

Supporting Information

Metal-Free Highly Chemo-Selective Bisphosphorylation and Deoxyphosphorylation of Carboxylic Acids

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General information

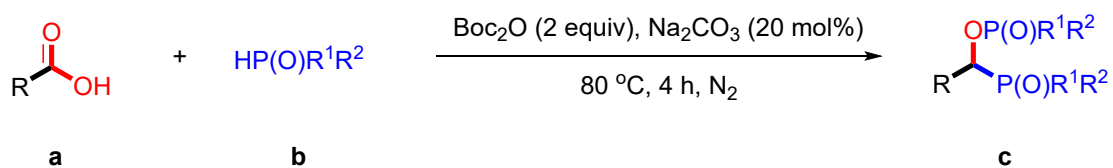
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1. General Information.

All experiments were carried out under a N₂ atmosphere using standard Schlenk techniques or in a dry glovebox. All heating (heating module) and stirring were conducted on the IKA (Model: RCT basic). Solvents were dried over Na metal or CaH₂, and were distilled under nitrogen prior to use. Reagents were of analytical grade, obtained from commercial suppliers and used without further purification. Flash column chromatography was performed by 200-300 mesh silica gel. The reactions were monitored by GC and GC-MS, GC-MS results were recorded on GC-MS QP2010, and GC analysis was performed on GC 2014. The ¹H, ¹³C and ³¹P NMR spectra were recorded on a Bruker ADVANCE III spectrometer at 400 MHz, 100 MHz and 162 MHz respectively, and chemical shifts were reported in parts per million (ppm). The following abbreviations were used to explain the multiplicities: s (singlet), d (doublet), dd (doublet of doublets), t (triplet), q (quartet), m (multiple), td (triplet of doublets). Melting points were measured using a melting point instrument and are uncorrected. High-resolution mass spectra (HRMS) were recorded on a TOF instrument by the ESI technique. All solvents, reagents and acids were purchased from Meryer, Energy Chemical and Aladdin. The electron spray ionization (ESI) method was used as the ionization method for the HRMS measurement, and the mass analyzer type is TOF for ESI. The authors thank the analytical and testing center of Hainan University for the great assistance.

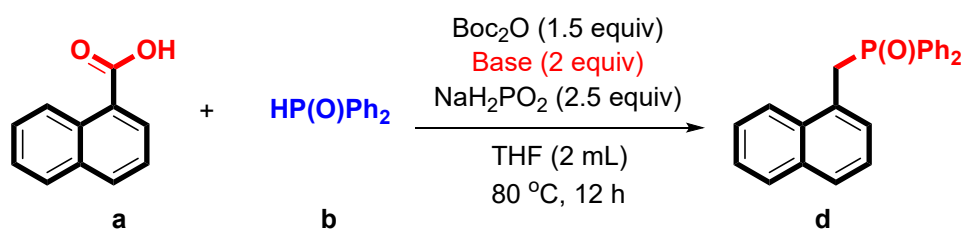
2. General procedure of bisphosphorylation.



Procedure A: To a 25 mL Schlenk tube, carboxylic acid (**a**, 0.2 mmol), P(O)H compound (**b**, 2 equiv, 0.4 mmol), Na₂CO₃ (4.2 mg, 20 mol%) and Boc₂O (92 μL, 2 equiv, 0.4 mmol) were added under N₂ atmosphere and stirred at 80 °C for 4 h. After being cooled to the room temperature, the analytically pure products were obtained by flash column chromatography.

3. Condition optimization of deoxyphosphorylation.

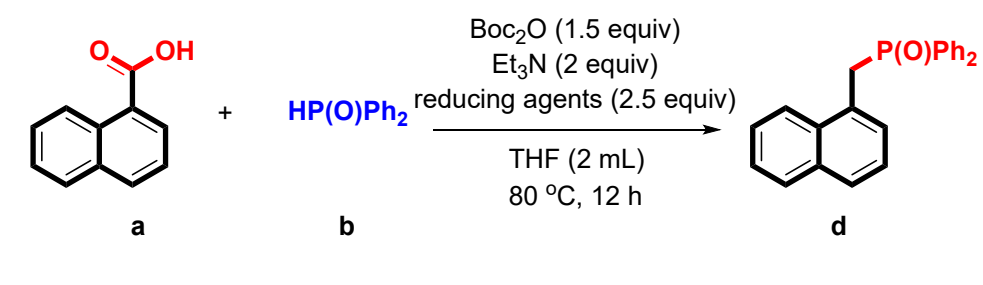
Table 1. Optimization of base^a.



Entry	Base	Yield/% ^b
1	K ₂ CO ₃	10
2	Na ₂ CO ₃	20
3	NaHCO ₃	7
4	NaOH	13
5	CH ₃ ONa	14
6	^t BuONa	14
7	Li ₂ CO ₃	19
8	Et ₃ N	36
9	ⁿ Pr ₃ N	18
10	DMF	N.D.

^aReaction conditions: **a** (34.4 mg, 0.2 mmol), **b** (80.9 mg, 0.4 mmol, 2 equiv), NaH₂PO₂ (44.0 mg, 0.5 mmol, 2.5 equiv), base (0.4 mmol) and Boc₂O (69 μL, 0.3 mmol, 1.5 equiv) were added into a 25 mL Schlenk tube and heated at 80 °C for 12 h. ^bGC yield using dodecane as an internal standard.

Table 2. Screening of reducing agents^a.

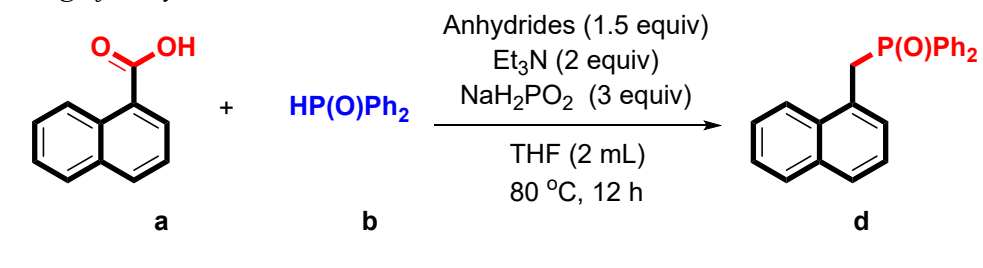


Entry	Reducing agents	Yield/% ^b
1	NaH ₂ PO ₂	36
2	HCOONa	32
3	Zn	26
4	Cu	17
5	Fe	13
6	NaH ₂ PO ₂	56 ^c
7	NaH ₂ PO ₂	57 ^d

^aReaction conditions: **a** (34.4 mg, 0.2 mmol), **b** (80.9 mg, 0.4 mmol, 2 equiv), Et₃N (56 μL, 0.4 mmol, 2 equiv), reducing agents (0.5 mmol, 2.5 equiv) and Boc₂O (69 μL, 0.3 mmol, 1.5 equiv) were added into a 25 mL Schlenk tube and heated at 80 °C for 12 h. ^bGC yield using dodecane as an internal standard.

^cNaH₂PO₂ (52.8 mg, 0.6 mmol, 3 equiv). ^dNaH₂PO₂ (61.6 mg, 0.7 mmol, 3.5 equiv).

Table 3. Screening of anhydrides^a.



Entry	Anhydrides	Yield/% ^b
1	Boc ₂ O	36
2	Piv ₂ O	32
3	Ac ₂ O	26

^aReaction conditions: **a** (34.4 mg, 0.2 mmol), **b** (80.9 mg, 0.4 mmol, 2equiv), Et₃N (56 μL, 0.4 mmol, 2 equiv), NaH₂PO₂ (52.8 mg, 0.6 mmol, 3 equiv) and anhydride (0.3 mmol, 1.5 equiv) were added into a 25 mL Schlenk tube and heated at 80 °C for 12 h. ^bGC yield using dodecane as an internal standard.

Table 4. Screening of solvents^a.

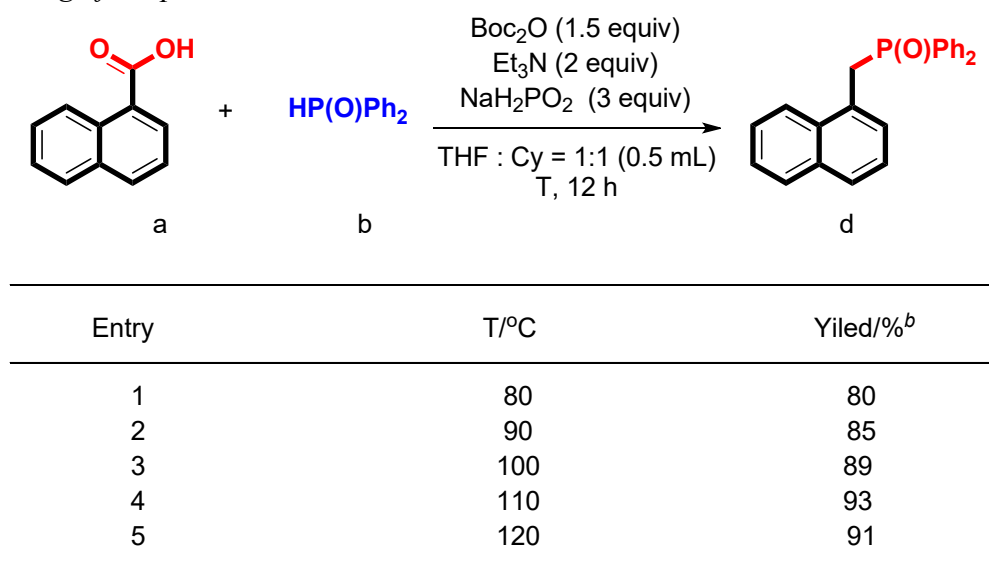
O=C(O)c1ccc2ccccc2c1 (a) + HP(=O)(c1ccccc1)c2ccccc2 (b)

 $\xrightarrow[\text{80 } ^\circ\text{C, 12 h}]{\text{Boc}_2\text{O (1.5 equiv), Et}_3\text{N (2 equiv), NaH}_2\text{PO}_2 \text{ (3 equiv), Solvents}}$
O=P(c1ccccc1)Cc2ccc3ccccc3c2 (d)

Entry	Solvents	Yield/% ^b
1	THF (2 mL)	56
2	Dioxane (2 mL)	26
3	DMF (2 mL)	48
4	Cy (2 mL)	58
5	MeCN (2 mL)	32
6	Et ₂ O (2 mL)	33
7	<i>i</i> PrOH (2 mL)	27
8	THF (1 mL)	60
9	THF (3 mL)	52
10	Cy (1 mL)	62
11	Cy (3 mL)	45
12	THF : Cy (1 mL)	71
13	THF : Cy (0.5 mL)	80

^aReaction conditions: **a** (34.4 mg, 0.2 mmol), **b** (80.9 mg, 0.4 mmol, 2 equiv), Et₃N (56 μL, 0.4 mmol, 2equiv), NaH₂PO₂ (52.8 mg, 0.6 mmol, 3 equiv) and Boc₂O (69 μL, 0.3 mmol, 1.5 equiv) were added into a 25 mL Schlenk tube and heated at 80 °C for 12 h. ^bGC yield using dodecane as an internal standard.

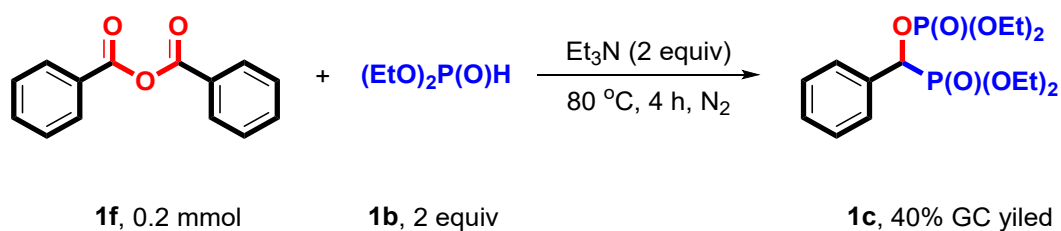
Table 5. Screening of temperature^a.



^aReaction conditions: **a** (34.4 mg, 0.2 mmol), **b** (80.9 mg, 0.4 mmol, 2equiv), Et₃N (56 μL, 0.4 mmol, 2equiv), NaH₂PO₂ (52.8 mg, 0.6 mmol, 3 equiv) and Boc₂O (69 μL, 0.3 mmol, 1.5 equiv) were added into a 25 mL Schlenk tube and heated at 80 °C for 12 h. ^bGC yield using dodecane as an internal standard.

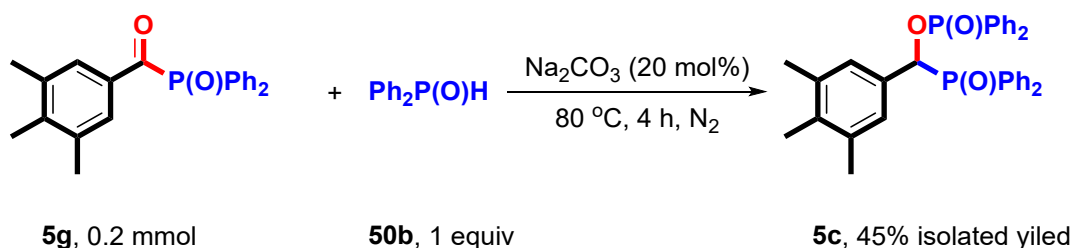
4. Mechanistic studies.

Scheme 1. The bisphosphorylation of benzoic anhydride.



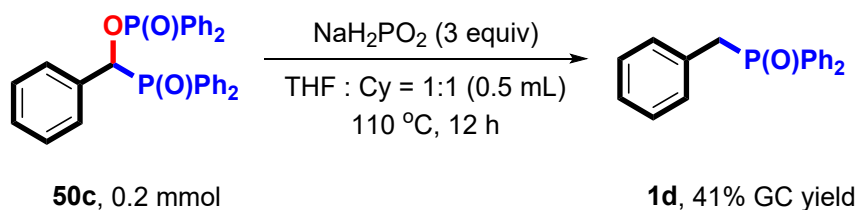
Procedure B: **1f** (45.2 mg, 0.2 mmol), **1b** (52 μ L, 0.4 mmol, 2equiv) and Et₃N (56 μ L, 2 equiv) were added into a 25 mL Schlenk tube and heated at 80 °C for 4 hours. **1c** was detected by GC in 40% yield.

Scheme 2. The addition and isomerization of acyl phosphoryl compound with P(O)H compound.



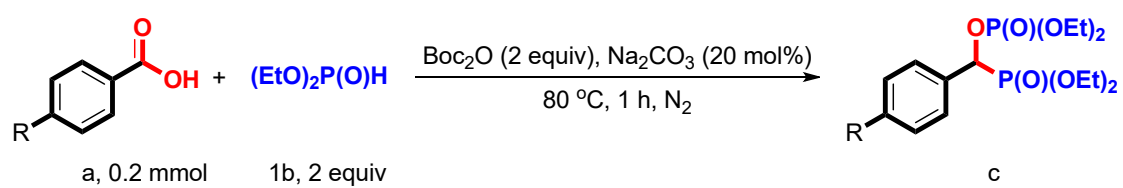
Procedure C: **5g** (69.6 mg, 0.2 mmol), **50b** (40.5 mg, 0.2 mmol, 1 equiv) and Na₂CO₃ (4.2 mg, 0.04 mmol, 20 mol%) were added into a 25 mL Schlenk tube under N₂ and heated at 80 °C for 4 hours. **5c** were purified by column chromatography.

Scheme 3. The reduction reaction of bisphosphorylation compound.



Procedure D: **50c** (101.6 mg, 0.2 mmol) and NaH₂PO₂ (52.8 mg, 0.6 mmol, 3 equiv) were added into a 25 mL Schlenk tube under N₂ and heated at 110 °C for 12 hours. **1d** was observed by GC.

Scheme 4. The experiment of Hammett analysis ^a.



Entry	-R	Yield/% ^a
1	-OMe	33
2	-Me	32
3	-H	30
4	-F	30
5	-Cl	29
6	-Br	23
7	-CF ₃	22
8	-NO ₂	20

^aReaction condition: To a 25 mL Schlenk tube, carboxylic acid (**a**, 0.2 mmol), P(O)H compound (**b**, 2 equiv, 0.4 mmol), Na₂CO₃ (4.2 mg, 20 mol%) and Boc₂O (92 μL, 2 equiv, 0.4 mmol) were added under N₂ atmosphere and stirred in 80 °C for 1 hour. ^bGC yield using dodecane as an internal standard.

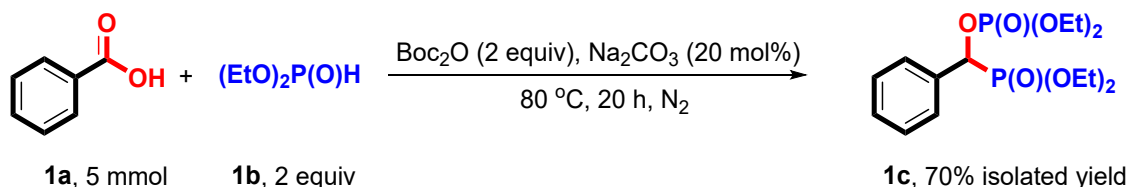
5. Application experiments.

Scheme 5. The reducing reaction of bisphosphorylation compound by silane.



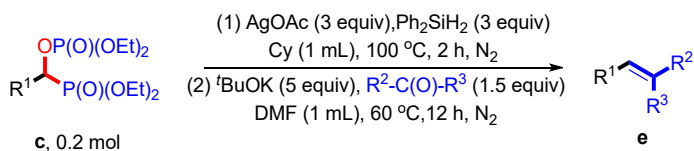
Procedure E: To a 25 mL Schlenk tube were added **1c** (76.0 mg, 0.2 mmol), AgOAc (100.2 mg, 0.6 mmol, 3 equiv), Ph₂SiH₂ (111 μL, 0.6 mmol, 3 equiv) and Cy (1 mL). the mixture was stirred at 100 °C for 2 hours. The reducing product was obtained by column chromatography.

Scheme 6. The gram scale reaction.



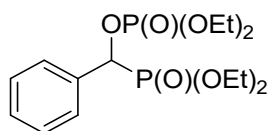
Procedure F: Under N₂ atmosphere, to a 250 mL three-necked flask was added **1a** (610.0 mg, 5 mmol), Na₂CO₃ (106.0 mg, 1 mmol, 20 mol%), Boc₂O (2.3 mL, 10 mmol, 2 equiv) and (EtO)₂P(O)H (1.3 mL, 10 mmol, 2 equiv). The mixture was stirred at 80 °C for 20 hours. **1c** was obtained by column chromatography in 70% (1.33 g) isolated yield.

Scheme 7. The preparation of olefin in one pot.



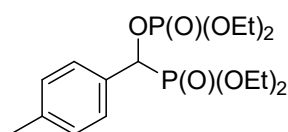
Procedure G: The compound **c** was obtained like the procedure A, to a 25 mL Schlenk tube, **c** (0.2 mmol), AgOAc (0.6 mmol, 100.2 mg, 3 equiv), Ph₂SiH₂ (0.6 mmol, 111 μL, 3 equiv) and cyclohexane (1 mL) were added and stirred at 100 °C for 2 hours. After cool to room temperature, ^tBuOK (1.0 mmol, 112.0 mg, 5 equiv) and aldehyde or ketone (0.3 mmol, 1.5 equiv) were added under N₂ atmosphere and heated at 60 °C for 12 hours. The desired products were obtained by column chromatography.

6. NMR Spectra



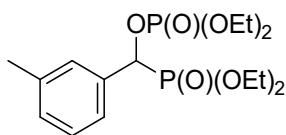
(diethoxyphosphoryl)(phenyl)methyl diethyl phosphate (1c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (72.5 mg, 95% yield). ^1H NMR (400 MHz): δ 7.53-7.51 (m, 2H), 7.40-7.32 (m, 3H), 5.55 (dd, $J = 13.6, 10.4$ Hz, 1H), 4.18-3.82 (m, 8H), 1.29-1.25 (m, 6H), 1.22 (td, $J = 6.8, 6.8$ Hz, 3H) 1.12 (td, $J = 6.8, 0.8$ Hz, 3H); ^{13}C NMR (100 MHz): δ 133.4, 128.9 (d, $J_{\text{C-P}} = 2.5$ Hz), 128.2 (d, $J_{\text{C-P}} = 1.6$ Hz), 127.9 (d, $J_{\text{C-P}} = 5.8$ Hz), 74.5 (dd, $J_{\text{C-P}} = 171.1, 6.9$ Hz), 63.9 (dd, $J_{\text{C-P}} = 21.0, 5.7$ Hz), 63.3 (dd, $J_{\text{C-P}} = 7.1, 2.5$ Hz), 16.1 (dd, $J_{\text{C-P}} = 11.1, 5.7$ Hz), 15.7 (dd, $J_{\text{C-P}} = 12.5, 7.2$ Hz); ^{31}P NMR (162 MHz): δ 16.70 (d, $J = 34.8$ Hz), -1.15 (d, $J = 35.0$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{15}\text{H}_{26}\text{O}_7\text{P}_2$: 381.1227; found: 381.1227.



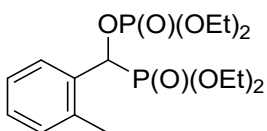
(diethoxyphosphoryl)(p-tolyl)methyl diethyl phosphate (2c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (72.0 mg, 91% yield). ^1H NMR (400 MHz): δ 7.40 (dd, $J = 8.0, 2.0$ Hz, 2H), 7.18 (d, $J = 8.0$ Hz, 2H) 5.51 (dd, $J = 13.2, 10.4$ Hz, 1H), 4.18-3.84 (m, 8H), 2.35 (d, $J = 1.2$ Hz, 3H), 1.30 (td, $J = 7.2, 7.2$ Hz, 3H), 1.27 (td, $J = 6.8, 1.2$ Hz, 3H), 1.22 (td, $J = 7.2, 7.2$ Hz, 3H) 1.13 (td, $J = 6.8, 1.2$ Hz, 3H); ^{13}C NMR (100 MHz): δ 138.8 (d, $J_{\text{C-P}} = 2.7$ Hz), 129.0 (d, $J_{\text{C-P}} = 1.6$ Hz), 127.9 (d, $J_{\text{C-P}} = 1.6$ Hz), 127.8 (d, $J_{\text{C-P}} = 6.0$ Hz), 74.5 (dd, $J_{\text{C-P}} = 172.0, 6.9$ Hz), 63.9 (dd, $J_{\text{C-P}} = 20.1, 5.7$ Hz), 63.3 (d, $J_{\text{C-P}} = 6.7$ Hz), 21.1, 16.2 (dd, $J_{\text{C-P}} = 10.9, 5.8$ Hz), 15.7 (dd, $J_{\text{C-P}} = 12.4, 7.2$ Hz); ^{31}P NMR (162 MHz): δ 16.9 (d, $J = 35.3$ Hz), -1.15 (d, $J = 35.3$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{16}\text{H}_{28}\text{O}_7\text{P}_2$: 385.1383; found: 381.1382.



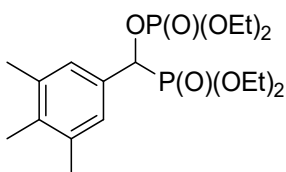
(diethoxyphosphoryl)(m-tolyl)methyl diethyl phosphate (3c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (71.8 mg, 91% yield). ¹H NMR (400 MHz): δ 7.32-7.15 (m, 3H), 7.16 (d, *J* = 7.2 Hz, 1H), 5.51 (dd, *J* = 13.6, 10.8 Hz, 1H), 4.18-3.85 (m, 8H), 2.36 (s, 3H), 1.30 (td, *J* = 7.2, 7.2 Hz, 3H), 1.27 (td, *J* = 6.8, 1.2 Hz, 3H), 1.23 (td, *J* = 7.2, 7.2 Hz, 3H), 1.13 (td, *J* = 7.2, 1.2 Hz, 3H); ¹³C NMR (100 MHz): δ 137.9, 133.3, 129.6 (d, *J*_{C-P} = 2.4 Hz), 128.5 (d, *J*_{C-P} = 6.0 Hz), 128.1, 124.9 (d, *J*_{C-P} = 5.9 Hz), 74.6 (dd, *J*_{C-P} = 171.1, 6.9 Hz), 63.9 (dd, *J*_{C-P} = 20.2, 5.7 Hz), 63.3 (dd, *J*_{C-P} = 6.7, 5.1 Hz), 21.2, 16.2 (dd, *J*_{C-P} = 11.2, 5.7 Hz), 15.7 (dd, *J*_{C-P} = 13.2, 7.2 Hz); ³¹P NMR (162 MHz): δ 16.82 (d, *J* = 35.0 Hz), -1.17 (d, *J* = 5.0 Hz); HRMS (ESI) *m/z*: [M+H]⁺ calcd. for C₁₆H₂₈O₇P₂: 385.1383; found: 381.1385.



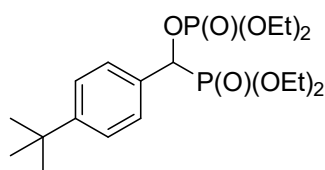
(diethoxyphosphoryl)(o-tolyl)methyl diethyl phosphate (4c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (76.4 mg, 97% yield). ¹H NMR (400 MHz): δ 7.65-7.64 (m, 1H), 7.27-7.21 (m, 2H), 7.18-7.16 (m, 1H), 5.83 (dd, *J* = 13.6, 10.4 Hz, 1H), 4.16-3.78 (m, 8H), 2.45 (s, 3H), 1.31 (td, *J* = 7.2, 7.2 Hz, 3H), 1.26 (td, *J* = 7.2, 0.8 Hz, 3H), 1.20 (td, *J* = 7.2, 7.2 Hz, 3H), 1.11 (td, *J* = 7.2, 0.8 Hz, 3H); ¹³C NMR (100 MHz): δ 136.1 (d, *J*_{C-P} = 7.3 Hz), 131.9, 130.1, 128.6 (d, *J*_{C-P} = 2.6 Hz), 128.1 (d, *J*_{C-P} = 4.0 Hz), 125.8 (d, *J*_{C-P} = 2.5 Hz), 70.7 (dd, *J*_{C-P} = 173.0, 6.8 Hz), 63.8 (dd, *J*_{C-P} = 22.7, 5.6 Hz), 63.2 (dd, *J*_{C-P} = 7.8, 7.8 Hz), 19.3, 16.1 (dd, *J*_{C-P} = 17.1, 5.8 Hz), 15.6 (dd, *J*_{C-P} = 12.0, 7.2 Hz); ³¹P NMR (162 MHz): δ 17.40 (d, *J* = 36.9 Hz), -0.98 (d, *J* = 36.9 Hz); HRMS (ESI) *m/z*: [M+H]⁺ calcd. for C₁₅H₂₆O₇P₂: 385.1383; found: 385.1385.



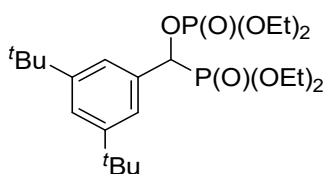
(diethoxyphosphoryl)(3,4,5-trimethylphenyl)methyl diethyl phosphate (5c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light white solid (72.6 mg, 86% yield) ^1H NMR (400 MHz): δ 7.06 (b, 2H), 5.38 (dd, $J = 13.2, 10.8$ Hz, 1H), 4.08-3.80 (m, 8H), 2.20 (s, 6H), 2.08 (s, 3H), 1.24 (td, $J = 7.2, 7.2$ Hz, 3H), 1.21 (td, $J = 6.8, 1.2$ Hz, 3H), 1.17 (td, $J = 7.2, 7.2$ Hz, 3H), 1.07 (td, $J = 7.2, 1.2$ Hz, 3H); ^{13}C NMR (100 MHz): δ 136.1, 135.8, 129.7, 126.8 (d, $J_{\text{C-P}} = 5.9$ Hz), 74.5 (dd, $J_{\text{C-P}} = 171.7, 6.8$ Hz), 63.7 (d, $J_{\text{C-P}} = 18.3, 5.6$ Hz), 63.1 (dd, $J_{\text{C-P}} = 7.2, 7.2$ Hz), 20.3, 16.1 (d, $J_{\text{C-P}} = 10.9, 5.7$ Hz), 15.6 (dd, $J_{\text{C-P}} = 14.9, 7.2$ Hz), 15.0; ^{31}P NMR (162 MHz): δ 17.08 (d, $J = 35.5$ Hz), -1.19 (d, $J = 35.3$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{18}\text{H}_{32}\text{O}_7\text{P}_2$: 423.1696; found: 423.1695.



(4-(tert-butyl)phenyl)(diethoxyphosphoryl)methyl diethyl phosphate (6c).

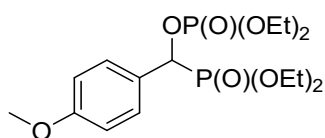
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (84.6 mg, 97% yield). ^1H NMR (400 MHz): δ 7.45-7.38 (m, 4H), 5.52 (dd, $J = 13.2, 10.0$ Hz, 1H), 4.17-3.82 (m, 8H), 1.31 (s, 9H), 1.30 (td, $J = 7.2, 7.2$ Hz, 3H), 1.26 (td, $J = 7.2, 1.2$ Hz, 3H), 1.22 (td, $J = 7.2, 7.2$ Hz, 3H), 1.09 (td, $J = 7.2, 0.8$ Hz, 3H); ^{13}C NMR (100 MHz): δ 151.9 (d, $J_{\text{C-P}} = 2.6$ Hz), 130.3, 127.6 (d, $J_{\text{C-P}} = 6.1$ Hz), 125.2, 74.4 (dd, $J_{\text{C-P}} = 172.0, 6.9$ Hz), 63.8 (d, $J_{\text{C-P}} = 18.3, 5.7$ Hz), 63.2 (dd, $J_{\text{C-P}} = 6.4, 6.4$ Hz), 34.4, 31.1, 16.1 (dd, $J_{\text{C-P}} = 14.5, 5.7$ Hz), 15.6 (dd, $J_{\text{C-P}} = 15.4, 7.2$ Hz); ^{31}P NMR (162 MHz): δ 16.97 (d, $J = 36.3$ Hz), -1.18 (d, $J = 36.1$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{19}\text{H}_{34}\text{O}_7\text{P}_2$: 437.1853; found: 437.1852.



(3,5-di-tert-butylphenyl)(diethoxyphosphoryl)methyl diethyl phosphate (7c).

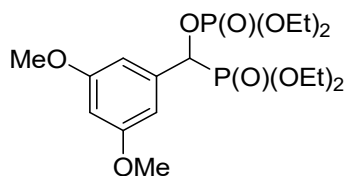
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (90.5 mg, 93% yield). ^1H NMR (400 MHz): δ 7.39-7.34 (m, 3H), 5.55 (dd, $J_{\text{C-P}} = 12.8, 10.8$ Hz, 1H), 4.18-3.83 (m, 8H), 1.32 (s, 18H), 1.29 (td, $J = 7.2, 7.2$ Hz, 3H), 1.25 (td, $J = 7.2, 7.2$ Hz, 3H), 1.20 (td, J

= 7.2, 7.2 Hz), 1.08 (td, $J = 7.2, 7.2$ Hz, 3H); ^{13}C NMR (100 MHz): δ 150.4, 132.3, 122.4, 122.1 (d, $J_{\text{C-P}} = 5.9$ Hz), 75.1 (dd, $J_{\text{C-P}} = 171.3, 6.9$ Hz), 63.6 (dd, $J_{\text{C-P}} = 22.2, 5.5$ Hz), 63.0 (dd, $J_{\text{C-P}} = 7.8, 7.8$ Hz), 34.5, 31.1, 16.0 (dd, $J_{\text{C-P}} = 17.4, 12.8$ Hz), 15.5 (dd, $J_{\text{C-P}} = 11.1, 7.3$ Hz); ^{31}P NMR (162 MHz): δ 16.98 (d, $J = 35.8$ Hz), -1.19 (d, $J = 35.6$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{32}\text{H}_{42}\text{O}_7\text{P}_2$: 493.2479; found: 493.2480.



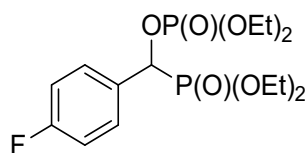
(diethoxyphosphoryl)(4-methoxyphenyl)methyl diethyl phosphate (8c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (75.4 mg, 92% yield). ^1H NMR (400 MHz): δ 7.46 (dd, $J = 8.4, 1.6$ Hz, 2H), 6.91 (d, $J = 7.2$ Hz, 2H), 5.49 (dd, $J = 13.2, 10.4$ Hz, 1H), 4.18-3.81 (m, 8H), 3.81 (s, 3H), 1.31 (td, $J = 7.2, 7.2$ Hz, 3H), 1.28 (td, $J = 7.2, 0.8$ Hz, 3H), 1.21 (td, $J = 7.2, 7.2$ Hz, 3H), 1.13 (td, $J = 7.2, 1.2$ Hz, 3H); ^{13}C NMR (100 MHz): δ 160.0 (d, $J_{\text{C-P}} = 2.3$ Hz), 129.4 (d, $J_{\text{C-P}} = 6.3$ Hz), 125.3, 113.6, 74.1 (dd, $J_{\text{C-P}} = 173.9, 7.0$ Hz), 63.7 (dd, $J_{\text{C-P}} = 21.0, 5.7$ Hz), 63.2 (db, $J_{\text{C-P}} = 6.2$ Hz), 16.1 (dd, $J_{\text{C-P}} = 11.8, 5.8$ Hz), 15.6 (dd, $J_{\text{C-P}} = 13.2, 7.2$ Hz); ^{31}P NMR (162 MHz): δ 16.98 (d, $J = 36.6$ Hz), -1.14 (d, $J = 36.6$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{16}\text{H}_{28}\text{O}_8\text{P}_2$: 411.1332; found: 411.1331.



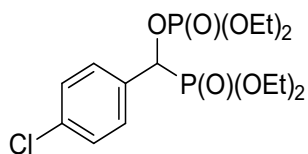
(diethoxyphosphoryl)(3,5-dimethoxyphenyl)methyl diethyl phosphate (9c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (77.5 mg, 88% yield). ^1H NMR (400 MHz): δ 6.67 (b, 3H), 6.44 (b, 3H), 5.49 (dd, $J = 13.2, 10.8$ Hz, 1H), 4.20-3.88 (m, 8H), 3.79 (s, 6H), 1.33-1.24 (m, 9H), 1.18 (td, $J = 7.2, 7.2$ Hz, 3H); ^{13}C NMR (100 MHz): δ 160.4 (d, $J_{\text{C-P}} = 1.8$ Hz), 135.4, 105.6 (d, $J_{\text{C-P}} = 5.7$ Hz), 101.0 (d, $J_{\text{C-P}} = 2.5$ Hz), 74.5 (dd, $J_{\text{C-P}} = 170.5, 6.9$ Hz), 63.8 (dd, $J_{\text{C-P}} = 17.5, 5.7$ Hz), 63.3 (dd, $J_{\text{C-P}} = 7.0, 4.1$ Hz), 55.1, 16.1, (dd, $J_{\text{C-P}} = 9.4, 5.7$ Hz), 15.6 (dd, $J_{\text{C-P}} = 8.6, 7.3$ Hz); ^{31}P NMR (162 MHz): δ 16.5 (d, $J = 34.0$ Hz), -1.20 (d, $J = 34.0$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{17}\text{H}_{30}\text{O}_9\text{P}_2$: 441.1438; found: 441.1439.



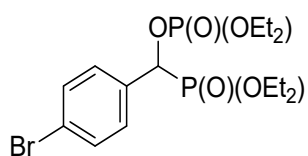
(diethoxyphosphoryl)(4-fluorophenyl)methyl diethyl phosphate (10c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (70.0 mg, 88% yield). ¹H NMR (400 MHz): δ 7.54-7.50 (m, 2H), 7.08 (dt, *J* = 8.8, 8.8 Hz, 2H), 5.53 (dd, *J* = 13.2 Hz, 10.4 Hz, 1H), 4.19-3.86 (m, 8H), 1.32-1.27 (m, 6H), 1.23 (td, *J* = 7.2, 7.2 Hz, 3H), 1.14 (td, *J* = 7.2, 0.8 Hz, 3H); ¹³C NMR (100 MHz): δ 163.0 (dd, *J*_{C-F} = 246.6 Hz, *J*_{C-P} = 2.7 Hz), 129.8 (dd, *J*_{C-F} = 35.2 Hz, *J*_{C-P} = 6.2 Hz), 129.5 (b), 115.3 (dd, *J*_{C-F} = 21.6 Hz, *J*_{C-P} = 1.3 Hz), 73.8 (dd, *J*_{C-P} = 172.6, 6.9 Hz), 64.0 (dd, *J*_{C-P} = 22.2, 5.8 Hz), 63.4 (dd, *J*_{C-P} = 6.6, 6.6 Hz), 16.2 (dd, *J*_{C-P} = 10.3, 5.7 Hz), 15.8 (dd, *J*_{C-P} = 11.7, 7.2 Hz); ³¹P NMR (162 MHz): δ 16.47 (dd, *J*_{P-P} = 35.2 Hz, *J*_{F-P} = 4.4 Hz), -1.094 (d, *J* = 35.3 Hz); HRMS (ESI) *m/z*: [M+H]⁺ calcd. for C₁₅H₂₅O₇FP₂: 399.1132; found: 399.1127.



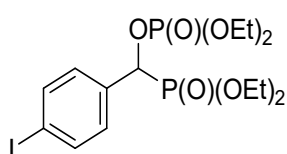
(4-chlorophenyl)(diethoxyphosphoryl)methyl diethyl phosphate (11c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (77.0 mg, 93% yield). ¹H NMR (400 MHz): δ 7.48-7.45 (dd, *J* = 8.4, 2.0 Hz, 2H), 7.36 (d, 8.4 Hz, 1H), 5.53 (dd, *J* = 13.6, 10.4 Hz, 1H), 4.20-3.89 (m, 8H), 1.30 (td, *J* = 7.2, 7.2 Hz, 3H), 1.30 (td, *J* = 7.2, 1.2 Hz, 3H), 1.25 (td, *J* = 7.2, 7.2 Hz, 3H), 1.16 (td, *J* = 7.2, 0.8 Hz, 3H); ¹³C NMR (100 MHz): δ 134.8 (d, *J*_{C-P} = 3.3 Hz), 132.2, 129.1 (d, *J*_{C-P} = 5.8 Hz), 128.5 (d, *J*_{C-P} = 1.6 Hz), 73.8 (dd, *J*_{C-P} = 171.5, 6.9 Hz), 64.0 (dd, *J*_{C-P} = 21.0, 5.8 Hz), 63.4 (dd, *J*_{C-P} = 7.3, 7.3 Hz), 16.2 (dd, *J*_{C-P} = 8.0, 5.8 Hz), 15.7 (dd, *J*_{C-P} = 9.4, 7.2 Hz); ³¹P NMR (162 MHz): δ 16.17 (d, *J* = 34.3 Hz), -1.13 (d, *J* = 34.3 Hz); HRMS (ESI) *m/z*: [M+H]⁺ calcd. for C₁₅H₂₅O₇ClP₂: 415.0837; found: 415.0837.



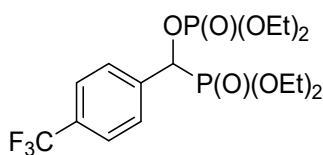
(4-bromophenyl)(phosphoryl)methyl phosphenite compound with ethoxyethane (12c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (72.4 mg, 85% yield). ^1H NMR (400 MHz): δ 7.52 (d, J = 8.4 Hz, 2H), 7.39 (d, J = 8.4, 2.0 Hz, 2H), 5.51 (dd, J = 14.0, 10.8 Hz, 1H), 4.20-3.86 (m, 8H), 1.30 (td, J = 7.2, 7.2 Hz, 3H), 1.29 (td, J = 7.2, 7.2 Hz, 3H), 1.25 (td, J = 7.2, 7.2 Hz, 3H), 1.17 (td, J = 7.2, 0.8 Hz, 3H); ^{13}C NMR (100 MHz): δ 132.7, 131.4 (d, $J_{\text{C-P}}$ = 1.6 Hz), 129.4 (d, $J_{\text{C-P}}$ = 5.7 Hz), 123.0 (d, $J_{\text{C-P}}$ = 3.4 Hz), 73.8 (dd, $J_{\text{C-P}}$ = 171.3, 6.9 Hz), 64.0 (dd, $J_{\text{C-P}}$ = 20.6, 5.8 Hz), 63.4 (dd, $J_{\text{C-P}}$ = 7.4, 7.4 Hz), 16.2 (dd, $J_{\text{C-P}}$ = 7.7, 5.9 Hz), 15.8 (dd, $J_{\text{C-P}}$ = 8.7, 7.4 Hz); ^{31}P NMR (162 MHz): δ 16.00 (d, J = 34.0 Hz), -1.13 (d, J = 34.2 Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{15}\text{H}_{25}\text{O}_7\text{BrP}_2$: 459.0332; found: 459.0334.



(diethoxyphosphoryl)(4-iodophenyl)methyl diethyl phosphate (13c).

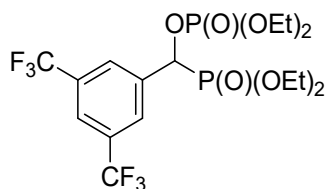
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (84.0 mg, 83% yield). ^1H NMR (400 MHz): δ 7.72 (d, J = 8.0 Hz, 2H), 7.25 (dd, J = 8.4, 2.0 Hz, 2H), 5.49 (dd, J = 13.6, 10.4 Hz, 1H), 4.19-3.86 (m, 8H), 1.31-1.28 (m, 6H), 1.25 (td, J = 7.2, 7.2 Hz, 3H), 1.16 (td, J = 7.2, 0.8 Hz, 3H); ^{13}C NMR (100 MHz): δ 137.4 (d, $J_{\text{C-P}}$ = 1.8 Hz), 133.4, 129.5 (d, $J_{\text{C-P}}$ = 5.7 Hz), 94.8 (d, $J_{\text{C-P}}$ = 3.6 Hz), 74.0 (dd, $J_{\text{C-P}}$ = 171.0, 6.9 Hz), 64.1 (dd, $J_{\text{C-P}}$ = 19.8, 5.8 Hz), 63.5 (dd, $J_{\text{C-P}}$ = 7.7, 7.7 Hz), 16.3 (dd, $J_{\text{C-P}}$ = 7.3, 6.1 Hz), 15.9 (dd, $J_{\text{C-P}}$ = 7.9, 7.9 Hz); ^{31}P NMR (162 MHz): δ 16.0 (d, J = 33.9 Hz), -1.12 (d, J = 32.4 Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{15}\text{H}_{25}\text{O}_7\text{IP}_2$: 507.0193; found: 507.0191.



(diethoxyphosphoryl)(4-(trifluoromethyl)phenyl)methyl diethyl phosphate (14c).

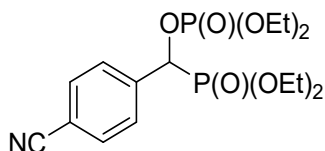
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (78.9 mg, 88% yield). ^1H NMR (400 MHz): δ 7.67-7.62 (m, 4H), 5.62 (dd, J = 14.0, 10.4 Hz, 1H), 4.19-3.91 (m, 8H), 1.30 (td, J = 7.2, 0.8 Hz, 3H), 1.29 (td, J = 7.2, 7.2 Hz, 3H), 1.27 (td, J = 7.2, 7.2 Hz, 3H), 1.17 (td, J = 7.2, 0.8 Hz, 3H); ^{13}C NMR (100 MHz): δ 137.9, 131.0 (dq, $J_{\text{C-F}}$ = 32.2, $J_{\text{C-P}}$ = 2.8 Hz), 128.1 (d, $J_{\text{C-P}}$ = 5.4 Hz), 125.3 (b), 123.8 (q, $J_{\text{C-F}}$ = 270.6 Hz), 74.0 (dd, $J_{\text{C-P}}$ = 169.6, 6.7

Hz), 64.3 (dd, $J_{C-P} = 20.8, 5.8$ Hz), 63.7 (dd, $J_{C-P} = 11.9, 7.1$ Hz), 16.3 (dd, $J_{C-P} = 6.3, 6.3$ Hz), 15.8 (dd, $J_{C-P} = 9.5, 7.1$ Hz); ^{31}P NMR (162 MHz): δ 15.80 (d, $J = 32.2$ Hz), -1.071 (d, $J = 32.7$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{16}\text{H}_{25}\text{F}_3\text{O}_7\text{P}_2$: 449.1100; found: 449.1101.



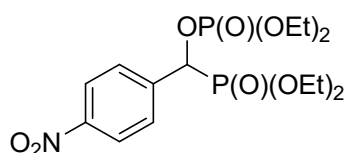
(3,5-bis(trifluoromethyl)phenyl)(diethoxyphosphoryl)methyl diethyl phosphate (15c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (82.6 mg, 80% yield). ^1H NMR (400 MHz): δ 7.96 (s, 2H), 7.87 (s, 1H), 5.70 (dd, $J = 14.4, 10.4$ Hz, 1H), 4.20-3.98 (m, 8H), 1.33 (td, $J = 6.8, 6.8$ Hz, 3H), 1.30 (td, $J = 7.2, 7.2$ Hz, 3H), 1.28 (td, $J = 7.2, 7.2$ Hz, 3H), 1.19 (td, $J = 7.2, 7.2$ Hz, 3H); ^{13}C NMR (100 MHz): δ 137.0, 131.7 (qd, $J_{C-F} = 50.1, J_{C-P} = 1.9$ Hz), 127.7 (b), 123.0 (q, $J_{C-F} = 270.1$ Hz), 122.5-122.4 (m), 73.4 (dd, $J_{C-P} = 169.7, 6.7$ Hz), 64.4 (dd, $J_{C-P} = 20.7, 5.9$ Hz), 63.9 (dd, $J_{C-P} = 6.4, 6.4$ Hz), 16.2 (db, $J_{C-P} = 5.6$ Hz), 15.7 (dd, $J_{C-P} = 14.5, 7.0$ Hz); ^{31}P NMR (162 MHz): δ 14.69 (d, $J = 32.4$ Hz), -1.07 (d, $J = 32.4$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{17}\text{H}_{24}\text{F}_6\text{O}_7\text{P}_2$: 517.0974; found: 517.0974.



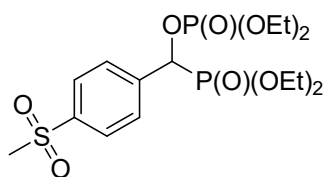
(4-cyanophenyl)(diethoxyphosphoryl)methyl diethyl phosphate (16c).

Like the **procedure A** but Et_3N (1equiv) instead of Na_2CO_3 (20 mol%). Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (45.4 mg, 56% yield). ^1H NMR (400 MHz): δ 7.69 (d, $J = 8.0$ Hz, 2H), 7.62 (dd, $J = 8.4, 1.6$ Hz, 2H), 5.61 (dd, $J = 14.8, 10.8$ Hz, 1H), 4.21-3.92 (m, 8H), 1.31 (td, $J = 7.2, 0.8$ Hz, 3H), 1.29 (td, $J = 7.2, 7.2$ Hz, 3H), 1.28 (td, $J = 7.2, 7.2$ Hz, 3H), 1.19 (td, $J = 7.8, 0.8$ Hz, 3H); ^{13}C NMR (100 MHz): δ 139.1, 131.9 (d, $J_{C-P} = 2.0$ Hz), 128.1 (d, $J_{C-P} = 5.3$ Hz), 118.2, 112.5 (d, $J_{C-P} = 2.8$ Hz), 73.8 (dd, $J_{C-P} = 168.8, 6.7$ Hz), 64.2 (dd, $J_{C-P} = 21.0, 5.9$ Hz), 63.6 (dd, $J_{C-P} = 13.1, 7.1$ Hz), 16.2 (dd, $J_{C-P} = 5.3, 5.3$ Hz), 15.8 (dd, $J_{C-P} = 6.6, 6.6$ Hz); ^{31}P NMR (162 MHz): δ 15.35 (d, $J = 32.4$ Hz), -1.04 (d, $J = 32.2$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{16}\text{H}_{25}\text{NO}_7\text{P}_2$: 437.1853; found: 437.1854.



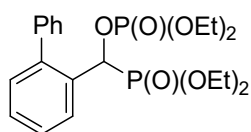
(diethoxyphosphoryl)(4-nitrophenyl)methyl diethyl phosphate (17c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (48.4 mg, 57% yield). ¹H NMR (400 MHz): δ 8.25 (d, *J* = 8.8 Hz, 2H), 7.69 (dd, *J* = 8.8, 2.0 Hz, 2H), 5.67 (dd, *J* = 14.8, 10.4 Hz, 1H), 4.23-3.93 (m, 8H), 1.32 (td, *J* = 7.2, 7.2 Hz, 3H), 1.31-1.27 (m, 6H), 1.20 (td, *J* = 7.2, 7.2 Hz, 3H); ¹³C NMR (100 MHz): δ 148.0 (d, *J*_{C-P} = 3.2 Hz), 141.1, 128.4 (d, *J*_{C-P} = 5.3 Hz), 123.4 (d, *J*_{C-P} = 2.1 Hz), 73.7 (dd, *J*_{C-P} = 168.3, 6.6 Hz), 64.4 (dd, *J*_{C-P} = 20.6, 5.9 Hz), 63.7 (dd, *J*_{C-P} = 12.5, 7.0 Hz), 16.3 (dd, *J*_{C-P} = 4.4, 4.4 Hz), 15.9 (dd, *J*_{C-P} = 6.9, 5.5 Hz); ³¹P NMR (162 MHz): δ 15.15 (d, *J* = 31.8 Hz), -1.04 (d, *J* = 31.9 Hz); HRMS (ESI) *m/z*: [M+H]⁺ calcd. for C₁₅H₂₅NO₉P₂: 426.1077; found: 426.1078.



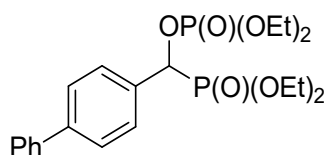
(diethoxyphosphoryl)(4-(methylsulfonyl)phenyl)methyl diethyl phosphate (18c).

Like the **procedure A** but Et₃N (1equiv) instead of Na₂CO₃ (20 mol%). Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (64.1 mg, 70% yield). ¹H NMR (400 MHz): δ 7.97 (d, *J* = 8.0 Hz, 2H), 7.71 (d, *J* = 6.8 Hz, 2H), 5.68 (dd, *J* = 14.8, 10.8 Hz, 1H), 4.19-3.97 (m, 8H), 3.07 (s, 3H), 1.34-1.28 (m, 9H), 1.20 (td, *J* = 7.2, 7.2 Hz, 3H); ¹³C NMR (100 MHz): δ 140.6 (d, *J*_{C-P} = 3.0 Hz), 139.9, 128.3 (d, *J*_{C-P} = 5.2 Hz), 127.3 (d, *J*_{C-P} = 1.3 Hz), 73.7 (dd, *J*_{C-P} = 168.4, 6.6 Hz), 64.3 (dd, *J*_{C-P} = 19.4, 5.9 Hz), 63.7 (dd, *J*_{C-P} = 18.6, 7.1 Hz), 44.3 16.2 (dd, *J*_{C-P} = 6.4, 6.4 Hz), 15.8 (dd, *J*_{C-P} = 6.3, 6.3 Hz); ³¹P NMR (162 MHz): δ 15.28 (d, *J* = 31.8 Hz), -1.17 (d, *J* = 31.9 Hz); HRMS (ESI) *m/z*: [M+H]⁺ calcd. for C₁₆H₂₈O₉P₂S: 459.1002; found: 459.1002.



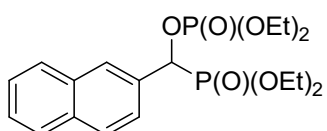
[1,1'-biphenyl]-2-yl(diethoxyphosphoryl)methyl diethyl phosphate (19c).

Like the **procedure A** but Et₃N (1equiv) instead of Na₂CO₃ (20 mol%). Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (79.3 mg, 87% yield). ¹H NMR (400 MHz): δ 7.83 (db, *J* = 7.2 Hz, 1H), 7.49-7.34 (m, 7H), 7.26 (db, *J* = 7.2 Hz, 1H), 5.74 (dd, *J* = 13.2, 10.0 Hz, 1H), 4.10-3.83 (m, 8H), 1.30-1.08 (m, 12H); ¹³C NMR (100 MHz): δ 142.1 (d, *J*_{C-P} = 8.2 Hz), 139.7, 131.2, 129.7, 129.4, 128.7 (d, *J*_{C-P} = 3.7 Hz), 128.6 (d, *J*_{C-P} = 2.4 Hz), 128.0, 127.3 (d, *J*_{C-P} = 2.4 Hz), 127.2, 70.9 (dd, *J*_{C-P} = 172.9, 7.3 Hz), 63.7 (dd, *J*_{C-P} = 8.5, 5.7 Hz), 63.1 (dd, *J*_{C-P} = 21.8, 7.0 Hz), 16.1 (dd, *J*_{C-P} = 14.1, 5.6 Hz), 15.5 (dd, *J*_{C-P} = 6.9, 5.2 Hz); ³¹P NMR (162 MHz): δ 17.1 (d, *J* = 38.39 Hz), -1.65 (d, *J* = 38.39 Hz); HRMS (ESI) *m/z*: [M+H]⁺ calcd. for C₂₁H₃₀O₇P₂: 457.1540; found: 457.1539.



[1,1'-biphenyl]-4-yl(diethoxyphosphoryl)methyl diethyl phosphate (20c).

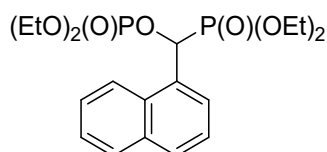
Like the **procedure A** but Et₃N (1equiv) instead of Na₂CO₃ (20 mol%). Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light white solid (65.7 mg, 72% yield) ¹H NMR (400 MHz): δ 7.63-7.57 (m, 6H), 7.45 (dd, *J* = 7.2, 7.2 Hz, 2H), 7.36 (dd, *J* = 7.2, 7.2 Hz, 1H), 5.60 (dd, *J* = 13.6, 10.8 Hz, 1H), 4.21-3.86 (m, 8H), 1.31 (td, *J* = 7.2, 7.2 Hz, 3H), 1.29 (td, *J* = 7.2, 7.2 Hz, 3H), 1.25 (td, *J* = 7.2, 7.2 Hz, 3H), 1.15 (td, *J* = 7.2, 7.2 Hz, 3H); ¹³C NMR (100 MHz): δ 141.7 (d, *J*_{C-P} = 2.7 Hz), 140.3, 132.6, 128.8, 128.4, 128.3, 127.5, 127.0, 74.5 (dd, *J*_{C-P} = 171.2, 6.8 Hz), 64.0 (dd, *J*_{C-P} = 18.9, 5.8 Hz), 63.5 (dd, *J*_{C-P} = 6.7, 6.7 Hz), 16.3 (dd, *J*_{C-P} = 10.2, 5.8 Hz), 15.8 (dd, *J*_{C-P} = 11.1, 7.2 Hz); ³¹P NMR (162 MHz): δ 16.70 (d, *J* = 35.0 Hz), -1.03 (d, *J* = 35.0 Hz); HRMS (ESI) *m/z*: [M+H]⁺ calcd. for C₂₁H₃₀O₇P₂: 457.1540; found: 457.1537.



(diethoxyphosphoryl)(naphthalen-2-yl)methyl diethyl phosphate (21c).

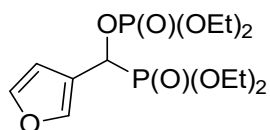
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (77.4 mg, 90% yield). ¹H NMR (400 MHz): δ 7.99 (b, 1H), 7.89-7.85 (m, 3H), 7.67 (db, *J* = 7.6 Hz, 1H), 7.54-7.50 (m, 2H), 5.73 (dd, *J* = 13.6, 10.8 Hz, 1H), 4.20-3.85 (m, 8H), 1.31 (tb, *J* =

7.2, 7.2 Hz, 3H), 1.29 (td, $J = 7.2, 0.8$ Hz, 3H), 1.23 (td, $J = 7.2, 0.8$ Hz, 3H), 1.09 (tb, $J = 7.2, 0.8$ Hz, 3H); ^{13}C NMR (100 MHz): δ 133.4 (d, $J_{\text{C-P}} = 1.6$ Hz), 132.8 (d, $J_{\text{C-P}} = 1.6$ Hz), 131.0, 128.1, 127.6, 127.6, 127.5, 126.6, 126.3, 125.1 (d, $J_{\text{C-P}} = 4.3$ Hz), 74.9 (dd, $J_{\text{C-P}} = 171.1, 6.8$ Hz), 64.0 (dd, $J_{\text{C-P}} = 22.7, 5.7$ Hz), 63.5 (dd, $J_{\text{C-P}} = 6.9, 4.4$ Hz), 16.3 (dd, $J_{\text{C-P}} = 10.3, 5.7$ Hz), 15.9 (dd, $J_{\text{C-P}} = 15.1, 7.1$ Hz); ^{31}P NMR (100 MHz): δ 16.69 (d, $J = 34.5$ Hz), -1.00 (d, $J = 34.5$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{19}\text{H}_{28}\text{O}_7\text{P}_2$: 431.1383; found: 431.1380.



(diethoxyphosphoryl)(naphthalen-1-yl)methyl diethyl phosphate (22c).

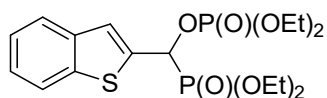
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (78.2 mg, 91% yield). ^1H NMR (400 MHz): δ 8.22 (d, $J = 8.0$ Hz, 1H), 7.87 (d, $J = 8.0$ Hz, 1H), 7.58-7.49 (m, 3H), 6.39 (dd, $J = 13.6, 11.2$ Hz, 1H), 4.19-3.73 (m, 8H), 1.29 (tb, $J = 7.2, 7.2$ Hz, 3H), 1.22 (tb, $J = 7.2, 1.2$ Hz, 3H), 1.05 (tb, $J = 7.2, 0.4$ Hz, 3H), 1.01 (tb, $J = 7.2, 0.8$ Hz, 3H); ^{13}C NMR (100 MHz): δ 133.5, 130.6 (d, $J_{\text{C-P}} = 5.4$ Hz), 129.6 (d, $J_{\text{C-P}} = 10.6$ Hz), 129.6, 128.5, 127.1, 126.4, 125.8, 125.0 (d, $J_{\text{C-P}} = 2.8$ Hz), 123.7, 73.4 (dd, $J_{\text{C-P}} = 177.5, 6.8$ Hz), 64.0 (dd, $J_{\text{C-P}} = 25.0, 5.6$ Hz), 63.5 (dd, $J_{\text{C-P}} = 6.9, 4.4$ Hz), 16.2 (dd, $J_{\text{C-P}} = 26.3, 5.8$ Hz), 15.7 (dd, $J_{\text{C-P}} = 17.4, 7.2$ Hz); ^{31}P NMR (162 MHz): δ 16.96 (d, $J = 34.67$ Hz), -1.04 (dd, $J = 34.83$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{19}\text{H}_{28}\text{O}_7\text{P}_2$: 431.1383; found: 431.1386.



(diethoxyphosphoryl)(thiophen-3-yl)methyl diethyl phosphate (23c).

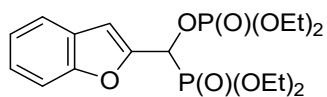
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (40.1 mg, 52% yield). ^1H NMR (400 MHz): δ 7.64 (b, 1H), 7.43 (b, 1H), 6.64 (b, 1H), 5.57 (dd, $J = 13.2, 10.0$ Hz, 1H), 4.24-3.90 (m, 8H), 1.34 (tb, $J_{\text{C-P}} = 7.2, 7.2$ Hz, 3H), 1.32 (tb, $J_{\text{C-P}} = 7.2, 0.8$ Hz, 3H), 1.28 (tb, $J_{\text{C-P}} = 7.2, 7.2$ Hz, 3H), 1.20 (tb, $J_{\text{C-P}} = 7.2, 1.2$ Hz, 3H); ^{13}C NMR (100 MHz): δ 143.4, 142.2 (d, $J_{\text{C-P}} = 11.3$ Hz), 118.7, 110.0 (d, $J_{\text{C-P}} = 3.3$ Hz), 67.2 (dd, $J_{\text{C-P}} = 178.4, 7.0$ Hz), 63.9 (dd, $J_{\text{C-P}} = 6.3, 3.4$ Hz), 63.4 (dd, $J_{\text{C-P}} = 6.3, 3.4$ Hz), 16.3 (dd, $J_{\text{C-P}} = 9.1, 5.9$ Hz), 15.8 (dd, $J_{\text{C-P}} = 14.1, 7.1$

Hz); ^{31}P NMR (162 MHz): δ 16.61 (d, $J = 34.2$ Hz), -1.09 (d, $J = 34.2$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{13}\text{H}_{24}\text{O}_8\text{P}_2$: 371.1019; found: 371.1018.



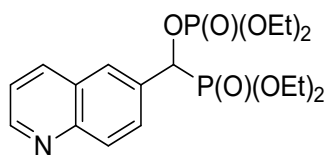
Benzo[b]thiophen-2-yl(diethoxyphosphoryl)methyl diethyl phosphate (24c).

Et_3N (1equiv) was used instead of Na_2CO_3 (20 mol%). Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (41.9 mg, 48% yield). ^1H NMR (400 MHz): δ 7.83-7.76 (m, 2H), 7.52 (d, $J_{\text{C-P}} = 2.8$ Hz, 1H), 7.38-7.31 (m, 2H), 5.93 (dd, $J = 14.0, 10.8$ Hz, 1H), 4.26-3.89 (m, 8H), 1.34 (tb, $J_{\text{C-P}} = 7.2, 7.2$ Hz, 3H), 1.32 (tb, $J_{\text{C-P}} = 7.2, 7.2$ Hz, 3H), 1.29 (tb, $J_{\text{C-P}} = 7.2, 7.2$ Hz, 3H), 1.13 (tb, $J_{\text{C-P}} = 7.2, 7.2$ Hz, 3H); ^{13}C NMR (100 MHz): δ 140.2, 138.6, 136.0, 125.3 (d, $J_{\text{C-P}} = 9.0$ Hz), 124.8, 124.3, 123.9, 122.1, 70.9 (dd, $J_{\text{C-P}} = 176.9, 7.1$ Hz), 64.1 (dd, $J_{\text{C-P}} = 17.4, 5.8$ Hz), 63.7 (dd, $J_{\text{C-P}} = 6.2, 4.1$ Hz), 16.2 (dd, $J_{\text{C-P}} = 7.2, 6.3$ Hz) 15.7 (dd, $J_{\text{C-P}} = 16.5, 7.1$ Hz); ^{31}P NMR (162 MHz): δ 15.05 (d, $J = 32.2$ Hz), -1.22 (d, $J = 32.1$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{17}\text{H}_{26}\text{O}_7\text{P}_2\text{S}$: 437.0947; found: 431.0948.



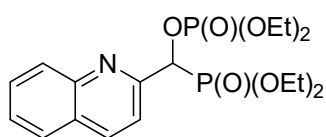
Benzofuran-2-yl(diethoxyphosphoryl)methyl diethyl phosphate (25c).

Et_3N (1equiv) was used instead of Na_2CO_3 (20 mol%). Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (41.9 mg, 48% yield). ^1H NMR (400 MHz): δ 7.29 (d, $J_{\text{C-P}} = 8.0$ Hz, 1H), 1.50 (d, $J_{\text{C-P}} = 8.4$ Hz, 1H), 7.33 (dd, $J_{\text{C-P}} = 7.2, 7.2$ Hz, 1H), 7.25 (dd, $J_{\text{C-P}} = 7.2, 7.2$ Hz, 1H), 7.02 (d, $J_{\text{C-P}} = 2.4$ Hz, 1H), 5.78 (dd, $J = 15.6, 10.4$ Hz, 1H), 4.30-3.92 (m, 8H), 1.37 (tb, $J_{\text{C-P}} = 7.2, 7.2$ Hz, 3H), 1.31 (tb, $J_{\text{C-P}} = 7.2, 7.2$ Hz, 3H), 1.31 (tb, $J_{\text{C-P}} = 7.2, 7.2$ Hz, 3H), 1.12 (tb, $J_{\text{C-P}} = 7.2, 0.8$ Hz, 3H); ^{13}C NMR (100 MHz): δ 155.1, 149.2, 127.5, 125.2, 123.1, 121.6, 111.4, 108.4 (d, $J_{\text{C-P}} = 6.8$ Hz), 68.2 (dd, $J_{\text{C-P}} = 176.1, 7.2$ Hz), 64.2 (dd, $J_{\text{C-P}} = 16.6, 5.9$ Hz), 63.9 (dd, $J_{\text{C-P}} = 32.5, 5.8$ Hz), 16.3 (dd, $J_{\text{C-P}} = 10.4, 5.6$ Hz), 15.8 (dd, $J_{\text{C-P}} = 18.2, 7.1$ Hz); ^{31}P NMR (162 MHz): δ 14.11 (d, $J = 32.2$ Hz), -1.20 (d, $J = 32.2$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{17}\text{H}_{26}\text{O}_8\text{P}_2$: 421.1176; found: 421.1176.



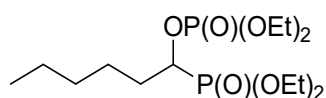
(diethoxyphosphoryl)(quinolin-6-yl)methyl diethyl phosphate (26c).

Et₃N (1equiv) was used instead of Na₂CO₃ (20 mol%). Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (69.0 mg, 80% yield). ¹H NMR (400 MHz): δ 8.94 (db, *J* = 3.2 Hz, 1H), 8.19 (d, *J* = 8.0 Hz, 1H), 8.14 (d, *J* = 8.8 Hz, 1H), 7.99 (b, 1H), 7.90 (db, *J* = 8.8 Hz, 1H), 7.45 (dd, *J* = 8.8, 4.0 Hz, 1H), 5.78 (dd, *J* = 13.6, 10.4 Hz, 1H), 4.23-3.87 (m, 8H), 1.31 (tb, *J* = 7.2, 7.2 Hz, 3H), 1.30 (tb, *J* = 7.2, 7.2 Hz, 3H), 1.25 (tb, *J* = 7.2, 7.2 Hz, 3H), 1.11 (tb, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz): δ 150.9, 148.0, 136.1, 131.9, 129.4, 128.4 (d, *J*_{C-P} = 4.1 Hz), 127.5, 127.2 (d, *J*_{C-P} = 7.5 Hz), 121.4, 74.2 (dd, *J*_{C-P} = 170.7, 6.9 Hz), 63.9 (dd, *J*_{C-P} = 23.6, 5.8 Hz), 63.5 (dd, *J*_{C-P} = 9.7, 7.3 Hz), 16.1 (dd, *J*_{C-P} = 8.3, 5.8 Hz), 15.7 (dd, *J*_{C-P} = 13.9, 7.0 Hz). ³¹P NMR (162 MHz): δ 16.1 (d, *J* = 34.0 Hz), -1.17 (d, *J* = 34.0 Hz); HRMS (ESI) *m/z*: [M+H]⁺ calcd. for C₁₈H₂₇NO₇P₂: 432.1336; found: 431.1336.



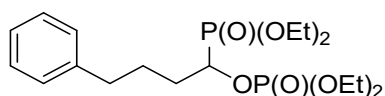
(diethoxyphosphoryl)(quinolin-2-yl)methyl diethyl phosphate (27c).

Et₃N (1equiv) was used instead of Na₂CO₃ (20 mol%). Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (35.3 mg, 41% yield). ¹H NMR (400 MHz): δ 8.22 (d, *J* = 8.8 Hz, 1H), 8.10 (d, *J* = 8.8 Hz, 1H), 7.83 (d, *J* = 8.4 Hz, 1H), 7.76-7.71 (m, 2H), 7.60 (dd, *J* = 7.6, 7.6 Hz, 1H), 5.89 (dd, *J* = 14.4, 10.8 Hz, 1H), 4.26-4.02 (m, 8H), 1.32 (tb, *J* = 7.2, 7.2 Hz, 3H), 1.26-1.23 (m, 6H), 1.18 (tb, *J* = 7.2, 7.2 Hz, 3H); ¹³C NMR (100 MHz): δ 154.1, 147.3, 136.8, 129.8, 129.3, 127.7, 127.6, 127.0, 119.8, 76.3 (dd, *J*_{C-P} = 172.1, 7.8 Hz), 64.3 (dd, *J*_{C-P} = 6.2, 6.2 Hz), 63.7 (dd, *J*_{C-P} = 11.4, 6.8 Hz), 16.3 (dd, *J*_{C-P} = 7.6, 6.0 Hz), 15.9 (db, *J*_{C-P} = 7.0 Hz); ³¹P NMR (162 MHz): δ 15.46 (d, *J* = 33.2 Hz), -1.29 (d, *J* = 33.3 Hz); HRMS (ESI) *m/z*: [M+H]⁺ calcd. for C₁₈H₂₇NO₇P₂: 432.1336; found: 431.1335.



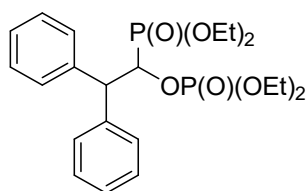
1-(diethoxyphosphoryl)hexyl diethyl phosphate (28c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (55.4 mg, 74% yield). ^1H NMR (400 MHz): δ 4.67-4.59 (m, 1H), 4.24-4.10 (m, 8H), 1.93-1.83 (m, 2H), 1.63-1.43 (m, 2H), 1.38-1.33 (m, 16H), 0.90 (t, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz): δ 73.1 (dd, $J_{\text{C-P}} = 168.8, 7.3$ Hz), 63.8 (db, $J_{\text{C-P}} = 5.9$ Hz), 62.7 (db, $J_{\text{C-P}} = 5.7$ Hz), 31.2, 30.8 (d, $J_{\text{C-P}} = 2.1$ Hz), 24.8 (d, $J_{\text{C-P}} = 10.4$ Hz), 22.2, 16.3 (dd, $J_{\text{C-P}} = 5.3, 5.3$ Hz), 15.9 (dd, $J_{\text{C-P}} = 7.0, 3.0$ Hz), 13.8; ^{31}P NMR (162 MHz): δ 20.22 (d, $J = 21.7$ Hz), -1.11 (d, $J = 21.8$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{14}\text{H}_{32}\text{O}_7\text{P}_2$: 375.1696; found: 375.1697.



1-(diethoxyphosphoryl)-4-phenylbutyl diethyl phosphate (29c).

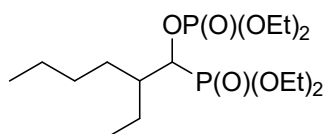
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (53.2 mg, 63% yield). ^1H NMR (400 MHz): δ 7.28-7.24 (m, 2H), 7.17 (d, $J = 7.6$ Hz, 3H), 4.70-4.62 (m, 1H), 4.21-4.04 (m, 8H), 2.70-2.63 (m, 2H), 1.97-1.80 (m, 4H), 1.33 (tb, $J = 7.2, 7.2$ Hz, 3H), 1.34-1.31 (m, 6H), 1.29 (tb, $J = 7.2, 0.8$ Hz, 3H); ^{13}C NMR (100 MHz): δ 141.5, 128.2, 128.1, 125.7, 72.7 (dd, $J_{\text{C-P}} = 169.1, 7.2$ Hz), 63.8 (db, $J_{\text{C-P}} = 5.9$ Hz), 62.6 (db, $J_{\text{C-P}} = 6.7$ Hz), 35.1, 30.3 (d, $J_{\text{C-P}} = 1.8$ Hz), 26.7 (d, $J_{\text{C-P}} = 10.3$ Hz), 16.3 (dd, $J_{\text{C-P}} = 5.2, 5.2$ Hz), 15.8 (db, $J_{\text{C-P}} = 7.1$ Hz); ^{31}P NMR (162 MHz): δ 19.9 (d, $J = 22.2$ Hz), -1.05 (d, $J = 22.2$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{18}\text{H}_{32}\text{O}_7\text{P}_2$: 423.1696; found: 423.1696.



1-(diethoxyphosphoryl)-2,2-diphenylethyl diethyl phosphate (30c).

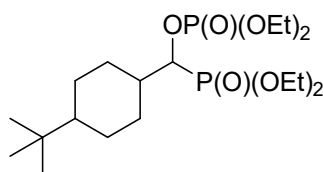
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (80.8 mg, 86% yield). ^1H NMR (400 MHz): δ 7.49 (db, $J = 7.2$ Hz, 2H), 7.37 (db, $J = 7.2$ Hz, 2H), 7.32-7.15 (m, 6H), 5.57-5.50 (m, 1H), 4.55 (dd, $J = 8.4, 8.4$ Hz, 1H), 4.10-3.17 (m, 8H), 1.27 (td, $J = 7.2, 1.8$ Hz, 3H), 1.13 (td, $J = 7.2, 7.2$ Hz, 3H), 1.11 (td, $J = 7.2, 7.2$ Hz, 3H), 0.94 (td, $J = 7.2, 1.2$ Hz, 3H); ^{13}C NMR (100 MHz): δ 139.9 (d, $J_{\text{C-P}} = 11.0$ Hz), 139.8 (d, $J_{\text{C-P}} = 5.1$ Hz), 129.3, 128.7, 128.4 (d, $J_{\text{C-P}} = 5.7$ Hz), 126.9, 75.5 (dd, $J_{\text{C-P}} = 165.1, 5.4$ Hz), 63.9 (db, $J_{\text{C-P}} = 6.1$ Hz), 63.1 (db,

$J_{C-P} = 4.0$ Hz), 62.5 (dd, $J_{C-P} = 24.5, 7.0$ Hz), 53.3 (dd, $J_{C-P} = 4.1, 4.1$ Hz), 16.1 (dd, $J_{C-P} = 5.2, 5.2$ Hz), 15.8 (dd, $J_{C-P} = 11.3, 7.5$ Hz); ^{31}P NMR (162 MHz): δ 18.87 (d, $J = 10.0$ Hz), -2.06 (d, $J = 10.0$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{22}\text{H}_{32}\text{O}_7\text{P}_2$: 471.1696; found: 471.1695.



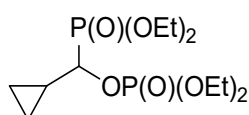
1-(diethoxyphosphoryl)-2-ethylhexyl diethyl phosphate (31c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (50.7 mg, 63% yield). The product is a mixture of two isomers (2:3). ^1H NMR (400 MHz): δ 4.79-4.71 (m, 1H), 4.23-4.09 (m, 8H), 1.82-1.26 (m, 21H), 0.98-0.89 (m, 6H); ^{13}C NMR (100 MHz): δ 74.8 (dd, $J_{C-P} = 166.9, 7.8$ Hz), 62.6-62.4 (m), 42.1, 29.6-29.0 (m), 23.2-22.6 (m), 16.3 (dd, $J_{C-P} = 4.7, 4.7$ Hz), 13.9, 11.9; ^{31}P NMR (162 MHz): δ 20.47-20.30(m), -0.92-(-1.05) (m); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{16}\text{H}_{36}\text{O}_7\text{P}_2$: 403.2009; found: 403.2009.



(4-(tert-butyl)cyclohexyl)(diethoxyphosphoryl)methyl diethyl phosphate (32c).

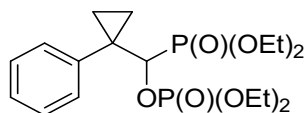
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (63.7 mg, 72% yield). The product is a mixture of two isomers (8:2). ^1H NMR (400 MHz): δ 4.55-4.49 (m, 1H), 4.25-4.09 (m, 8H), 2.22-0.8 (m, 22H), 0.85-0.84 (m, 9H); ^{13}C NMR (100 MHz): δ 74.9 (dd, $J_{C-P} = 166.5, 7.8$ Hz), 63.9 (d, $J_{C-P} = 6.0$ Hz), 47.4, 39.5, 32.2, 27.8-26.7 (m), 21.8 (d, $J_{C-P} = 76.7$ Hz), 16.3 (dd, $J_{C-P} = 6.1, 6.1$ Hz), 16.0 (dd, $J_{C-P} = 6.9, 3.2$ Hz); ^{31}P NMR (162 MHz): δ 19.85 (d, $J = 19.9$ Hz), -1.03 (d, $J = 19.6$ Hz), HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{19}\text{H}_{40}\text{O}_7\text{P}_2$: 443.2322; found: 443.2318.



Cyclopropyl(diethoxyphosphoryl)methyl diethyl phosphate (33c).

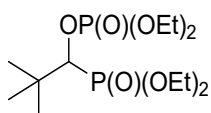
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a

light yellow oil (53.7 mg, 78% yield). ^1H NMR (400 MHz): δ 4.27-4.12 (m, 8H), 3.94-3.91 (m, 1H), 1.38-1.29 (m, 12H), 0.78-0.71 (m, 3H), 0.59-0.54 (m, 1H); ^{13}C NMR (100 MHz): δ 77.7 (dd, $J_{\text{C-P}} = 174.1, 7.0$ Hz), 63.6 (db, $J_{\text{C-P}} = 5.8$ Hz), 62.6 (dd, $J_{\text{C-P}} = 5.8, 5.8$ Hz), 16.2 (db, $J_{\text{C-P}} = 5.7$ Hz), 15.7 (db, $J_{\text{C-P}} = 7.2$ Hz), 11.3, 4.6 (d, $J_{\text{C-P}} = 13.4$ Hz), 3.7; ^{31}P NMR (162 MHz): δ 18.63 (d, $J = 30.0$ Hz), -0.98 (d, $J = 30.1$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{12}\text{H}_{26}\text{O}_7\text{P}_2$: 361.1540; found: 361.1540.



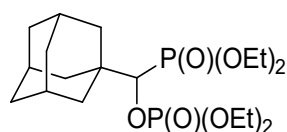
(diethoxyphosphoryl)(1-phenylcyclopropyl)methyl diethyl phosphate (34c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (67.2 mg, 80% yield). ^1H NMR (400 MHz): δ 7.53 (dd, $J = 8.0, 1.2$ Hz, 2H), 7.36-7.20 (m, 3H), 1.43 (td, $J = 7.2, 1.2$ Hz, 3H), 1.34 (td, $J = 7.2, 0.8$ Hz, 3H), 1.29-1.19 (m, 3H), 1.16 (td, $J = 7.2, 7.2$ Hz, 3H), 1.04 (td, $J = 7.2, 7.2$ Hz, 3H), 0.90-0.85 (m, 1H); ^{13}C NMR (100 MHz): δ 139.8, 131.1, 127.2, 126.7, 81.0 (dd, $J_{\text{C-P}} = 171.5, 7.4$ Hz), 63.7 (dd, $J_{\text{C-P}} = 13.3, 5.9$ Hz), 62.1 (dd, $J_{\text{C-P}} = 6.6, 5.5$ Hz), 26.2, 15.8 (dd, $J_{\text{C-P}} = 5.4, 5.4$ Hz), 15.7 (dd, $J_{\text{C-P}} = 7.4, 2.4$ Hz), 13.6 (d, $J_{\text{C-P}} = 13.0$ Hz), 11.7; ^{31}P NMR (162 MHz): δ 16.53 (d, $J = 27.9$ Hz), -0.35 (dd, $J = 26.4$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{18}\text{H}_{30}\text{NO}_7\text{P}_2$: 421.1540; found: 421.1540.



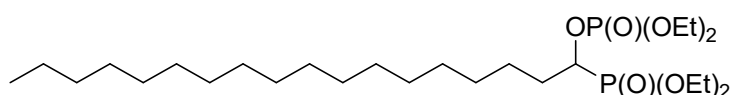
1-(diethoxyphosphoryl)-2,2-dimethylpropyl diethyl phosphate (35c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (45.4 mg, 63% yield). ^1H NMR (400 MHz): δ 4.44 (d, $J = 11.6, 8.4$ Hz, 1H), 4.23-4.11 (m, 8H), 1.37-1.33 (m, 12H), 1.14 (s, 9H); ^{13}C NMR (100 MHz): δ 81.2 (dd, $J_{\text{C-P}} = 162.3, 8.3$ Hz), 63.8 (dd, $J_{\text{C-P}} = 12.5, 6.0$ Hz), 62.3 (dd, $J_{\text{C-P}} = 8.2, 8.2$ Hz), 34.9 (dd, $J_{\text{C-P}} = 2.9, 2.9$ Hz), 26.7 (d, $J_{\text{C-P}} = 6.1$ Hz), 16.2 (dd, $J_{\text{C-P}} = 9.2, 5.9$ Hz), 15.9 (dd, $J_{\text{C-P}} = 7.0, 3.6$ Hz); ^{31}P NMR (162 MHz): δ 19.4 (d, $J = 15.2$ Hz), -1.13 (d, $J = 15.2$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{13}\text{H}_{30}\text{O}_7\text{P}_2$: 361.1540; found: 361.1539.



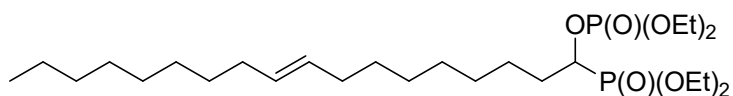
Adamantan-1-yl(diethoxyphosphoryl)methyl diethyl phosphate (36c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (70.1 mg, 80% yield). ¹H NMR (400 MHz): δ 4.30 (dd, $J = 11.6, 8.8$ Hz, 1H) 4.24-4.11 (m, 8H), 2.01 (b, 3H), 1.83 (d, $J = 2.0$ Hz, 6H), 1.69-1.69 (b, 6H), 1.36 (tb, $J = 7.2$ Hz, 12H); ¹³C NMR (100 MHz): δ 81.5 (dd, $J_{C-P} = 161.4, 8.4$ Hz), 63.5 (dd, $J_{C-P} = 20.3, 6.0$ Hz), 62.1 (dd, $J_{C-P} = 15.9, 7.4$ Hz), 37.9 (d, $J_{C-P} = 5.7$ Hz), 36.5, 36.3, 27.8, 16.1 (dd, $J_{C-P} = 12.1, 5.6$ Hz), 15.7 (dd, $J_{C-P} = 6.8, 6.8$ Hz); ³¹P NMR (162 MHz): δ 19.18 (d, $J = 15.6$ Hz), -1.09 (d, $J = 15.6$ Hz); HRMS (ESI) m/z : $[M+H]^+$ calcd. for C₁₉H₃₆O₇P₂: 439.2009; found: 439.2010.



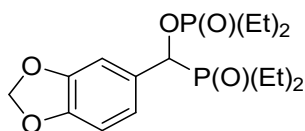
1-(diethoxyphosphoryl)octadecyl diethyl phosphate (37c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (81.3 mg, 75% yield). ¹H NMR (400 MHz): δ 4.66-4.60 (m, 1H), 4.24-4.10 (m, 8H), 1.92-1.82 (m, 2H), 1.59-1.45 (m, 2H), 1.37-1.32 (m, 15H), 1.25-0.86 (m, 25H), 0.88 (td, $J = 7.2, 7.2$ Hz, 3H); ¹³C NMR (100 MHz): δ 73.2 (dd, $J_{C-P} = 168.7, 7.4$ Hz), 63.3 (dd, $J_{C-P} = 118.7, 5.9$ Hz), 31.8, 30.9 (d, $J_{C-P} = 2.2$ Hz), 29.6 (b), 29.5, 29.3, 29.3, 29.2, 25.3 (d, $J_{C-P} = 10.5$ Hz), 22.6, 16.3 (dd, $J_{C-P} = 5.4, 5.4$ Hz), 16.0 (dd, $J_{C-P} = 6.9, 3.8$ Hz) 14.0; ³¹P NMR (162 MHz): δ 20.28 (d, $J = 21.7$ Hz), -1.06 (dd, $J = 21.7$ Hz); HRMS (ESI) m/z : $[M+H]^+$ calcd. for C₂₆H₅₆O₇P₂: 543.3574; found: 543.3574.



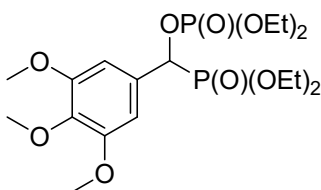
(E)-1-(diethoxyphosphoryl)octadec-9-en-1-yl diethyl phosphate (38c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (81.0 mg, 75% yield). ¹H NMR (400 MHz): δ 5.38-5.32 (m, 2H), 4.67-4.59 (m, 1H), 4.24-4.10 (m, 8H), 2.03-1.84 (m, 4H), 1.60-1.45 (m, 2H), 1.37-1.27 (m, 34H), 0.88 (td, $J = 7.2, 7.2$ Hz, 3H); ¹³C NMR (100 MHz): δ 129.9, 129.7, 73.2 (dd, $J_{C-P} = 168.7, 7.2$ Hz), 63.9 (db, $J_{C-P} = 5.9$ Hz), 62.7 (db, $J_{C-P} = 6.6$ Hz), 31.8, 30.9, 30.9, 29.7, 29.7, 29.6, 29.6, 29.4, 29.2, 29.1, 27.1, 25.3, 25.2, 22.6, 16.4 (dd, $J_{C-P} = 5.3, 5.3$ Hz), 16.0 (dd, $J_{C-P} = 6.9, 4.0$ Hz), 14.0; ³¹P NMR (162 MHz): δ 20.28 (d, $J = 21.7$ Hz), -1.03 (d, $J = 21.7$ Hz); HRMS (ESI) m/z : $[M+H]^+$ calcd. for C₂₆H₅₄O₇P₂: 541.3418; found: 541.3419.



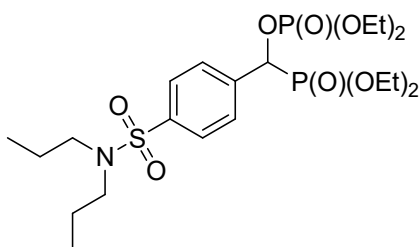
Benzo[d][1,3]dioxol-5-yl(diethylphosphoryl)methyl diethylphosphate (39c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (67.0 mg, 93% yield). ¹H NMR (400 MHz): δ 7.05 (b, 1H), 6.97 (db, J = 8.0 Hz, 1H), 6.79 (db, J = 8.0 Hz, 1H), 5.98 (dd, J = 7.2, 3.2 Hz, 2H), 5.46 (dd, J = 13.2, 10.4 Hz), 4.19-3.87 (m, 8H), 1.32 (td, J = 7.2, 7.2 Hz, 3H), 1.30 (td, J = 6.8, 0.8 Hz, 3H), 1.24 (td, J = 7.2, 7.2 Hz, 3H), 1.17 (td, J = 7.2, 1.2 Hz, 3H); ¹³C NMR (100 MHz): δ 148.0 (d, J_{C-P} = 2.5 Hz), 147.5 (d, J_{C-P} = 1.4 Hz), 126.9, 121.9 (d, J_{C-P} = 7.6 Hz), 108.2 (d, J_{C-P} = 5.1 Hz), 107.8, 101.0, 74.2 (dd, J_{C-P} = 173.8, 6.9 Hz), 63.8 (dd, J_{C-P} = 19.3, 5.8 Hz), 63.1 (d, J_{C-P} = 6.9 Hz), 16.0 (dd, J_{C-P} = 9.5, 5.7 Hz), 15.6 (dd, J_{C-P} = 11.4, 7.1 Hz); ³¹P NMR (162 MHz): δ 16.7 (d, J = 36.0 Hz), -1.22 (d, J = 35.8 Hz); HRMS (ESI) m/z : [M+H]⁺ calcd. for C₁₆H₂₆O₉P₂: 425.1125; found: 425.1124.



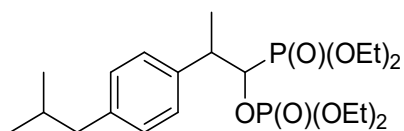
(diethoxyphosphoryl)(3,4,5-trimethoxyphenyl)methyl diethyl phosphate (40c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (71.4 mg, 76% yield). ¹H NMR (400 MHz): δ 6.75 (d, J = 1.6 Hz, 2H), 5.47 (dd, J = 13.2, 10.4 Hz, 1H), 4.19-3.89 (m, 8H), 3.86 (s, 6H), 3.84 (s, 3H), 1.32 (td, J = 7.2, 7.2 Hz, 3H), 1.29 (td, J = 7.2, 1.2 Hz, 3H), 1.24 (td, J = 7.2, 7.2 Hz, 3H), 1.15 (td, J = 7.2, 1.2 Hz, 3H); ¹³C NMR (100 MHz): δ 153.0, 138.4, 128.9, 105.1 (d, J_{C-P} = 6.1 Hz), 74.5 (dd, J_{C-P} = 172.3, 6.8 Hz), 63.9 (dd, J_{C-P} = 19.8, 5.8 Hz), 63.3 (dd, J_{C-P} = 7.0, 4.6 Hz), 60.6 (d, J_{C-P} = 1.0 Hz), 56.0, 16.2 (dd, J_{C-P} = 13.2, 5.7 Hz), 15.7 (dd, J_{C-P} = 9.9, 7.3 Hz); ³¹P NMR (162 MHz): δ 16.74 (d, J = 35.5 Hz), -1.02 (d, J = 35.6 Hz); HRMS (ESI) m/z : [M+H]⁺ calcd. for C₁₈H₃₂O₁₀P₂: 471.1543; found: 471.1547.

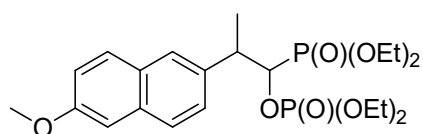


(diethoxyphosphoryl)(4-(N,N-dipropylsulfamoyl)phenyl)methyl diethyl phosphate (41c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (55.4 mg, 51% yield). ¹H NMR (400 MHz): δ 7.82 (d, *J* = 8.0 Hz, 2H), 7.63 (dd, *J* = 8.4, 1.6 Hz, 2H), 5.63 (dd, *J* = 14.8, 10.8 Hz, 1H), 4.21-3.90 (m, 8H), 3.10-3.06 (m, 4H), 1.59-1.50 (m, 4H), 1.30 (td, *J* = 7.2, 0.8 Hz, 3H), 1.28 (td, *J* = 7.6, 7.6 Hz, 3H), 1.26 (td, *J* = 7.2, 7.2 Hz, 3H), 1.17 (td, *J* = 7.2, 1.2 Hz, 3H), 0.86 (t, *J* = 7.2 Hz, 6H); ¹³C NMR (100 MHz): δ 140.7, 138.3, 128.2 (d, *J*_{C-P} = 5.4 Hz), 126.9 (d, *J*_{C-P} = 1.7 Hz), 73.9 (dd, *J*_{C-P} = 169.3, 6.8 Hz), 64.2 (dd, *J*_{C-P} = 23.6, 5.8 Hz), 63.6 (d, *J*_{C-P} = 12.9, 7.1 Hz), 49.9, 21.9, 16.2 (dd, *J*_{C-P} = 5.8, 5.8 Hz), 15.9 (dd, *J*_{C-P} = 7.0, 7.0 Hz), 11.1; ³¹P NMR (162 MHz): δ 15.6 (d, *J* = 32.4 Hz), -1.10 (d, *J* = 32.6 Hz); HRMS (ESI) *m/z*: [M+H]⁺ calcd. for C₂₁H₃₉NO₉P₂S: 544.1894; found: 544.1894.



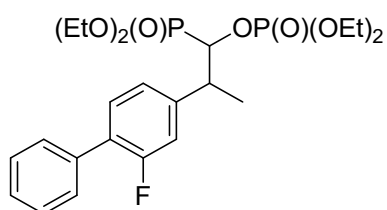
1-(diethoxyphosphoryl)-2-(4-isobutylphenyl)propyl diethyl phosphate (42c). Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (77.1 mg, 83% yield). The product is a mixture of two isomers (1:1). ¹H NMR (400 MHz): δ 7.24 (dd, *J* = 8.4 Hz, 2H), 7.08 (dd, *J* = 8.0, 2.0 Hz, 2H), 4.92-4.81 (m, 1H), 4.15-3.68 (m, 8H), 3.45-3.34 (m, 1H), 2.43 (d, *J* = 7.2 Hz, 2H), 1.88-1.78 (m, 1H), 1.34-1.16 (m, 15H), 0.89 (dd, *J*_{C-P} = 6.8, 2.4 Hz, 3H); ¹³C NMR (100 MHz): δ 140.0 (d, *J*_{C-P} = 4.1 Hz), 139.0 (dd, *J*_{C-P} = 30.6, 8.9 Hz), 128.8 (d, *J*_{C-P} = 8.7 Hz), 127.9 (d, *J*_{C-P} = 44.4 Hz), 78.2 (dd, *J*_{C-P} = 7.6, 6.0 Hz), 63.8-63.4 (m), 62.5-62.3 (m), 44.8 (d, *J*_{C-P} = 2.0 Hz), 40.6 (d, *J*_{C-P} = 99.7 Hz), 30.0, 22.1 (d, *J*_{C-P} = 3.7 Hz), 18.8 (d, *J*_{C-P} = 7.5 Hz), 16.3-16.0 (m), 115.9-15.8 (m); ³¹P NMR (162 MHz): δ 19.18-18.96 (m), -1.13-(1.46) (m)



1-(diethoxyphosphoryl)-2-(6-methoxynaphthalen-2-yl)propyl diethyl phosphate (43c).

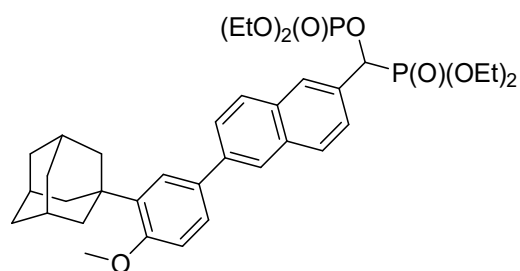
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (39.0 mg, 40% yield). The product is a mixture of two isomers (8:2). ¹H NMR (400 MHz): δ 7.73-7.12 (m, 6H), 5.00-4.93 (m, 1H), 4.24-3.50 (m, 8H), 3.94 (s, 3H), 1.61 (d, *J* = 7.2 Hz, 3H), 1.38-1.12 (m, 12H), 1.01-0.91 (m, 1H); ¹³C NMR (100 MHz): δ 157.4, 137.1 (d, *J*_{C-P} = 9.3 Hz), 126.8 (d,

$J_{C-P} = 28.4$ Hz), 126.5, 118.7, 105.4, 77.3 (dd, $J_{C-P} = 164.8, 7.7$ Hz), 63.8 (dd, $J_{C-P} = 15.1, 5.9$ Hz), 62.6 (d, $J_{C-P} = 18.1, 7.2$ Hz), 55.2, 40.5, 29.6, 16.2 (dd, $J_{C-P} = 12.9, 6.2$ Hz), 15.9 (dd, $J_{C-P} = 7.4, 7.4$ Hz); ^{31}P NMR (162 MHz): δ 19.19-19.07 (m), -0.95-(-1.40) (m). HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{22}\text{H}_{34}\text{O}_8\text{P}_2$: 489.1802; found: 489.1805.



1-(diethoxyphosphoryl)-2-(2-fluoro-[1,1'-biphenyl]-4-yl)propyl diethyl phosphate (44c).

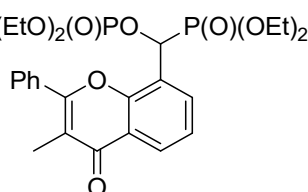
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (65.4 mg, 67% yield). The product is a mixture of two isomers (8:2). ^1H NMR (400 MHz): δ 7.54-7.14 (m, 8H), 4.94-4.83 (m, 1H), 4.20-3.85 (m, 8H), 3.53-3.44 (m, 1H), 1.55-1.52 (m, 3H), 1.36-1.23 (m, 12H); ^{13}C NMR (100 MHz): δ 160.6 (d, $J_{C-P} = 246.2$ Hz), 143.8 (dd, $J_{C-P} = 9.7, 7.5$ Hz), 143.4, 135.6, 130.4 (d, $J_{C-P} = 3.8$ Hz), 128.8, 128.4, 127.6, 127.4 (d, $J_{C-P} = 13.4$ Hz), 124.3 (d, $J_{C-P} = 3.1$ Hz), 115.9 (d, $J_{C-P} = 23.3$ Hz), 77.1 (dd, $J_{C-P} = 165.3, 7.6$ Hz), 64.1-63.8 (m), 62.9-62.6 (m), 40.2, 18.8 (d, $J_{C-P} = 8.0$ Hz), 16.3-16.2 (m), 16.1-16.0 (m); ^{31}P NMR (162 MHz): δ 18.66 (d, $J = 15.55$ Hz), -1.00 (d, $J = 15.71$ Hz). HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{23}\text{H}_{33}\text{O}_7\text{P}_2$: 503.1758; found: 503.1758.



(6-(3-adamantan-1-yl-4-methoxyphenyl)naphthalen-2-yl)(diethoxyphosphoryl) methyl diethyl phosphate (45c).

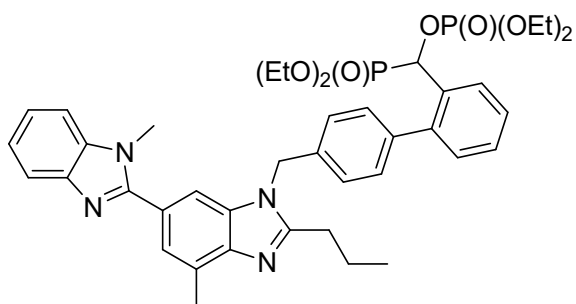
Like the **procedure A** but Et_3N (1equiv) instead of Na_2CO_3 (20 mol%). Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (111.2 mg, 83% yield). ^1H NMR (400 MHz): δ 7.99 (s, 2H), 7.91 (dd, $J = 8.8, 2.0$ Hz, 2H), 7.76 (dd, $J = 8.4, 0.8$ Hz, 1H), 7.67 (db, $J = 8.8$ Hz, 1H), 7.58 (d, $J = 2.4$ Hz, 1H), 7.51 (dd, $J = 8.4, 2.0$ Hz, 1H), 6.97 (d, $J = 8.4$ Hz, 1H), 5.75 (dd, $J = 13.6, 10.8$ Hz, 1H), 4.24-3.82 (m, 8H), 3.87 (s, 3H), 2.18 (b, 6H), 1.80 (b, 6H), 1.36-1.32-1.26 (m, 9H), 1.22 (td, $J = 7.2, 7.2$ Hz, 3H), 1.09 (td, $J = 7.2, 0.8$ Hz, 3H); ^{13}C NMR (100 MHz): δ

167.5, 158.4, 139.6, 138.6, 133.6, 132.5, 132.2, 131.5, 130.7, 128.4 (d, $J_{C-P} = 20.0$ Hz), 127.2 (d, $J_{C-P} = 7.8$ Hz), 126.1, 125.6, 125.3 (d, $J_{C-P} = 4.2$ Hz), 124.5, 111.8, 74.7 (dd, $J_{C-P} = 171.5, 6.9$ Hz), 67.9, 63.9 (dd, $J_{C-P} = 22.8, 5.7$ Hz), 63.3 (db, $J_{C-P} = 6.1$ Hz), 54.9, 40.4, 38.5, 36.9, 30.1, 28.9, 28.7, 23.5, 22.8, 16.1 (dd, $J_{C-P} = 10.2, 5.7$ Hz), 15.7 (dd, $J_{C-P} = 14.2, 7.1$ Hz), 13.8, 10.7; ^{31}P NMR (162 MHz): δ 16.76 (d, $J = 34.5$ Hz), -0.97 (d, $J = 34.7$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{36}\text{H}_{48}\text{O}_8\text{P}_2$: 671.2897; found: 671.2894.



(diethoxyphosphoryl)(3-methyl-4-oxo-2-phenyl-4H-chromen-8-yl)methyl diethyl phosphate (46c).

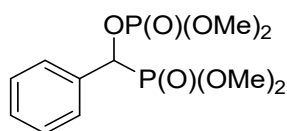
Like the **procedure A** but Et_3N (1equiv) instead of Na_2CO_3 (20 mol%). Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (59.2 mg, 55% yield). ^1H NMR (400 MHz): δ 8.29 (dd, $J = 7.2, 4.4$ Hz, 1H), 8.00 (dd, $J = 6.8, 4.0$ Hz, 1H), 7.81-7.78 (m, 2H), 7.48 (dd, $J = 8.0, 8.0$ Hz, 1H), 6.28 (dd, $J = 14.0, 10.8$ Hz, 1H), 4.18-3.89 (m, 8H), 2.21 (s, 1H), 1.25 (td, $J = 7.2, 1.2$ Hz, 3H), 1.24 (td, $J = 7.2, 7.2$ Hz, 3H), 1.20 (td, $J = 7.2, 7.2$ Hz, 3H), 1.13 (td, $J = 7.2, 1.2$ Hz, 3H); ^{13}C NMR (100 MHz): δ 178.4, 160.6, 152.6 (d, $J_{C-P} = 5.8$ Hz), 132.9, 132.6 (d, $J_{C-P} = 4.1$ Hz), 130.2, 129.0, 128.3, 126.6 (d, $J_{C-P} = 2.3$ Hz), 124.2 (d, $J_{C-P} = 2.5$ Hz), 123.3, 122.1, 117.3, 67.3 (dd, $J_{C-P} = 173.0, 4.4$ Hz), 64.1 (dd, $J_{C-P} = 20.3, 5.9$ Hz), 63.5 (dd, $J_{C-P} = 10.6, 7.1$ Hz), 16.1 (dd, $J_{C-P} = 10.9, 5.7$ Hz), 15.7 (dd, $J_{C-P} = 6.9, 6.9$ Hz), 11.5; ^{31}P NMR (162 MHz): δ 15.92 (d, $J = 34.2$ Hz), -1.21 (d, $J = 34.0$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{25}\text{H}_{32}\text{O}_9\text{P}_2$: 539.1594; found: 539.1596.



(diethoxyphosphoryl)(4'-((1,7'-dimethyl-2'-propyl-1H,3'H-[2,5'-bibenzo[d]imidazol]-3'-yl)methyl)-[1,1'-biphenyl]-2-yl)methyl diethyl phosphate (47c).

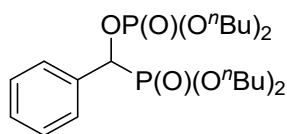
Flash column chromatography (silica gel: EA) afforded the title compound as a yellow oil (143.6 mg, 93% yield). ^1H NMR (400 MHz): δ 7.70-7.66 (m, 2H), 7.32 (d, $J = 8.4$ Hz, 4H), 7.28-7.23 (m, 2H),

7.18-7.10 (m, 3H), 7.04 (m, 3H), 5.56 (dd, $J = 13.2, 10.0$ Hz, 1H), 5.30 (s, 2H), 3.95-3.66 (m, 8H), 3.60 (s, 3H), 2.80 (t, $J = 7.6$ Hz), 2.64 (b, 3H), 1.75-1.70 (m, 2H), 1.10 (td, $J = 7.2, 7.2$ Hz, 3H), 1.02 (td, $J = 6.8, 6.8$ Hz, 3H), 1.00 (td, $J = 7.2, 7.2$ Hz, 3H), 0.92 (t, $J = 7.2$ Hz, 3H), 0.90 (td, $J = 7.2, 7.2$ Hz, 3H); ^{13}C NMR (100 MHz): δ 156.3, 154.4, 142.8, 142.6, 141.3 (d, $J_{\text{C-P}} = 8.1$ Hz), 139.5, 136.4, 134.9 (d, $J_{\text{C-P}} = 5.7$ Hz), 131.1, 130.1, 130.0, 129.3, 128.8, 128.8, 128.7, 128.7, 127.6, 125.9, 123.7 (d, $J_{\text{C-P}} = 8.3$ Hz), 122.4, 122.2, 119.2, 109.4, 108.8, 70.9 (dd, $J_{\text{C-P}} = 173.2, 6.9$ Hz), 63.7 (dd, $J_{\text{C-P}} = 16.7, 5.7$ Hz), 63.2 (dd, $J_{\text{C-P}} = 15.2, 7.1$ Hz), 46.8, 31.1, 29.6, 21.6, 16.7, 16.1 (dd, $J_{\text{C-P}} = 13.9, 5.6$ Hz), 15.6 (dd, $J_{\text{C-P}} = 11.0, 7.1$ Hz), 13.9; ^{31}P NMR (162 MHz): δ 16.99 (d, $J = 38.2$ Hz), -1.60 (d, $J = 38.4$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{41}\text{H}_{50}\text{N}_4\text{O}_7\text{P}_2$: 773.3227; found: 773.3227.



(dimethoxyphosphoryl)(phenyl)methyl dimethyl phosphate (48c).

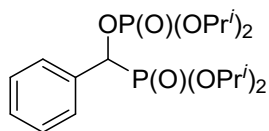
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (58.3 mg, 93% yield). ^1H NMR (400 MHz): δ 7.52 (d, $J = 7.2$ Hz, 2H), 7.42-7.34 (m, 3H), 5.59 (dd, $J = 13.2, 10.8$ Hz, 1H), 3.76 (d, $J = 10.4$ Hz, 3H), 3.76 (d, $J = 11.6$ Hz, 3H), 3.68 (db, $J = 10.4$ Hz, 3H), 3.54 (db, $J = 11.2$ Hz, 3H); ^{13}C NMR (100 MHz): δ 133.1, 129.1 (d, $J_{\text{C-P}} = 2.4$ Hz), 128.5, 127.7 (d, $J_{\text{C-P}} = 5.8$ Hz), 74.4 (dd, $J_{\text{C-P}} = 171.2, 6.6$ Hz) 54.3 (dd, $J_{\text{C-P}} = 22.1, 5.9$ Hz), 53.9 (dd, $J_{\text{C-P}} = 7.4, 7.4$ Hz); ^{31}P NMR (162 MHz): δ 18.96 (d, $J = 32.7$ Hz), -1.35 (d, $J = 32.4$ Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{11}\text{H}_{18}\text{O}_7\text{P}_2$: 325.0601 found: 325.0601.



Dibutyl ((dibutoxyphosphoryl)(phenyl)methyl) phosphate (49c).

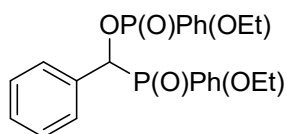
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (92.5 mg, 94% yield). ^1H NMR (400 MHz): δ 7.53-7.50 (m, 2H), 7.39-7.33 (m, 3H), 5.55 (dd, $J = 13.6, 10.4$ Hz), 4.12-3.73 (m, 8H) 1.63-1.50 (m, 17H), 0.91 (td, $J = 7.2, 7.2$ Hz, 6H), 0.87 (td, $J = 7.2, 7.2$ Hz, 3H), 0.81 (td, $J = 7.2, 7.2$ Hz, 3H); ^{13}C NMR (100 MHz): δ 133.5, 128.7 (d, $J_{\text{C-P}} = 2.4$ Hz), 128.1 (d, $J_{\text{C-P}} = 1.5$ Hz), 127.9 (d, $J_{\text{C-P}} = 5.8$ Hz), 74.5 (dd, $J_{\text{C-P}} = 171.5, 6.9$ Hz), 67.5 (dd, $J_{\text{C-P}}$

= 21.4, 6.0 Hz), 66.9 (dd, J_{C-P} = 2.4, 1.3 Hz), 32.3 (dd, J_{C-P} = 10.0, 5.7 Hz), 31.8 (dd, J_{C-P} = 13.8, 7.3 Hz), 18.4, 18.3 (d, J_{C-P} = 10.1 Hz), 13.3, 13.3 (d, J_{C-P} = 6.0 Hz); ^{31}P NMR (162 MHz): δ 16.66 (d, J = 35.6 Hz), -0.86 (d, J = 35.6 Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{23}\text{H}_{42}\text{O}_7\text{P}_2$: 493.2479; found: 493.2479.



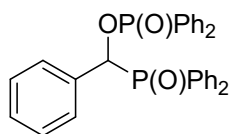
(diisopropoxyphosphoryl)(phenyl)methyl diisopropyl phosphate (50c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (72.4 mg, 83% yield) ^1H NMR (400 MHz): δ 7.53-7.51 (m, 2H), 7.37-7.32 (m, 3H), 5.48 (dd, J = 14.4, 10.4 Hz, 1H), 4.73-4.32 (m, 4H), 1.34 (d, J = 6.0 Hz, 3H), 1.30 (d, J = 2.8 Hz, 3H), 1.29 (b, 3H), 1.28 (d, J = 6.4 Hz, 3H), 1.12 (dd, J = 6.4, 6.4 Hz, 3H), 1.02 (d, J = 6.0 Hz, 3H); ^{13}C NMR (100 MHz): δ 133.2, 129.3, 128.7, 127.9 (d, J_{C-P} = 5.9 Hz), 74.5 (dd, J_{C-P} = 171.1, 6.7 Hz), 54.4 (dd, J_{C-P} = 21.9, 5.9 Hz), 54.0 (dd, J_{C-P} = 6.7, 6.7 Hz), 29.7, 14.1; ^{31}P NMR (162 MHz): δ 15.15 (d, J = 38.6 Hz), -2.80 (d, J = 38.6 Hz); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{19}\text{H}_{34}\text{O}_7\text{P}_2$: 437.1853; found: 437.1857.



(ethoxy(phenyl)phosphoryl)(phenyl)methyl ethyl phenylphosphonate (51c).

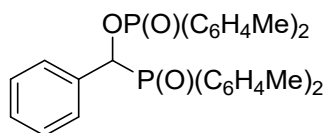
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light yellow oil (56.8 mg, 64% yield) ^1H NMR (400 MHz): δ 7.53-7.05 (m, 15H), 5.89-5.61 (m, 1H), 4.04-3.57 (m, 4H), 1.24-0.95 (m, 6H); ^{13}C NMR (100 MHz): δ 133.6-127.8 (m), 77.2-74.3 (m), 62.2-61.6 (m), 16.4-15.8 (m); ^{31}P NMR (162 MHz): δ 34.2-33.6 (m), 19.9-19.1 (m); HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{23}\text{H}_{26}\text{O}_5\text{P}_2$: 445.1328; found: 445.1329.



(diphenylphosphoryl)(phenyl)methyl diphenylphosphinate (52c).

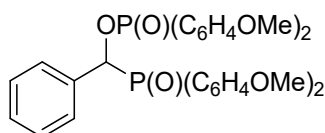
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light white solid (92.5 mg, 91% yield) ^1H NMR (400 MHz): δ 8.03-7.01 (m, 25H), 6.31 (dd, J = 10.0,

2.4 Hz, 1H); ^{13}C NMR (100 MHz): δ 132.4 (d, $J_{\text{C-P}} = 9.0$ Hz), 132.2 (d, $J_{\text{C-P}} = 2.8$ Hz), 132.0, 132.0 (d, $J_{\text{C-P}} = 5.4$ Hz), 131.8 (d, $J_{\text{C-P}} = 2.8$ Hz), 131.7, , 131.6, 131.5, 131.3 (d, $J_{\text{C-P}} = 10.8$ Hz), 131.1 (d, $J_{\text{C-P}} = 10.5$ Hz), 130.3 (d, $J_{\text{C-P}} = 68.4$ Hz), 129.8 (d, $J_{\text{C-P}} = 24.8$ Hz), 129.1 (d, $J_{\text{C-P}} = 4.8$ Hz), 129.0, 128.5 (d, $J_{\text{C-P}} = 2.1$ Hz), 128.4, 128.2 (d, $J_{\text{C-P}} = 2.4$ Hz), 128.0 (d, $J_{\text{C-P}} = 22.3$ Hz), 127.7, 127.6 (d, $J_{\text{C-P}} = 1.2$ Hz), 74.7 (dd, $J_{\text{C-P}} = 85.4, 7.5$ Hz); ^{31}P NMR (162 MHz): δ 35.43 (d, $J = 24.5$ Hz) 28.95 (d, $J = 24.7$ Hz). HRMS (ESI) m/z: $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{31}\text{H}_{26}\text{O}_3\text{P}_2$: 509.1430; found: 509.1429.



(di-p-tolylphosphoryl)(phenyl)methyl di-p-tolylphosphinate (53c).

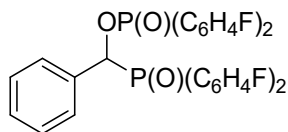
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light white solid (84.6 mg, 75% yield) ^1H NMR (400 MHz): δ 7.83 (dd, $J = 10.8, 2.8$ Hz, 2H), 7.47 (dd, $J = 11.2, 8.0$ Hz, 2H), 7.27-7.02 (m, 15H), 6.94 (dd, $J = 8.0, 3.2$ Hz, 2H), 6.21 (dd, $J = 10.4, 3.6$ Hz), 2.39 (s, 3H), 2.35 (s, 3H), 2.32 (s, 3H), 2.24 (s, 3H); ^{13}C NMR (100 MHz): δ 142.5 (dd, $J_{\text{C-P}} = 10.7, 2.6$ Hz), 142.3 (dd, $J_{\text{C-P}} = 9.0, 2.7$ Hz), 132.8-124.7 (m), 74.8 (dd, $J_{\text{C-P}} = 85.7, 7.8$ Hz), 21.5 (d, $J_{\text{C-P}} = 2.8$ Hz), 21.4 (d, $J_{\text{C-P}} = 8.7$ Hz); ^{31}P NMR (162 MHz): δ 36.41 (d, $J = 24.30$ Hz), 29.7 (d, $J = 24.30$ Hz); HRMS (ESI) m/z: $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{35}\text{H}_{34}\text{O}_3\text{P}_2$: 565.2056; found: 565.2054.



(bis(4-methoxyphenyl)phosphoryl)(phenyl)methyl bis(4-methoxyphenyl) phosphinate (54c).

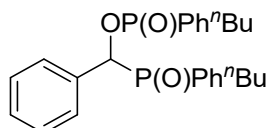
Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light white solid (88.0 mg, 75% yield) ^1H NMR (400 MHz): δ 7.85 (dd, $J = 7.2, 0.8$ Hz, 2H), 7.52 (d, $J = 8.8$ Hz, 1H), 7.49 (d, $J = 8.4$ Hz, 1H), 7.33 (d, $J = 8.8$ Hz, 2H), 7.31 (d, $J = 8.8$ Hz, 2H), 7.15-7.04 (m, 5H), 6.94 (dd, $J = 8.8, 2.0$ Hz, 2H), 6.82 (dd, $J = 8.8, 2.0$ Hz, 2H), 6.77 (dd, $J = 8.8, 2.4$ Hz, 2H), 6.65 (dd, $J = 8.8, 2.4$ Hz, 2H), 6.17 (dd, $J = 10.8, 3.6$ Hz, 1H), 3.82 (s, 3H), 3.79 (s, 3H), 3.76 (s, 3H), 3.70 (s, 3H); ^{13}C NMR (100 MHz): δ 162.4 (dd, $J_{\text{C-P}} = 21.8, 2.9$ Hz), 162.2 (dd, $J_{\text{C-P}} = 10.9, 3.0$ Hz), 134.2 (d, $J_{\text{C-P}} = 9.9$ Hz), 133.5 (d, $J_{\text{C-P}} = 11.1$ Hz), 133.2 (d, $J_{\text{C-P}} = 12.1$ Hz), 128.9 (d, $J_{\text{C-P}} = 4.7$ Hz), 128.2 (d, $J_{\text{C-P}} = 1.7$ Hz), 127.6 (d, $J_{\text{C-P}} = 0.8$ Hz), 122.0 (d, $J_{\text{C-P}} = 59.9$ Hz), 121.4 (d, $J_{\text{C-P}} = 42.6$ Hz), 119.5 (d, $J_{\text{C-P}} = 105.7$ Hz), 113.9-113.2 (m), 74.7 (dd, $J_{\text{C-P}} = 86.4, 7.7$ Hz), 55.1-55.0 (m); ^{31}P NMR (162 MHz): δ 36.23

(d, $J = 24.1$ Hz), 29.63 (d, $J = 24.1$ Hz); HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{35}H_{34}O_7P_2$: 629.1853; found: 629.1852.



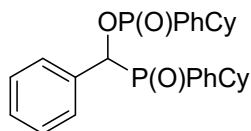
(bis(4-fluorophenyl)phosphoryl)(phenyl)methyl bis(4-fluorophenyl)phosphinate (55c).

Flash column chromatography (silica gel: DCM to DCM : EA = 2:1) afforded the title compound as a light white solid (98.6 mg, 85% yield). 1H NMR (400 MHz): δ 8.03-7.97 (m, 2H), 7.57-7.51 (m, 2H), 7.47-7.36 (m, 4H), 7.20-7.15 (m, 5H), 7.10-7.06 (m, 2H), 7.04-6.99 (m, 4H), 6.84 (dd, $J = 8.4, 2.8$ Hz, 1H), 6.82 (dd, $J = 8.8, 2.8$ Hz, 2H), 6.27 (dd, $J = 10.8, 1.2$ Hz, 1H); ^{13}C NMR (100 MHz): δ 166.3 (ddd, $J = 26.8, 20.0, 3.0$ Hz), 163.8 (ddd, $J = 26.8, 21.1, 3.2$ Hz), 134.8-133.5 (m), 131.8, 129.1-123.8 (m), 116.2-115.1 (m), 74.5 (dd, $J_{C-P} = 86.8, 7.3$ Hz); ^{31}P NMR (162 MHz): δ 33.94 (d, $J = 23.6$ Hz), 27.73 (d, $J = 23.9$ Hz); HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{31}H_{22}F_4O_3P_2$: 581.1053; found: 581.1053.



(butyl(phenyl)phosphoryl)(phenyl)methyl butyl(phenyl)phosphinate (56c).

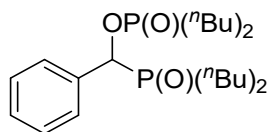
Flash column chromatography (silica gel: EA) afforded the title compound as a yellow oil (65.5 mg, 70% yield). The product is a mixture of isomers. 1H NMR (400 MHz): δ 7.58-6.88 (m, 15H), 6.10-5.76 (m, 1H), 2.05-0.87 (m, 18H); ^{13}C NMR (100 MHz): δ 165.4-165.1 (m), 133.4-128.2 (m), 69.5-66.7 (m), 26.2-22.7 (m), 13.9-13.3 (m); ^{31}P NMR (162 MHz): δ 51.23-49.24 (m), 41.79-40.36 (m); HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{27}H_{34}O_3P_2$: 469.2056; found: 469.2055.



(cyclohexyl(phenyl)phosphoryl)(phenyl)methyl cyclohexyl(phenyl)phosphinate (57c).

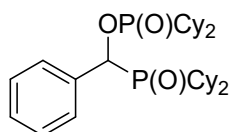
Flash column chromatography (silica gel: EA) afforded the title compound as a light yellow oil (72.8 mg, 70% yield). The product is a mixture of isomers. 1H NMR (400 MHz): δ 7.72-6.88 (m, 15H), 6.10-5.76 (m, 1H), 1.91-0.84 (m, 22H); ^{13}C NMR (100 MHz): δ 134.1-127.0 (m), 71.6 (dd, $J_{C-P} = 77.8, 8.0$ Hz), 39.2 (d, $J_{C-P} = 93.5$ Hz), 34.2 (d, $J_{C-P} = 67.7$ Hz), 26.3-23.9 (m); ^{31}P NMR (162 MHz): δ 50.68-

49.29 (m), 41.89-40.68 (m). HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{31}H_{38}O_3P_2$: 521.2369; found: 521.2369.



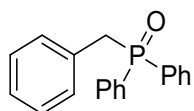
(dibutylphosphoryl)(phenyl)methyl dibutylphosphinate (58c).

The reaction temperature is 100 °C. Flash column chromatography (silica gel: EA) afforded the title compound as a light yellow oil (61.6 mg, 72% yield) 1H NMR (400 MHz): δ 7.48 (d, $J = 7.2$ Hz, 2H), 7.42-7.36 (m, 3H), 5.57 (dd, $J = 11.2, 4.4$ Hz, 1H) 1.85-1.00 (m, 24H), 0.95 (td, $J = 7.2, 7.2$ Hz, 3H), 0.92 (td, $J = 7.2, 7.2$ Hz, 3H), 0.87 (td, $J = 7.2, 7.2$ Hz, 3H), 0.66 (td, $J = 7.2, 7.2$ Hz, 3H); ^{13}C NMR (100 MHz): δ 134.0, 128.9 (d, $J_{C-P} = 1.2$ Hz), 128.6, 127.8 (d, $J_{C-P} = 4.1$ Hz), 72.3 (dd, $J_{C-P} = 76.0, 7.5$ Hz), 28.4 (d, $J_{C-P} = 82.4$ Hz), 27.8 (d, $J_{C-P} = 90.4$ Hz), 25.1-23.0 (m); ^{31}P NMR (162 MHz): δ 63.20 (d, $J = 17.5$ Hz), 48.04 (d, $J = 17.8$ Hz); HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{23}H_{42}O_3P_2$: 429.2682; found: 429.2681.



(dicyclohexylphosphoryl)(phenyl)methyl dicyclohexylphosphinate (59c).

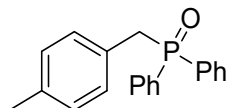
The reaction temperature is 100 °C. Flash column chromatography (silica gel: EA) afforded the title compound as a light yellow oil (74.5 mg, 72% yield) 1H NMR (400 MHz): δ 7.54 (d, $J = 7.6$ Hz, 2H), 7.41-7.33 (m, 3H), 5.74 (dd, $J = 10.4, 4.4$ Hz), 2.10-0.64 (m, 44H); ^{13}C NMR (100 MHz): δ 135.0, 128.7, 128.4, 128.3, 71.0 (dd, $J_{C-P} = 69.4, 8.5$ Hz), 37.9 (d, $J_{C-P} = 80.1$ Hz), 36.1 (d, $J_{C-P} = 87.9$ Hz), 35.7 (d, $J_{C-P} = 73.3$ Hz), 35.2 (d, $J_{C-P} = 96.8$ Hz), 26.9-25.0 (m); ^{31}P NMR (162 MHz): δ 63.23 (d, $J = 15.6$ Hz), 49.02 (d, $J = 15.7$ Hz); HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{31}H_{50}O_3P_2$: 533.3308; found: 533.3308.



Benzyldiphenylphosphine oxide (1d).

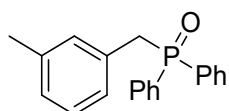
Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (52.0 mg, 89% yield) 1H NMR (400 MHz): δ 7.71-7.66 (m, 4H), 7.48-7.46 (m, 2H), 7.43-7.38 (m, 4H),

7.11-7.08 (m, 5 H), 3.64 (d, $J = 13.6$ Hz, 2H); ^{13}C NMR (100 MHz): δ 131.5 ($J_{\text{C-P}} = 98.6$ Hz), 131.6 (d, $J_{\text{C-P}} = 2.6$ Hz), 130.9 (d, $J_{\text{C-P}} = 9.1$ Hz), 130.8 (d, $J_{\text{C-P}} = 7.5$ Hz), 129.9 (d, $J_{\text{C-P}} = 5.2$ Hz), 128.3 (d, $J_{\text{C-P}} = 11.7$ Hz), 128.1 (d, $J_{\text{C-P}} = 2.3$ Hz), 126.5 (d, $J_{\text{C-P}} = 2.8$ Hz), 37.8 (d, $J_{\text{C-P}} = 66.1$ Hz); ^{31}P NMR (162 MHz): δ 29.8. This compound is known^[1].



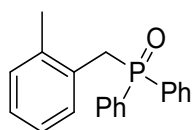
(4-methylbenzyl)diphenylphosphine oxide (2d).

Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (58.1 mg, 95% yield) ^1H NMR (400 MHz): δ 7.7-7.67 (m, 4H), 7.50-7.39 (m, 6H), 7.00-7.69 (m, 4H), 3.60 (d, $J = 13.6$ Hz, 2H), 2.24 (d, $J = 1.2$ Hz, 3H); ^{13}C NMR (100 MHz): δ 136.1 (d, $J_{\text{C-P}} = 3.0$ Hz), 132.2 (d, $J_{\text{C-P}} = 98.1$ Hz), 131.5 (d, $J_{\text{C-P}} = 2.5$ Hz), 131.0 (d, $J_{\text{C-P}} = 9.0$ Hz), 129.8 (d, $J_{\text{C-P}} = 5.2$ Hz), 128.9 (d, $J_{\text{C-P}} = 2.4$ Hz), 128.3 (d, $J_{\text{C-P}} = 11.6$ Hz), 127.7 (d, $J_{\text{C-P}} = 8.0$ Hz), 37.4 (d, $J_{\text{C-P}} = 66.6$ Hz), 20.8; ^{31}P NMR (162 MHz): δ 29.37. This compound is known^[1].



(3-methylbenzyl)diphenylphosphine oxide (3d).

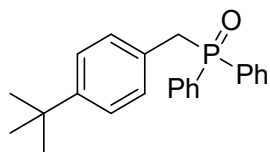
Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (56.3 mg, 92% yield) ^1H NMR (400 MHz): δ 7.71-7.67 (m, 4H), 7.50-7.40 (m, 6H), 7.07-6.85 (m, 4H), 3.60 (d, $J = 14.0$ Hz, 2H), 2.20 (s, 3H); ^{13}C NMR (100 MHz): δ 137.7, 132.2 (d, $J = 98.4$ Hz), 131.6 (d, $J = 2.4$ Hz), 131.0 (d, $J_{\text{C-P}} = 9.0$ Hz), 130.9 (d, $J = 5.3$ Hz), 130.8 (d, $J = 8.1$ Hz), 128.3 (d, $J = 11.6$ Hz), 128.0 (d, $J = 2.2$ Hz), 127.4 (d, $J = 2.8$ Hz), 127.0 (d, $J = 5.3$ Hz), 37.9 (d, $J = 66.3$ Hz), 21.1; ^{31}P NMR (162 MHz): δ 29.58. This compound is known^[1].



(2-methylbenzyl)diphenylphosphine oxide (4d).

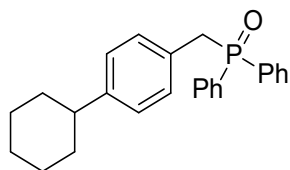
Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (56.9 mg, 92% yield) ^1H NMR (400 MHz): δ 7.69-7.65 (m, 4H), 7.51-7.40 (m, 6H), 7.08 (db, $J = 3.6$ Hz), 6.96-6.95 (m, 2H), 3.66 (d, $J = 14.0$ Hz, 2H), 2.13 (s, 3H); ^{13}C NMR (100 MHz): δ 137.3 (d, $J_{\text{C-P}} = 5.4$ Hz), 132.3 (d, $J_{\text{C-P}} = 97.8$ Hz), 131.7 (d, $J_{\text{C-P}} = 2.5$ Hz), 131.1 (d, $J_{\text{C-P}} = 9.0$ Hz), 130.6 (d, $J_{\text{C-P}} = 4.5$

Hz), 130.3 (d, $J_{C-P} = 2.4$ Hz), 129.5 (d, $J_{C-P} = 8.1$ Hz), 128.3 (d, $J_{C-P} = 11.5$ Hz), 126.8 (d, $J_{C-P} = 3.0$ Hz), 125.6 (d, $J_{C-P} = 2.7$ Hz), 35.1 (d, $J_{C-P} = 66.2$ Hz), 19.9; ^{31}P NMR (162 MHz): δ 29.67. This compound is known^[1].



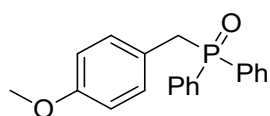
(4-(tert-butyl)benzyl)diphenylphosphine oxide (5d).

Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (65.4 mg, 92% yield) ^1H NMR (400 MHz): δ 7.72-7.67 (m, 4H), 7.52-7.41 (m, 6H), 7.20 (d, $J = 8.0$ Hz, 2H), 7.03 (dd, $J = 8.0, 2.0$ Hz, 2H), 3.62 (d, $J = 13.6$ Hz, 2H), 1.25 (s, 9H); ^{13}C NMR (100 MHz): δ 149.6, 132.3 (d, $J_{C-P} = 98.1$ Hz), 131.7 (d, $J_{C-P} = 2.5$ Hz), 131.1 (d, $J_{C-P} = 9.0$ Hz), 129.7 (d, $J_{C-P} = 5.1$ Hz), 128.4 (d, $J_{C-P} = 11.6$ Hz), 127.8 (d, $J_{C-P} = 8.0$ Hz), 125.3 (d, $J_{C-P} = 2.3$ Hz), 37.4 (d, $J_{C-P} = 66.6$ Hz), 34.2, 31.2; ^{31}P NMR (162 MHz): δ 29.74. This compound is known^[1].



(4-cyclohexylbenzyl)diphenylphosphine oxide (6d).

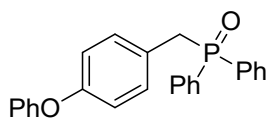
Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (69.5 mg, 93% yield) ^1H NMR (400 MHz, CDCl_3) δ 7.71-7.66 (m, 4H), 7.52-7.47 (m, 2H), 7.44-7.40 (m, 4H), 7.04-6.99 (m, 4H), 3.62 (d, $J = 13.6$ Hz, 2H), 2.41 (t, $J = 8.0$ Hz, 1H), 1.80 (d, $J = 8.8$ Hz, 4H), 1.73-1.70 (m, 1H), 1.40-1.28 (m, 4H), 1.27-1.19 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 146.54 (d, $J_{C-P} = 3.2$ Hz), 132.4 (d, $J_{C-P} = 98.2$ Hz), 131.67 (d, $J_{C-P} = 2.8$ Hz), 131.13 (d, $J_{C-P} = 9.2$ Hz), 129.95 (d, $J_{C-P} = 5.1$ Hz), 128.39 (d, $J_{C-P} = 11.6$ Hz), 128.16 (d, $J_{C-P} = 8.0$ Hz), 126.82 (d, $J_{C-P} = 2.5$ Hz), 44.06, 37.57 (d, $J_{C-P} = 66.5$ Hz), 34.32, 26.80, 26.07; ^{31}P NMR (162 MHz, CDCl_3) δ 29.63. This compound is known^[1].



(4-methoxybenzyl)diphenylphosphine oxide (7d).

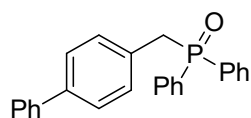
Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid

(59.2 mg, 92% yield) ^1H NMR (400 MHz): δ 7.71-7.66 (m, 4H), 7.50-7.41 (m, 6H), 7.01 (d, $J = 6.8$ Hz, 2H), 6.72 (d, $J = 8.4$ Hz, 2H), 3.72 (s, 3H), 3.59 (d, $J = 13.6$ Hz, 2H); ^{13}C NMR (100 MHz): δ 158.4, 132.2 (d, $J_{\text{C-P}} = 98.8$ Hz), 131.7 (d, $J_{\text{C-P}} = 2.5$ Hz), 131.1 (d, $J_{\text{C-P}} = 9.0$ Hz), 131.0 (b), 128.4 (d, $J_{\text{C-P}} = 11.5$ Hz), 122.7 (d, $J_{\text{C-P}} = 8.1$ Hz), 113.8, 55.1, 36.9 (d, $J_{\text{C-P}} = 67.1$ Hz); ^{31}P NMR (162 MHz): δ 29.80. This compound is known^[1].



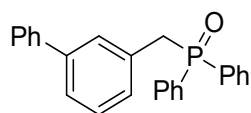
(4-phenoxybenzyl)diphenylphosphine oxide (8d).

Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (73.7 mg, 96% yield) ^1H NMR (400 MHz, CDCl_3) δ 7.73-7.68 (m, 4H), 7.54-7.49 (m, 2H), 7.47-7.42 (m, 4H), 7.33-7.28 (m, 2H), 7.10-7.04 (m, 3H), 6.96-6.92 (m, 2H), 6.84-6.81 (m, 2H), 3.63 (d, $J = 13.6$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 157.12, 156.07 (d, $J_{\text{C-P}} = 3.3$ Hz), 132.2 (d, $J_{\text{C-P}} = 98.3$ Hz), 131.81 (d, $J_{\text{C-P}} = 2.7$ Hz), 131.38 (d, $J_{\text{C-P}} = 5.1$ Hz), 131.13 (d, $J_{\text{C-P}} = 9.1$ Hz), 129.64, 128.49 (d, $J_{\text{C-P}} = 11.6$ Hz), 125.85 (d, $J_{\text{C-P}} = 8.1$ Hz), 123.15, 118.84 (d, $J_{\text{C-P}} = 2.7$ Hz), 118.73, 37.27 (d, $J_{\text{C-P}} = 66.5$ Hz); ^{31}P NMR (162 MHz, CDCl_3) δ 29.44. This compound is known^[1].



([1,1'-biphenyl]-4-ylmethyl)diphenylphosphine oxide (9d).

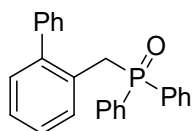
Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (44.1 mg, 60% yield) ^1H NMR (400 MHz, CDCl_3) δ 7.75-7.70 (m, 4H), 7.54-7.50 (m, 4H), 7.48-7.38 (m, 8H), 7.33-7.30 (m, 1H), 7.18-7.16 (m, 2H), 3.70 (d, $J = 13.2$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 140.62, 139.57 (d, $J_{\text{C-P}} = 3.0$ Hz), 132.2 (d, $J_{\text{C-P}} = 98.5$ Hz), 131.85 (d, $J_{\text{C-P}} = 2.5$ Hz), 131.16 (d, $J_{\text{C-P}} = 9.2$ Hz), 131.2 (d, $J_{\text{C-P}} = 9.2$ Hz), 130.1 (d, $J_{\text{C-P}} = 8.0$ Hz), 128.69, 128.5 (d, $J_{\text{C-P}} = 11.7$ Hz), 127.19, 127.07 (d, $J_{\text{C-P}} = 2.5$ Hz), 126.93, 37.73 (d, $J_{\text{C-P}} = 66.1$ Hz); ^{31}P NMR (162 MHz, CDCl_3) δ 29.64. This compound is known^[1].



([1,1'-biphenyl]-3-ylmethyl)diphenylphosphine oxide (10d).

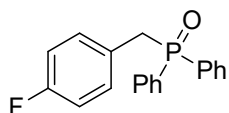
Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid

(45.6 mg, 62% yield) ^1H NMR (400 MHz, CDCl_3) δ 7.74-7.69 (m, 4H), 7.51-7.46 (m, 2H), 7.44-7.32 (m, 9H), 7.30-7.23 (m, 3H), 7.13-7.11 (m, 1H), 3.69 (d, $J = 13.6$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 140.97 (d, $J_{\text{C-P}} = 2.7$ Hz), 140.58, 132.1 (d, $J_{\text{C-P}} = 98.4$ Hz), 131.69 (d, $J_{\text{C-P}} = 2.7$ Hz), 131.47 (d, $J_{\text{C-P}} = 7.9$ Hz), 131.08 (d, $J_{\text{C-P}} = 9.0$ Hz), 128.96 (d, $J_{\text{C-P}} = 5.1$ Hz), 128.85 (d, $J_{\text{C-P}} = 5.2$ Hz), 128.67 (d, $J_{\text{C-P}} = 2.3$ Hz), 128.45 (d, $J_{\text{C-P}} = 1.1$ Hz), 128.36, 127.11, 126.94, 125.47 (d, $J_{\text{C-P}} = 2.9$ Hz), 38.07 (d, $J_{\text{C-P}} = 65.8$ Hz); ^{31}P NMR (162 MHz, CDCl_3) δ 29.52. This compound is known^[1].



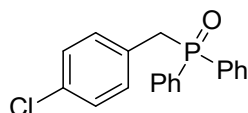
[(1,1'-biphenyl)-2-ylmethyl)diphenylphosphine oxide (11d).

Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (38.3 mg, 52% yield) ^1H NMR (400 MHz, CDCl_3) δ 7.61-7.58 (m, 1H), 7.49-7.41 (m, 6H), 7.37-7.29 (m, 7H), 7.28-7.21 (m, 2H), 7.12 -7.10 (m, 1H), 6.98-6.96 (m, 2H), 3.72 (d, $J = 14.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 142.57 (d, $J_{\text{C-P}} = 6.1$ Hz), 140.90, 132.4 (d, $J_{\text{C-P}} = 98.3$ Hz), 131.60 (d, $J_{\text{C-P}} = 2.7$ Hz), 131.08 (d, $J_{\text{C-P}} = 9.2$ Hz), 130.82 (d, $J_{\text{C-P}} = 4.3$ Hz), 130.22 (d, $J = 2.2$ Hz), 129.45, 128.73 (d, $J_{\text{C-P}} = 7.5$ Hz), 128.37 (d, $J_{\text{C-P}} = 11.7$ Hz), 128.10, 127.41 (d, $J_{\text{C-P}} = 2.7$ Hz), 126.90, 126.81 (d, $J_{\text{C-P}} = 2.8$ Hz), 34.32 (d, $J_{\text{C-P}} = 66.2$ Hz); ^{31}P NMR (162 MHz, CDCl_3) δ 29.89. This compound is known^[1].



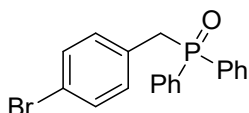
(4-fluorobenzyl)diphenylphosphine oxide (12d).

Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (32.9 mg, 53% yield) ^1H NMR (400 MHz): δ 7.71-7.66 (m, 4H), 7.51-7.49 (m, 2H), 7.46-7.42 (m, 4H), 7.09-7.05 (m, 2H), 6.87 (dd, $J = 8.4, 8.4$ Hz, 2H), 3.61 (d, $J = 13.2$ Hz, 2H); ^{13}C NMR (100 MHz): δ 161.8 (dd, $J_{\text{C-P}} = 243.9, J_{\text{C-F}} = 3.2$ Hz), 132.0 (d, $J_{\text{C-P}} = 97.8$ Hz), 131.8 (d, $J_{\text{C-P}} = 2.5$ Hz), 131.5 (dd, $J_{\text{C-P}} = 3.4, J_{\text{C-F}} = 3.4$ Hz), 131.0 (d, $J_{\text{C-P}} = 9.1$ Hz), 128.5 (d, $J_{\text{C-P}} = 9.6$ Hz), 126.7 (dd, $J_{\text{C-F}} = 7.9, J_{\text{C-P}} = 3.0$ Hz), 115.2 (dd, $J_{\text{C-P}} = 21.3, J_{\text{C-F}} = 2.2$ Hz), 37.1 (d, $J_{\text{C-P}} = 66.4$ Hz); ^{31}P NMR (162 MHz): δ 29.39 (d, $J = 4.7$ Hz). This compound is known^[1].



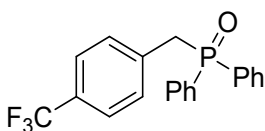
(4-chlorobenzyl)diphenylphosphine oxide (13d).

Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (35.6 mg, 56% yield) ^1H NMR (400 MHz): δ 7.71-7.66 (m, 4H), 7.54-7.51 (m, 2H), 7.47-7.43 (m, 4H), 7.15 (d, $J = 8.4$ Hz, 2H), 7.05-7.03 (m, 2H), 3.61 (d, $J = 13.6$ Hz, 2H); ^{13}C NMR (100 MHz): δ 132.8 (d, $J_{\text{C-P}} = 3.4$ Hz), 132.0 (d, $J_{\text{C-P}} = 98.9$ Hz), 131.9 (d, $J_{\text{C-P}} = 2.3$ Hz), 131.3 (d, $J_{\text{C-P}} = 5.1$ Hz), 131.0 (d, $J_{\text{C-P}} = 9.1$ Hz), 129.6 (d, $J_{\text{C-P}} = 7.9$ Hz), 128.6 (b), 128.5 (b), 37.4 (d, $J_{\text{C-P}} = 65.9$ Hz); ^{31}P NMR (162 MHz): δ 29.18. This compound is known^[1].



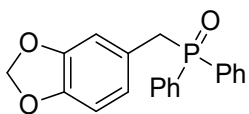
(4-bromobenzyl)diphenylphosphine oxide (14d).

Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (33.3 mg, 45% yield) ^1H NMR (400 MHz): δ 7.69 (dd, $J = 11.2, 7.2$ Hz, 4H), 7.55-7.51 (m, 2H), 7.48-7.43 (m, 4H), 7.30 (d, $J = 8.0$ Hz, 2H), 6.98 (dd, $J = 8.4, 2.0$ Hz, 2H), 3.59 (d, $J = 13.2$ Hz, 1H); ^{13}C NMR (100 MHz): δ 132.0 (d, $J_{\text{C-P}} = 2.6$ Hz), 131.9 (d, $J_{\text{C-P}} = 96.1$ Hz), 131.7 (d, $J_{\text{C-P}} = 5.2$ Hz), 131.4 (d, $J_{\text{C-P}} = 2.4$ Hz), 131.0 (d, $J_{\text{C-P}} = 9.1$ Hz), 130.1 (d, $J_{\text{C-P}} = 8.0$ Hz), 128.5 (d, $J_{\text{C-P}} = 11.7$ Hz), 120.9 (d, $J_{\text{C-P}} = 3.5$ Hz), 37.5 (d, $J_{\text{C-P}} = 65.7$ Hz); ^{31}P NMR (162 MHz): δ 29.09. This compound is known^[1].



Diphenyl(4-(trifluoromethyl)benzyl)phosphine oxide (15d).

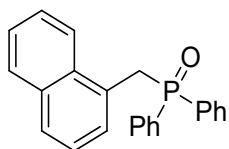
Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (21.6 mg, 30% yield) ^1H NMR (400 MHz): δ 7.72-7.67 (m, 4H), 7.55-7.51 (m, 2H), 7.48-7.43 (m, 6H), 7.22 (d, $J = 7.2$ Hz, 2H), 3.70 (d, $J = 13.6$ Hz); ^{13}C NMR (100 MHz): δ 135.4 (d, $J_{\text{C-P}} = 7.9$ Hz), 132.1 (d, $J_{\text{C-P}} = 2.5$ Hz), 131.8 (d, $J_{\text{C-P}} = 99.4$ Hz), 131.1 (d, $J_{\text{C-P}} = 9.1$ Hz), 130.0 (d, $J_{\text{C-P}} = 5.0$ Hz), 129.0 (q, $J_{\text{C-P}} = 29.3$ Hz), 128.6 (d, $J_{\text{C-P}} = 11.8$ Hz), 125.3 (b), 124.2 (q, $J_{\text{C-F}} = 271.1$ Hz), 37.9 (d, $J_{\text{C-P}} = 64.9$ Hz); ^{31}P NMR (162 MHz): δ 29.13. This compound is known^[1].



(benzo[d][1,3]dioxol-5-ylmethyl)diphenylphosphine oxide (16d).

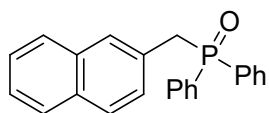
Flash column chromatography (silica gel: 25% DCM in EA) afforded the title compound as a white

solid (46.3 mg, 69% yield) ^1H NMR (400 MHz): δ 7.75-7.70 (m, 4H), 7.55-7.51 (m, 2H), 7.48-7.44 (m, 4H), 6.67-6.54 (m, 3H), 5.89 (s, 2H), 3.58 (d, $J = 13.2$ Hz, 2H); ^{13}C NMR (100 MHz): δ 147.5 (d, $J_{\text{C-P}} = 2.4$ Hz), 146.5 (d, $J_{\text{C-P}} = 2.9$ Hz), 132.2 (d, $J_{\text{C-P}} = 98.5$ Hz), 131.7 (d, $J_{\text{C-P}} = 2.2$ Hz), 131.0 (d, $J_{\text{C-P}} = 9.0$ Hz), 128.4 (d, $J_{\text{C-P}} = 11.6$ Hz), 124.4 (d, $J_{\text{C-P}} = 8.2$ Hz), 123.2 (d, $J_{\text{C-P}} = 6.1$ Hz), 110.4 (d, $J_{\text{C-P}} = 4.6$ Hz), 108.1 (d, $J_{\text{C-P}} = 2.5$ Hz), 100.8, 37.6 (d, $J_{\text{C-P}} = 48.9$ Hz); ^{31}P NMR (162 MHz): δ 29.34. This compound is known^[1].



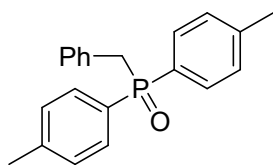
(naphthalen-1-ylmethyl)diphenylphosphine oxide (17d).

Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (60.9 mg, 89% yield) ^1H NMR (400 MHz): δ 7.92 (db, $J = 8.0$ Hz, 1H), 7.78 (d, $J = 7.6$ Hz, 1H), 7.71-7.66 (m, 5H), 7.47-7.46 (m, 2H), 7.40-7.36 (m, 6H), 7.26-7.24 (m, 2H), 4.12 (d, $J = 14.0$ Hz, 2H); ^{13}C NMR (100 MHz): δ 133.7 (d, $J_{\text{C-P}} = 1.7$ Hz), 132.4 (d, $J_{\text{C-P}} = 4.1$ Hz), 132.3 (d, $J_{\text{C-P}} = 98.9$ Hz), 131.7 (d, $J_{\text{C-P}} = 2.2$ Hz), 131.1 (d, $J_{\text{C-P}} = 9.0$ Hz), 128.6 (d, $J_{\text{C-P}} = 6.1$ Hz), 128.5, 128.4 (d, $J_{\text{C-P}} = 11.6$ Hz), 127.7 (d, $J_{\text{C-P}} = 10.2$ Hz), 127.6, 125.8, 125.5, 125.0 (d, $J_{\text{C-P}} = 6.1$ Hz), 124.2, 34.8 (d, $J_{\text{C-P}} = 66.4$ Hz); ^{31}P NMR (162 MHz): δ 29.73. This compound is known^[1].



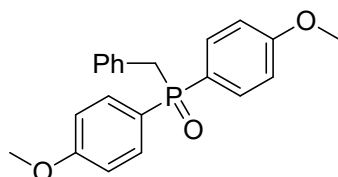
(naphthalen-2-ylmethyl)diphenylphosphine oxide (18d).

Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (53.3 mg, 78% yield) ^1H NMR (400 MHz): δ 7.76-7.65 (m, 7H), 7.56 (s, 1H), 7.52-7.48 (m, 2H), 7.45-7.39 (m, 6H), 7.23 (d, $J = 8.4$ Hz, 1H), 3.81 (d, $J = 13.6$ Hz); ^{13}C NMR (100 MHz): δ 133.3 (d, $J_{\text{C-P}} = 2.4$ Hz), 132.3 (d, $J_{\text{C-P}} = 98.4$ Hz), 132.2 (d, $J_{\text{C-P}} = 1.8$ Hz), 131.9 (d, $J_{\text{C-P}} = 2.4$ Hz), 131.8, 131.2 (d, $J_{\text{C-P}} = 9.1$ Hz), 129.0 (d, $J_{\text{C-P}} = 6.6$ Hz), 128.7 (d, $J_{\text{C-P}} = 8.2$ Hz), 128.5 (d, $J_{\text{C-P}} = 11.6$ Hz), 128.2 (d, $J_{\text{C-P}} = 4.2$ Hz), 127.9 (d, $J_{\text{C-P}} = 1.5$ Hz), 127.6 (d, $J_{\text{C-P}} = 6.9$ Hz), 126.0, 125.7, 38.3 (d, $J_{\text{C-P}} = 66.0$ Hz); ^{31}P NMR (162 MHz): δ 29.51. This compound is known^[1].



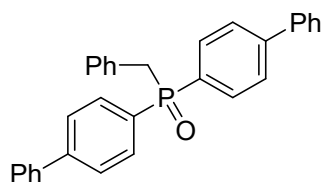
Benzyldi-p-tolylphosphine oxide (19d).

Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (46.7 mg, 73% yield). ^1H NMR (400 MHz): δ 7.58-7.53 (m, 4H), 7.23-7.22 (m, 4H), 7.18-7.09 (m, 5H), 3.61 (d, $J = 14.0$ Hz, 2H), 2.36 (s, 6H); ^{13}C NMR (100 MHz): δ 142.0 (d, $J_{\text{C-P}} = 2.6$ Hz), 131.3 (d, $J_{\text{C-P}} = 7.9$ Hz), 131.0 (d, $J_{\text{C-P}} = 9.4$ Hz), 130.0 (d, $J_{\text{C-P}} = 5.3$ Hz), 129.0 (d, $J_{\text{C-P}} = 100.9$ Hz), 129.0 (d, $J_{\text{C-P}} = 12.0$ Hz), 128.2 (d, $J_{\text{C-P}} = 2.5$ Hz), 126.5 (d, $J_{\text{C-P}} = 2.8$ Hz), 38.1 (d, $J_{\text{C-P}} = 66.3$ Hz), 21.4; ^{31}P NMR (162 MHz): δ 29.92. This compound is known^[1].



Benzylbis(4-methoxyphenyl)phosphine oxide (20d).

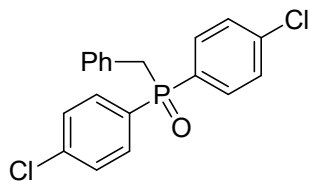
Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (45.7 mg, 65% yield). ^1H NMR (400 MHz): δ 7.51-7.46 (m, 4H), 7.07-7.00 (m, 5H), 6.84-6.82 (m, 4H), 3.71 (s, 6H), 3.50 (d, $J = 14.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 162.2 (d, $J_{\text{C-P}} = 2.7$ Hz), 133.0 (d, $J_{\text{C-P}} = 10.4$ Hz), 131.6 (d, $J_{\text{C-P}} = 7.8$ Hz), 130.1 (d, $J_{\text{C-P}} = 5.2$ Hz), 128.3 (d, $J_{\text{C-P}} = 2.3$ Hz), 126.6 (d, $J_{\text{C-P}} = 2.8$ Hz), 123.7 (d, $J_{\text{C-P}} = 104.9$ Hz), 113.9 (d, $J_{\text{C-P}} = 12.6$ Hz), 55.3, 38.6 (d, $J_{\text{C-P}} = 66.9$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 29.63. This compound is known^[1]. This compound is known^[1].



Di([1,1'-biphenyl]-4-yl)(benzyl)phosphine oxide (21d).

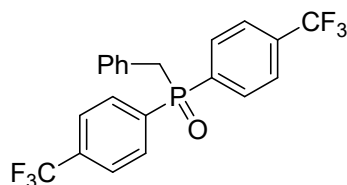
Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (55.9 mg, 63% yield). ^1H NMR (400 MHz): δ 7.81-7.76 (m, 4H), 7.69-7.66 (m, 4H), 7.61-7.59 (m, 4H), 7.48-7.44 (m, 4H), 7.41-7.36 (m, 2H), 7.22-7.16 (m, 5H), 3.72 (d, $J = 14.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 144.5 (d, $J_{\text{C-P}} = 2.7$ Hz), 139.8, 131.7 (d, $J_{\text{C-P}} = 9.4$ Hz), 131.1 (d, $J_{\text{C-P}} = 7.9$ Hz), 130.8

(d, $J_{C-P} = 99.7$ Hz), 130.1 (d, $J_{C-P} = 5.1$ Hz), 128.9, 128.4 (d, $J_{C-P} = 2.3$ Hz), 128.1, 127.2, 127.1, 126.8 (d, $J_{C-P} = 2.8$ Hz), 38.2 (d, $J_{C-P} = 66.4$ Hz); ^{31}P NMR (162 MHz, CDCl_3): 29.64. This compound is known^[1].



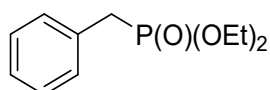
Benzylbis(4-chlorophenyl)phosphine oxide (22d).

Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (43.2 mg, 60% yield). ^1H NMR (400 MHz): δ 7.62-7.57 (m, 4H), 7.44-7.41 (m, 4H), 7.21-7.20 (m, 3H), 7.10-7.08 (m, 2H), 3.63 (d, $J = 14.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 138.6 (d, $J_{C-P} = 3.3$ Hz), 132.4 (d, $J_{C-P} = 10.0$ Hz), 130.3 (d, $J_{C-P} = 99.9$ Hz), 130.3 (d, $J_{C-P} = 8.1$ Hz), 130.0 (d, $J_{C-P} = 5.3$ Hz), 128.5 (d, $J_{C-P} = 2.3$ Hz), 127.1 (d, $J_{C-P} = 2.9$ Hz), 37.9 (d, $J_{C-P} = 67.1$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 28.56. This compound is known^[1].



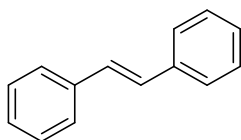
Benzylbis(4-(trifluoromethyl)phenyl)phosphine oxide (23d).

Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a white solid (45.4mg, 53% yield). ^1H NMR (400 MHz): δ 7.86-7.81 (m, 4H), 7.73-7.70 (m, 4H), 7.27-7.20 (m, 3H), 7.13-7.10 (m, 2H), 3.72 (d, $J = 13.6$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 135.9 (d, $J_{C-P} = 96.0$ Hz), 134.0 (qd, $J_{C-F} = 65.5$ Hz, $J_{C-P} = 5.4$ Hz), 131.6 (d, $J_{C-P} = 9.3$ Hz), 130.0 (d, $J_{C-P} = 5.3$ Hz), 129.8 (d, $J_{C-P} = 8.2$ Hz), 128.7 (d, $J_{C-P} = 2.6$ Hz), 127.3 (d, $J_{C-P} = 3.1$ Hz), 125.6-125.4 (m), 123.4 (q, $J_{C-F} = 271.1$ Hz), 37.6 (d, $J_{C-P} = 67.0$ Hz); ^{31}P NMR (162 MHz, CDCl_3): 29.58; HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{21}\text{H}_{15}\text{F}_6\text{OP}$: 492.0837; found: 492.0838.



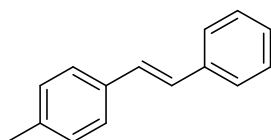
Diethyl benzylphosphonate (24d).

Flash column chromatography (silica gel: 50% PE in EA) afforded the title compound as a light yellow solid (38.7 mg, 85% yield). ^1H NMR (400 MHz): δ 7.313-7.243 (m, 5H), 4.04-3.97 (m, 4H), 3.15 (d, J = 21.6 Hz, 2H), 1.24 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz): δ 131.4 (d, $J_{\text{C-P}}$ = 9.1 Hz), 129.7 (d, $J_{\text{C-P}}$ = 6.5 Hz), 128.4 (d, $J_{\text{C-P}}$ = 2.9 Hz), 126.8 (d, $J_{\text{C-P}}$ = 3.5 Hz), 62.1 (d, $J_{\text{C-P}}$ = 6.7 Hz), 33.7 (d, $J_{\text{C-P}}$ = 137.3 Hz), 16.2 (d, $J_{\text{C-P}}$ = 14.1 Hz); ^{31}P NMR (162 MHz): δ 26.53. This compound is known^[1].



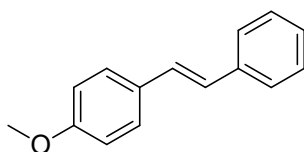
(E)-1,2-diphenylethene (1e).

Flash column chromatography (silica gel: PE) afforded the title compound as a light white solid (25.2 mg, 70% yield). ^1H NMR (400 MHz): δ 7.49 (db, J = 7.2 Hz, 4H), 7.35-7.31 (m, 4H), 7.25-7.23 (m, 2H), 7.08 (s, 2H); ^{13}C NMR (100 MHz): δ 137.2, 128.6, 127.5, 126.4. This compound is known^[2].



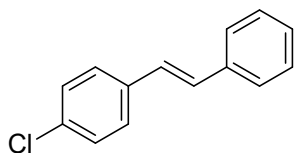
(E)-1-methyl-4-styrylbenzene (2e).

Flash column chromatography (silica gel: PE) afforded the title compound as a light white solid (27.9 mg, 72% yield). ^1H NMR (400 MHz): δ 7.51-7.49 (m, 2H), 7.41 (d, J = 8.0 Hz, 2H), 7.36-7.32 (m, 2H), 7.26-7.22 (m, 1H), 7.16 (d, J = 8.0 Hz, 2H), 7.07 (d, J = 2.8 Hz, 2H), 2.35 (s, 3H); ^{13}C NMR (100 MHz): δ 137.4, 134.5, 129.3, 128.6, 128.5, 127.6, 127.3, 126.4, 126.3, 21.2. This compound is known^[2].



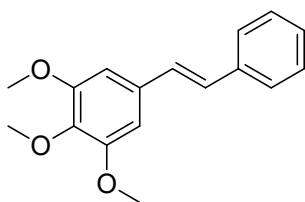
(E)-1-methoxy-4-styrylbenzene (3e).

Flash column chromatography (silica gel: PE) afforded the title compound as a light white solid (22.7 mg, 72% yield). ^1H NMR (400 MHz): δ 7.49-7.43 (m, 4H), 7.35-7.31 (m, 2H), 7.24-7.20 (m, 1H), 7.08-6.94 (m, 2H), 6.91-6.88 (m, 2H), 3.80 (s, 3H); ^{13}C NMR (100 MHz): δ 159.2, 137.6, 130.1, 128.6, 128.1, 127.6, 127.1, 126.5, 126.2, 114.1, 55.2. This compound is known^[2].



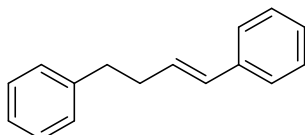
(E)-1-chloro-4-styrylbenzene (4e).

Flash column chromatography (silica gel: PE) afforded the title compound as a light white solid (19.3 mg, 46% yield). ^1H NMR (400 MHz): δ 7.50-7.48 (m, 2H), 7.43-7.40 (m, 2H), 7.37-7.33 (m, 2H), 7.32-7.30 (m, 3H), 7.28-7.23 (m, 1H), 7.05 (d, $J = 2.8$ Hz, 2H); ^{13}C NMR (100 MHz): δ 136.9, 135.8, 133.1, 129.2, 128.8, 128.7, 127.8, 127.6, 127.3, 126.5. This compound is known^[2].



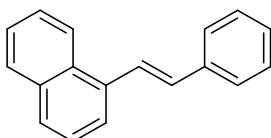
(E)-1,2,3-trimethoxy-5-styrylbenzene (5e).

Flash column chromatography (silica gel: PE) afforded the title compound as a light white solid (41.8 mg, 77% yield). ^1H NMR (400 MHz): δ 7.52-7.50 (m, 2H), 7.38-7.34 (m, 2H), 7.27-7.25 (m, 1H), 7.02 (d, $J = 2.8$ Hz, 2H), 6.74 (s, 2H), 3.91 (s, 6H), 3.87 (s, 3H); ^{13}C NMR (100 MHz): δ 153.3, 137.9, 137.1, 133.0, 128.6, 128.5, 128.1, 127.5, 126.3, 103.5, 60.9, 56.0. This compound is known^[3].



(E)-but-1-ene-1,4-diyl dibenzene (6e).

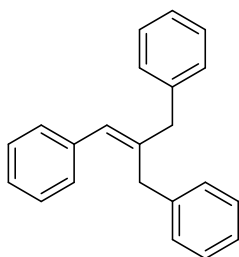
Flash column chromatography (silica gel: PE) afforded the title compound as a light white solid (34.0 mg, 74% yield). ^1H NMR (400 MHz): δ 7.34-7.26 (m, 6H), 7.23-7.17 (m, 4H), 6.42 (d, $J = 15.6$ Hz, 1H), 6.29-6.21 (m, 1H), 2.79 (t, $J = 7.6$ Hz, 2H), 2.56-2.50 (m, 2H); ^{13}C NMR (100 MHz): δ 141.7, 137.7, 130.3, 129.9, 128.4 (b), 128.3, 126.9, 125.9, 125.8, 35.8, 34.8. This compound is known^[4].



(E)-1-styrylnaphthalene (7e).

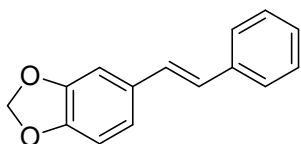
Flash column chromatography (silica gel: PE) afforded the title compound as a light white solid (34.0 mg, 74% yield). ^1H NMR (400 MHz): δ 7.83-7.78 (m, 4H), 7.72 (dd, $J = 8.8, 1.6$ Hz, 1H), 7.56-7.54 (m,

2H), 7.48-7.41 (m, 2H), 7.39-7.35 (m, 2H), 7.29-7.19 (m, 3H); ^{13}C NMR (100 MHz): δ 137.3, 134.7, 133.6, 133.0, 129.0, 128.7, 128.7, 128.2, 127.9, 127.6, 127.6, 126.6, 126.5, 126.3, 125.8, 123.4. This compound is known^[2].



(2-benzylprop-1-ene-1,3-diyl)dibenzene (8e).

Flash column chromatography (silica gel: PE) afforded the title compound as a light white solid (25.5 mg, 45% yield). ^1H NMR (400 MHz): δ 7.49-7.44 (m, 6H), 7.36-7.32 (m, 3H), 7.24-7.23 (m, 2H), 7.08-6.95 (m, 3H), 6.90-6.88 (m, 3H), 3.81 (s, 4H); ^{13}C NMR (100 MHz): δ 159.2, 137.6, 130.1, 128.6, 128.1, 127.6, 127.1, 126.5, 126.2, 114.1, 55.2. This compound is known^[5].



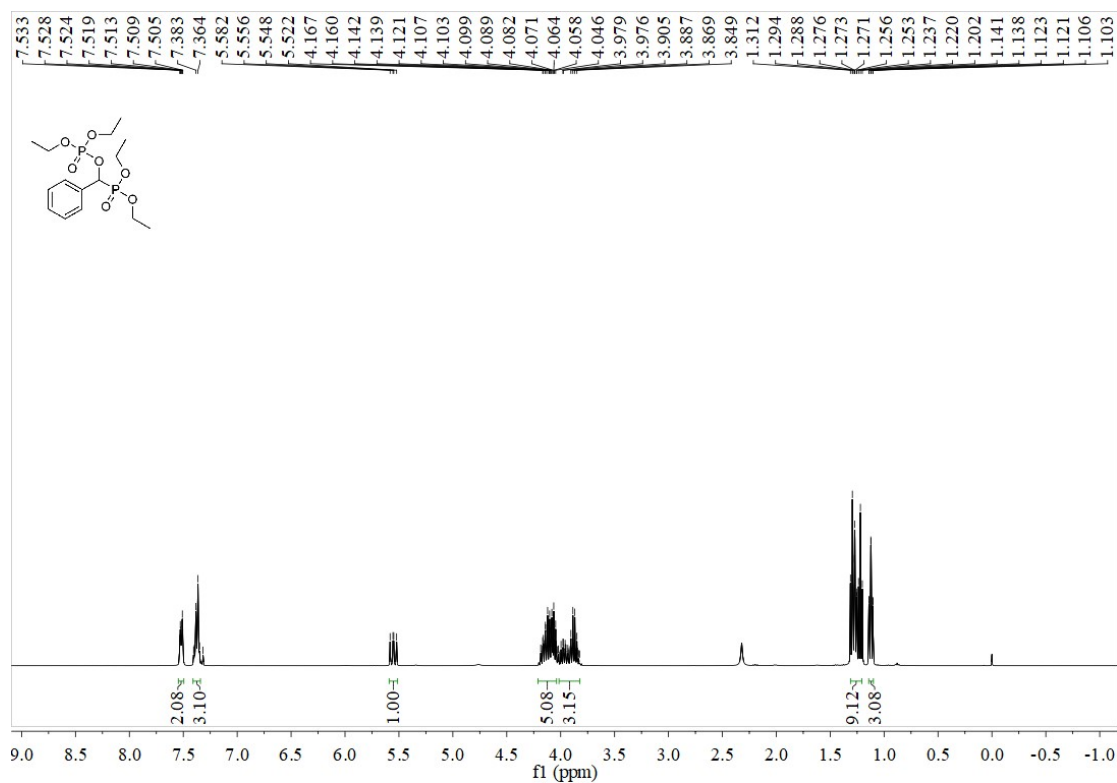
(E)-5-styrylbenzo[d][1,3]dioxole (9e).

Flash column chromatography (silica gel: PE) afforded the title compound as a light white solid (34.0 mg, 74% yield). ^1H NMR (400 MHz): δ 7.48-7.46 (m, 2H), 7.35-7.32 (m, 2H), 7.25-7.23 (m, 1H), 7.06 (d, $J = 1.6$ Hz, 1H), 7.04-7.00 (m, 1H), 6.94-6.90 (m, 2H), 6.79 (d, $J = 8.0$ Hz, 1H), 5.95 (s, 2H); ^{13}C NMR (100 MHz): δ 148.1, 147.2, 137.3, 131.8, 128.6, 128.3, 127.3, 126.9, 126.2, 126.1, 121.4, 108.3, 105.5, 101.0. This compound is known^[3].

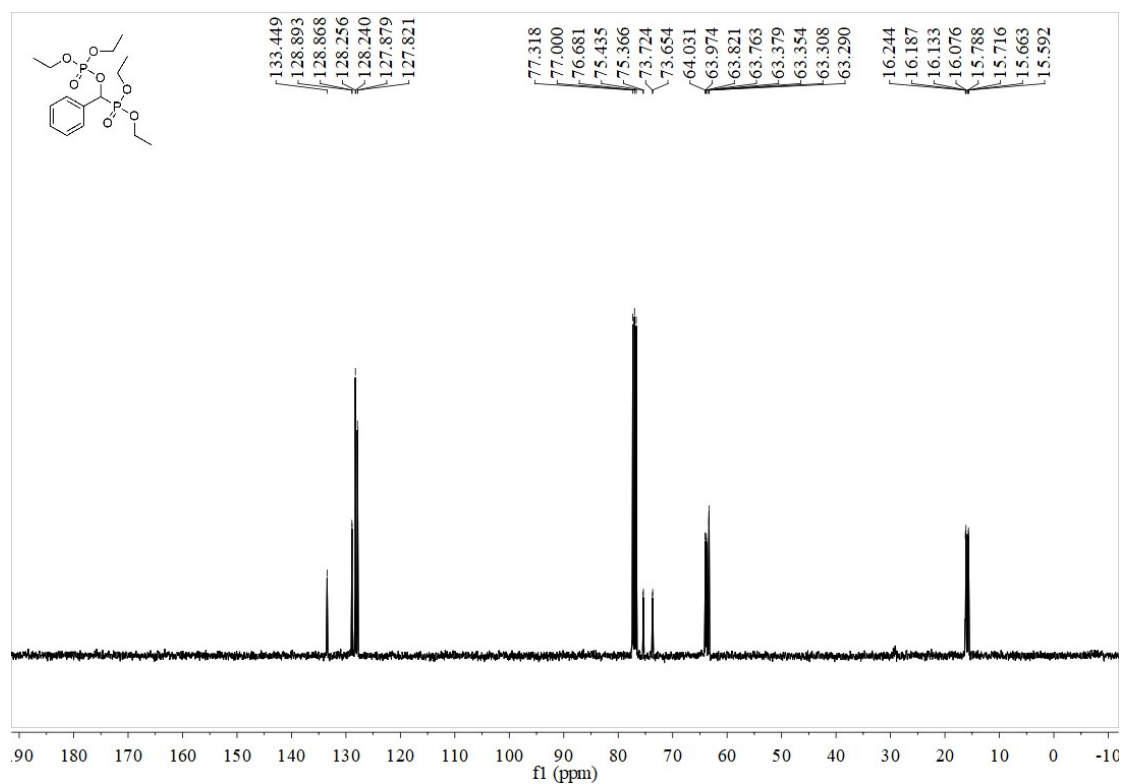
Reference

- (1) L. Liu, Y. Tang, K. Wang, T. Huang, T. Chen, *J. Org. Chem.* **2021**, *86*, 4159–4170.
- (2) J. Luo, Y. Liang, M. Montag, Y. Diskin-Posner, L. Avram, D. Milstein, *J. Am. Chem. Soc.* **2022**, *144*, 13266–13275.
- (3) J. McNulty, P. Das, *Eur. J. Org. Chem.* **2009**, *24*, 4031-4035.
- (4) L. Chen, Y. Hisaeda, H. Shimakoshi, *Adv. Syn. Cat.* **2019**, *361*, 2877-2884.
- (5) S. G. Mahamulkar, I. Cisarova, U. Jahna, *Adv. Syn. Cat.* **2014**, *4*, 793-799.

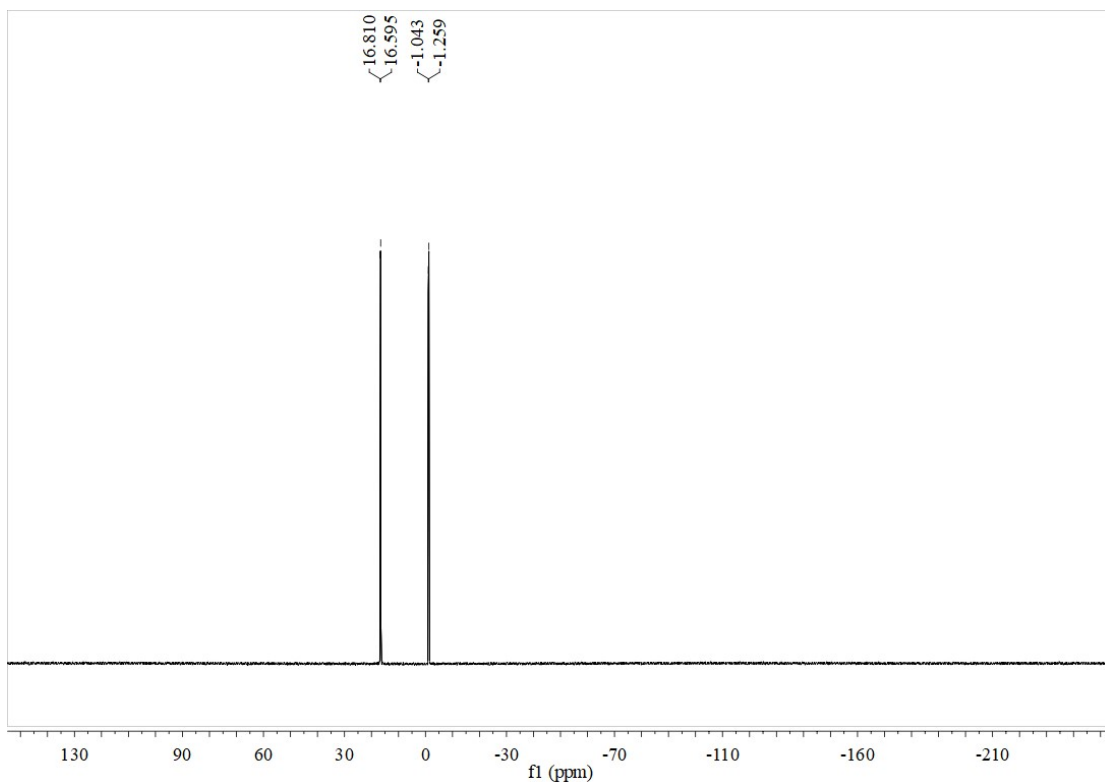
7. Copies of ^1H NMR, ^{13}C NMR and ^{31}P NMR spectra of products



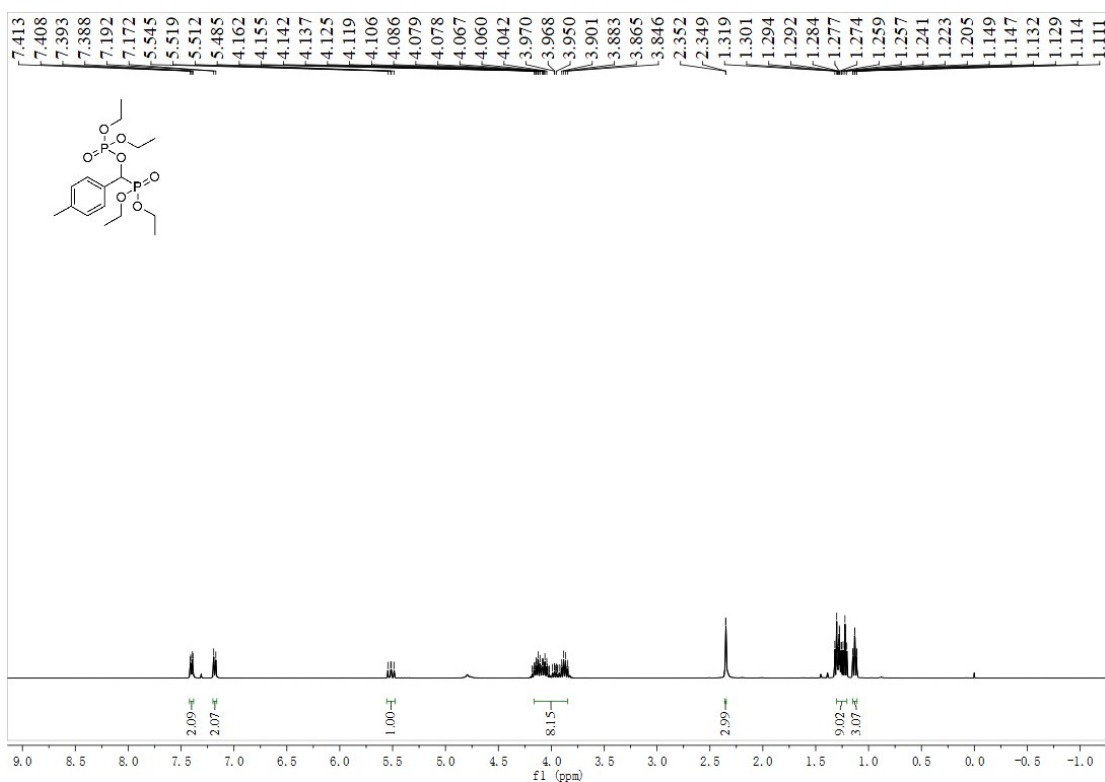
^1H NMR spectra of (diethoxyphosphoryl)(phenyl)methyl diethyl phosphate (**1c**).



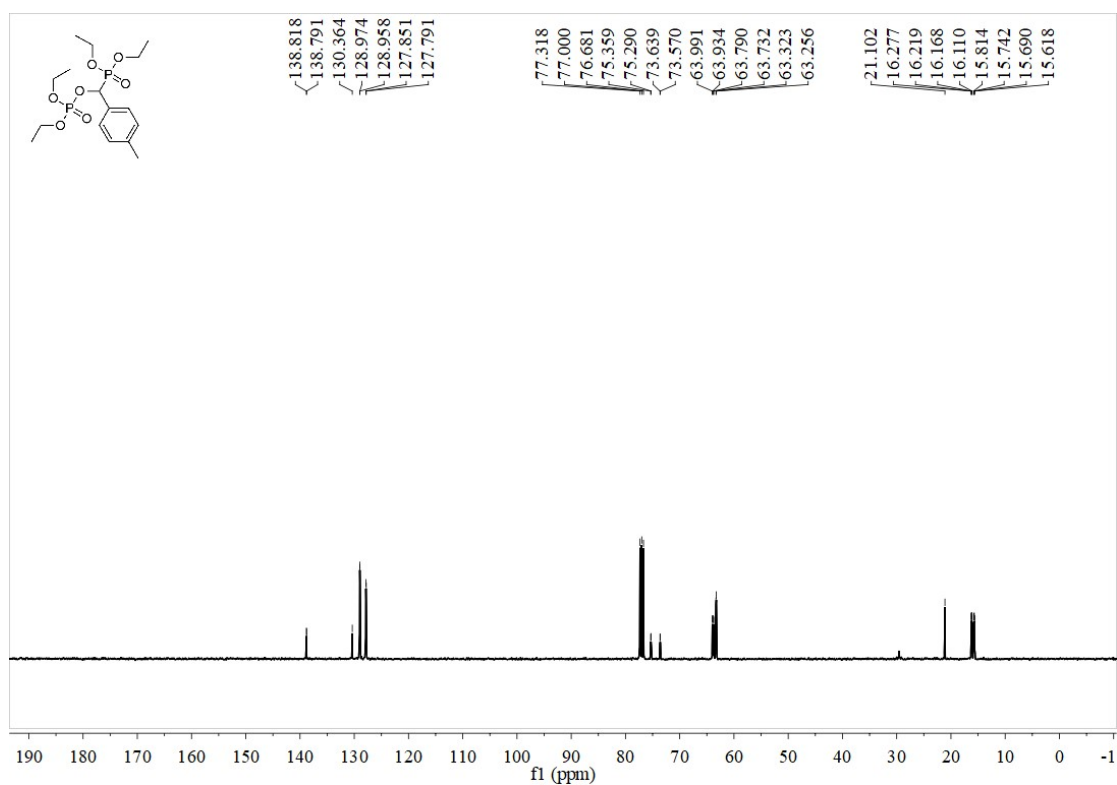
^{13}C NMR spectra of (diethoxyphosphoryl)(phenyl)methyl diethyl phosphate (**1c**).



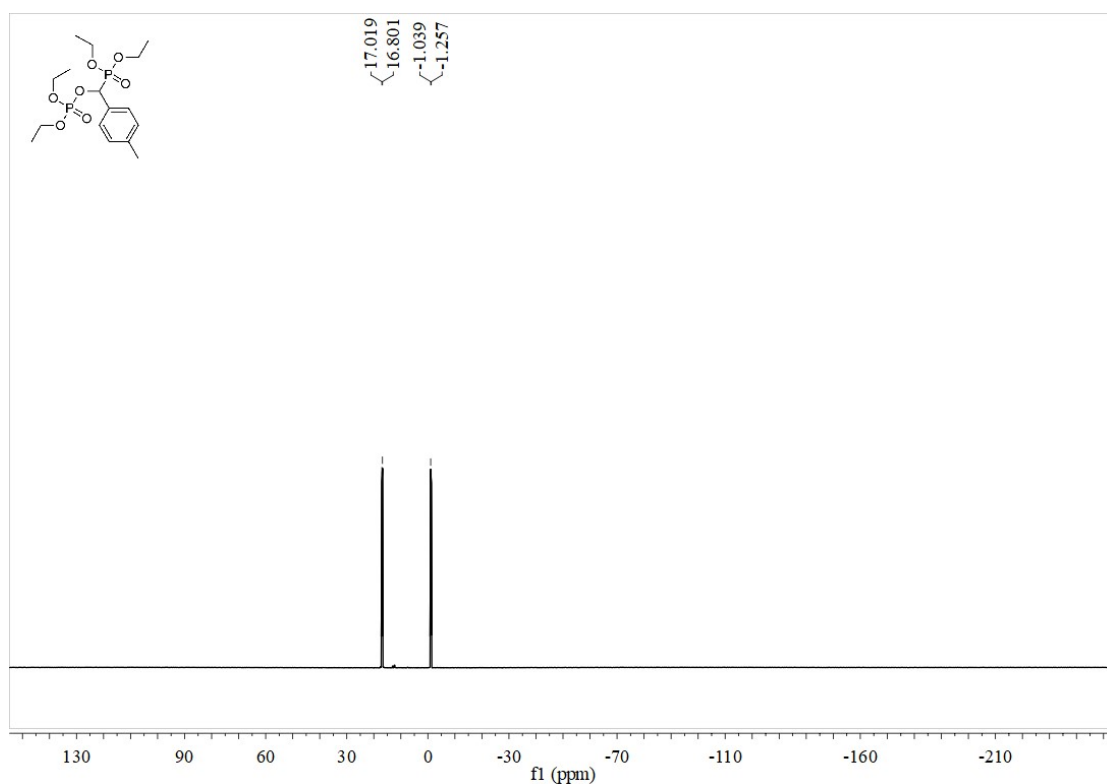
^{31}P NMR spectra of (diethoxyphosphoryl)(phenyl)methyl diethyl phosphate (**1c**)



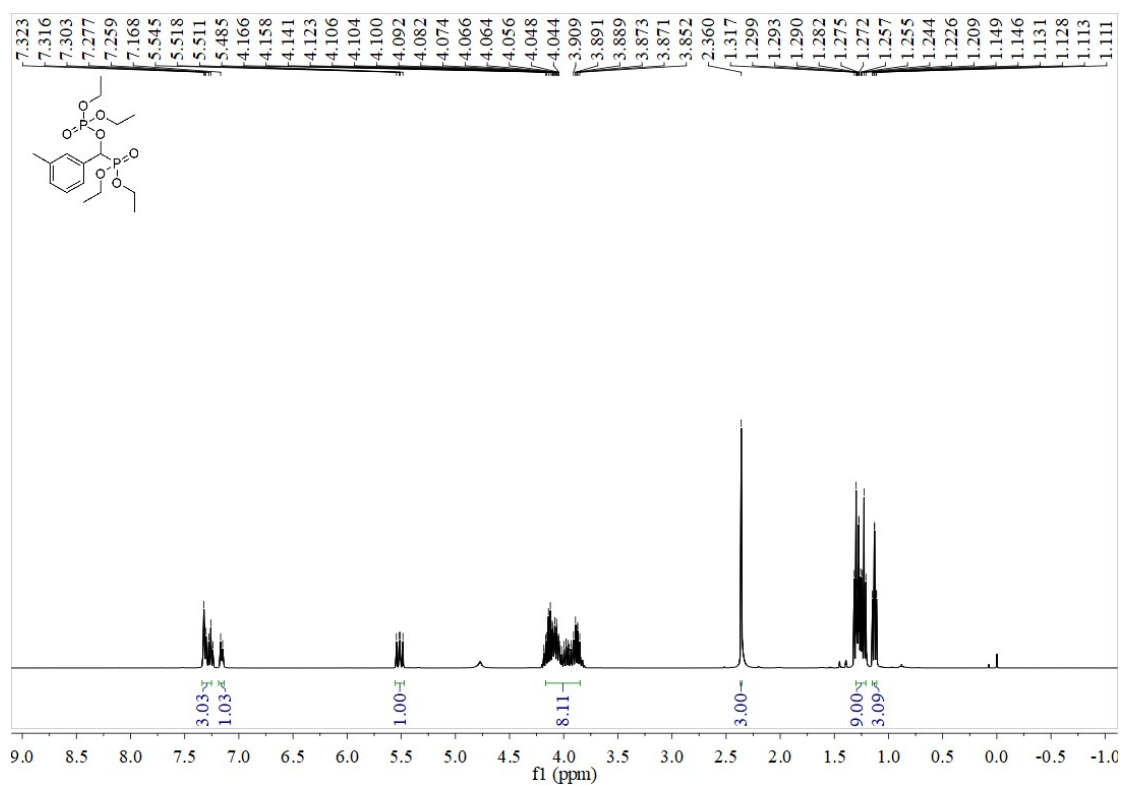
^1H NMR spectra of (diethoxyphosphoryl)(p-tolyl)methyl diethyl phosphate (**2c**).



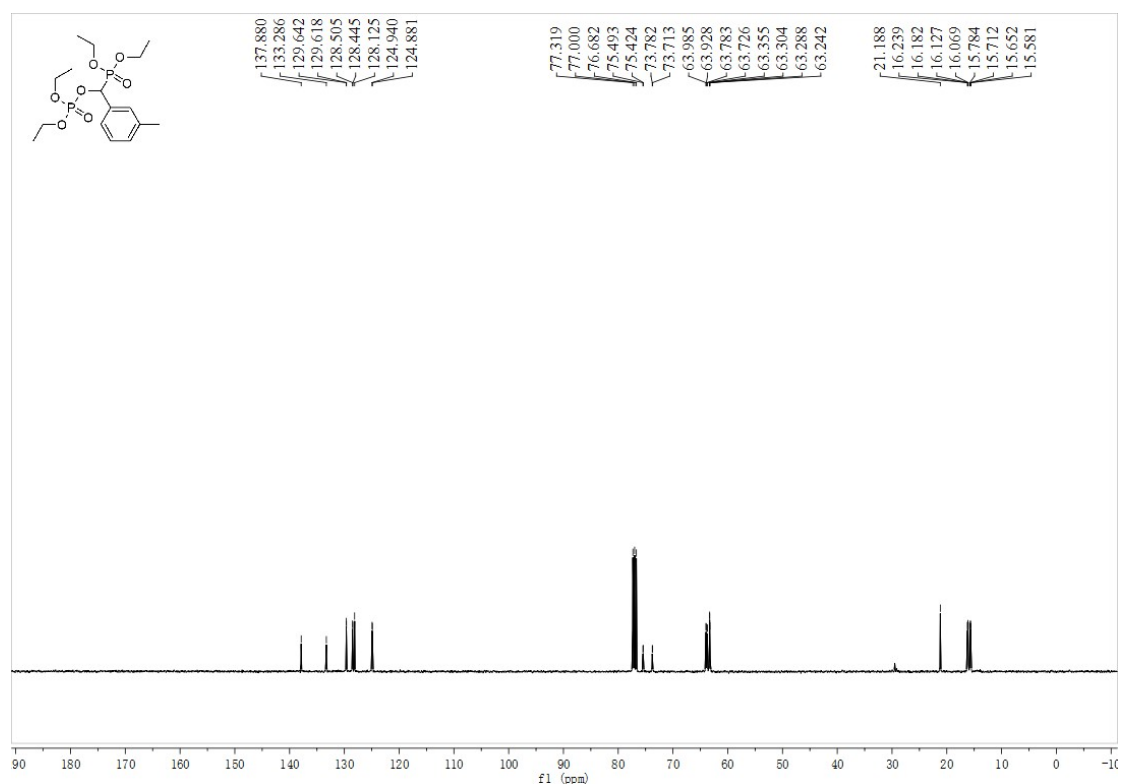
¹³C NMR spectra of (diethoxyphosphoryl)(p-tolyl)methyl diethyl phosphate (**2c**).



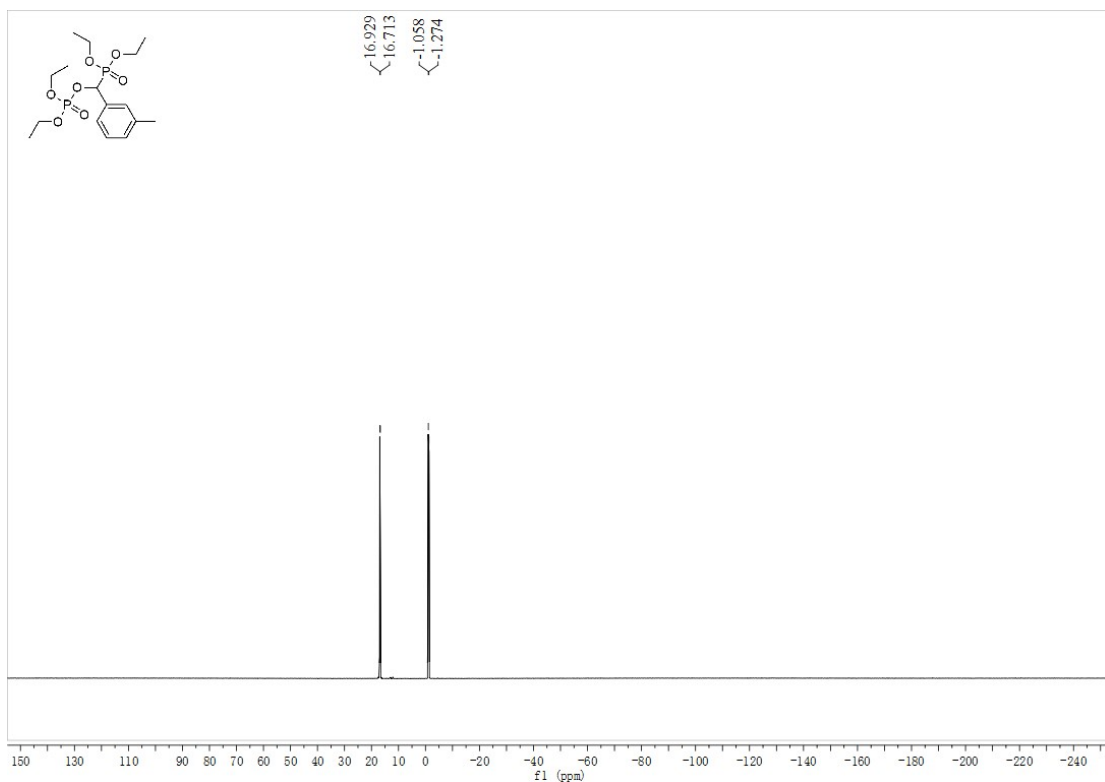
³¹P NMR spectra of (diethoxyphosphoryl)(p-tolyl)methyl diethyl phosphate (**2c**).



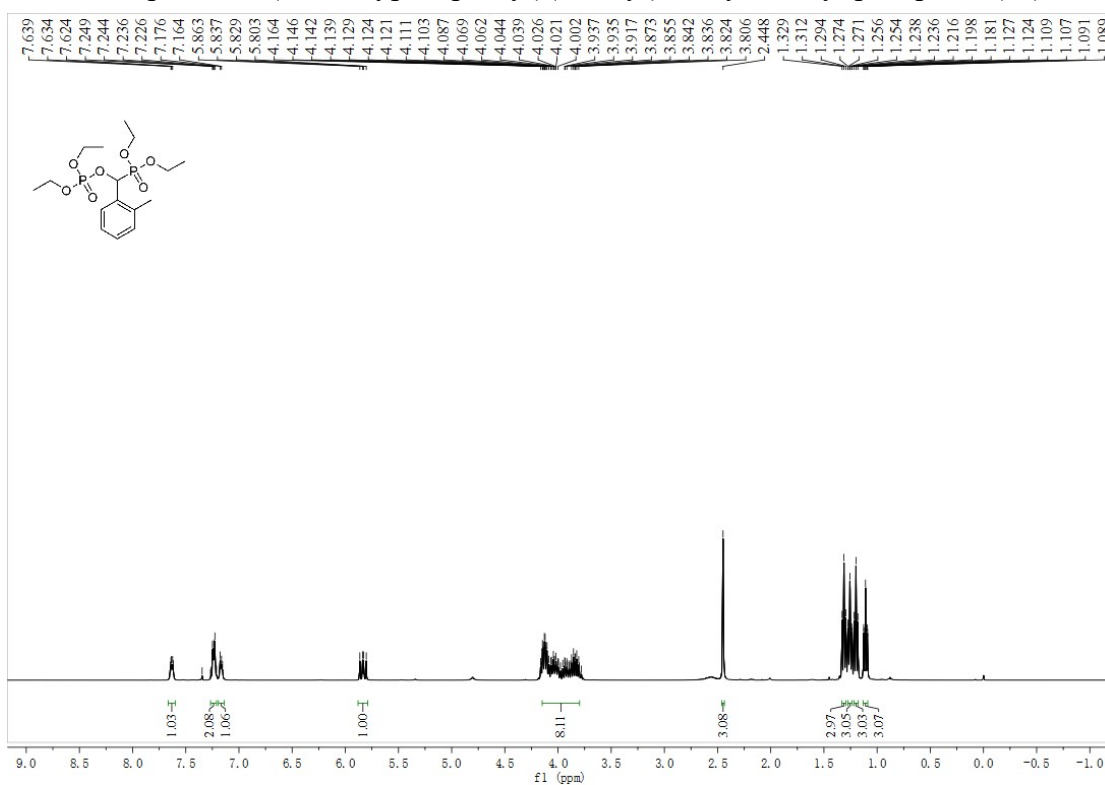
¹H NMR spectra of (diethoxyphosphoryl)(m-tolyl)methyl diethyl phosphate (3c).



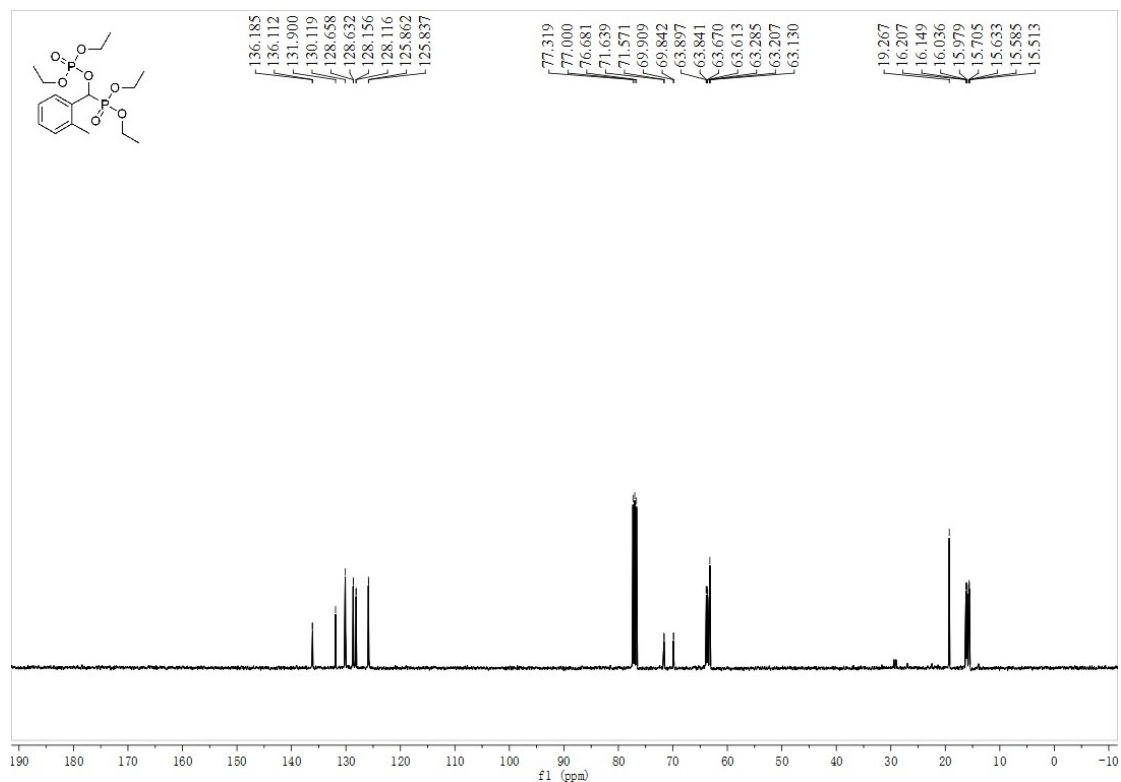
¹³C NMR spectra of (diethoxyphosphoryl)(m-tolyl)methyl diethyl phosphate (3c).



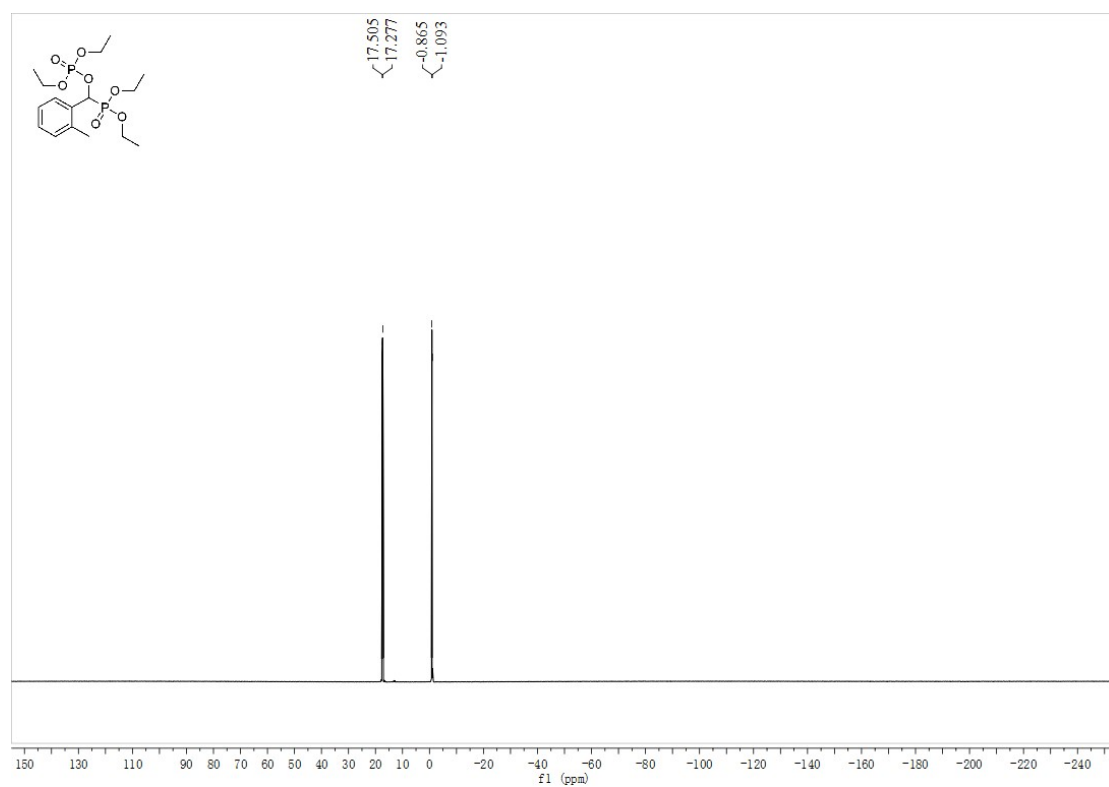
^{31}P NMR spectra of (diethoxyphosphoryl)(m-tolyl)methyl diethyl phosphate (**3c**).



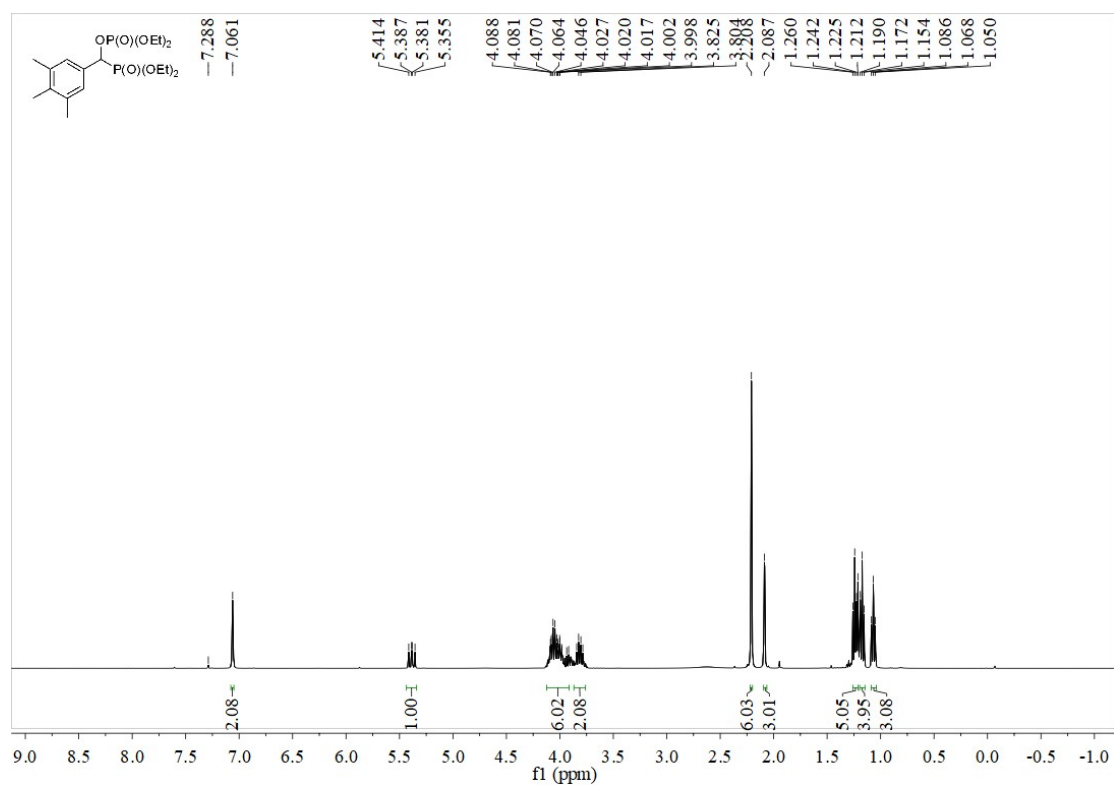
^1H NMR spectra of (diethoxyphosphoryl)(o-tolyl)methyl diethyl phosphate (**4c**).



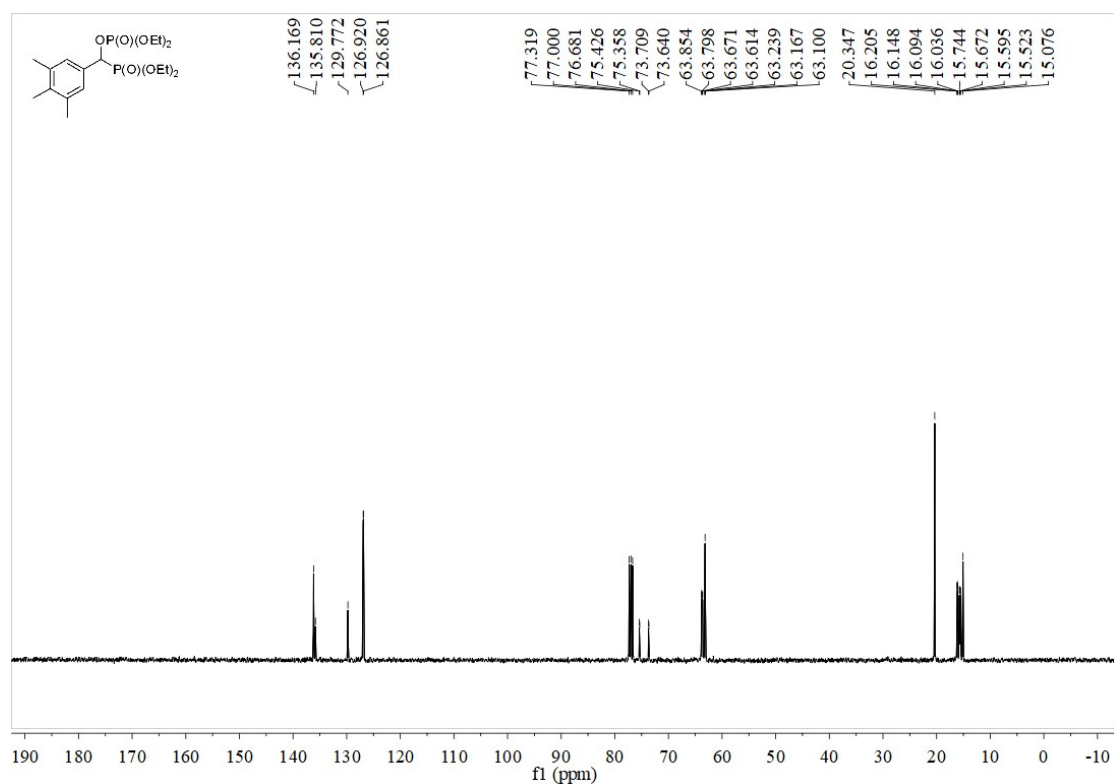
^{13}C NMR spectra of (diethoxyphosphoryl)(o-tolyl)methyl diethyl phosphate (**4c**).



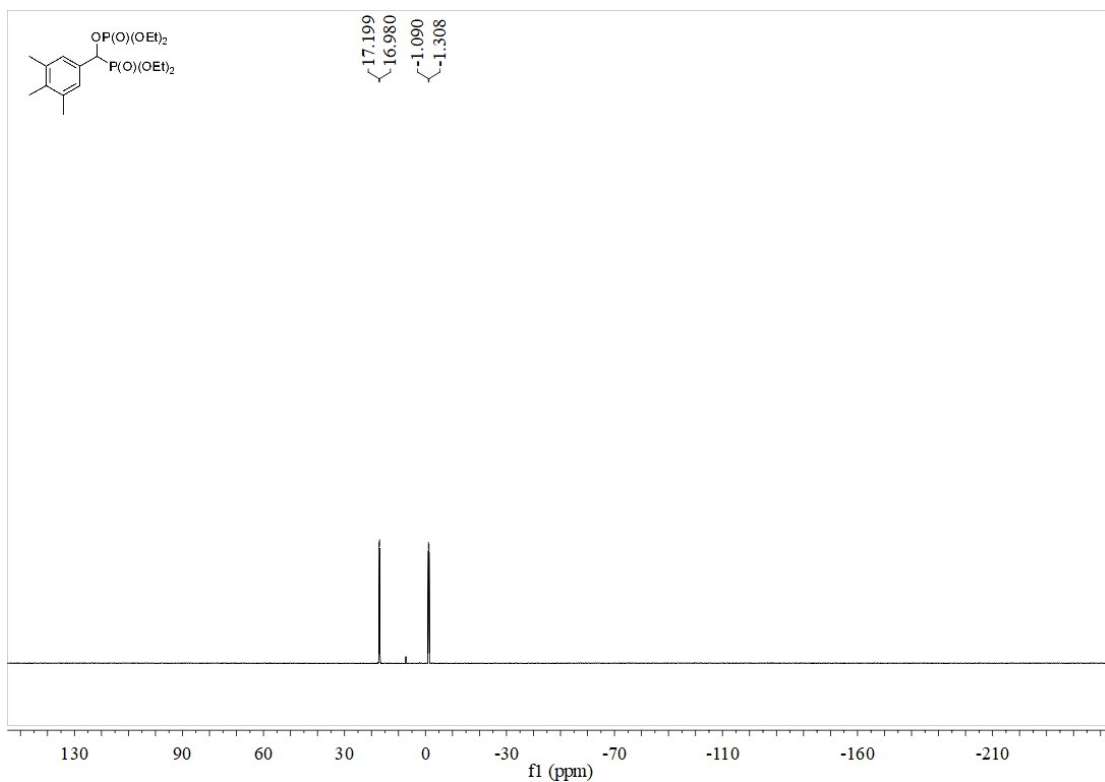
^{31}P NMR spectra of (diethoxyphosphoryl)(o-tolyl)methyl diethyl phosphate (**4c**).



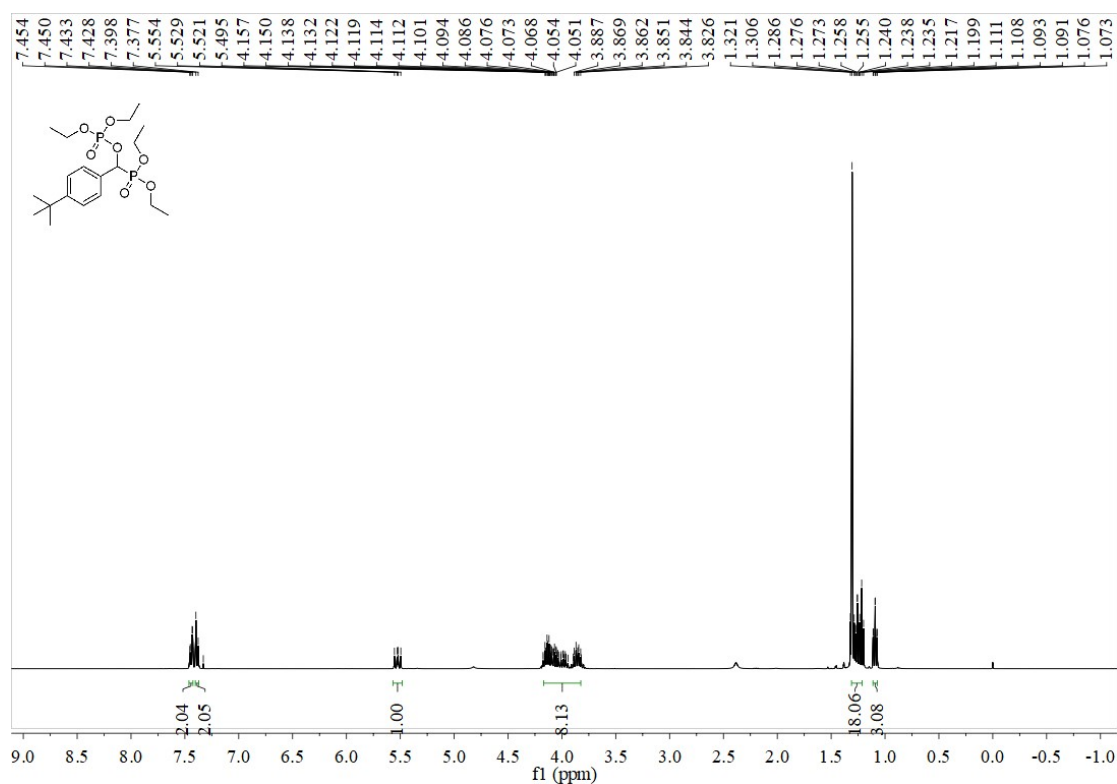
¹H NMR spectra of (diethoxyphosphoryl)(3,4,5-trimethylphenyl)methyl diethyl phosphate (**5c**).



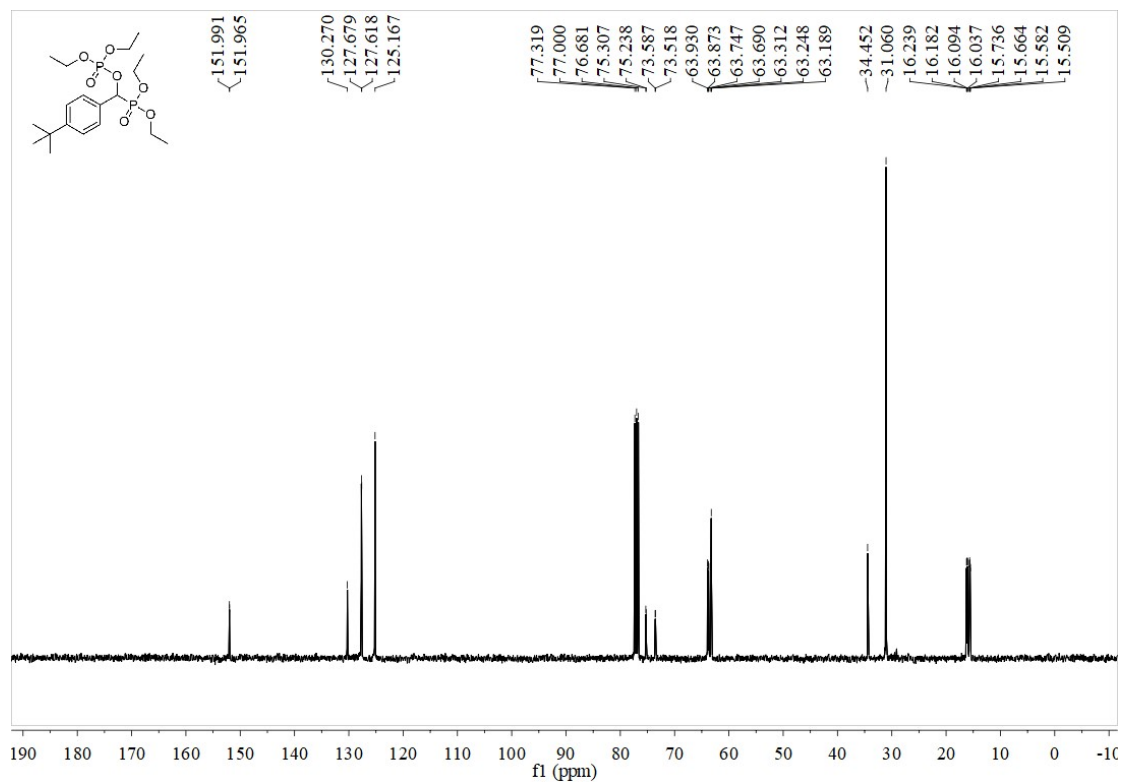
¹³C NMR spectra of (diethoxyphosphoryl)(3,4,5-trimethylphenyl)methyl diethyl phosphate (**5c**).



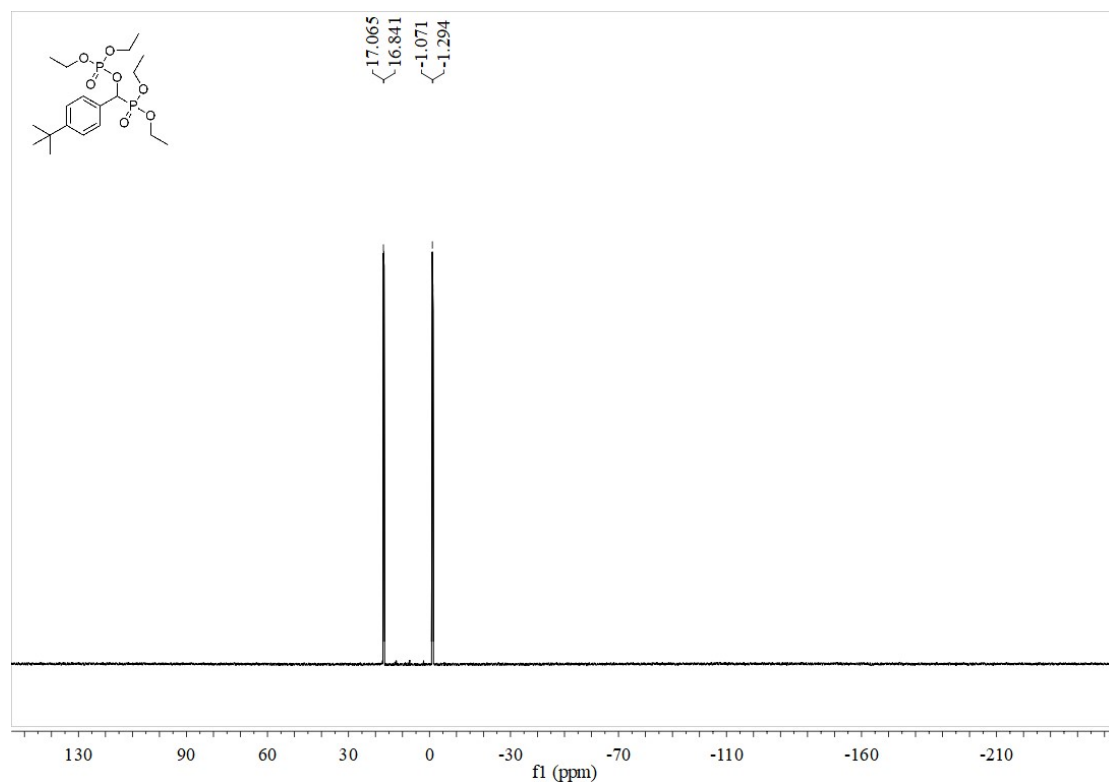
³¹P NMR spectra of (diethoxyphosphoryl)(3,4,5-trimethylphenyl)methyl diethyl phosphate (**5c**).



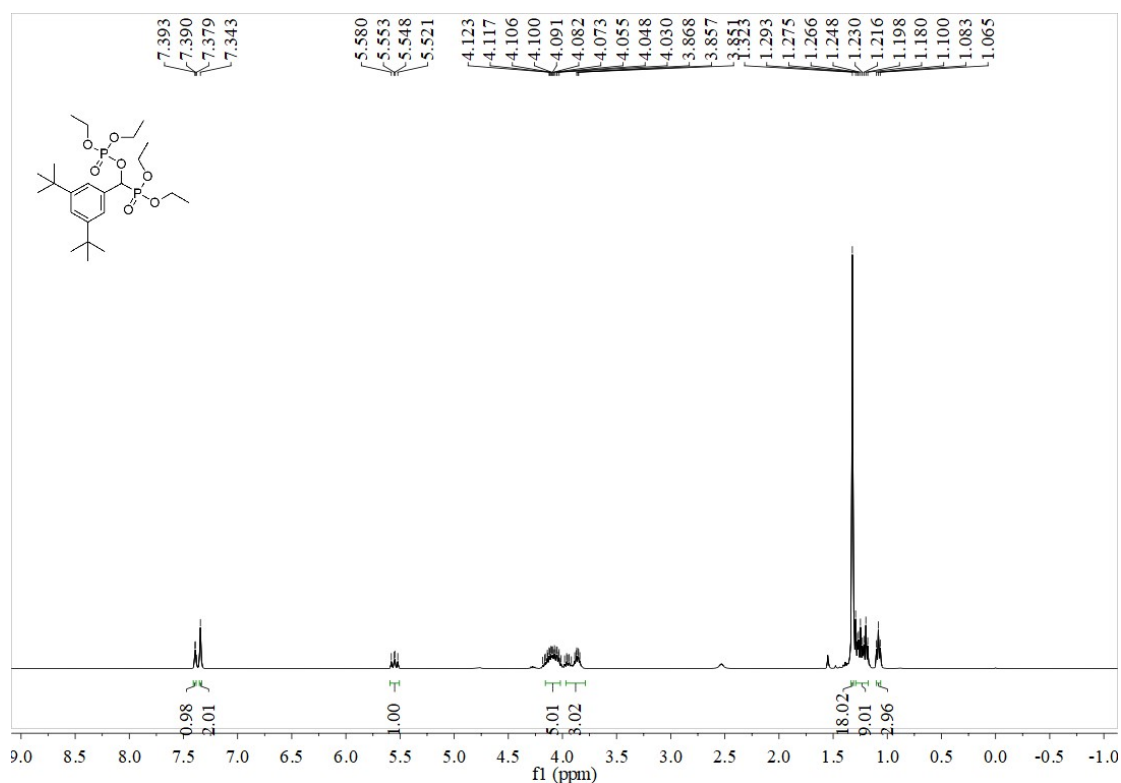
¹H NMR spectra of (4-(tert-butyl)phenyl)(diethoxyphosphoryl)methyl diethyl phosphate (**6c**).



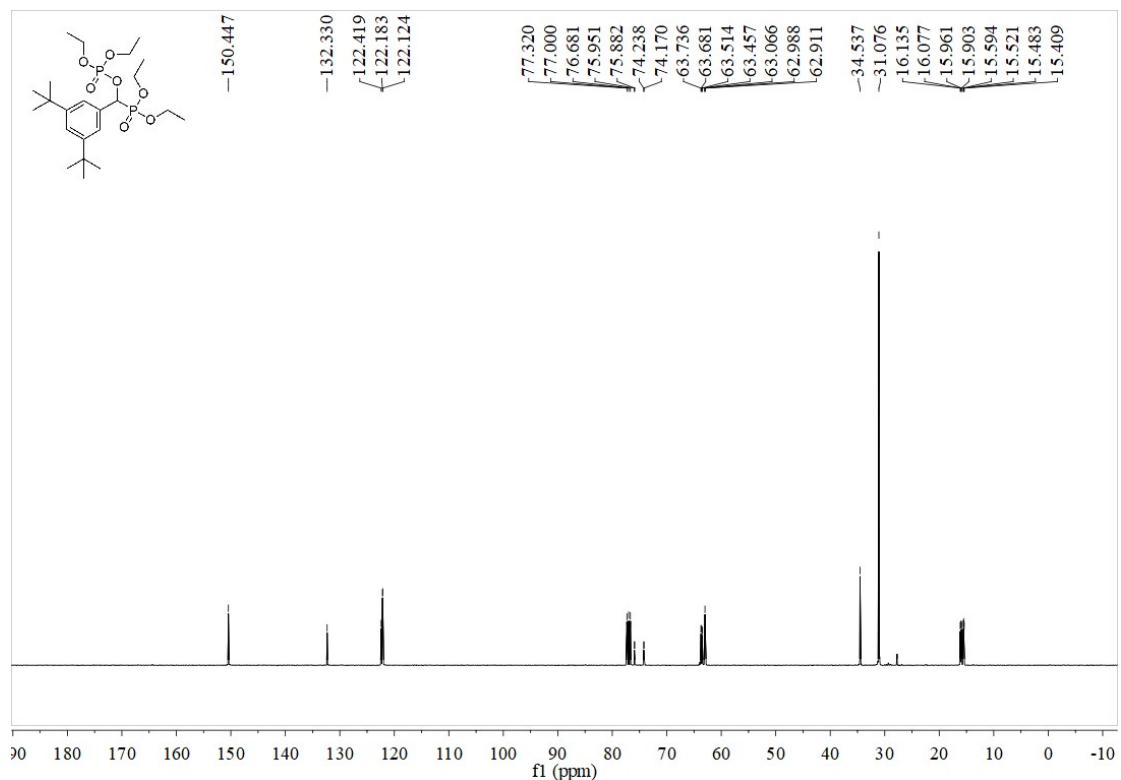
¹³C NMR spectra of (4-(tert-butyl)phenyl)(diethoxyphosphoryl)methyl diethyl phosphate (**6c**).



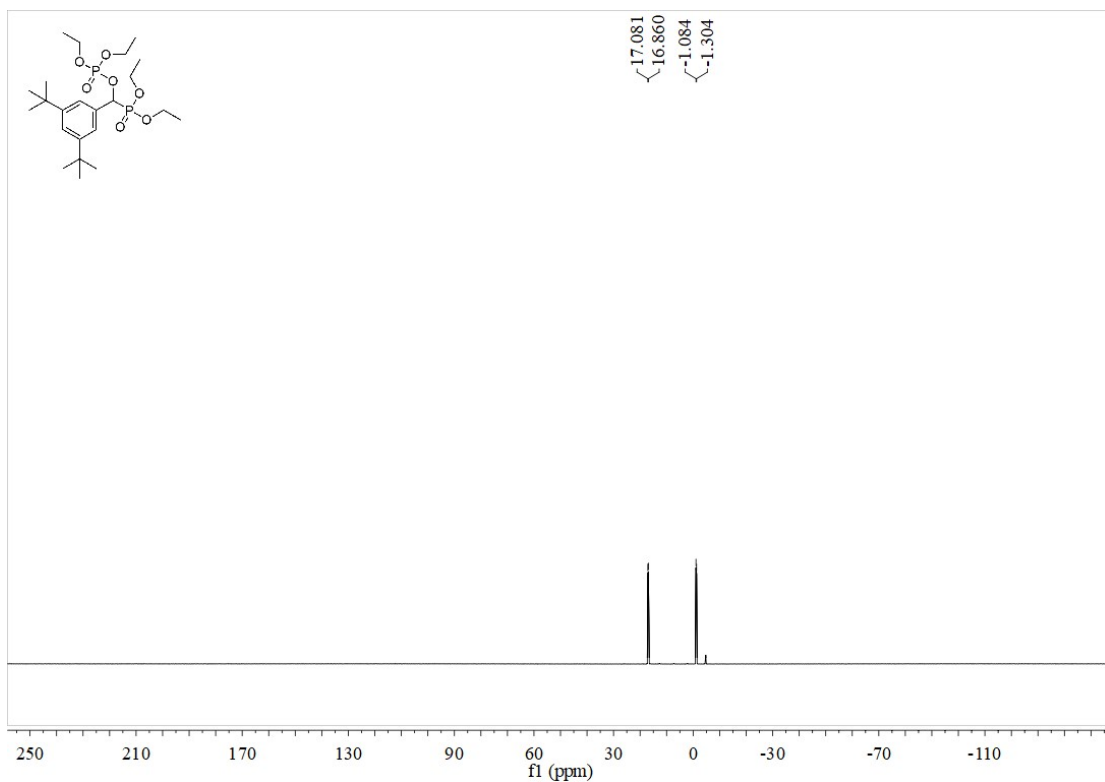
³¹P NMR spectra of (4-(tert-butyl)phenyl)(diethoxyphosphoryl)methyl diethyl phosphate (**6c**).



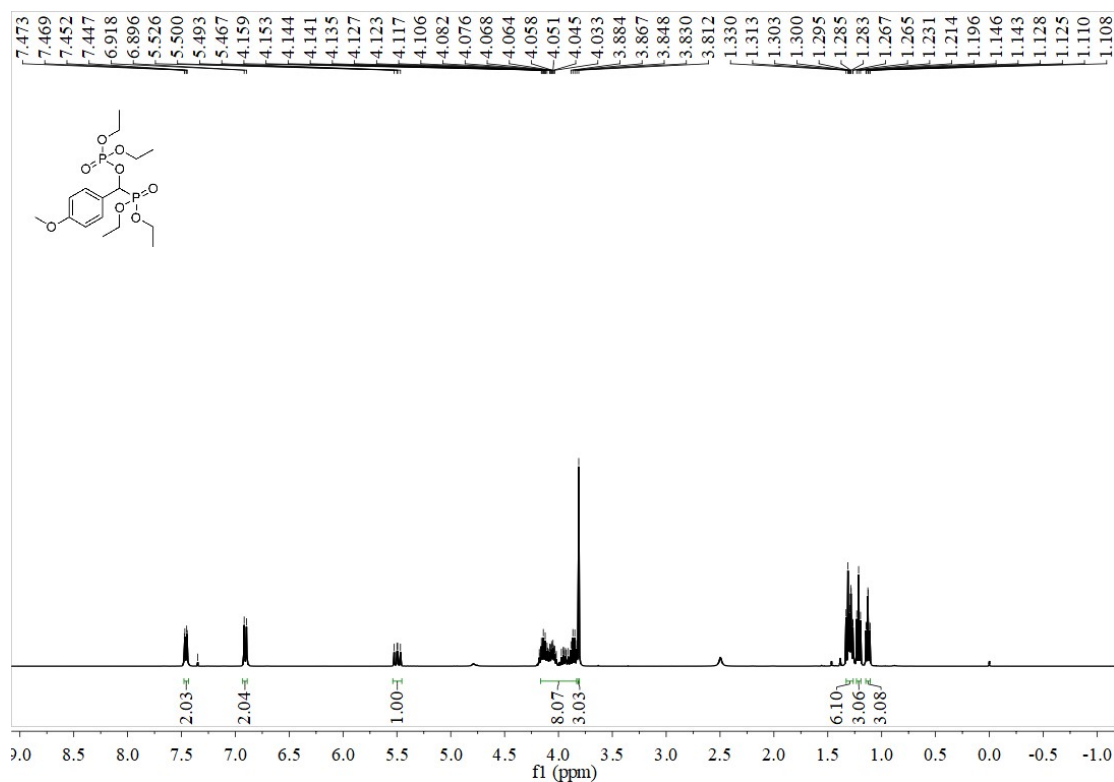
¹H NMR spectra of (3,5-di-tert-butylphenyl)(diethoxyphosphoryl)methyl diethyl phosphate (7c).



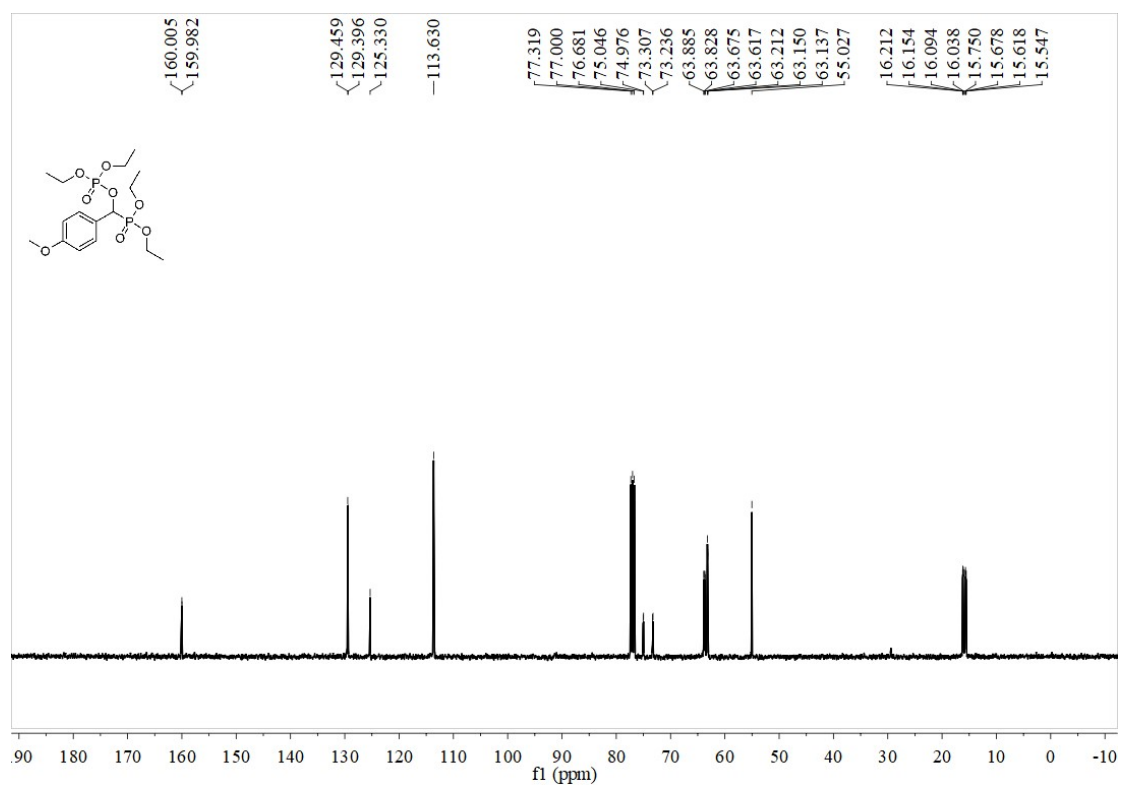
¹³C NMR spectra of (3,5-di-tert-butylphenyl)(diethoxyphosphoryl)methyl diethyl phosphate (7c).



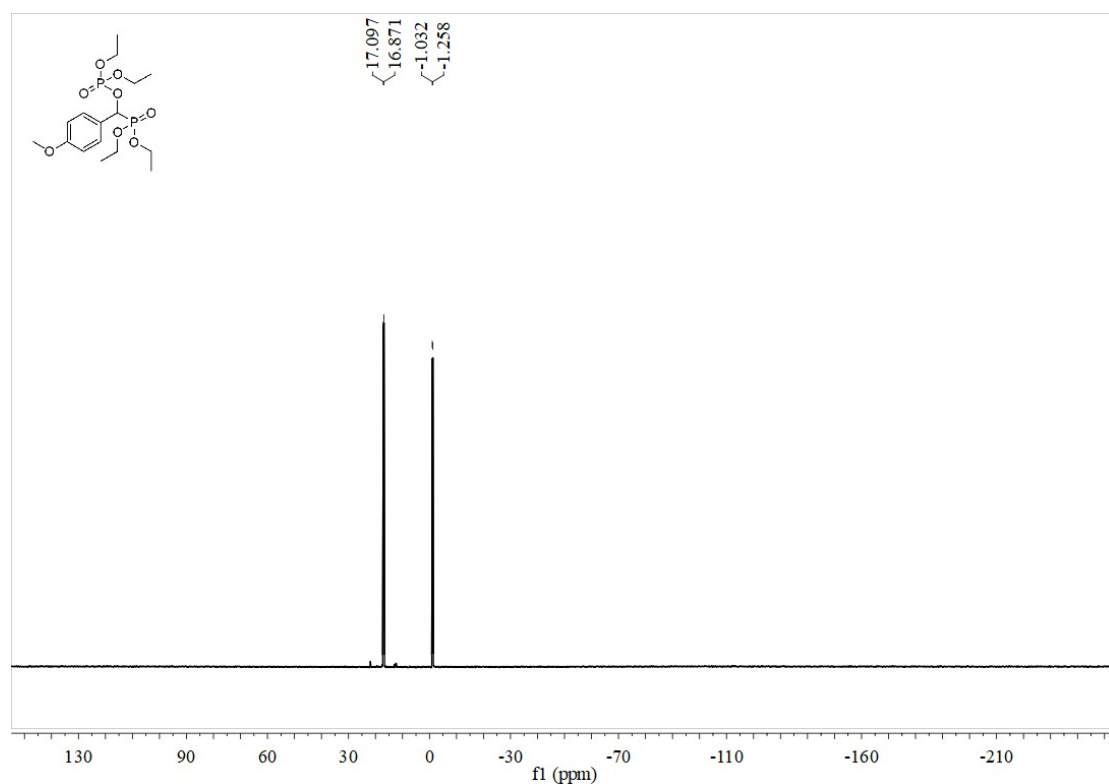
³¹P NMR spectra of (3,5-di-tert-butylphenyl)(diethoxyphosphoryl)methyl diethyl phosphate (7c).



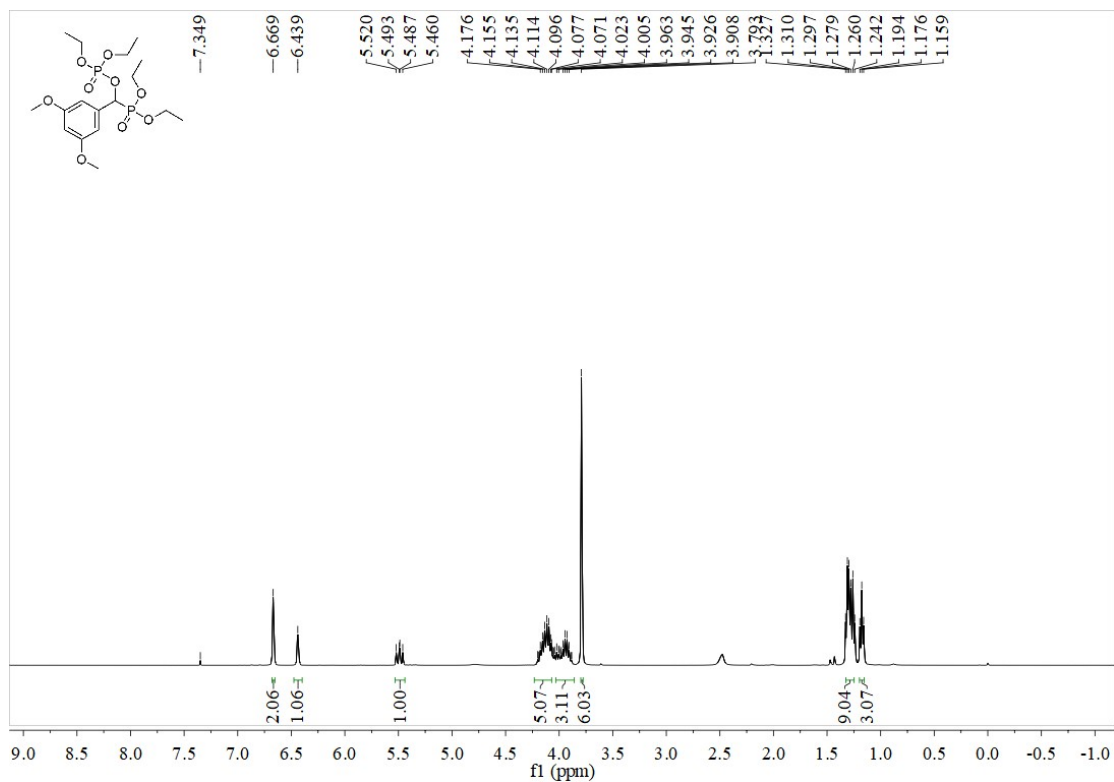
¹H NMR spectra of (diethoxyphosphoryl)(4-methoxyphenyl)methyl diethyl phosphate (8c).



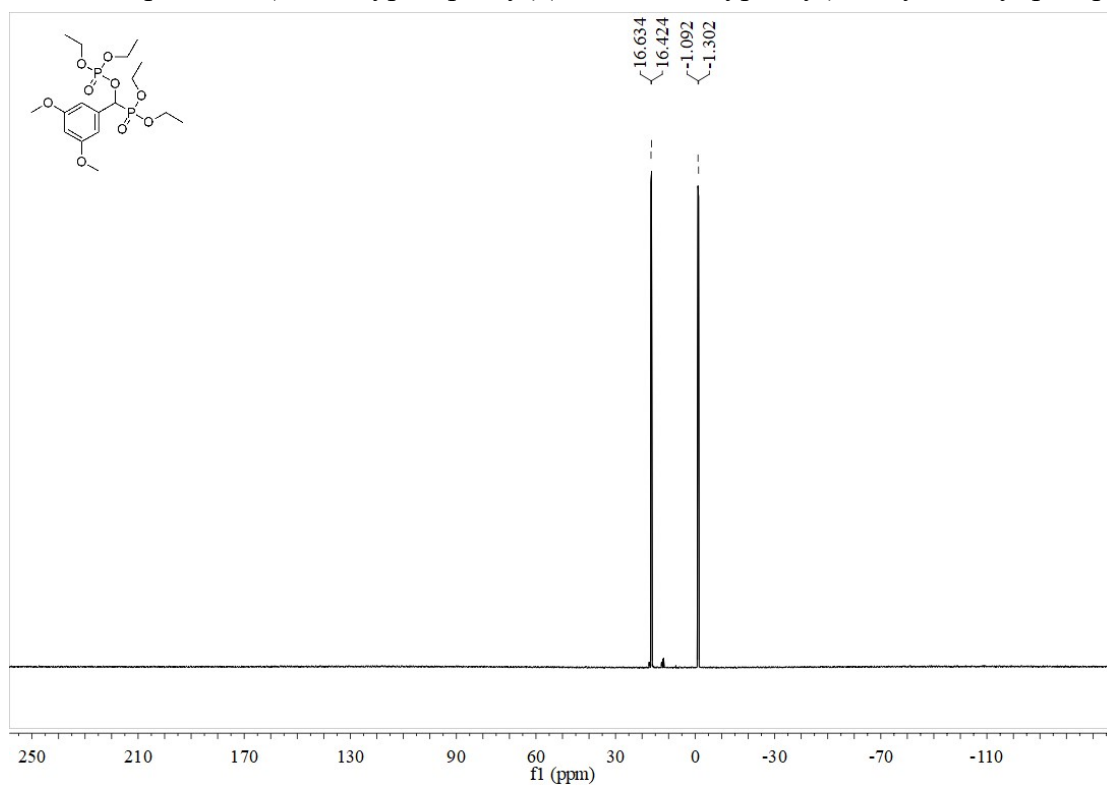
¹³C NMR spectra of (diethoxyphosphoryl)(4-methoxyphenyl)methyl diethyl phosphate (**8c**).



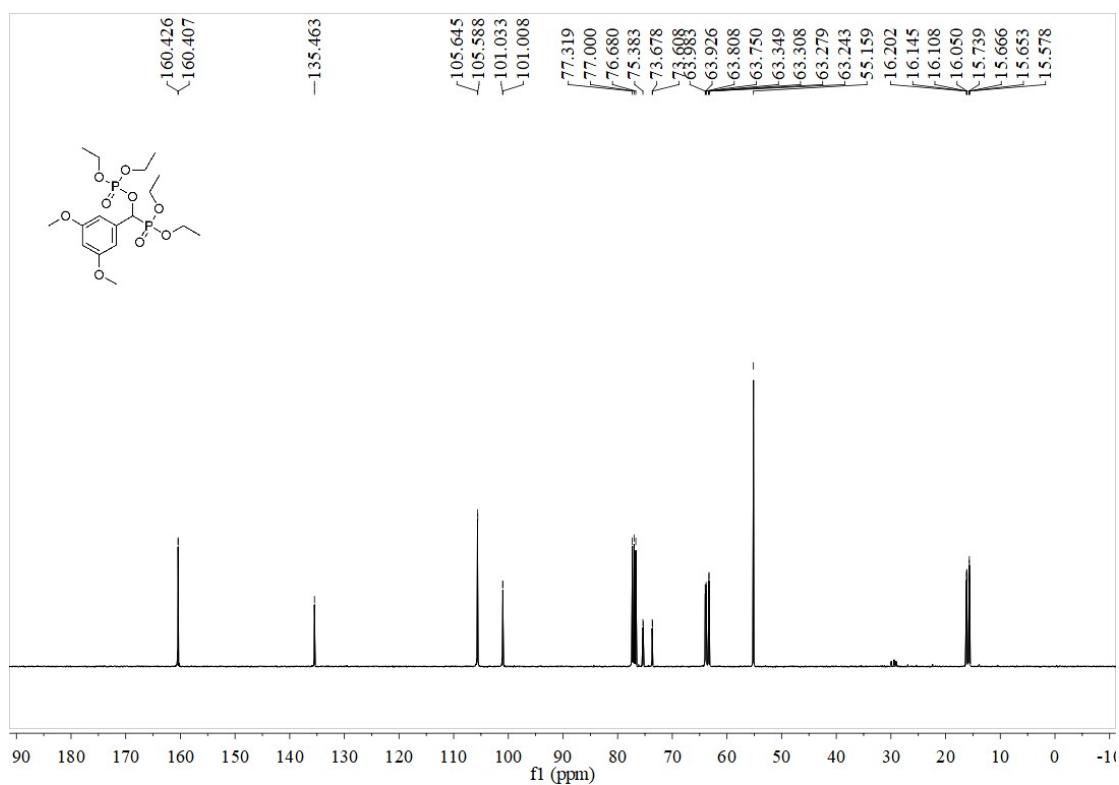
³¹P NMR spectra of (diethoxyphosphoryl)(4-methoxyphenyl)methyl diethyl phosphate (**8c**).



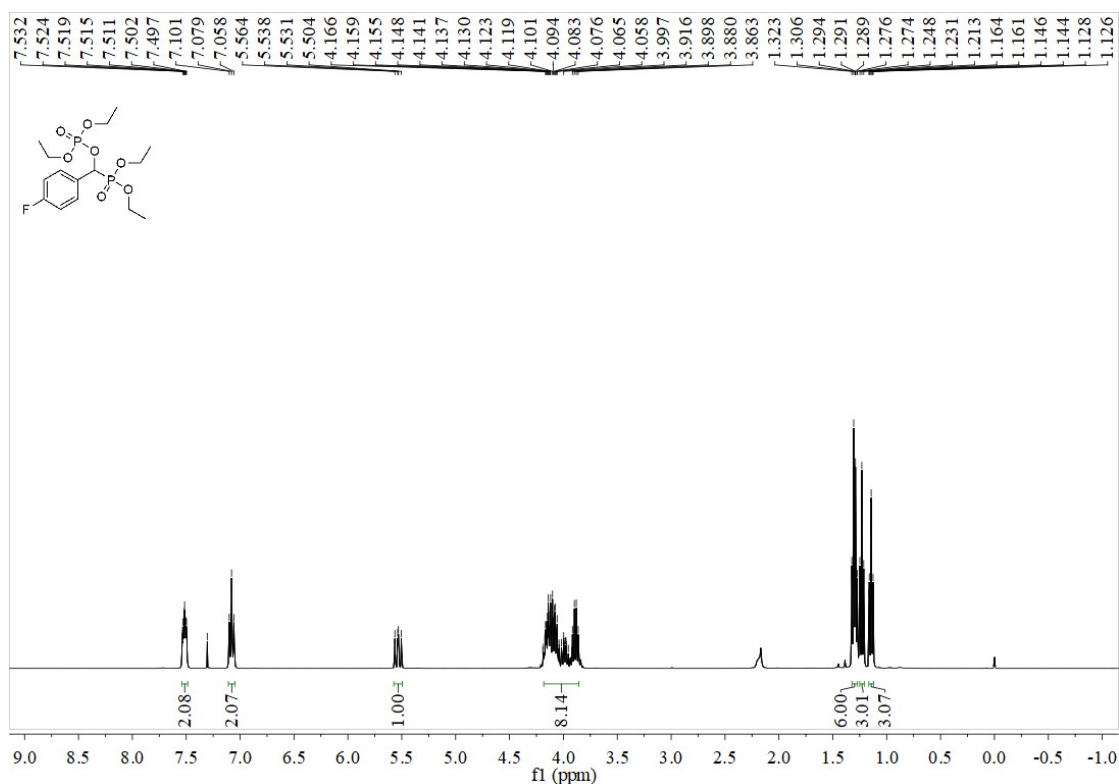
¹H NMR spectra of (diethoxyphosphoryl)(3,5-dimethoxyphenyl)methyl diethyl phosphate (**9c**).



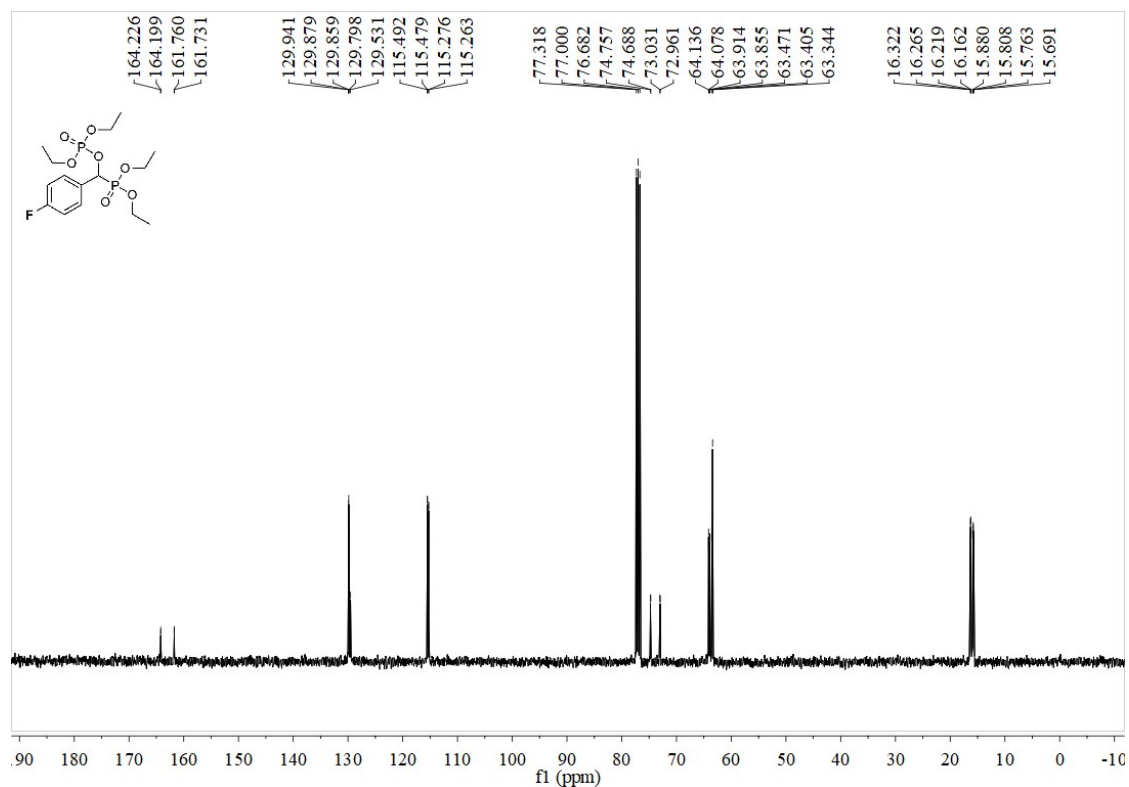
¹³C NMR spectra of (diethoxyphosphoryl)(3,5-dimethoxyphenyl)methyl diethyl phosphate (**9c**).



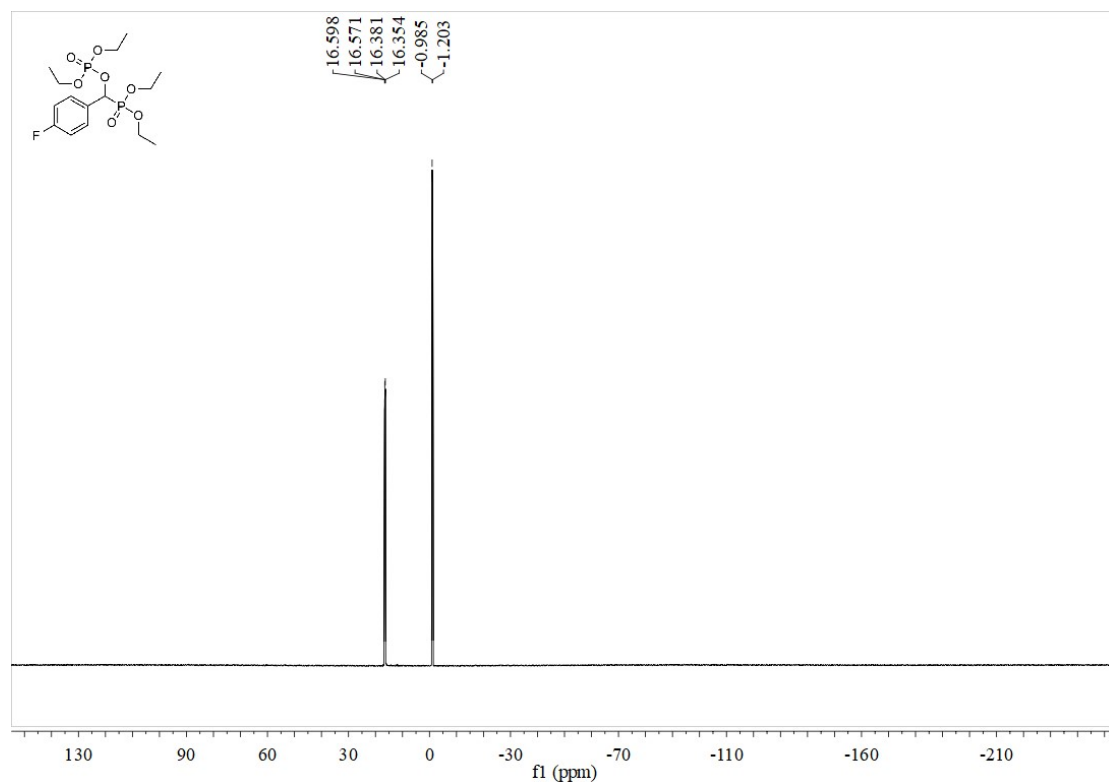
³¹P NMR spectra of (diethoxyphosphoryl)(3,5-dimethoxyphenyl)methyl diethyl phosphate (9c).



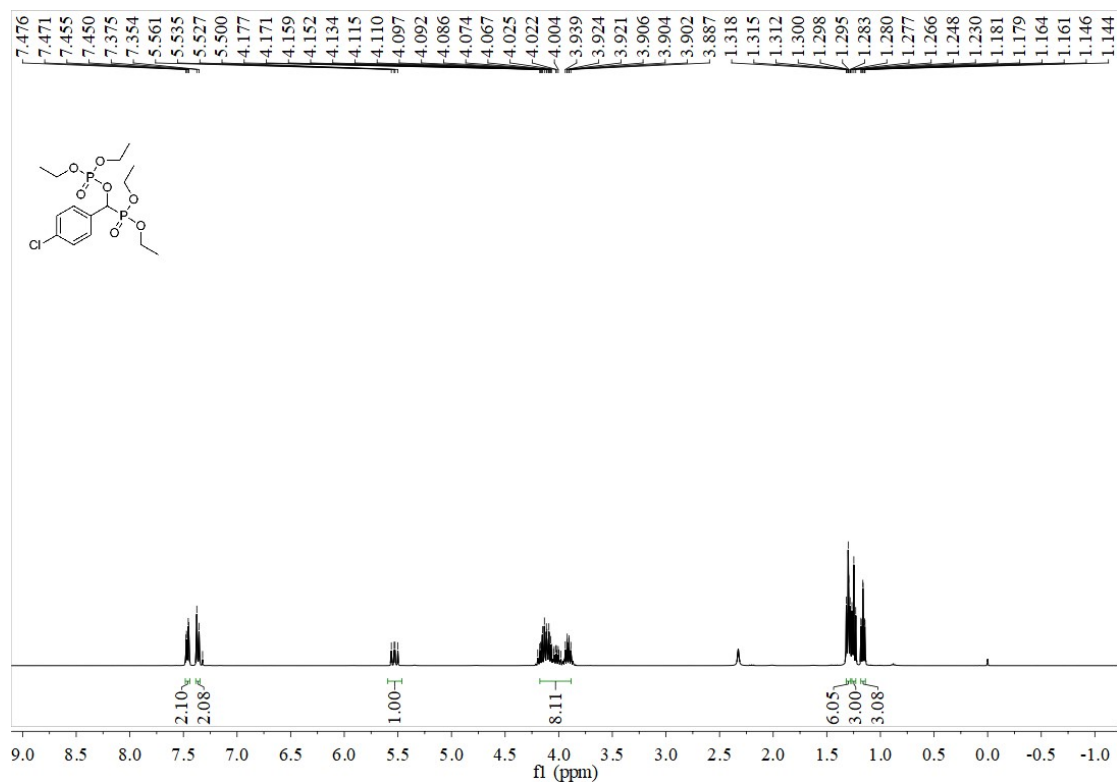
¹H NMR spectra of (diethoxyphosphoryl)(4-fluorophenyl)methyl diethyl phosphate (10c).



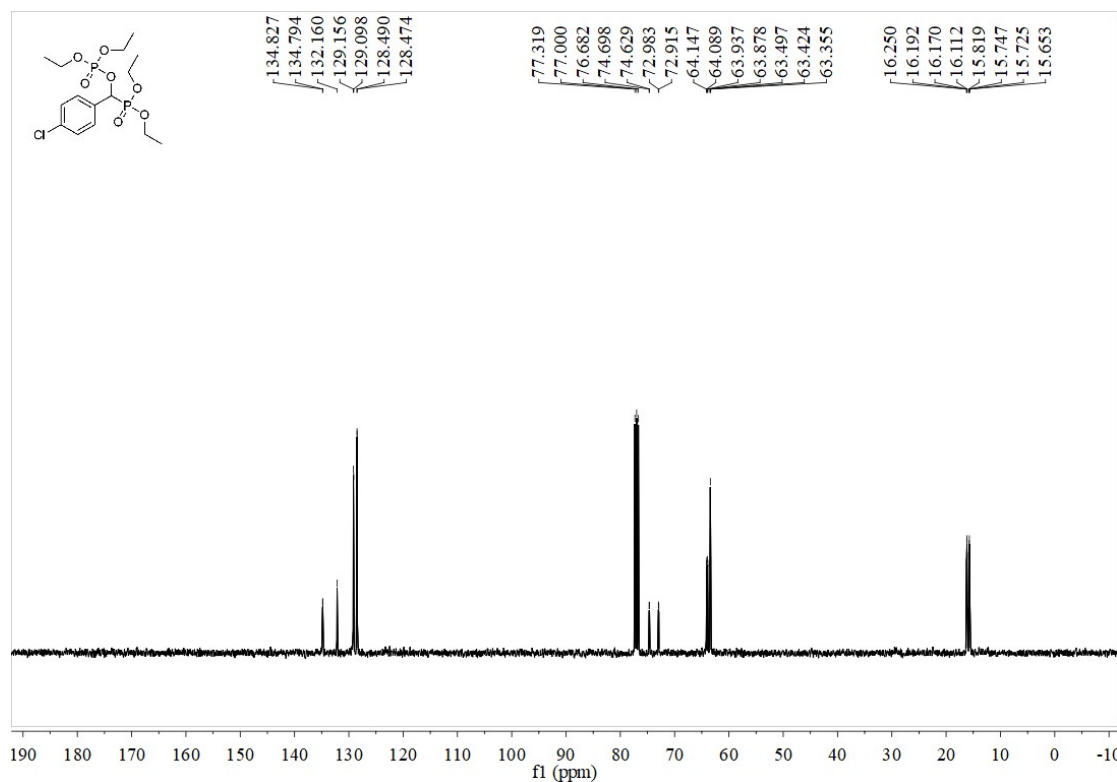
¹³C NMR spectra of (diethoxyphosphoryl)(4-fluorophenyl)methyl diethyl phosphate (**10c**).



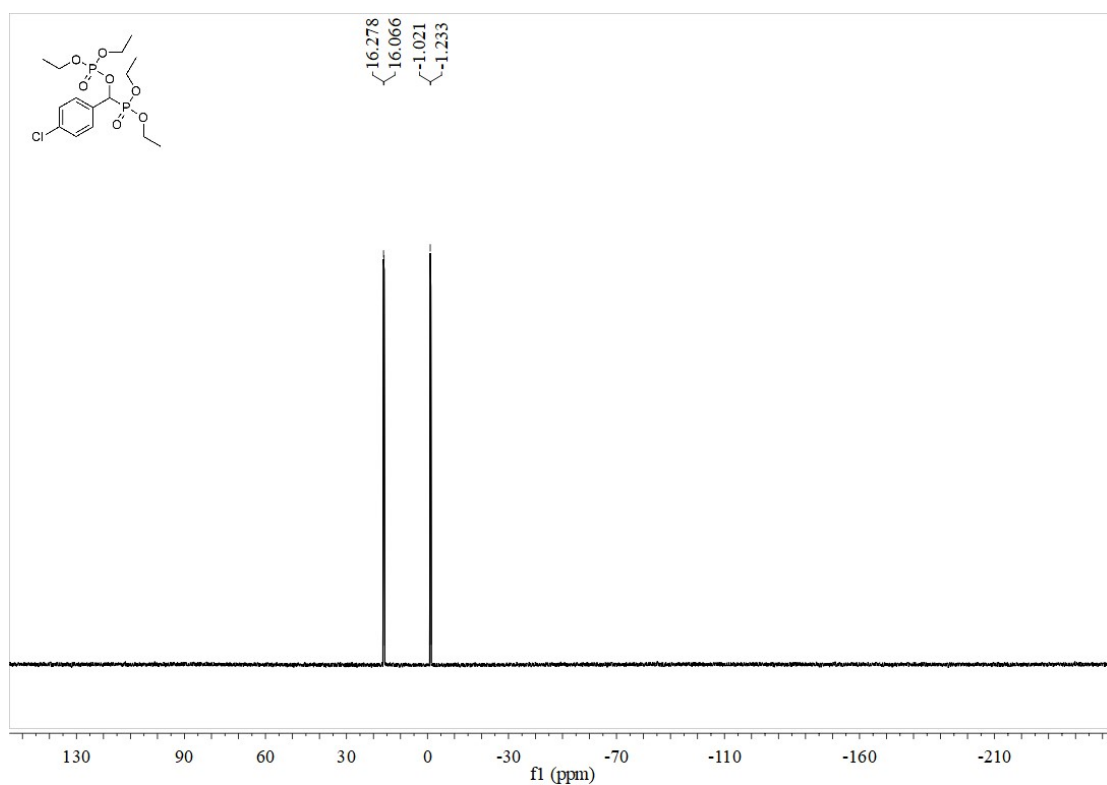
³¹P NMR spectra of (diethoxyphosphoryl)(4-fluorophenyl)methyl diethyl phosphate (**10c**).



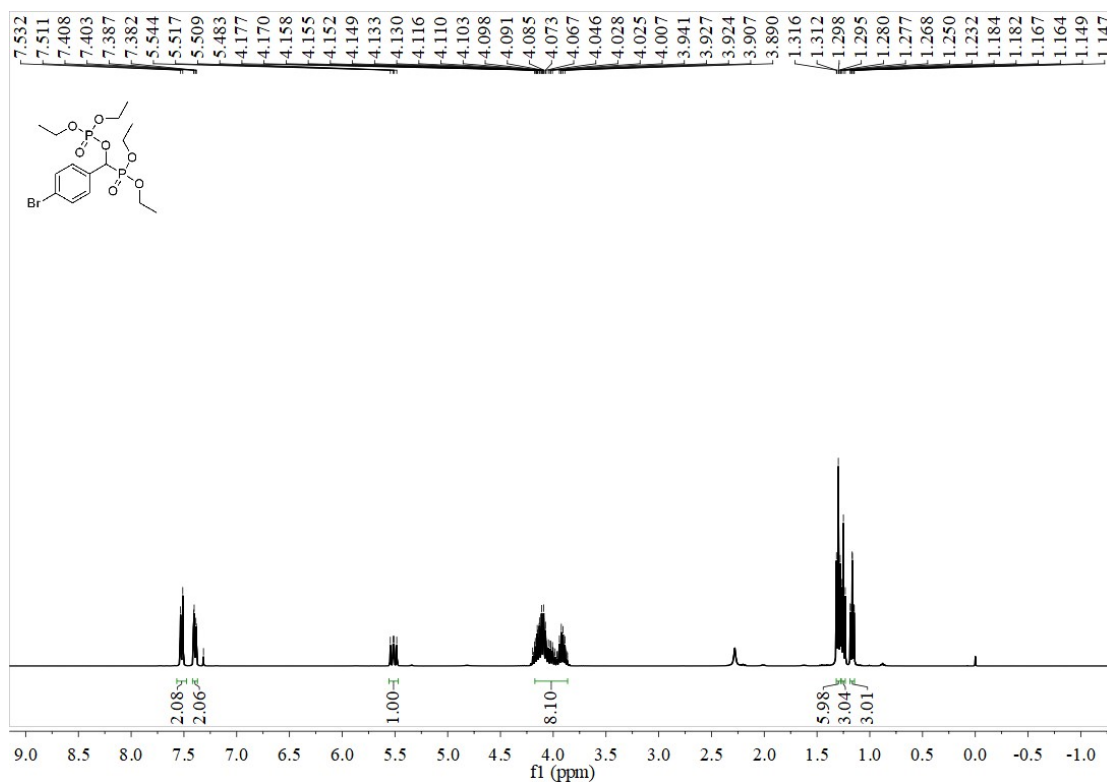
¹H NMR spectra of (4-chlorophenyl)(diethoxyphosphoryl)methyl diethyl phosphate (**11c**).



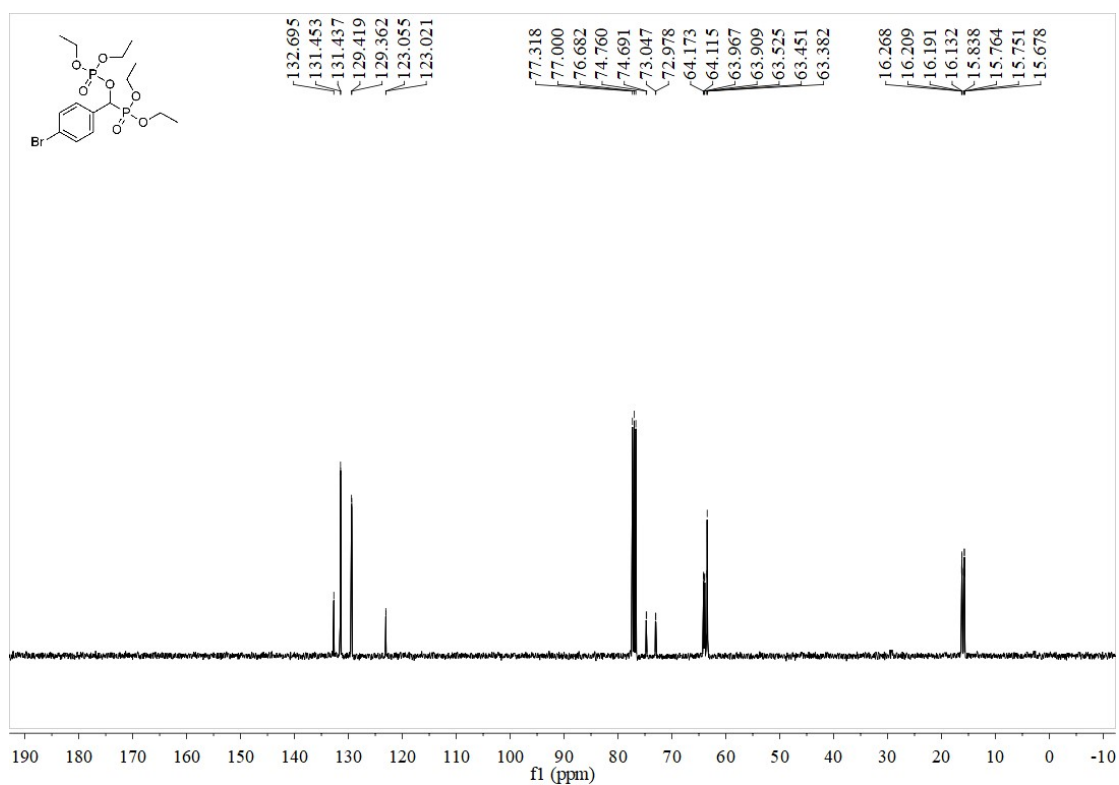
¹³C NMR spectra of (4-chlorophenyl)(diethoxyphosphoryl)methyl diethyl phosphate (**11c**).



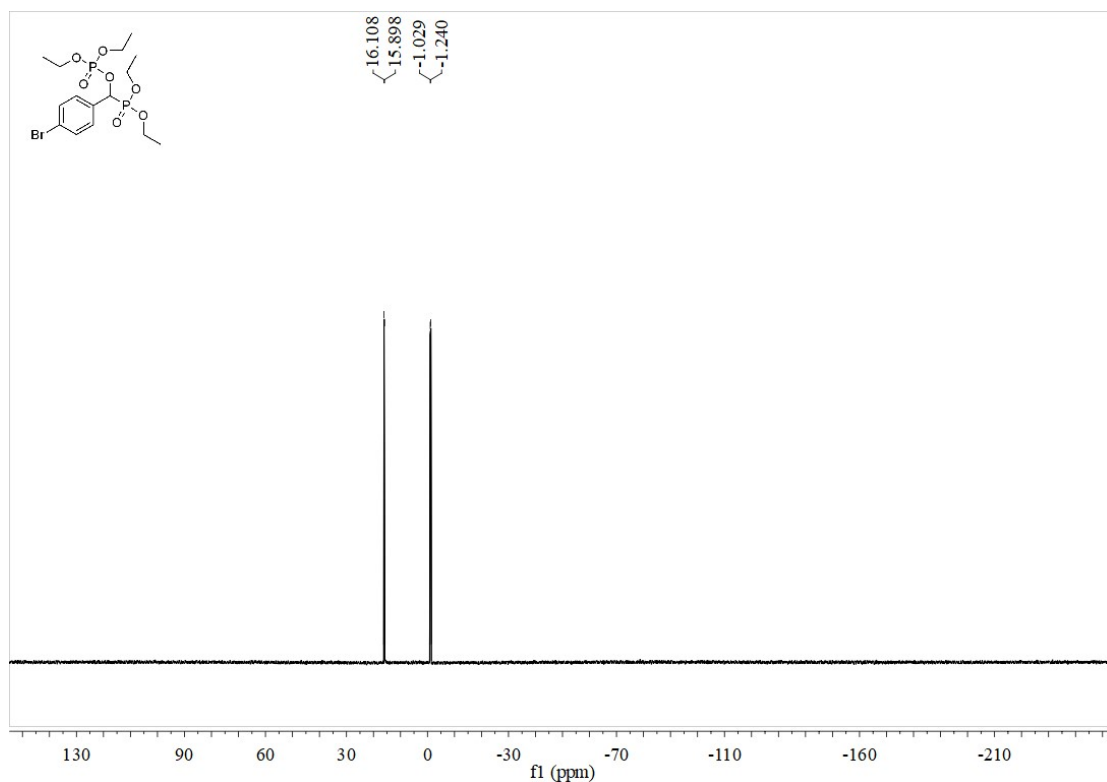
^{31}P NMR spectra of (4-chlorophenyl)(diethoxyphosphoryl)methyl diethyl phosphate (**11c**).



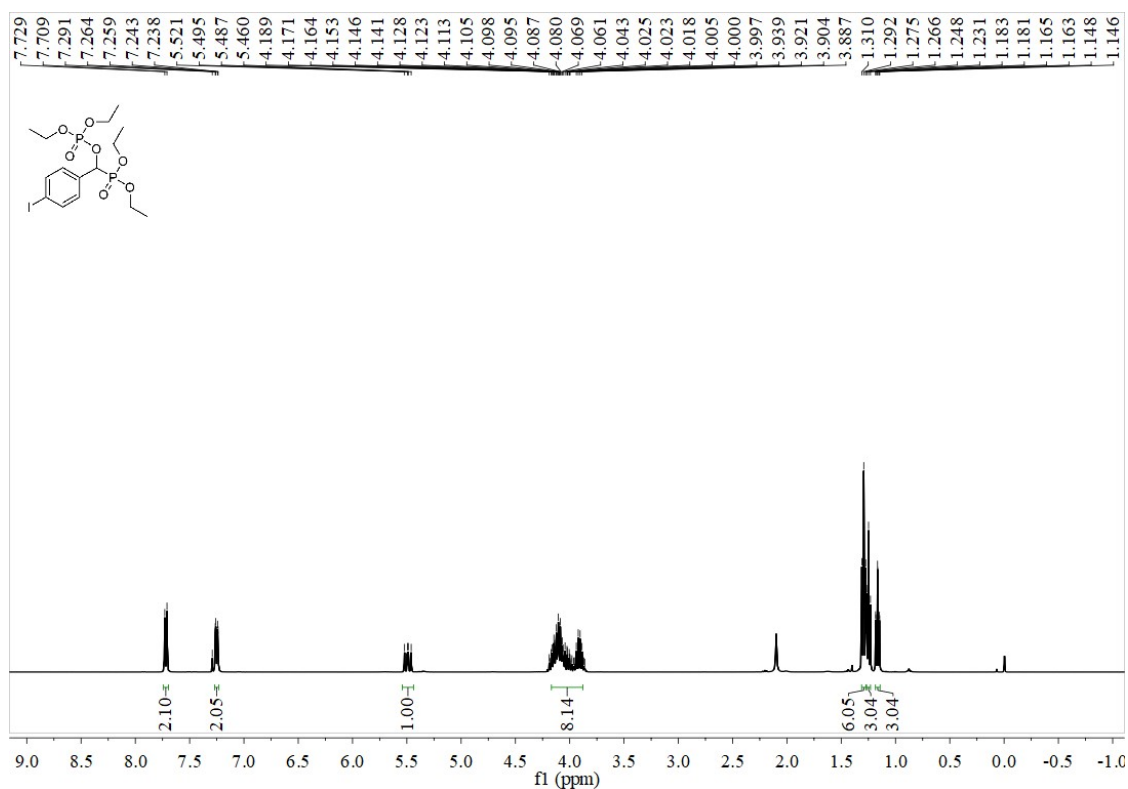
^1H NMR spectra of (4-bromophenyl)(phosphoryl)methyl phosphenite compound with ethoxyethane (**12c**).



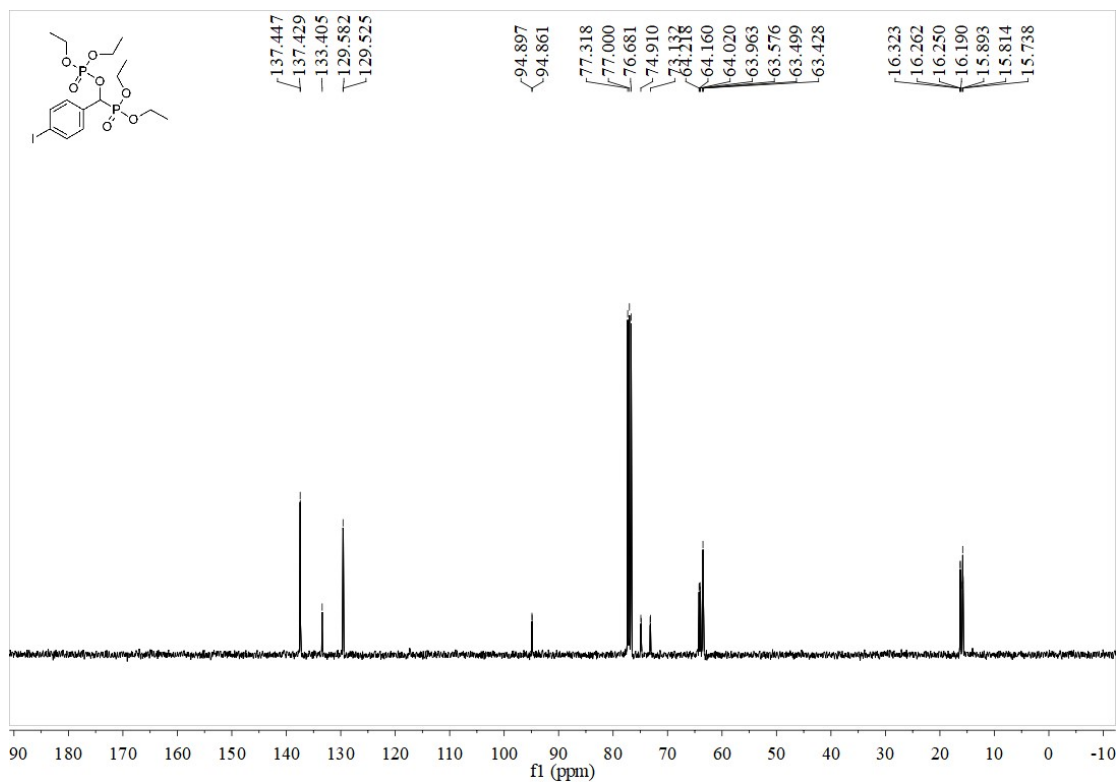
¹³C NMR spectra of (4-bromophenyl)(phosphoryl)methyl phosphenite compound with ethoxyethane (**12c**).



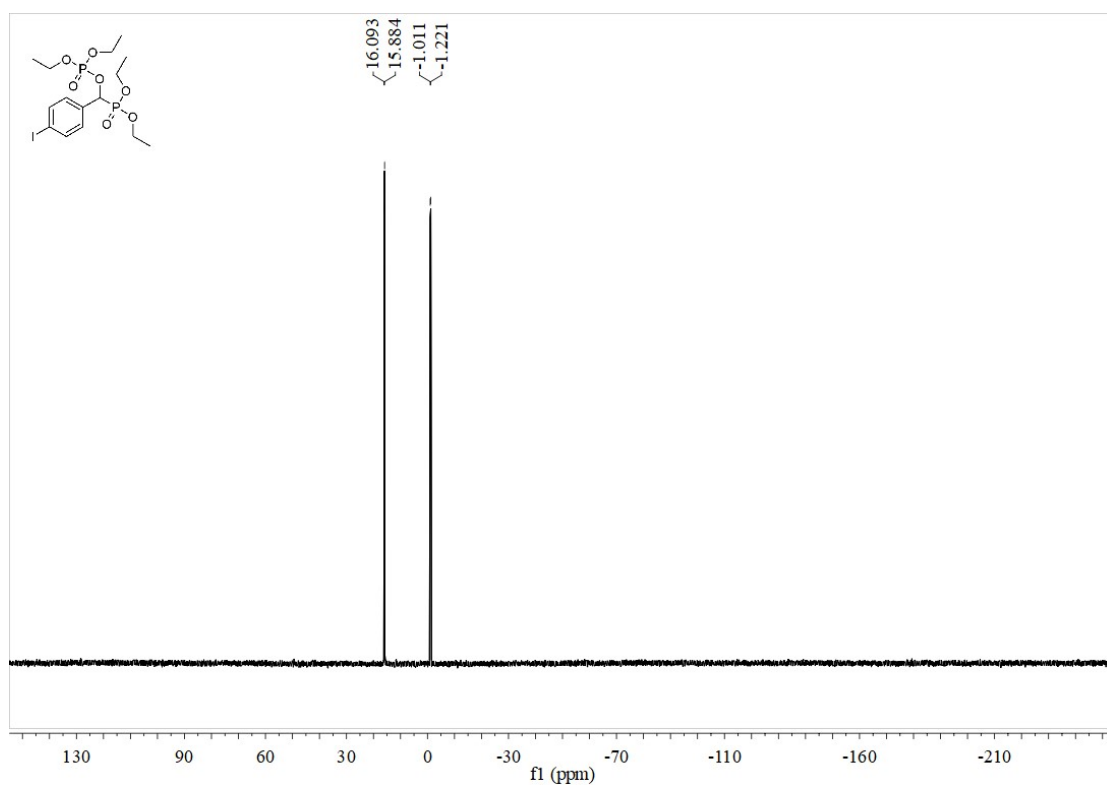
³¹P NMR spectra of (4-bromophenyl)(phosphoryl)methyl phosphenite compound with ethoxyethane (**12c**).



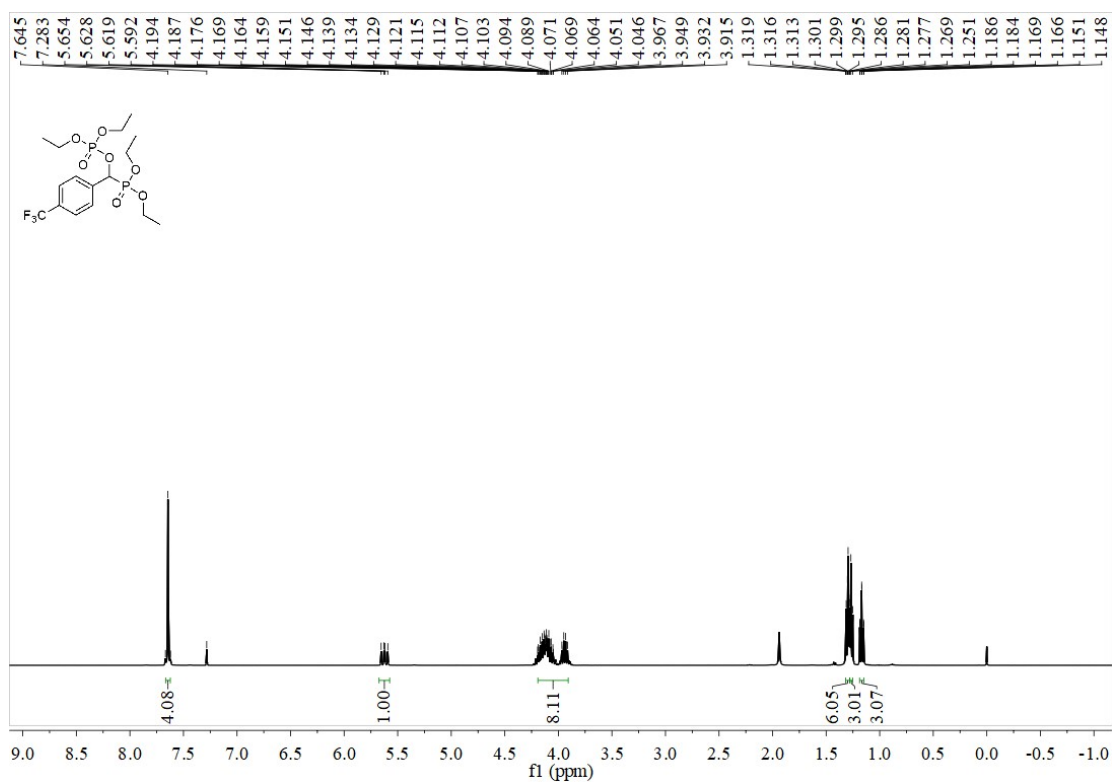
¹H NMR spectra of (diethoxyphosphoryl)(4-iodophenyl)methyl diethyl phosphate (13c).



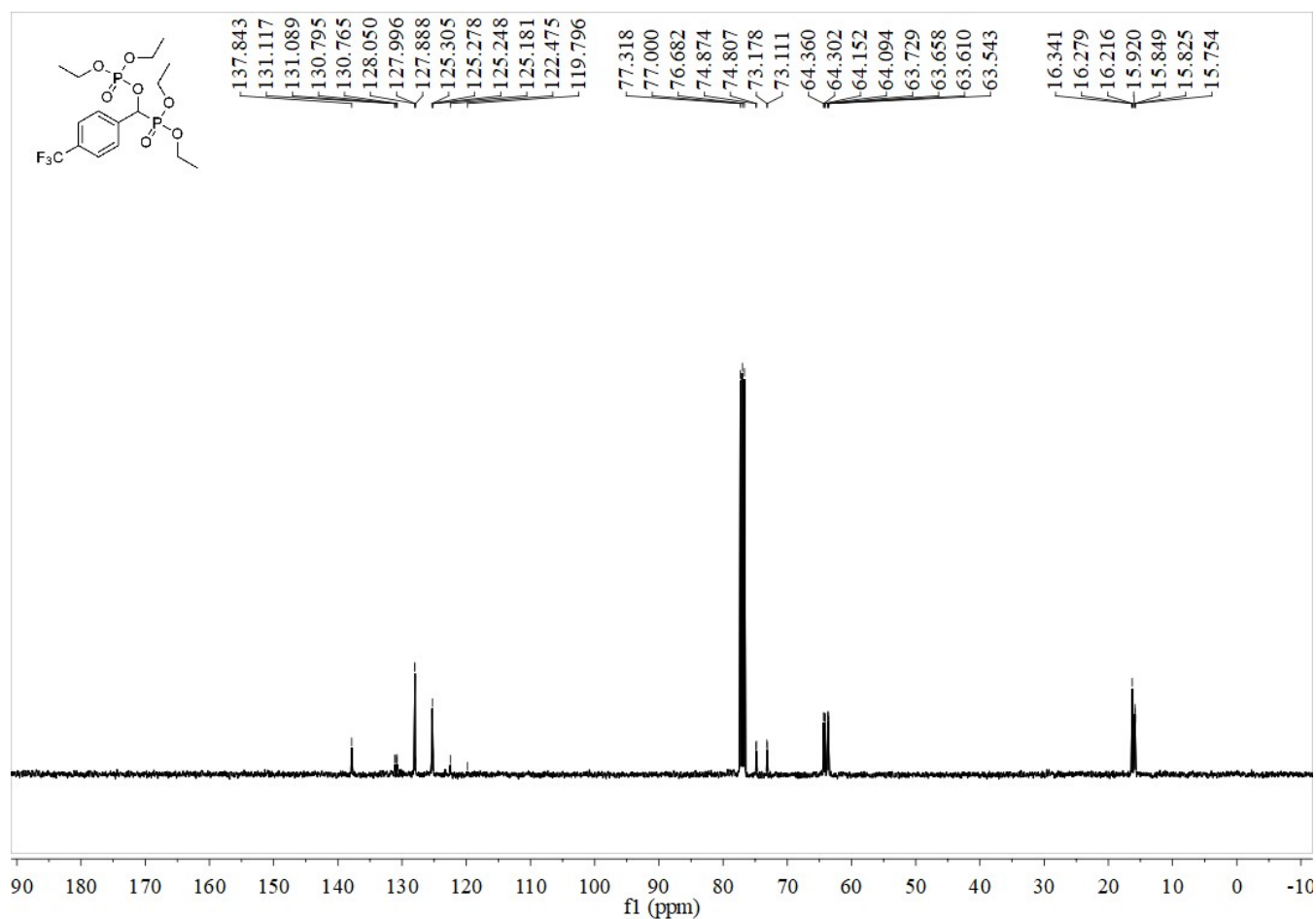
¹³C NMR spectra of (diethoxyphosphoryl)(4-iodophenyl)methyl diethyl phosphate (13c).



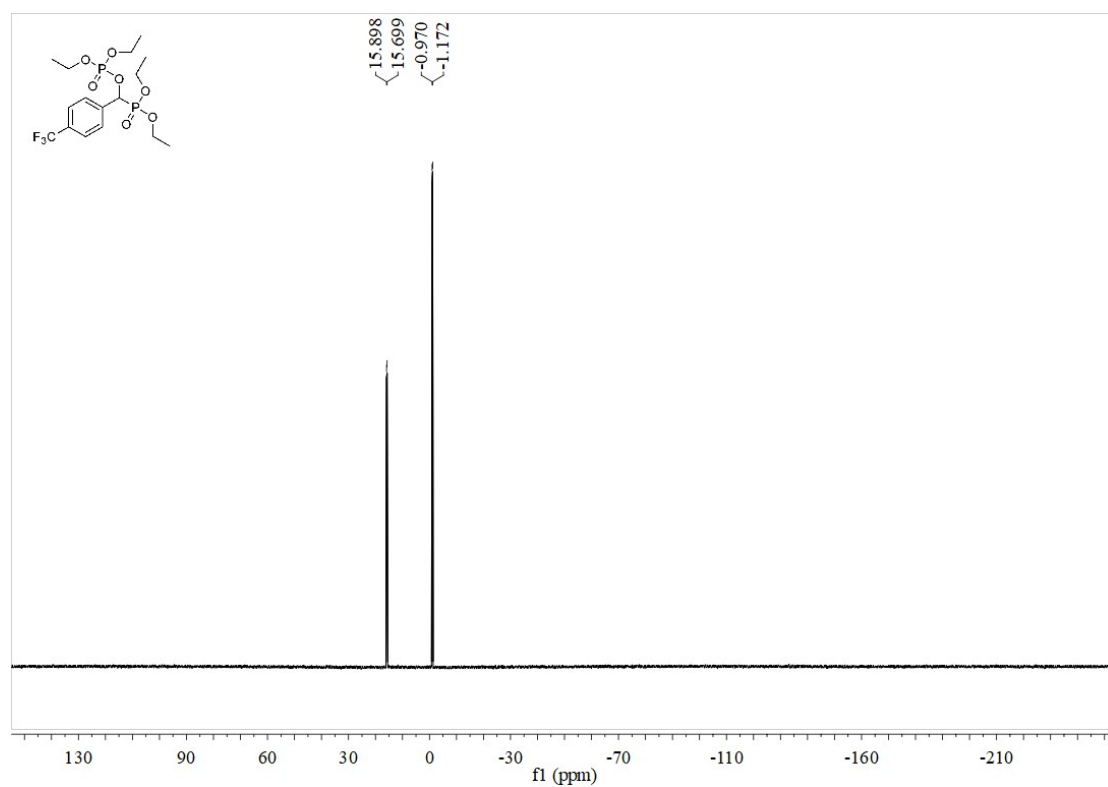
³¹P NMR spectra of (diethoxyphosphoryl)(4-iodophenyl)methyl diethyl phosphate (**13c**).



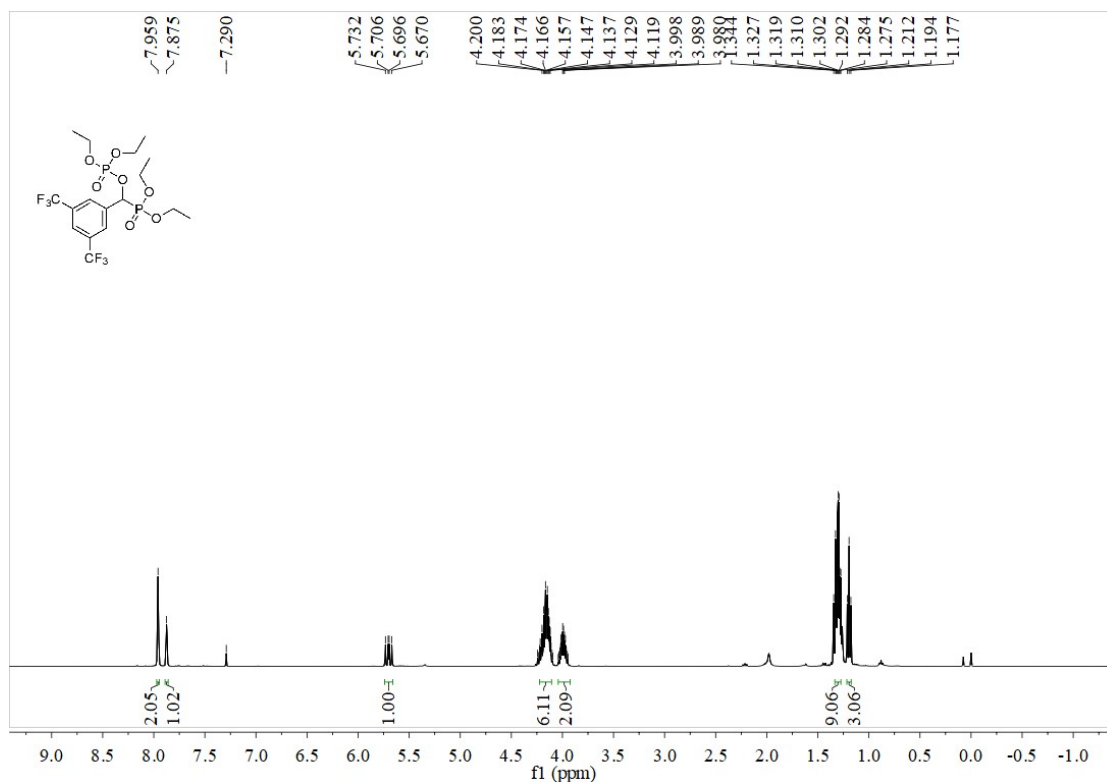
¹H NMR spectra of (diethoxyphosphoryl)(4-(trifluoromethyl)phenyl)methyl diethyl phosphate (**14c**).



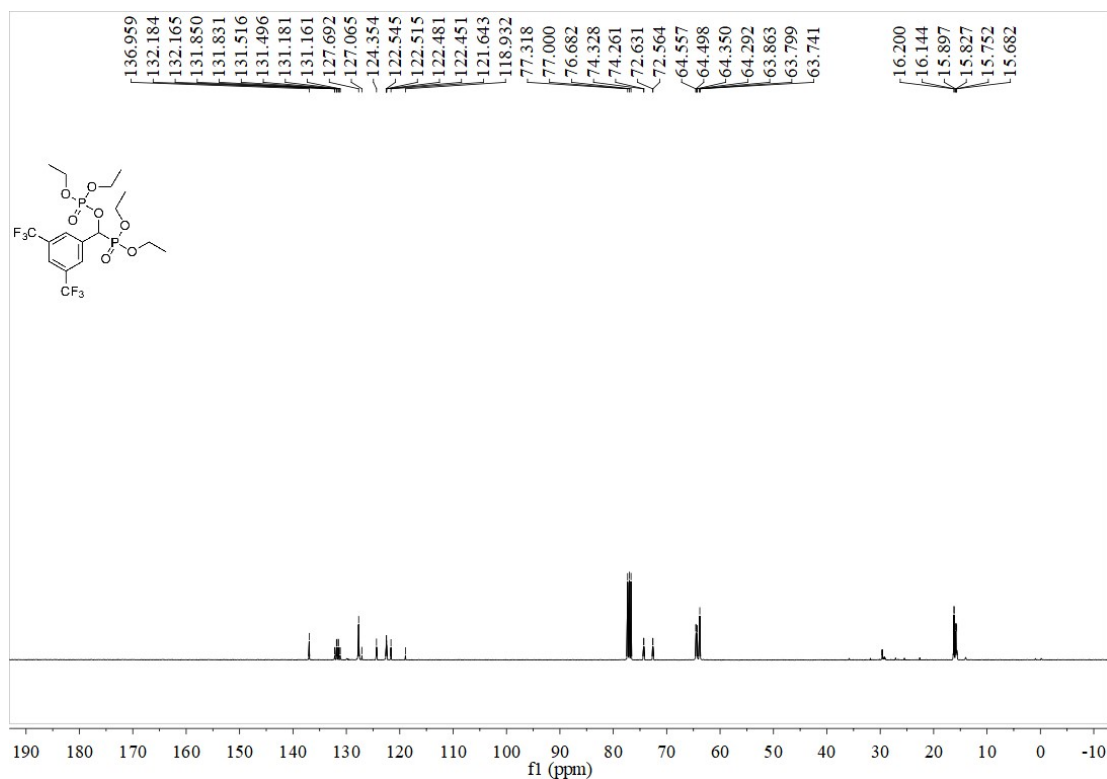
¹³C NMR spectra of (diethoxyphosphoryl)(4-(trifluoromethyl)phenyl)methyl diethyl phosphate (**14c**).



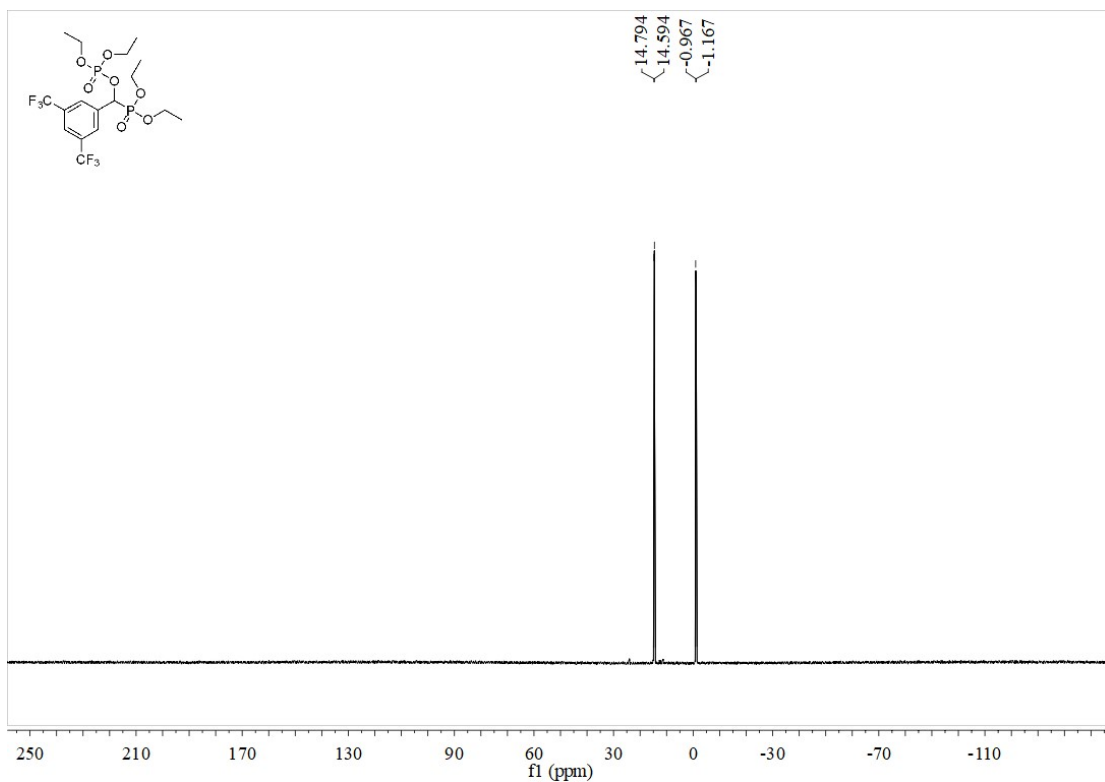
³¹P NMR spectra of (diethoxyphosphoryl)(4-(trifluoromethyl)phenyl)methyl diethyl phosphate (**14c**).



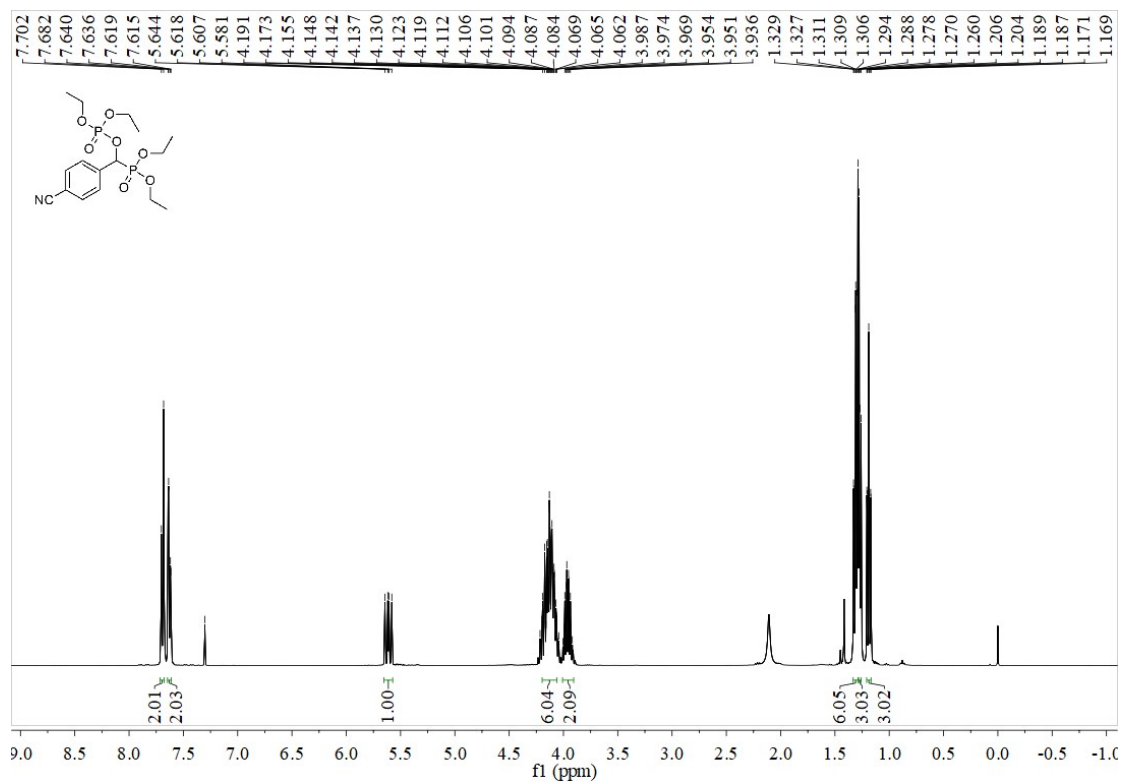
¹H NMR spectra of (3,5-bis(trifluoromethyl)phenyl)(diethoxyphosphoryl)methyl diethyl phosphate (**15c**).



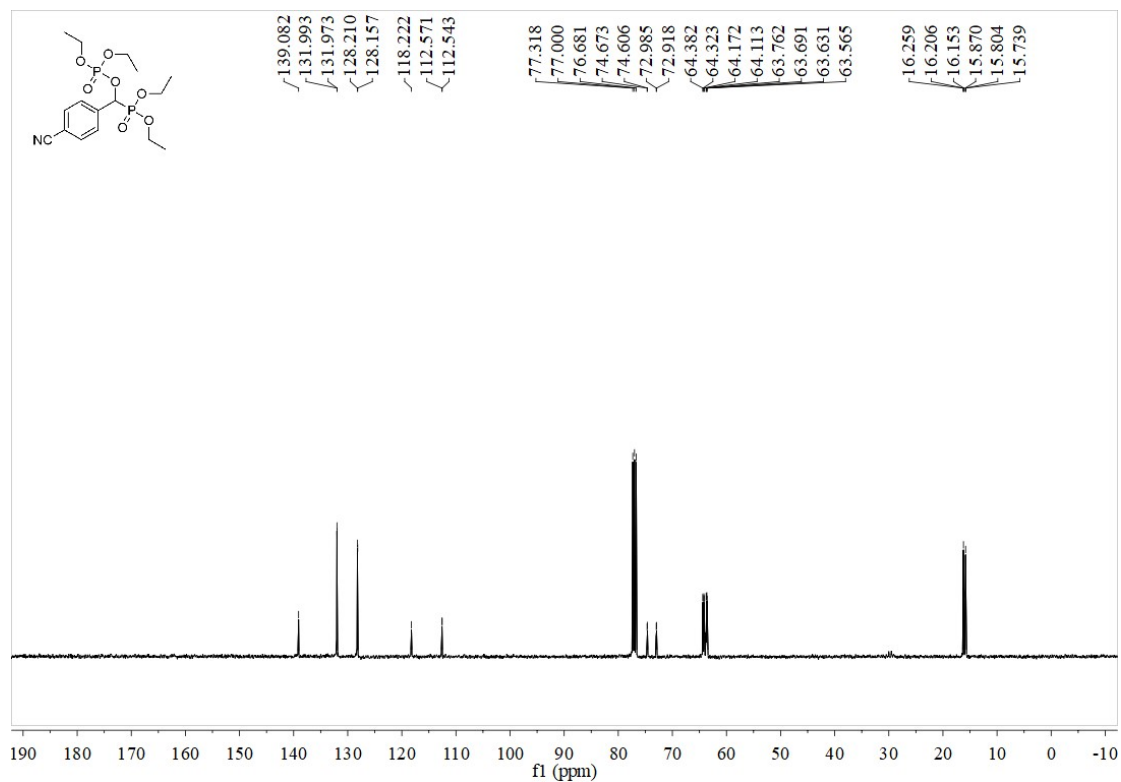
¹³C NMR spectra of (3,5-bis(trifluoromethyl)phenyl)(diethoxyphosphoryl)methyl diethyl phosphate (**15c**).



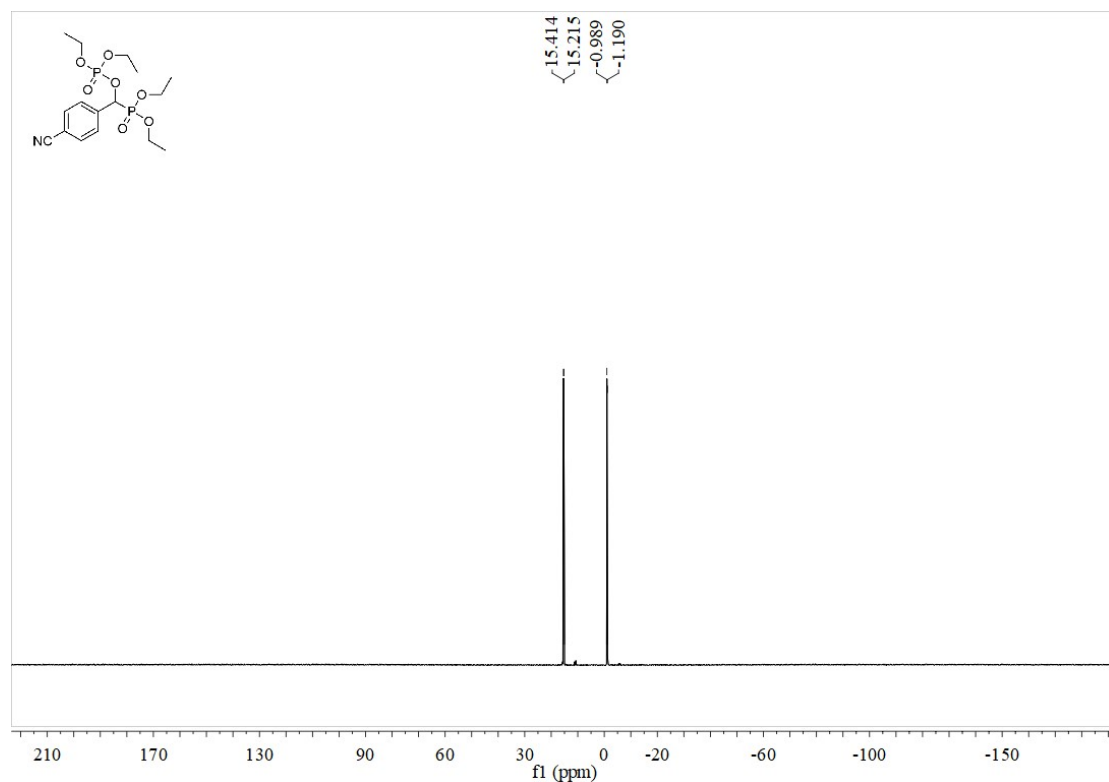
³¹P NMR spectra of (3,5-bis(trifluoromethyl)phenyl)(diethoxyphosphoryl)methyl diethyl phosphate (**15c**).



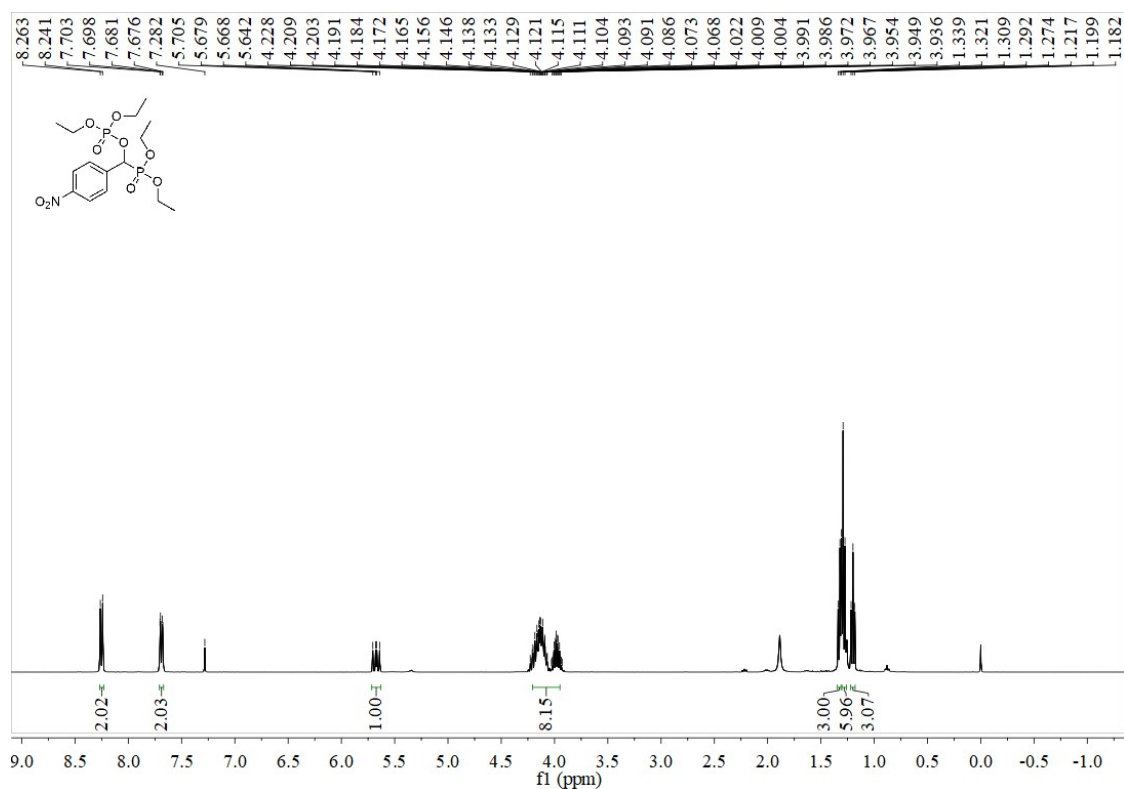
¹H NMR spectra of (4-cyanophenyl)(diethoxyphosphoryl)methyl diethyl phosphate (**16c**).



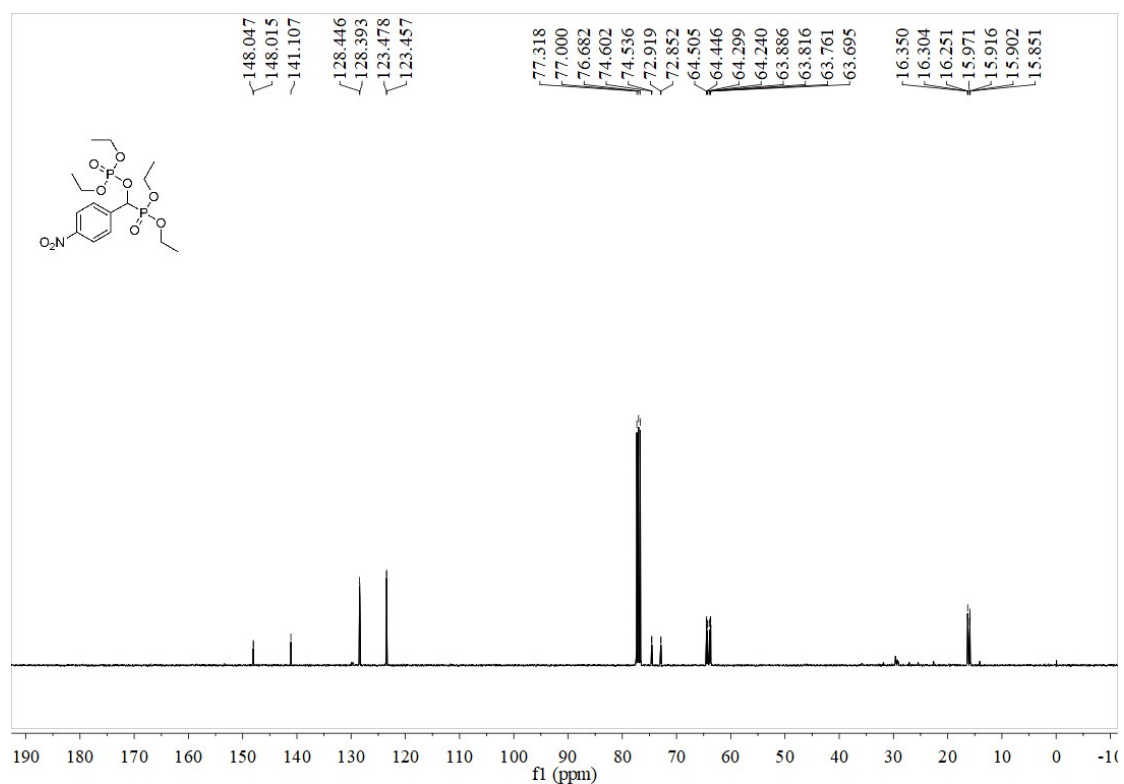
¹³C NMR spectra of (4-cyanophenyl)(diethoxyphosphoryl)methyl diethyl phosphate (**16c**).



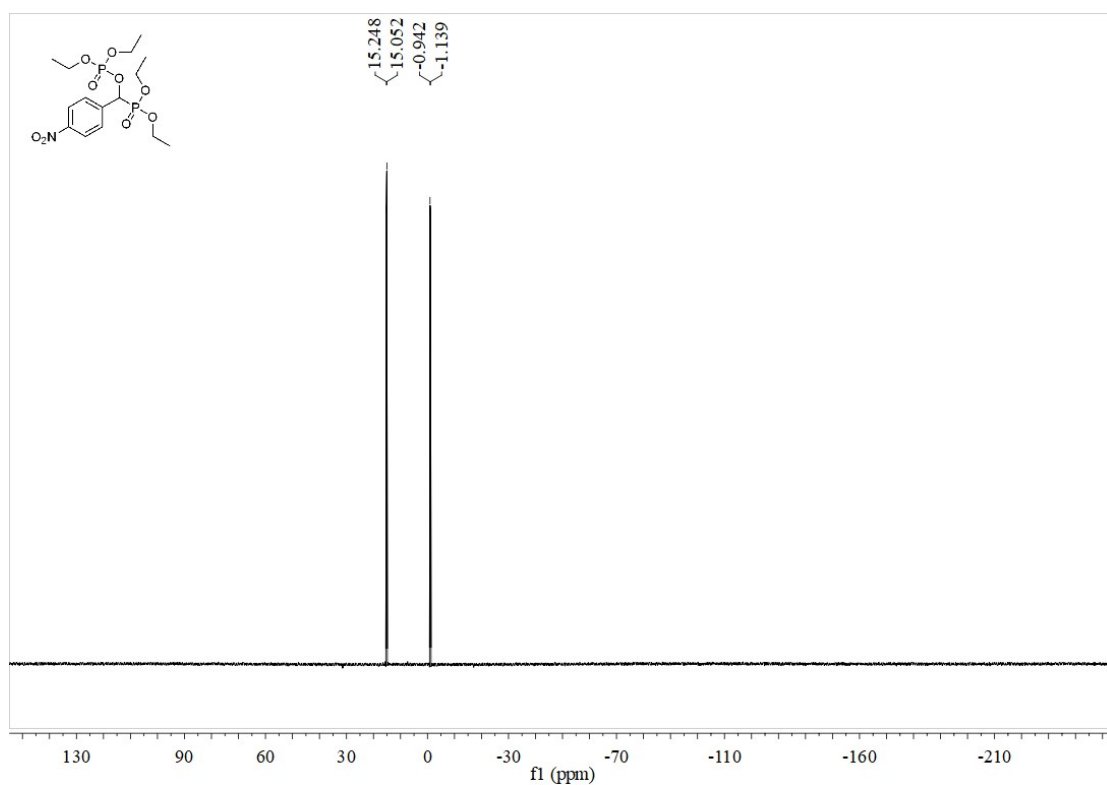
³¹P NMR spectra of (4-cyanophenyl)(diethoxyphosphoryl)methyl diethyl phosphate (**16c**).



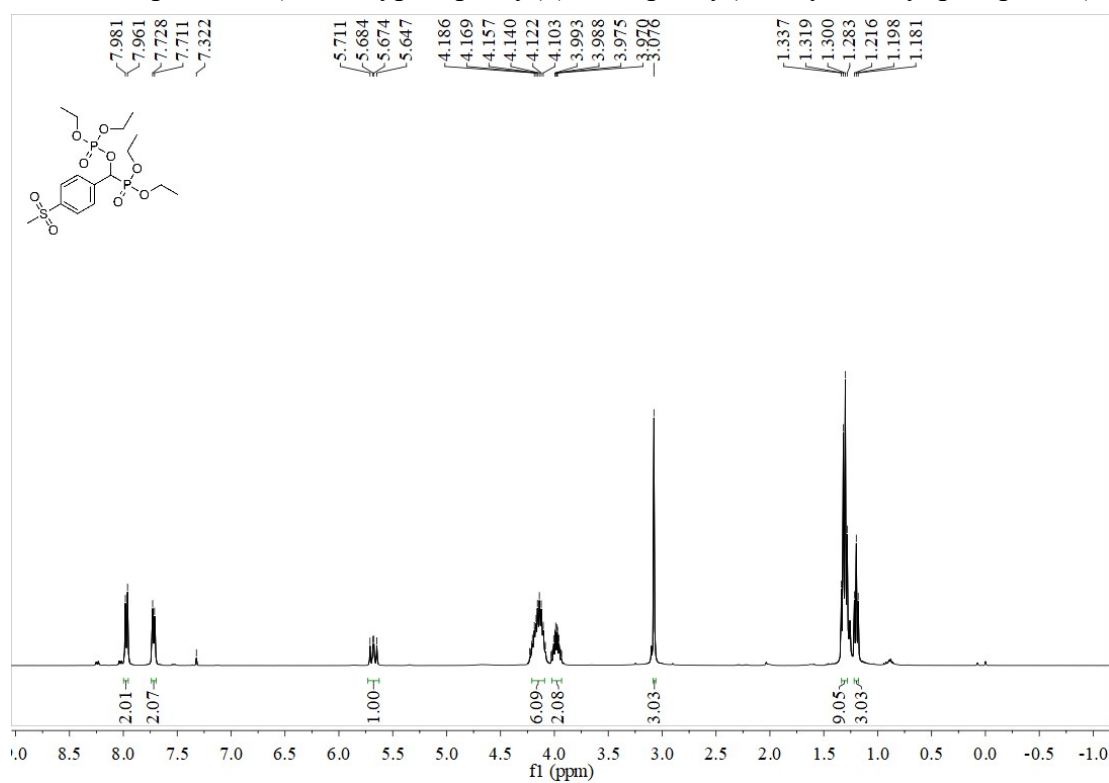
¹H NMR spectra of (diethoxyphosphoryl)(4-nitrophenyl)methyl diethyl phosphate (17c).



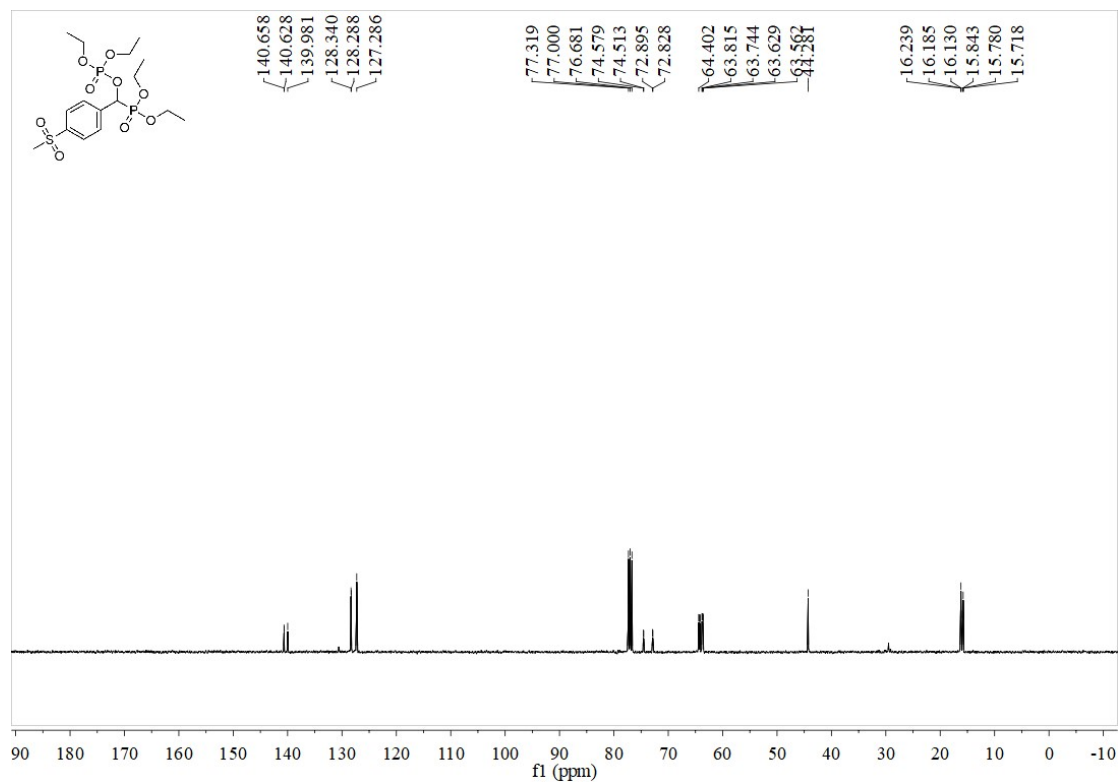
¹³C NMR spectra of (diethoxyphosphoryl)(4-nitrophenyl)methyl diethyl phosphate (17c).



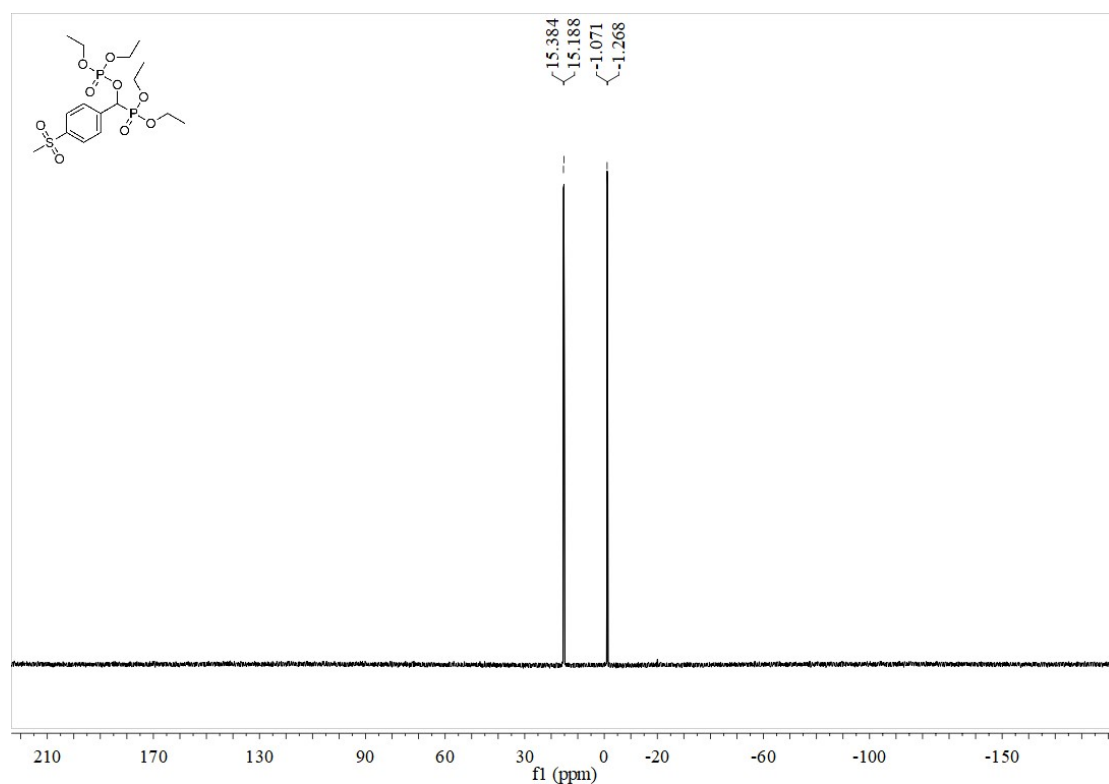
^{31}P NMR spectra of (diethoxyphosphoryl)(4-nitrophenyl)methyl diethyl phosphate (**17c**).



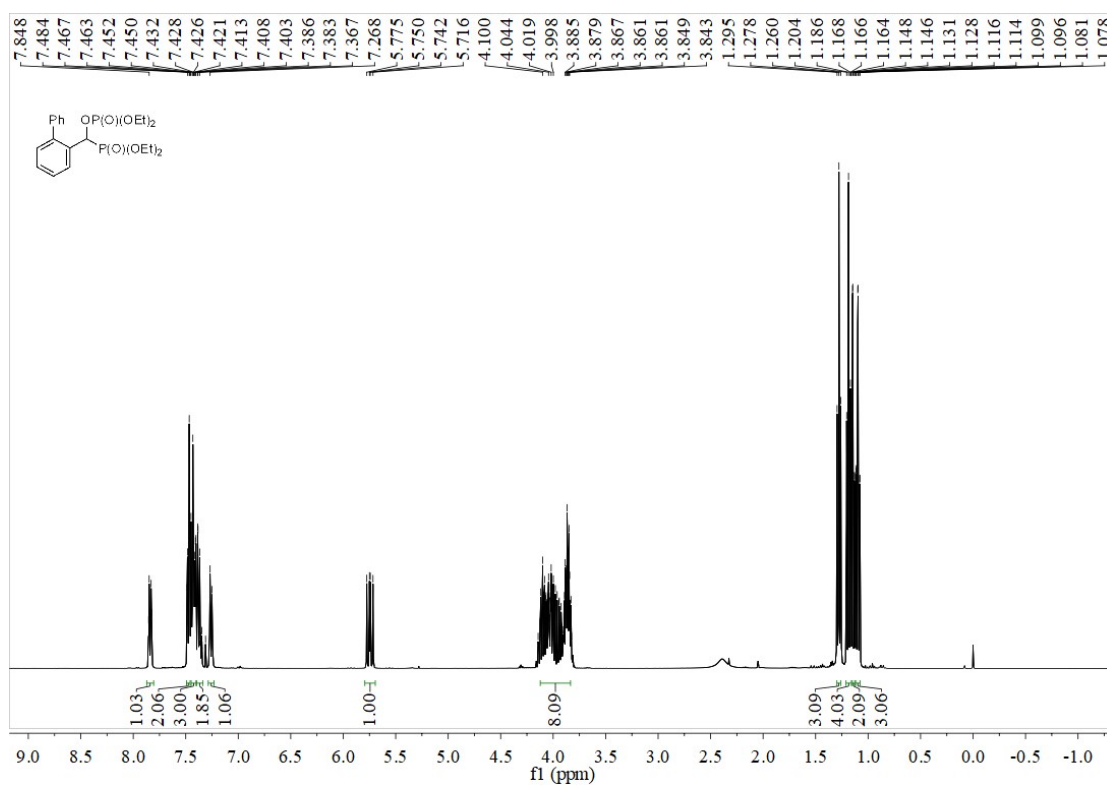
^1H NMR spectra of (diethoxyphosphoryl)(4-(methylsulfonyl)phenyl)methyl diethyl phosphate (**18c**).



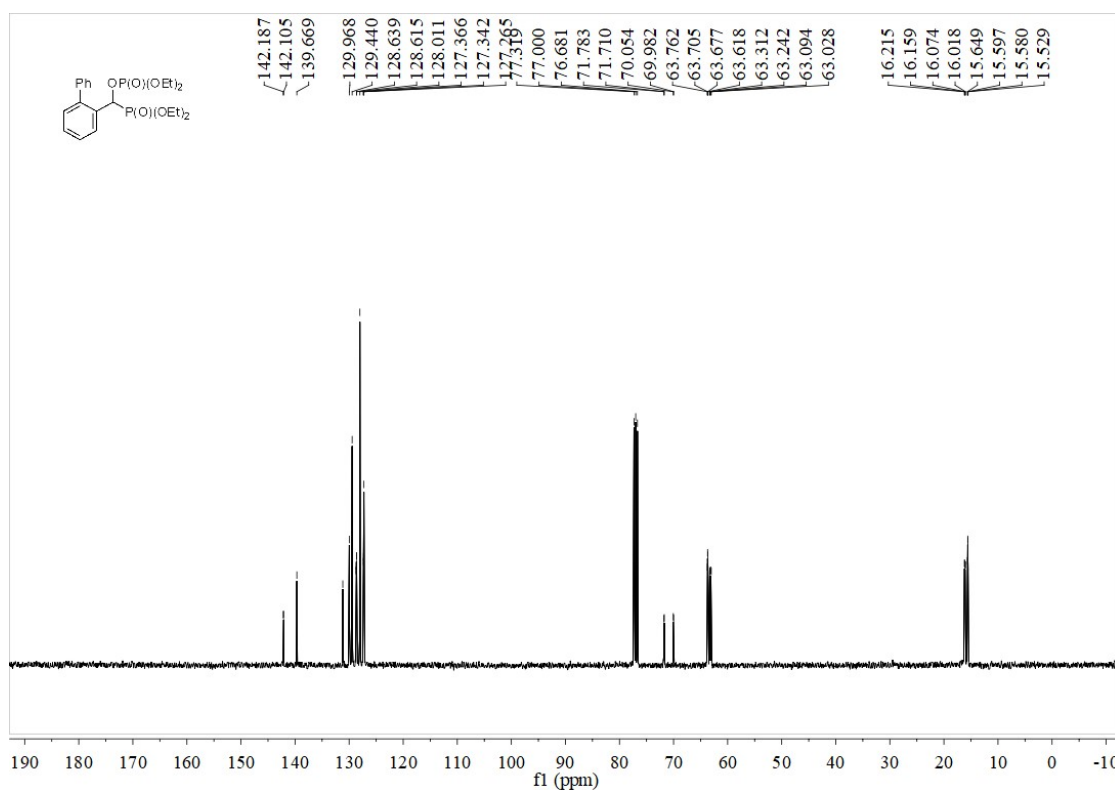
¹³C NMR spectra of (diethoxyphosphoryl)(4-(methylsulfonyl)phenyl)methyl diethyl phosphate (**18c**).



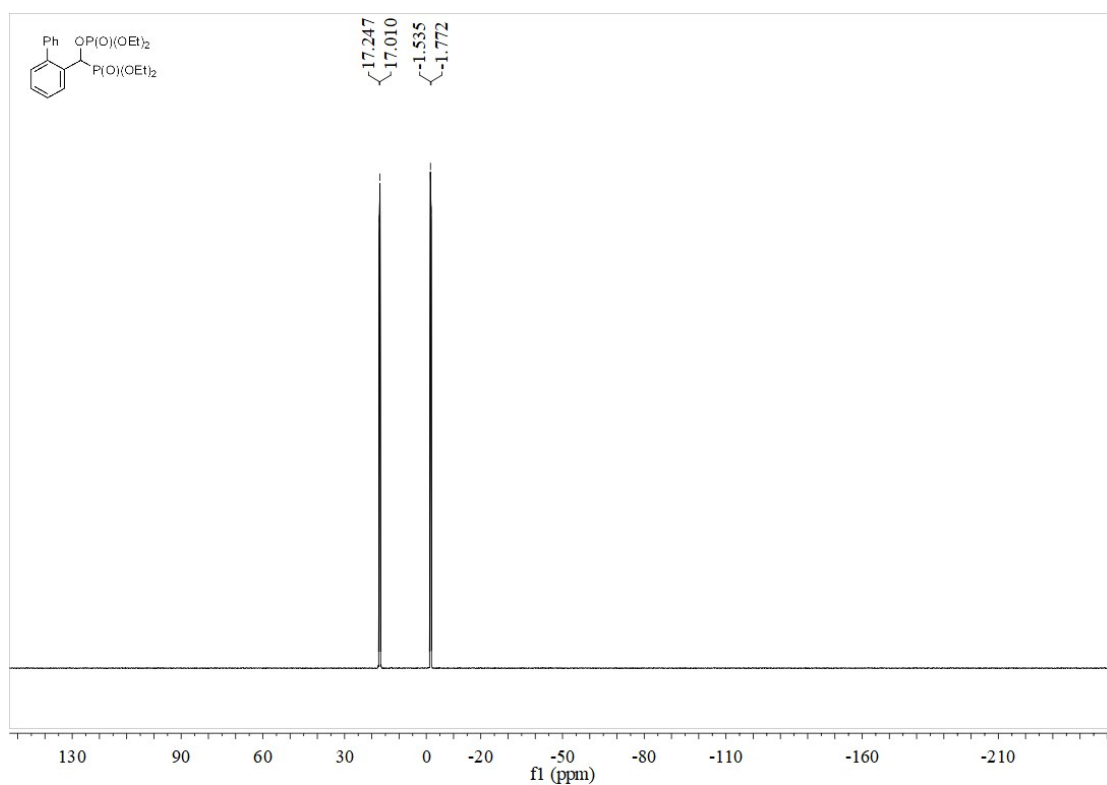
³¹P NMR spectra of (diethoxyphosphoryl)(4-(methylsulfonyl)phenyl)methyl diethyl phosphate (**18c**).



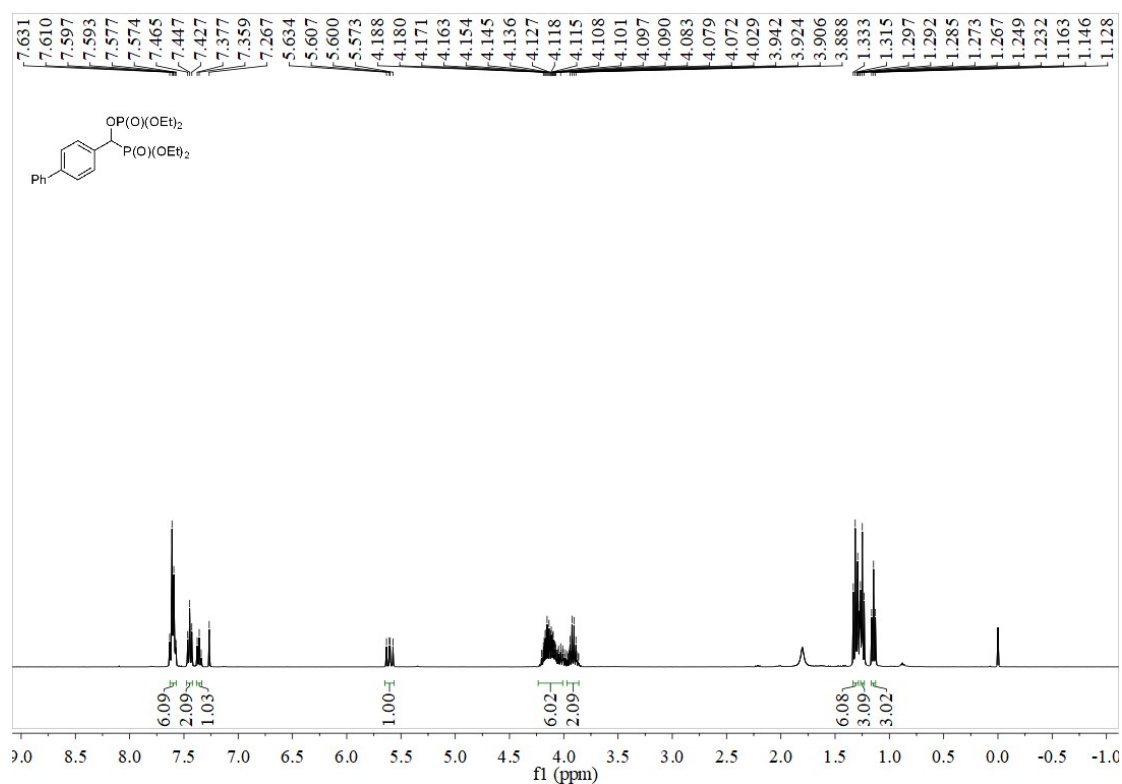
¹H NMR spectra of [1,1'-biphenyl]-2-yl(diethoxyphosphoryl)methyl diethyl phosphate (**19c**).



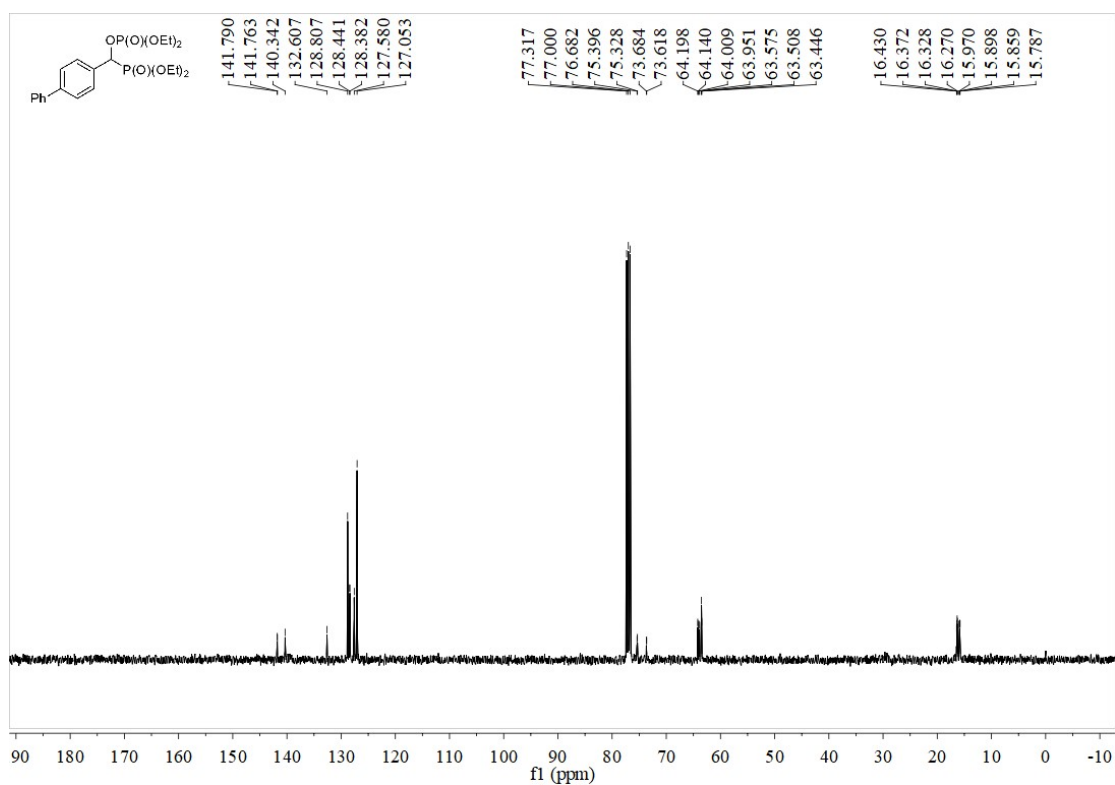
¹³C NMR spectra of [1,1'-biphenyl]-2-yl(diethoxyphosphoryl)methyl diethyl phosphate (**19c**).



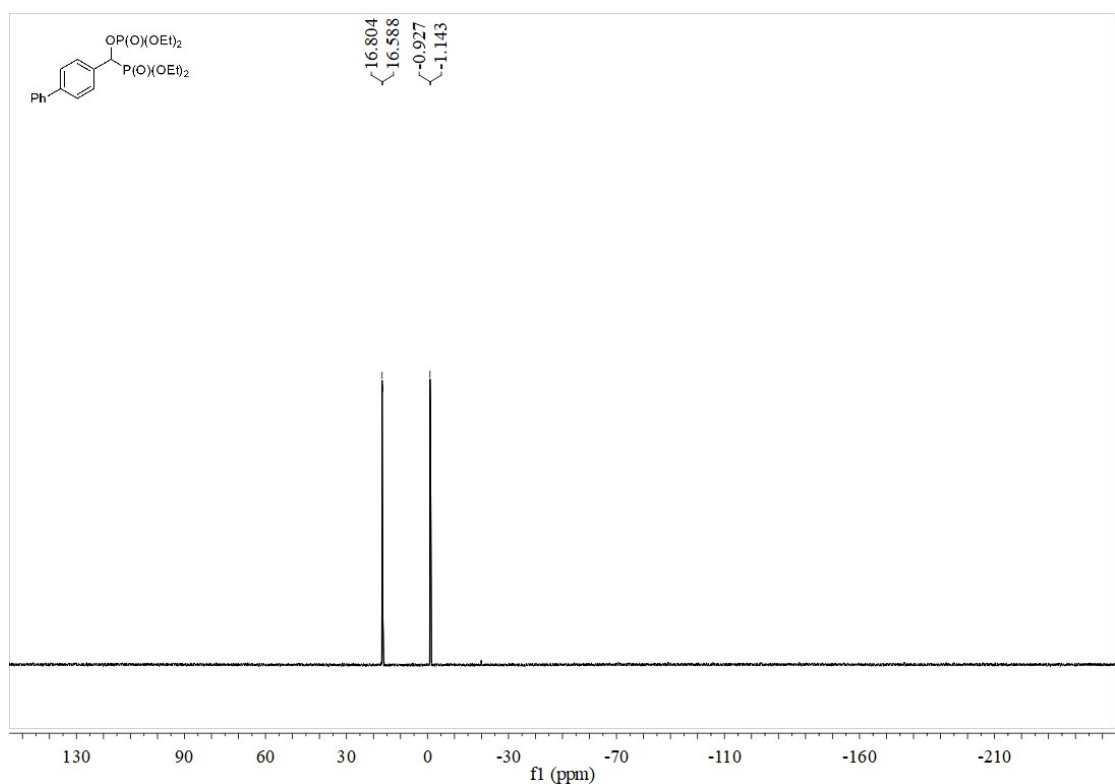
³¹P NMR spectra of [1,1'-biphenyl]-2-yl(diethoxyphosphoryl)methyl diethyl phosphate (19c).



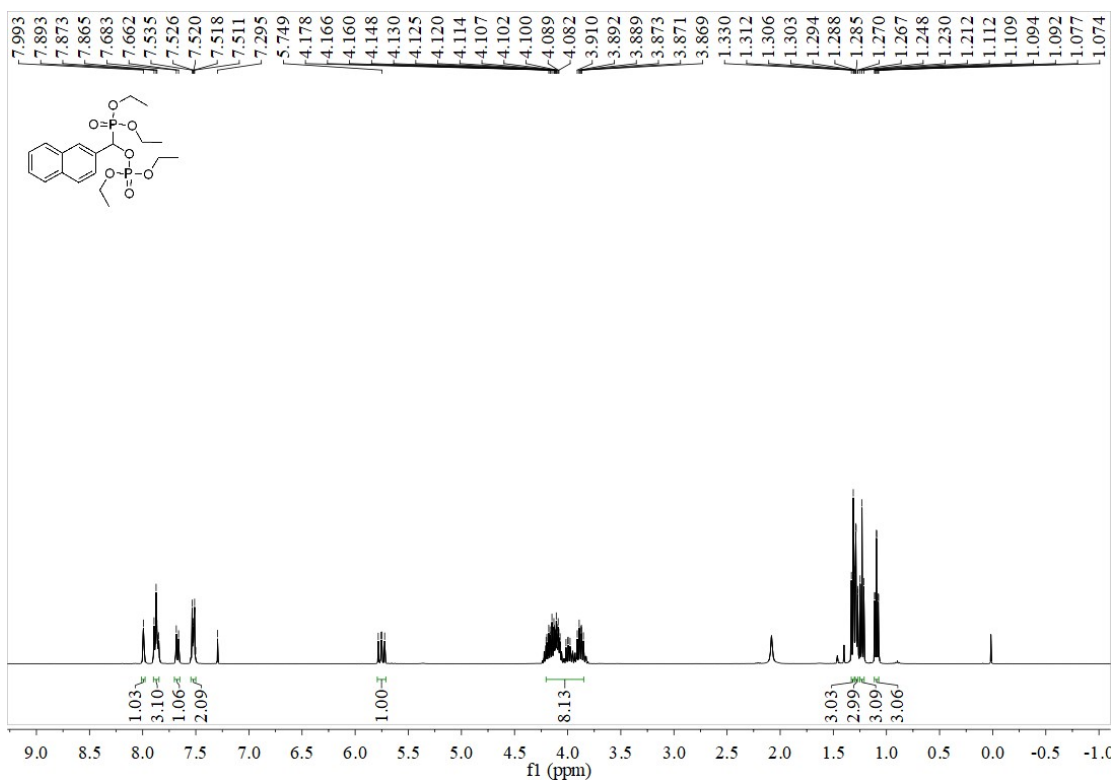
¹H NMR spectra of [1,1'-biphenyl]-4-yl(diethoxyphosphoryl)methyl diethyl phosphate (20c).



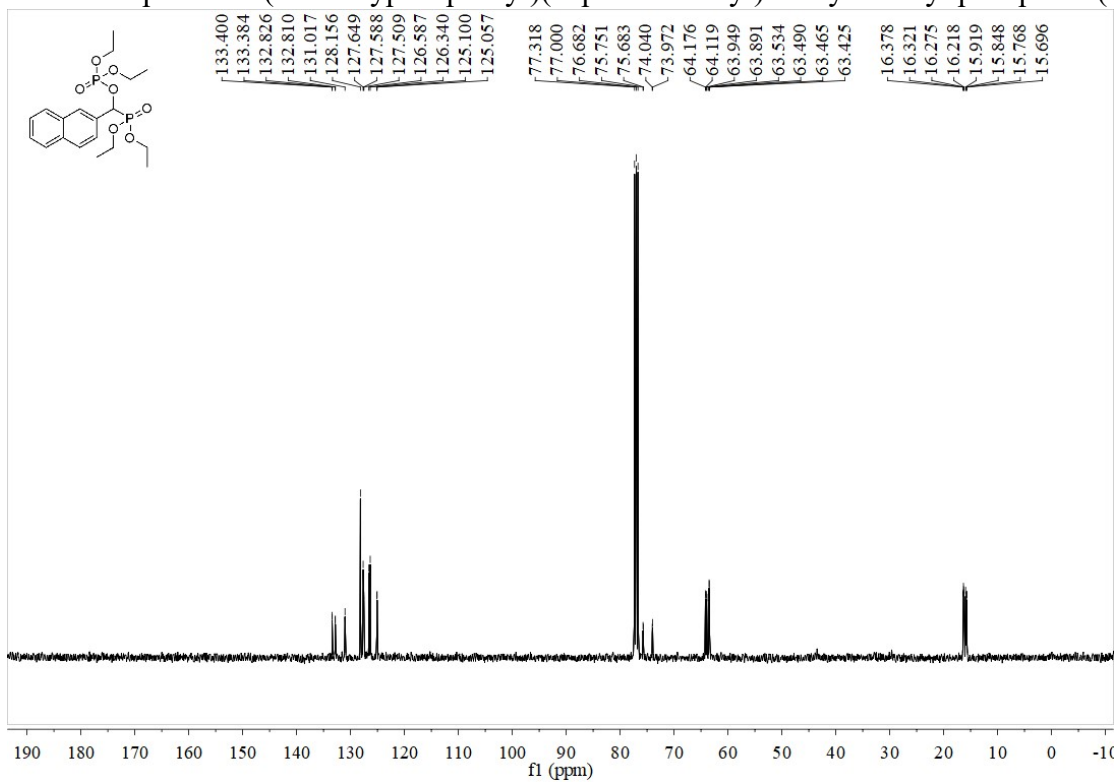
¹³C NMR spectra of [1,1'-biphenyl]-4-yl(diethoxyphosphoryl)methyl diethyl phosphate (**20c**).



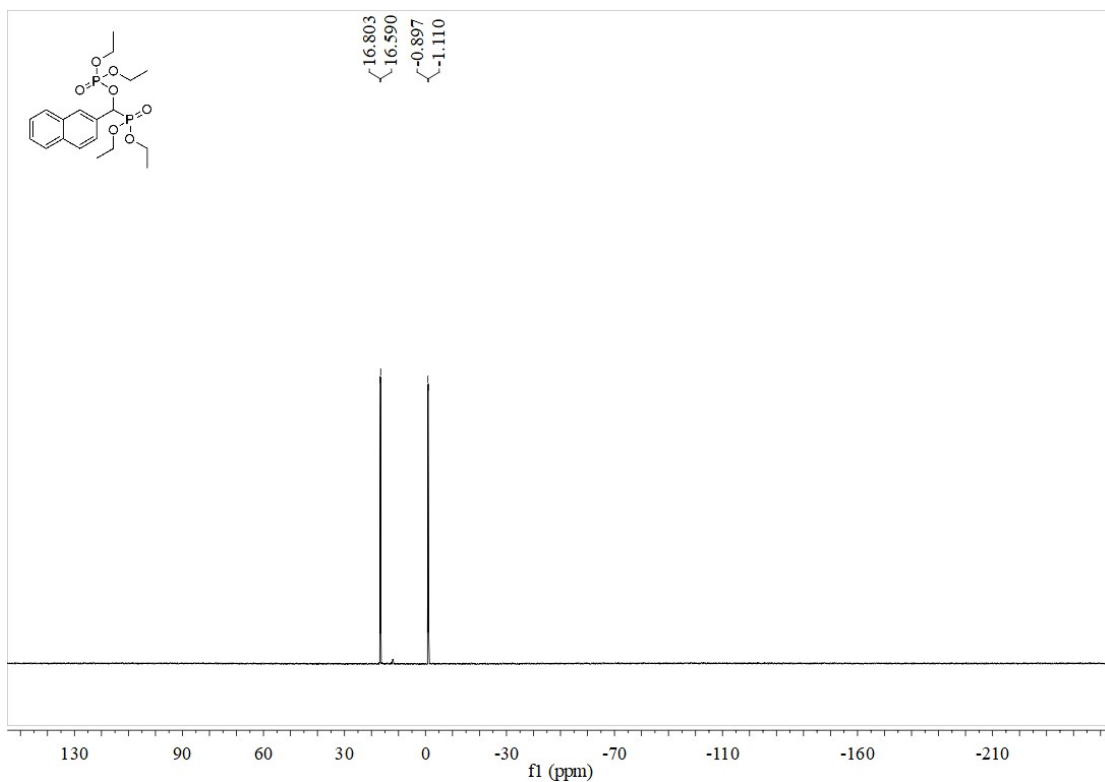
³¹P NMR spectra of [1,1'-biphenyl]-4-yl(diethoxyphosphoryl)methyl diethyl phosphate (**20c**).



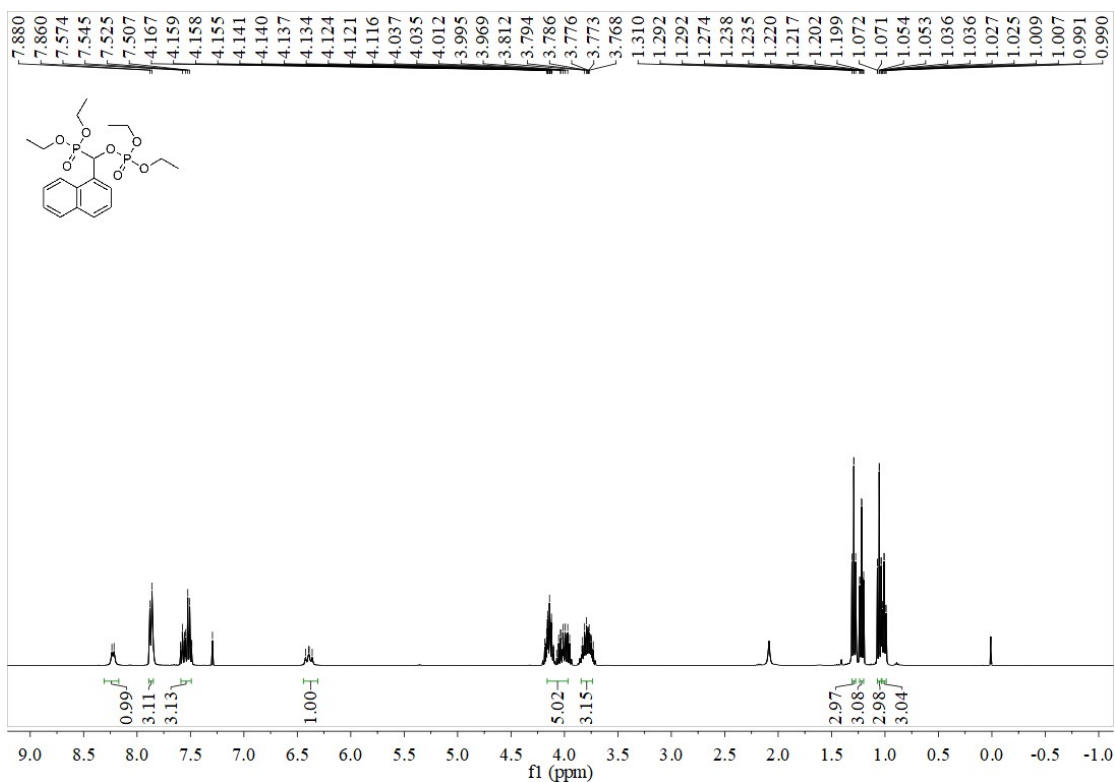
¹H NMR spectra of (diethoxyphosphoryl)(naphthalen-2-yl)methyl diethyl phosphate (21c).



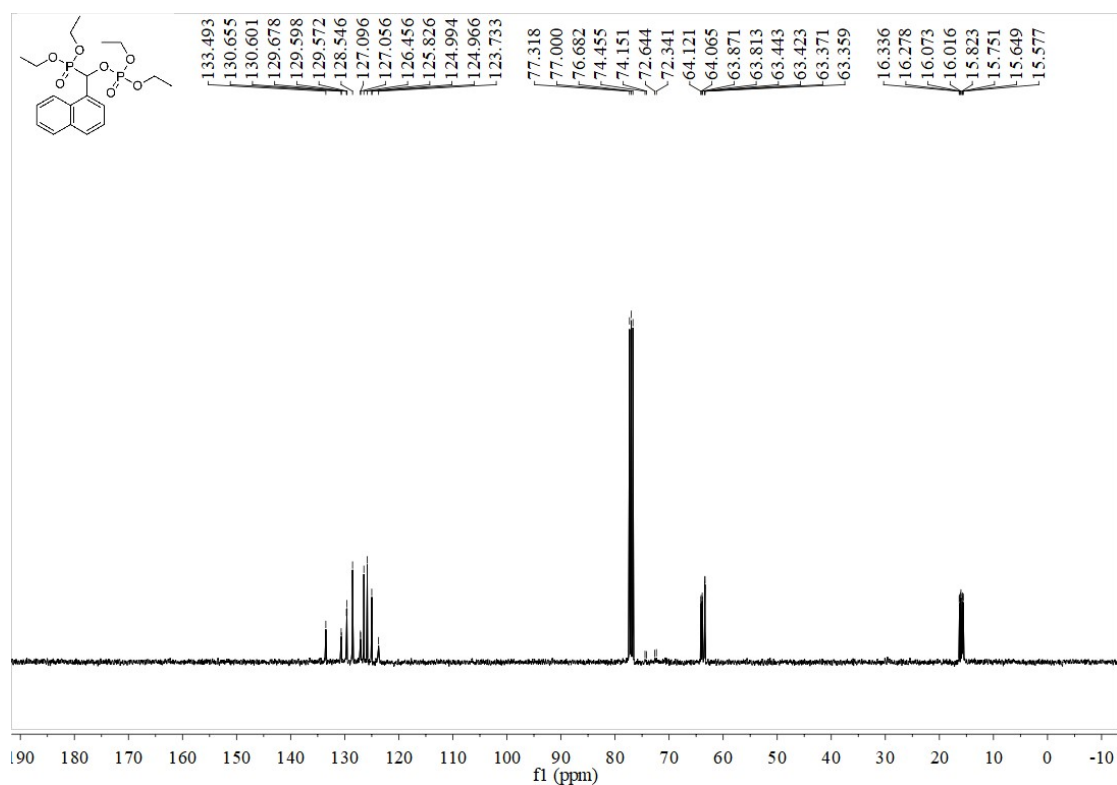
¹³C NMR spectra of (diethoxyphosphoryl)(naphthalen-2-yl)methyl diethyl phosphate (21c).



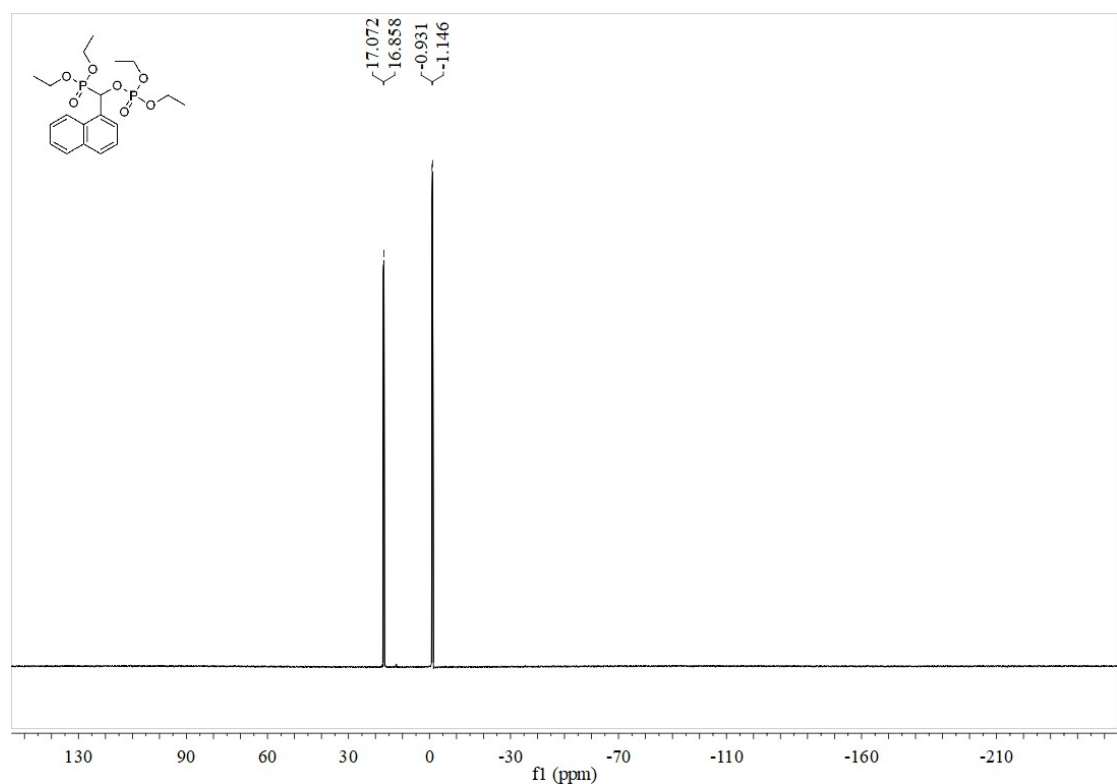
³¹P NMR spectra of (diethoxyphosphoryl)(naphthalen-2-yl)methyl diethyl phosphate (**21c**).



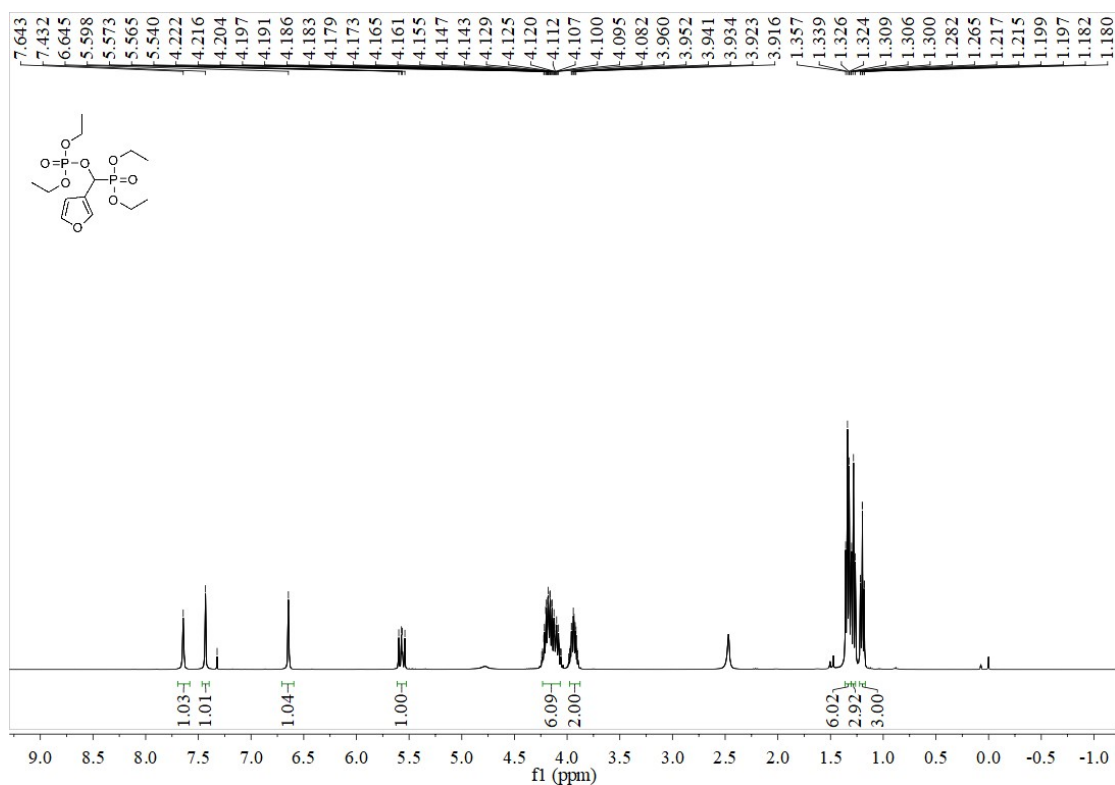
¹H NMR spectra of (diethoxyphosphoryl)(naphthalen-1-yl)methyl diethyl phosphate (**22c**).



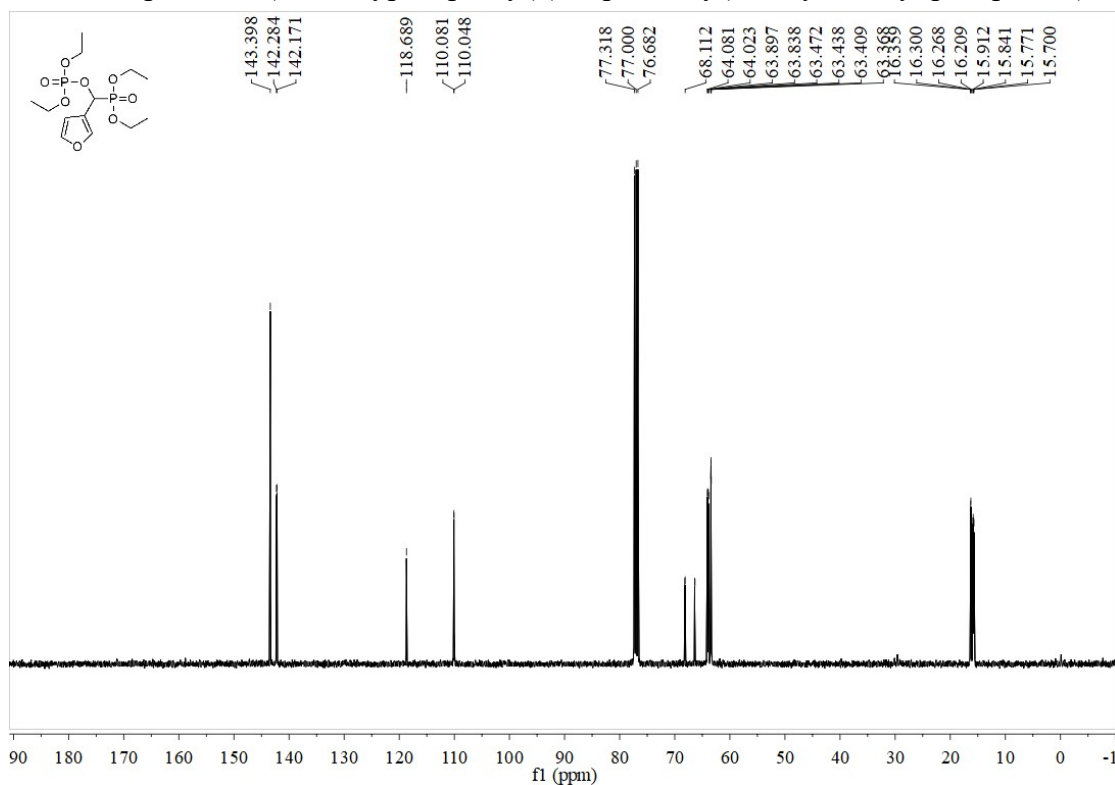
^{13}C NMR spectra of (diethoxyphosphoryl)(naphthalen-1-yl)methyl diethyl phosphate (**22c**).



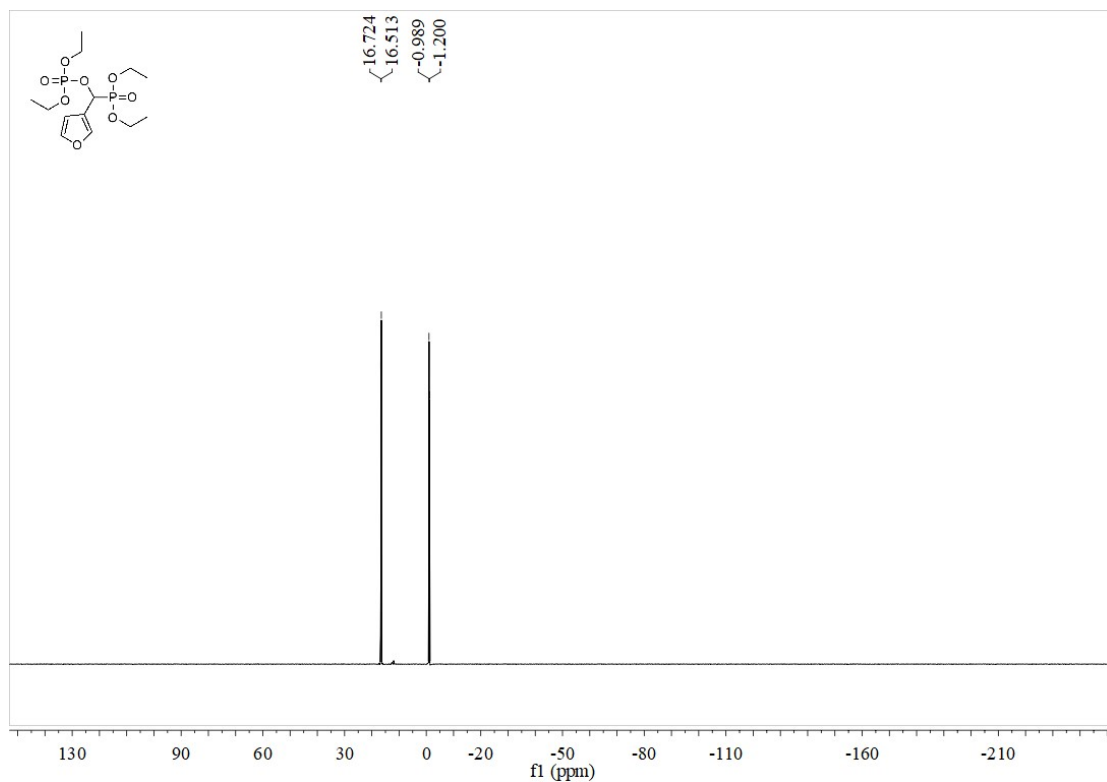
^{31}P NMR spectra of (diethoxyphosphoryl)(naphthalen-1-yl)methyl diethyl phosphate (**22c**).



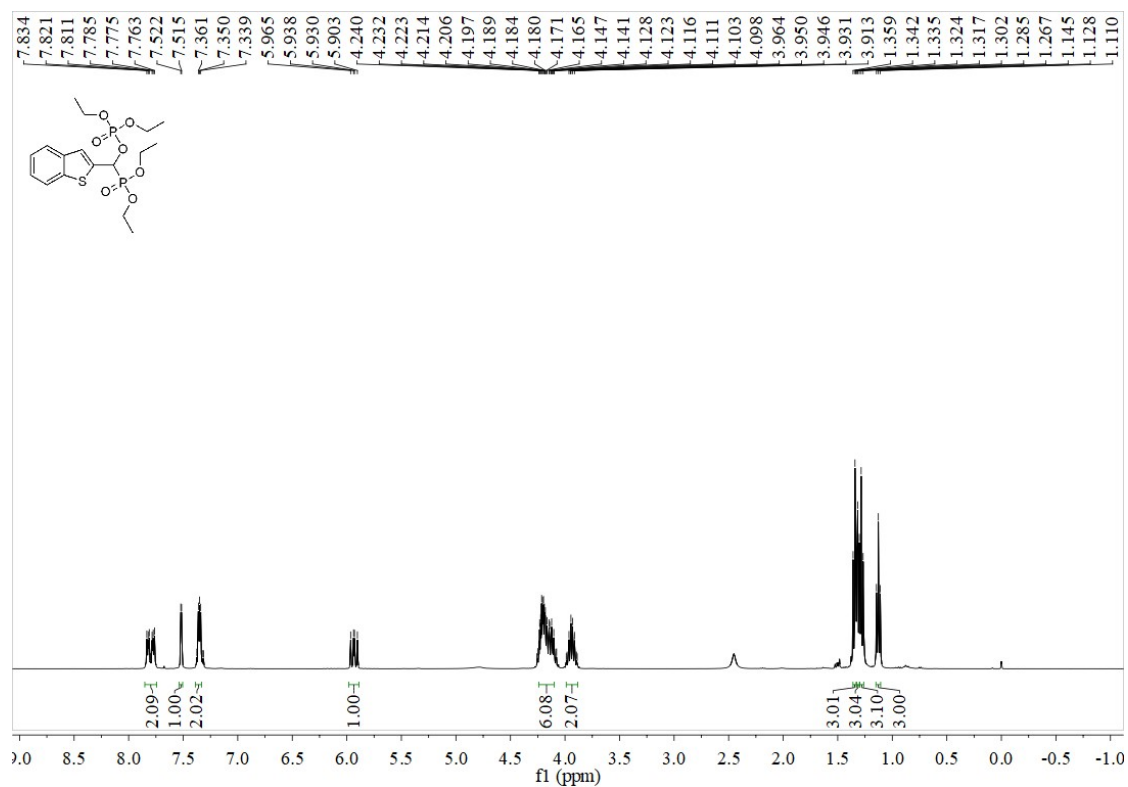
¹H NMR spectra of (diethoxyphosphoryl)(thiophen-3-yl)methyl diethyl phosphate (**23c**).



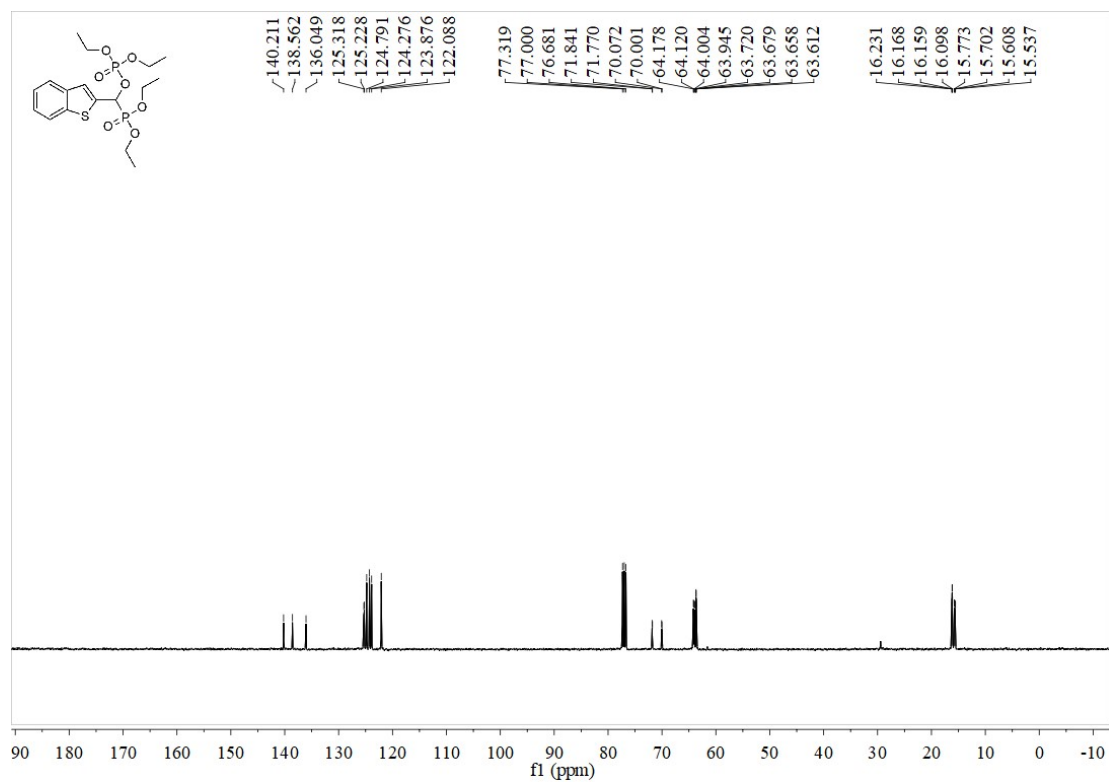
¹³C NMR spectra of (diethoxyphosphoryl)(thiophen-3-yl)methyl diethyl phosphate (**23c**).



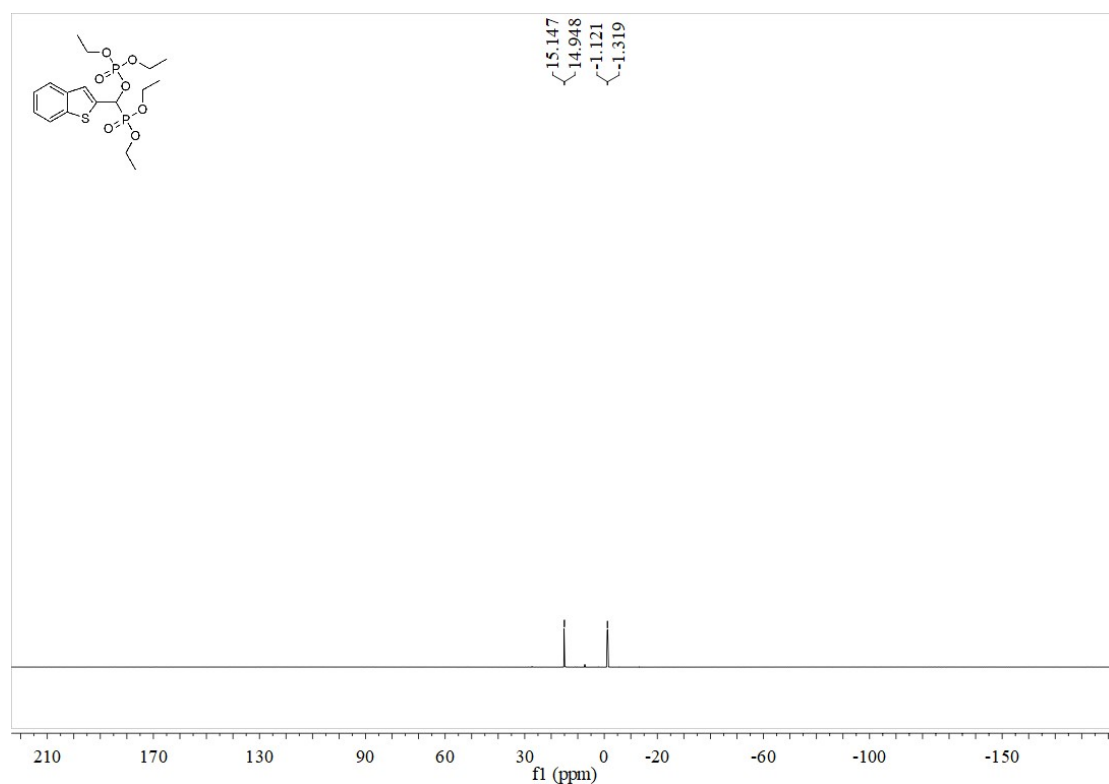
^{31}P NMR spectra of (diethoxyphosphoryl)(thiophen-3-yl)methyl diethyl phosphate (**23c**).



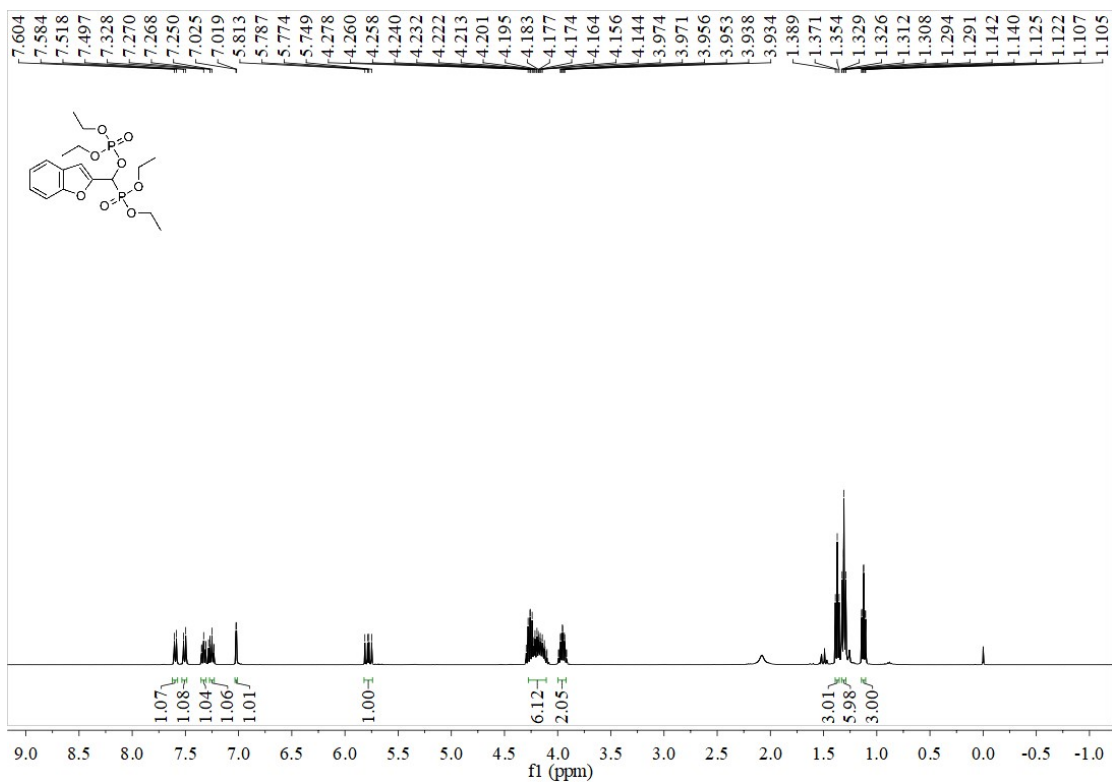
^1H NMR spectra of Benzo[b]thiophen-2-yl(diethoxyphosphoryl)methyl diethyl phosphate (**24c**).



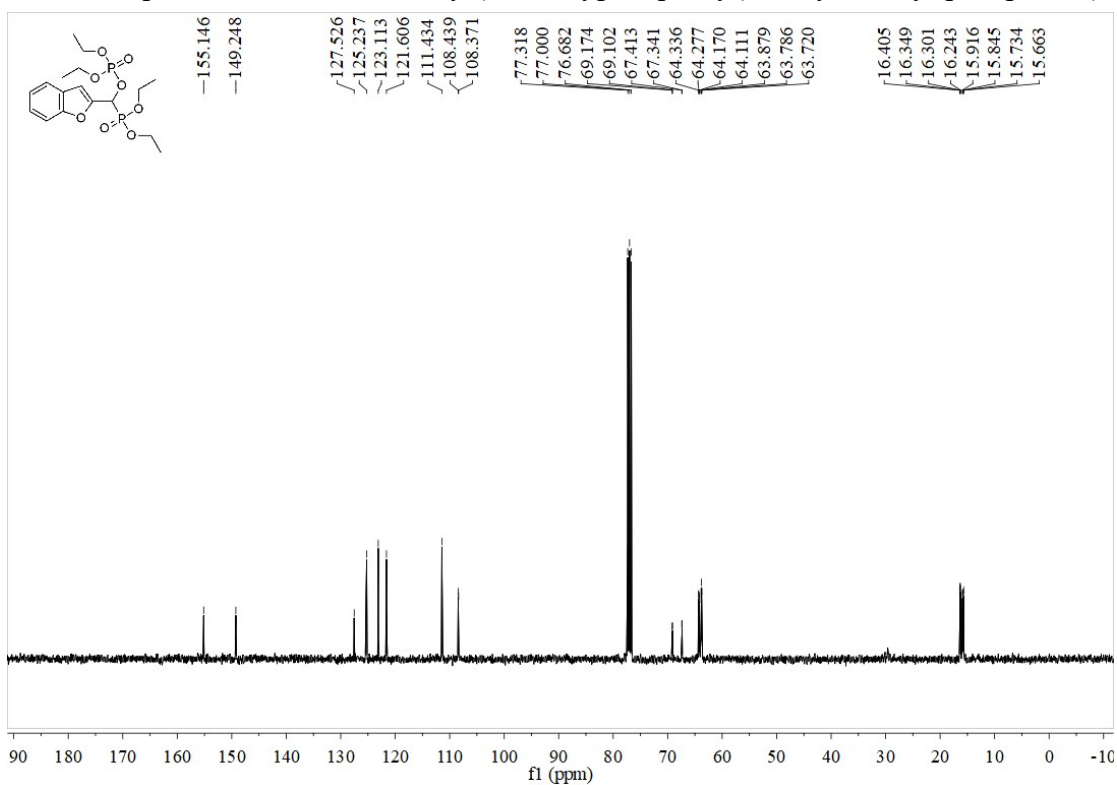
¹³C NMR spectra of Benzo[b]thiophen-2-yl(diethoxyphosphoryl)methyl diethyl phosphate (24c).



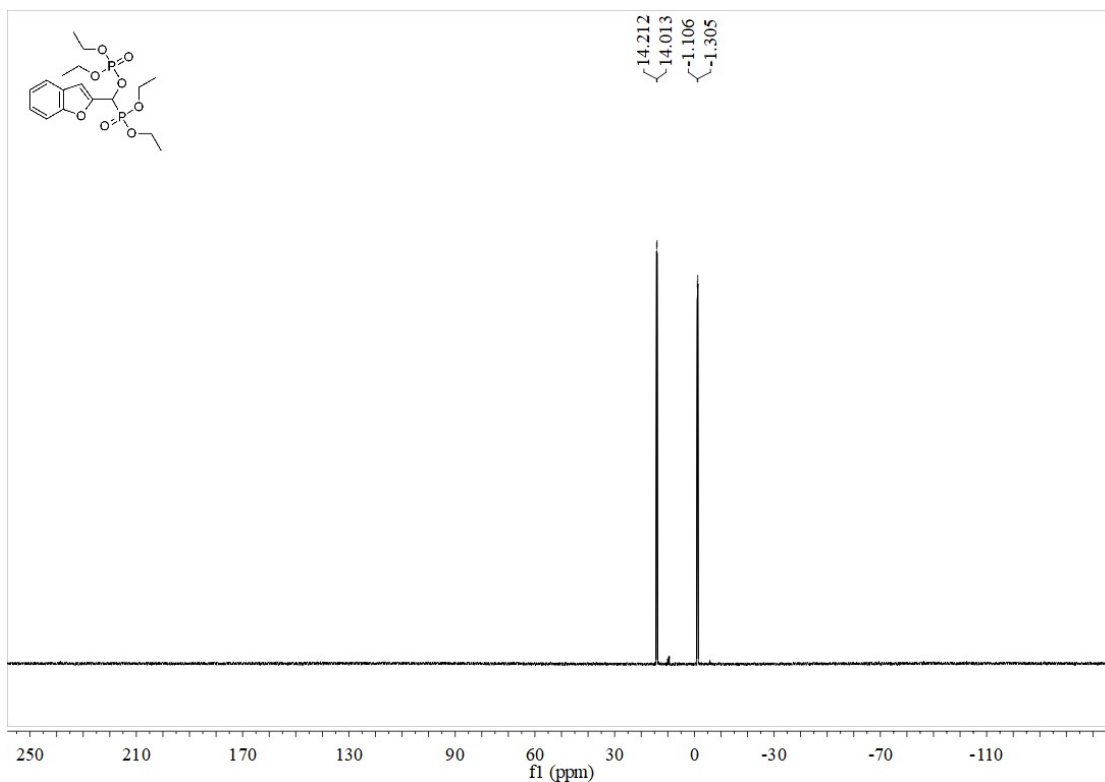
³¹P NMR spectra of Benzo[b]thiophen-2-yl(diethoxyphosphoryl)methyl diethyl phosphate (24c).



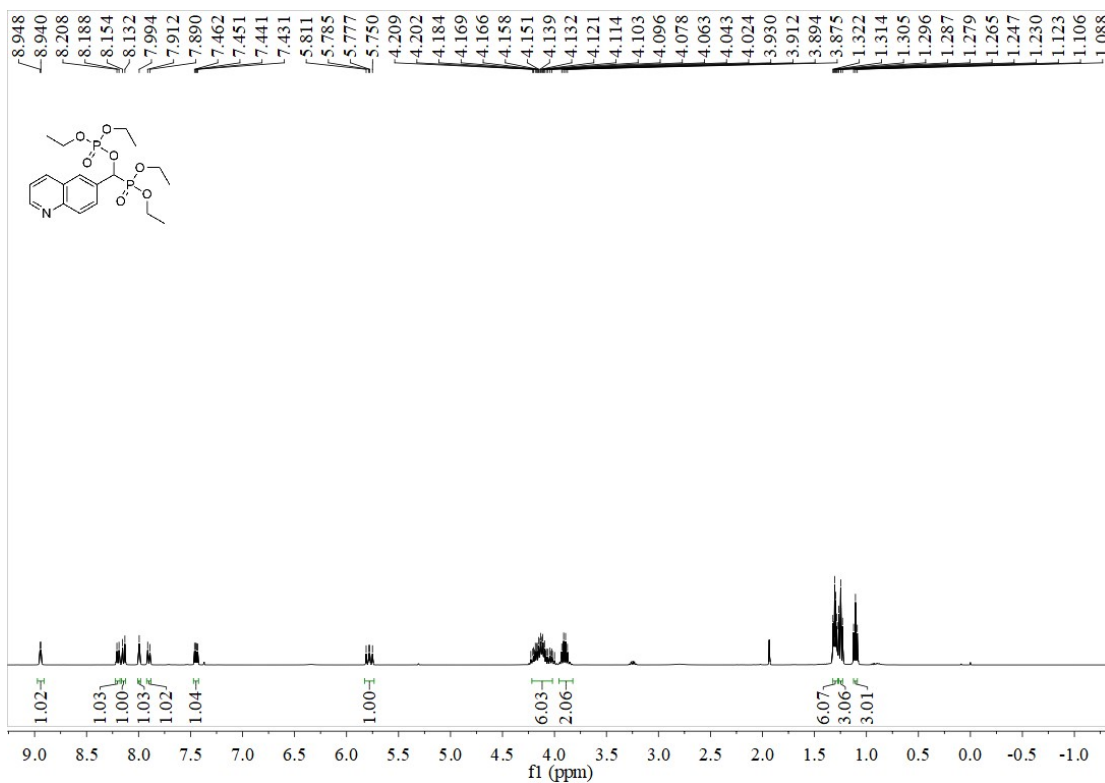
¹H NMR spectra of Benzofuran-2-yl(diethoxyphosphoryl)methyl diethyl phosphate (25c).



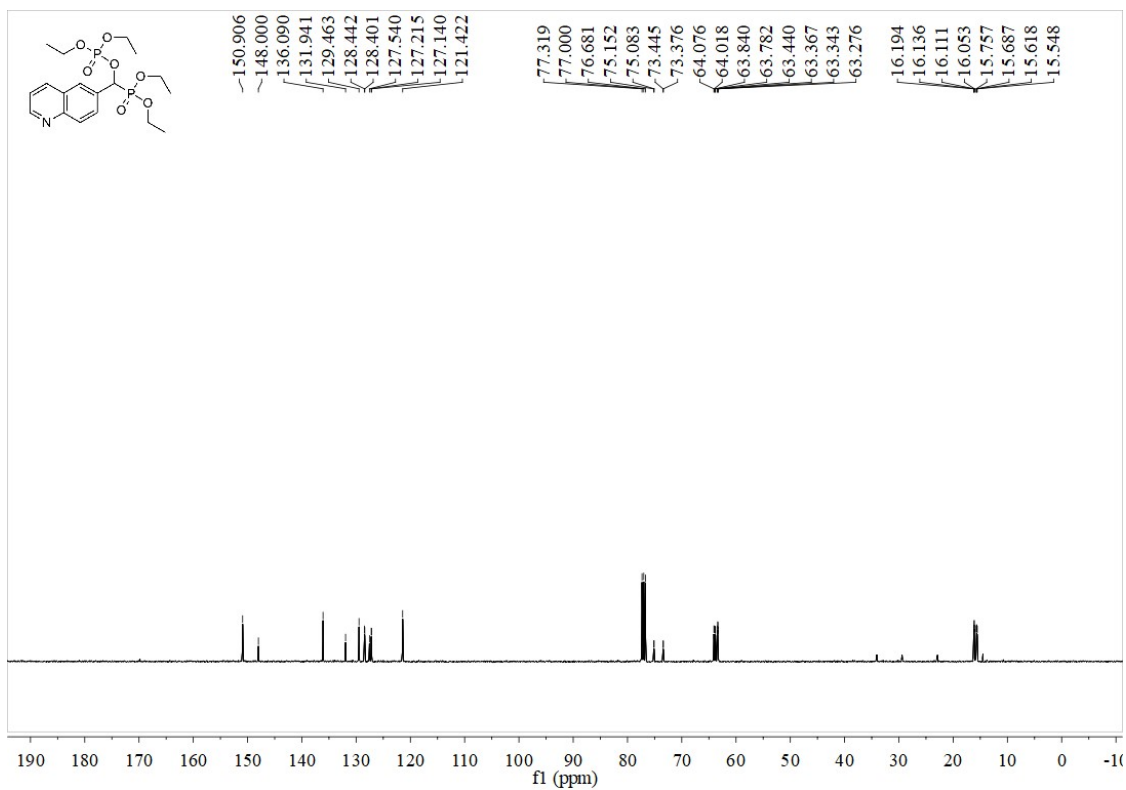
¹³C NMR spectra of Benzofuran-2-yl(diethoxyphosphoryl)methyl diethyl phosphate (25c).



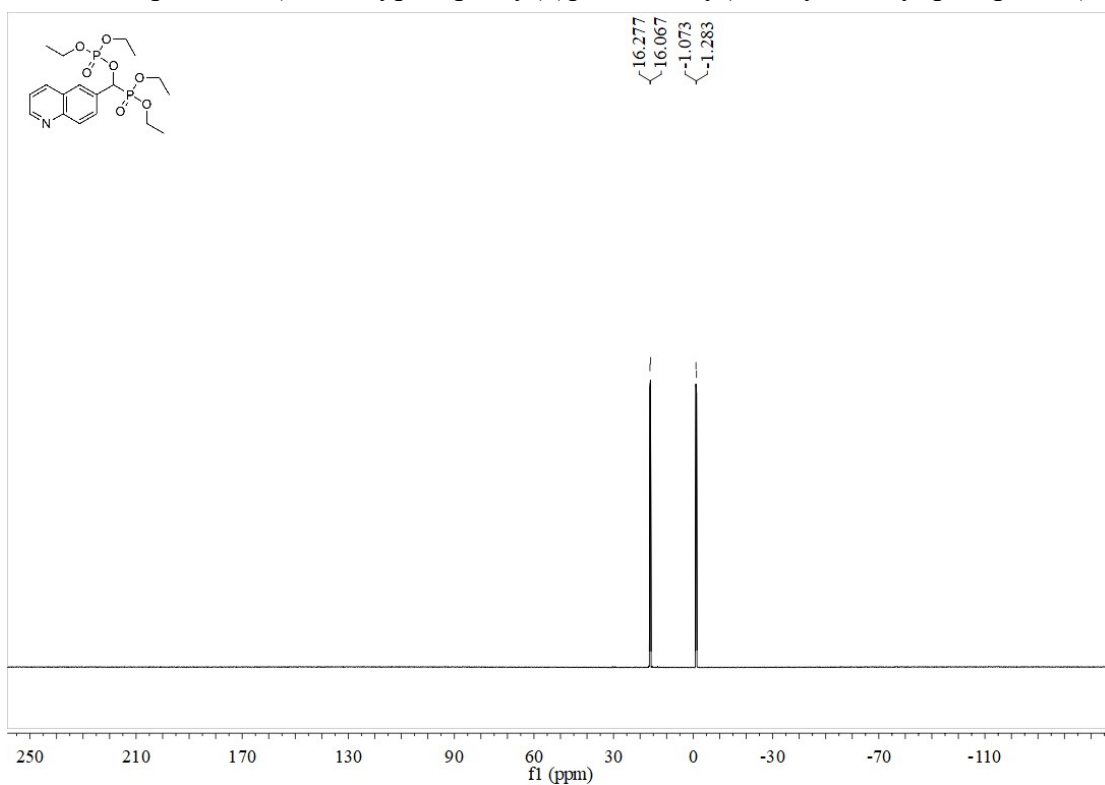
³¹P NMR spectra of Benzofuran-2-yl(diethoxyphosphoryl)methyl diethyl phosphate (**25c**).



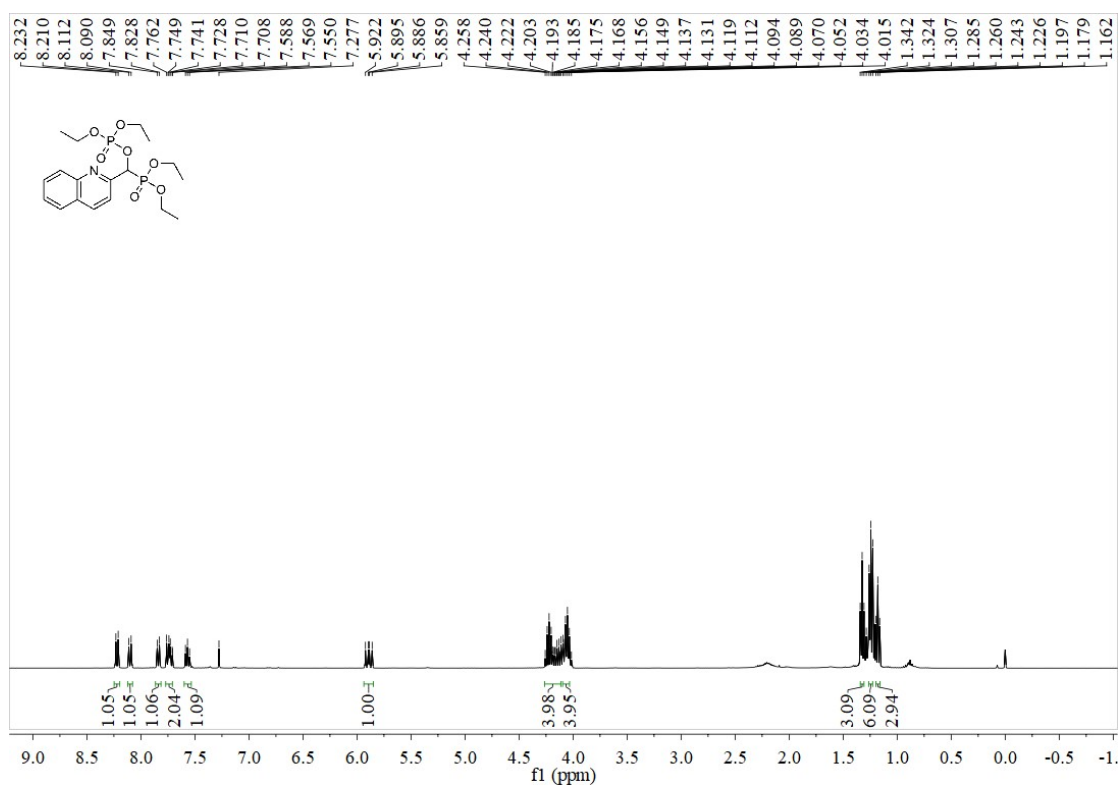
¹H NMR spectra of (diethoxyphosphoryl)(quinolin-6-yl)methyl diethyl phosphate (**26c**).



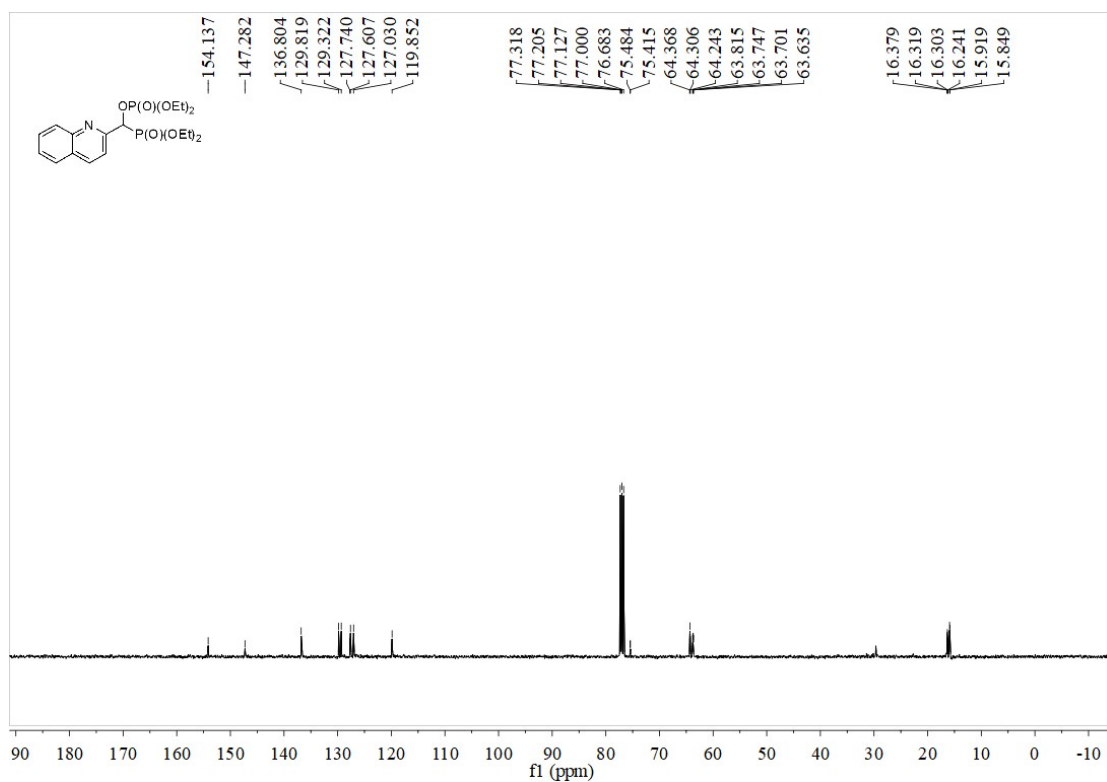
¹³C NMR spectra of (diethoxyphosphoryl)(quinolin-6-yl)methyl diethyl phosphate (**26c**).



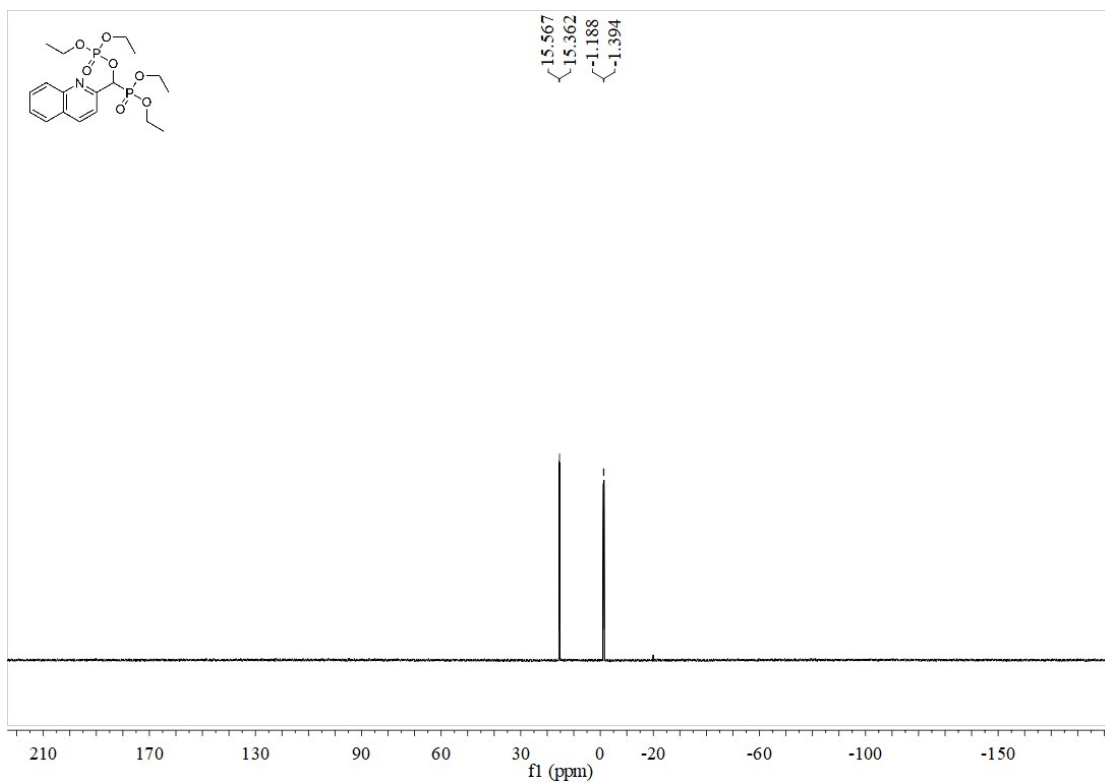
³¹P NMR spectra of (diethoxyphosphoryl)(quinolin-6-yl)methyl diethyl phosphate (**26c**).



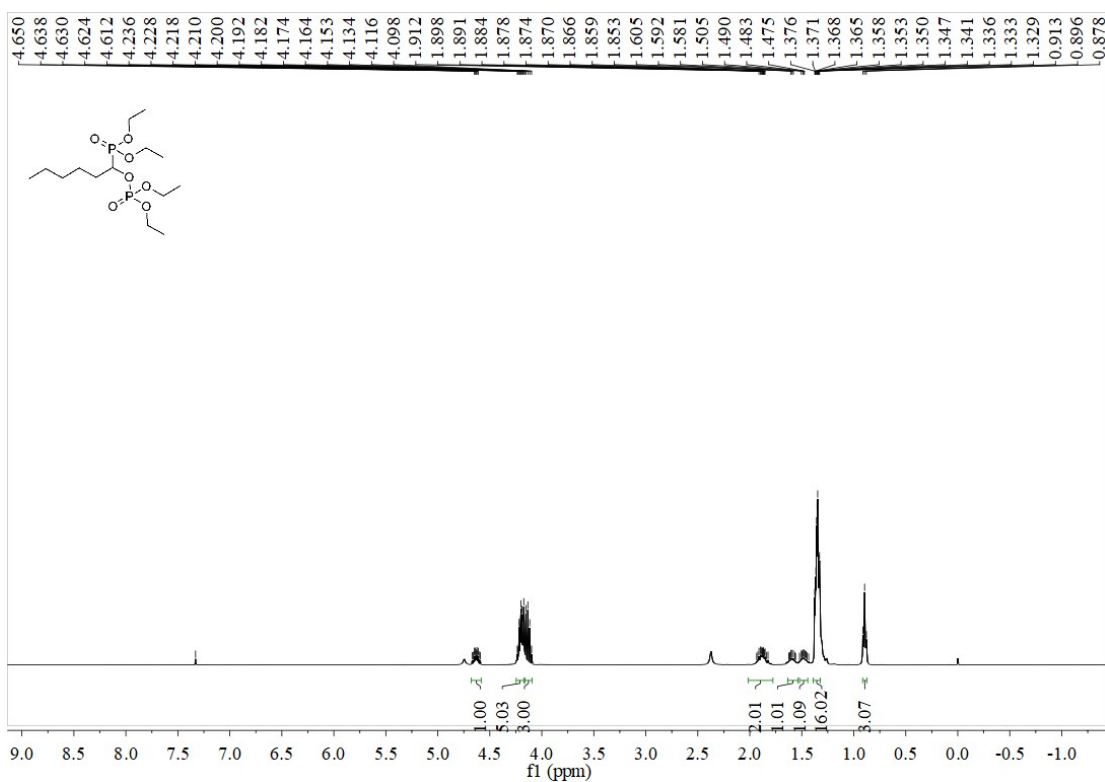
¹H NMR spectra of (diethoxyphosphoryl)(quinolin-2-yl)methyl diethyl phosphate (27c).



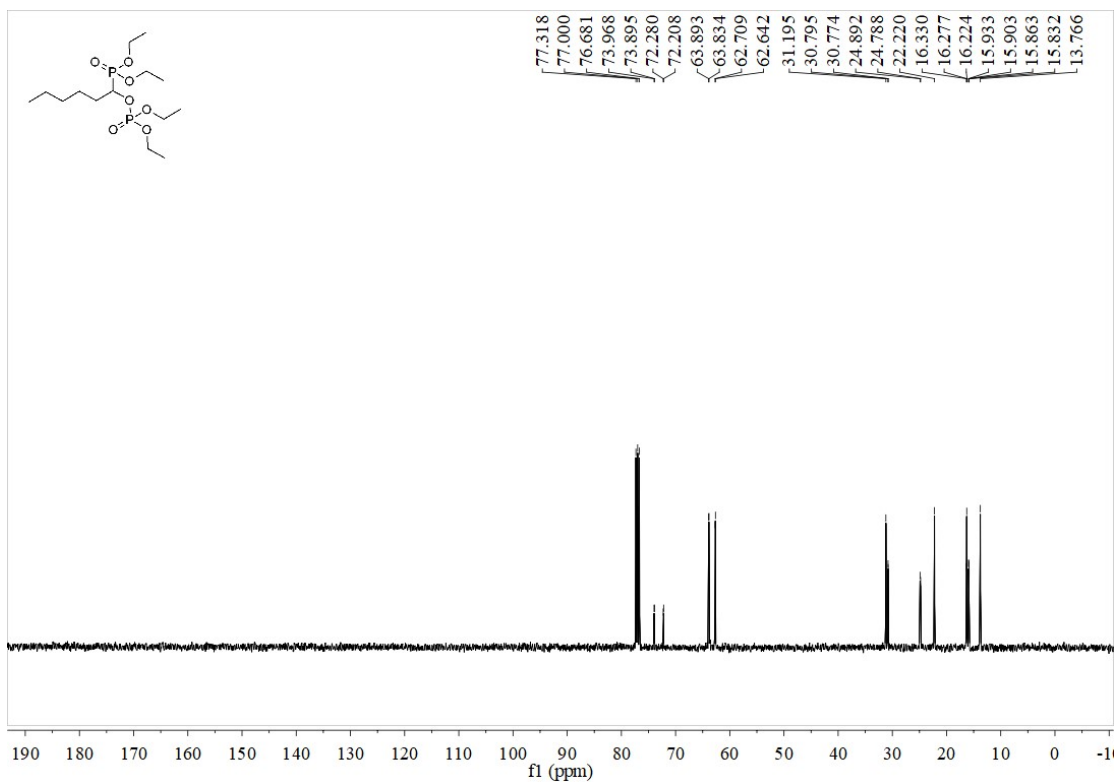
¹³C NMR spectra of (diethoxyphosphoryl)(quinolin-2-yl)methyl diethyl phosphate (27c).



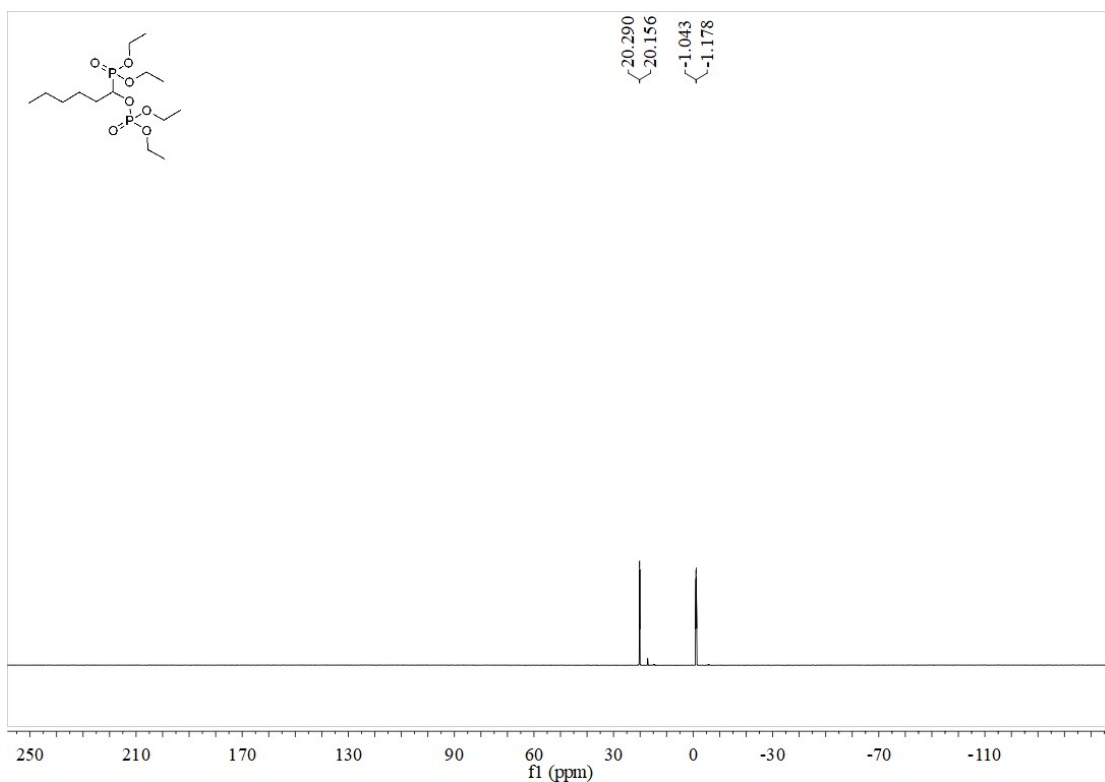
³¹P NMR spectra of (diethoxyphosphoryl)(quinolin-2-yl)methyl diethyl phosphate (**27c**).



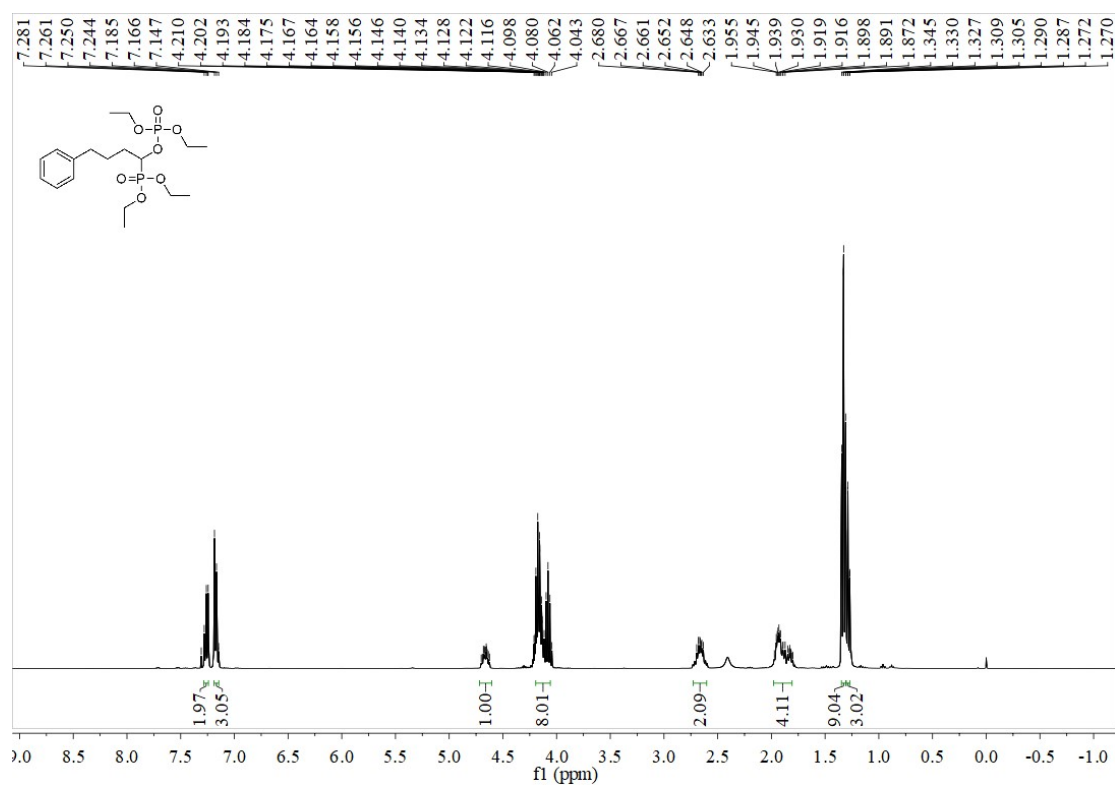
¹H NMR spectra of 1-(diethoxyphosphoryl)hexyl diethyl phosphate (**28c**).



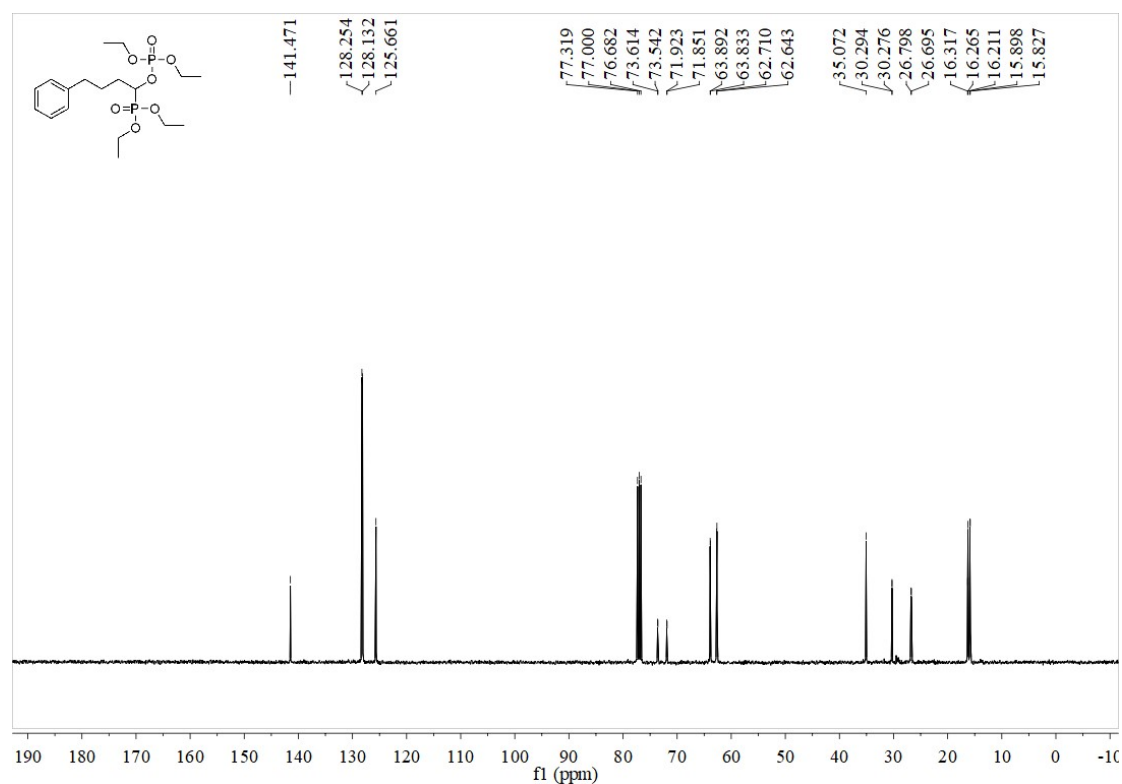
¹³C NMR spectra of 1-(diethoxyphosphoryl)hexyl diethyl phosphate (**28c**).



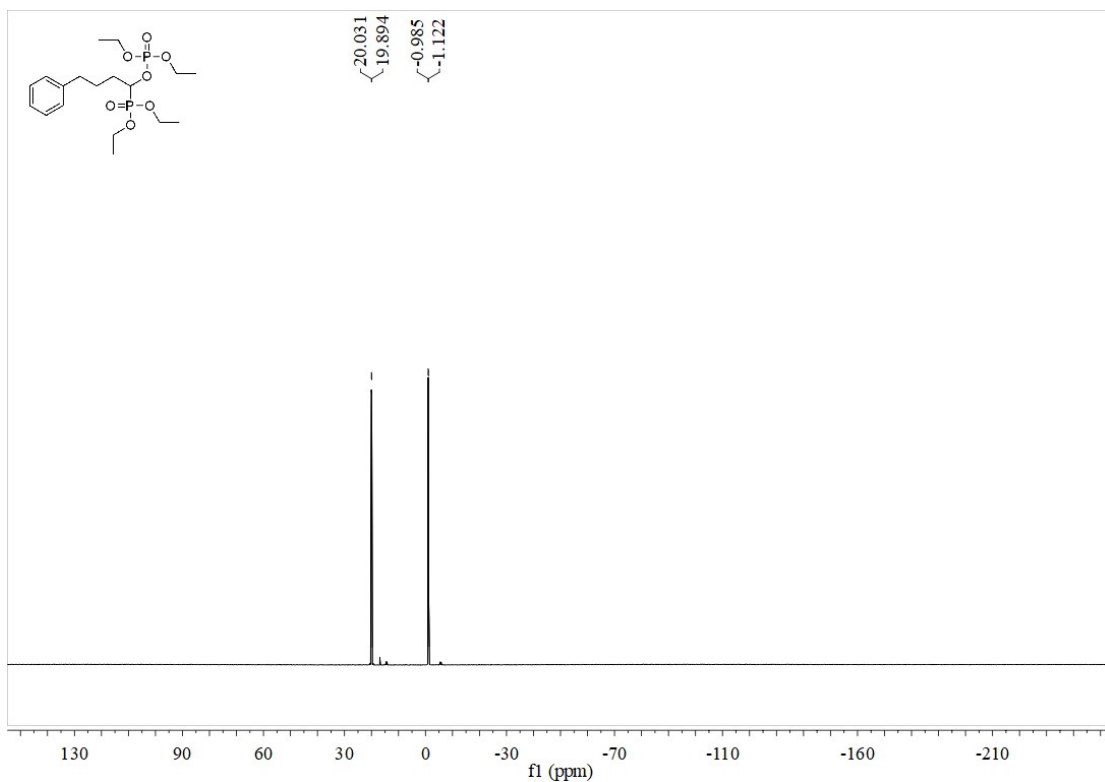
³¹P NMR spectra of 1-(diethoxyphosphoryl)hexyl diethyl phosphate (**28c**).



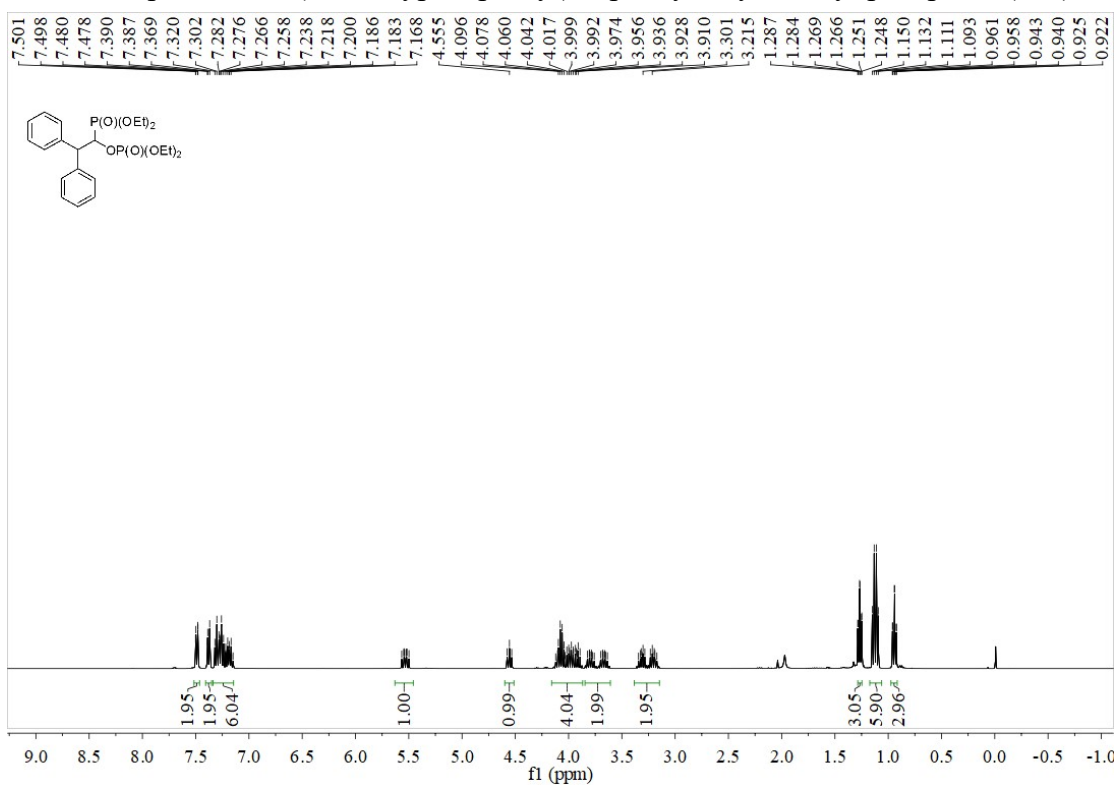
¹H NMR spectra of 1-(diethoxyphosphoryl)-4-phenylbutyl diethyl phosphate (**29c**).



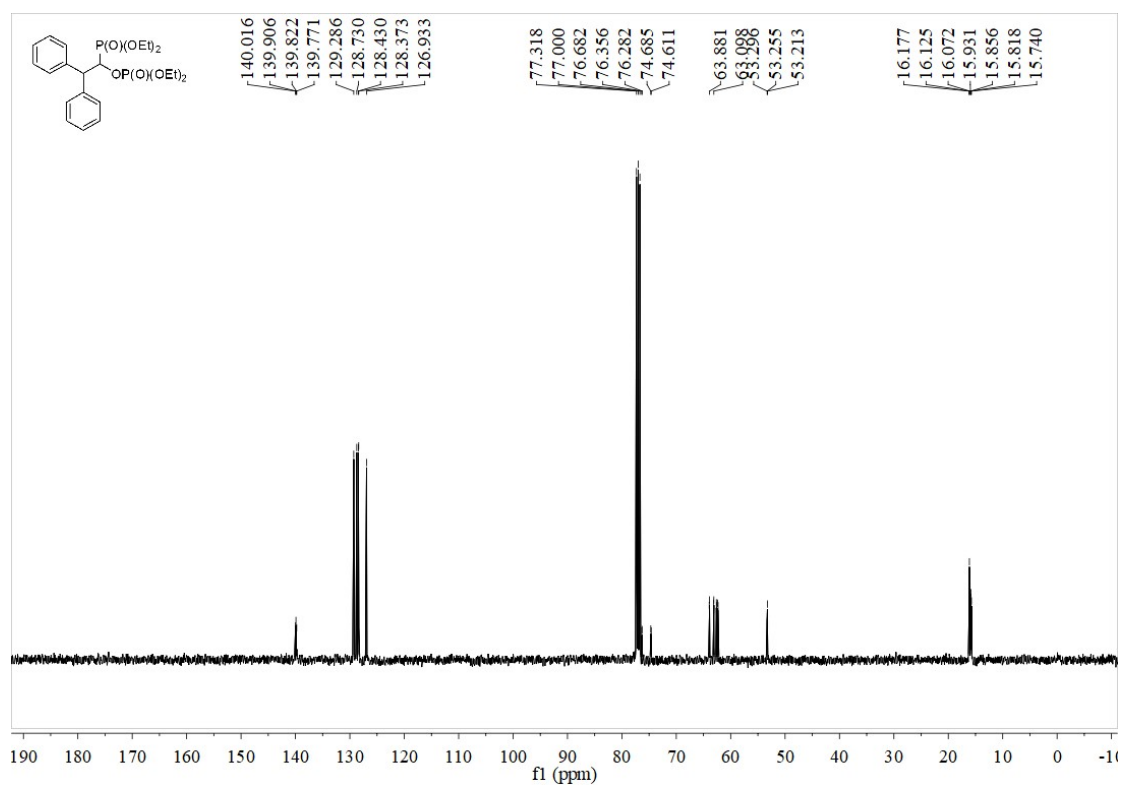
¹³C NMR spectra of 1-(diethoxyphosphoryl)-4-phenylbutyl diethyl phosphate (**29c**).



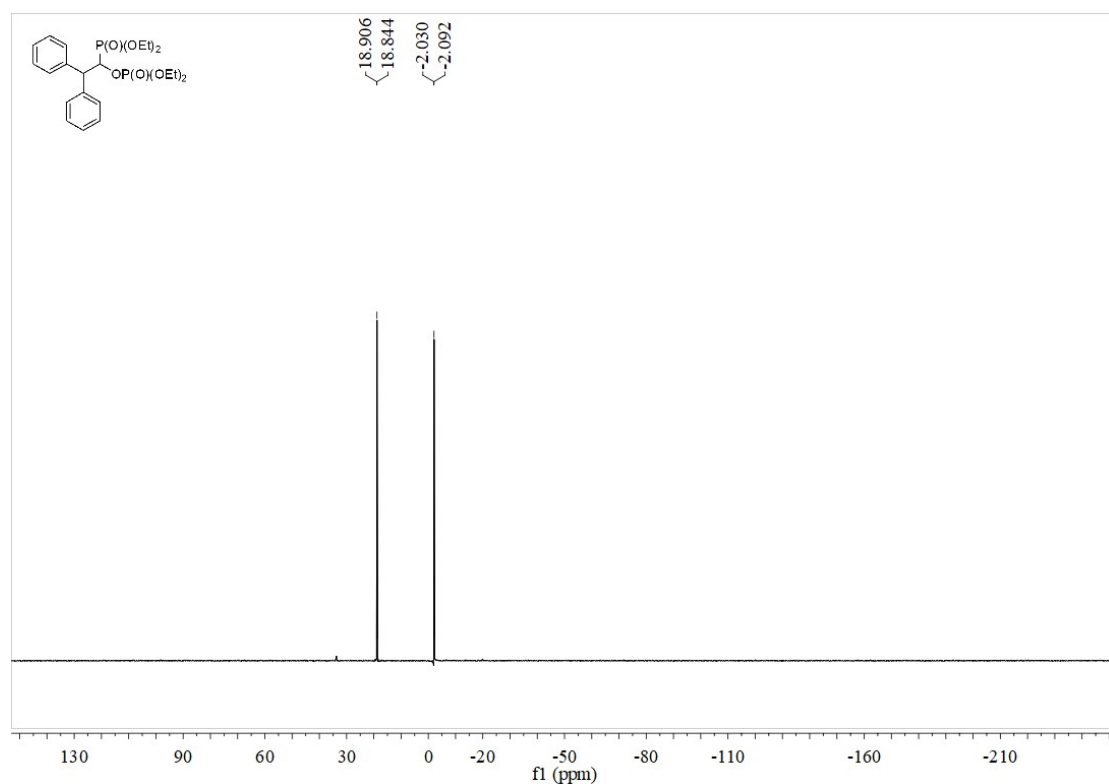
^{31}P NMR spectra of 1-(diethoxyphosphoryl)-4-phenylbutyl diethyl phosphate (**29c**).



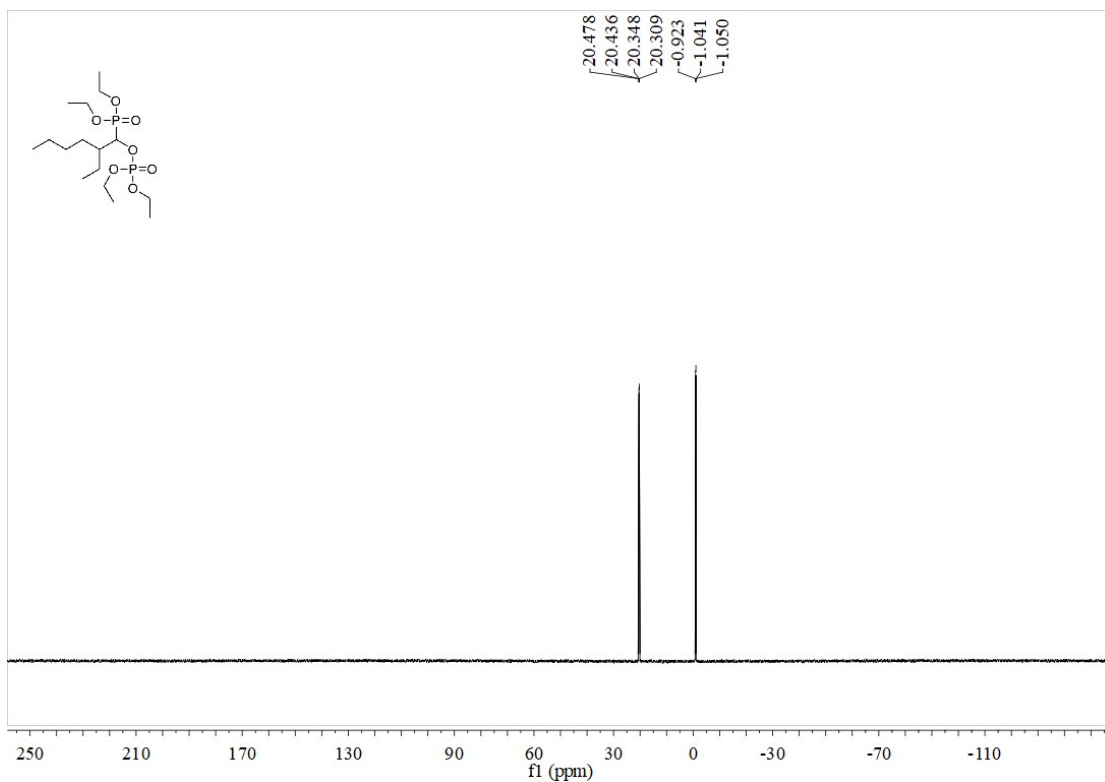
^1H NMR spectra of 1-(diethoxyphosphoryl)-2,2-diphenylethyl diethyl phosphate (**30c**).



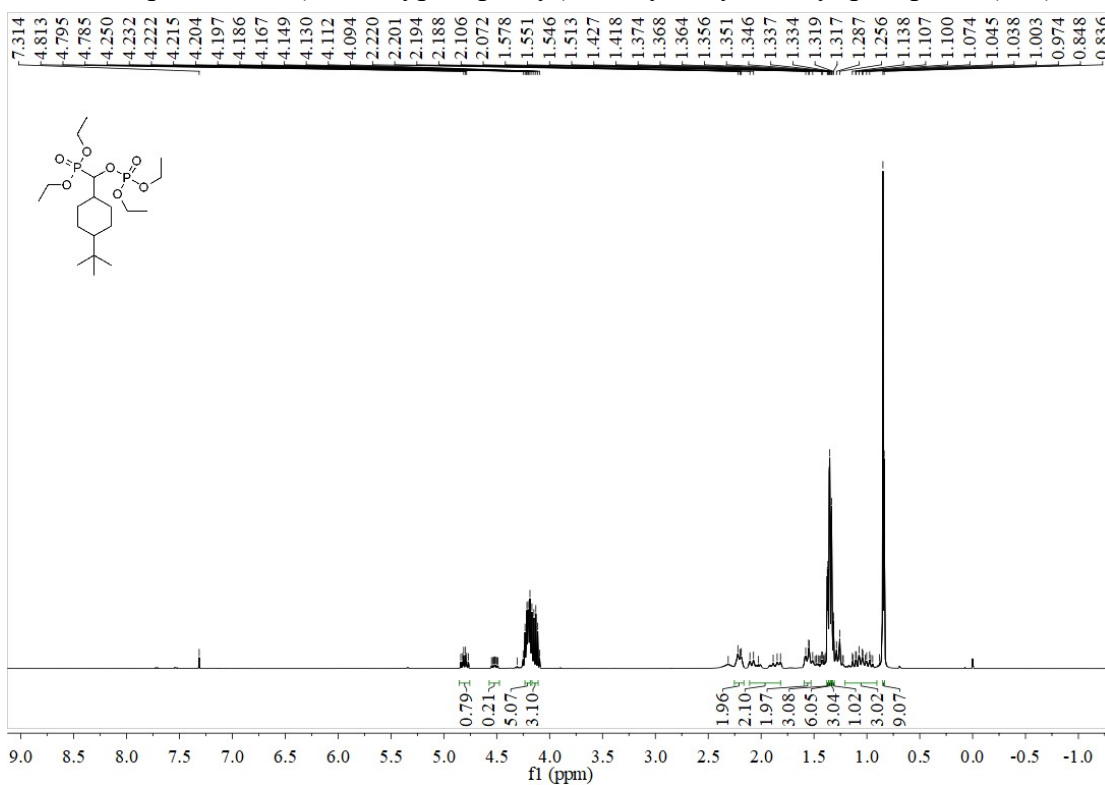
¹³C NMR spectra of 1-(diethoxyphosphoryl)-2,2-diphenylethyl diethyl phosphate (30c).



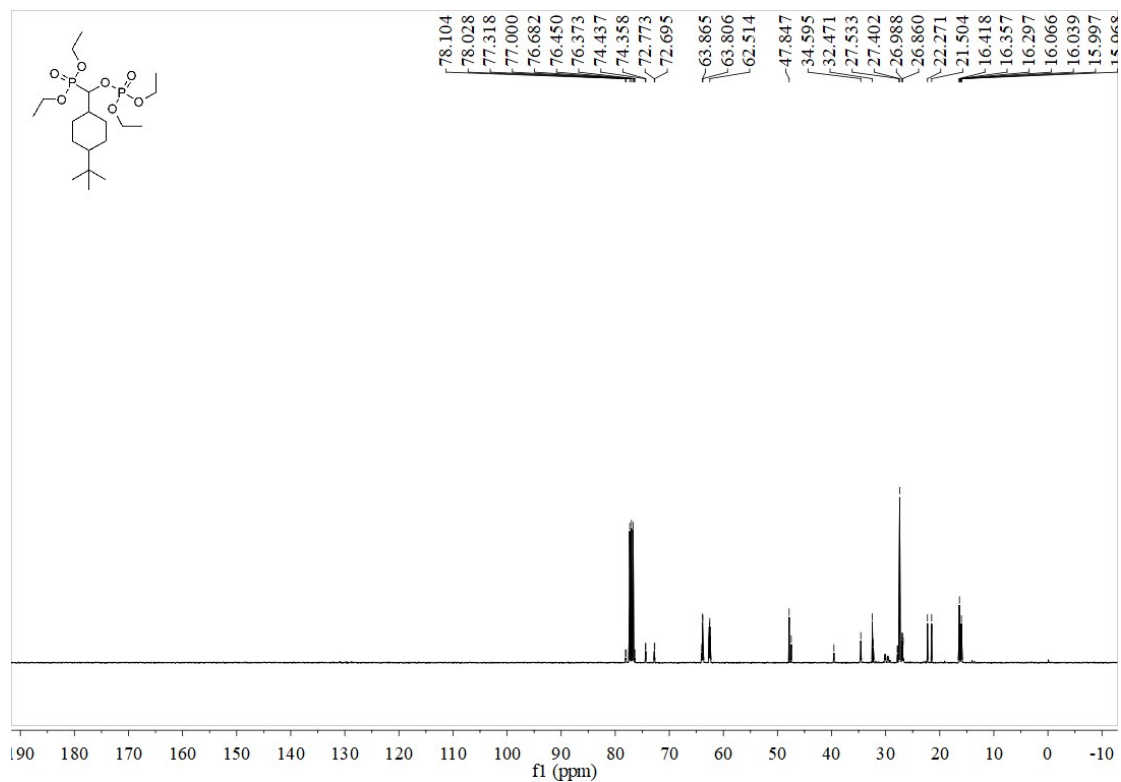
³¹P NMR spectra of 1-(diethoxyphosphoryl)-2,2-diphenylethyl diethyl phosphate (30c).



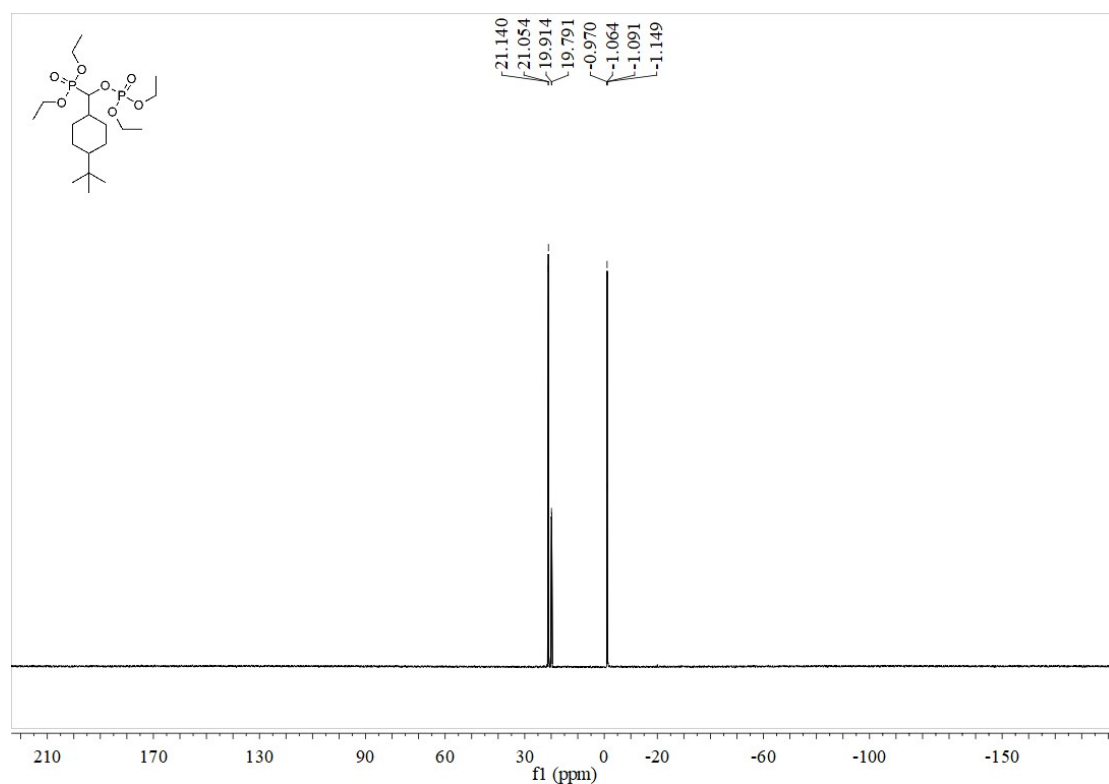
³¹P NMR spectra of 1-(diethoxyphosphoryl)-2-ethylhexyl diethyl phosphate (**31c**).



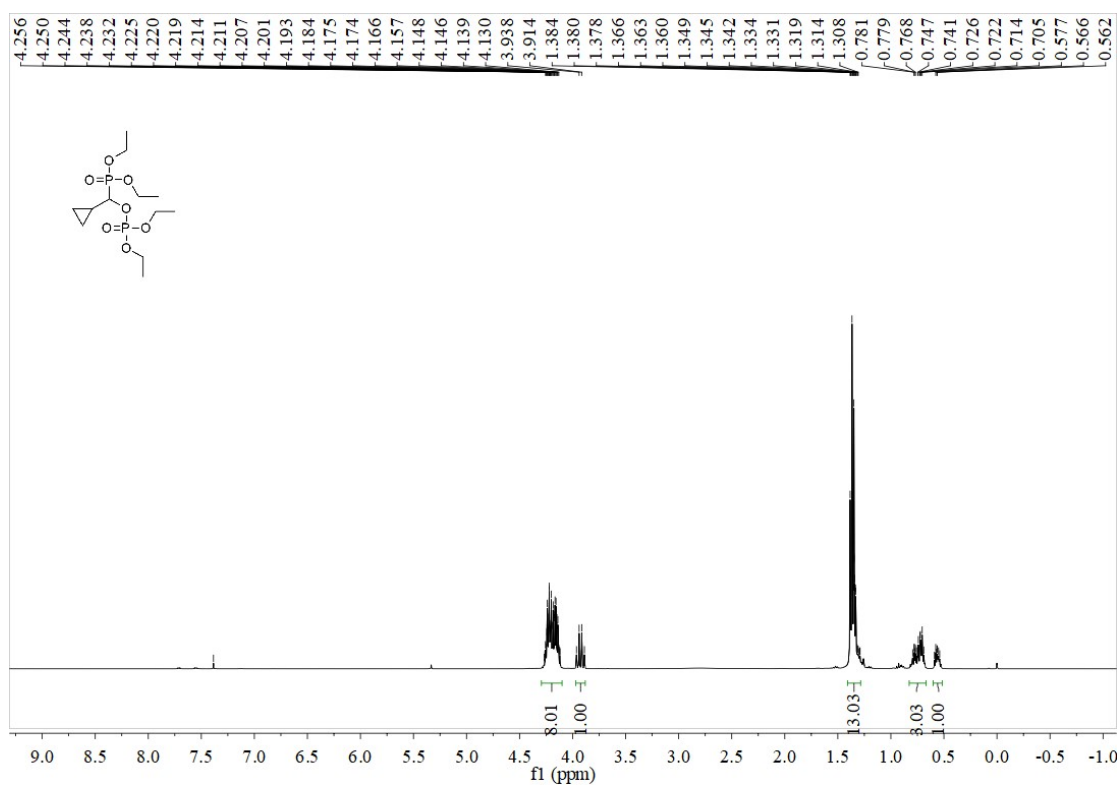
¹H NMR spectra of (4-(tert-butyl)cyclohexyl)(diethoxyphosphoryl)methyl diethyl phosphate (**32c**).



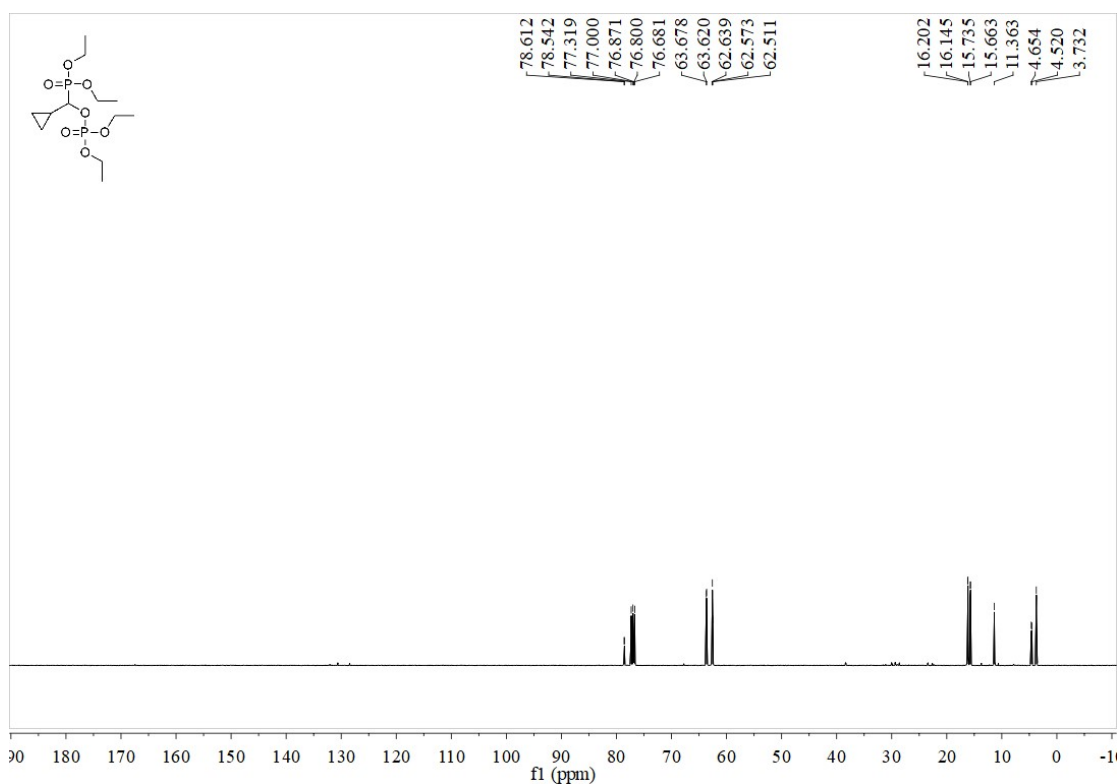
¹³C NMR spectra of (4-(tert-butyl)cyclohexyl)(diethoxyphosphoryl)methyl diethyl phosphate (**32c**).



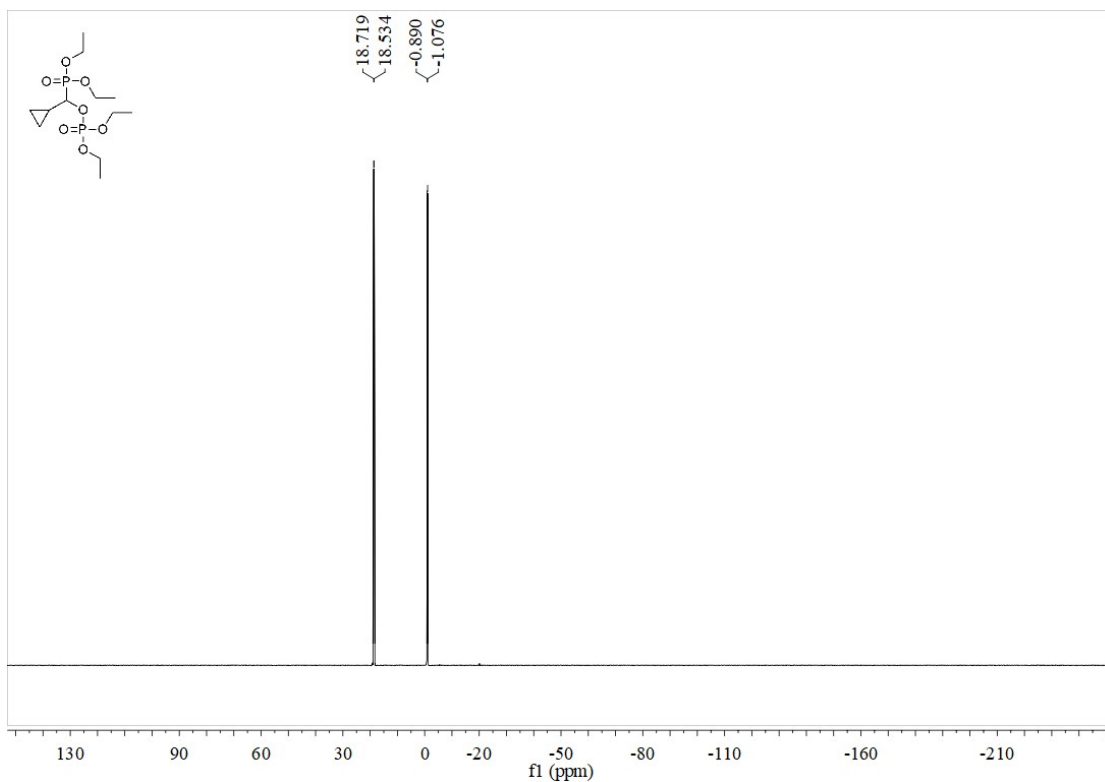
³¹P NMR spectra of (4-(tert-butyl)cyclohexyl)(diethoxyphosphoryl)methyl diethyl phosphate (**32c**).



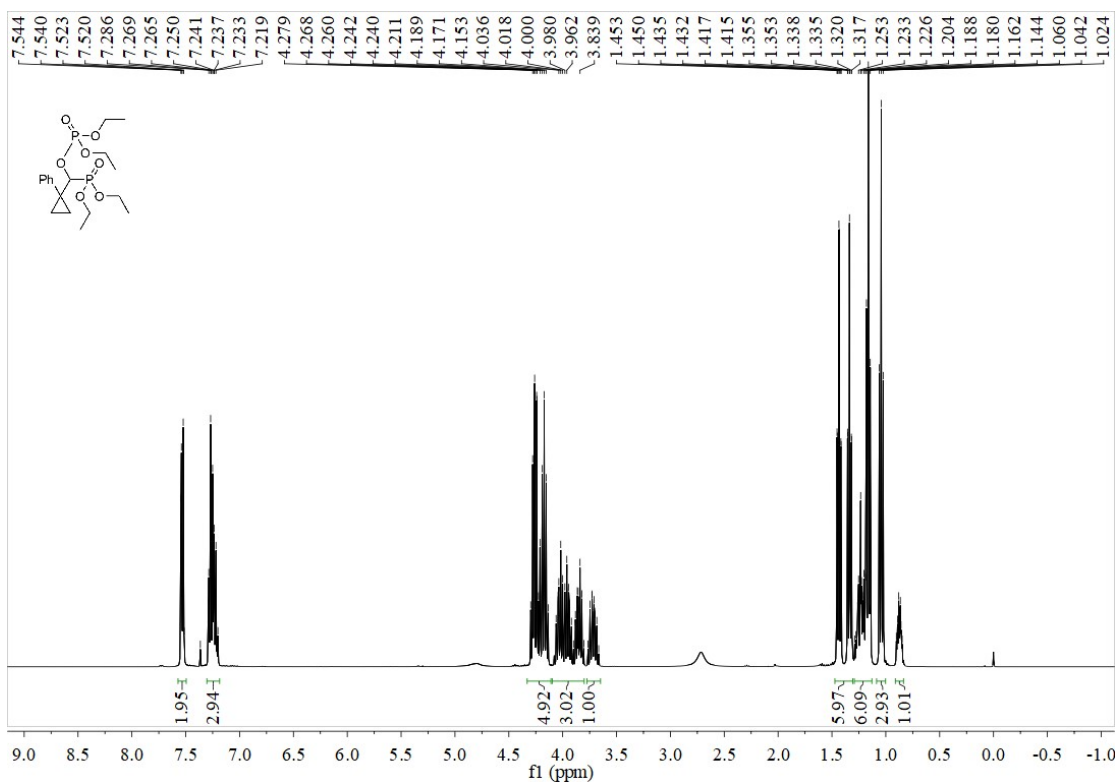
¹H NMR spectra of Cyclopropyl(diethoxyphosphoryl)methyl diethyl phosphate (**33c**).



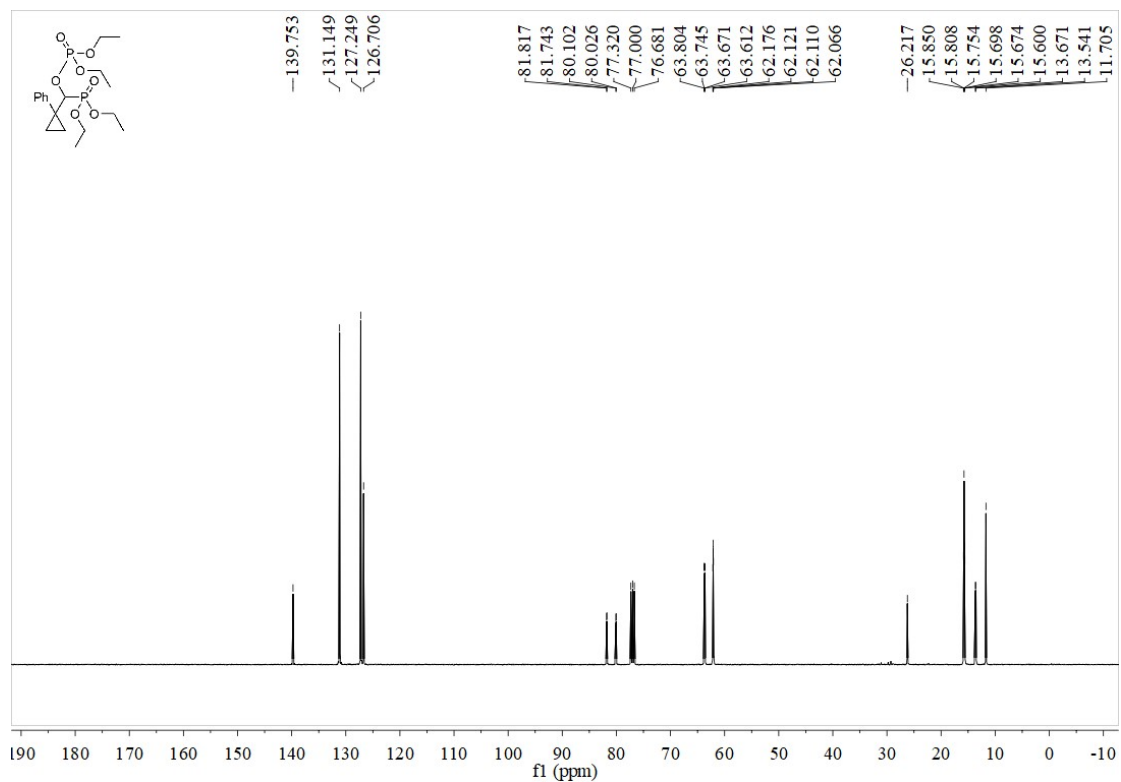
¹³C NMR spectra of Cyclopropyl(diethoxyphosphoryl)methyl diethyl phosphate (**33c**).



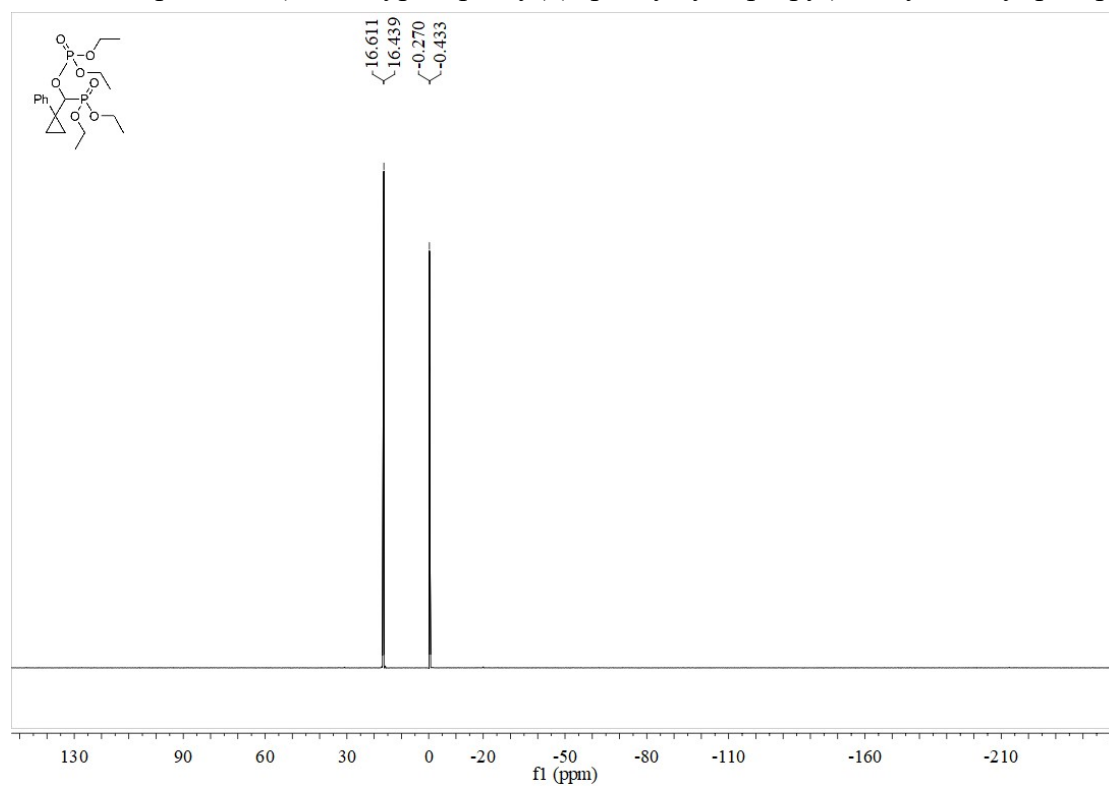
^{31}P NMR spectra of Cyclopropyl(diethoxyphosphoryl)methyl diethyl phosphate (**33c**).



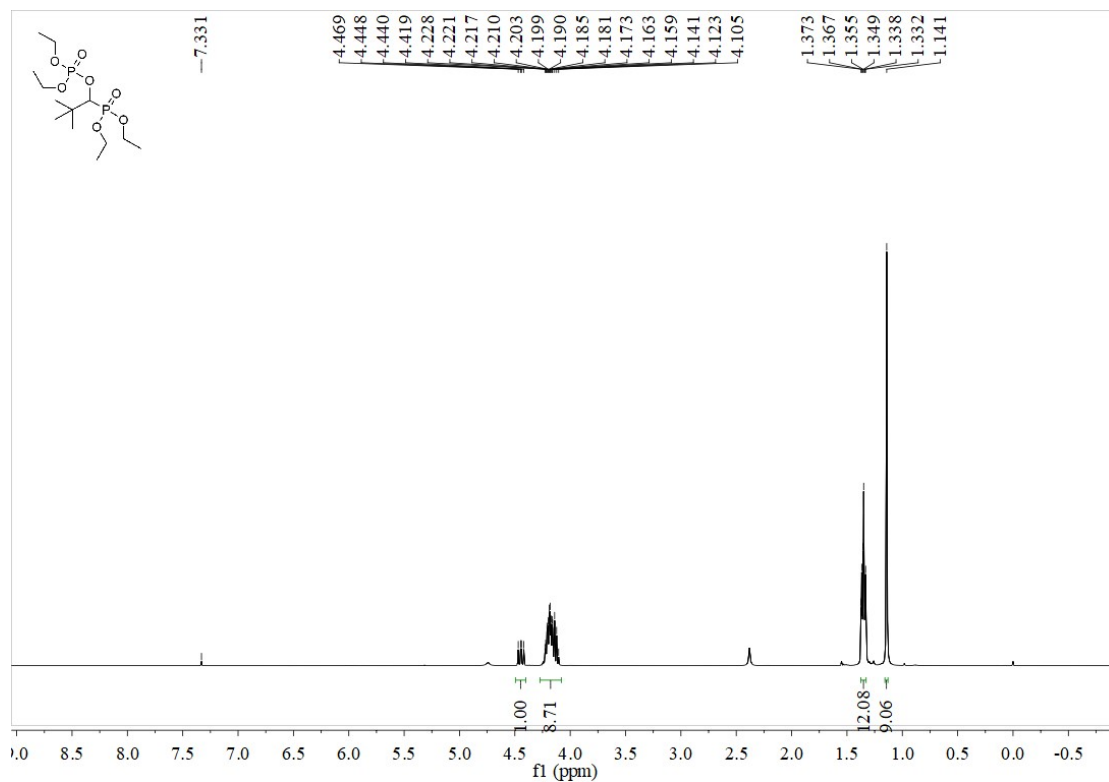
^1H NMR spectra of (diethoxyphosphoryl)(1-phenylcyclopropyl)methyl diethyl phosphate (**34c**).



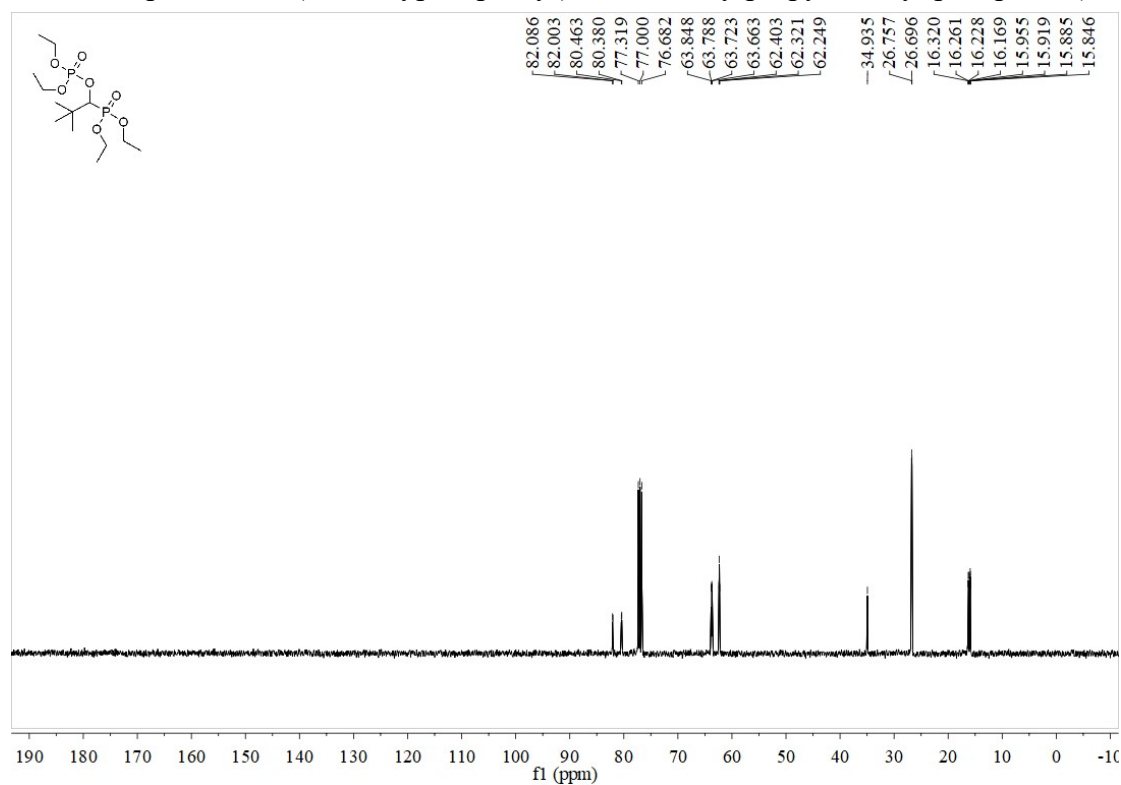
¹³C NMR spectra of (diethoxyphosphoryl)(1-phenylcyclopropyl)methyl diethyl phosphate (**34c**).



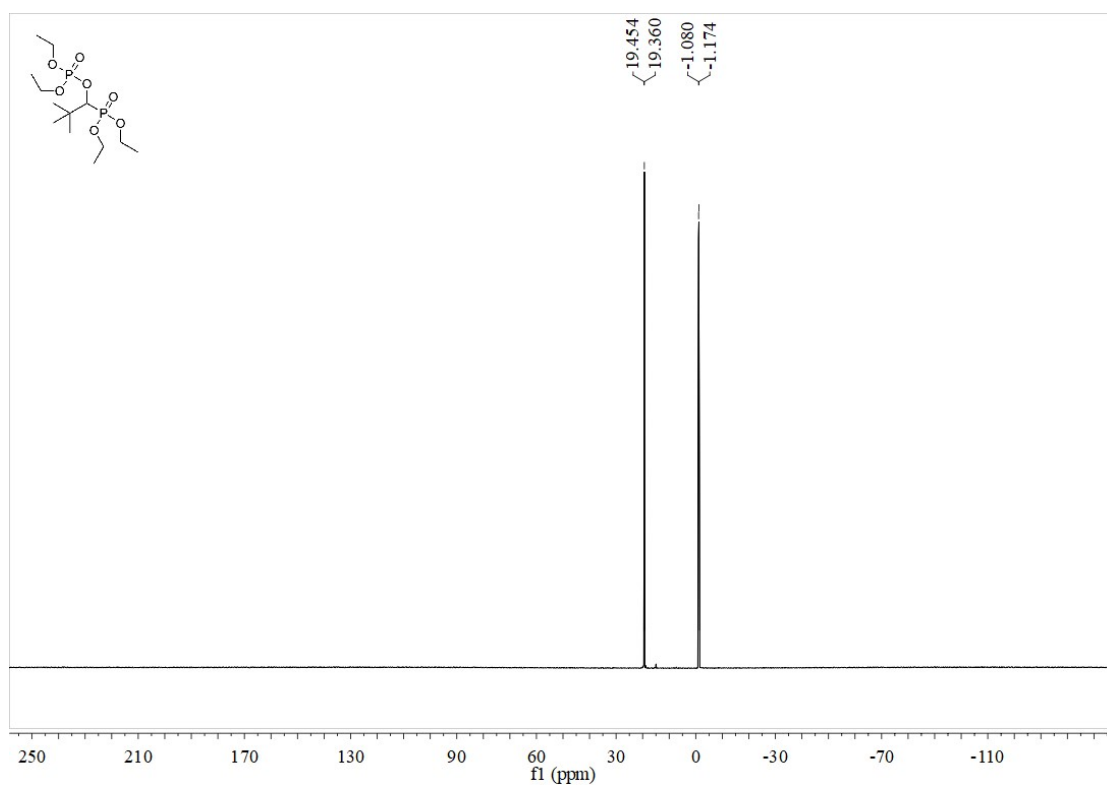
³¹P NMR spectra of (diethoxyphosphoryl)(1-phenylcyclopropyl)methyl diethyl phosphate (**34c**).



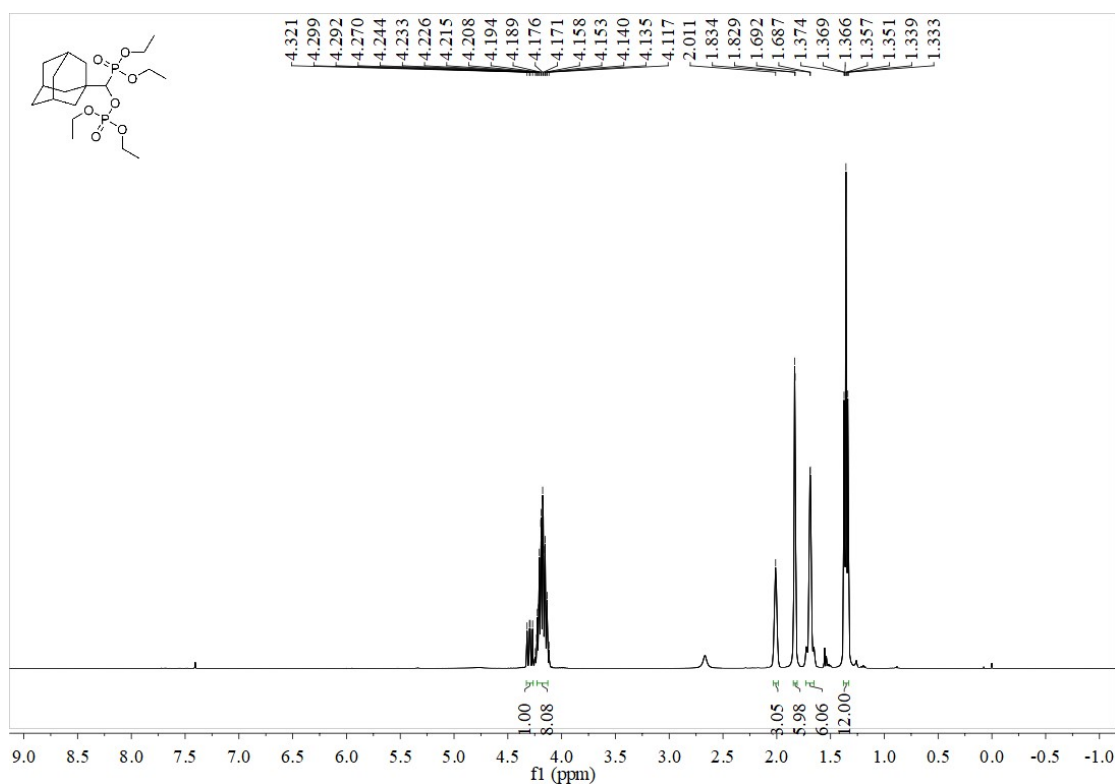
¹H NMR spectra of 1-(diethoxyphosphoryl)-2,2-dimethylpropyl diethyl phosphate (**35c**).



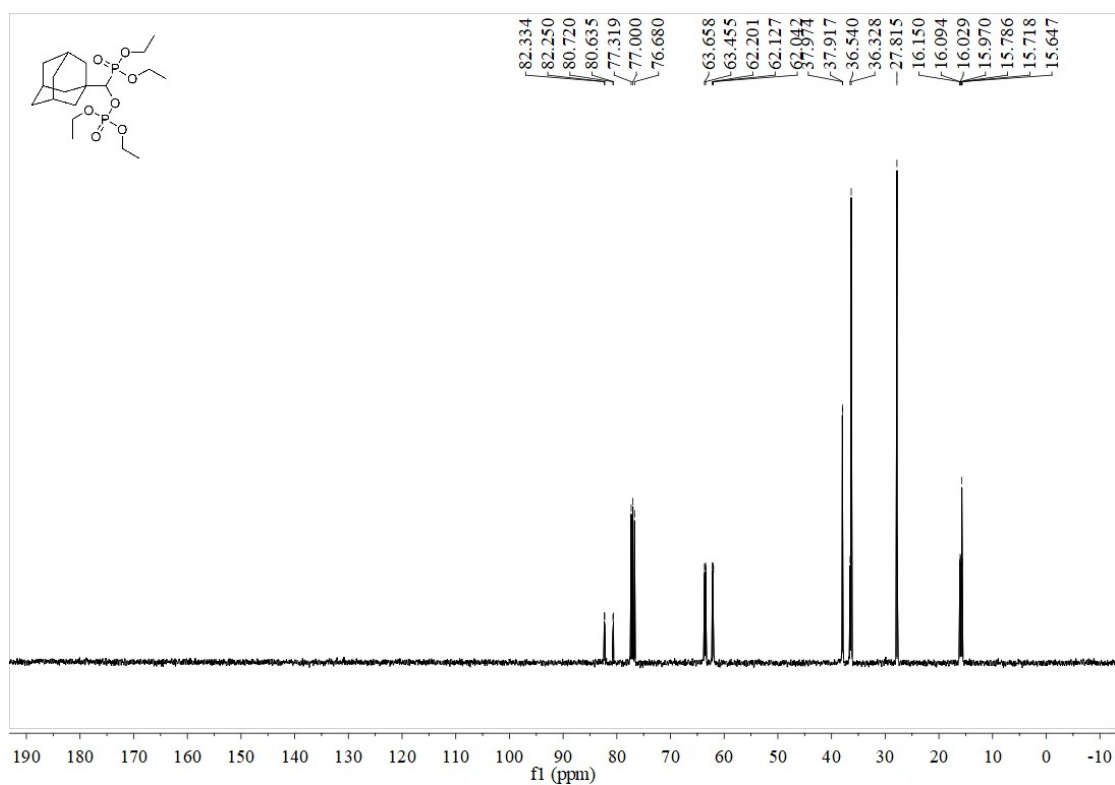
¹³C NMR spectra of 1-(diethoxyphosphoryl)-2,2-dimethylpropyl diethyl phosphate (**35c**).



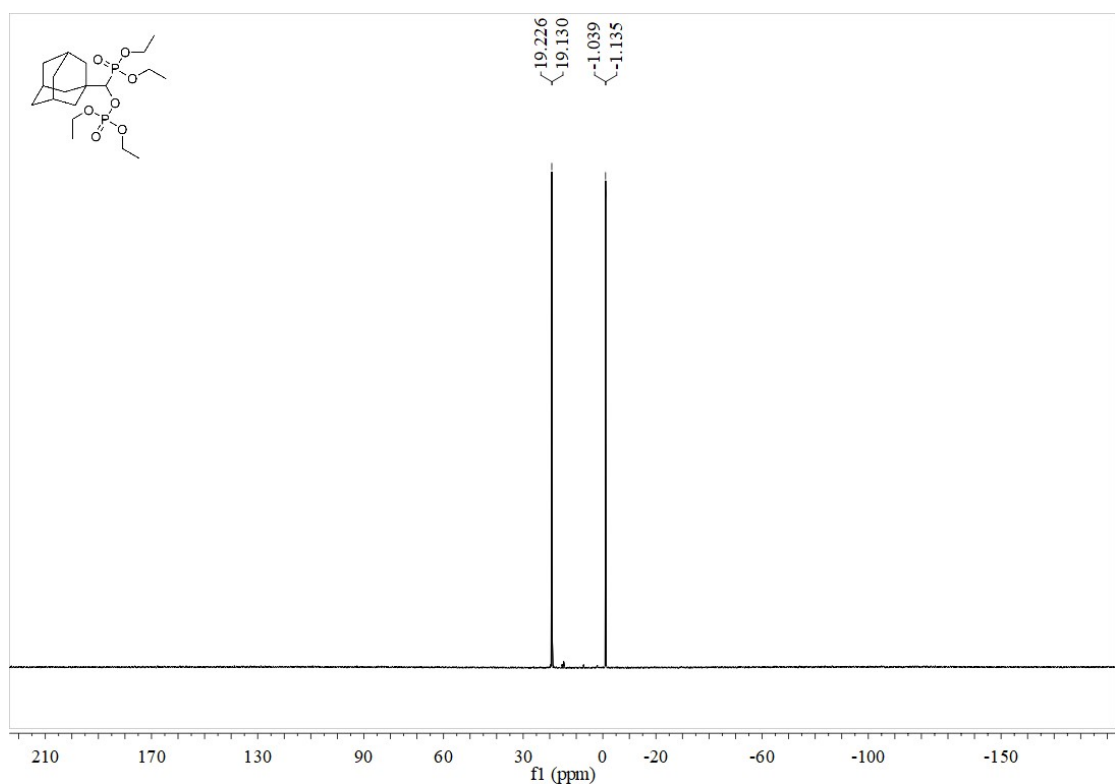
^{31}P NMR spectra of 1-(diethoxyphosphoryl)-2,2-dimethylpropyl diethyl phosphate (**35c**).



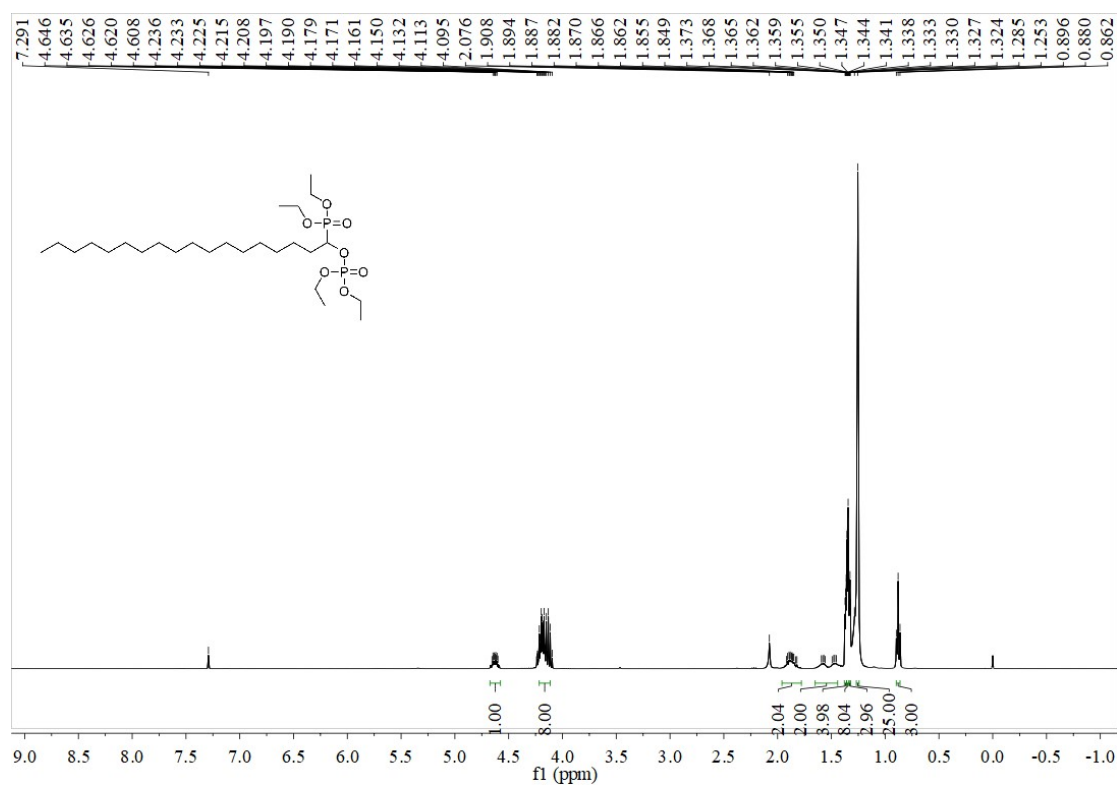
^1H NMR spectra of Adamantan-1-yl(diethoxyphosphoryl)methyl diethyl phosphate (**36c**).



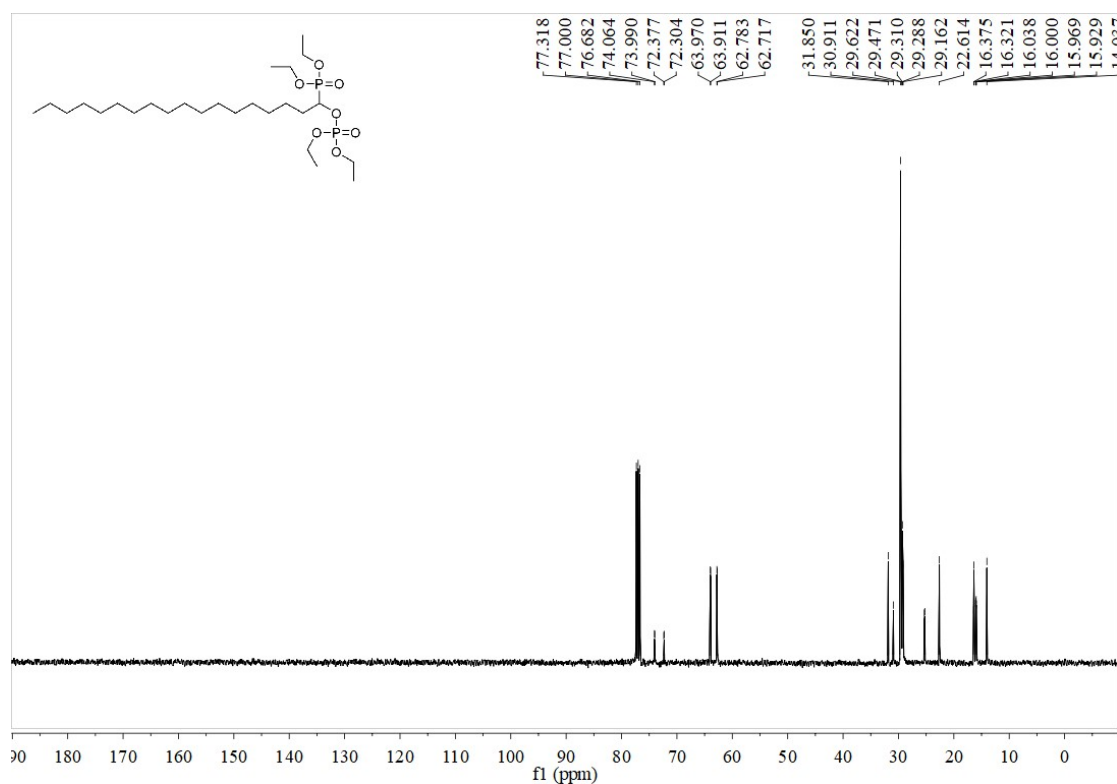
¹³C NMR spectra of Adamantan-1-yl(diethoxyphosphoryl)methyl diethyl phosphate (**36c**).



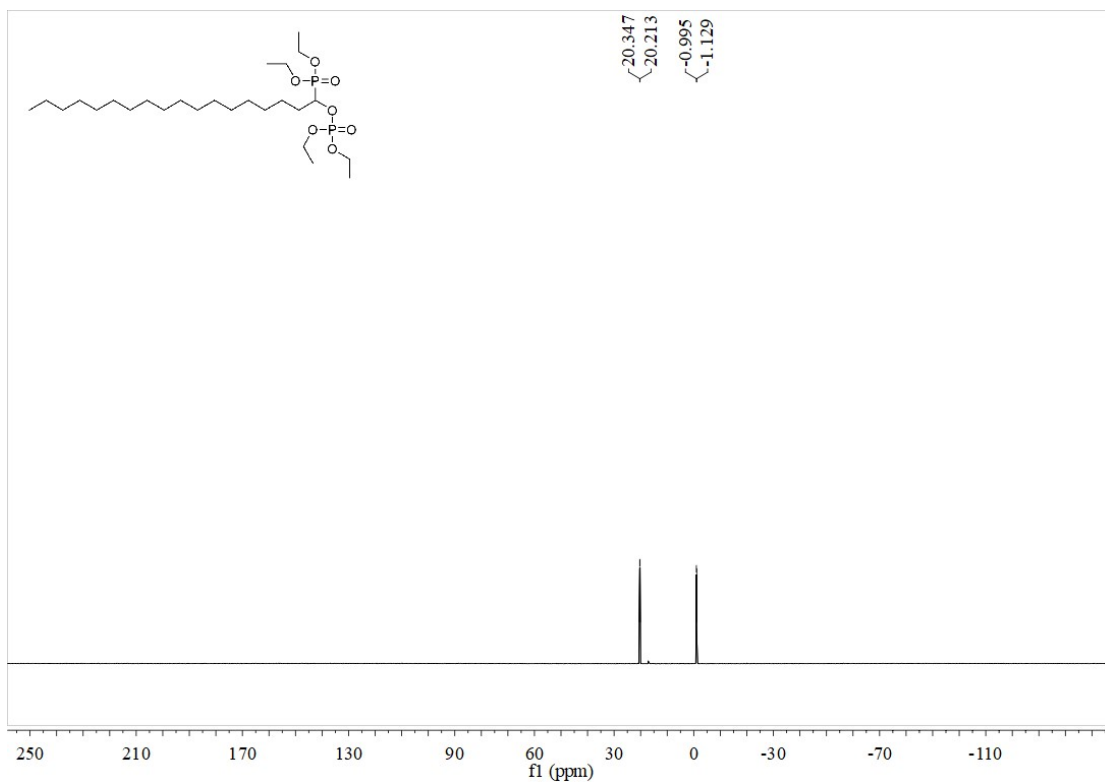
³¹P NMR spectra of Adamantan-1-yl(diethoxyphosphoryl)methyl diethyl phosphate (**36c**).



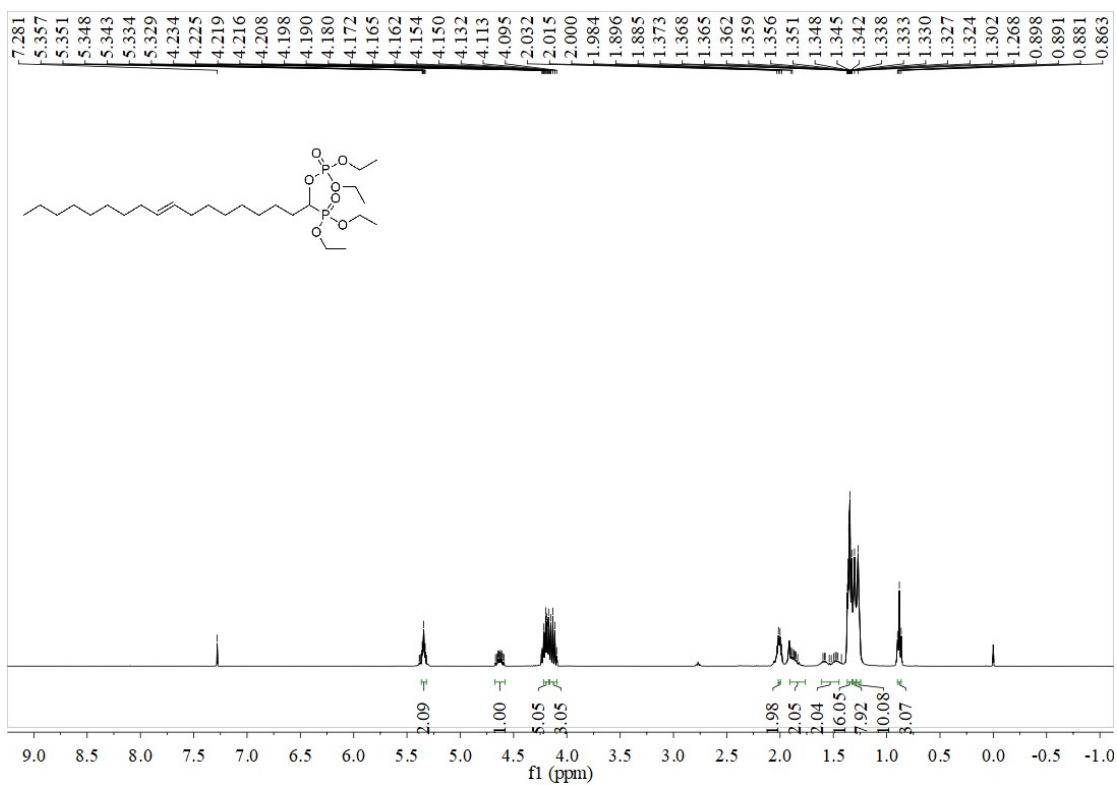
¹H NMR spectra of 1-(diethoxyphosphoryl)octadecyl diethyl phosphate (**37c**).



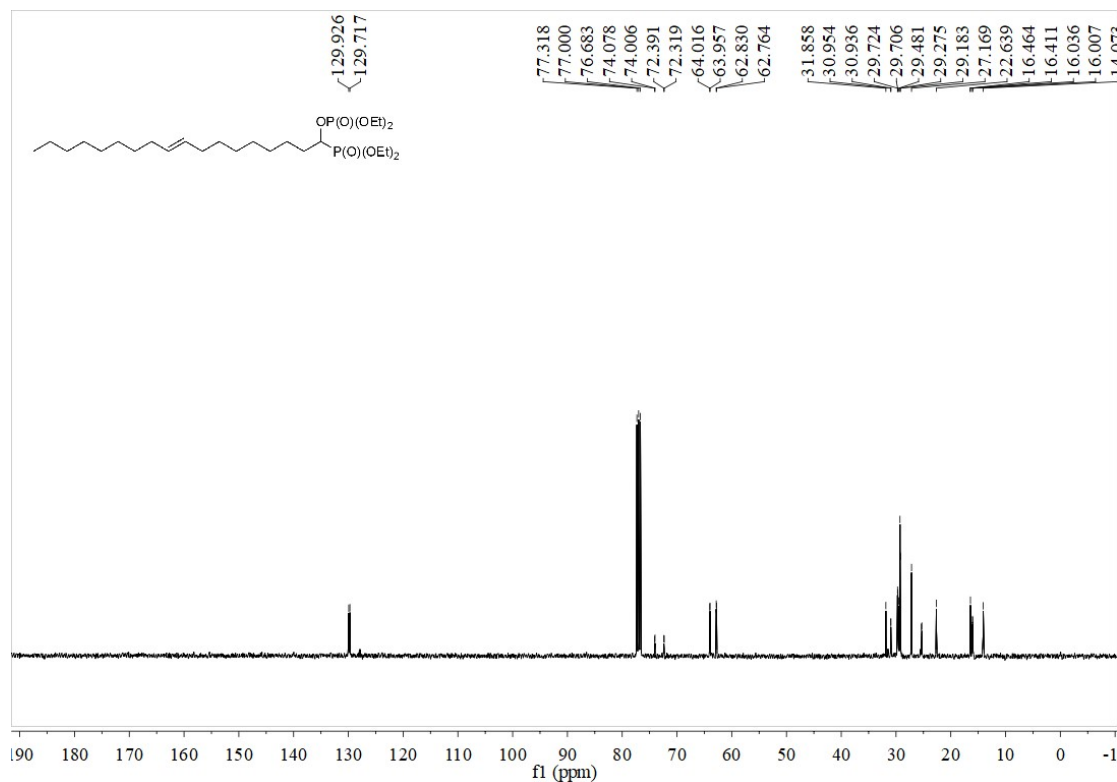
¹³C NMR spectra of 1-(diethoxyphosphoryl)octadecyl diethyl phosphate (**37c**).



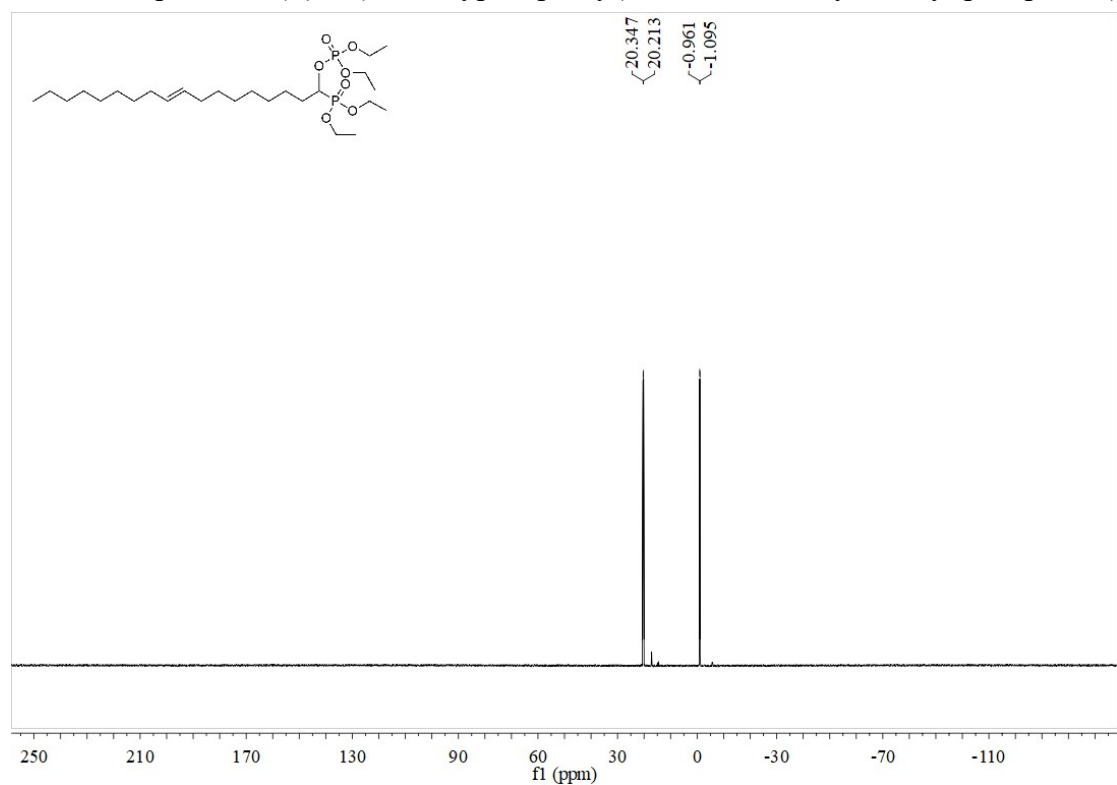
³¹P NMR spectra of 1-(diethoxyphosphoryl)octadecyl diethyl phosphate (**37c**).



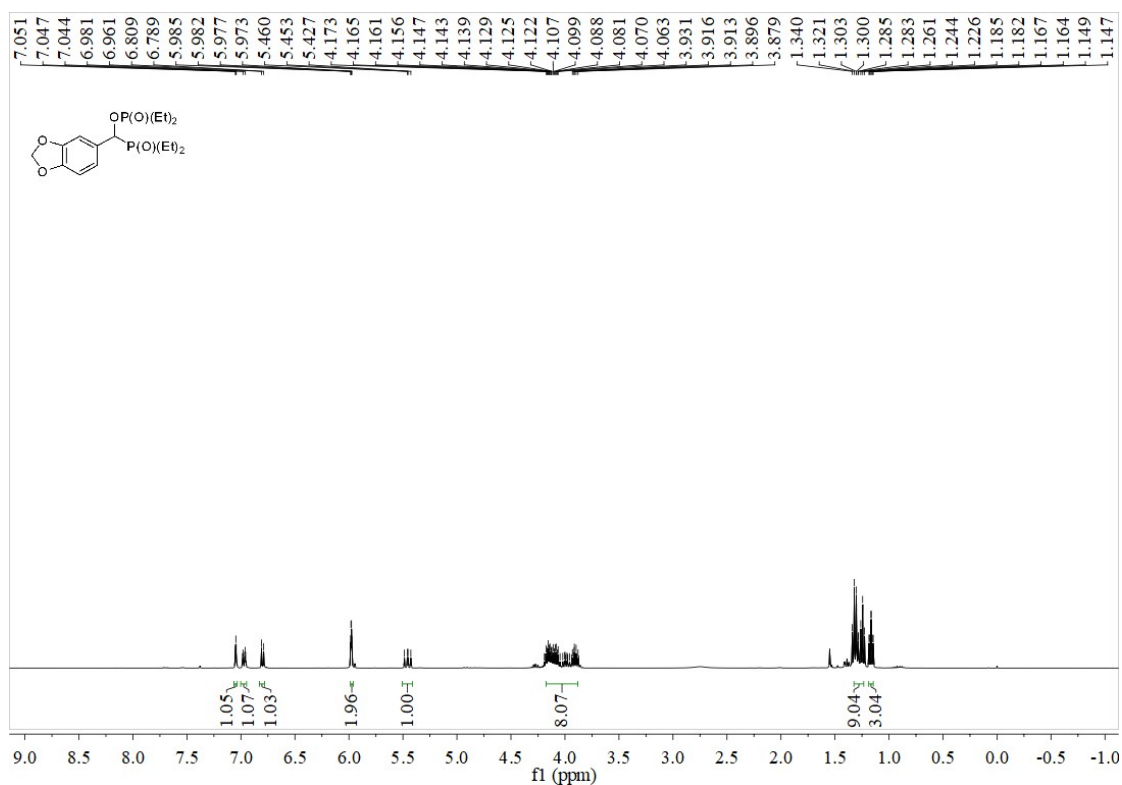
¹H NMR spectra of (E)-1-(diethoxyphosphoryl)octadec-9-en-1-yl diethyl phosphate (**38c**).



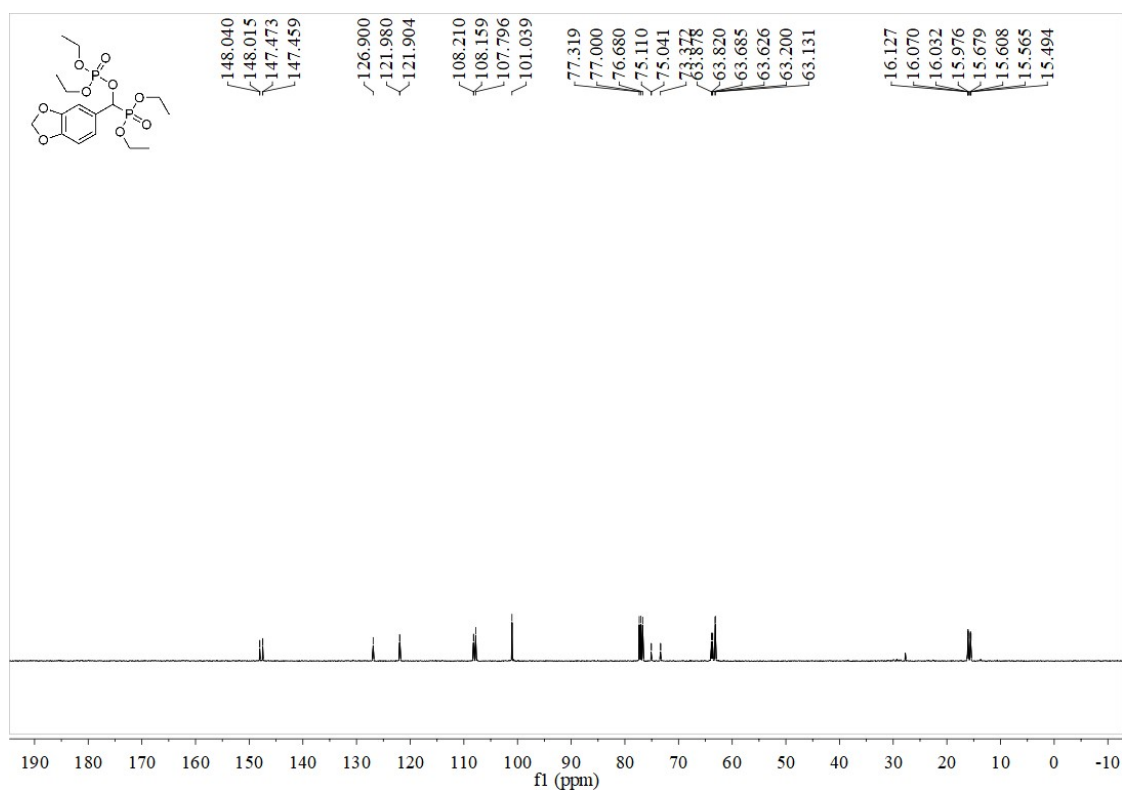
¹³C NMR spectra of (E)-1-(diethoxyphosphoryl)octadec-9-en-1-yl diethyl phosphate (**38c**).



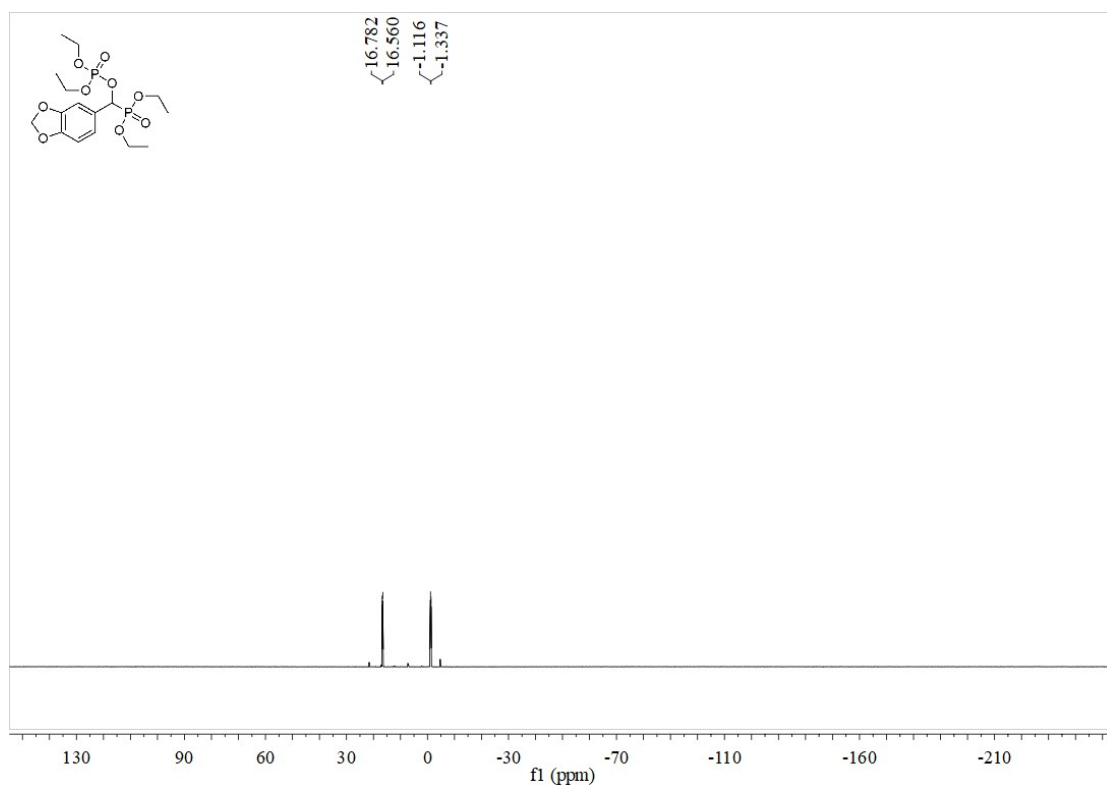
³¹P NMR spectra of (E)-1-(diethoxyphosphoryl)octadec-9-en-1-yl diethyl phosphate (**38c**).



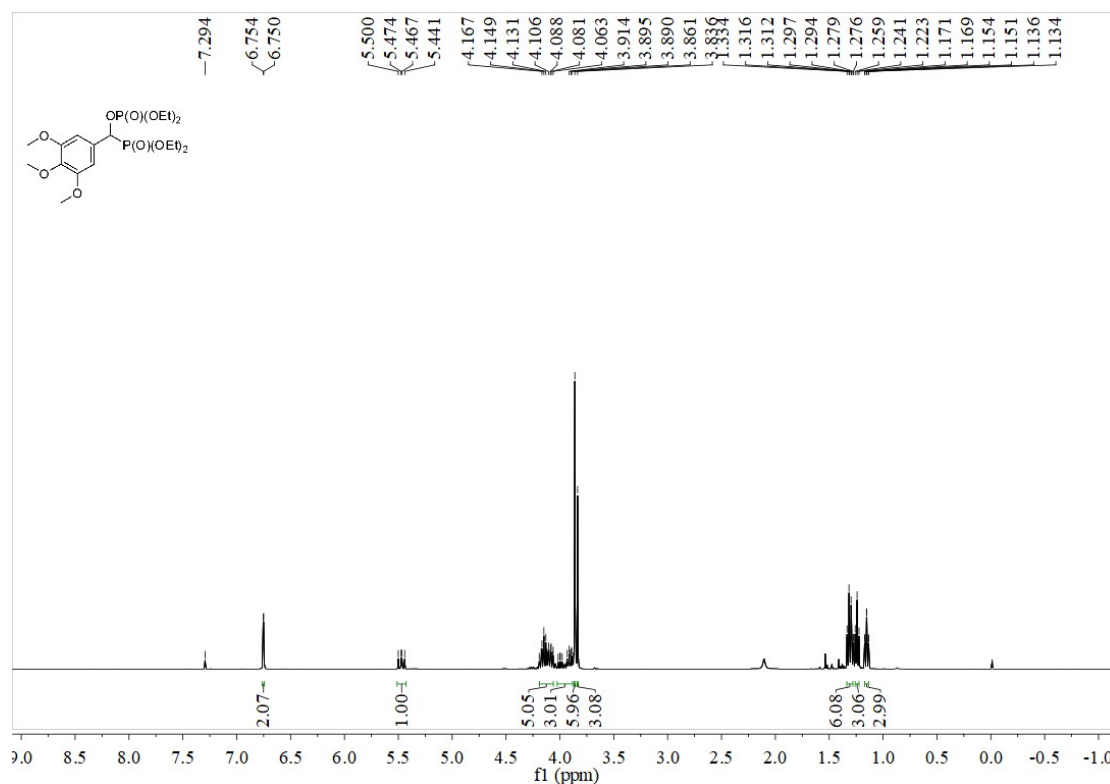
¹H NMR spectra of Benzo[d][1,3]dioxol-5-yl(diethylphosphoryl)methyl diethylphosphinate (**39c**).



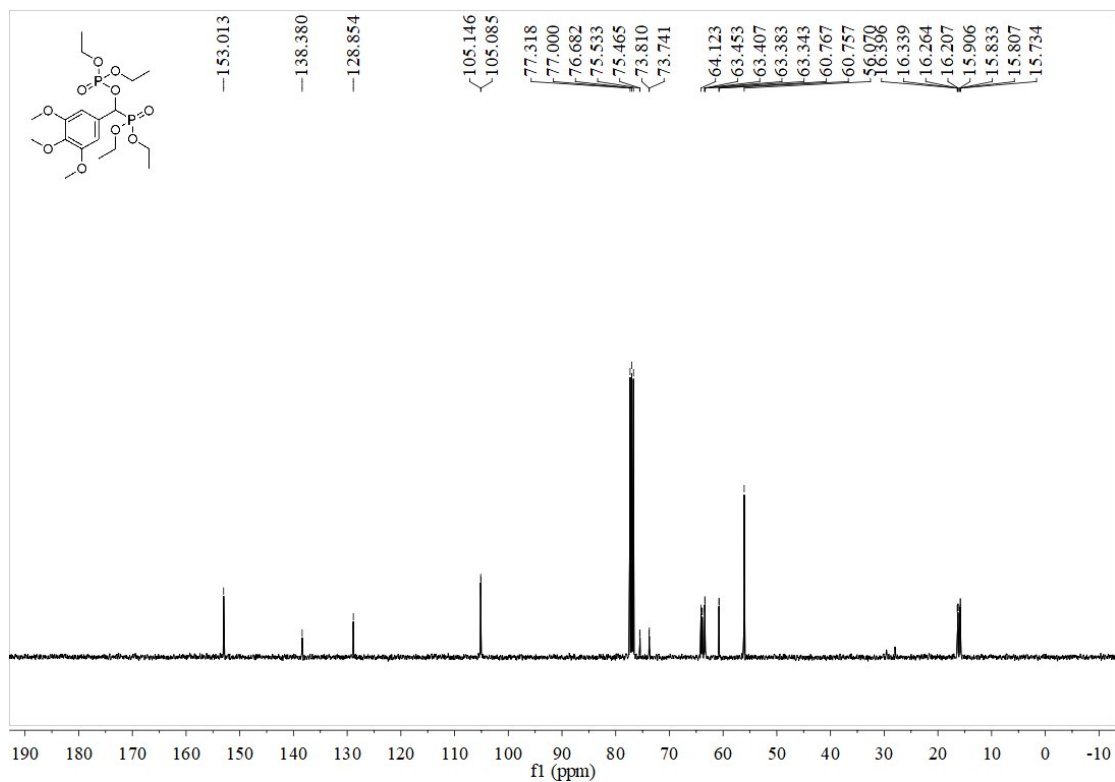
¹³C NMR spectra of Benzo[d][1,3]dioxol-5-yl(diethylphosphoryl)methyl diethylphosphinate (**39c**).



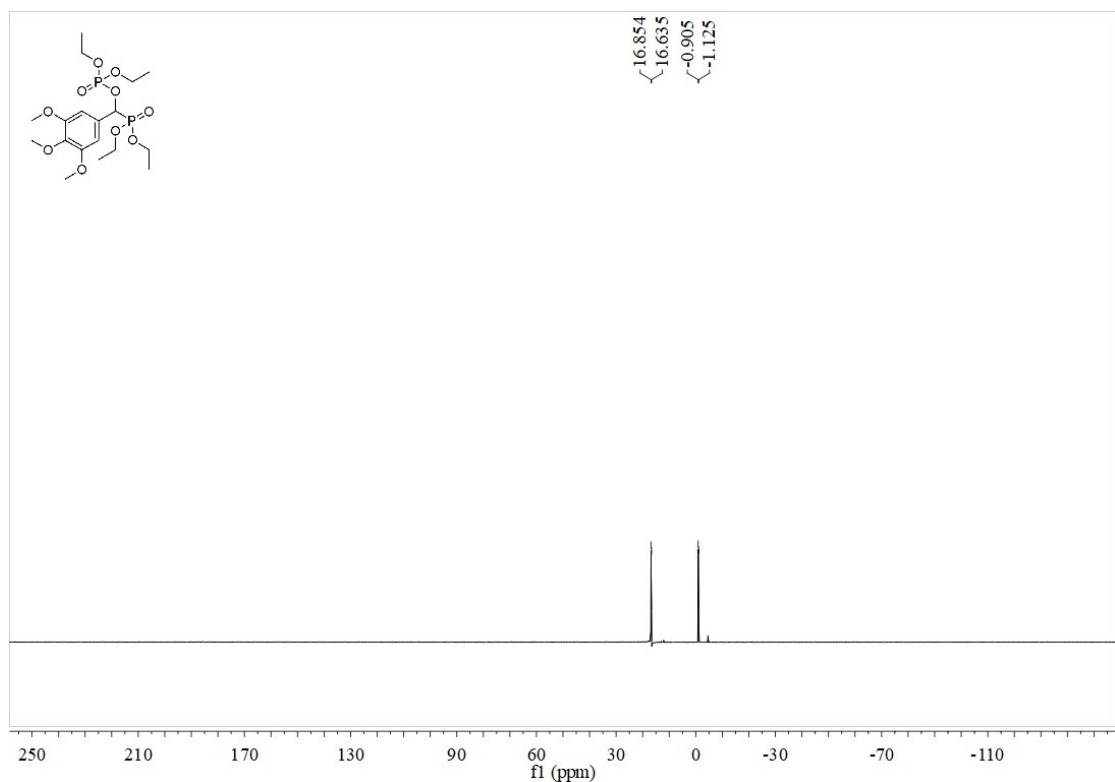
^{31}P NMR spectra of Benzo[d][1,3]dioxol-5-yl(diethylphosphoryl)methyl diethylphosphinate (**39c**).



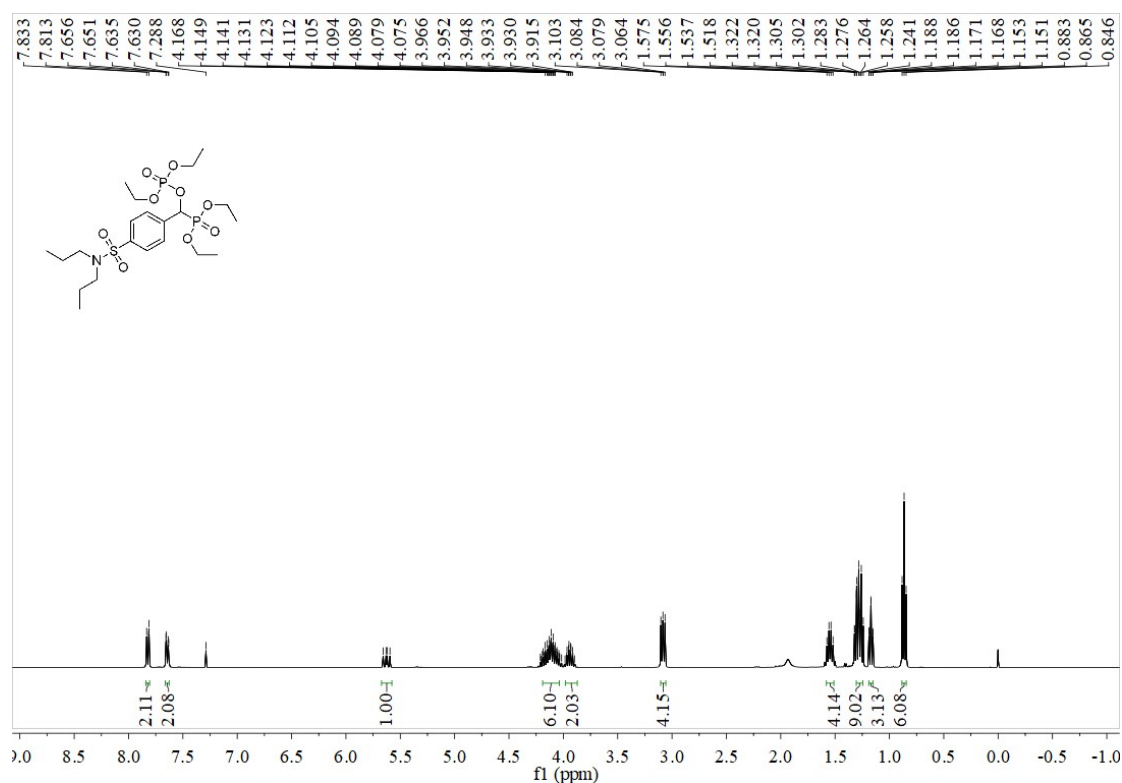
^1H NMR spectra of (diethoxyphosphoryl)(3,4,5-trimethoxyphenyl)methyl diethyl phosphate (**40c**).



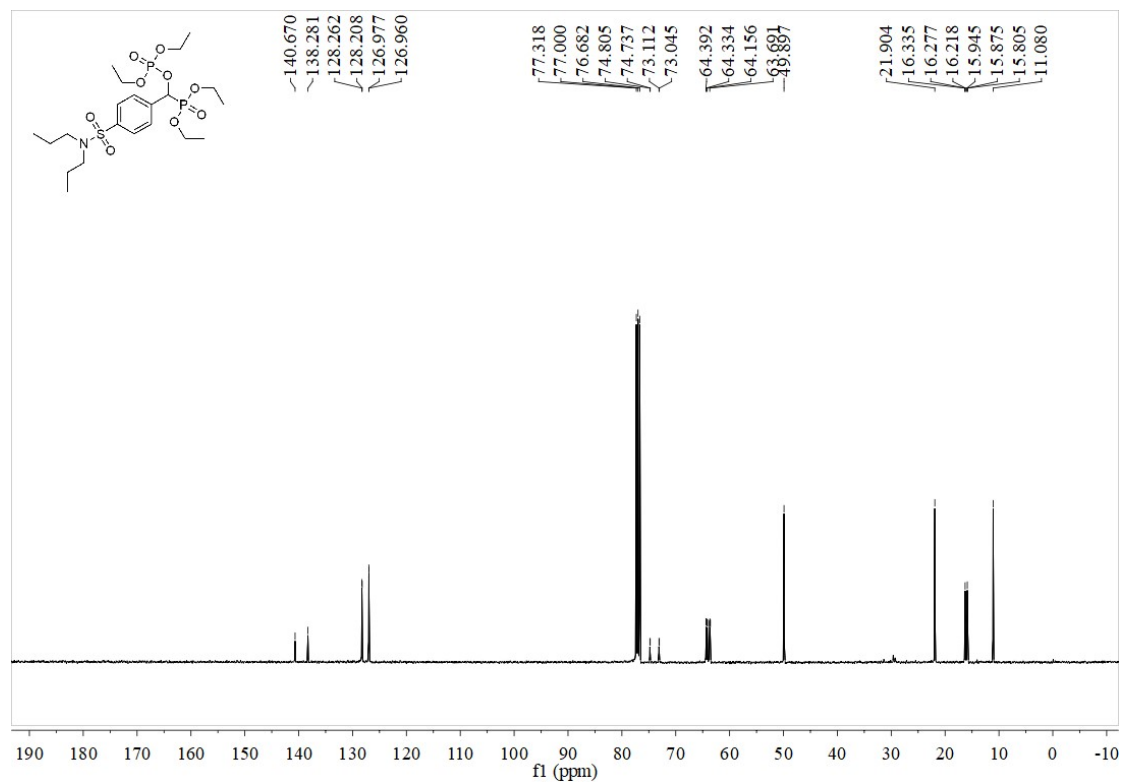
^{13}C NMR spectra of (diethoxyphosphoryl)(3,4,5-trimethoxyphenyl)methyl diethyl phosphate (**40c**).



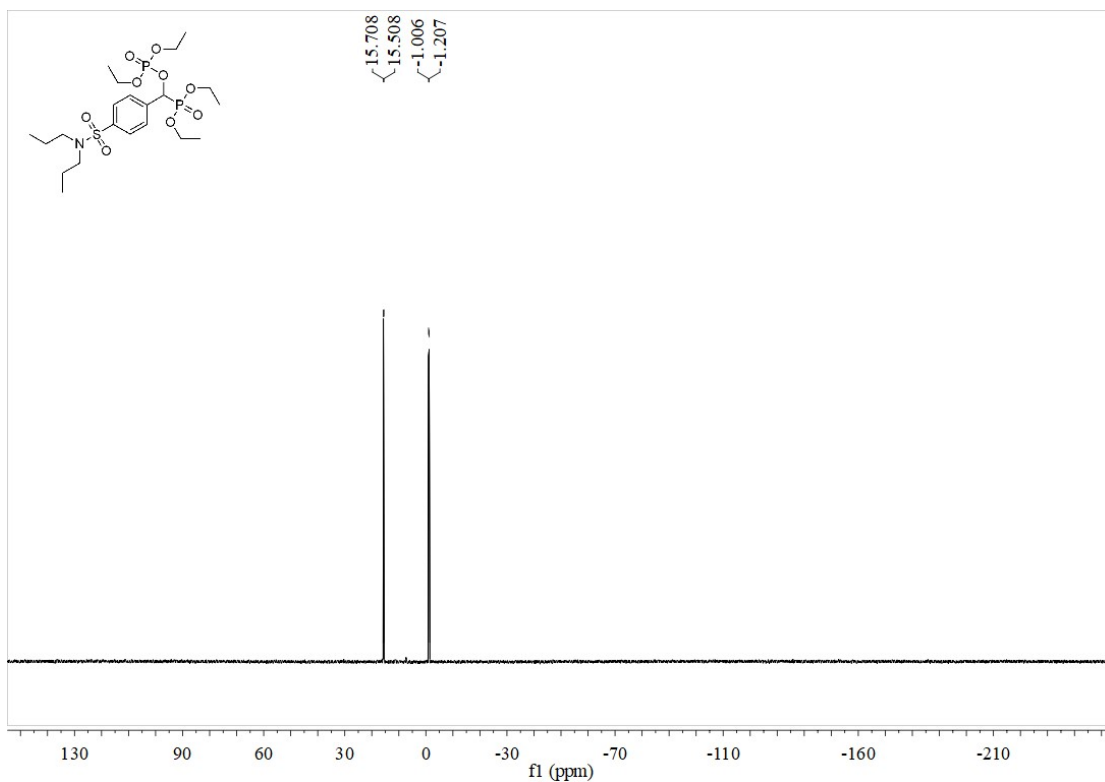
^{31}P NMR spectra of (diethoxyphosphoryl)(3,4,5-trimethoxyphenyl)methyl diethyl phosphate (**40c**).



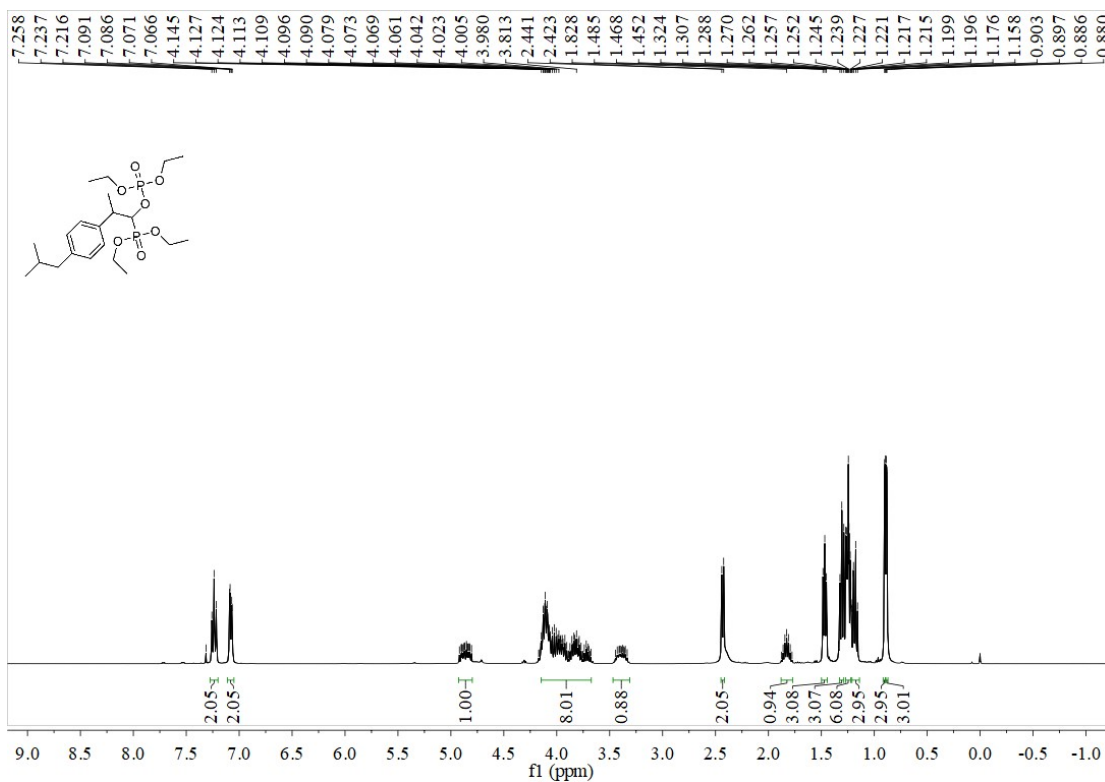
¹H NMR spectra of (diethoxyphosphoryl)(4-(N,N-dipropylsulfamoyl)phenyl)methyl diethyl phosphate (41c).



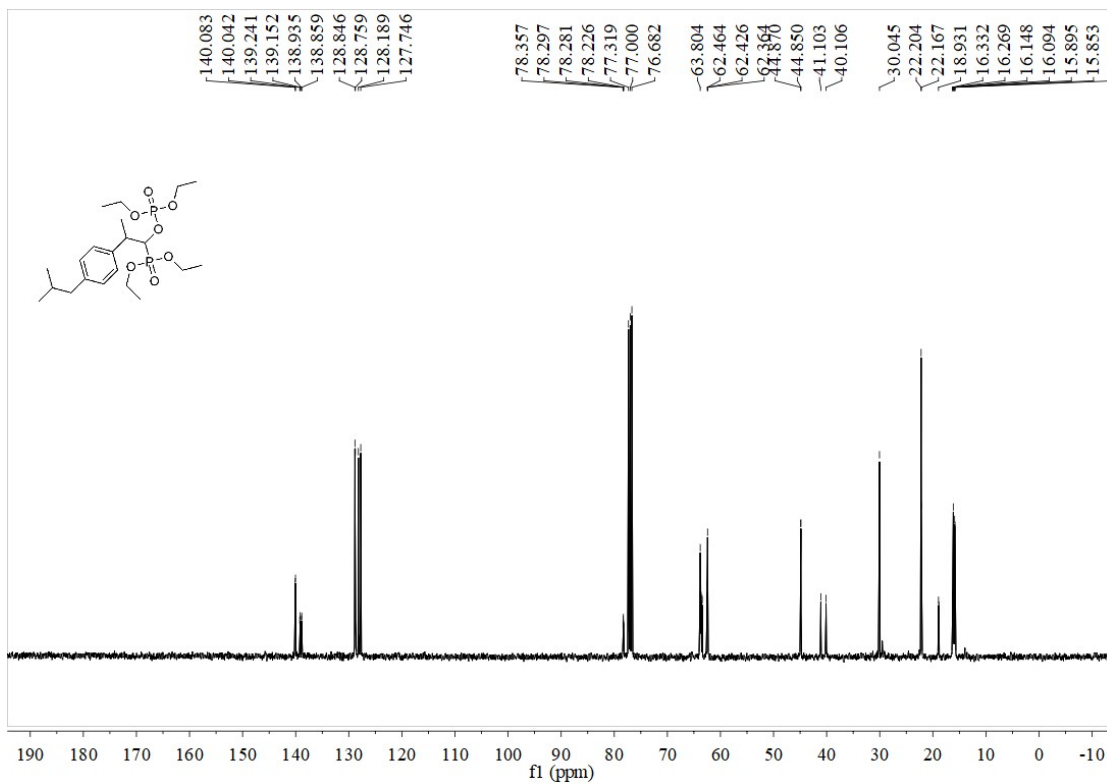
¹³C NMR spectra of (diethoxyphosphoryl)(4-(N,N-dipropylsulfamoyl)phenyl)methyl diethyl phosphate (41c).



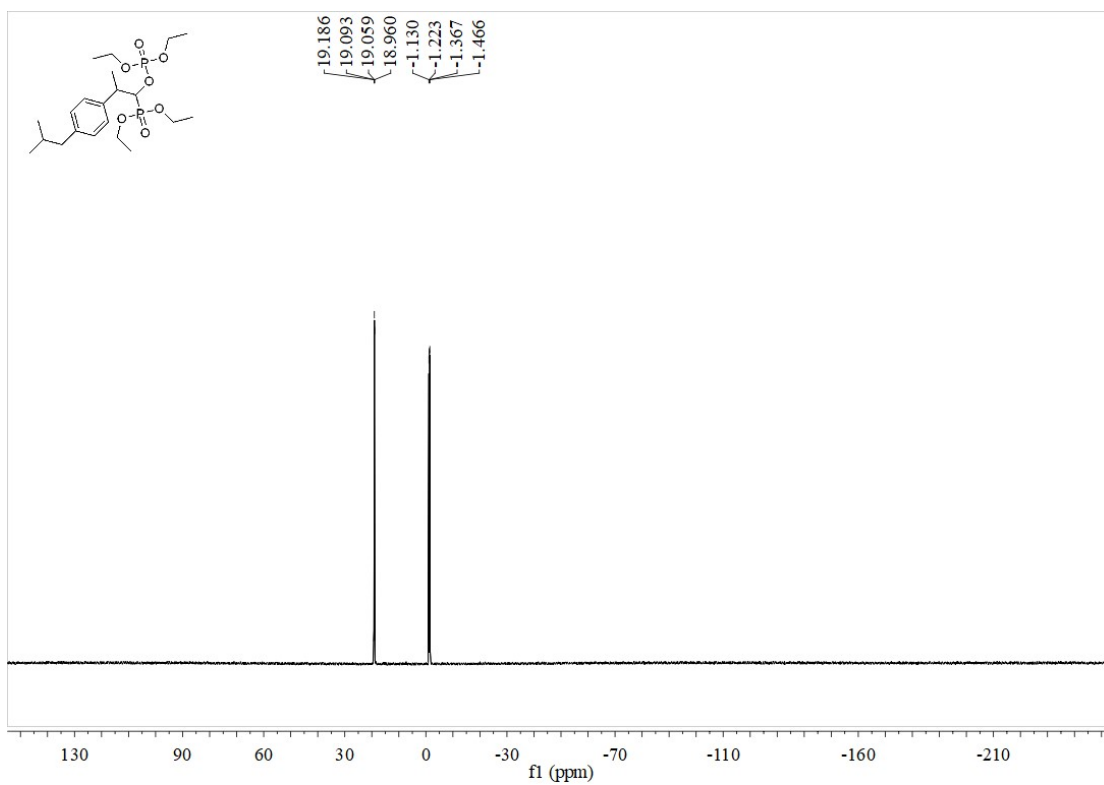
³¹P NMR spectra of (diethoxyphosphoryl)(4-(N,N-dipropylsulfamoyl)phenyl)methyl diethyl phosphate (41c).



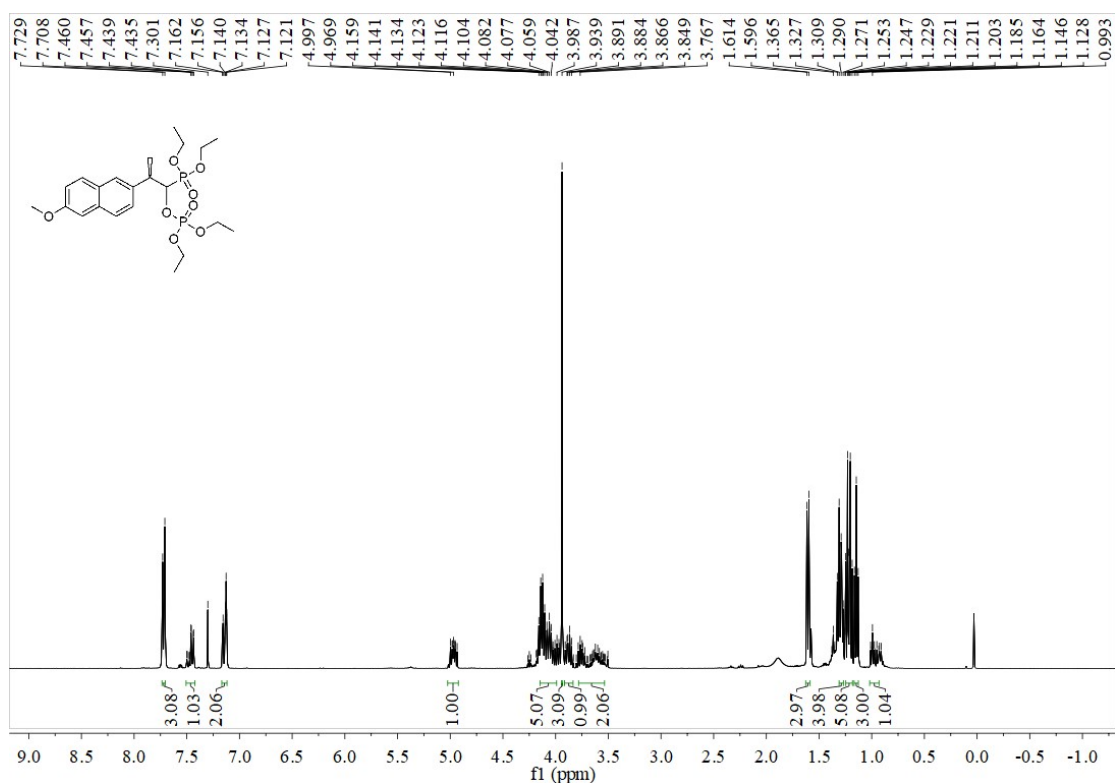
¹H NMR spectra of 1-(diethoxyphosphoryl)-2-(4-isobutylphenyl)propyl diethyl phosphate (42c).



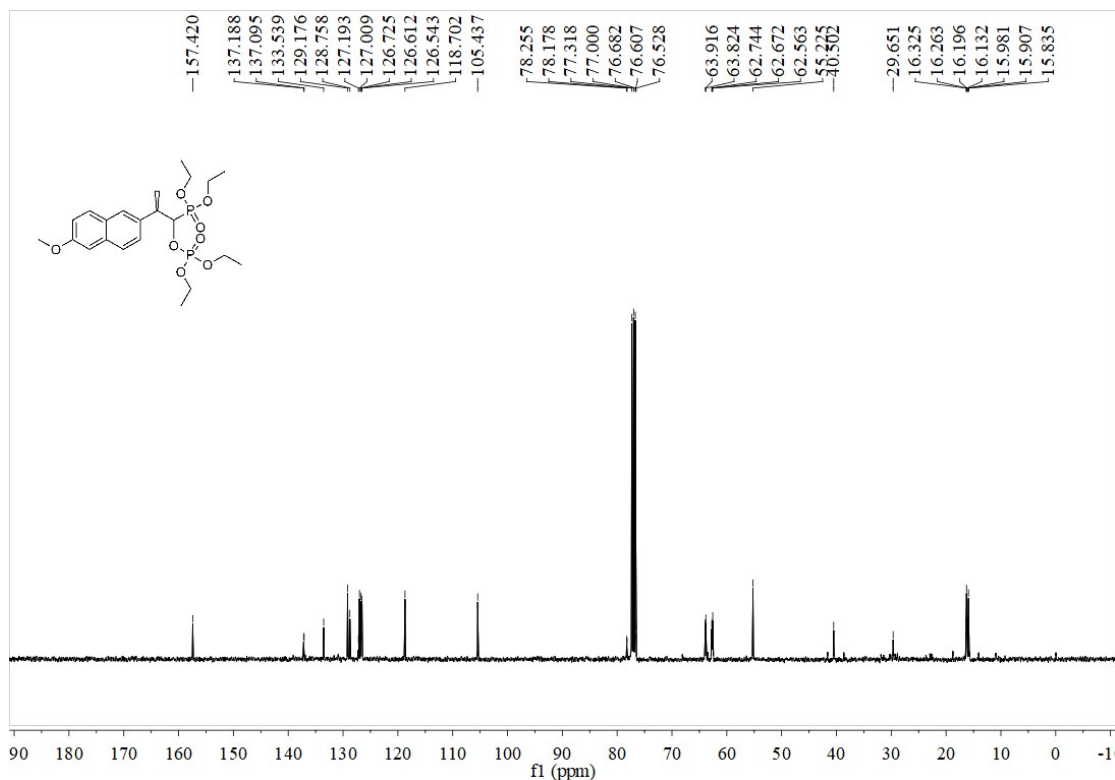
¹³C NMR spectra of 1-(diethoxyphosphoryl)-2-(4-isobutylphenyl)propyl diethyl phosphate (**42c**)



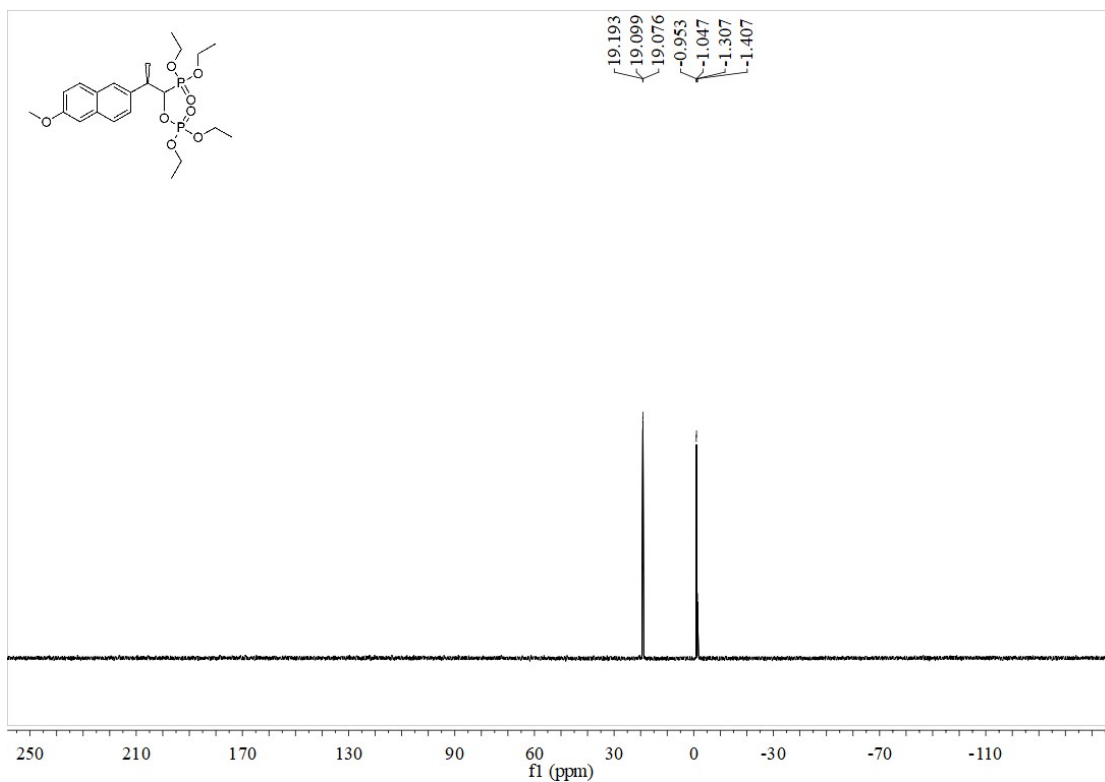
³¹P NMR spectra of 1-(diethoxyphosphoryl)-2-(4-isobutylphenyl)propyl diethyl phosphate (**42c**)



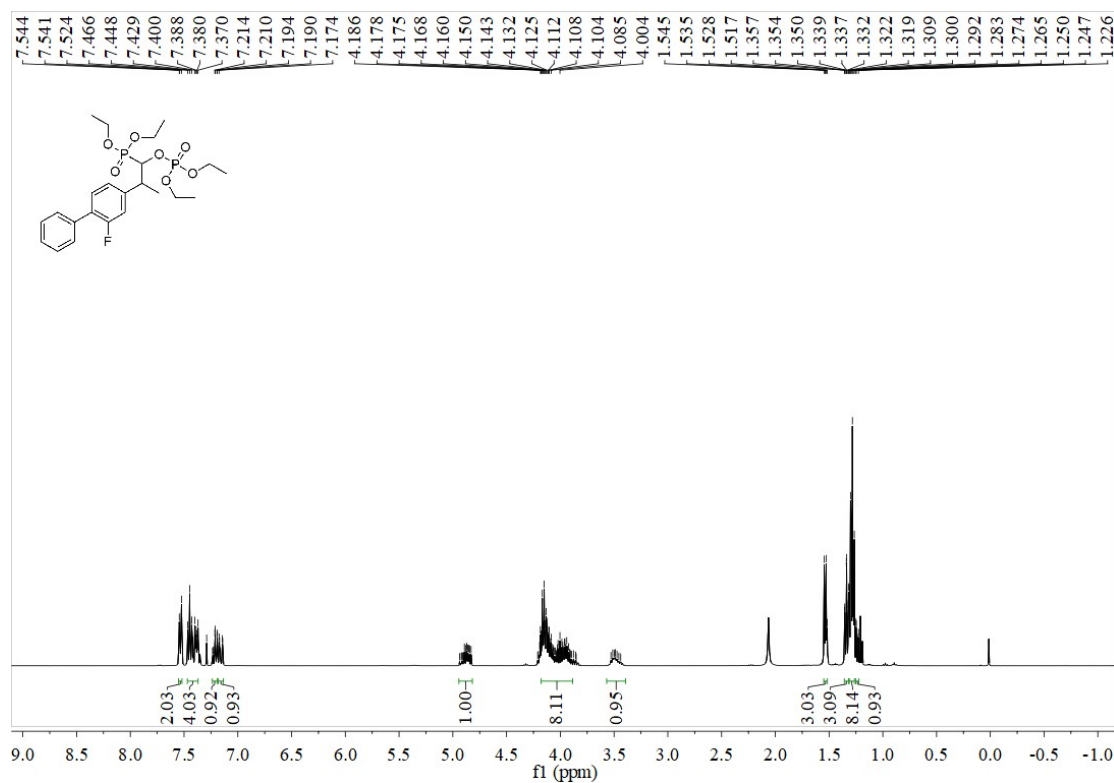
¹H NMR spectra of 1-(diethoxyphosphoryl)-2-(6-methoxynaphthalen-2-yl)propyl diethyl phosphate (43c).



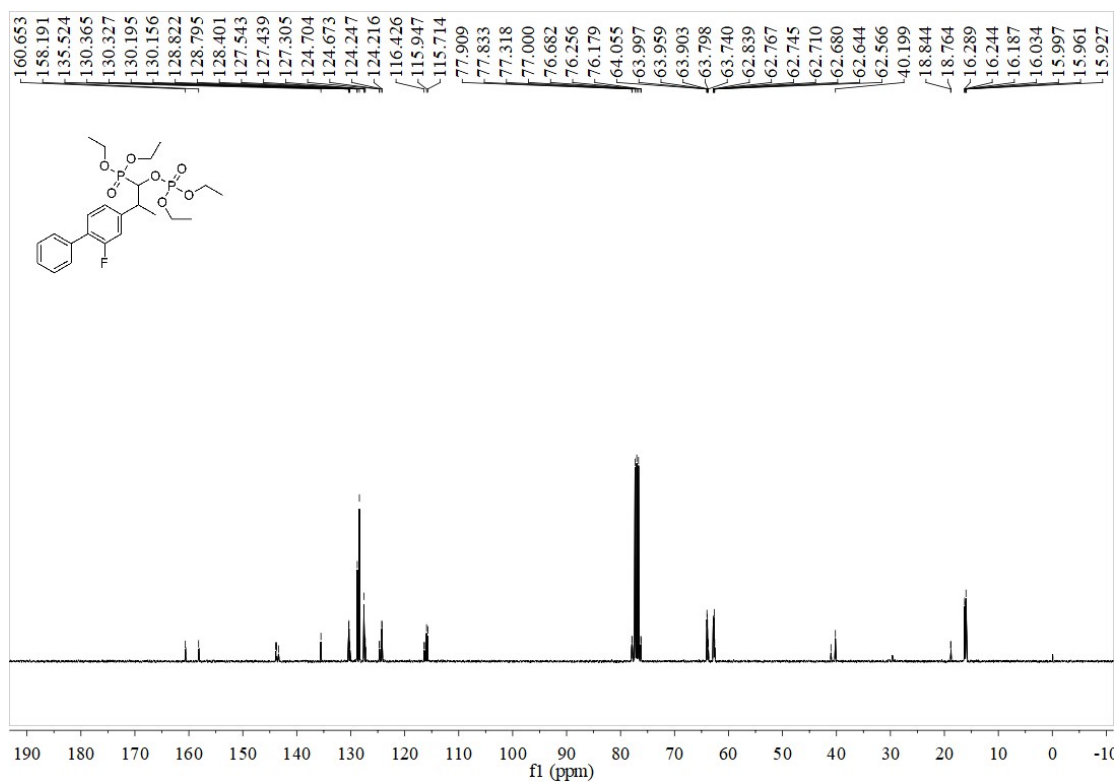
¹³C NMR spectra of 1-(diethoxyphosphoryl)-2-(6-methoxynaphthalen-2-yl)propyl diethyl phosphate (43c).



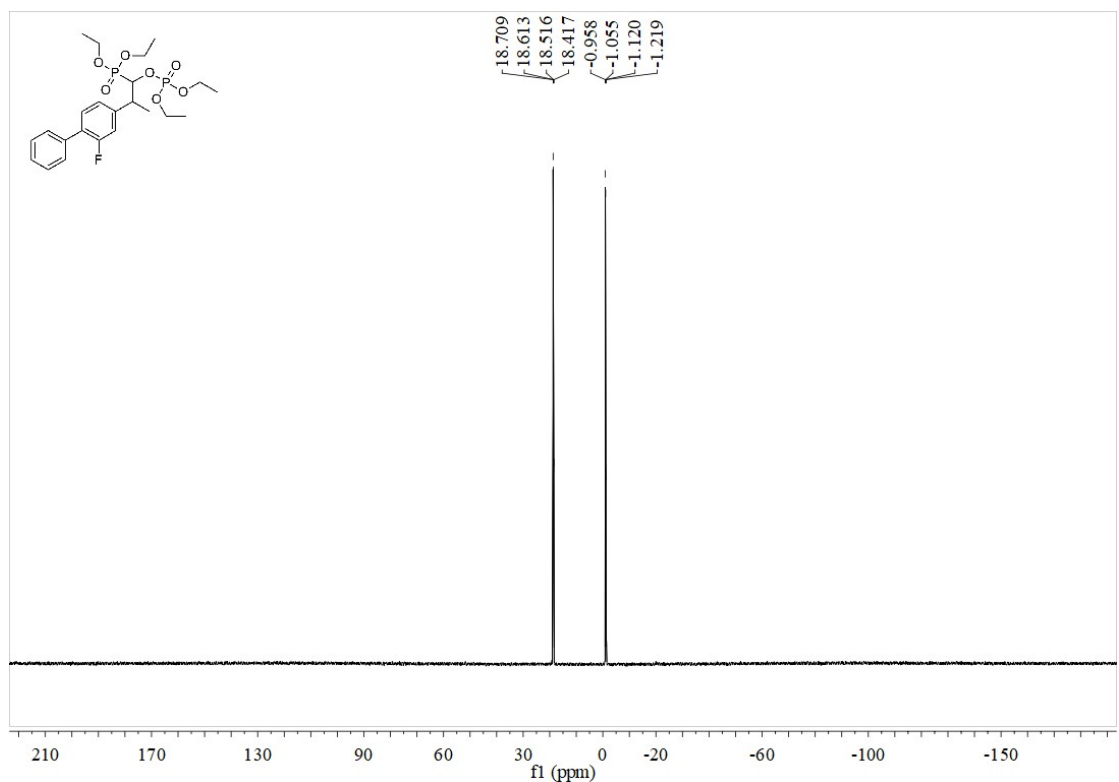
^{31}P NMR spectra of 1-(diethoxyphosphoryl)-2-(6-methoxynaphthalen-2-yl)propyl diethyl phosphate (43c).



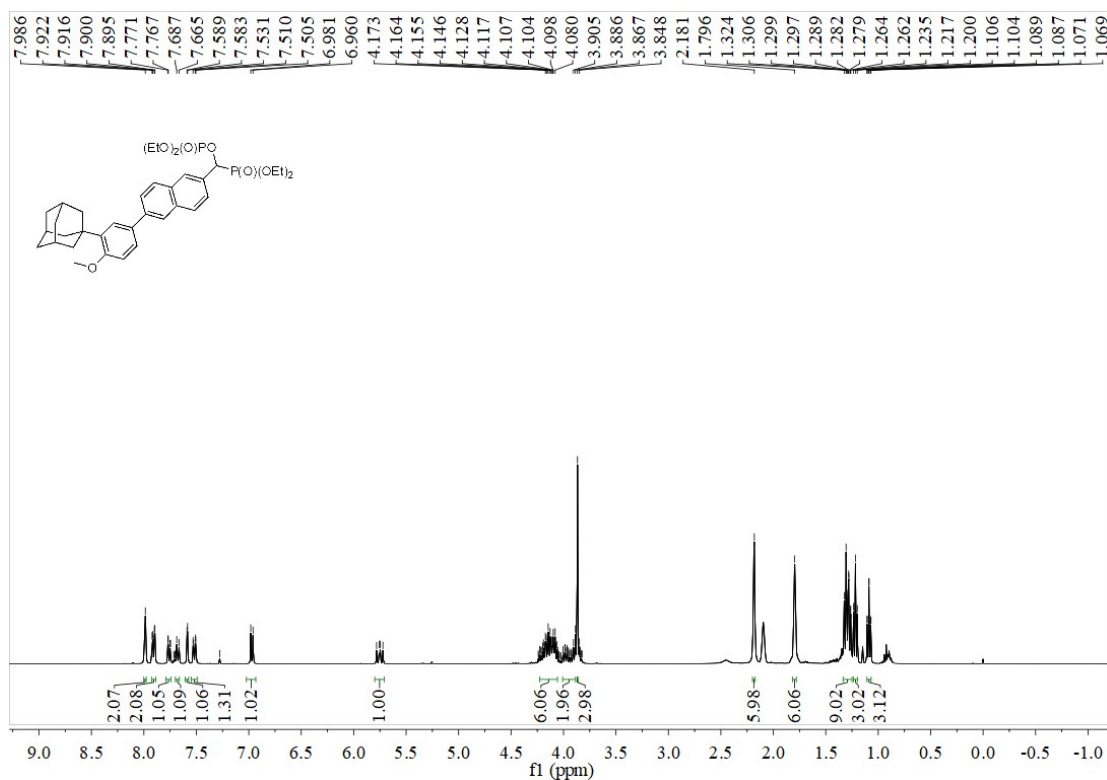
^1H NMR spectra of 1-(diethoxyphosphoryl)-2-(2-fluoro-[1,1'-biphenyl]-4-yl)propyl diethyl phosphate (44c).



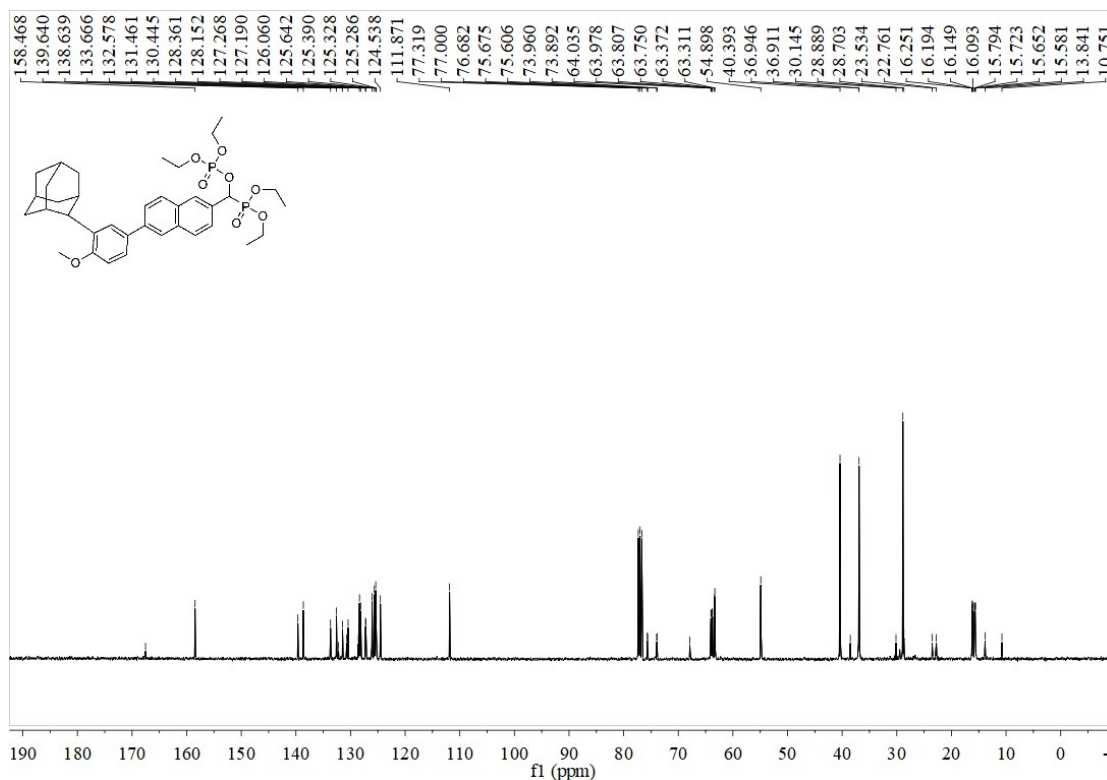
¹³C NMR spectra of 1-(diethoxyphosphoryl)-2-(2-fluoro-[1,1'-biphenyl]-4-yl)propyl diethyl phosphate (44c).



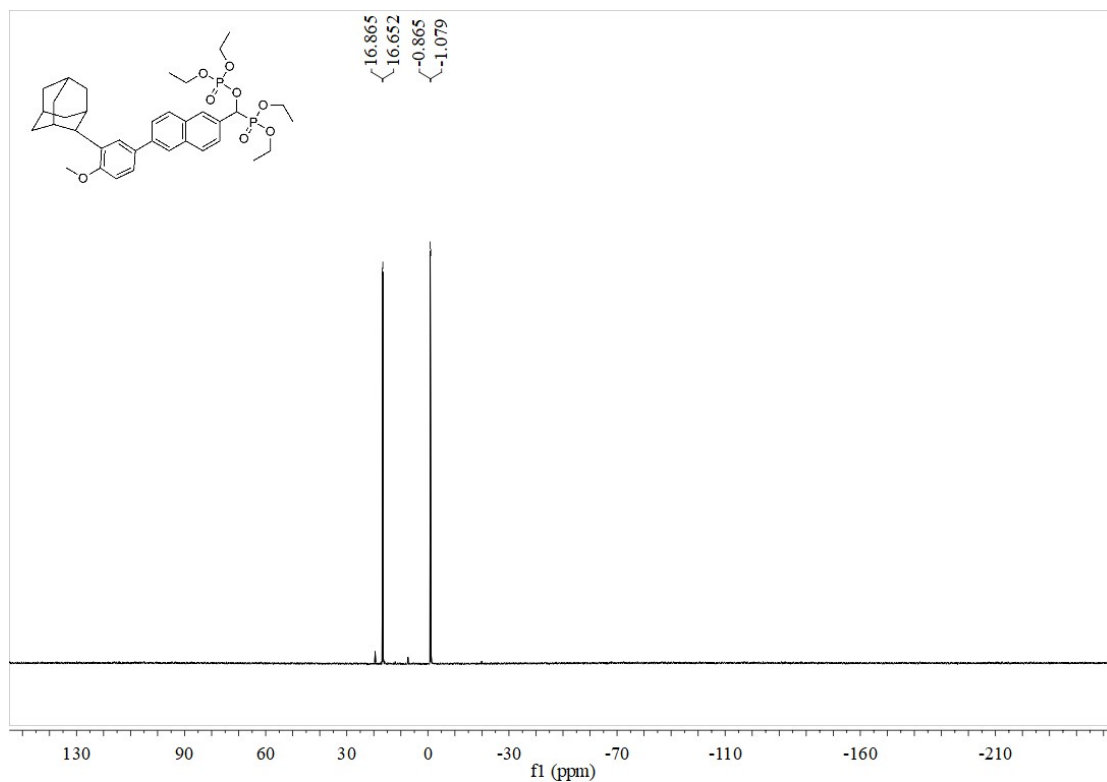
³¹P NMR spectra of 1-(diethoxyphosphoryl)-2-(2-fluoro-[1,1'-biphenyl]-4-yl)propyl diethyl phosphate (44c).



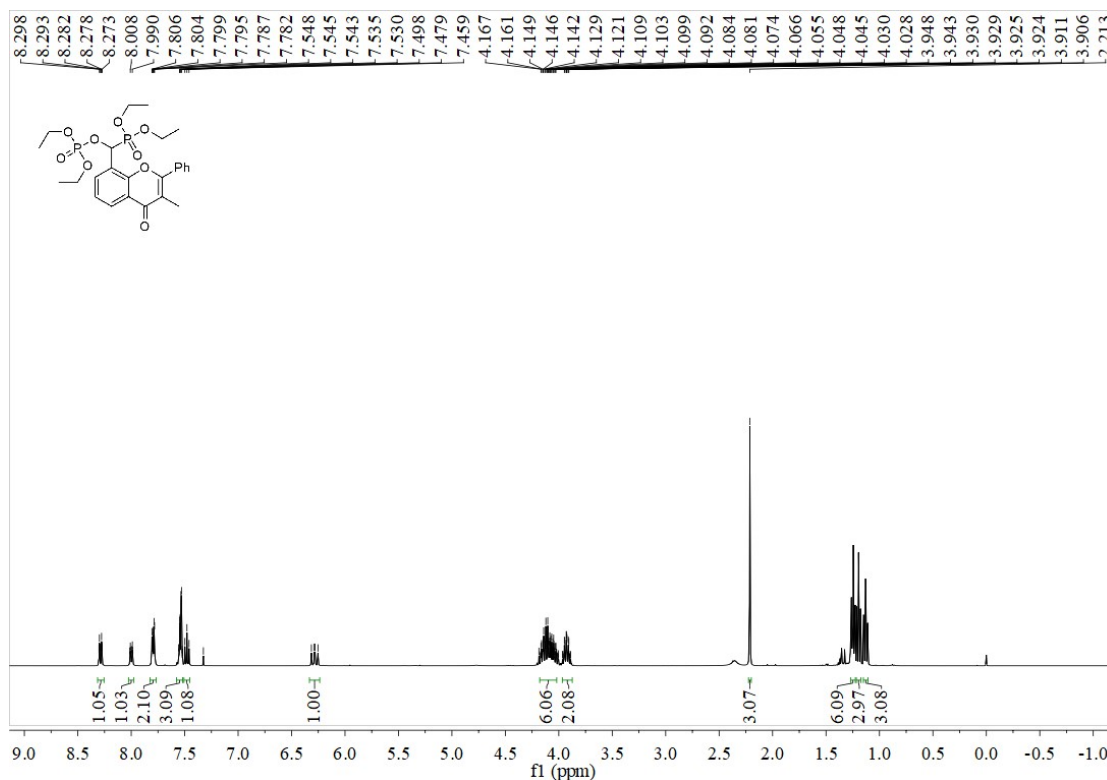
¹H NMR spectra of (6-(3-adamantan-1-yl-4-methoxyphenyl)naphthalen-2-yl)(diethoxyphosphoryl) methyl diethyl phosphate (**45c**).



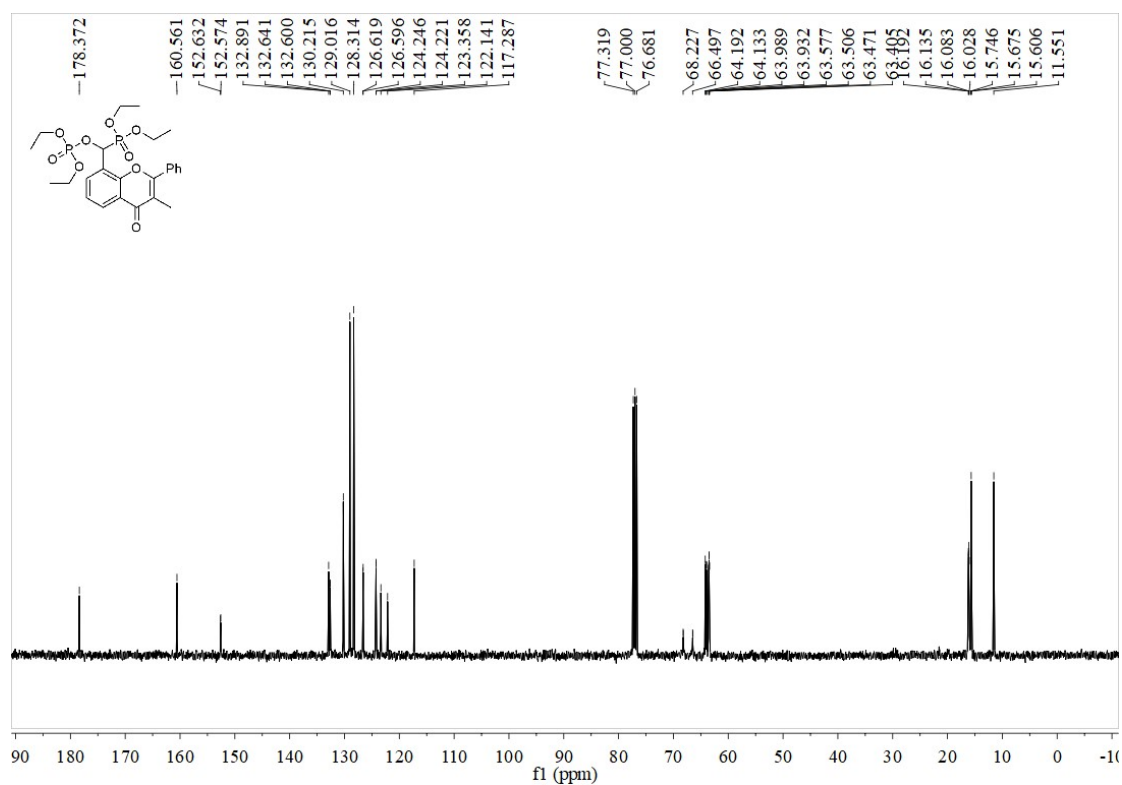
¹³C NMR spectra of 1-(6-(3-adamantan-1-yl-4-methoxyphenyl)naphthalen-2-yl)(diethoxyphosphoryl) methyl diethyl phosphate (**45c**).



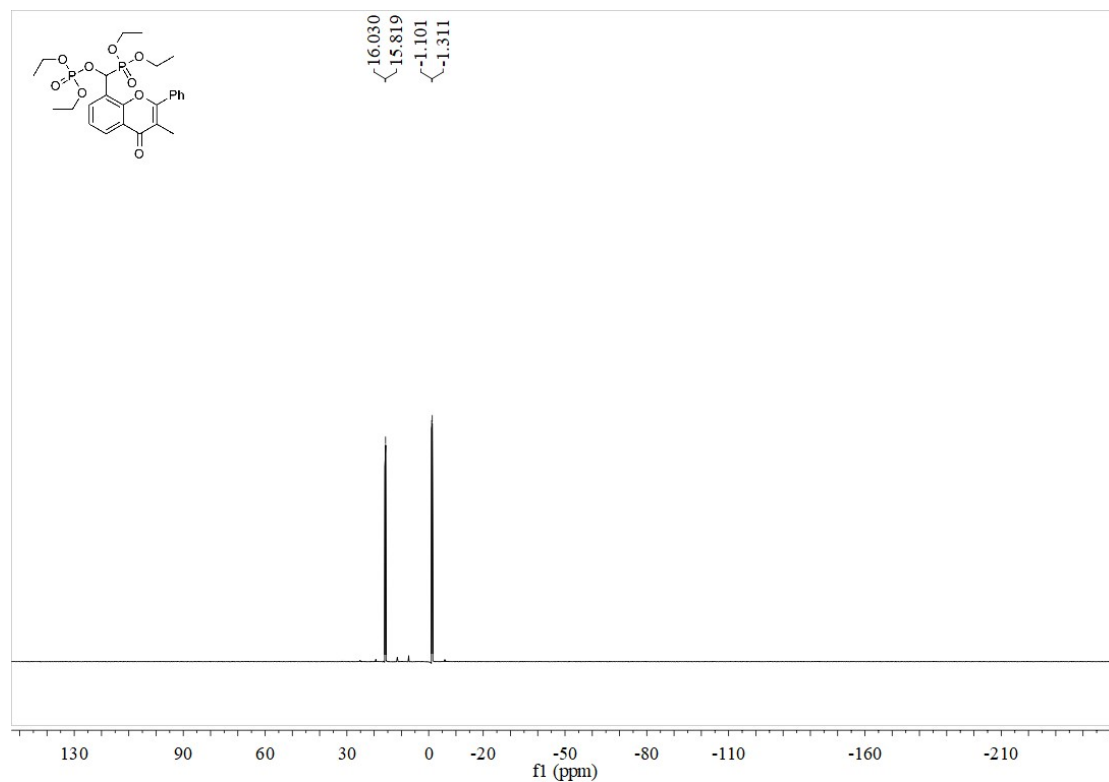
^{31}P NMR spectra of (6-(3-adamantan-1-yl-4-methoxyphenyl)naphthalen-2-yl)(diethoxyphosphoryl) methyl diethyl phosphate (**45c**).



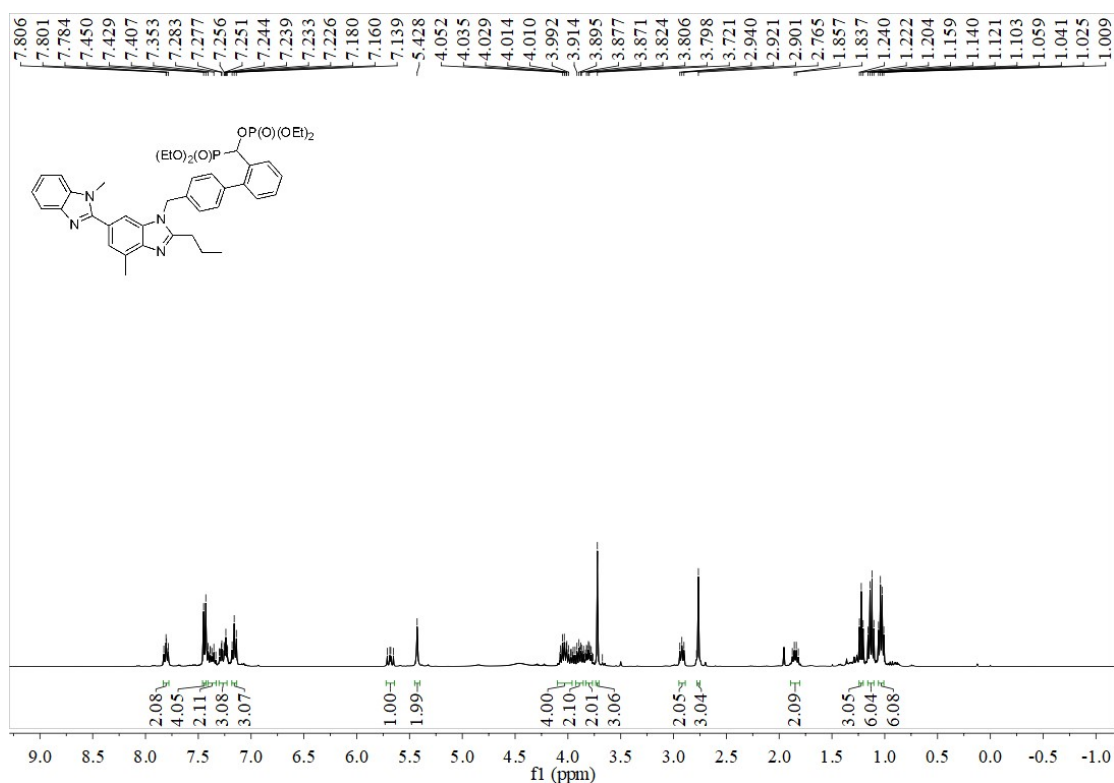
^1H NMR spectra of (diethoxyphosphoryl)(3-methyl-4-oxo-2-phenyl-4H-chromen-8-yl)methyl diethyl phosphate (**46c**).



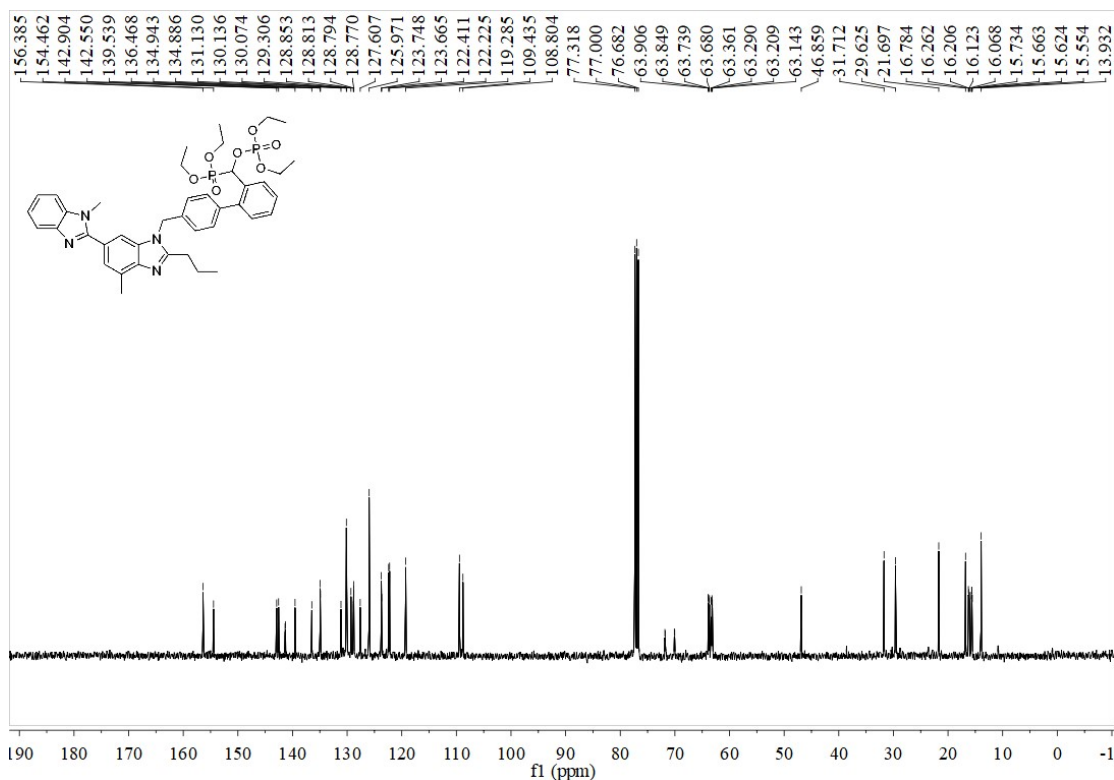
¹³C NMR spectra of (diethoxyphosphoryl)(3-methyl-4-oxo-2-phenyl-4H-chromen-8-yl)methyl diethyl phosphate (**46c**).



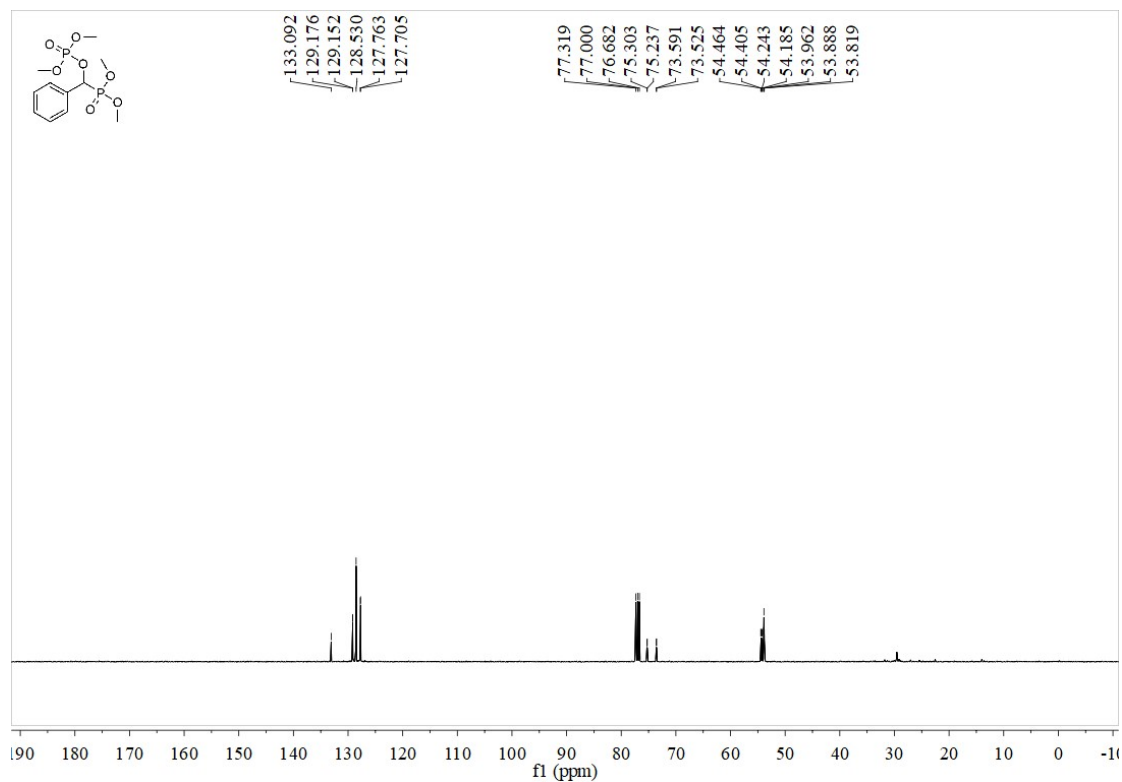
³¹P NMR spectra of (diethoxyphosphoryl)(3-methyl-4-oxo-2-phenyl-4H-chromen-8-yl)methyl diethyl phosphate (**46c**).



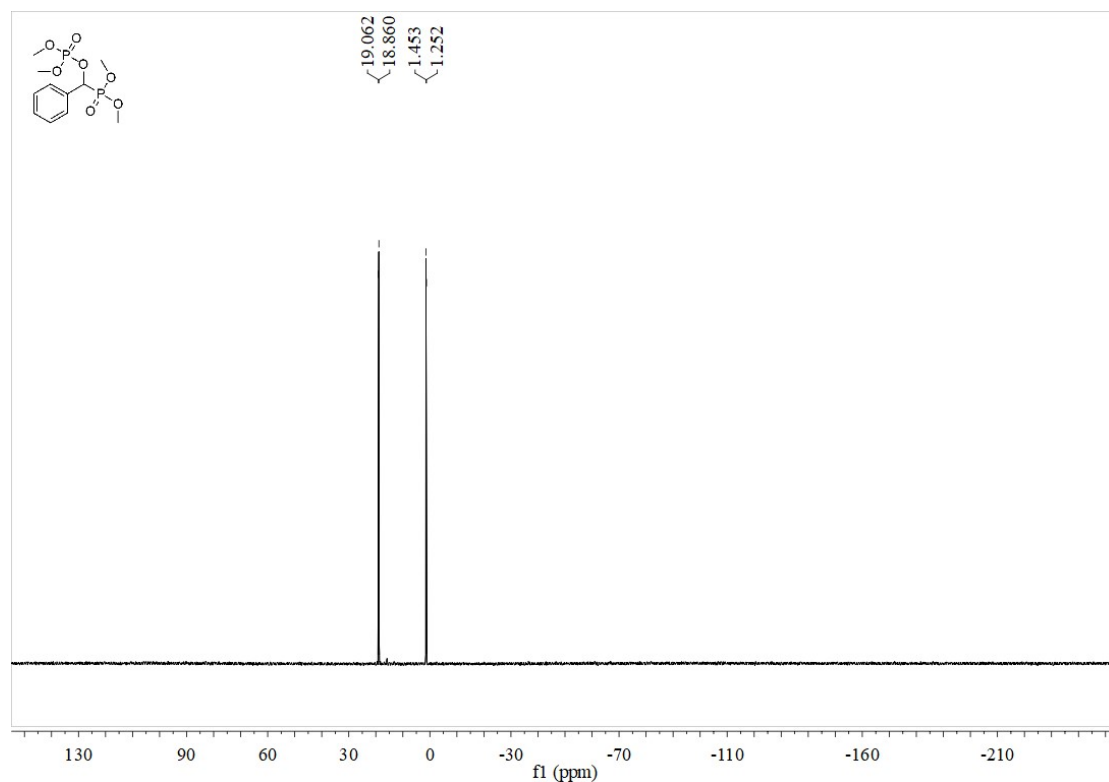
¹H NMR spectra of (diethoxyphosphoryl)(4'-((1,7'-dimethyl-2'-propyl-1H,3'H-[2,5'-bibenzo [d]imidazol]-3'-yl)methyl)-[1,1'-biphenyl]-2-yl)methyl diethyl phosphate (**47c**).



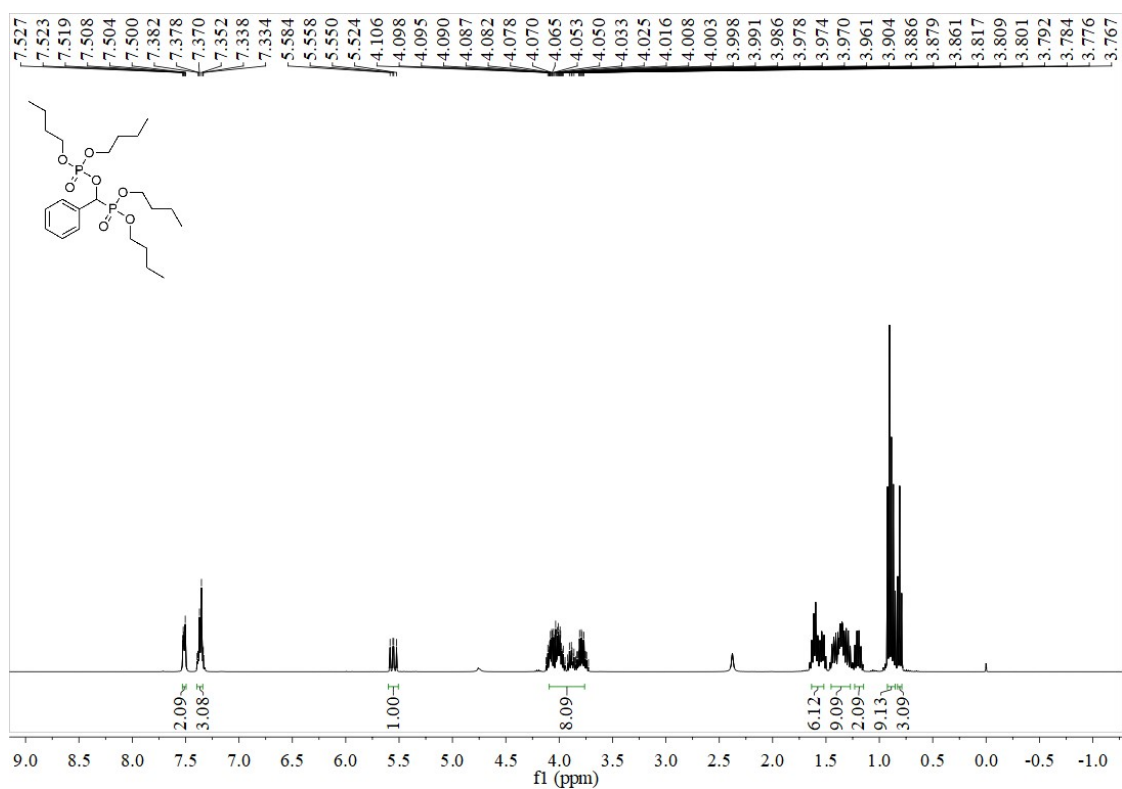
¹³C NMR spectra of (diethoxyphosphoryl)(4'-((1,7'-dimethyl-2'-propyl-1H,3'H-[2,5'-bibenzo [d]imidazol]-3'-yl)methyl)-[1,1'-biphenyl]-2-yl)methyl diethyl phosphate (**47c**).



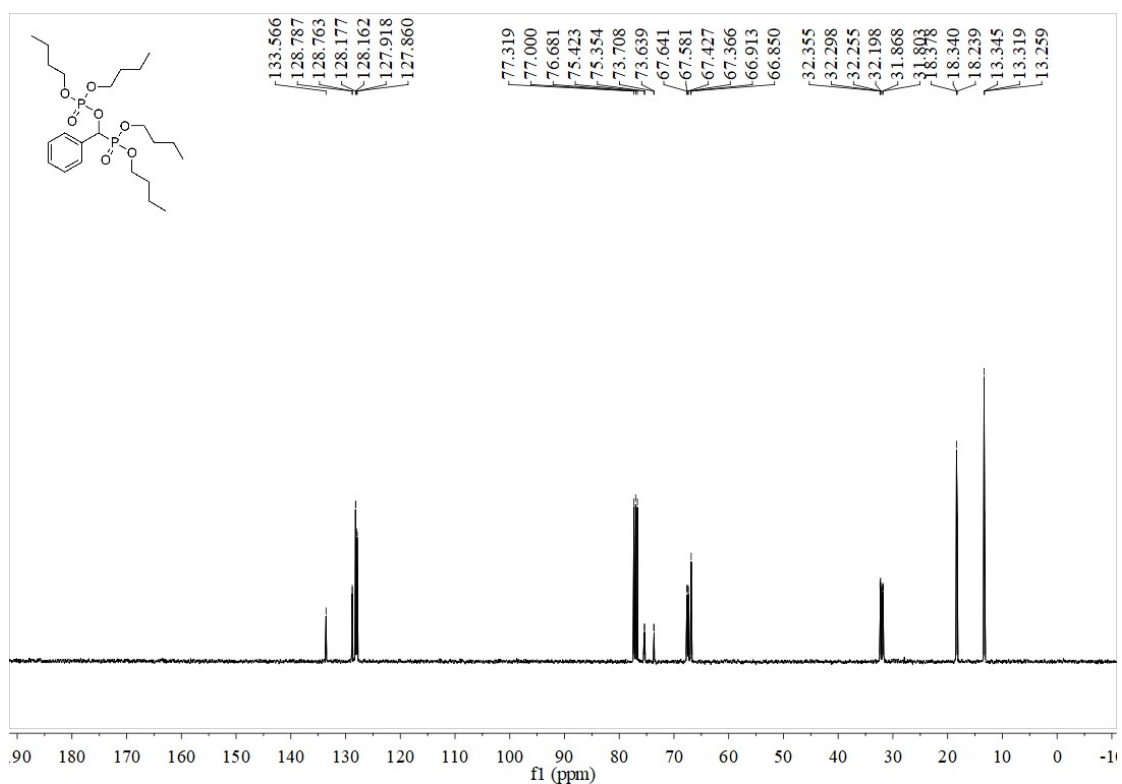
¹³C NMR spectra of (dimethoxyphosphoryl)(phenyl)methyl dimethyl phosphate (**48c**).



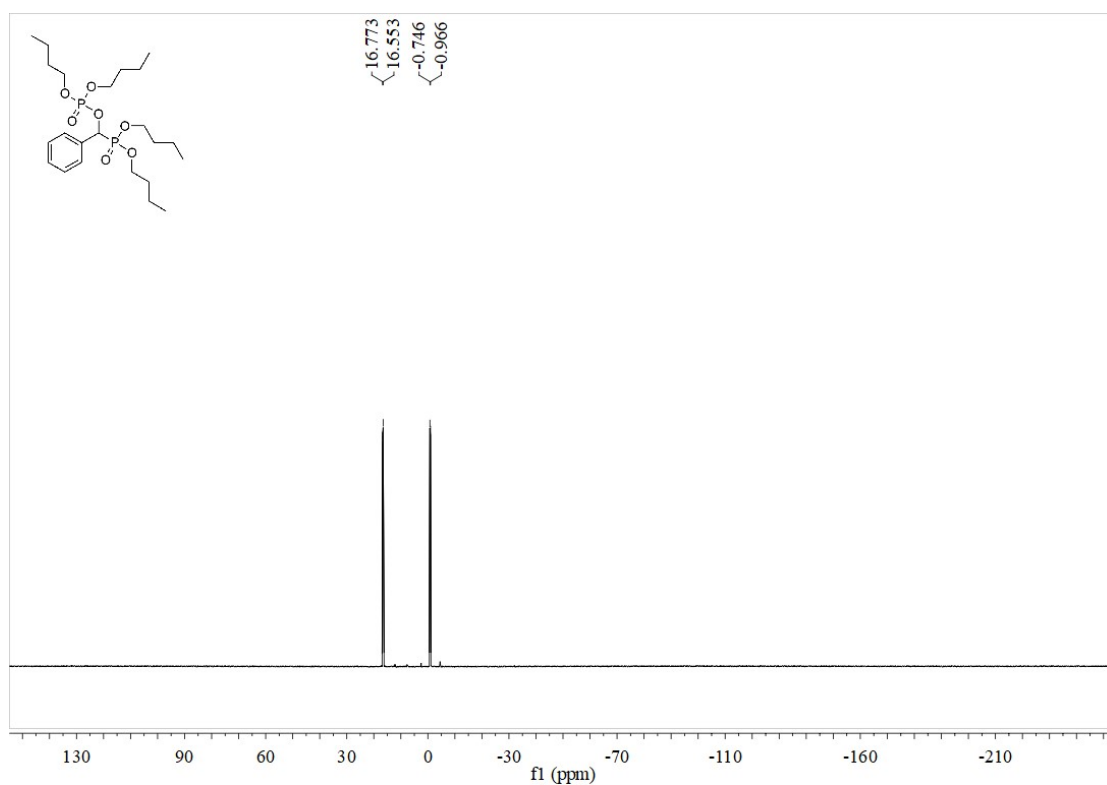
³¹P NMR spectra of (dimethoxyphosphoryl)(phenyl)methyl dimethyl phosphate (**48c**).



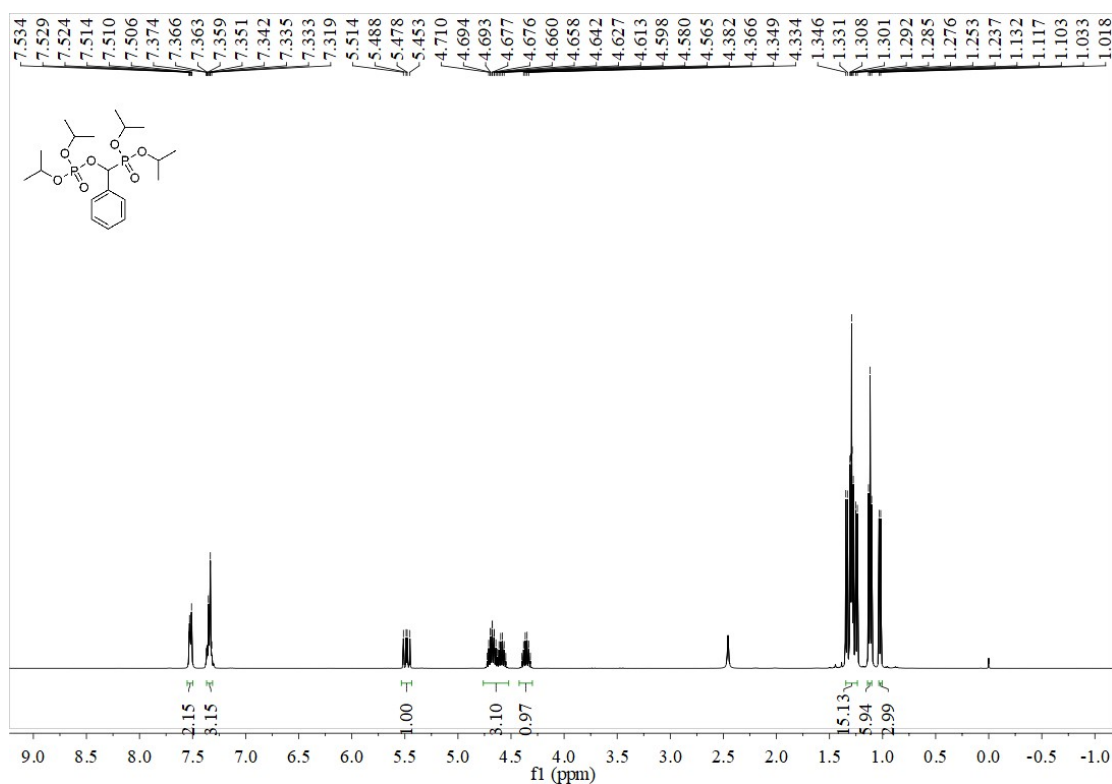
¹H NMR spectra of Dibutyl ((dibutoxyphosphoryl)(phenyl)methyl) phosphate (**49c**).



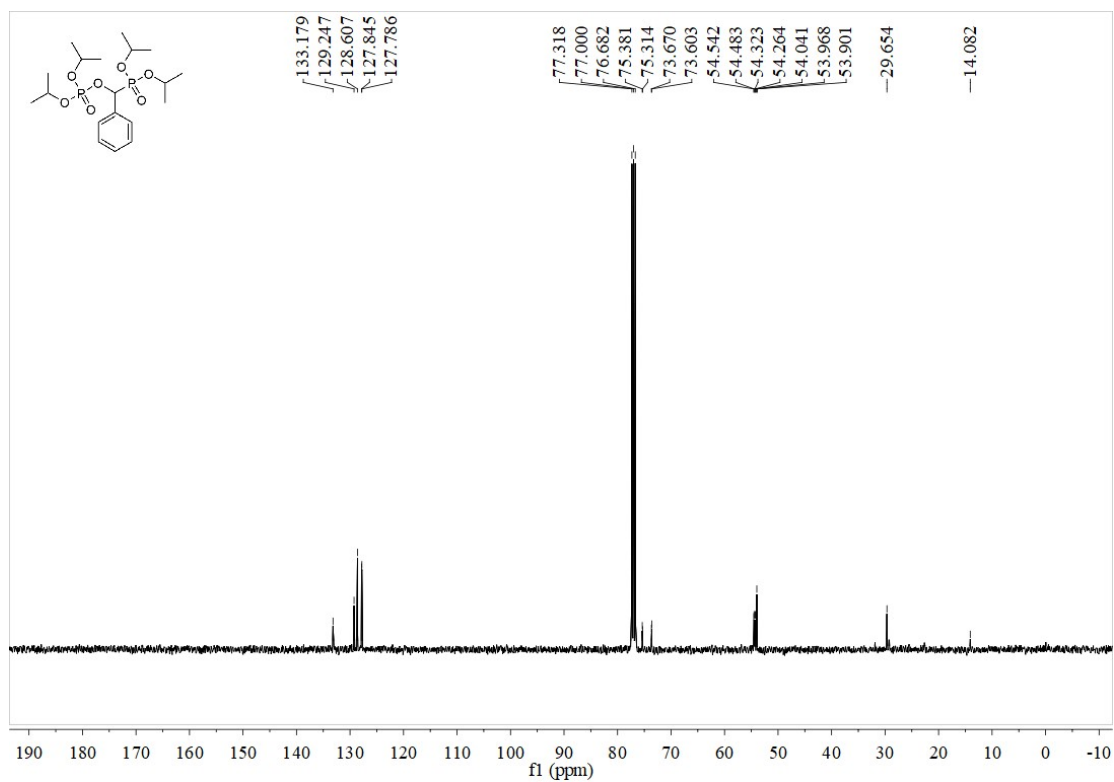
¹³C NMR spectra of Dibutyl ((dibutoxyphosphoryl)(phenyl)methyl) phosphate (**49c**).



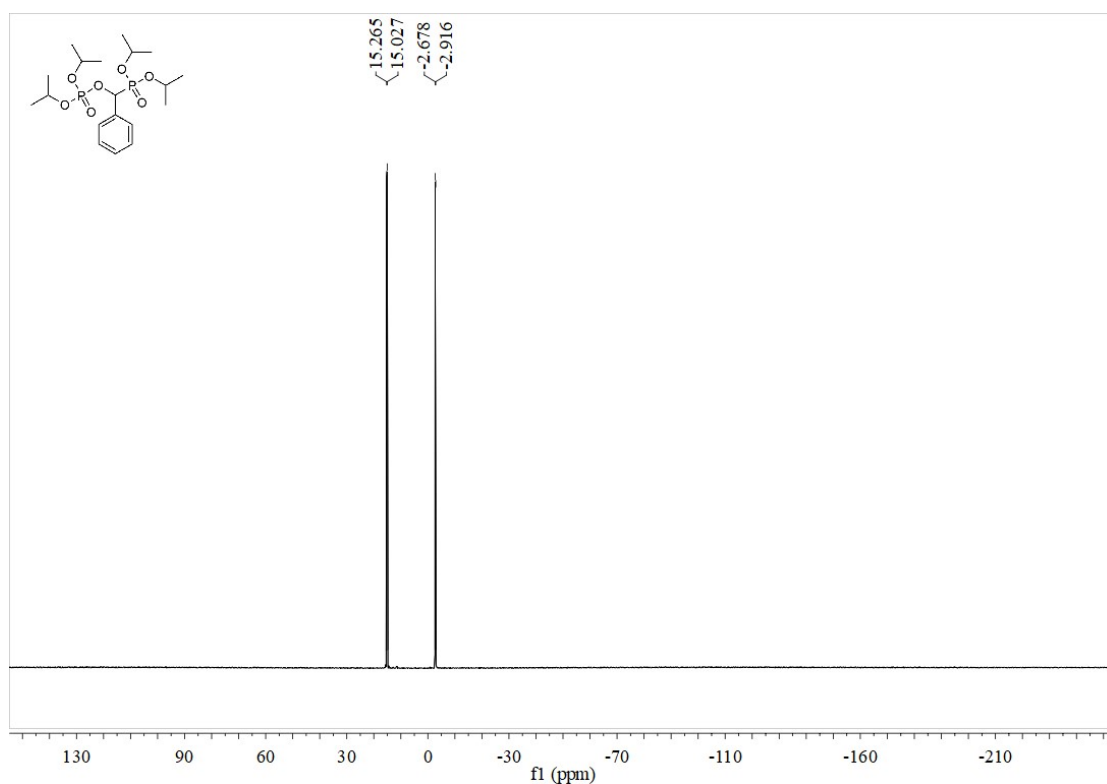
³¹P NMR spectra of Dibutyl ((dibutoxyphosphoryl)(phenyl)methyl) phosphate (**49c**).



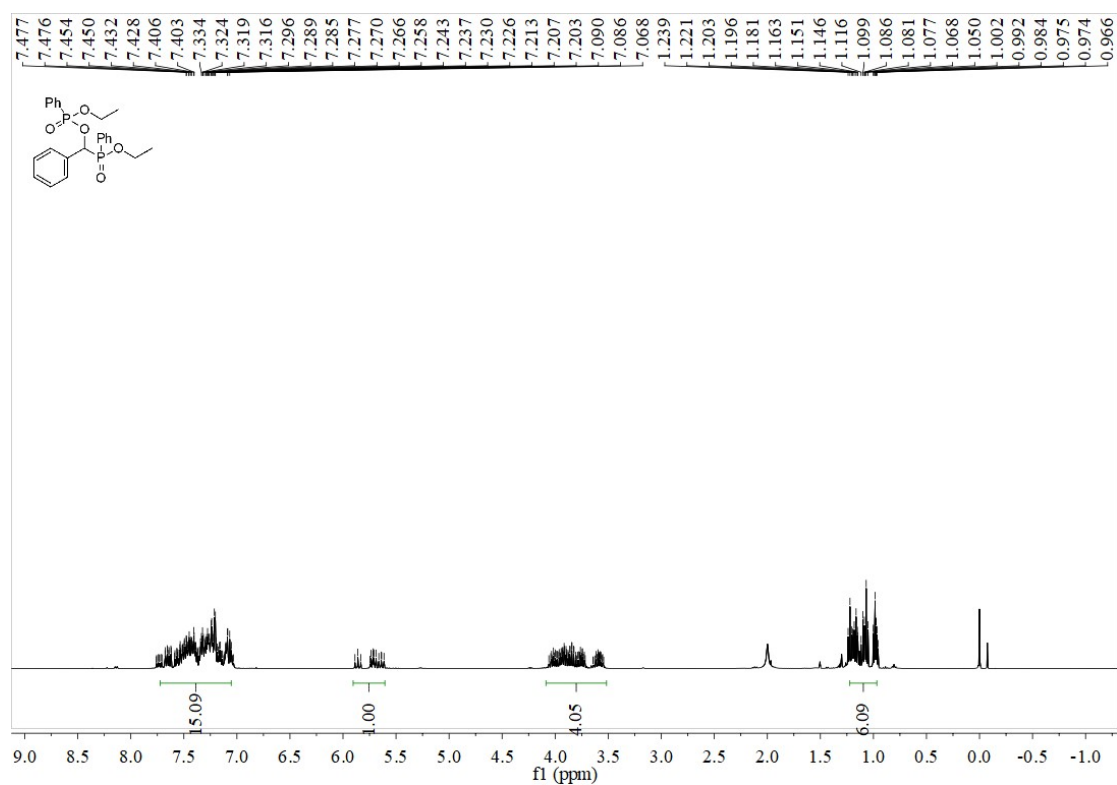
¹H NMR spectra of (diisopropoxyphosphoryl)(phenyl)methyl diisopropyl phosphate (**50c**).



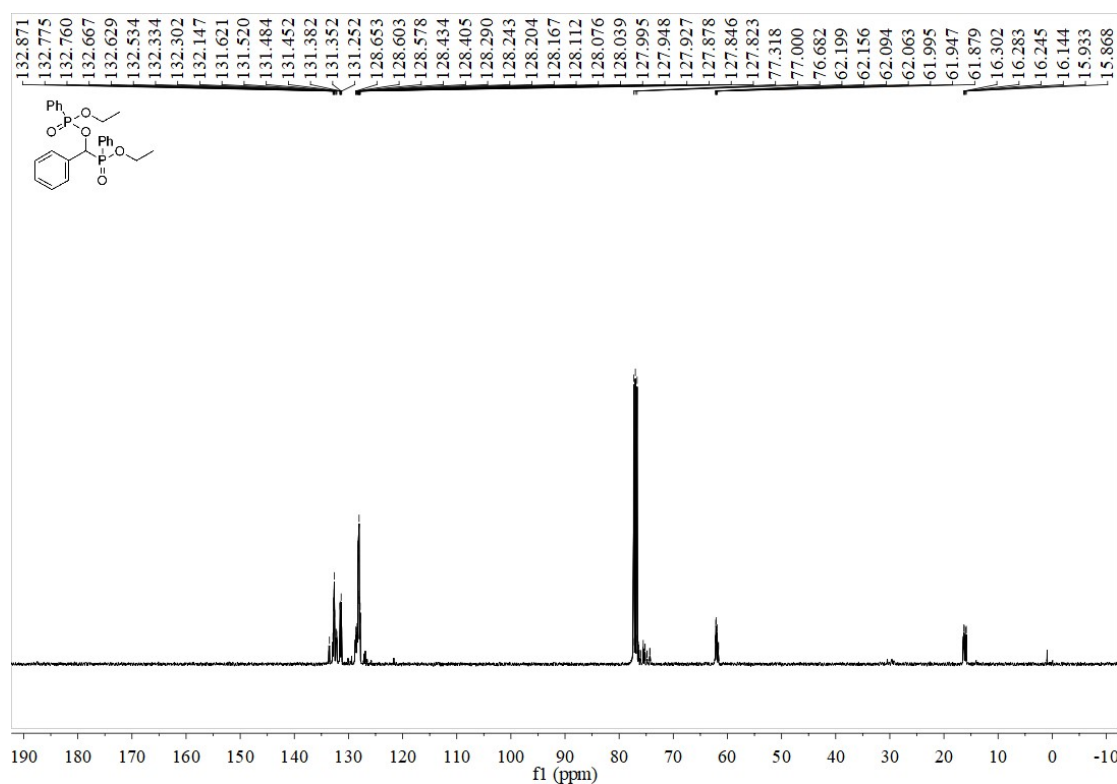
^{13}C NMR spectra of (diisopropoxyphosphoryl)(phenyl)methyl diisopropyl phosphate (**50c**).



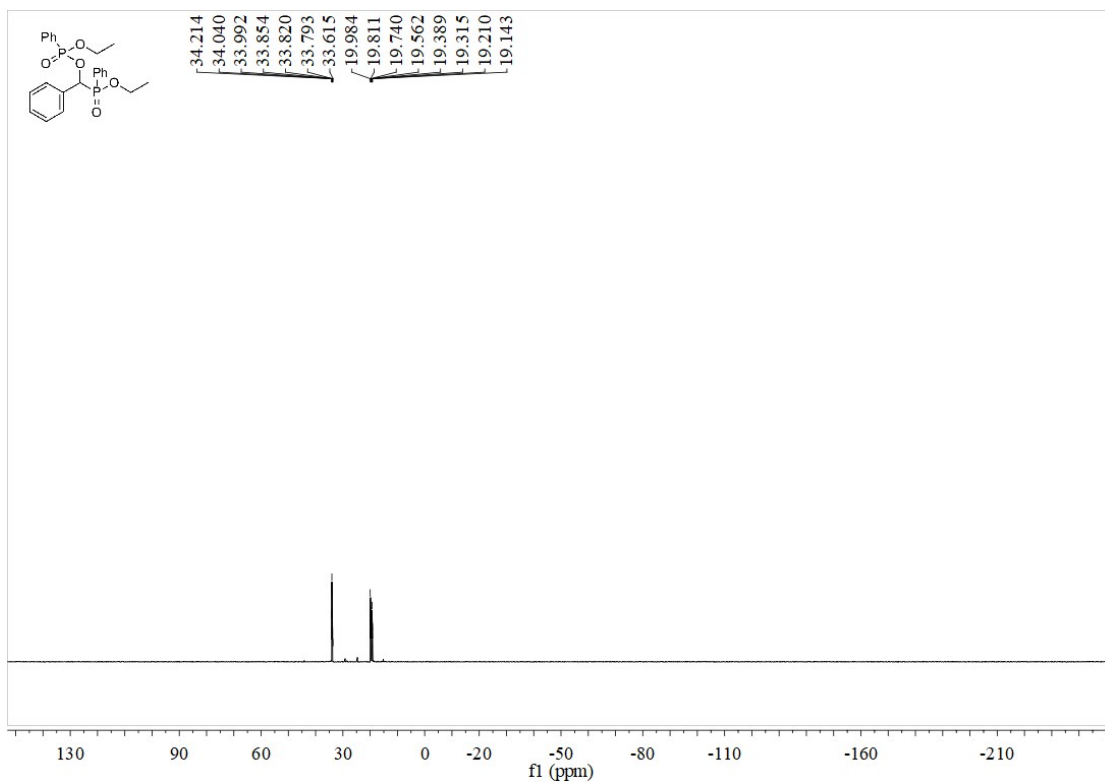
^{31}P NMR spectra of (diisopropoxyphosphoryl)(phenyl)methyl diisopropyl phosphate (**50c**).



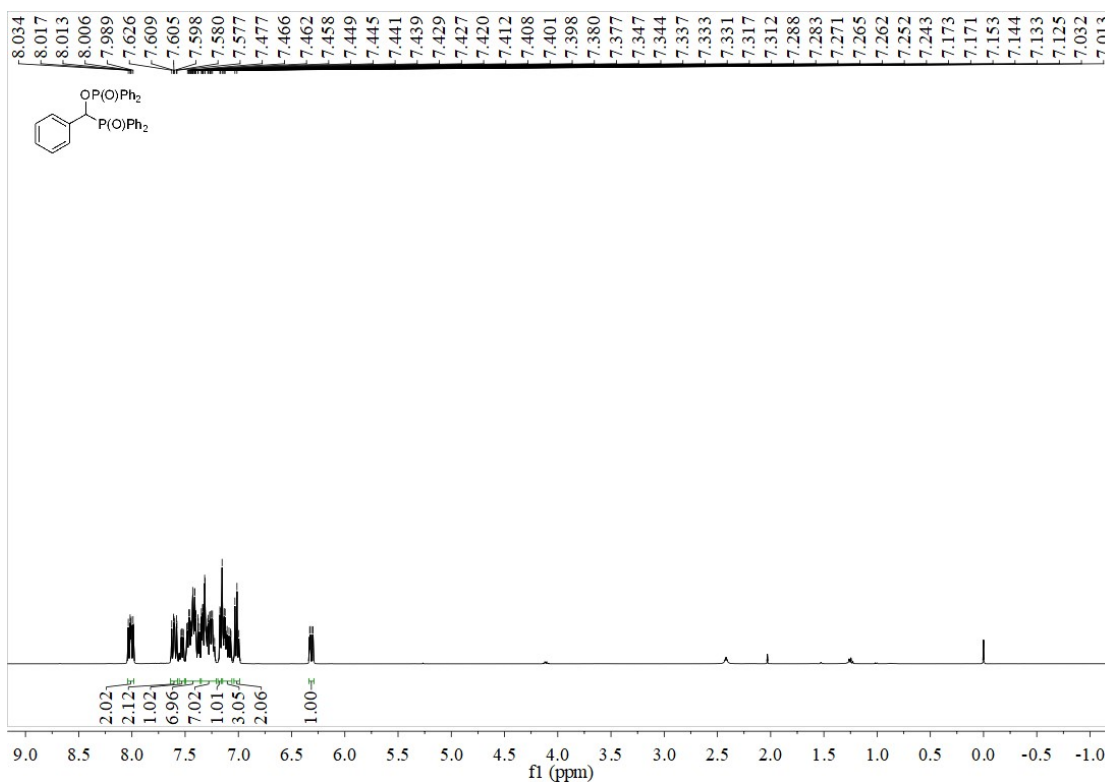
¹H NMR spectra of (ethoxy(phenyl)phosphoryl)(phenyl)methyl ethyl phenylphosphonate (**51c**).



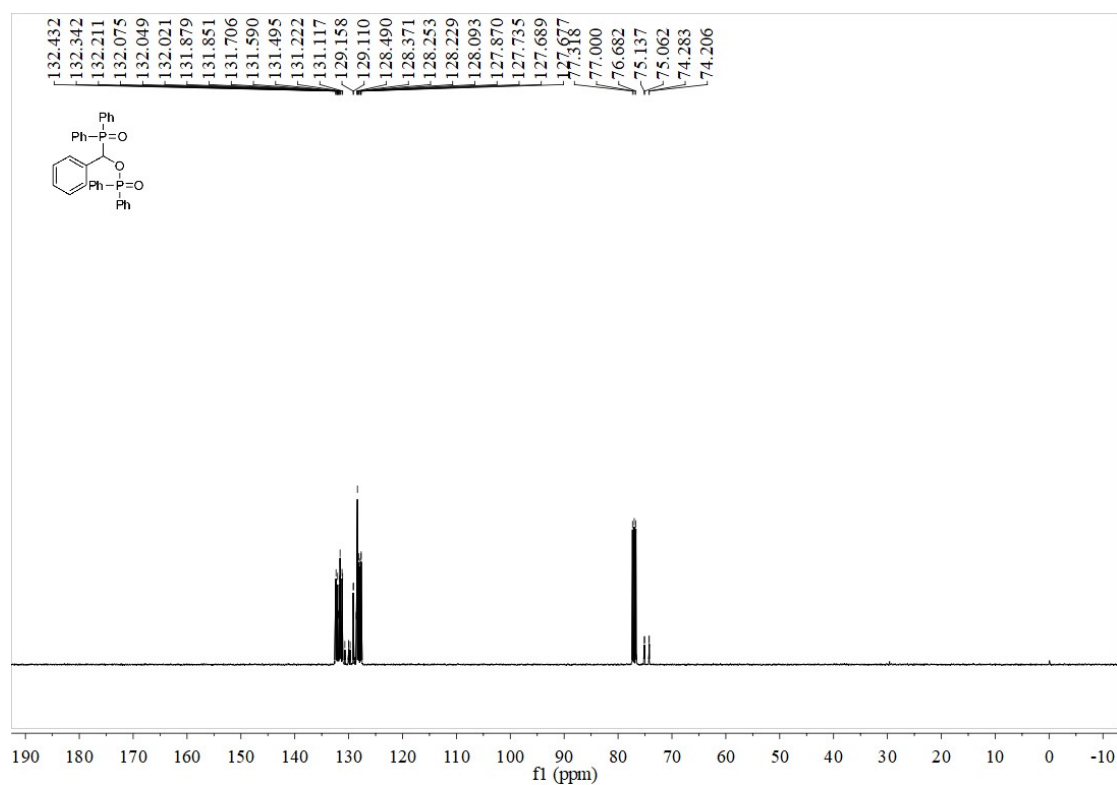
¹³C NMR spectra of (ethoxy(phenyl)phosphoryl)(phenyl)methyl ethyl phenylphosphonate (**51c**).



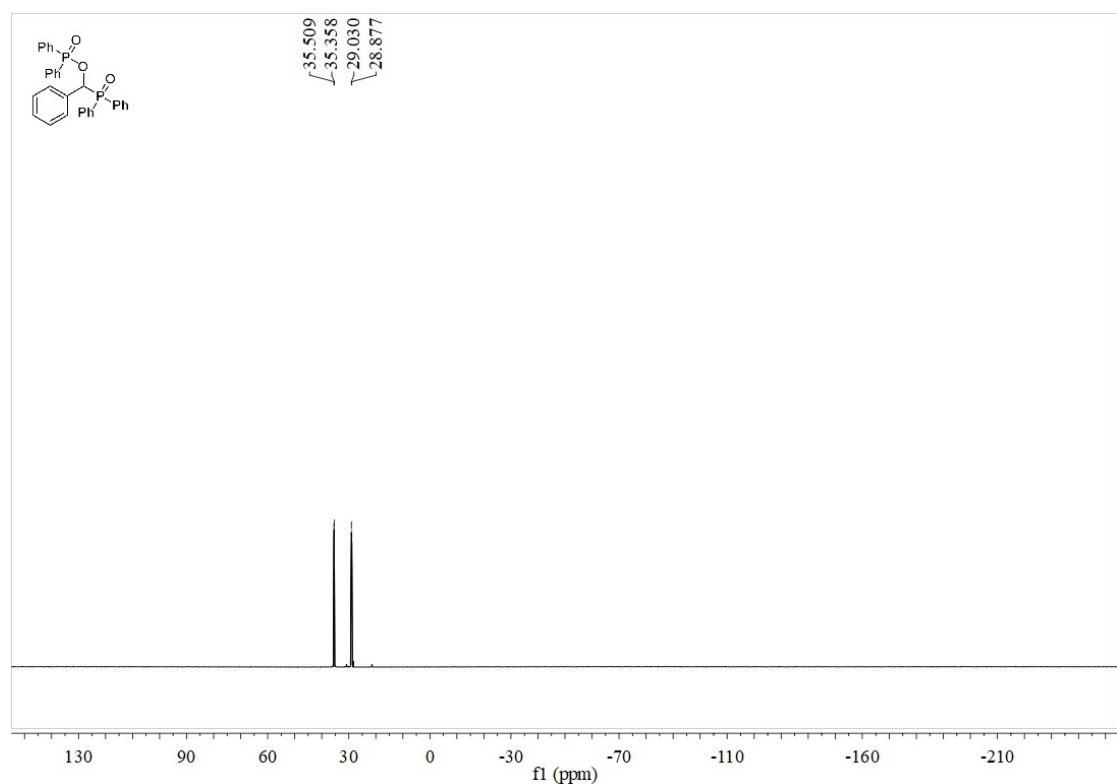
³¹P NMR spectra of (ethoxy(phenyl)phosphoryl)(phenyl)methyl ethyl phenylphosphonate (**51c**).



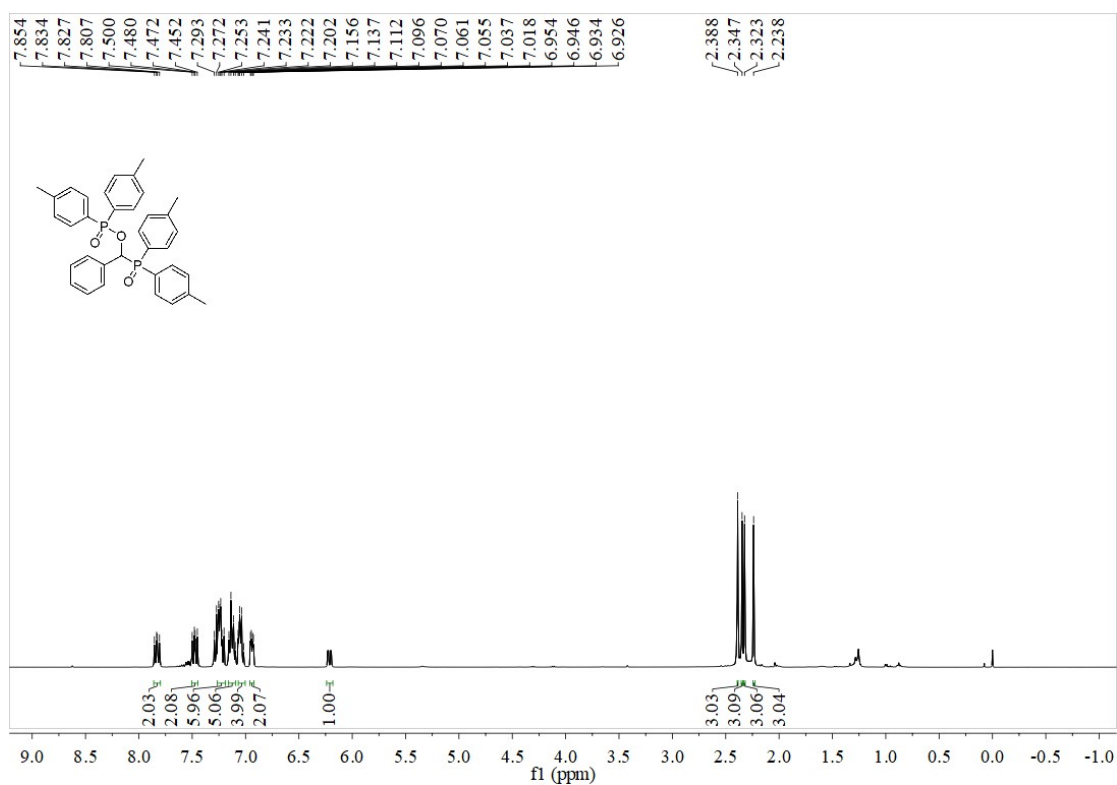
¹H NMR spectra of (diphenylphosphoryl)(phenyl)methyl diphenylphosphinate (**52c**).



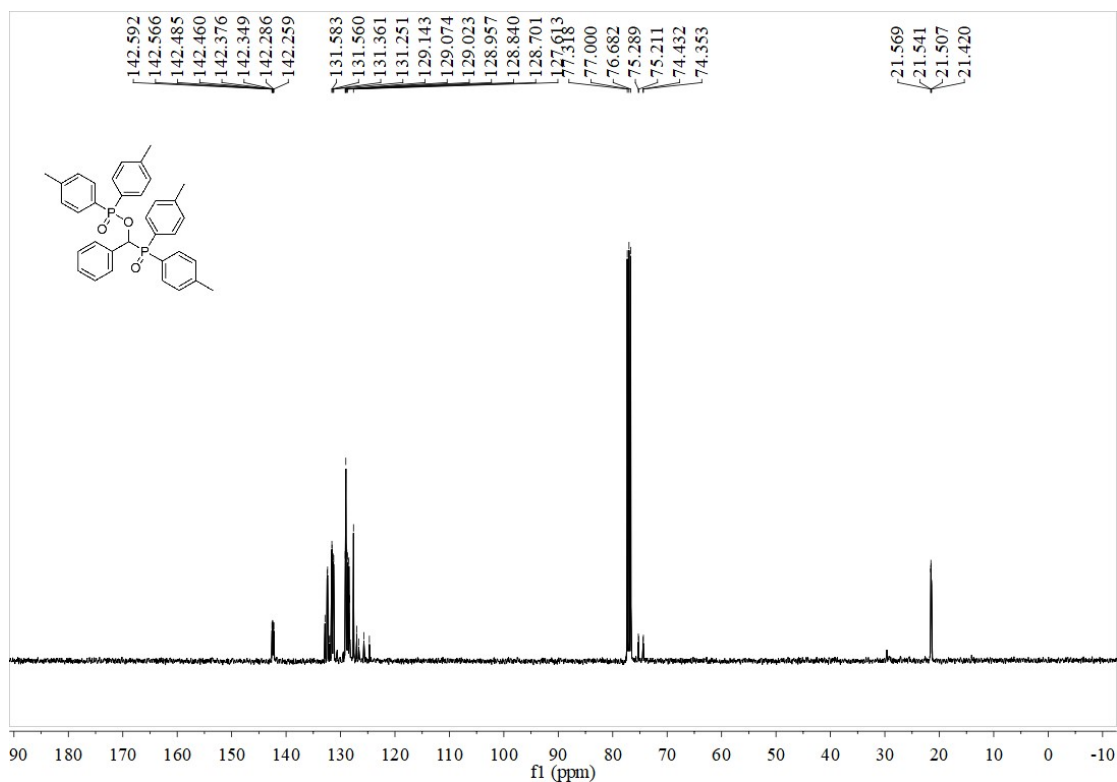
¹³C NMR spectra of (diphenylphosphoryl)(phenyl)methyl diphenylphosphinate (**52c**).



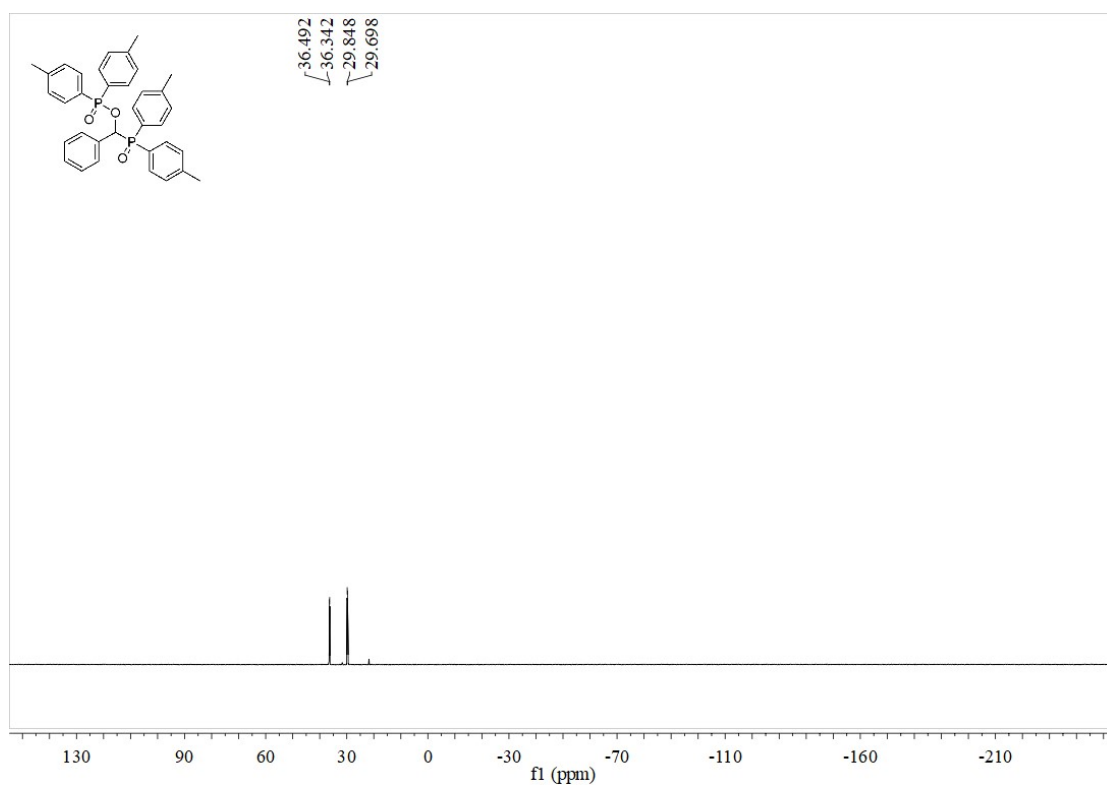
³¹P NMR spectra of (diphenylphosphoryl)(phenyl)methyl diphenylphosphinate (**52c**).



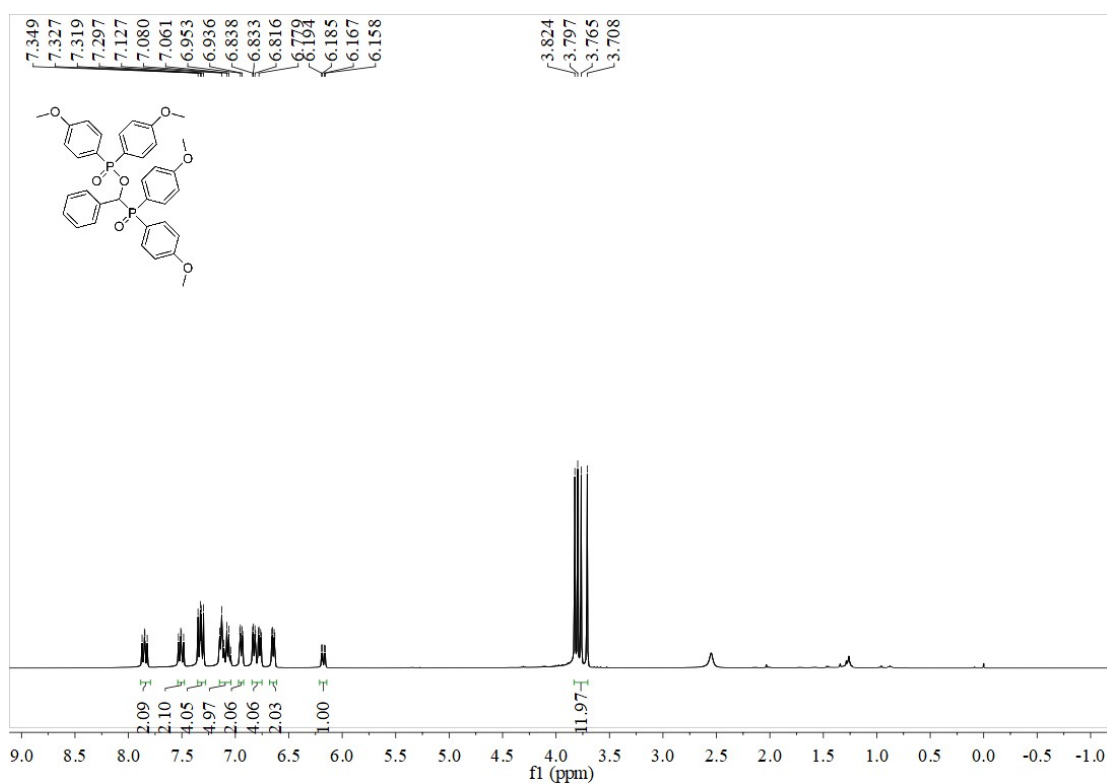
¹H NMR spectra of (di-p-tolylphosphoryl)(phenyl)methyl di-p-tolylphosphinate (**53c**).



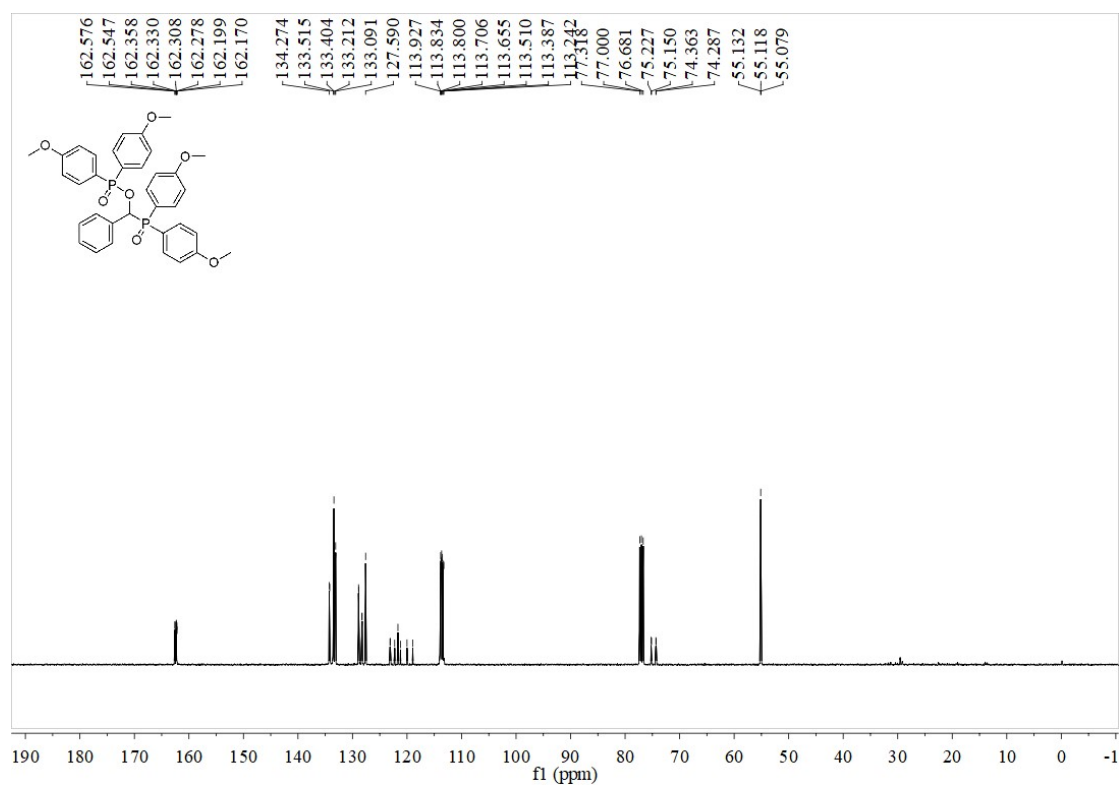
¹³C NMR spectra of (di-p-tolylphosphoryl)(phenyl)methyl di-p-tolylphosphinate (**53c**).



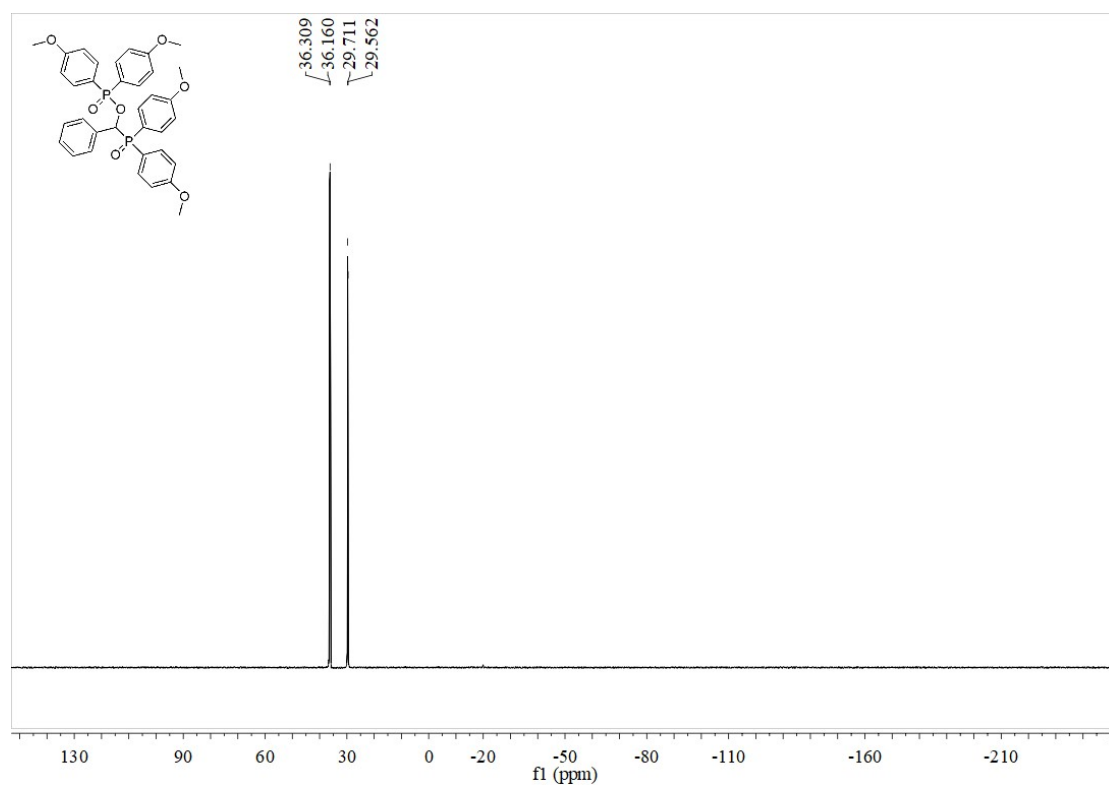
³¹P NMR spectra of (di-p-tolylphosphoryl)(phenyl)methyl di-p-tolylphosphinate (**53c**).



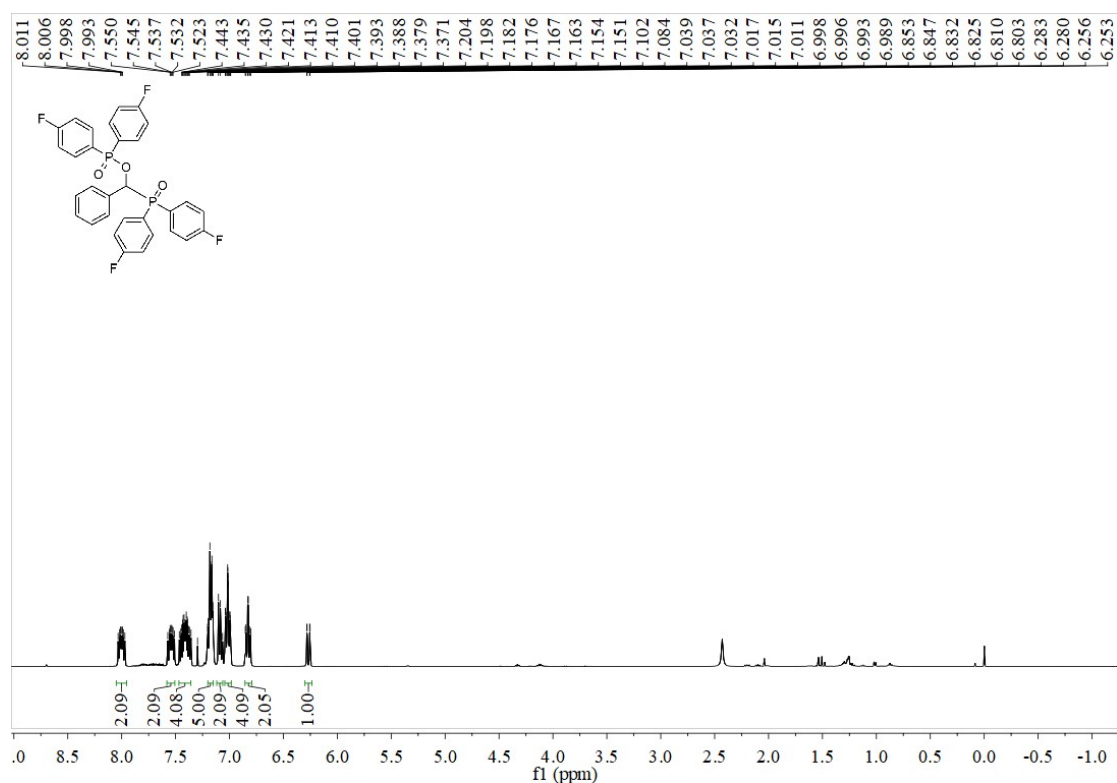
¹H NMR spectra of (bis(4-methoxyphenyl)phosphoryl)(phenyl)methyl bis(4-methoxyphenyl) phosphinate (**54c**).



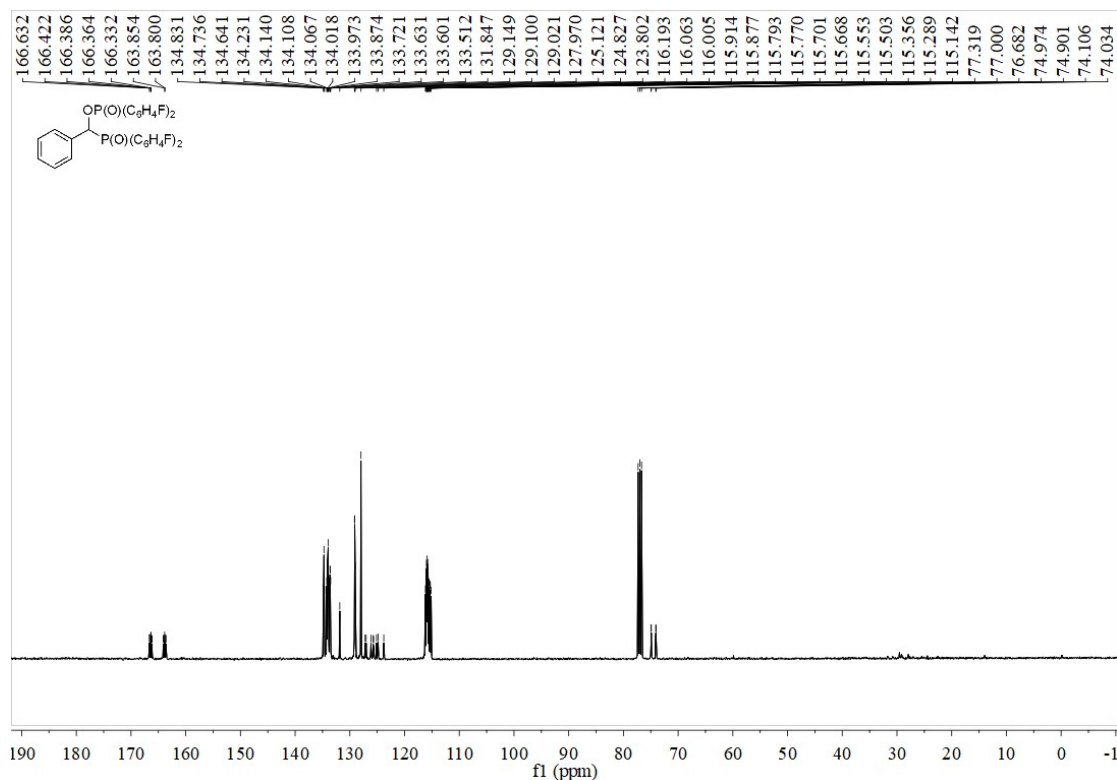
¹³C NMR spectra of (bis(4-methoxyphenyl)phosphoryl)(phenyl)methyl bis(4-methoxyphenyl) phosphinate (**54c**).



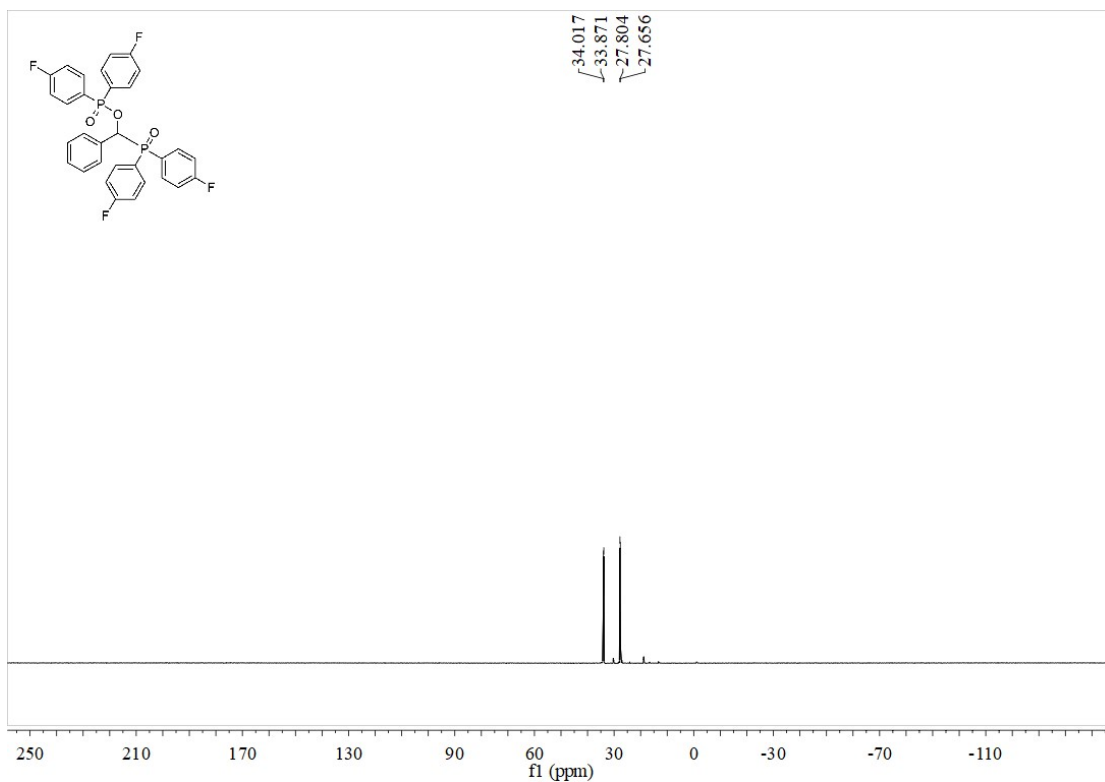
³¹P NMR spectra of (bis(4-methoxyphenyl)phosphoryl)(phenyl)methyl bis(4-methoxyphenyl) phosphinate (**54c**).



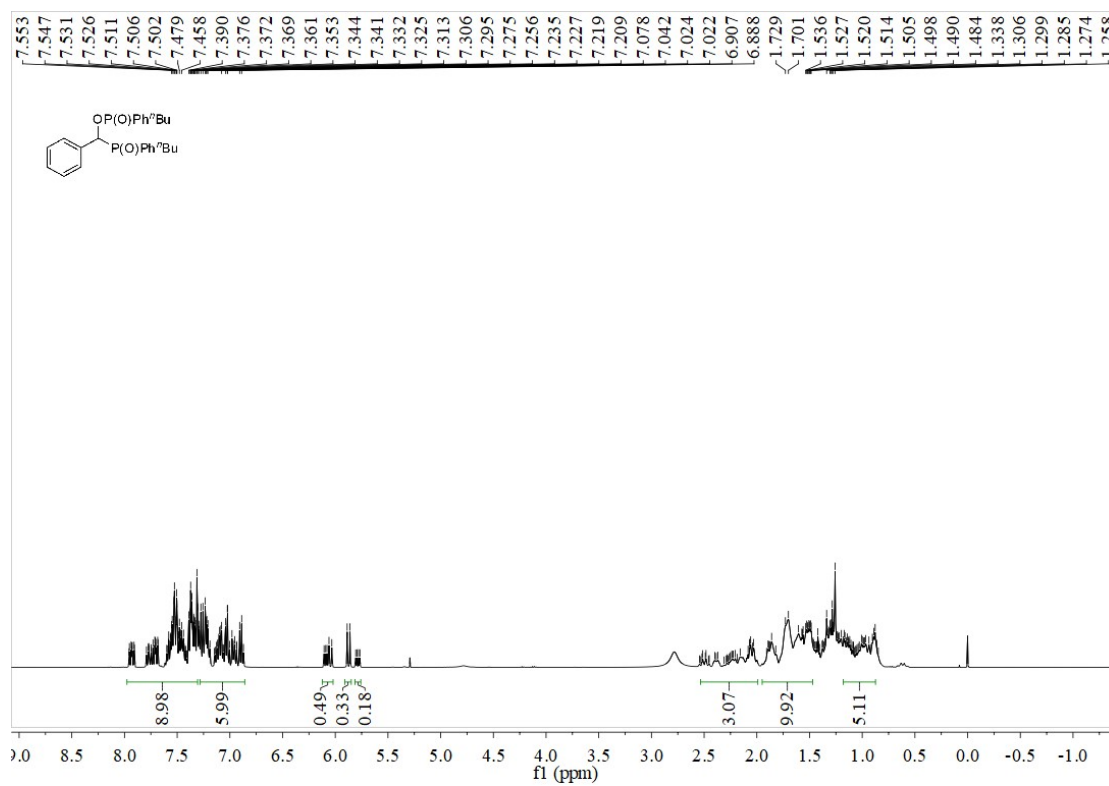
¹H NMR spectra of (bis(4-fluorophenyl)phosphoryl)(phenyl)methyl bis(4-fluorophenyl)phosphinate (55c).



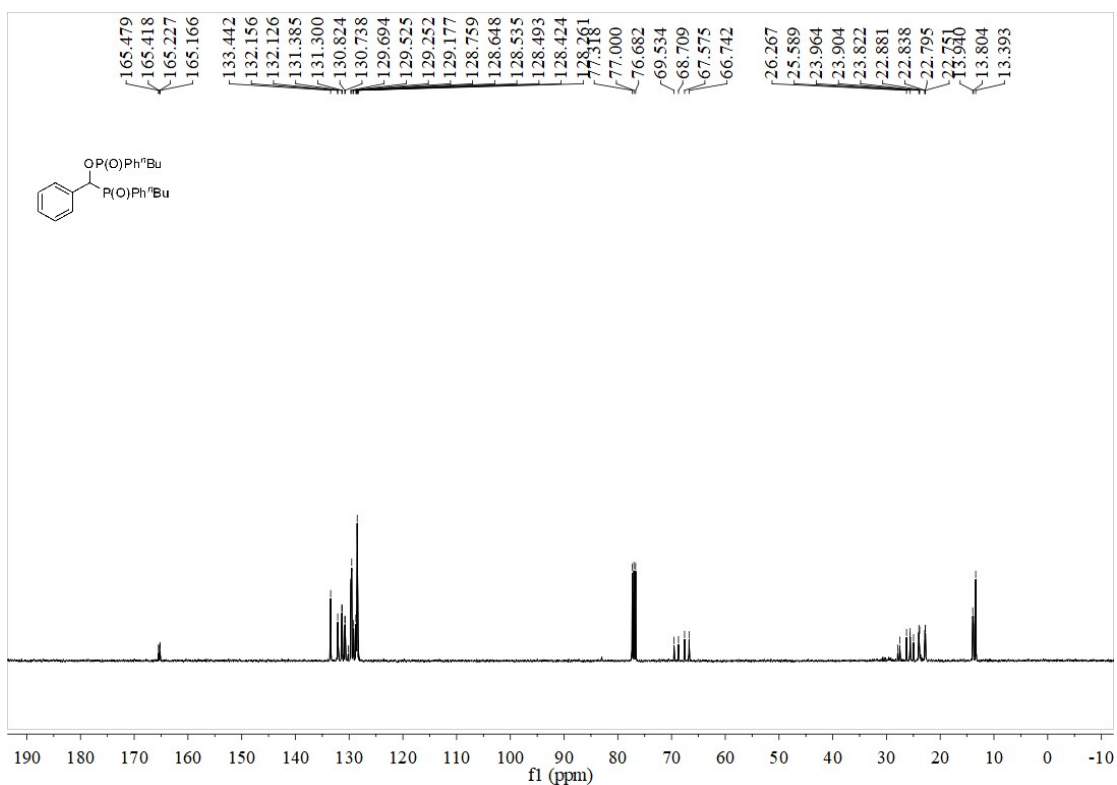
¹³C NMR spectra of (bis(4-fluorophenyl)phosphoryl)(phenyl)methyl bis(4-fluorophenyl)phosphinate (55c).



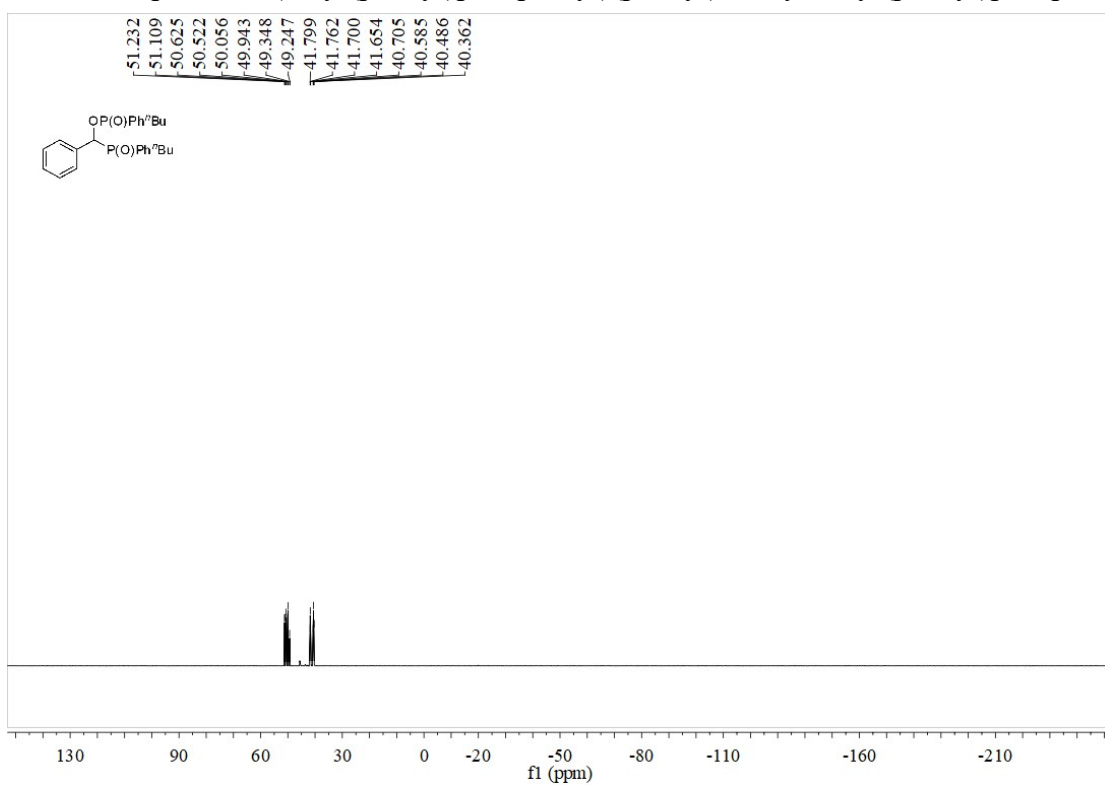
³¹P NMR spectra of (bis(4-fluorophenyl)phosphoryl)(phenyl)methyl bis(4-fluorophenyl)phosphinate (55c).



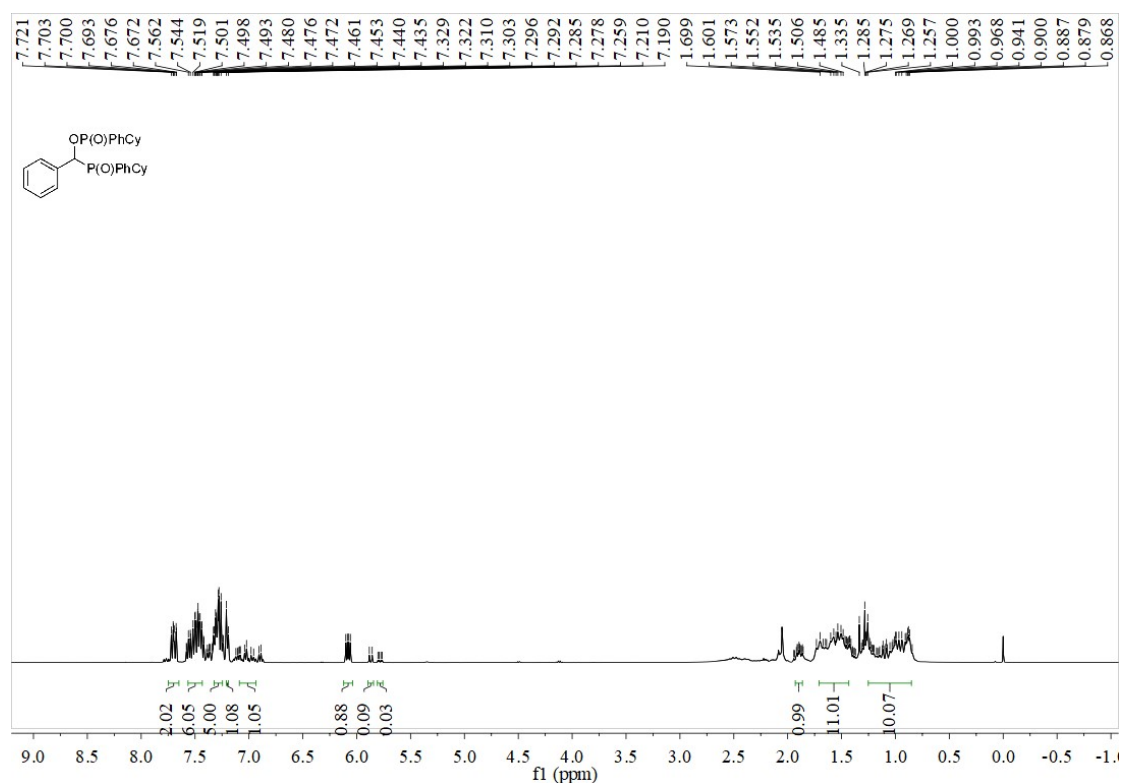
¹H NMR spectra of (butyl(phenyl)phosphoryl)(phenyl)methyl butyl(phenyl)phosphinate (56c).



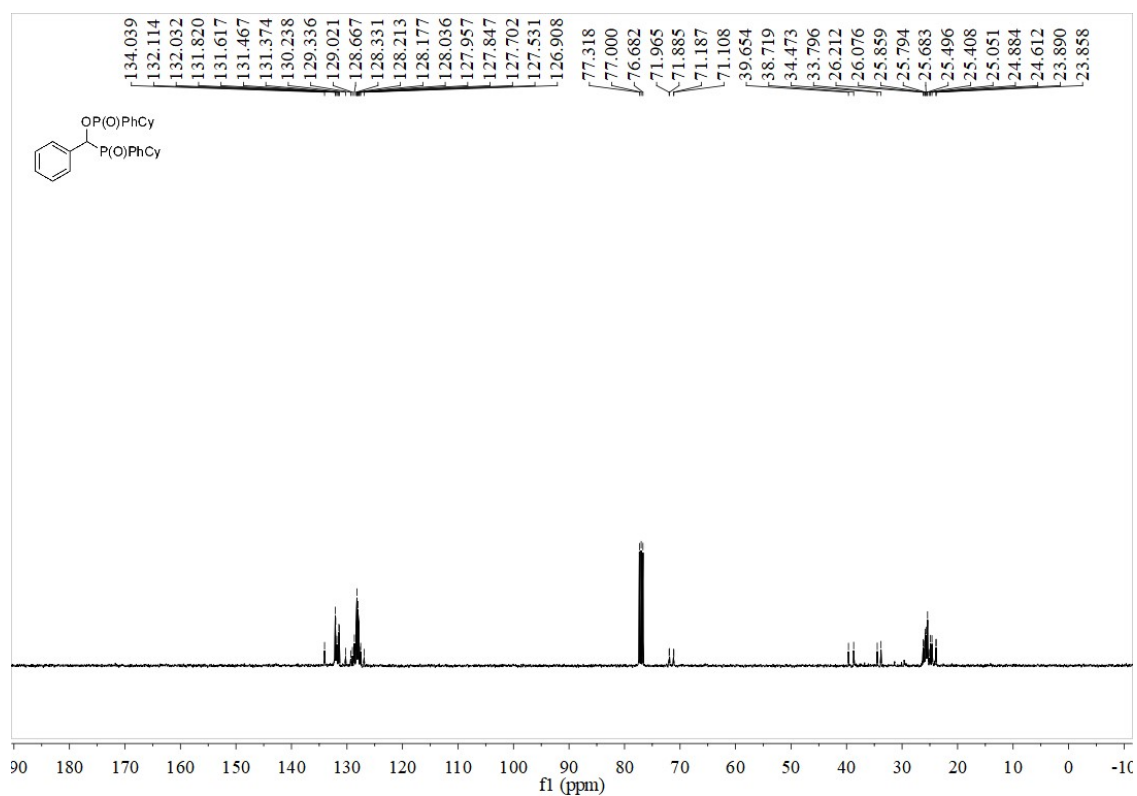
¹³C NMR spectra of (butyl(phenyl)phosphoryl)(phenyl)methyl butyl(phenyl)phosphinate (**56c**).



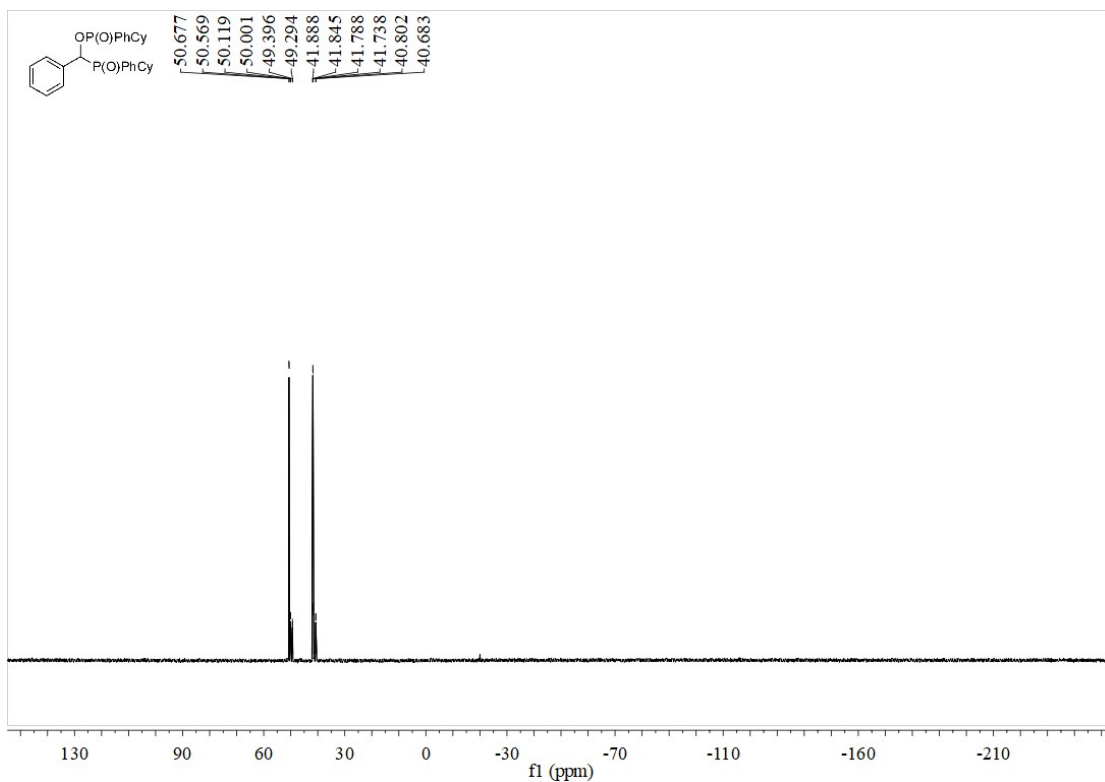
³¹P NMR spectra of (butyl(phenyl)phosphoryl)(phenyl)methyl butyl(phenyl)phosphinate (**56c**).



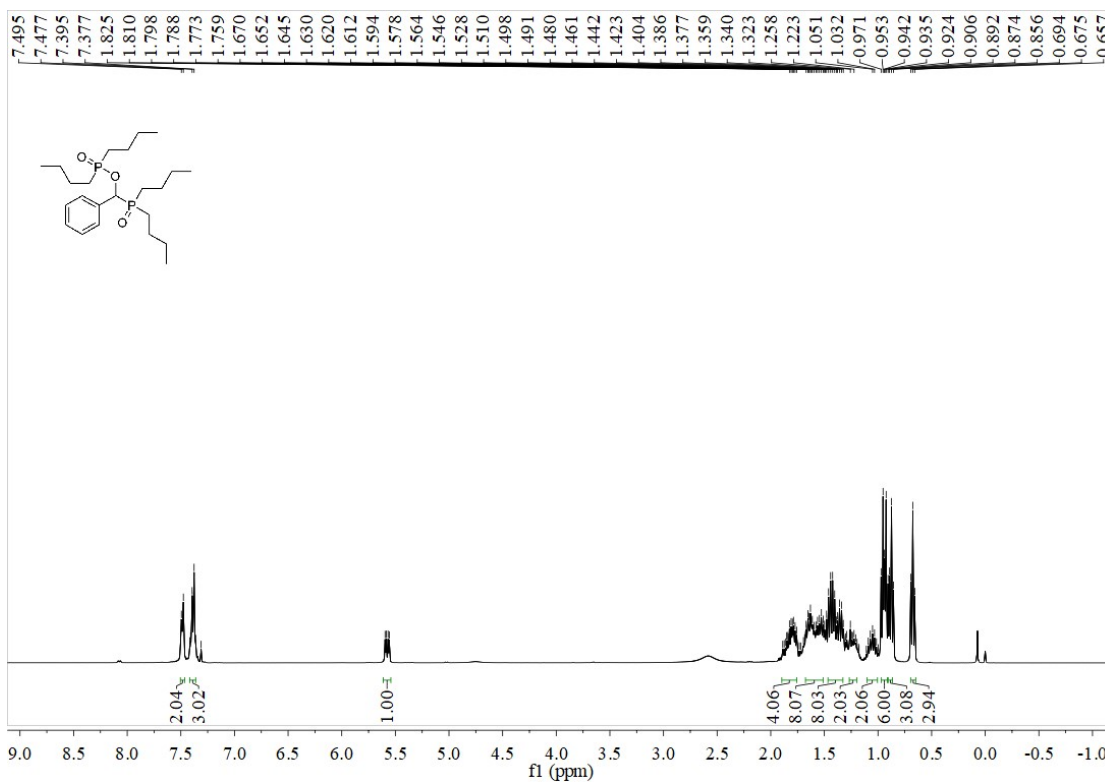
¹H NMR spectra of (cyclohexyl(phenyl)phosphoryl)(phenyl)methyl cyclohexyl(phenyl)phosphinate (57c).



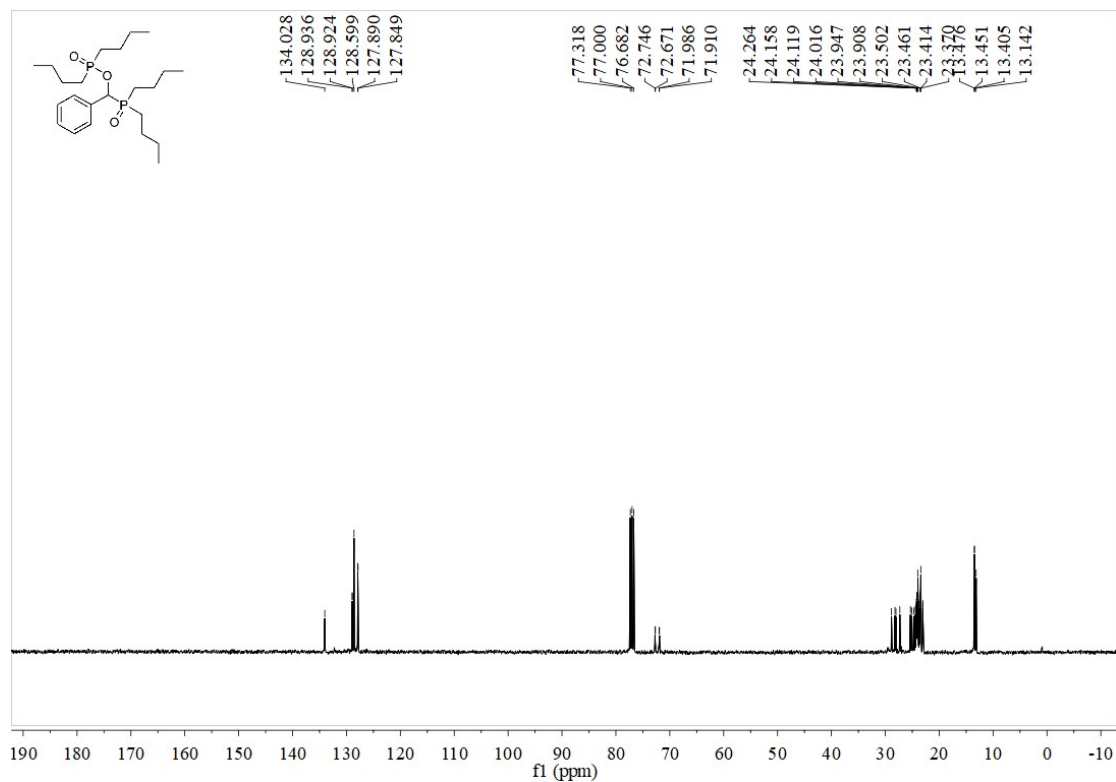
¹³C NMR spectra of (cyclohexyl(phenyl)phosphoryl)(phenyl)methyl cyclohexyl(phenyl)phosphinate (57c).



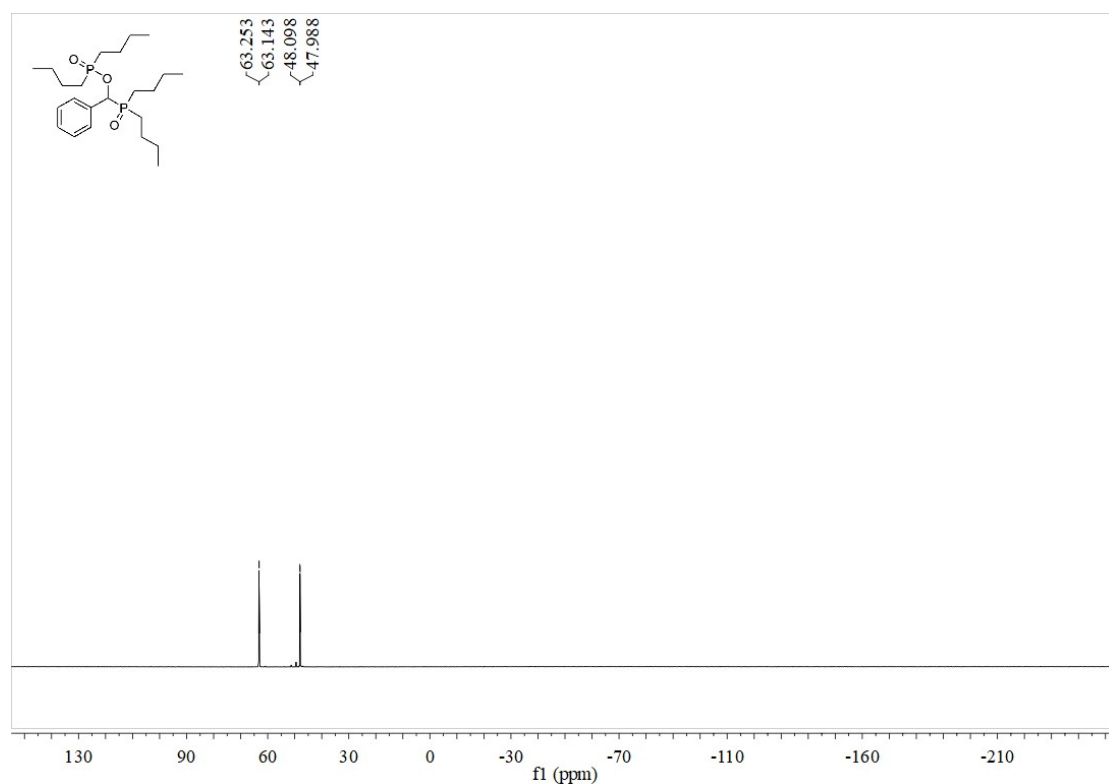
³¹P NMR spectra of (cyclohexyl(phenyl)phosphoryl)(phenyl)methyl cyclohexyl(phenyl)phosphinate (**57c**).



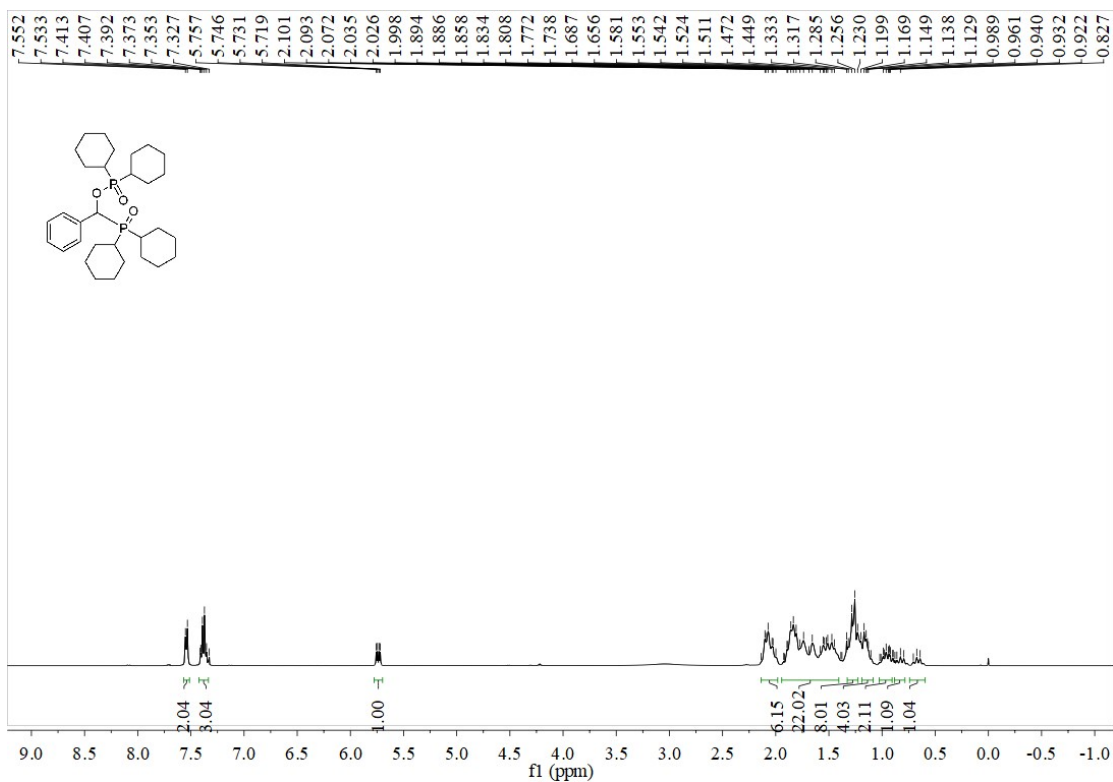
¹H NMR spectra of (dibutylphosphoryl)(phenyl)methyl dibutylphosphinate (**58c**).



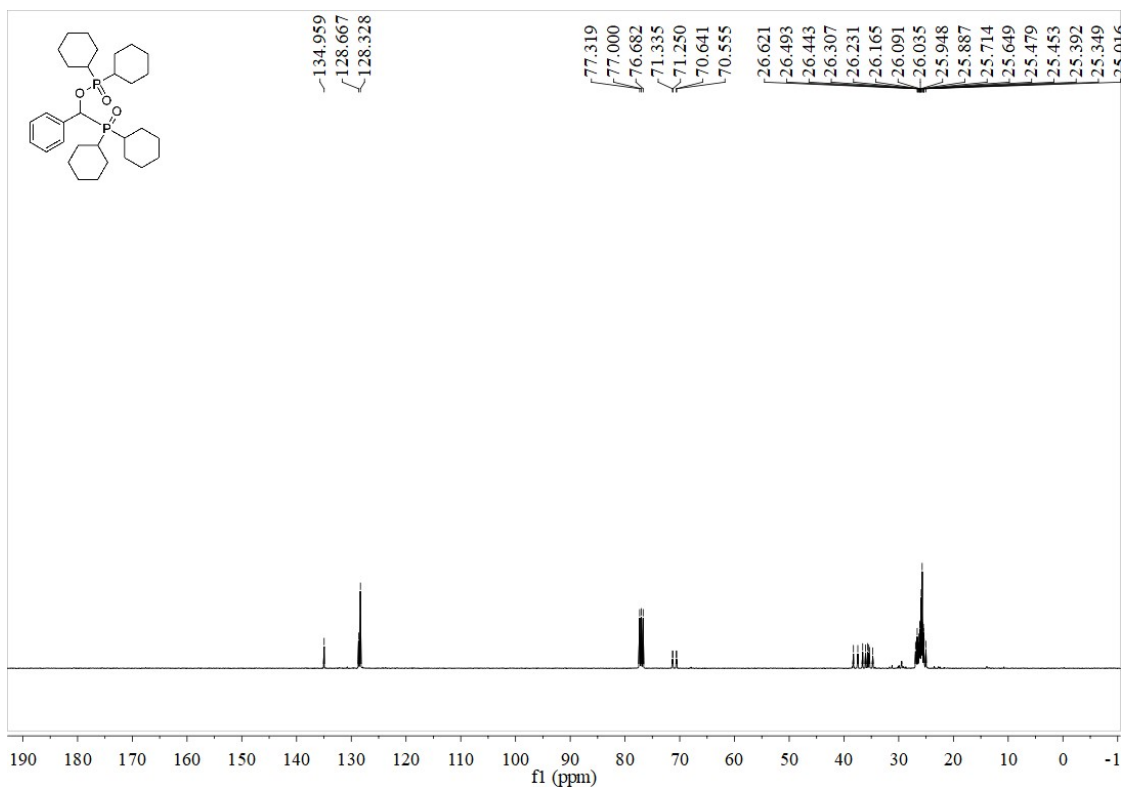
¹³C NMR spectra of (dibutylphosphoryl)(phenyl)methyl dibutylphosphate (**58c**).



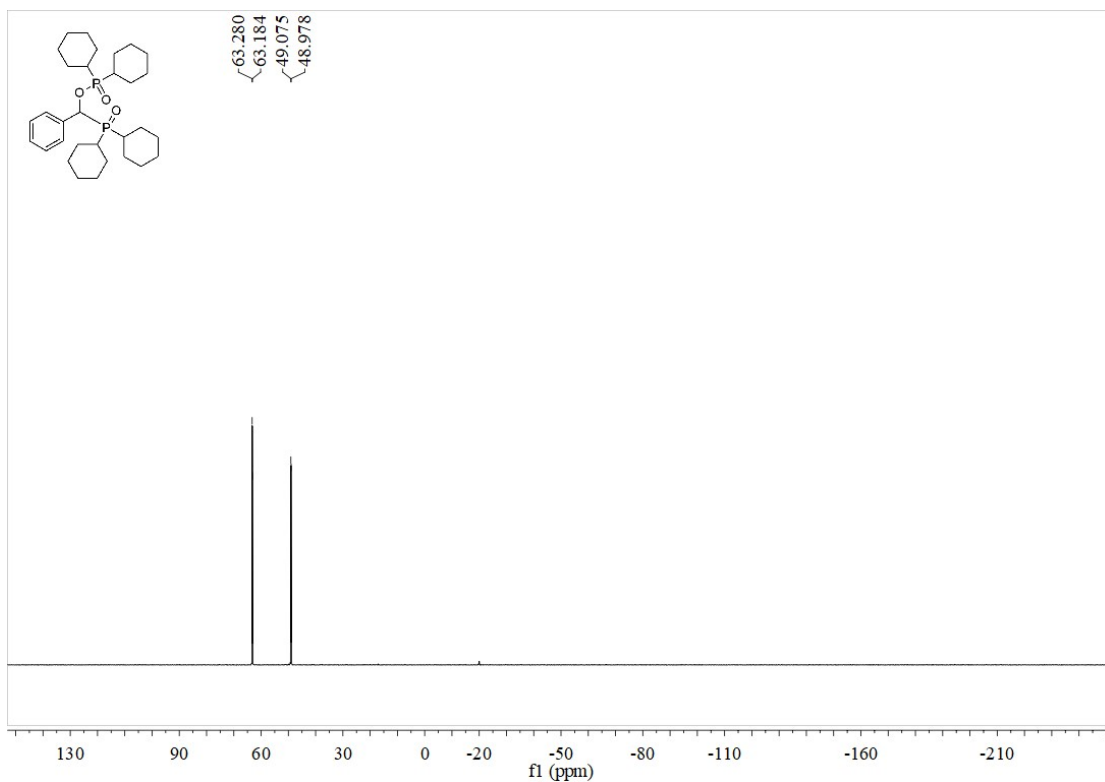
³¹P NMR spectra of (dibutylphosphoryl)(phenyl)methyl dibutylphosphate (**58c**).



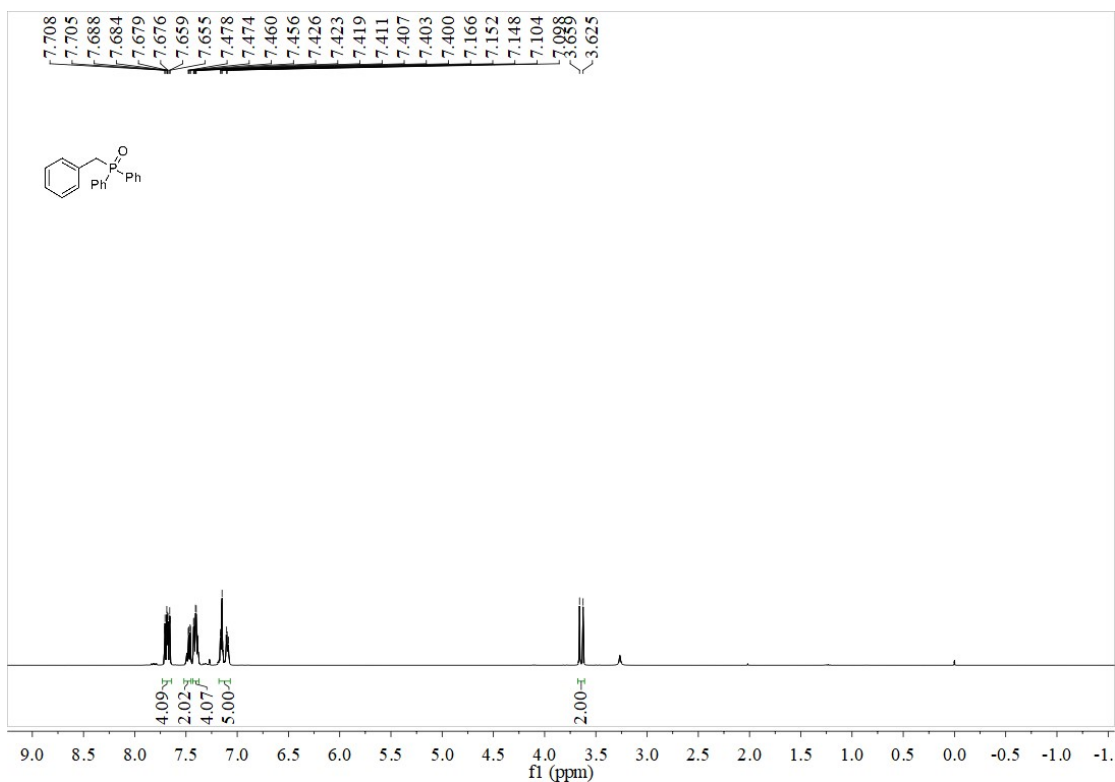
¹H NMR spectra of (dicyclohexylphosphoryl)(phenyl)methyl dicyclohexylphosphinate (**59c**).



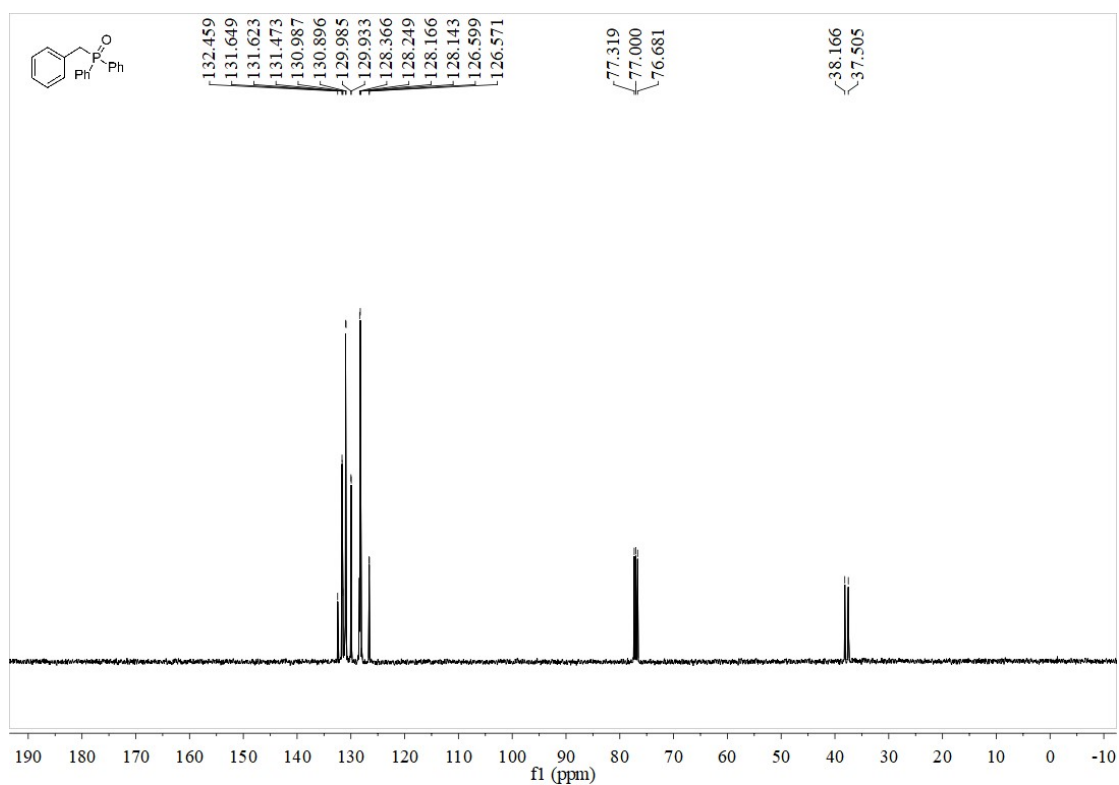
¹³C NMR spectra of (dicyclohexylphosphoryl)(phenyl)methyl dicyclohexylphosphinate (**59c**).



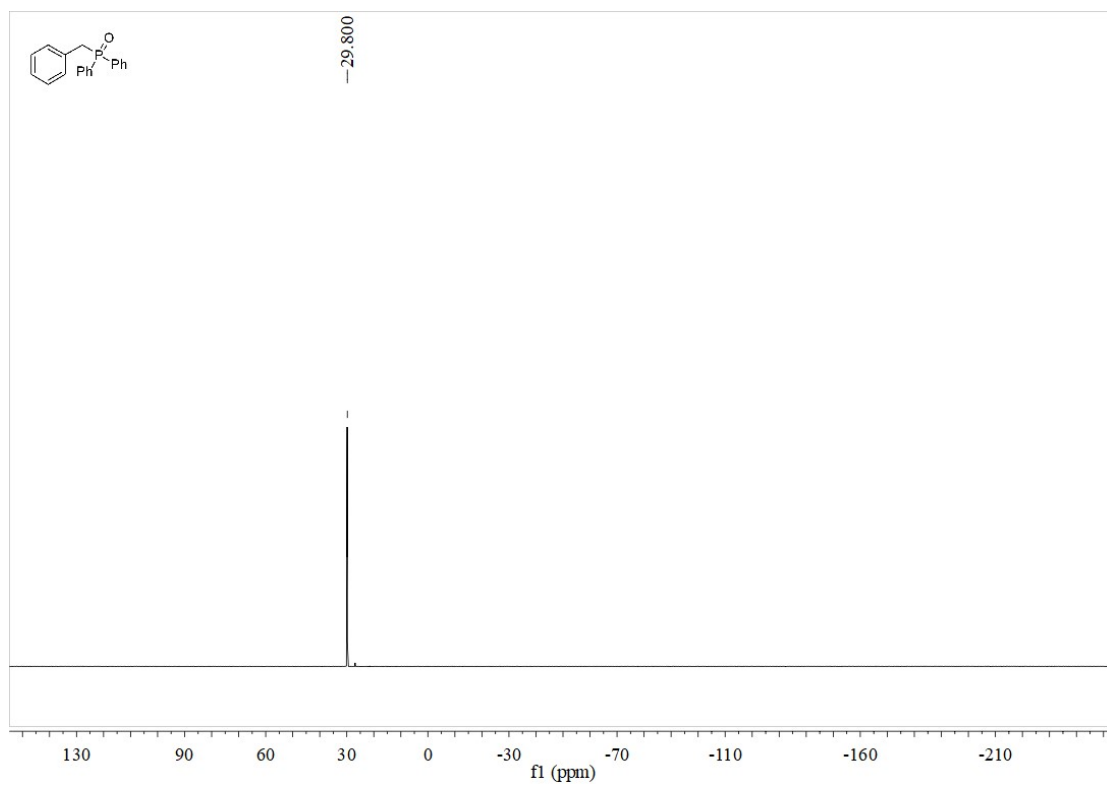
³¹P NMR spectra of (dicyclohexylphosphoryl)(phenyl)methyl dicyclohexylphosphinate (**59c**).



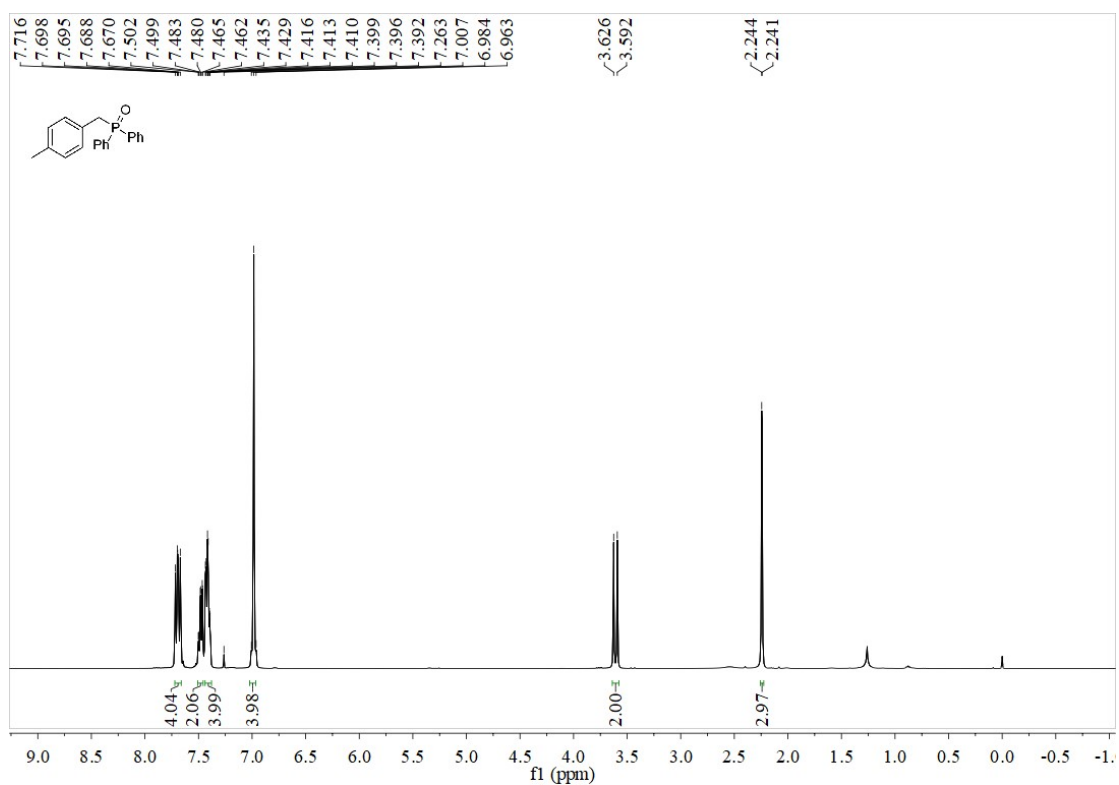
¹H NMR spectra of Benzylidene diphenylphosphine oxide (**1d**).



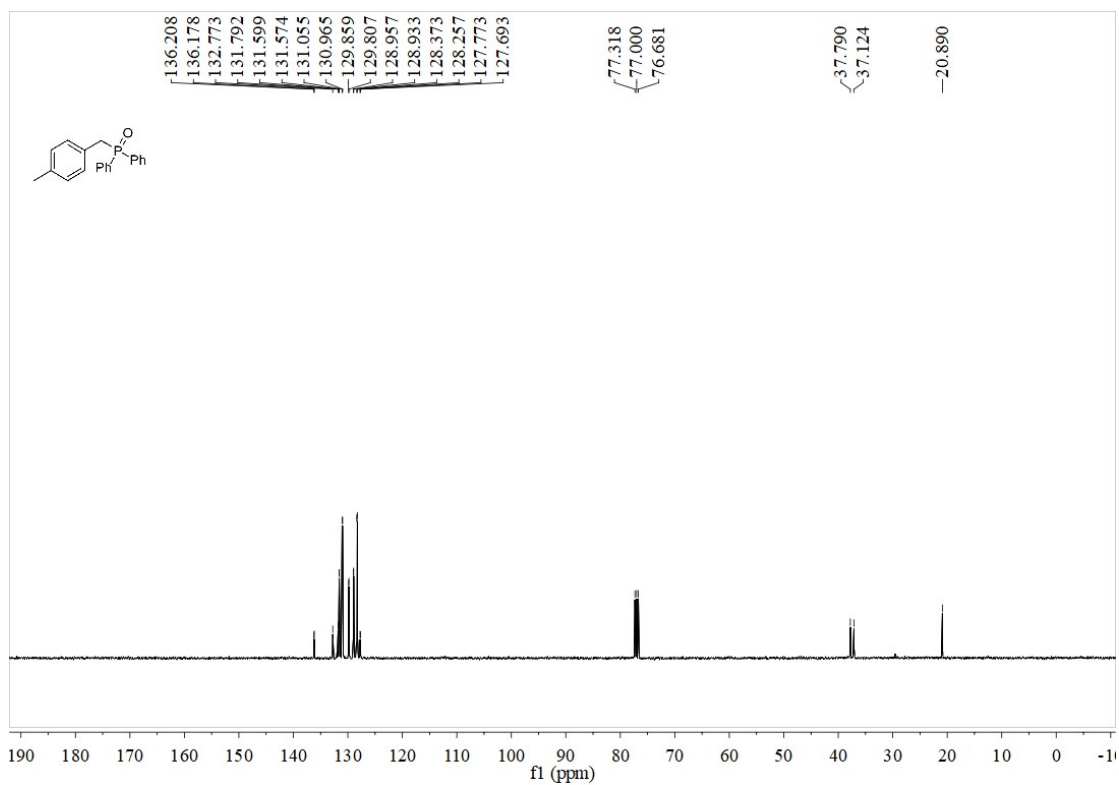
¹³C NMR spectra of Benzylidiphenylphosphine oxide (**1d**).



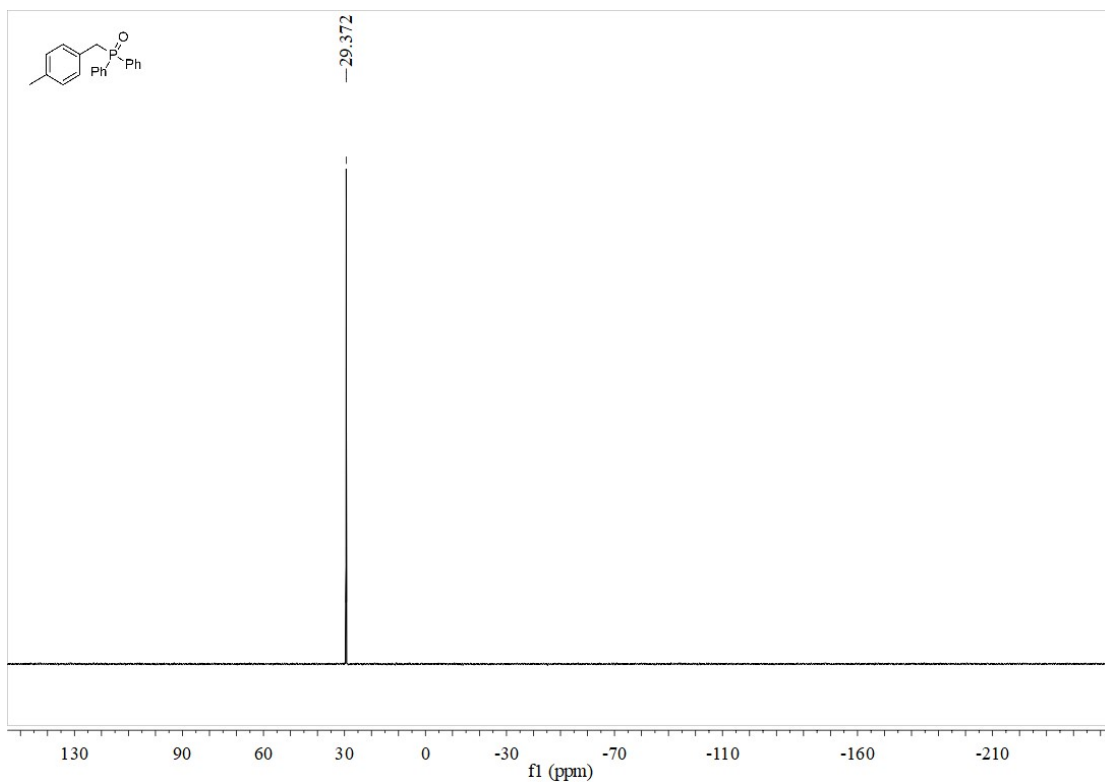
³¹P NMR spectra of Benzylidiphenylphosphine oxide (**1d**).



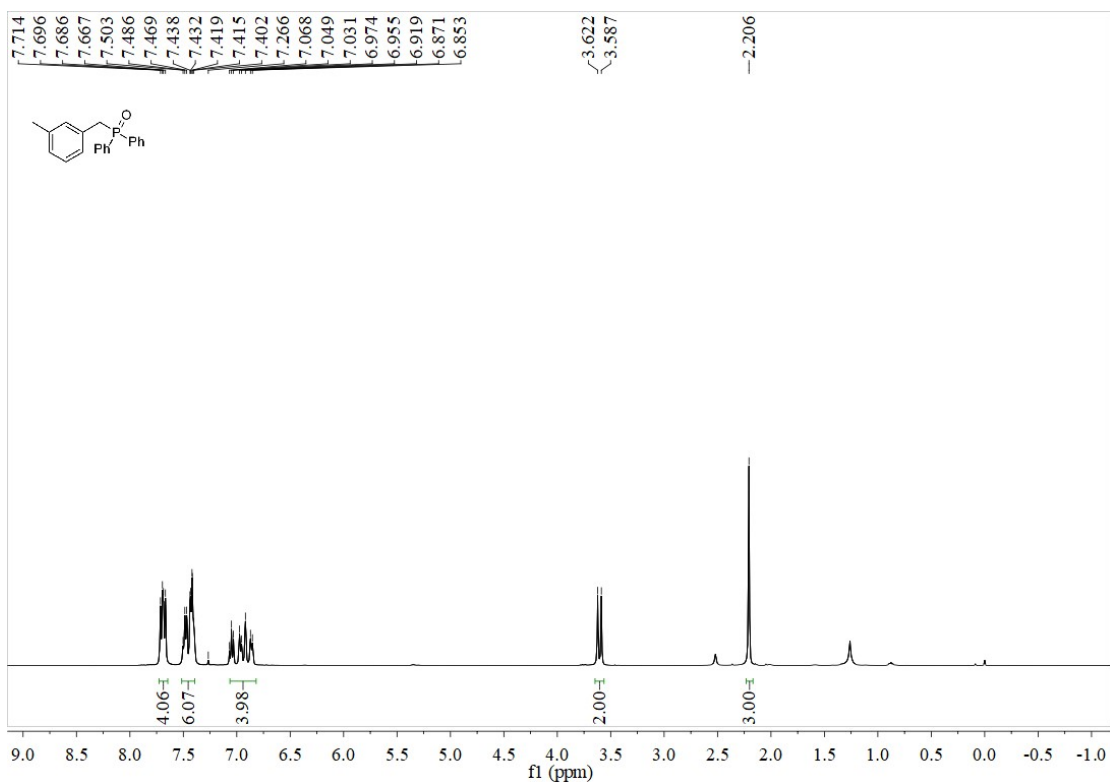
¹H NMR spectra of (4-methylbenzyl)diphenylphosphine oxide (**2d**).



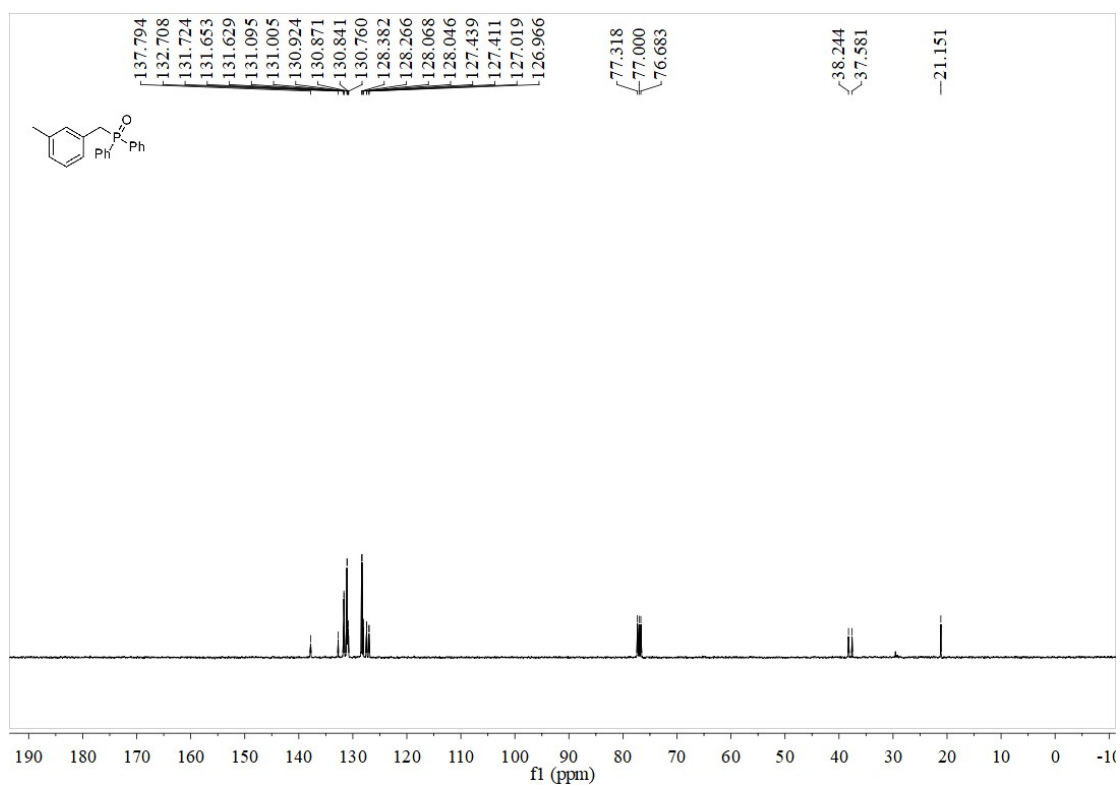
¹³C NMR spectra of (4-methylbenzyl)diphenylphosphine oxide (**2d**).



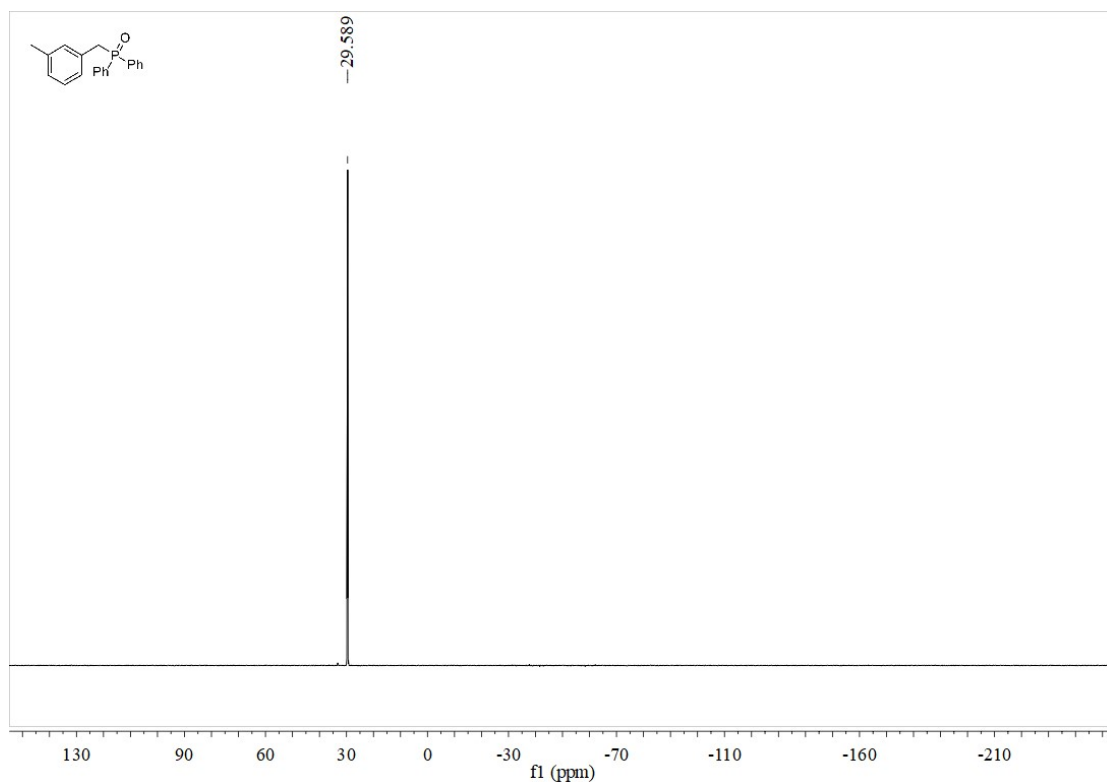
^{31}P NMR spectra of (4-methylbenzyl)diphenylphosphine oxide (**2d**).



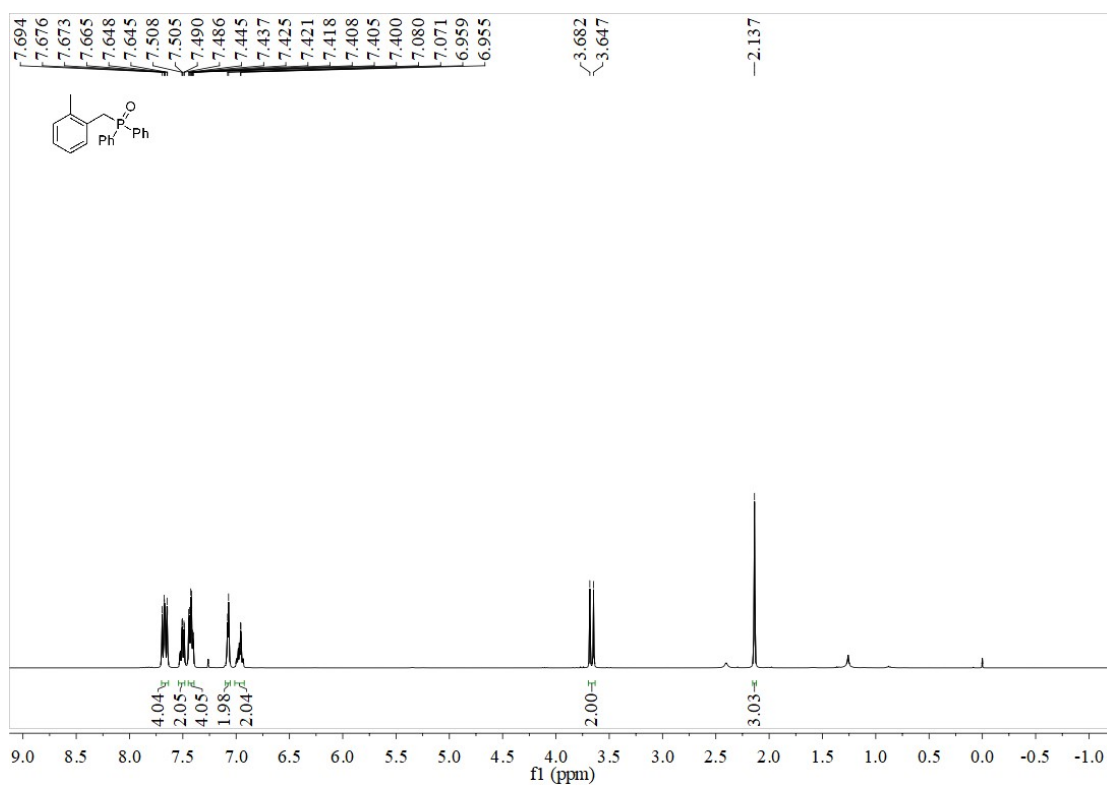
^1H NMR spectra of (3-methylbenzyl)diphenylphosphine oxide (**3d**).



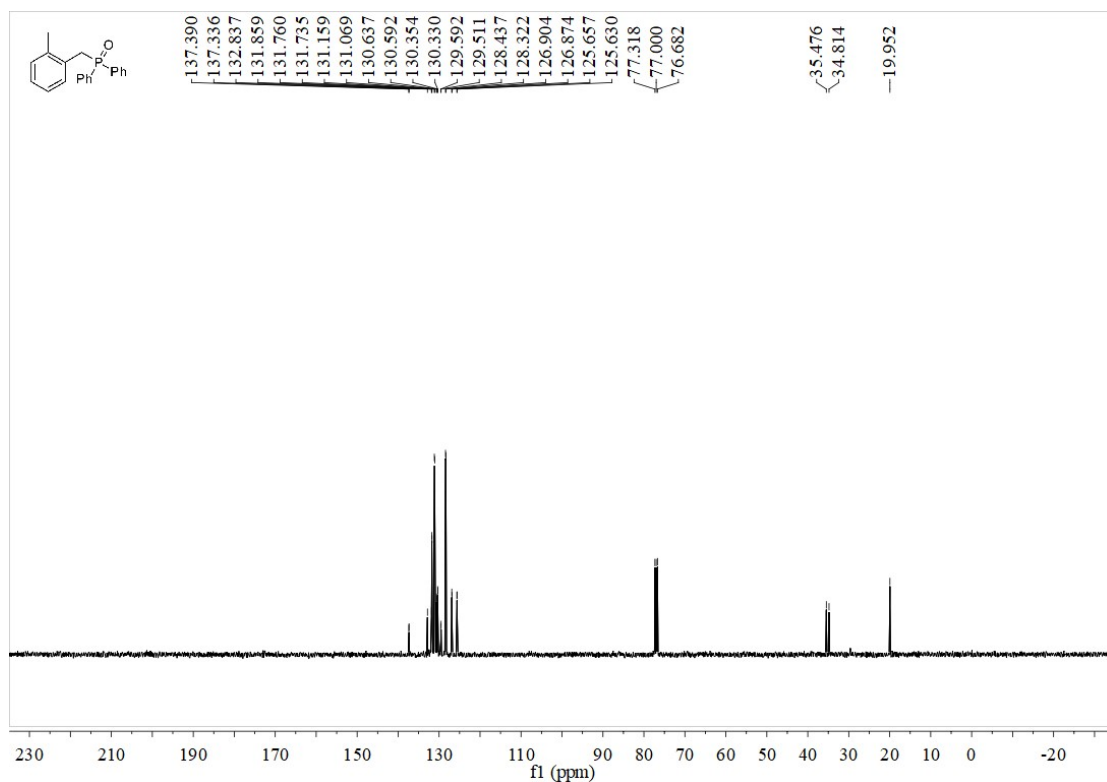
^{13}C NMR spectra of (3-methylbenzyl)diphenylphosphine oxide (**3d**).



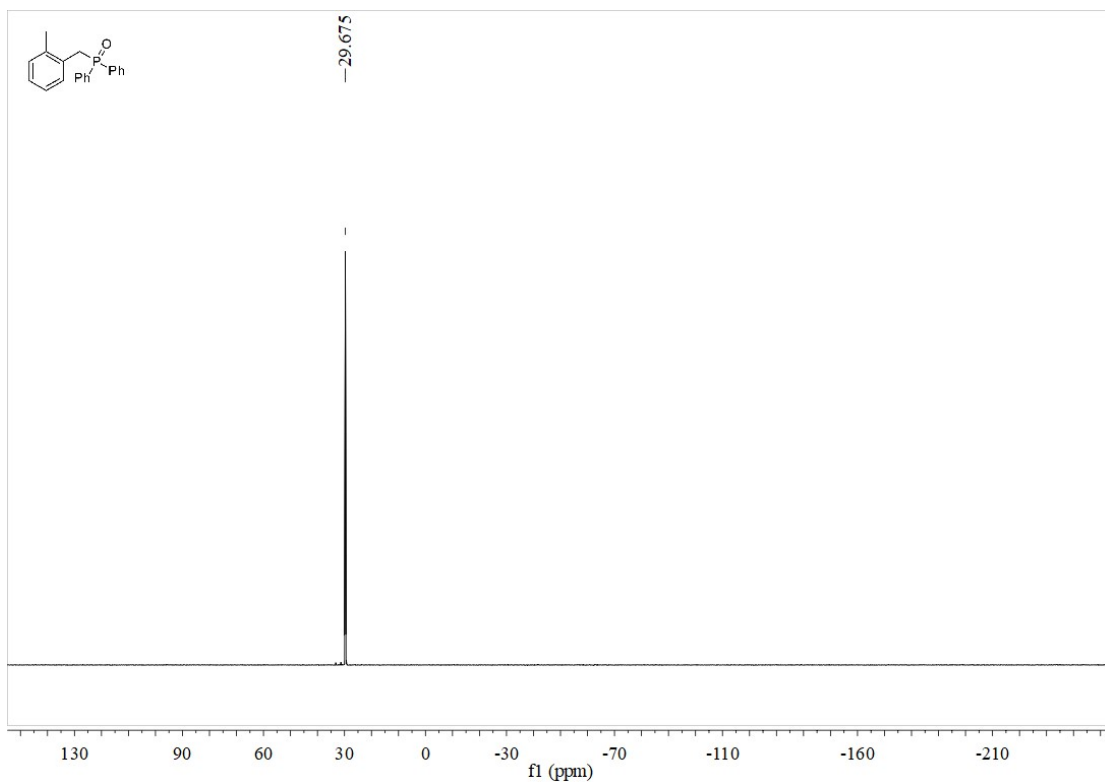
^{31}P NMR spectra of (3-methylbenzyl)diphenylphosphine oxide (**3d**).



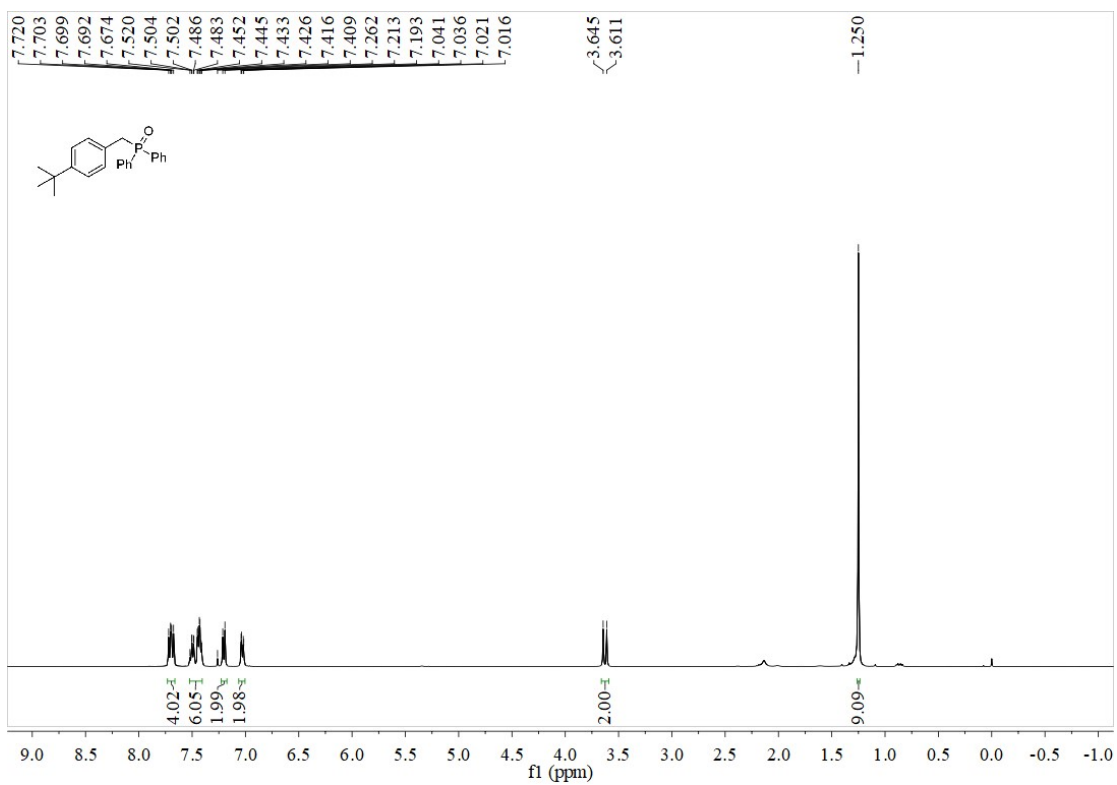
¹H NMR spectra of (2-methylbenzyl)diphenylphosphine oxide (**4d**).



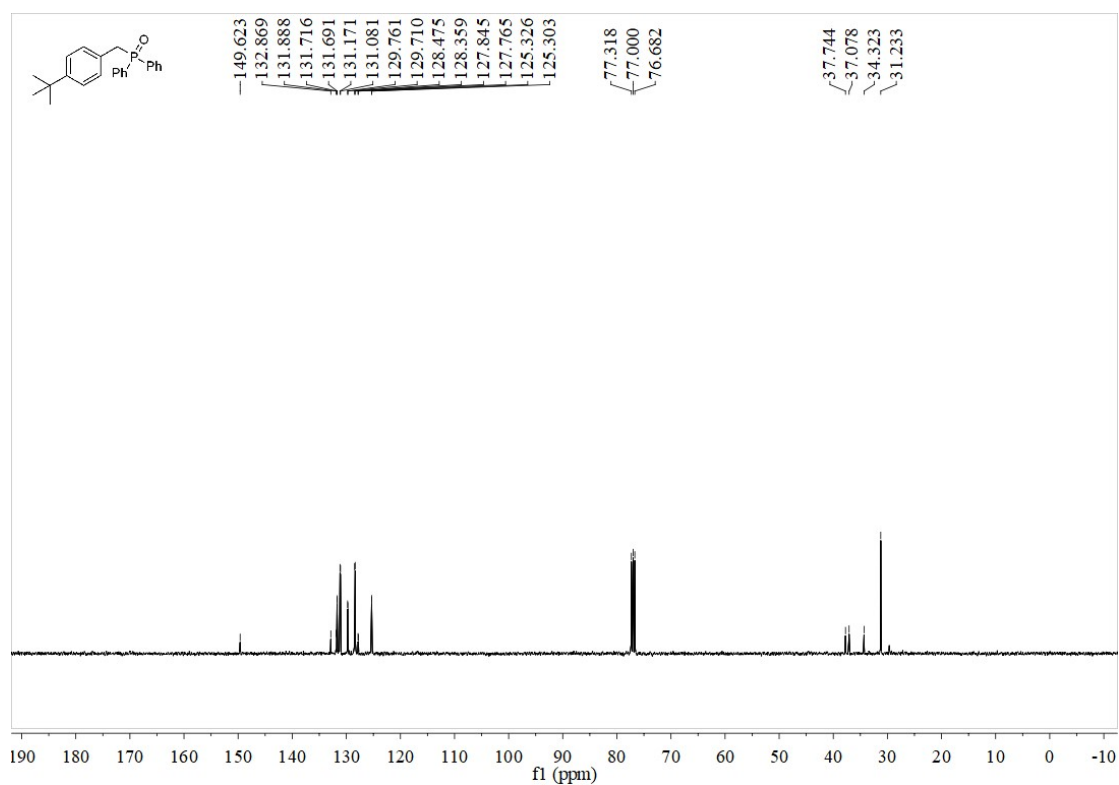
¹³C NMR spectra of (2-methylbenzyl)diphenylphosphine oxide (**4d**).



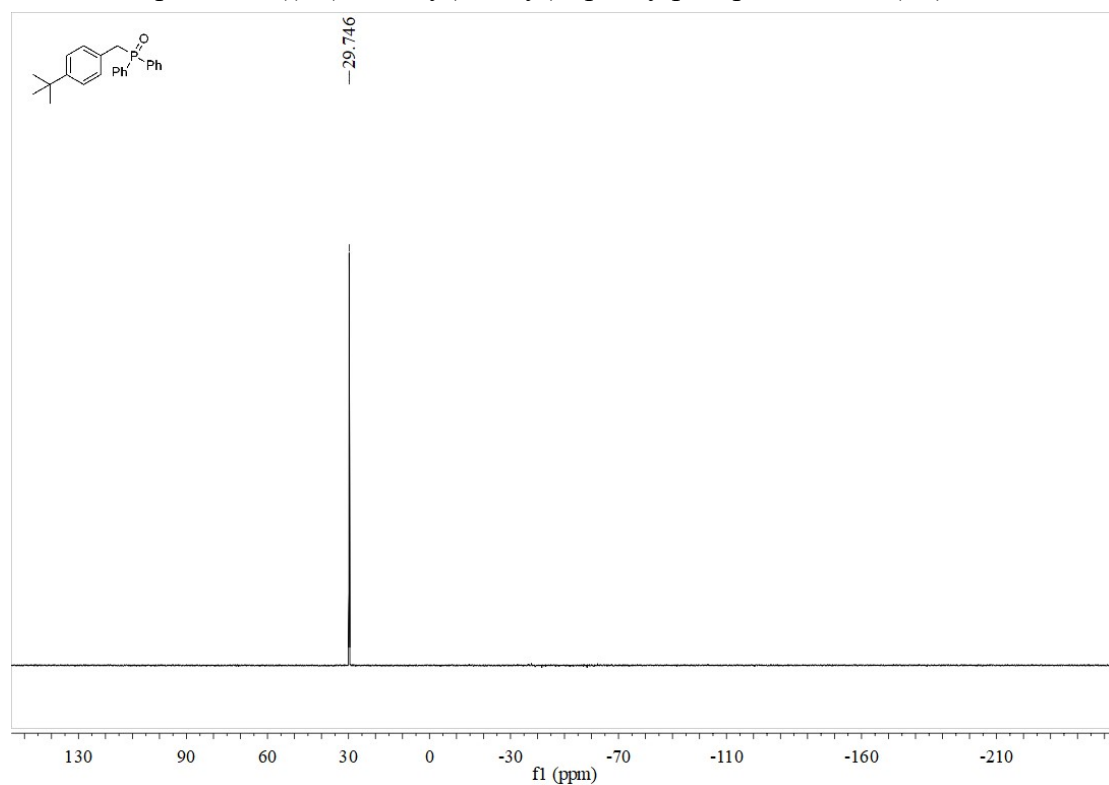
^{31}P NMR spectra of (2-methylbenzyl)diphenylphosphine oxide (**4d**).



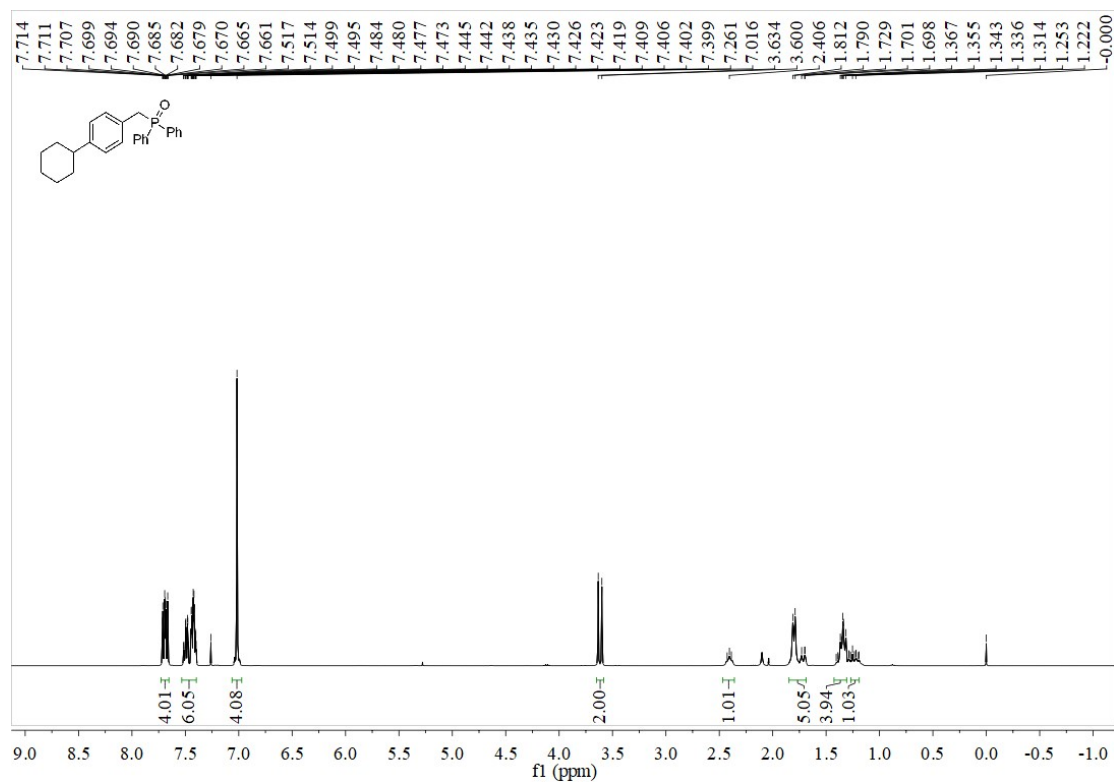
^1H NMR spectra of ((4-(tert-butyl)benzyl)diphenylphosphine oxide (**5d**)).



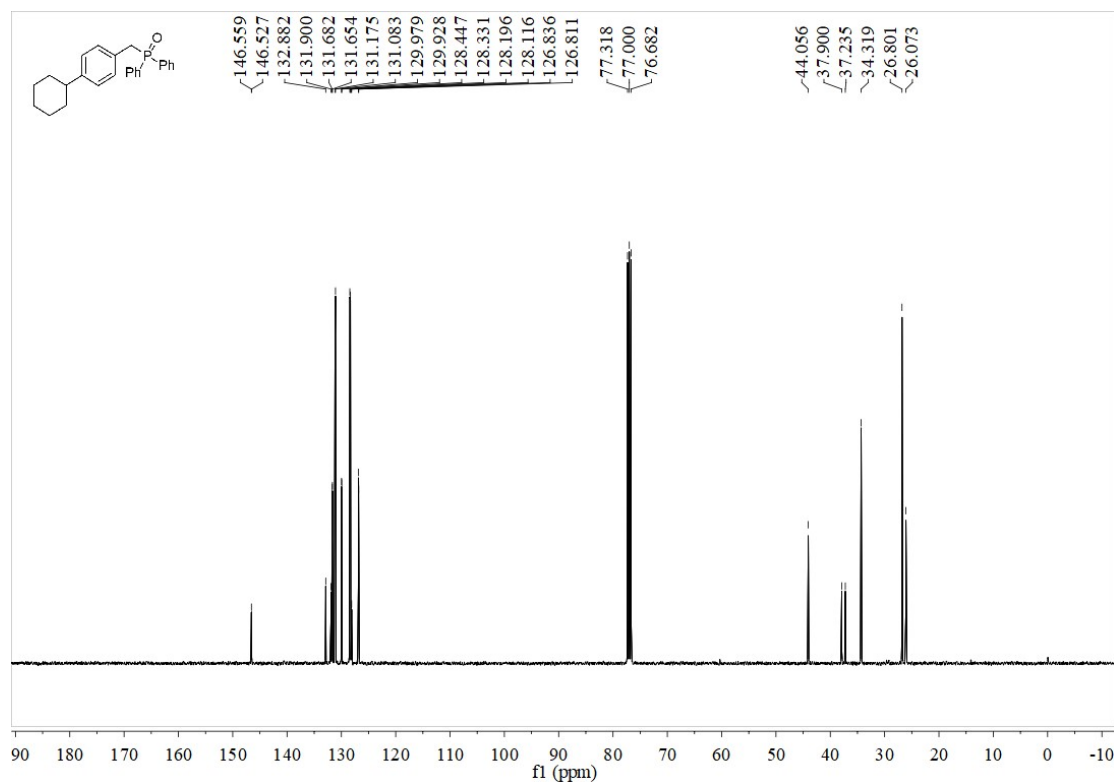
¹³C NMR spectra of ((4-(tert-butyl)benzyl)diphenylphosphine oxide (**5d**)).



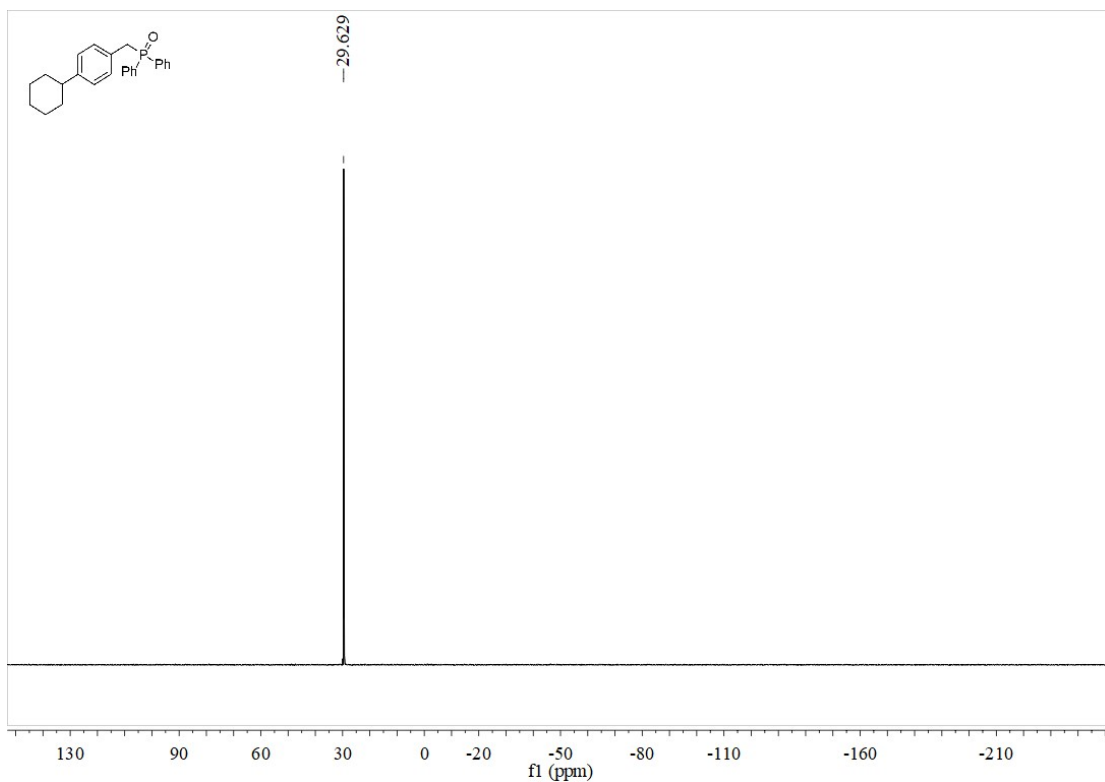
³¹P NMR spectra of ((4-(tert-butyl)benzyl)diphenylphosphine oxide (**5d**)).



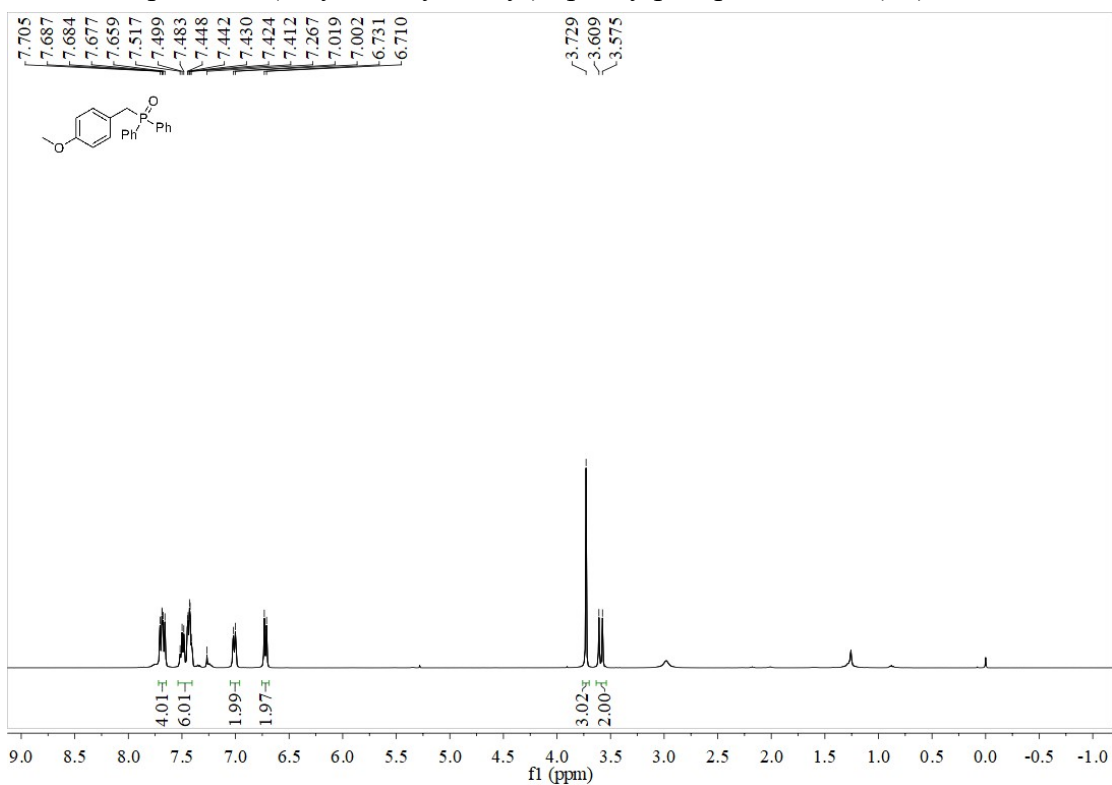
¹H NMR spectra of (4-cyclohexylbenzyl)diphenylphosphine oxide (**6d**).



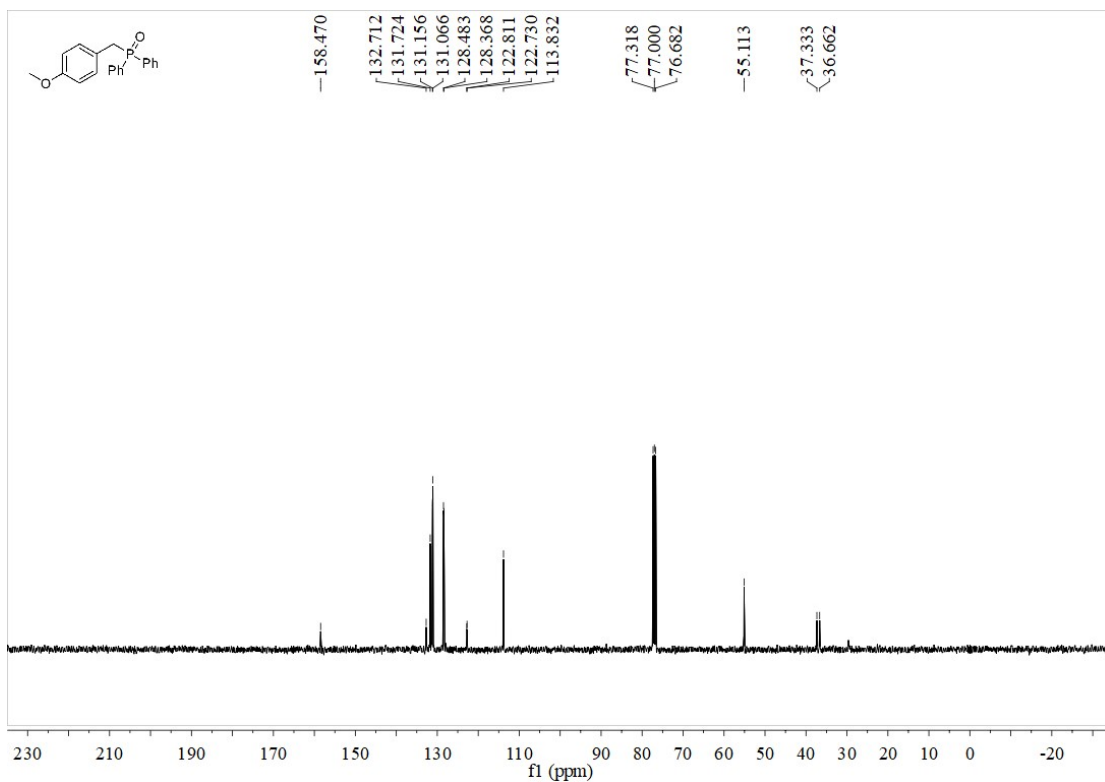
¹³C NMR spectra of (4-cyclohexylbenzyl)diphenylphosphine oxide (**6d**).



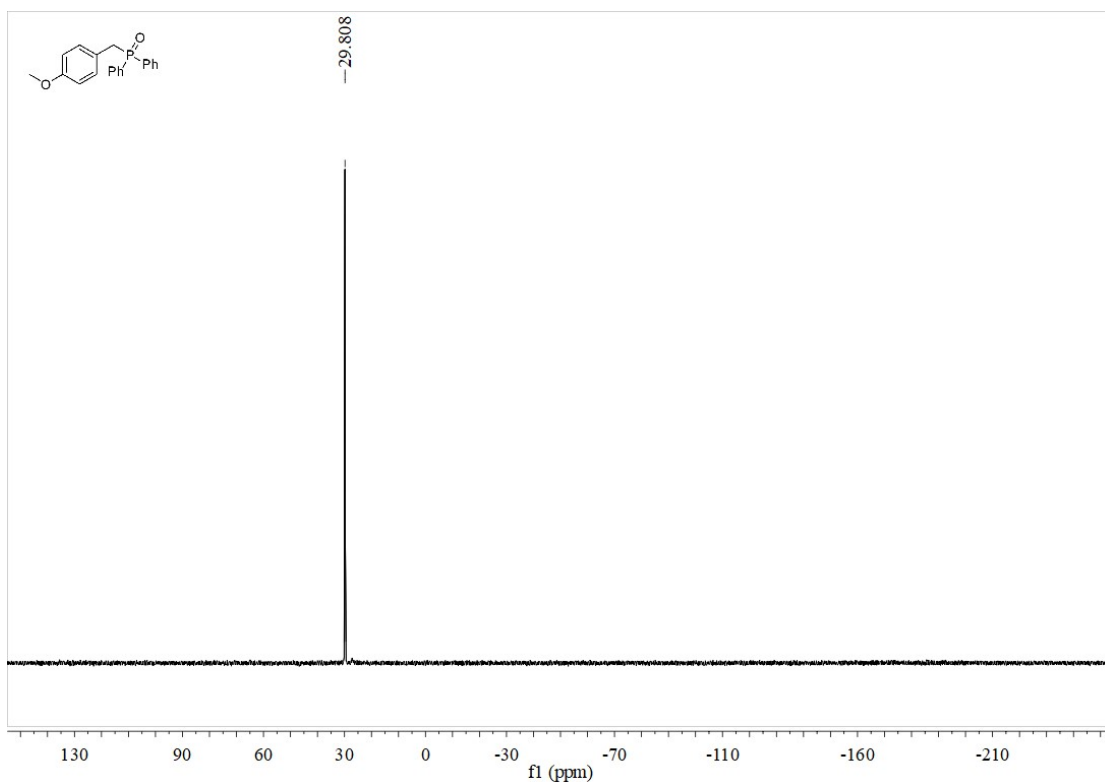
^{31}P NMR spectra of (4-cyclohexylbenzyl)diphenylphosphine oxide (**6d**).



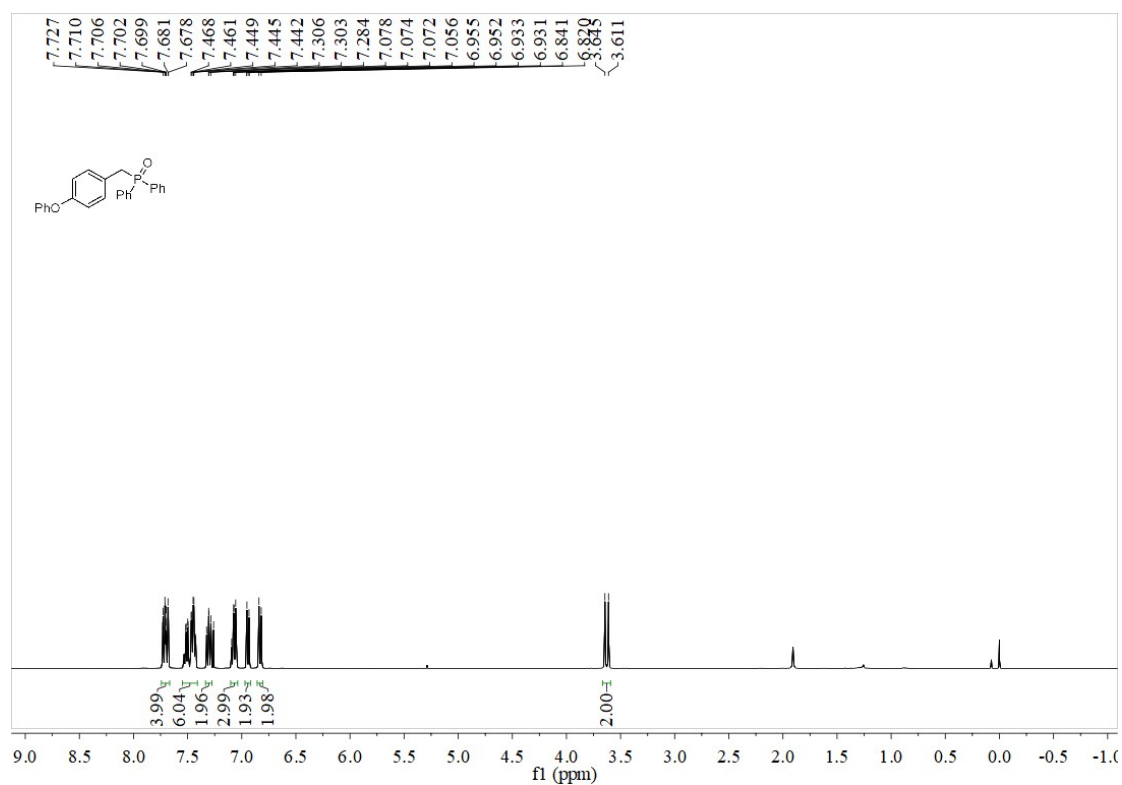
^1H NMR spectra of (4-methoxybenzyl)diphenylphosphine oxide (**7d**).



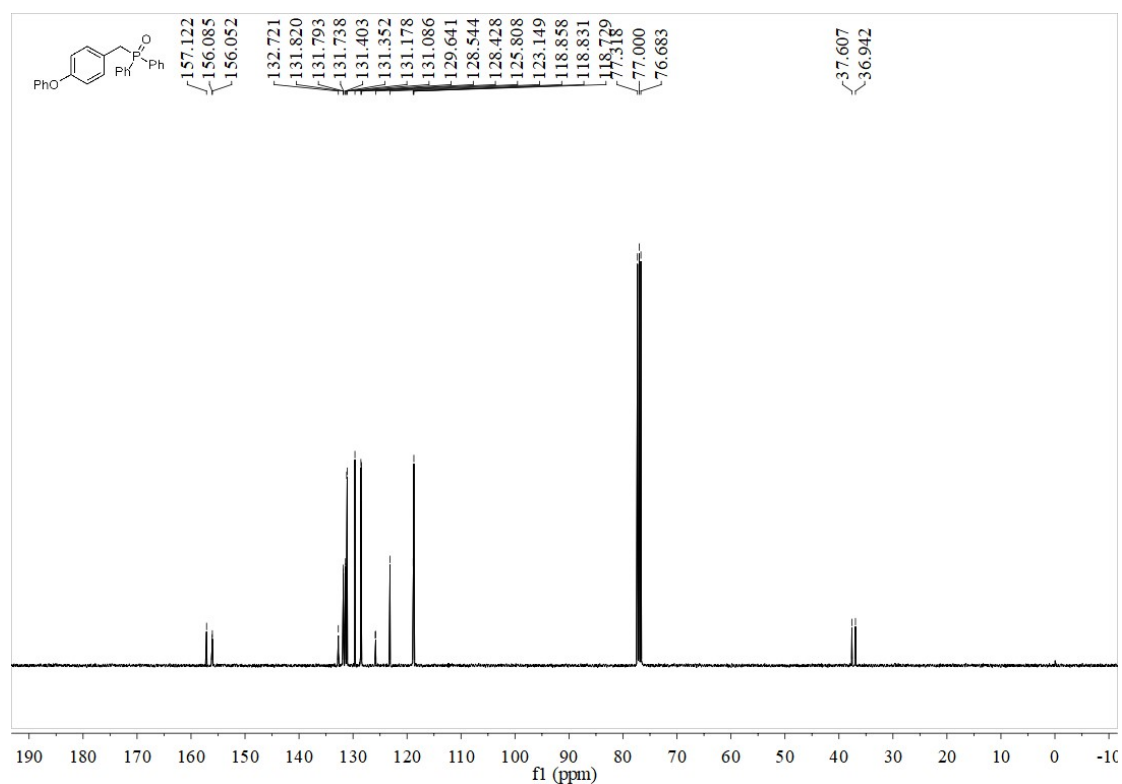
¹³C NMR spectra of (4-methoxybenzyl)diphenylphosphine oxide (**7d**).



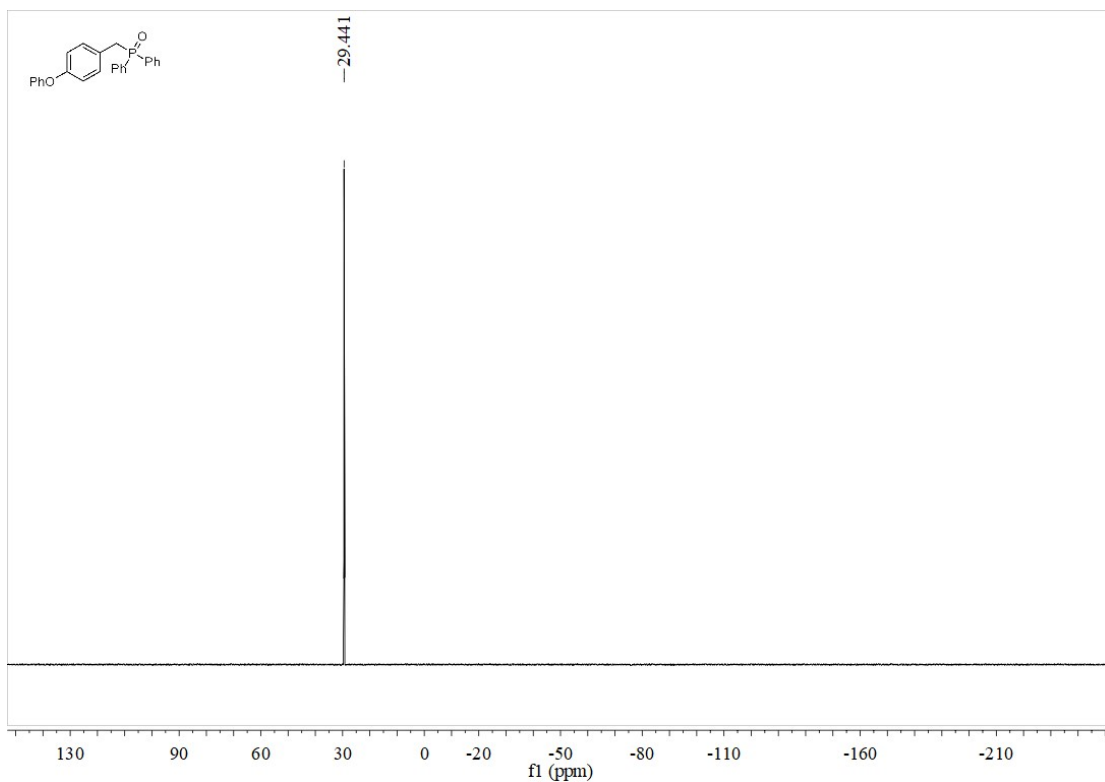
³¹P NMR spectra of (4-methoxybenzyl)diphenylphosphine oxide (**7d**).



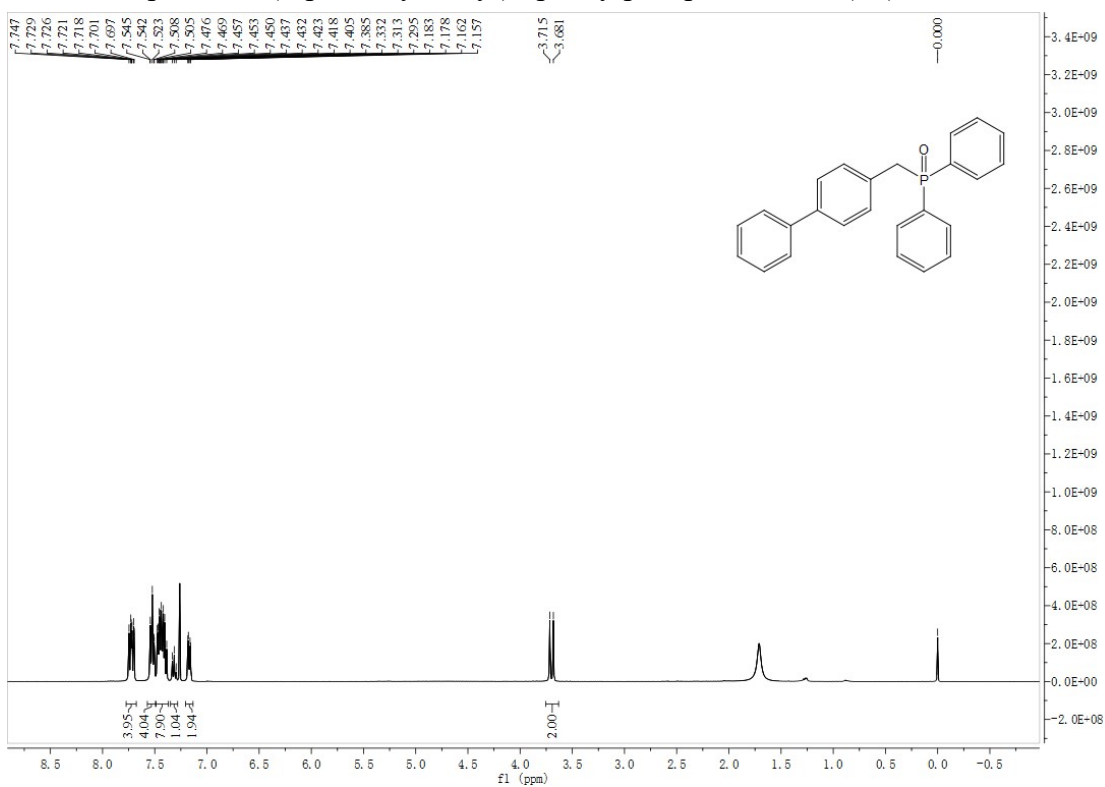
¹H NMR spectra of (4-phenoxybenzyl)diphenylphosphine oxide (**8d**).



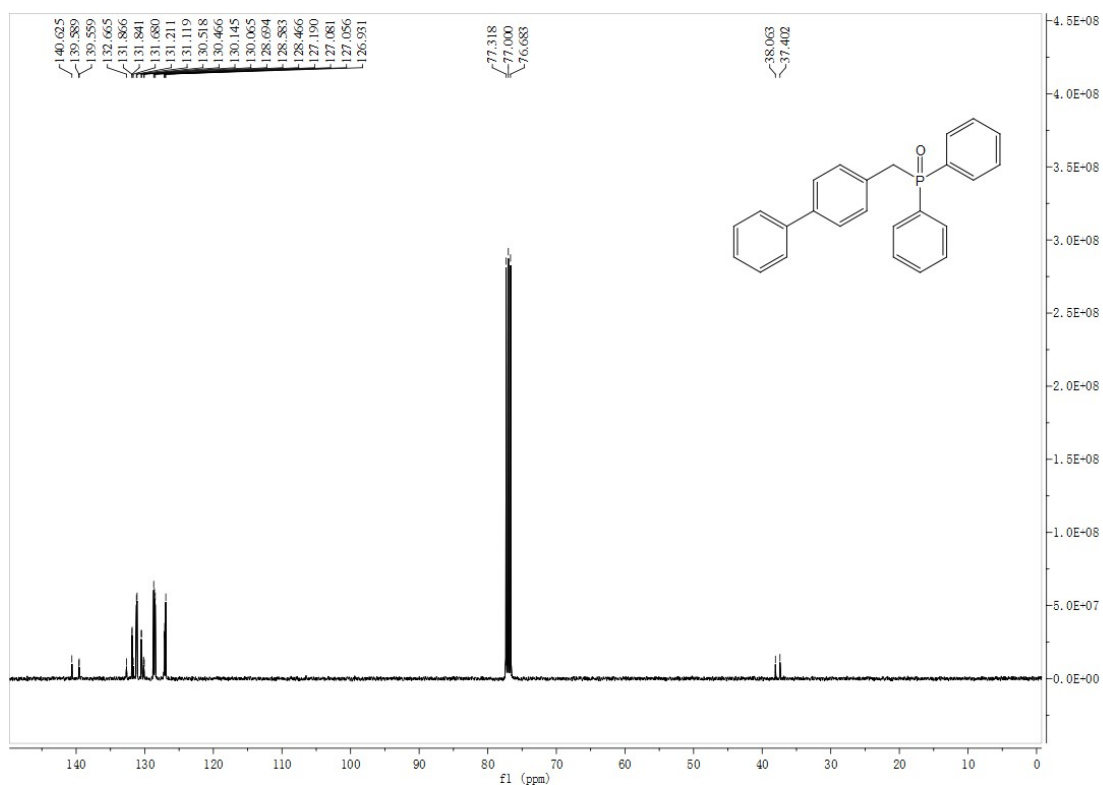
¹³C NMR spectra of (4-phenoxybenzyl)diphenylphosphine oxide (**8d**).



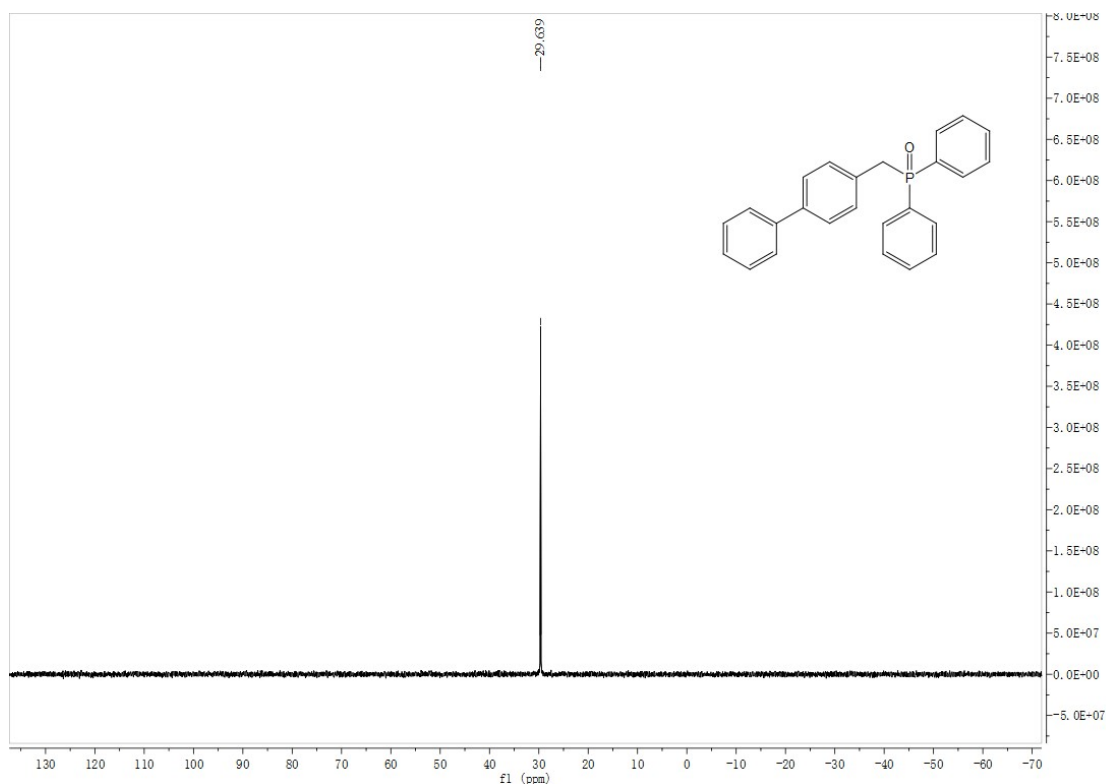
^{31}P NMR spectra of (4-phenoxybenzyl)diphenylphosphine oxide (**8d**).



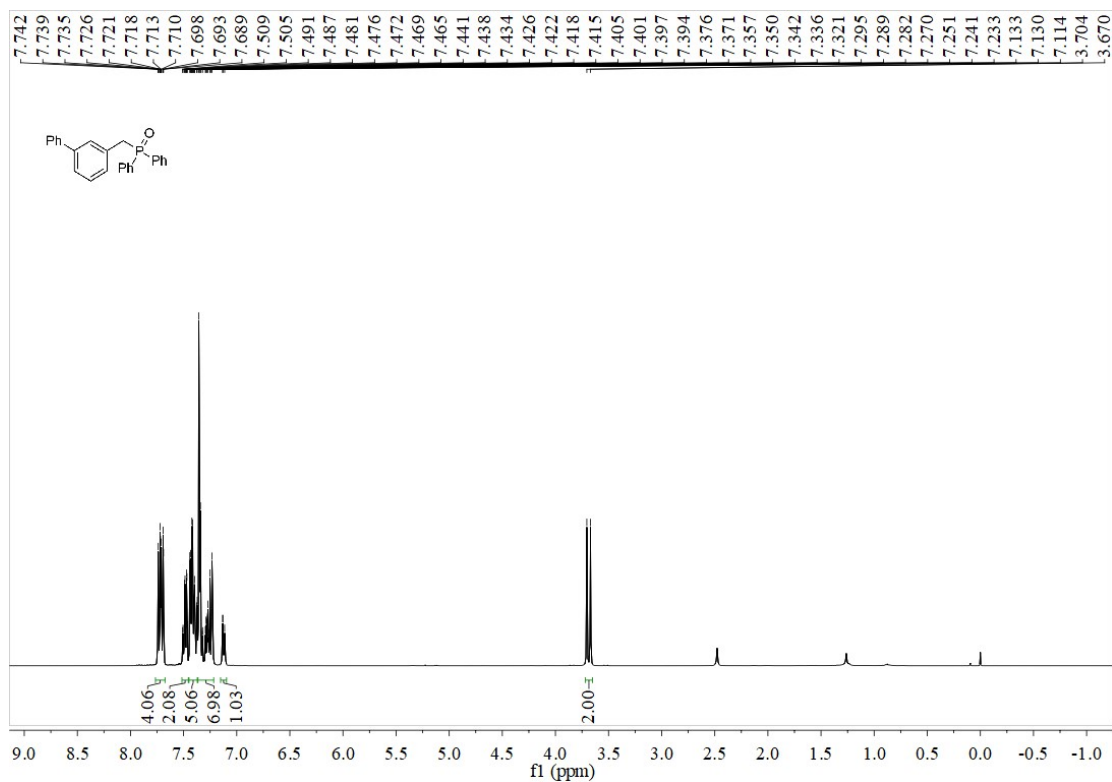
^1H NMR spectra of ([1,1'-biphenyl]-4-ylmethyl)diphenylphosphine oxide (**9d**).



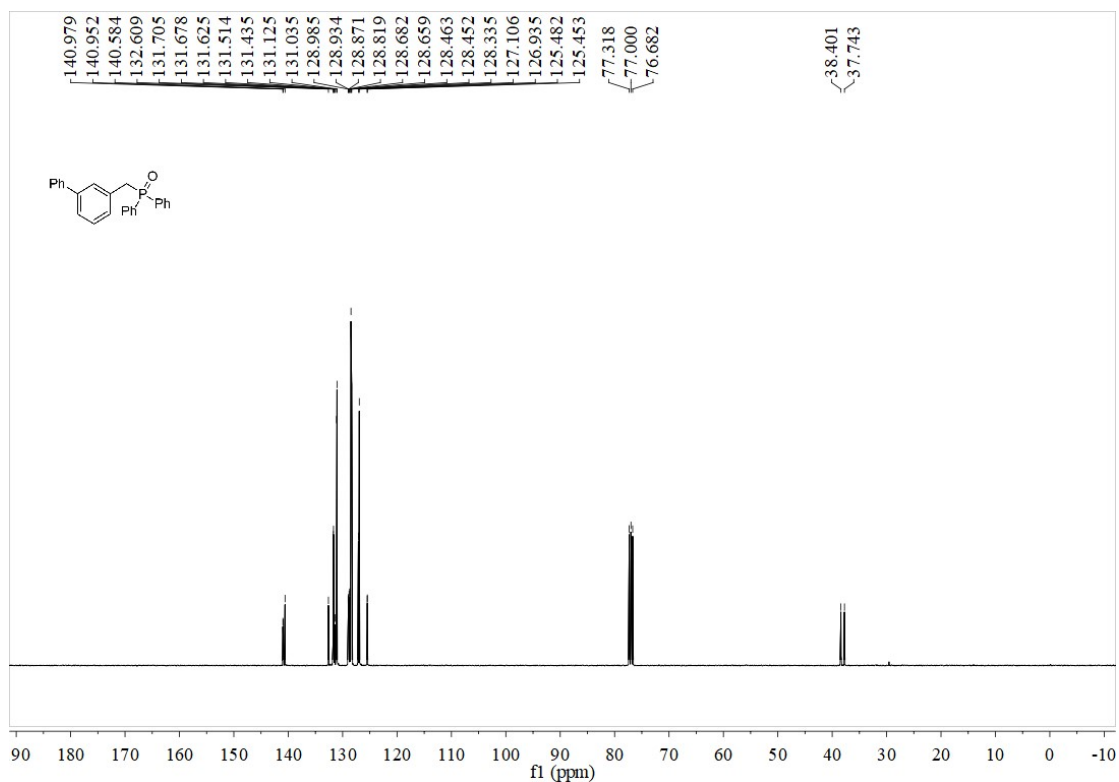
¹³C NMR spectra of ([1,1'-biphenyl]-4-ylmethyl)diphenylphosphine oxide (**9d**).



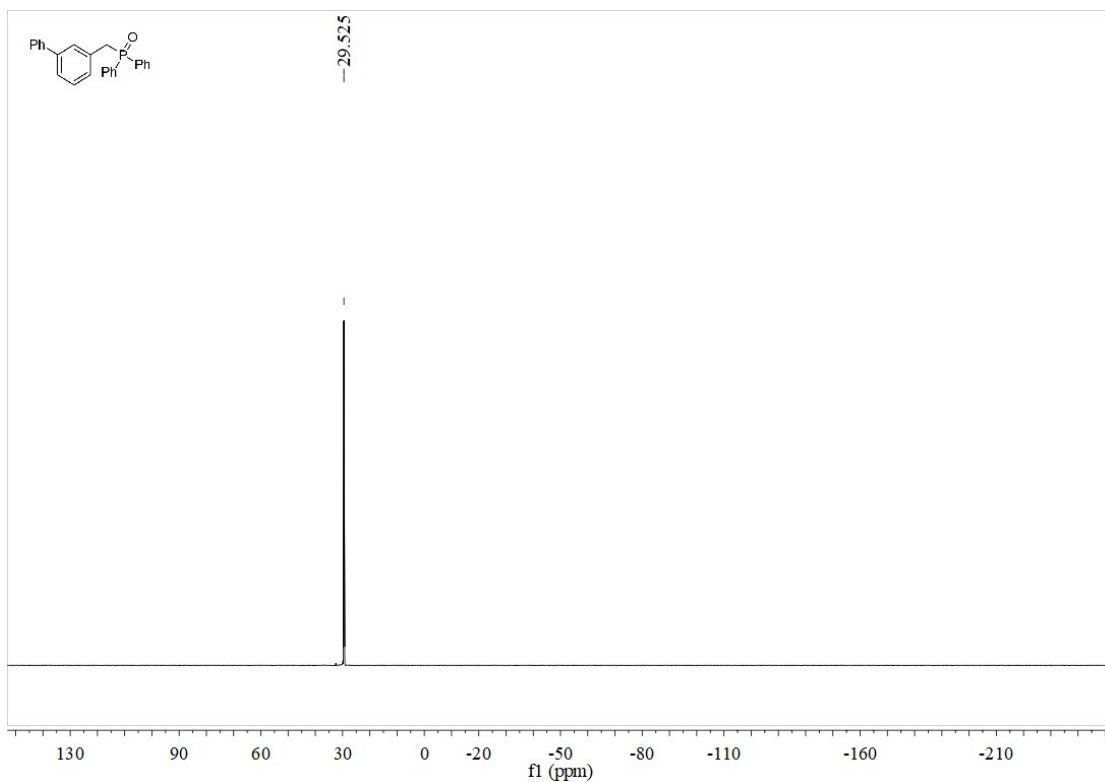
³¹P NMR spectra of ([1,1'-biphenyl]-4-ylmethyl)diphenylphosphine oxide (**9d**).



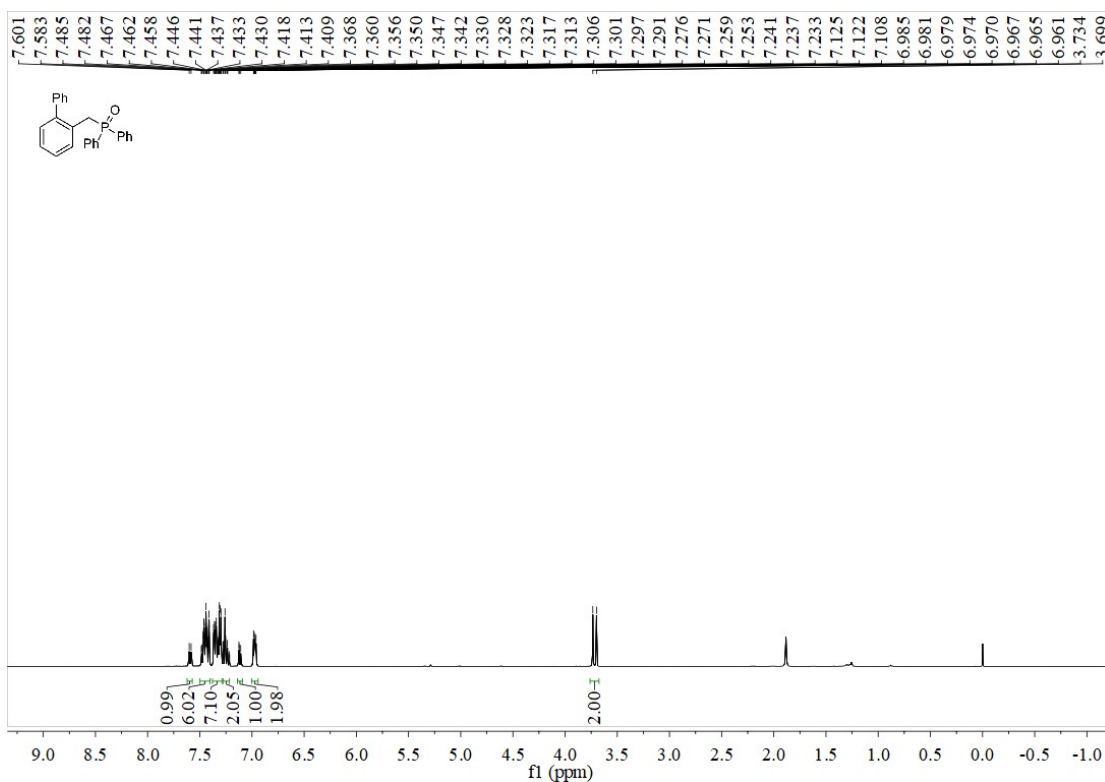
¹H NMR spectra of ([1,1'-biphenyl]-3-ylmethyl)diphenylphosphine oxide (**10d**).



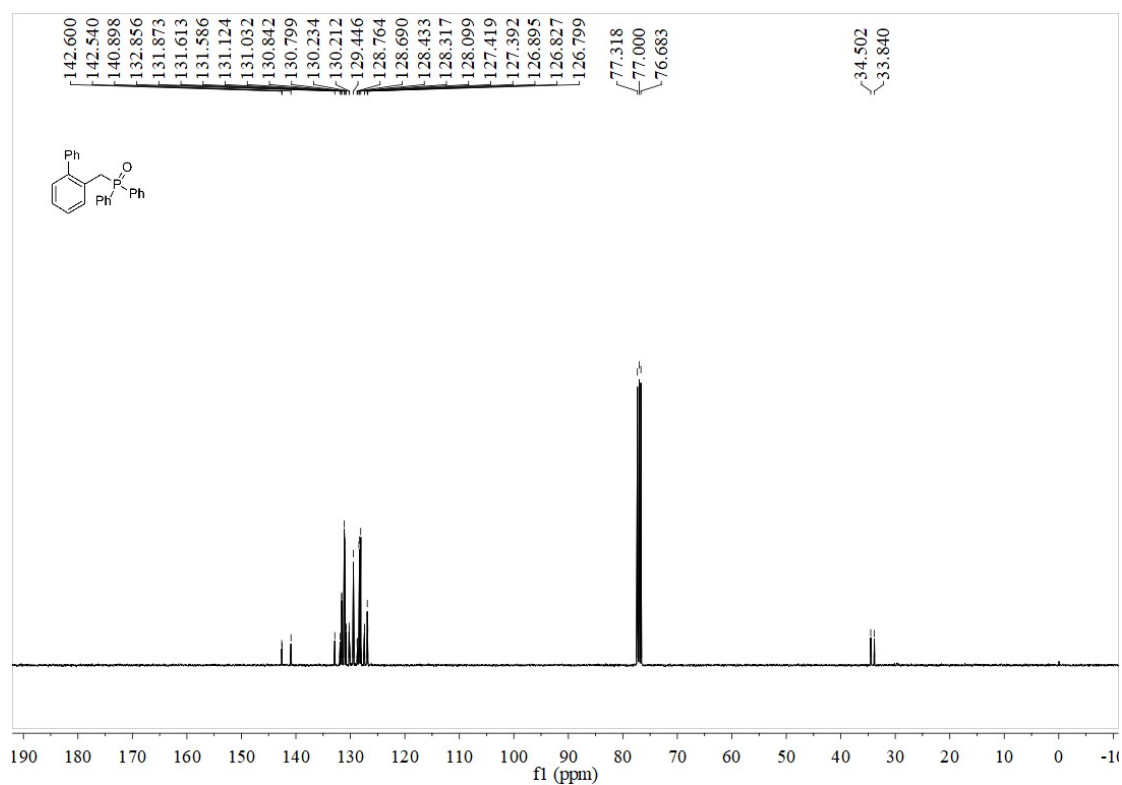
¹³C NMR spectra of ([1,1'-biphenyl]-3-ylmethyl)diphenylphosphine oxide (**10d**).



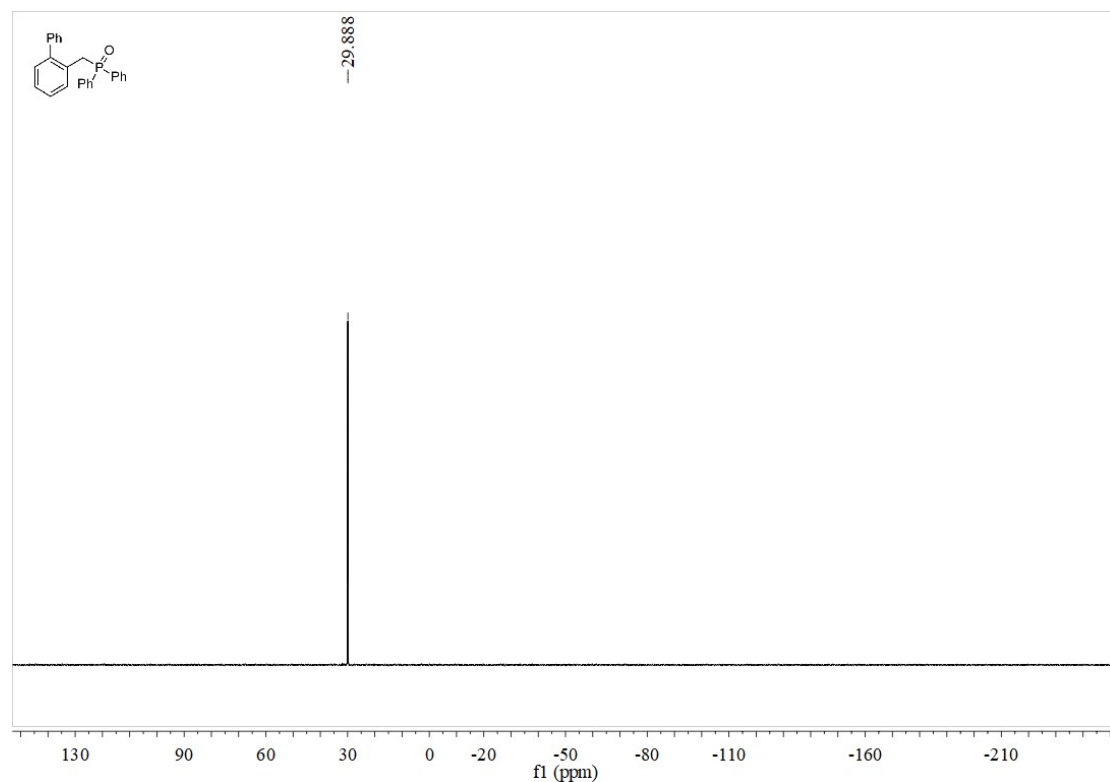
^{31}P NMR spectra of ([1,1'-biphenyl]-3-ylmethyl)diphenylphosphine oxide (10d).



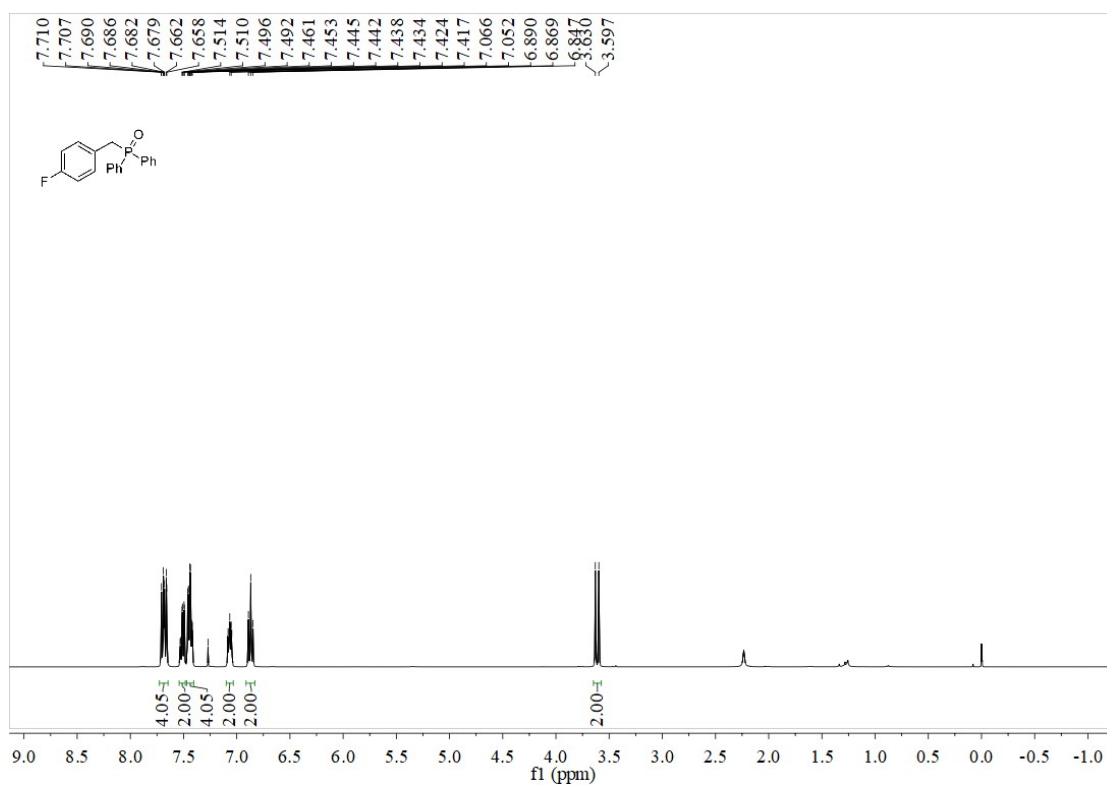
^1H NMR spectra of ([1,1'-biphenyl]-2-ylmethyl)diphenylphosphine oxide (11d).



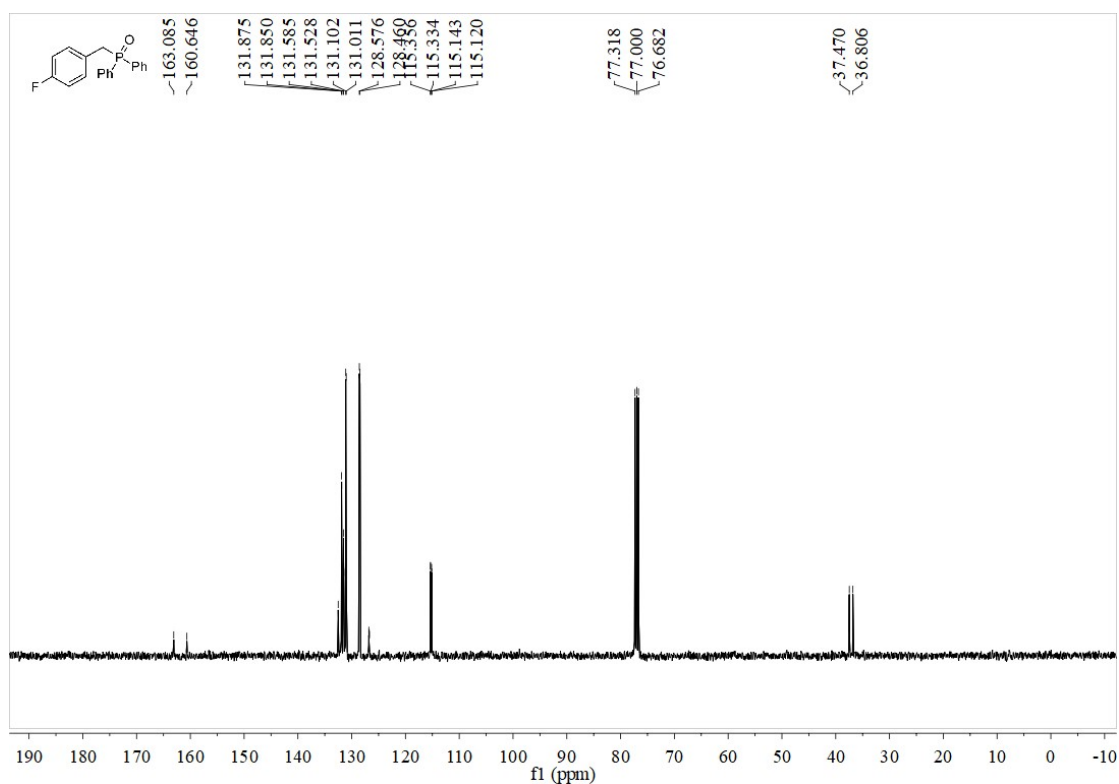
¹³C NMR spectra of ([1,1'-biphenyl]-2-ylmethyl)diphenylphosphine oxide (**11d**).



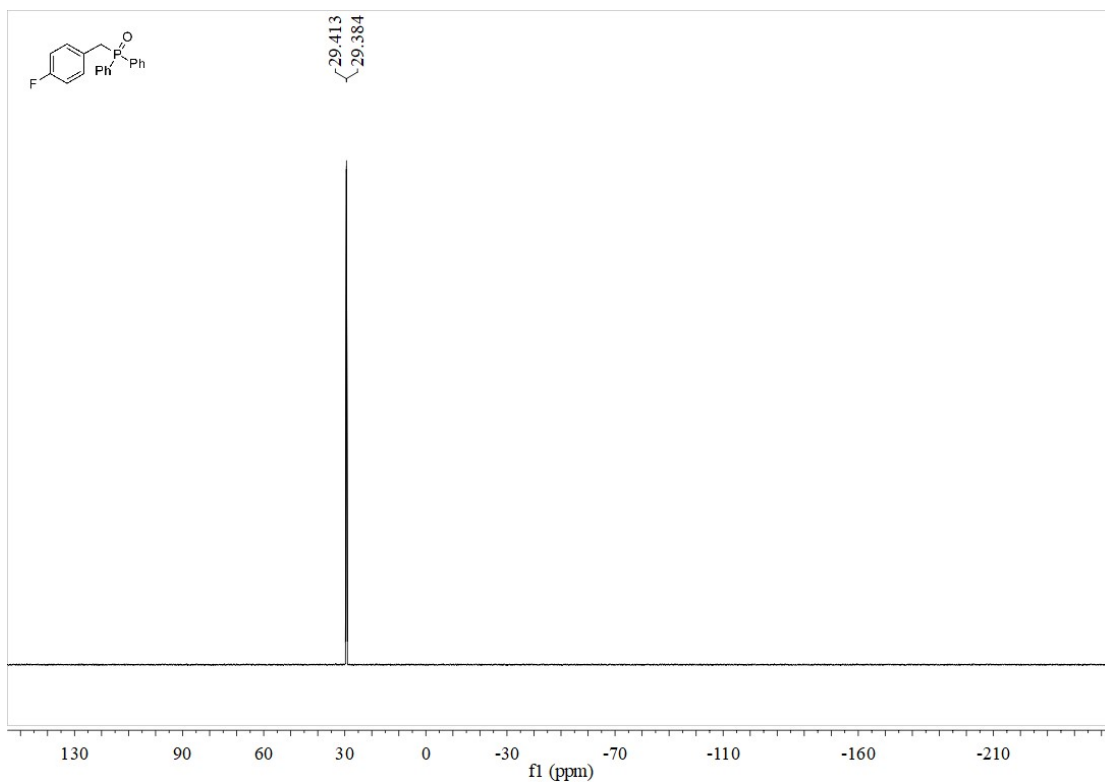
³¹P NMR spectra of ([1,1'-biphenyl]-2-ylmethyl)diphenylphosphine oxide (**11d**).



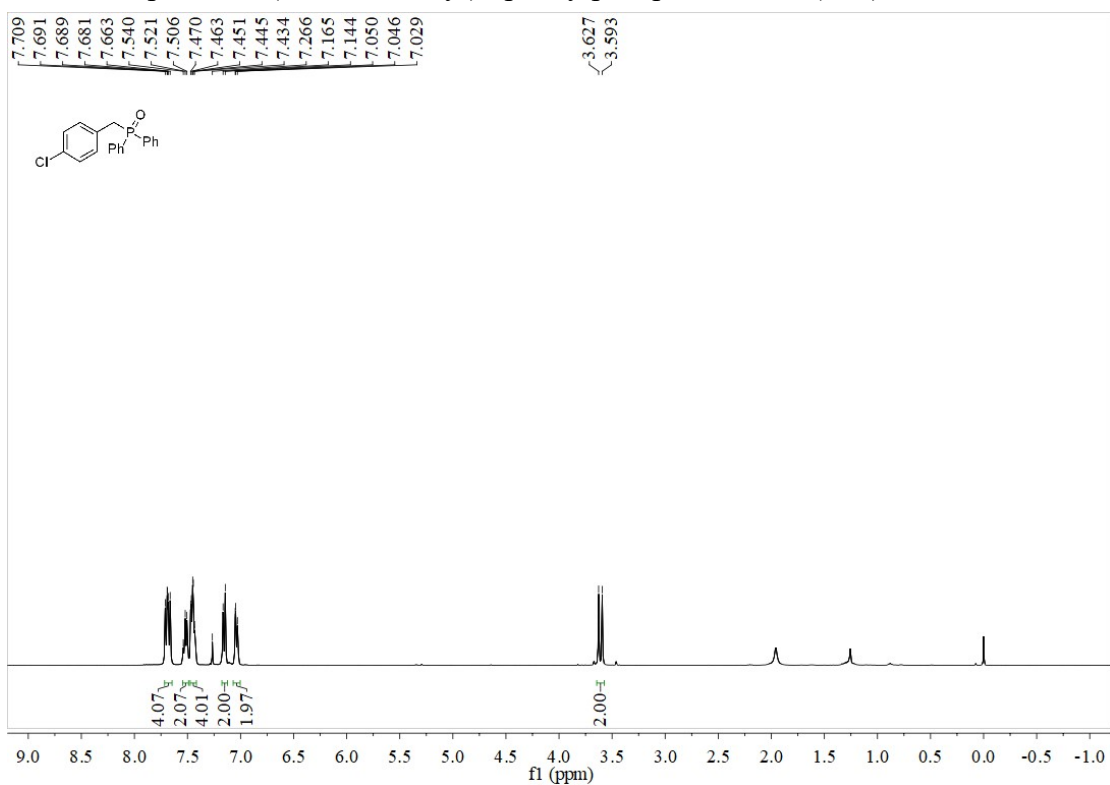
¹H NMR spectra of (4-fluorobenzyl)diphenylphosphine oxide (**12d**).



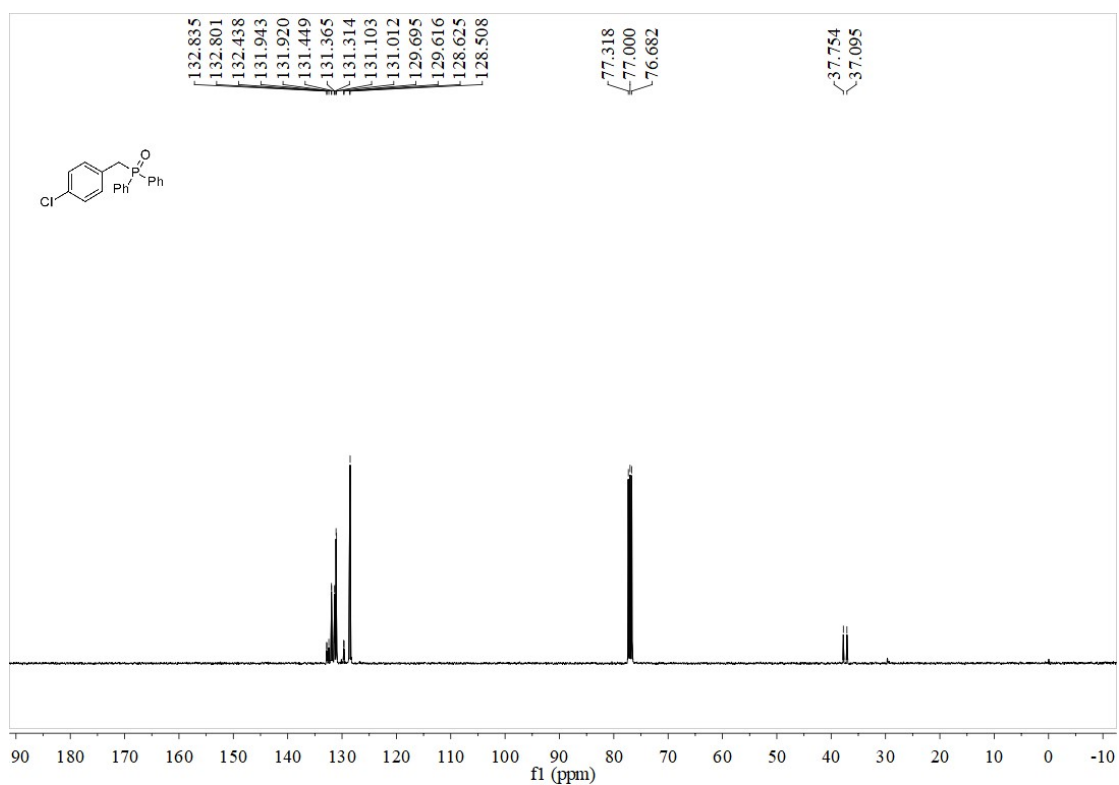
¹³C NMR spectra of (4-fluorobenzyl)diphenylphosphine oxide (**12d**).



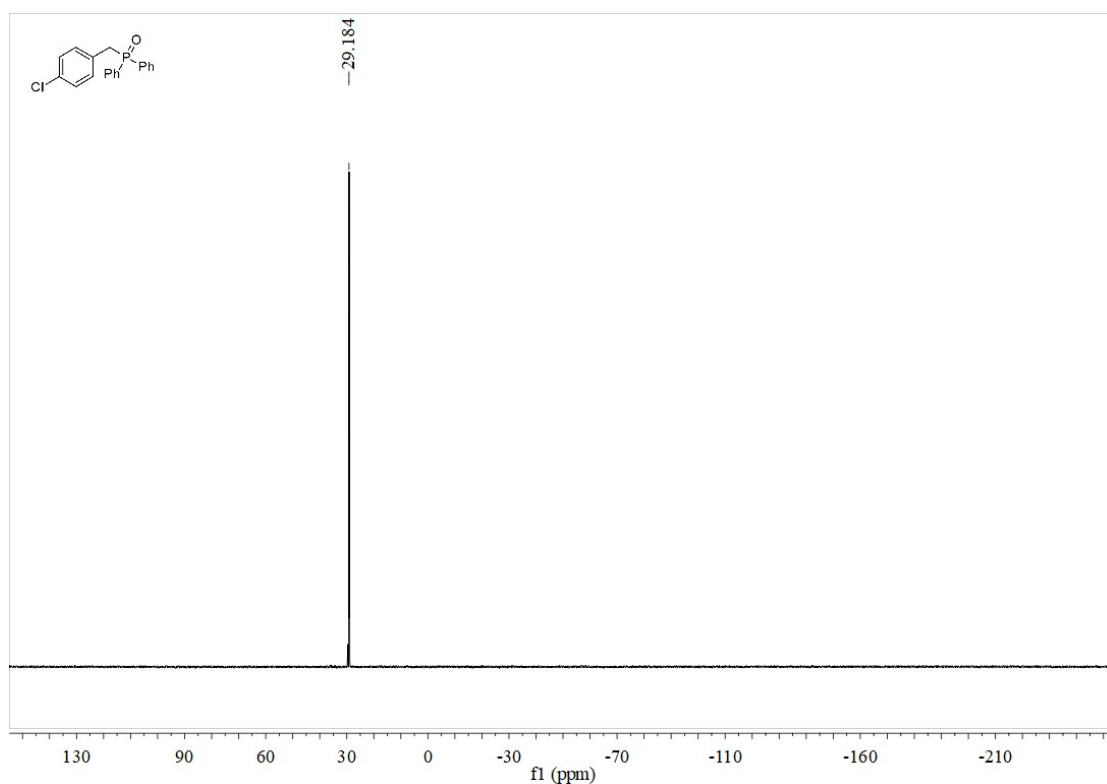
^{31}P NMR spectra of (4-fluorobenzyl)diphenylphosphine oxide (**12d**).



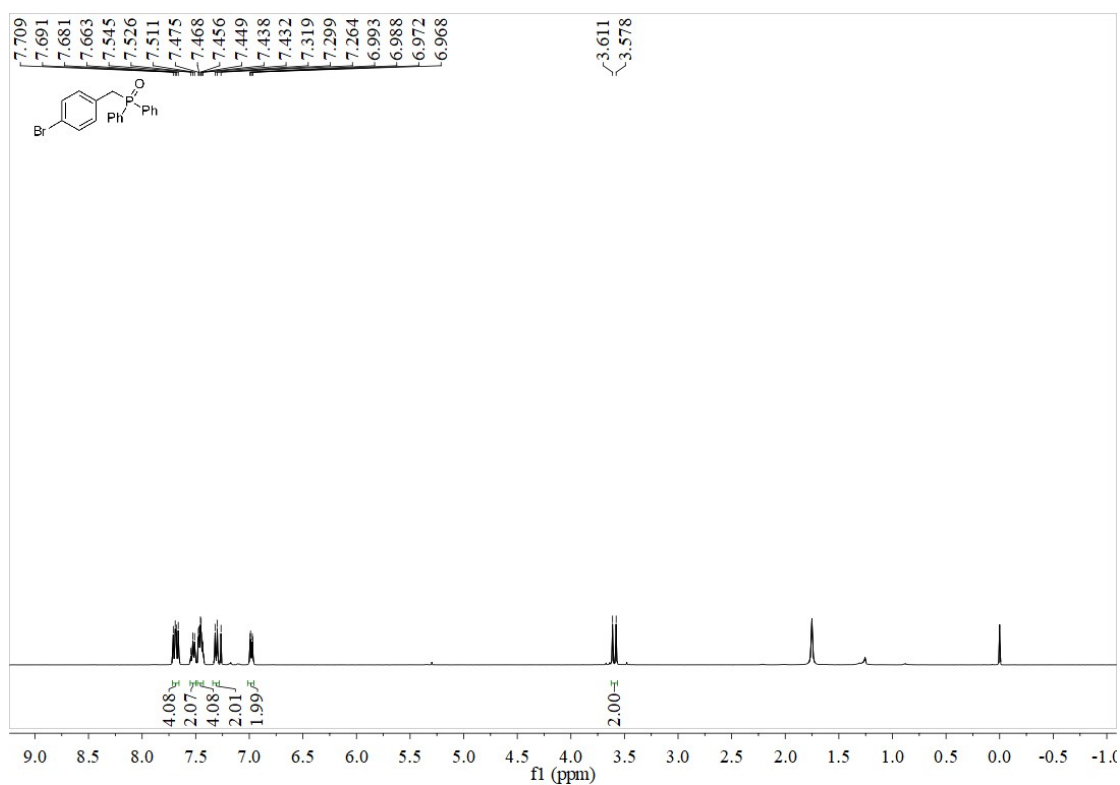
^1H NMR spectra of (4-chlorobenzyl)diphenylphosphine oxide (**13d**).



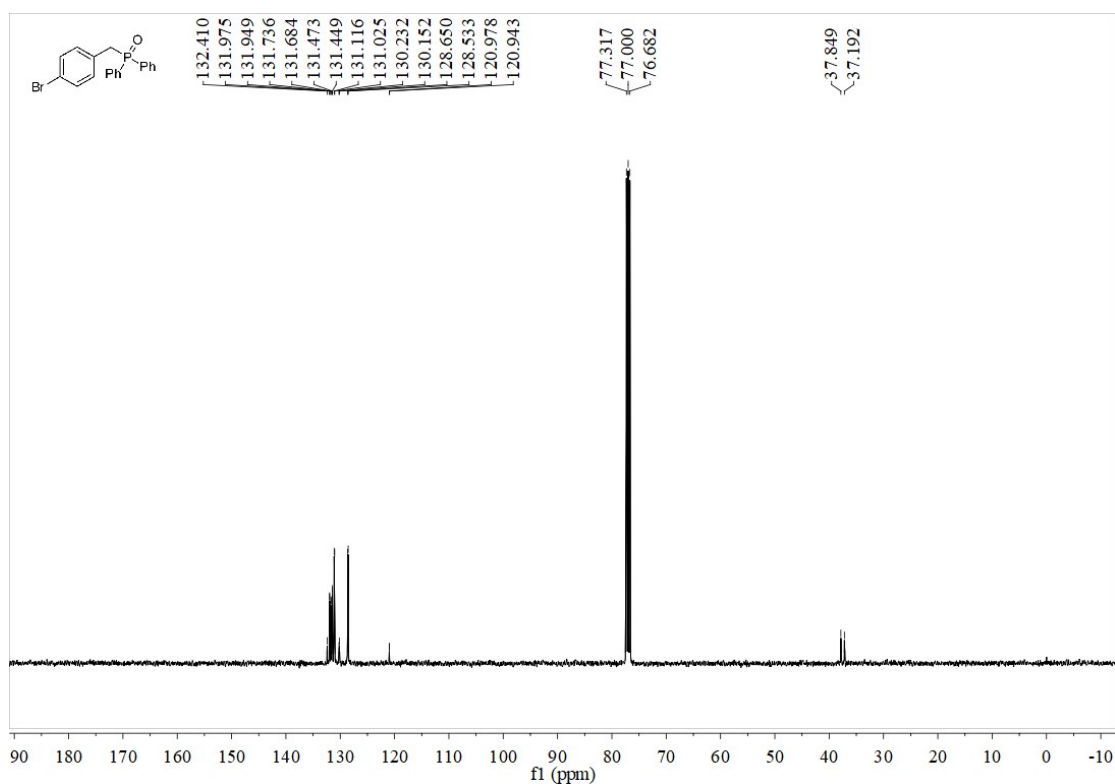
¹³C NMR spectra of (4-chlorobenzyl)diphenylphosphine oxide (**13d**).



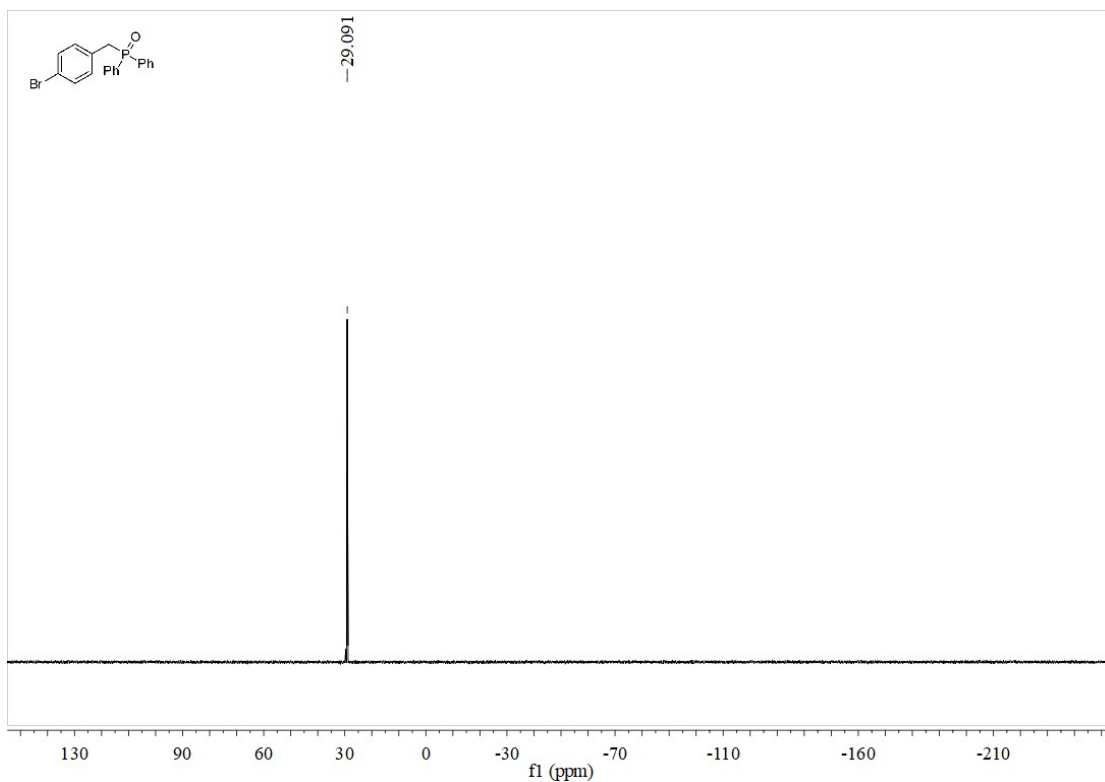
³¹P NMR spectra of (4-chlorobenzyl)diphenylphosphine oxide (**13d**).



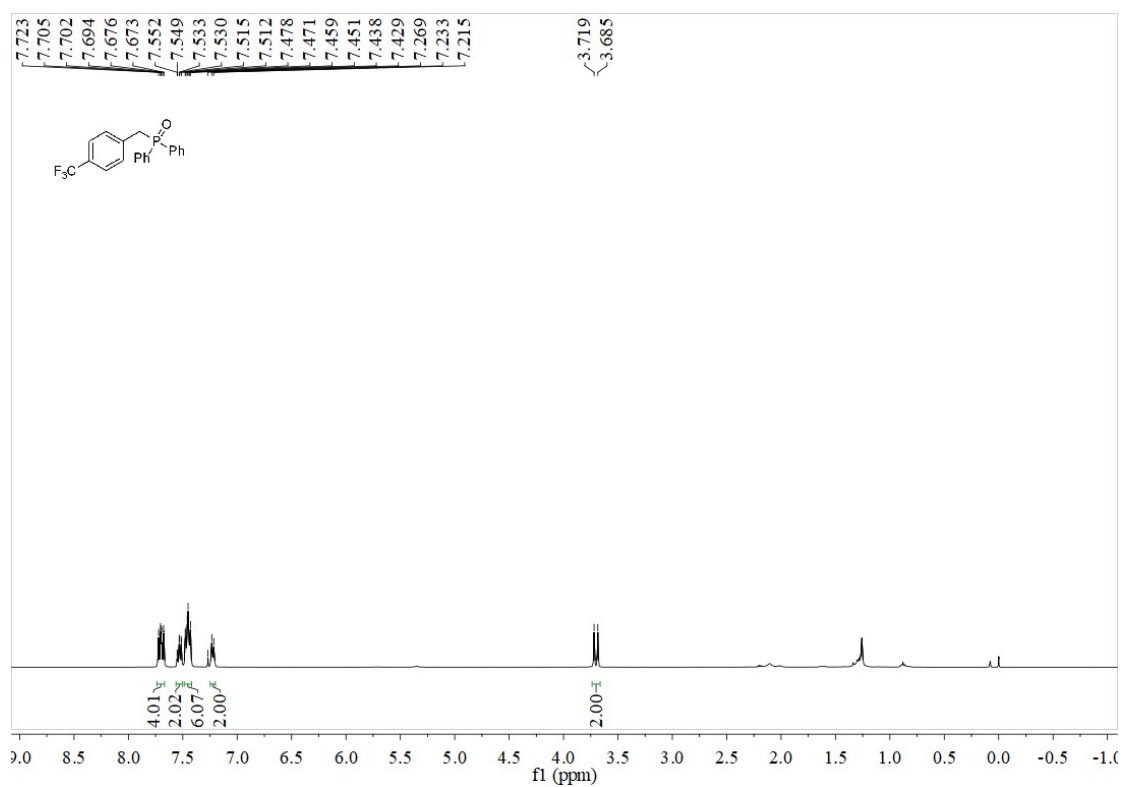
¹H NMR spectra of (4-bromobenzyl)diphenylphosphine oxide (**14d**).



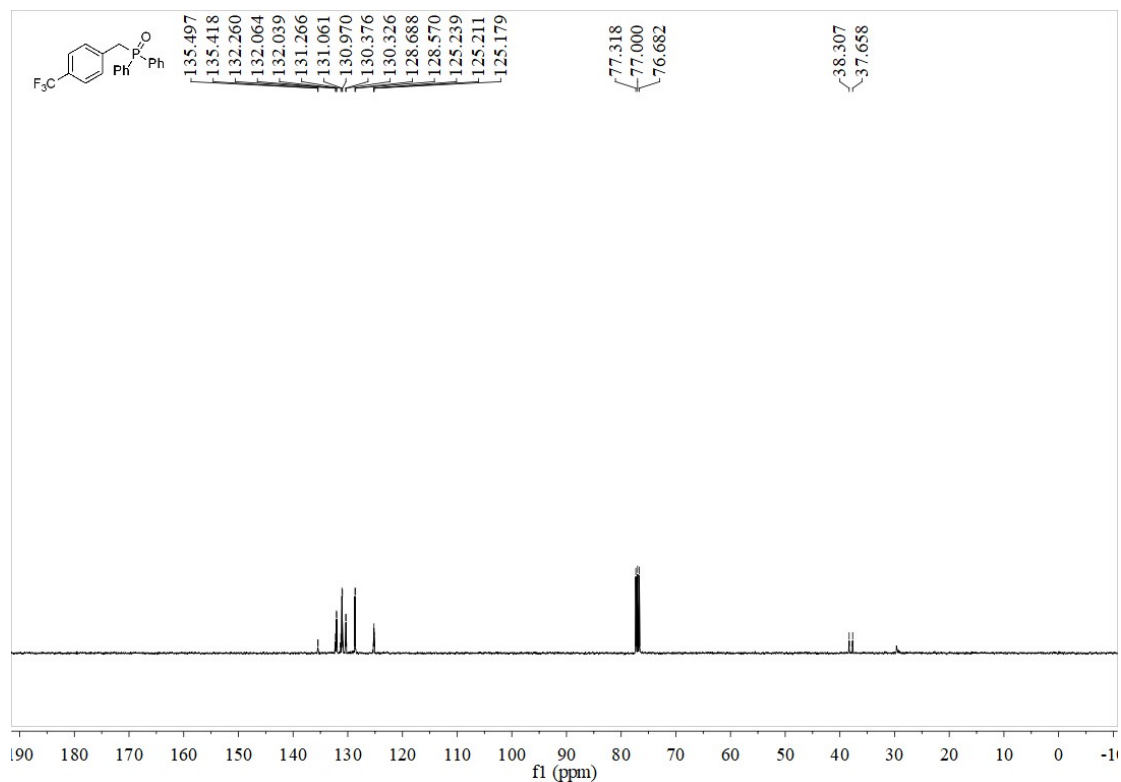
¹³C NMR spectra of (4-bromobenzyl)diphenylphosphine oxide (**14d**).



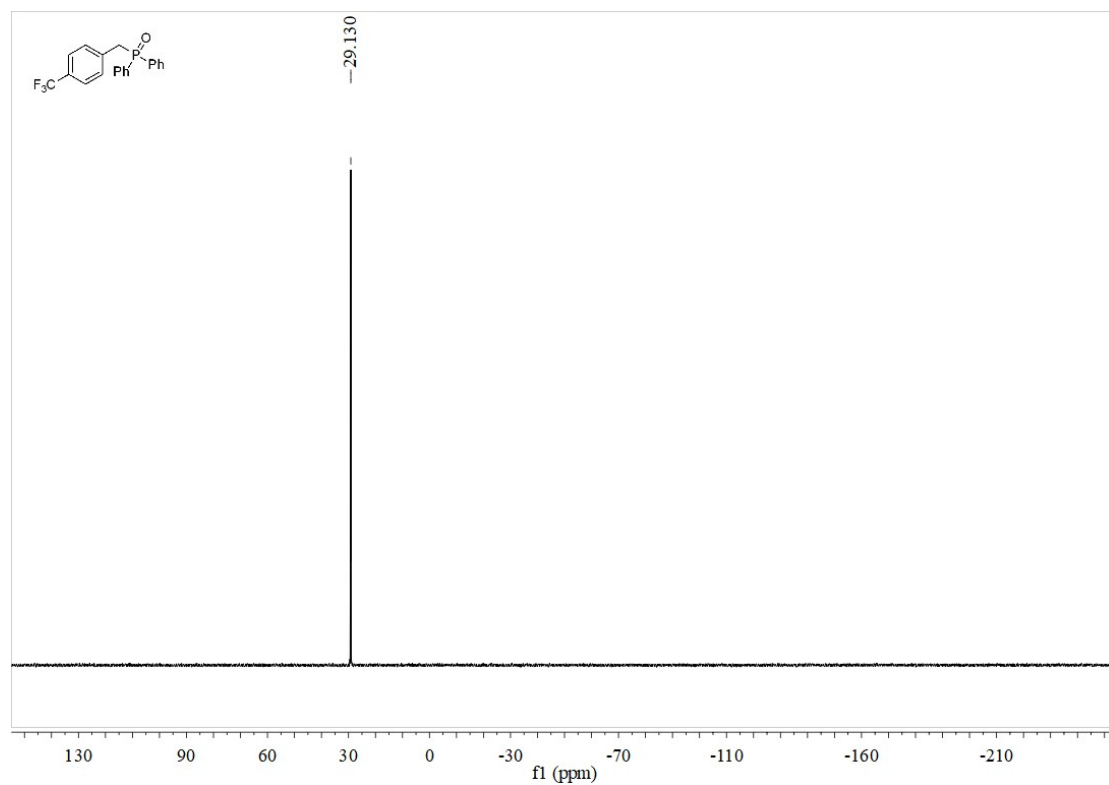
^{31}P NMR spectra of (4-bromobenzyl)diphenylphosphine oxide (**14d**).



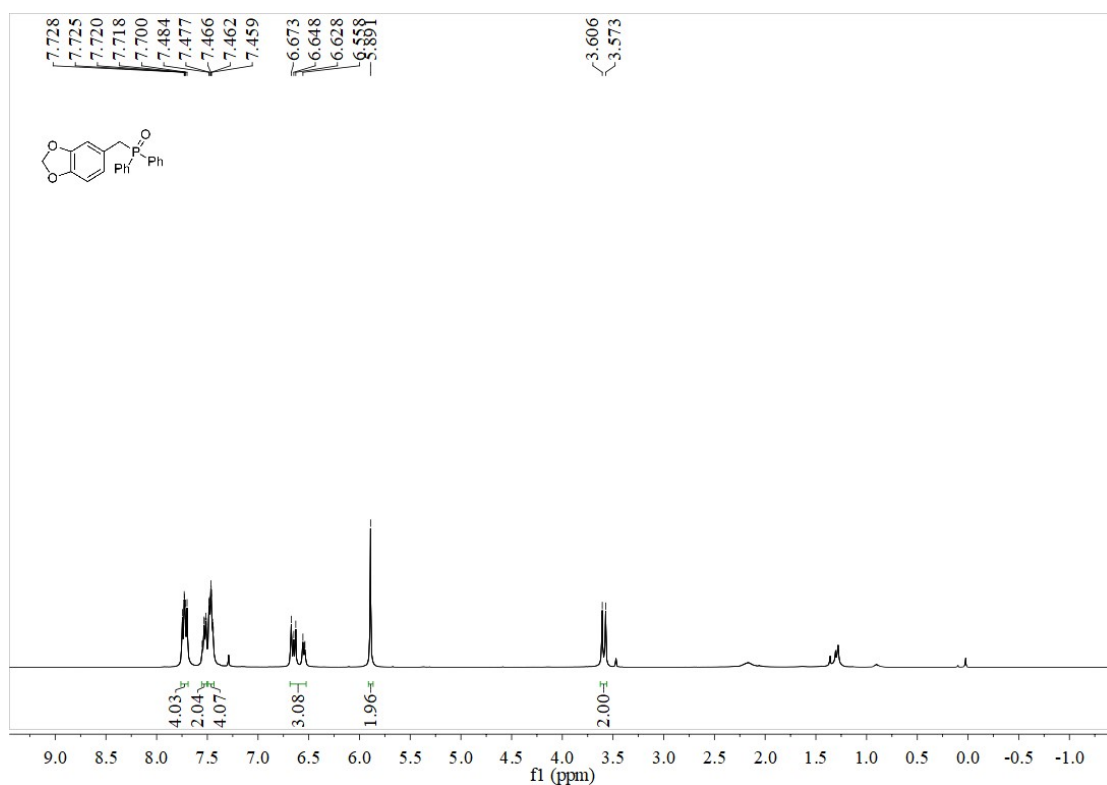
^1H NMR spectra of Diphenyl(4-(trifluoromethyl)benzyl)phosphine oxide (**15d**).



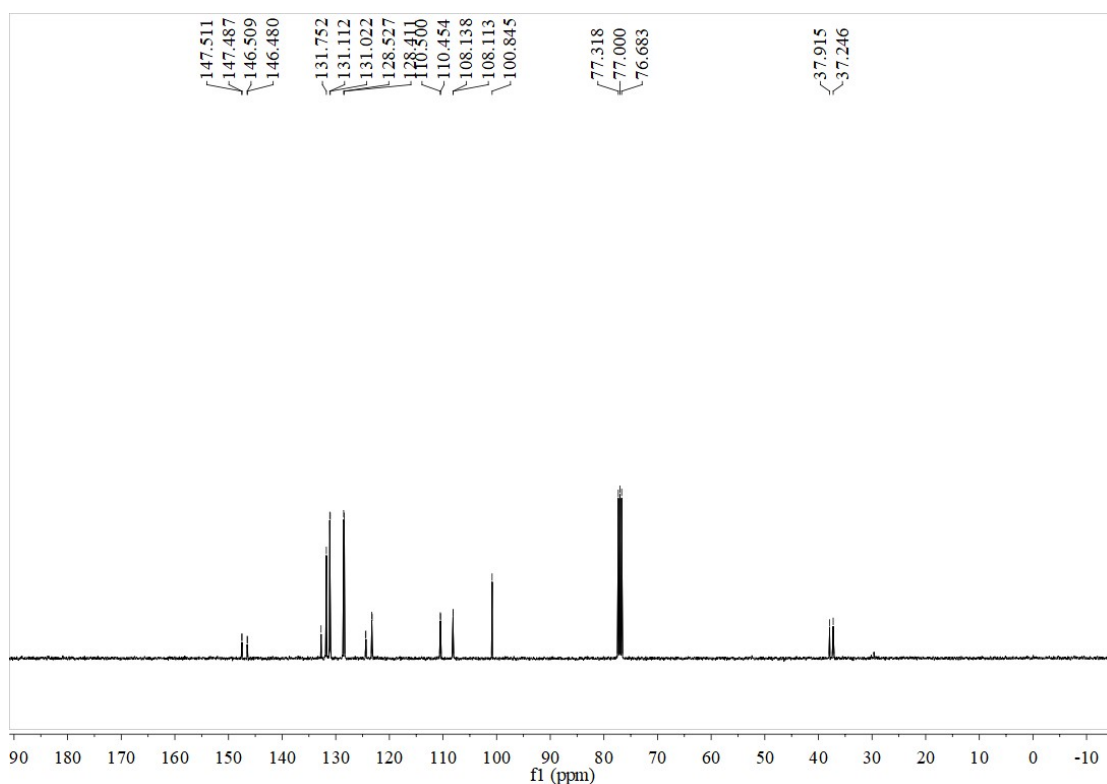
¹³C NMR spectra of Diphenyl(4-(trifluoromethyl)benzyl)phosphine oxide (**15d**).



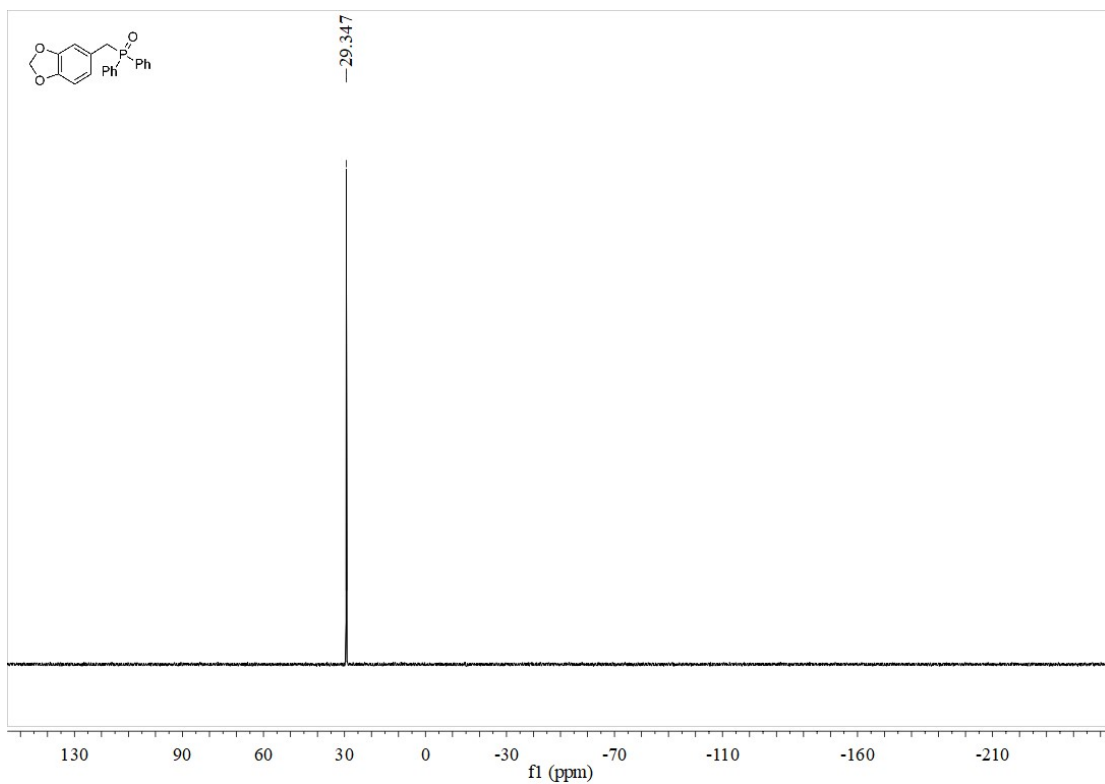
³¹P NMR spectra of Diphenyl(4-(trifluoromethyl)benzyl)phosphine oxide (**15d**).



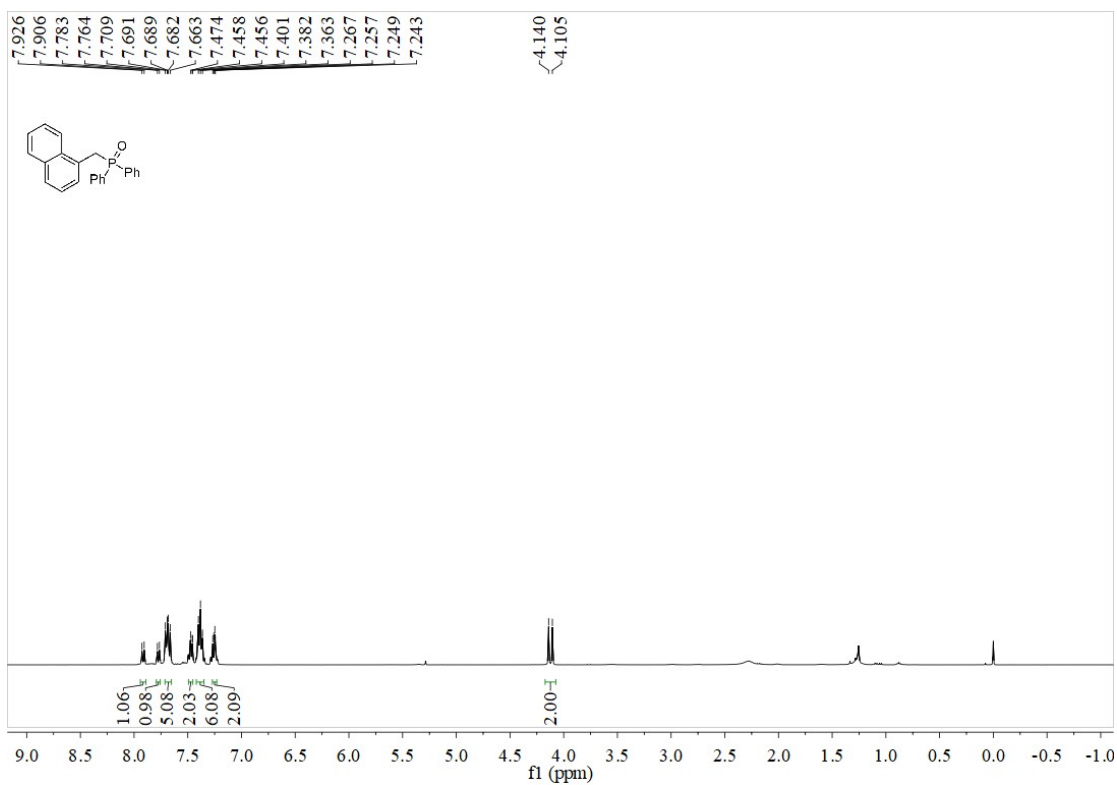
¹H NMR spectra of (benzo[d][1,3]dioxol-5-ylmethyl)diphenylphosphine oxide (**16d**).



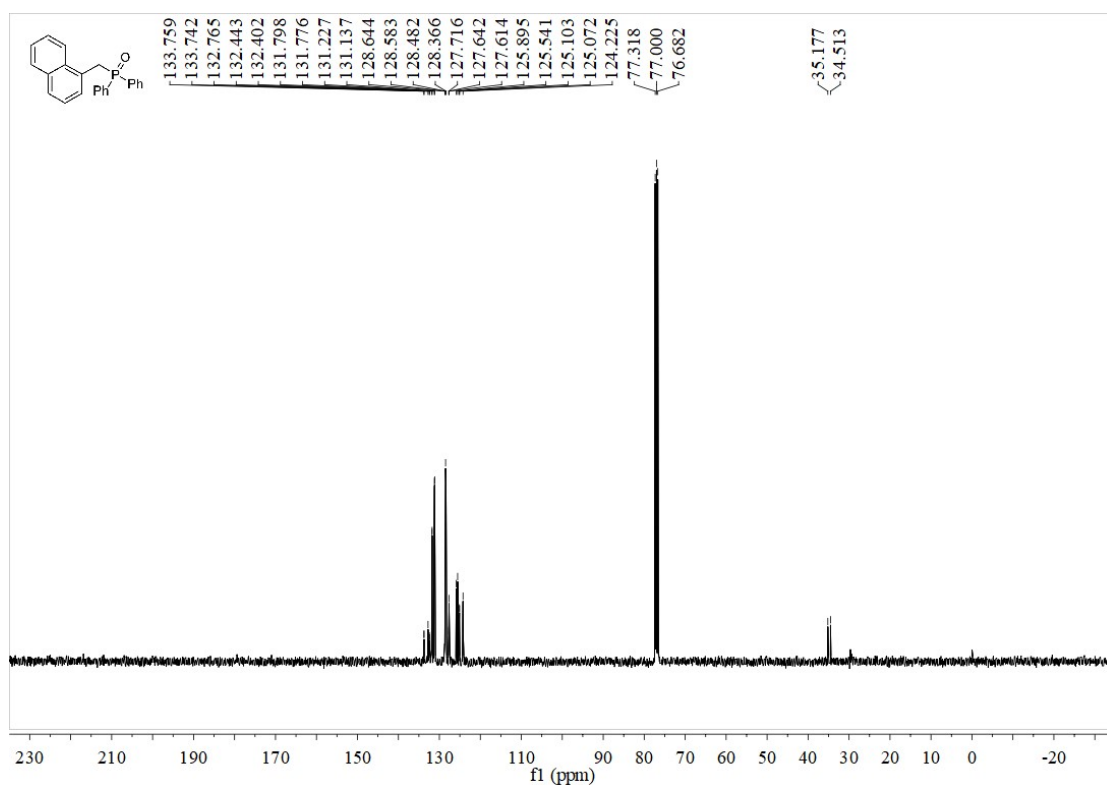
¹³C NMR spectra of (benzo[d][1,3]dioxol-5-ylmethyl)diphenylphosphine oxide (**16d**).



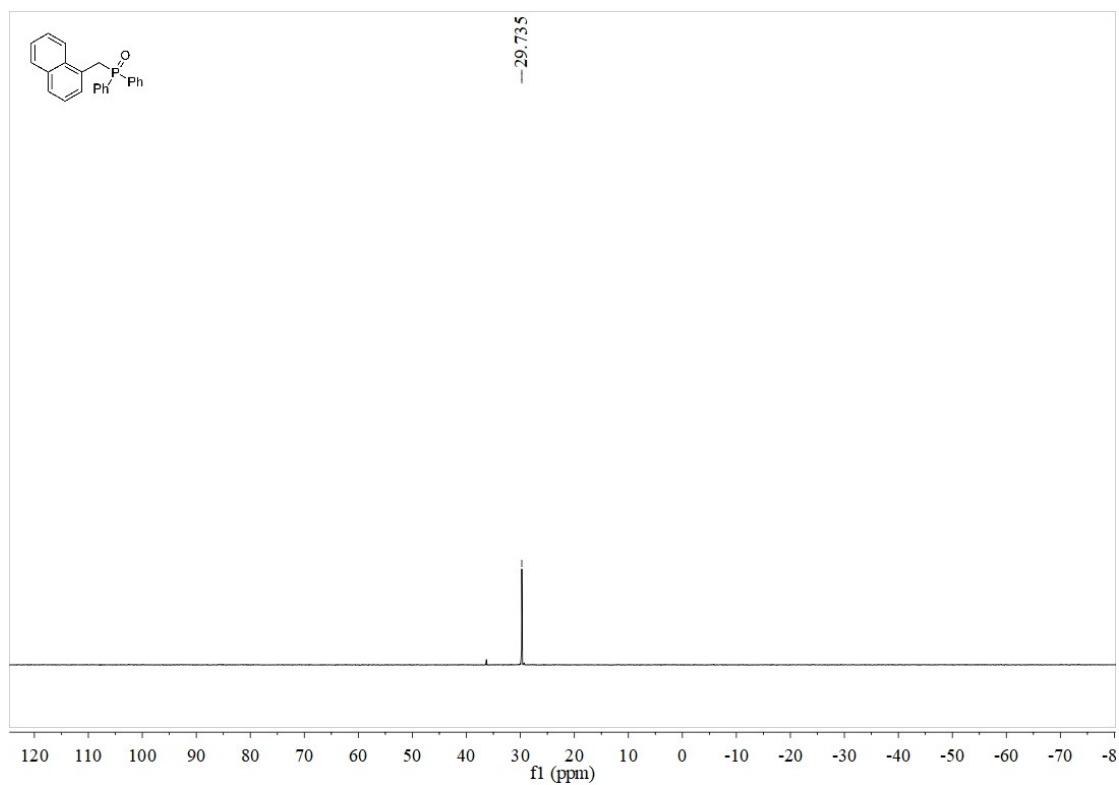
^{31}P NMR spectra of (benzo[d][1,3]dioxol-5-ylmethyl)diphenylphosphine oxide (**16d**).



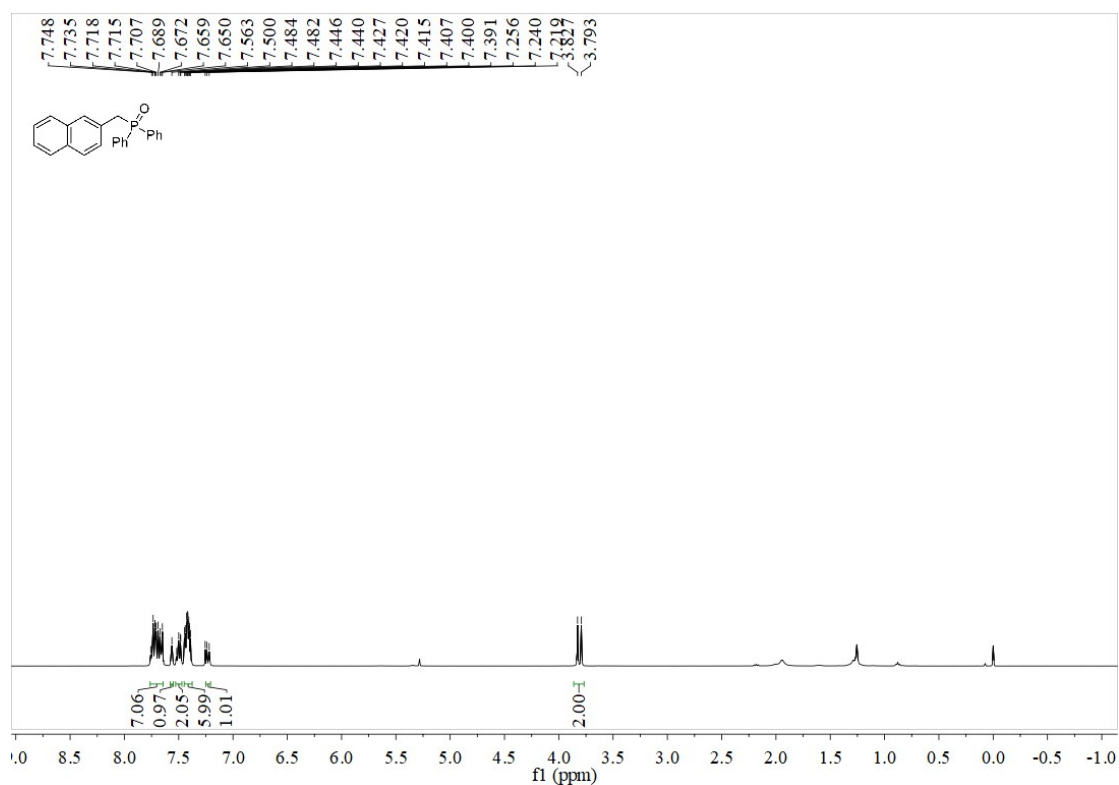
^1H NMR spectra of (naphthalen-1-ylmethyl)diphenylphosphine oxide (**17d**).



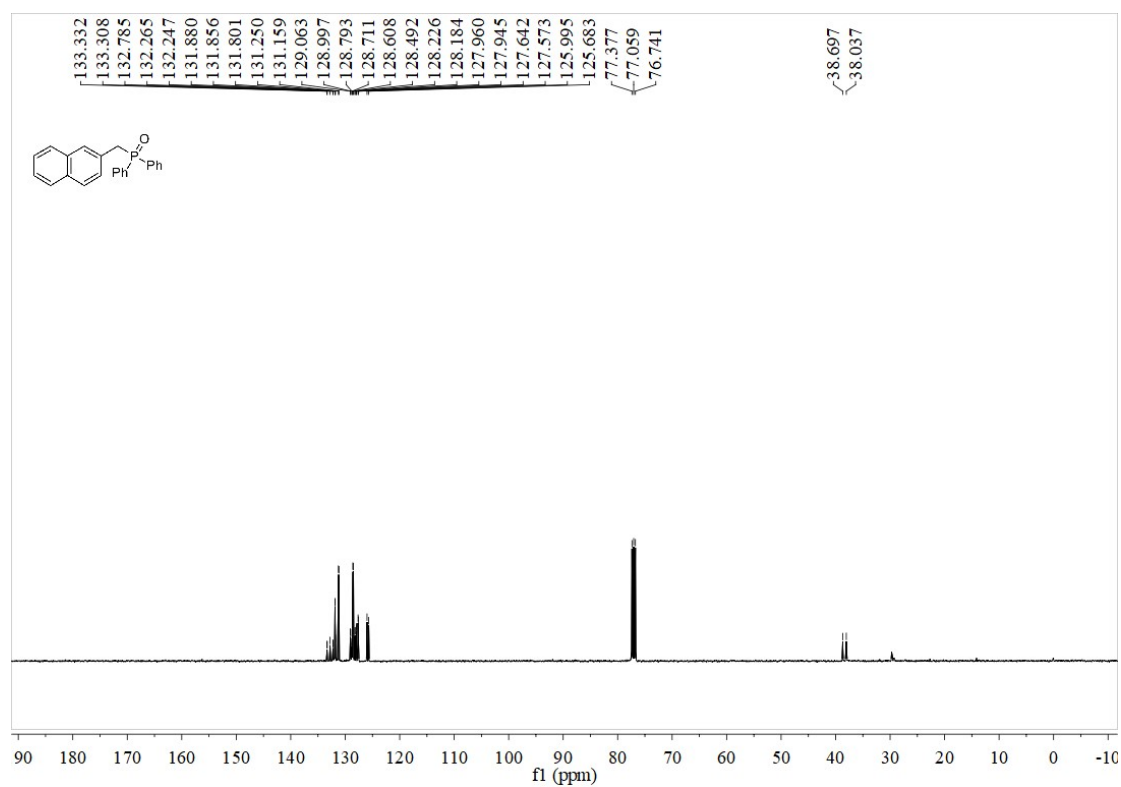
¹³C NMR spectra of (naphthalen-1-ylmethyl)diphenylphosphine oxide (**17d**).



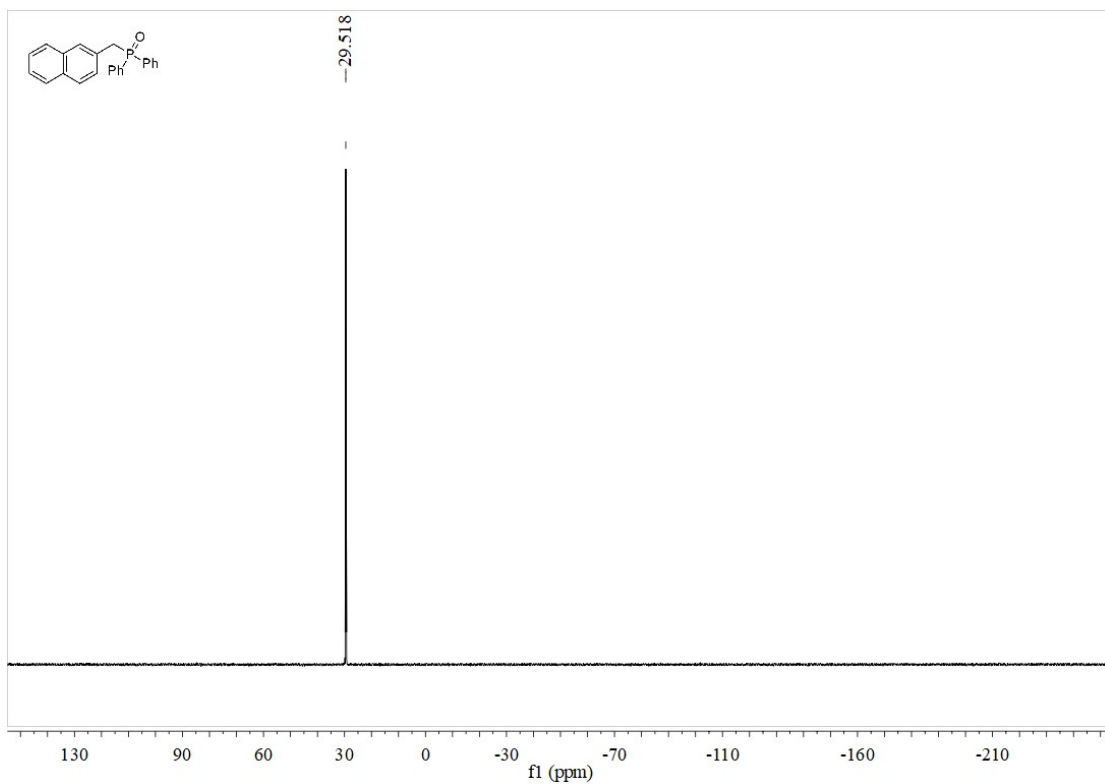
³¹P NMR spectra of (naphthalen-1-ylmethyl)diphenylphosphine oxide (**17d**).



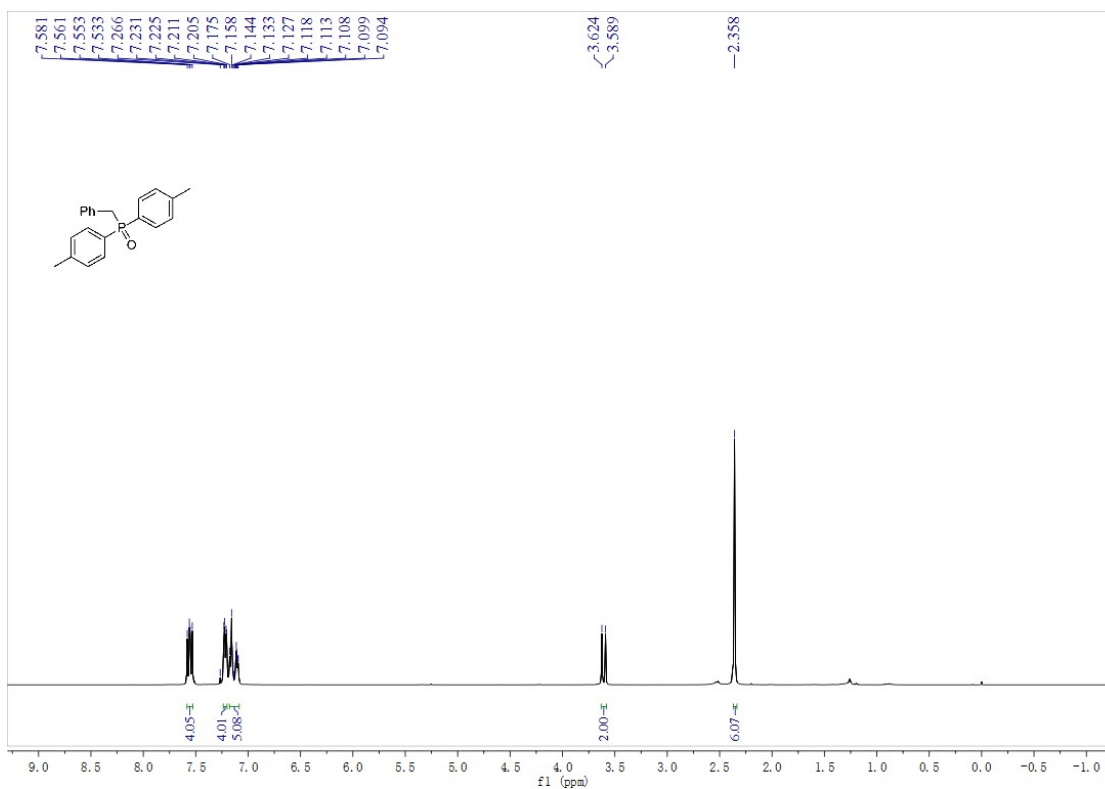
¹H NMR spectra of (naphthalen-2-ylmethyl)diphenylphosphine oxide (**18d**).



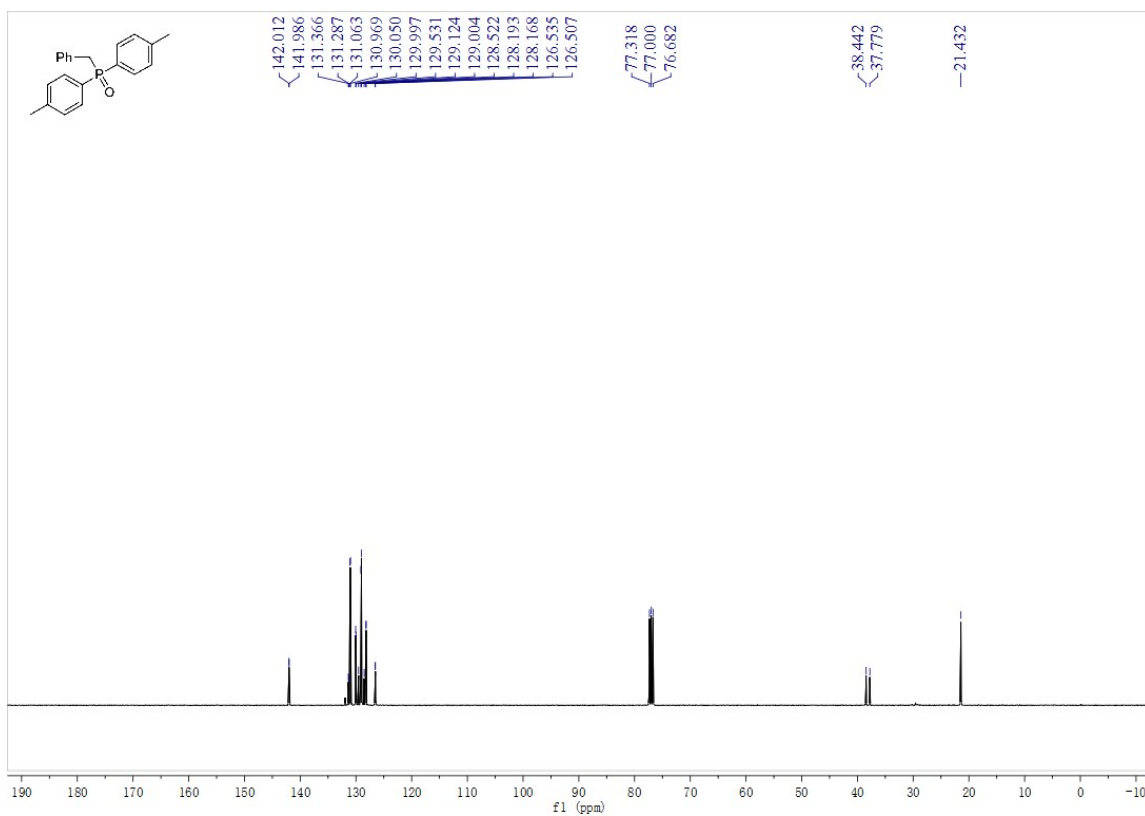
¹³C NMR spectra of (naphthalen-2-ylmethyl)diphenylphosphine oxide (**18d**).



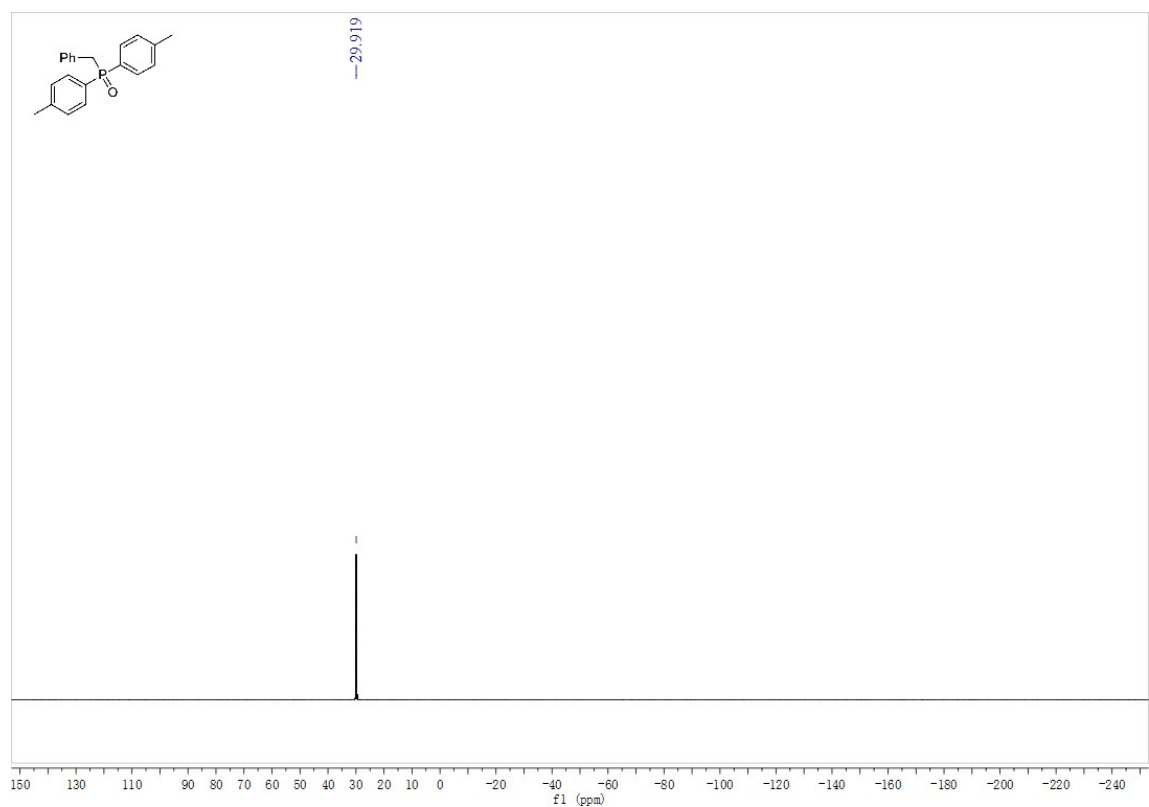
^{31}P NMR spectra of (naphthalen-2-ylmethyl)diphenylphosphine oxide (**18d**).



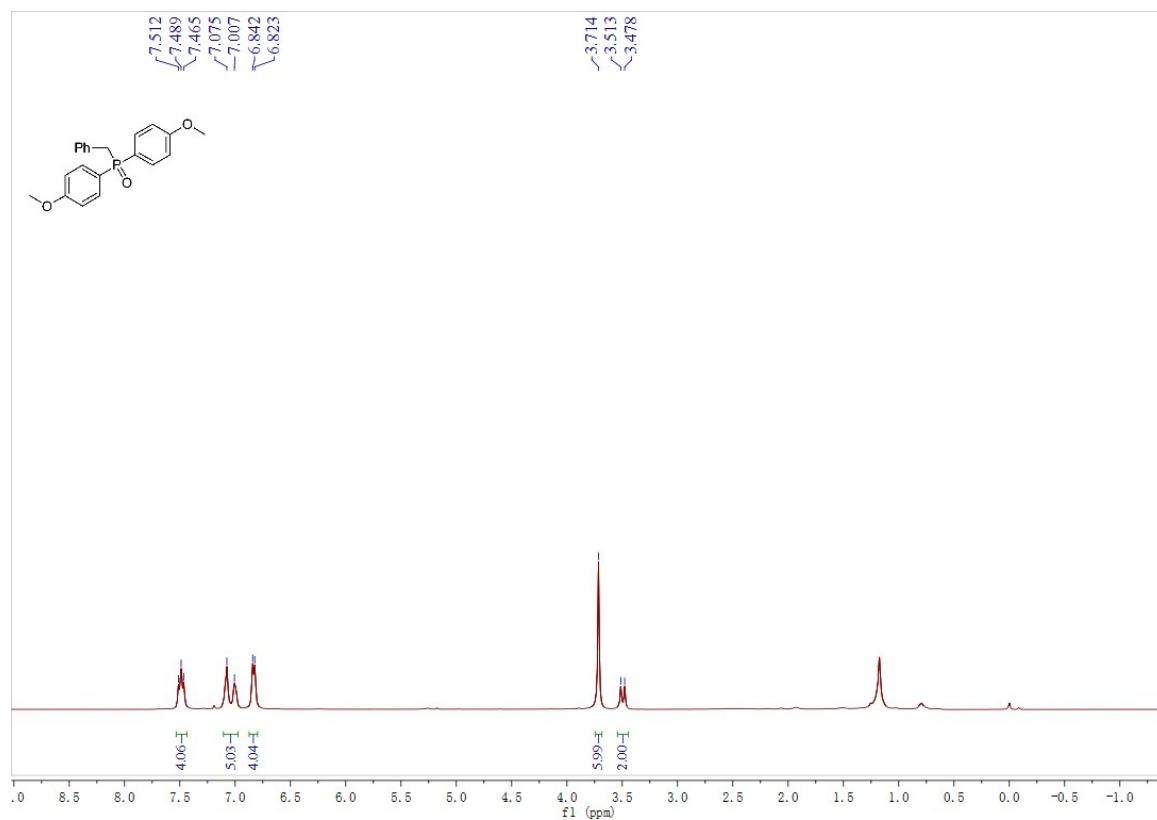
^1H NMR spectra of benzyl-di-p-tolylphosphine oxide (**19d**).



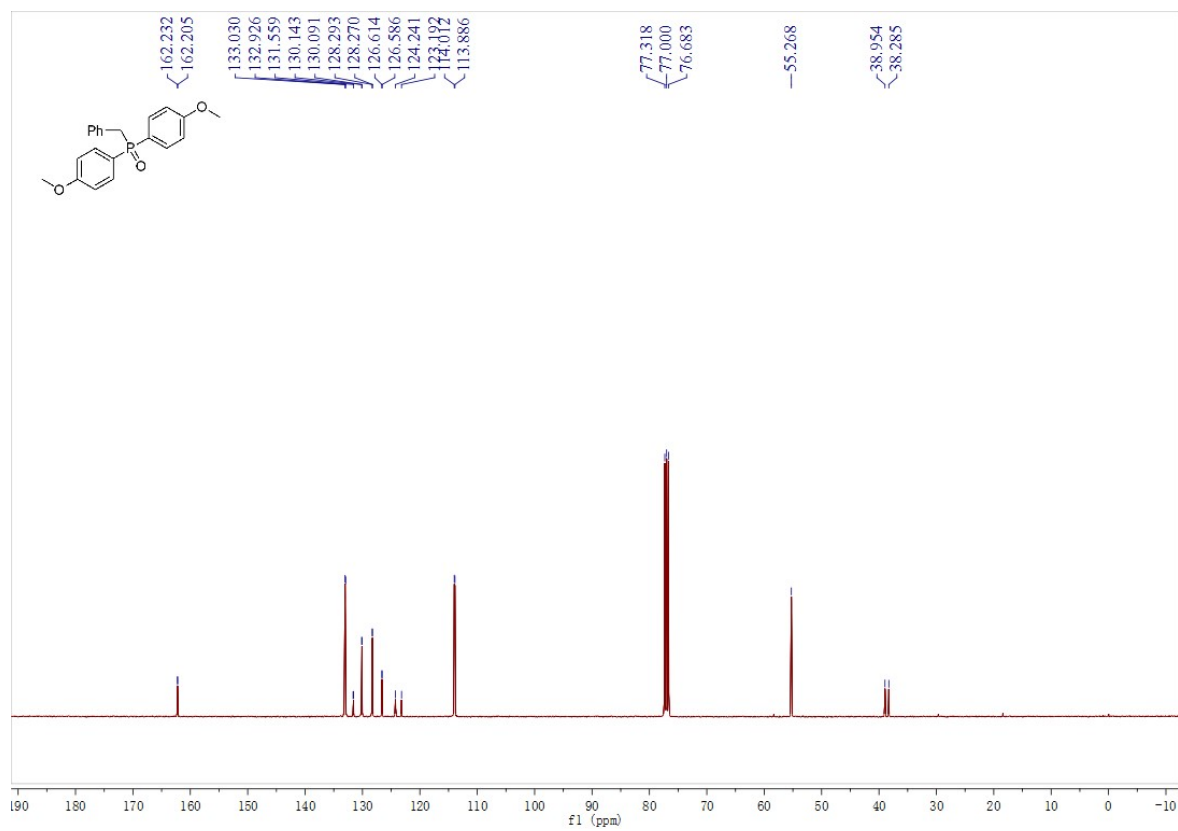
¹³C NMR spectra of benzyldi-p-tolylphosphine oxide (19d).



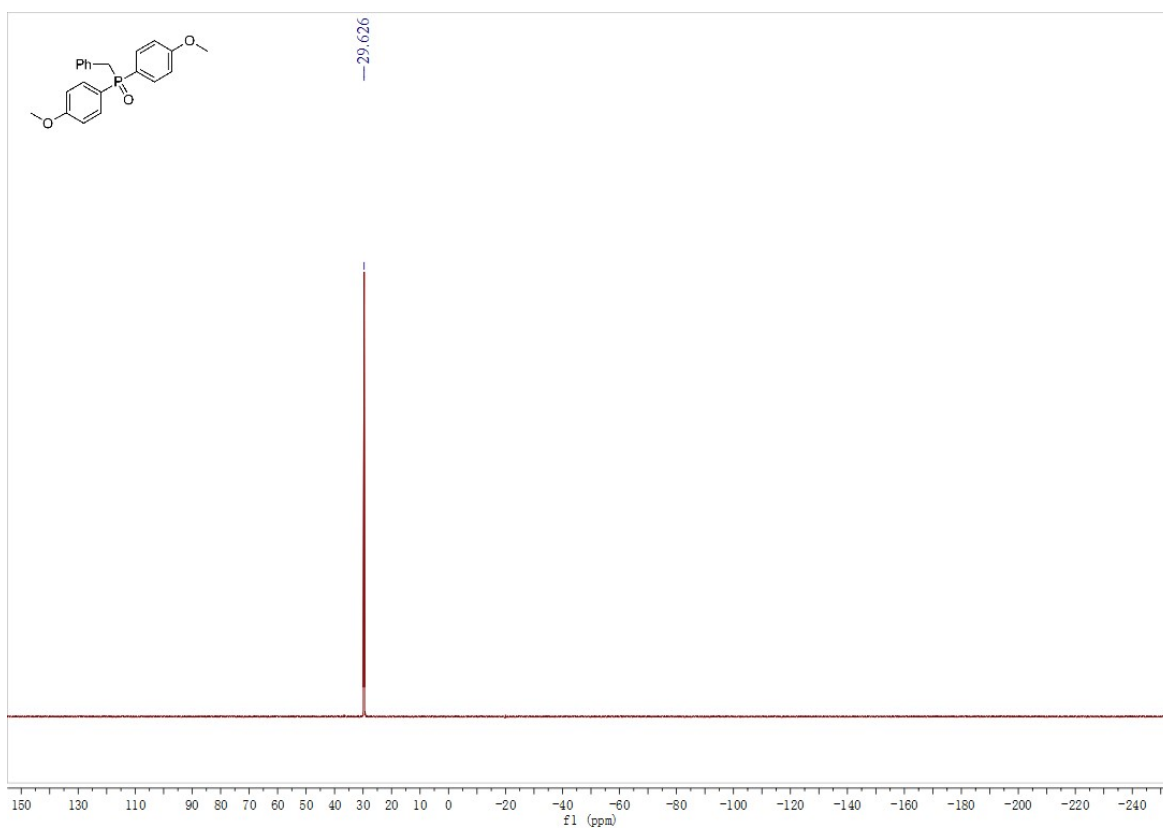
³¹P NMR spectra of benzyldi-p-tolylphosphine oxide (19d).



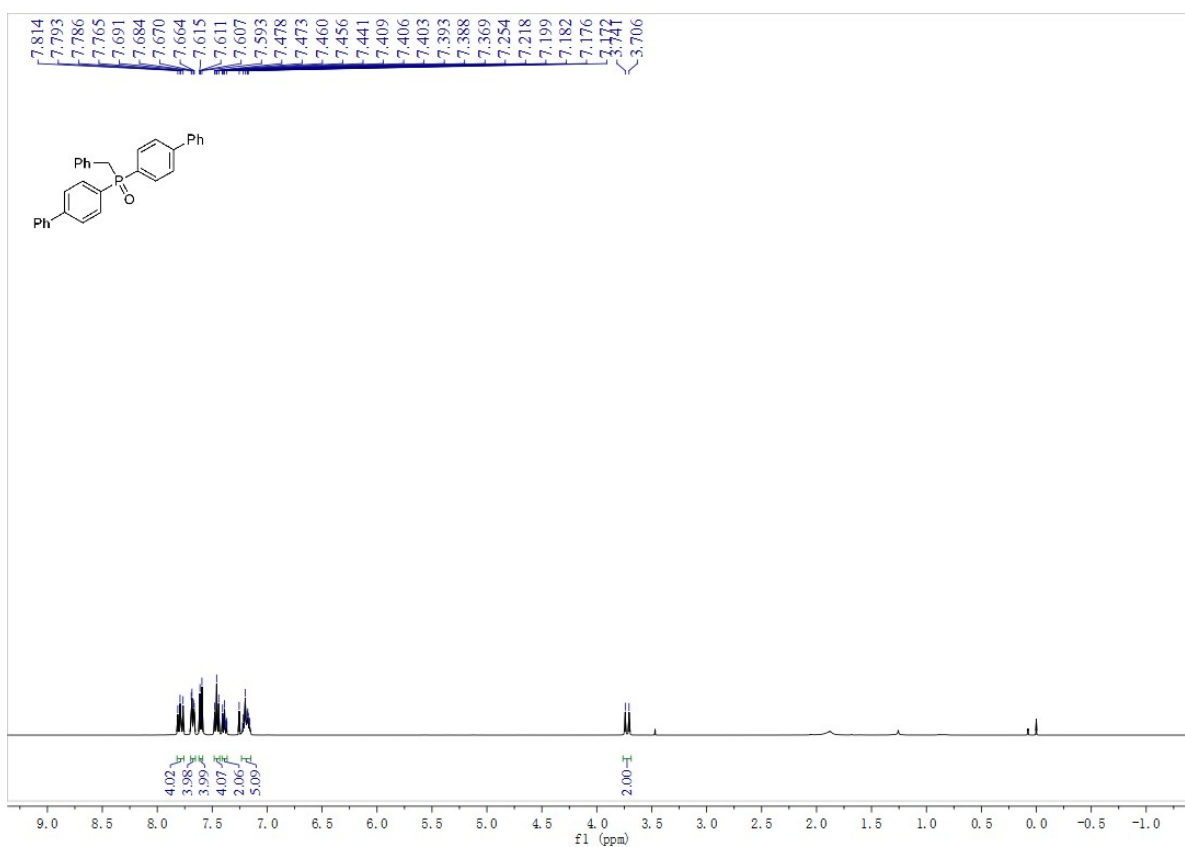
¹H NMR spectra of benzylbis(4-methoxyphenyl)phosphine oxide (**20d**).



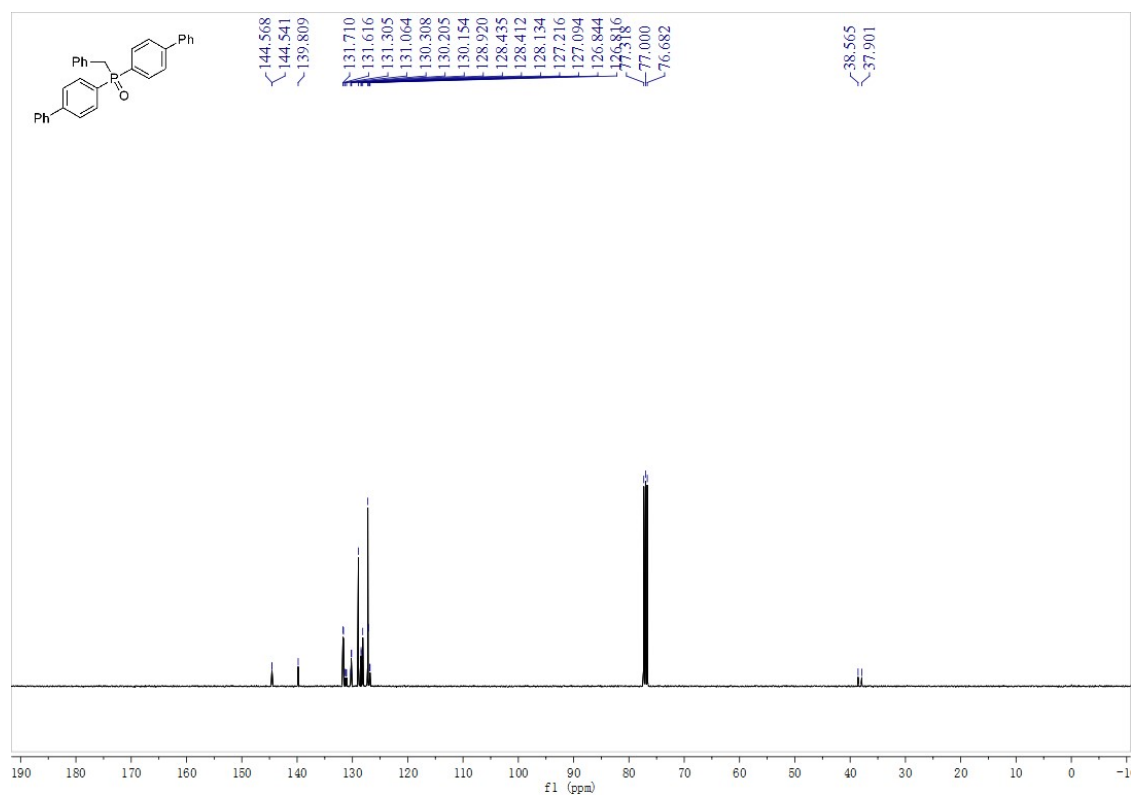
¹³C NMR spectra of benzylbis(4-methoxyphenyl)phosphine oxide (**20d**).



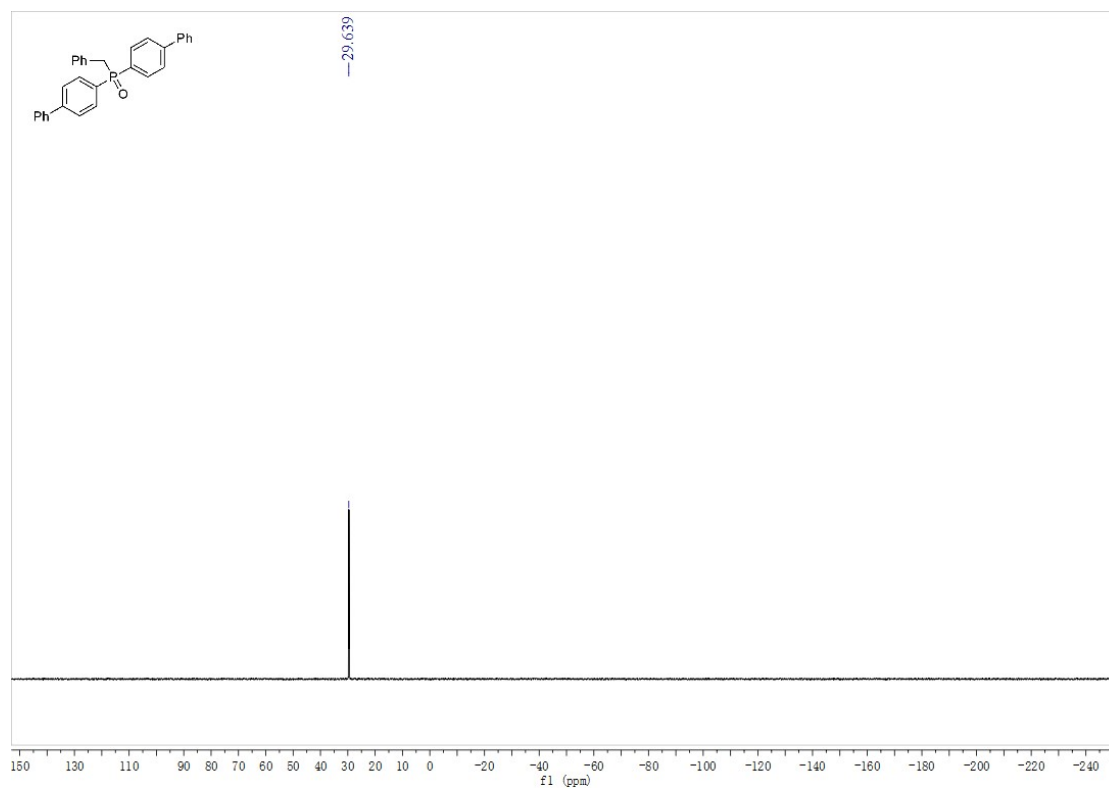
^{31}P NMR spectra of benzylbis(4-methoxyphenyl)phosphine oxide (**20d**).



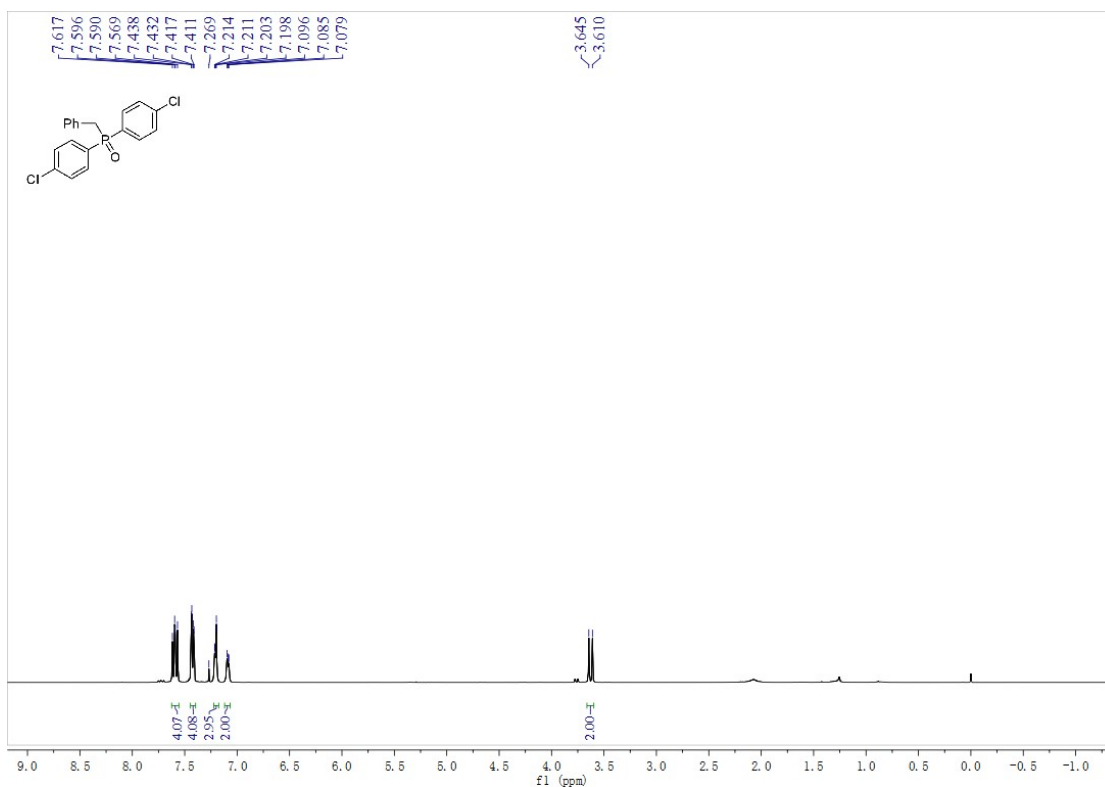
^1H NMR spectra of di([1,1'-biphenyl]-4-yl)(benzyl)phosphine oxide (**21d**).



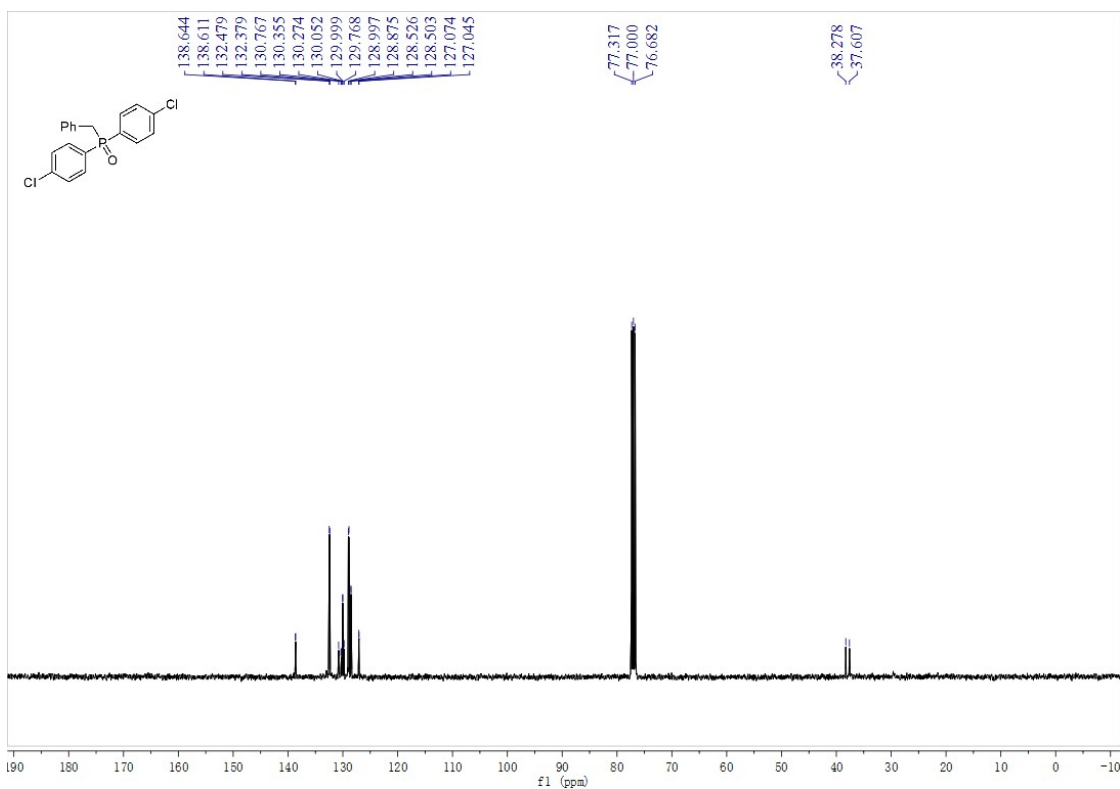
¹³C NMR spectra of di([1,1'-biphenyl]-4-yl)(benzyl)phosphine oxide (**21d**).



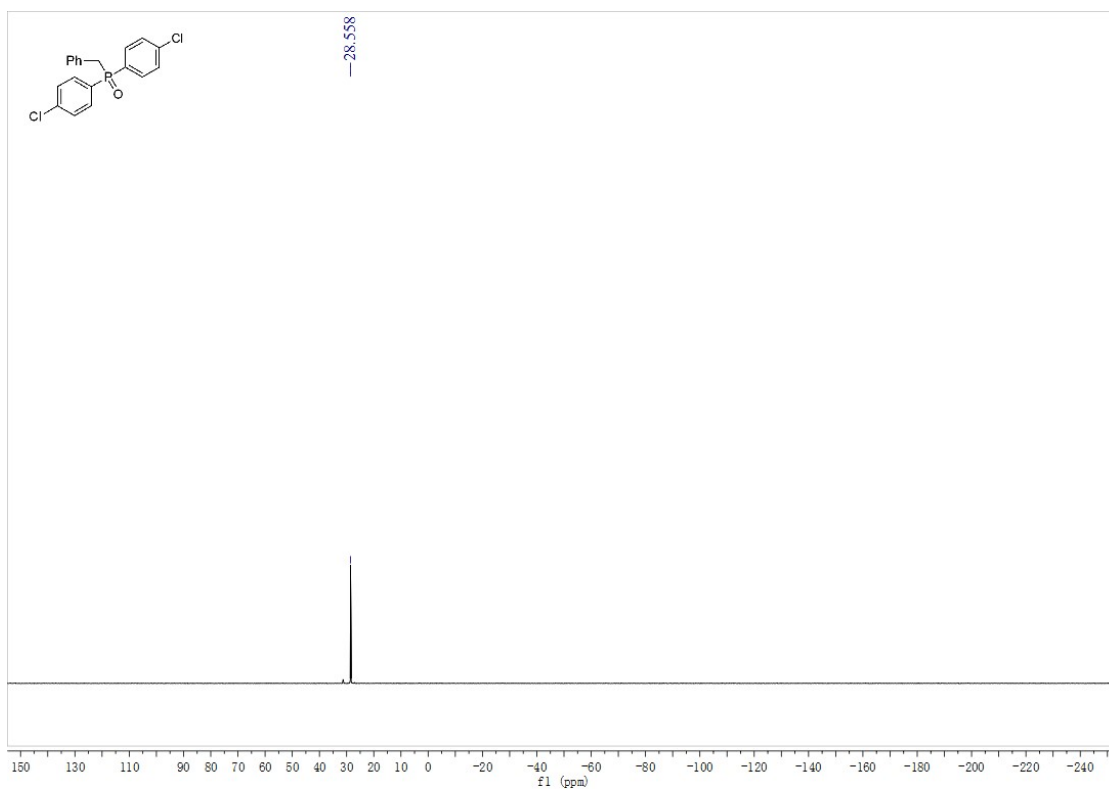
³¹P NMR spectra of di([1,1'-biphenyl]-4-yl)(benzyl)phosphine oxide (**21d**).



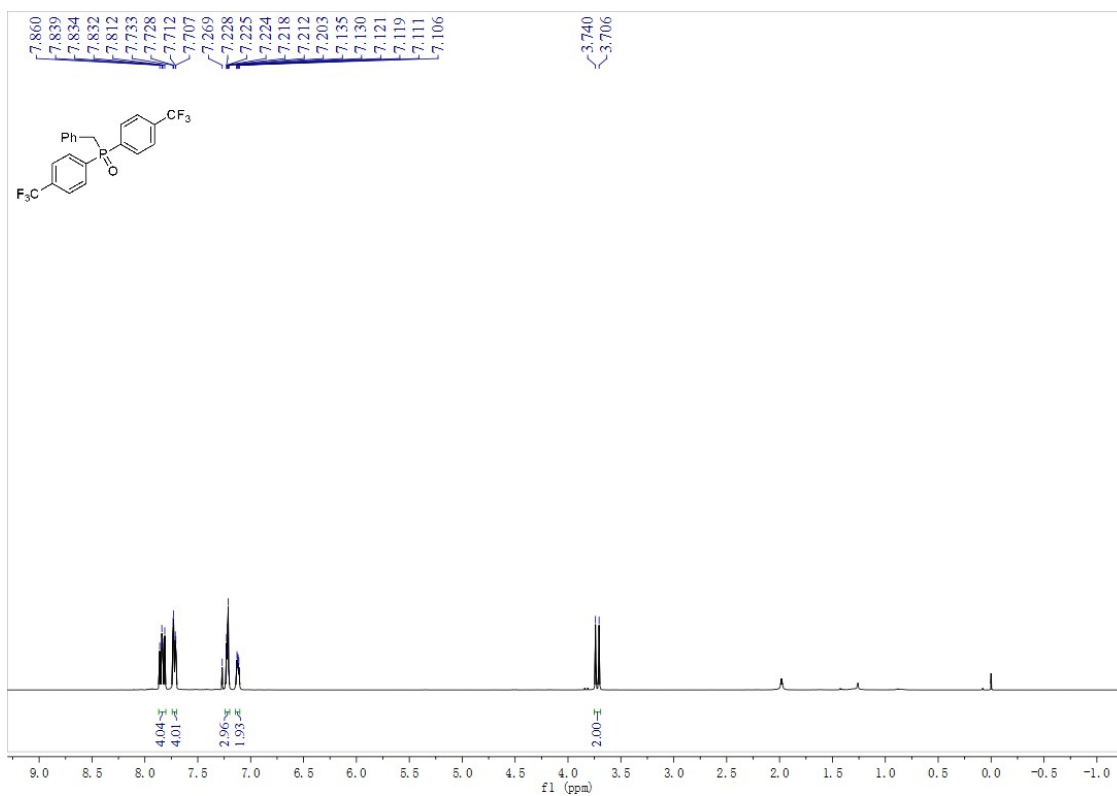
¹H NMR spectra of benzylbis(4-chlorophenyl)phosphine oxide (**22d**).



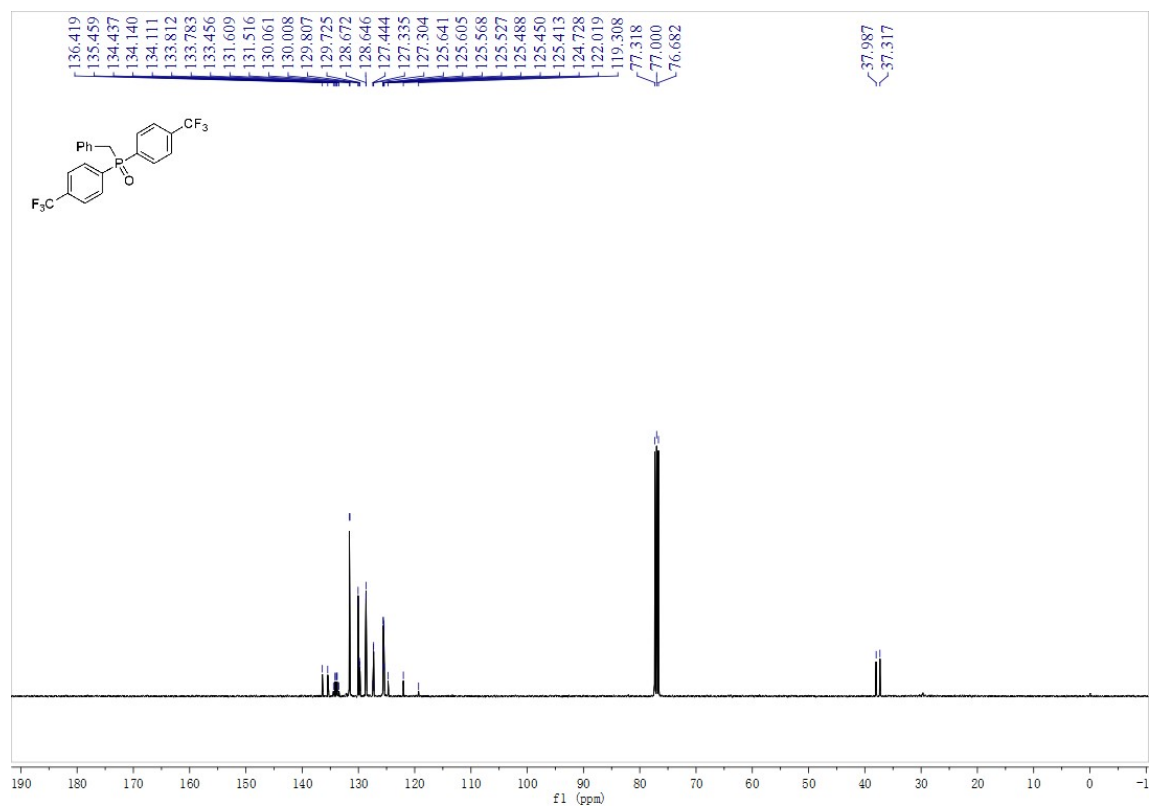
¹³C NMR spectra of benzylbis(4-chlorophenyl)phosphine oxide (**22d**).



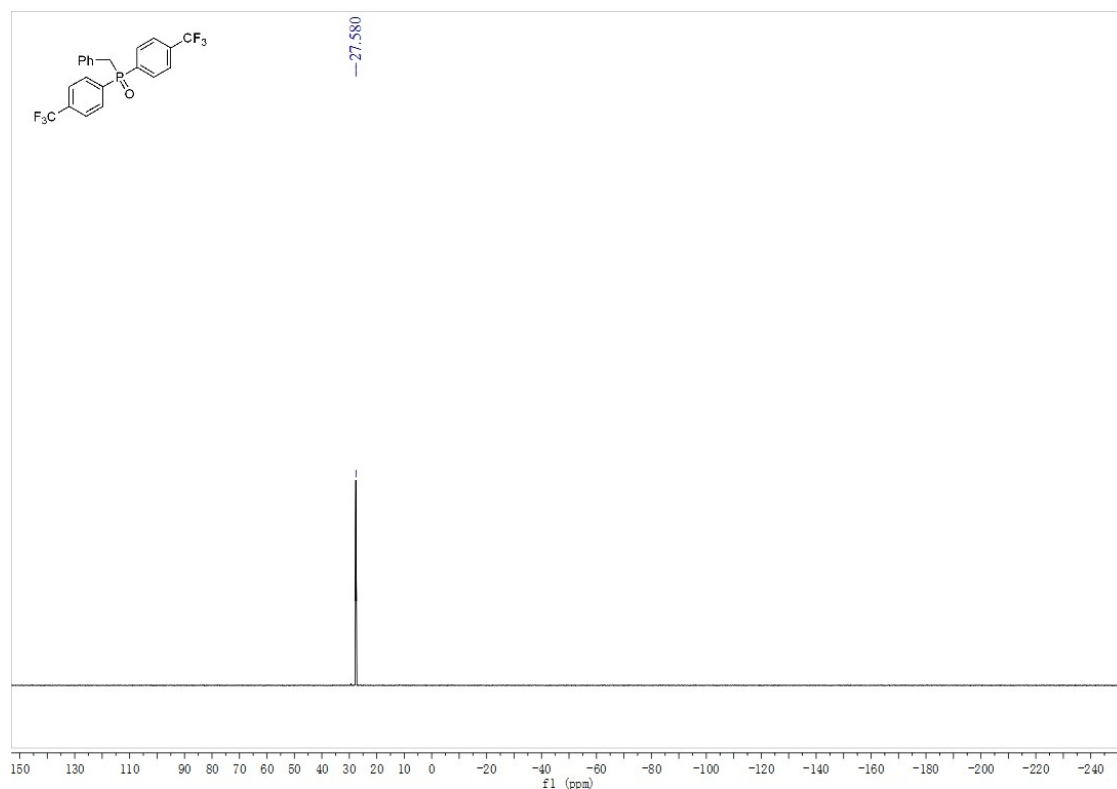
^{31}P NMR spectra of benzylbis(4-chlorophenyl)phosphine oxide (22d).



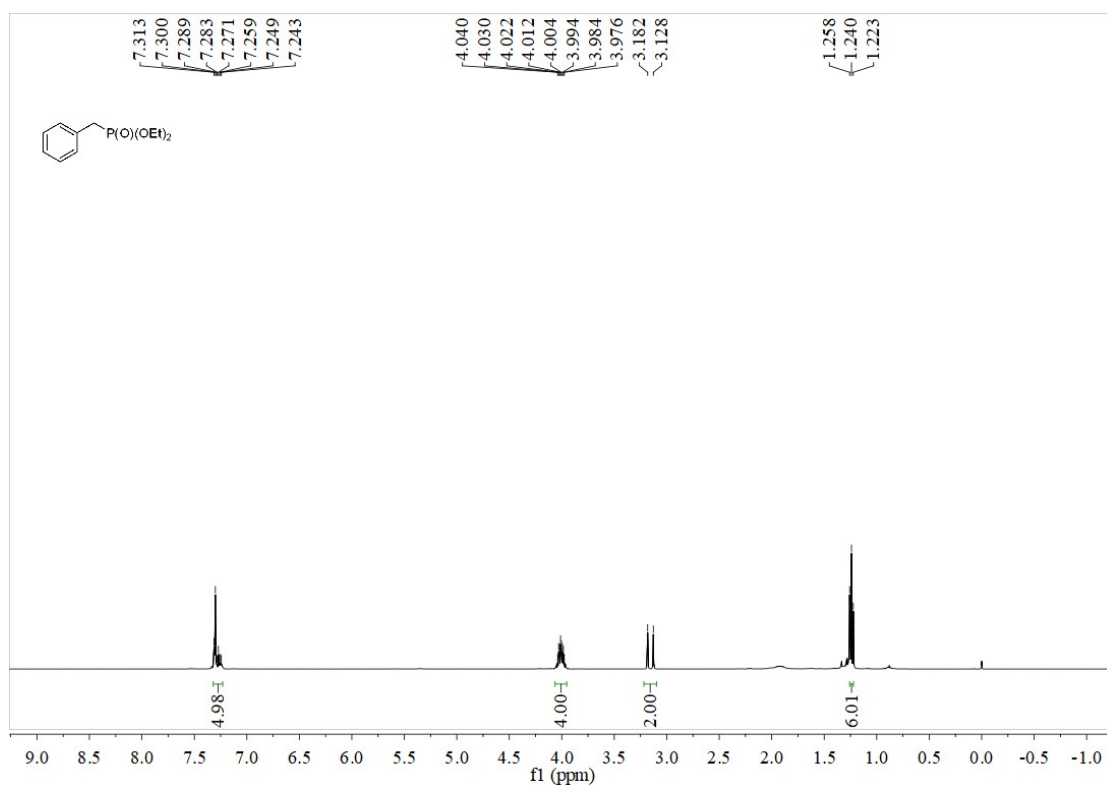
^1H NMR spectra of benzylbis(4-(trifluoromethyl)phenyl)phosphine oxide (23d).



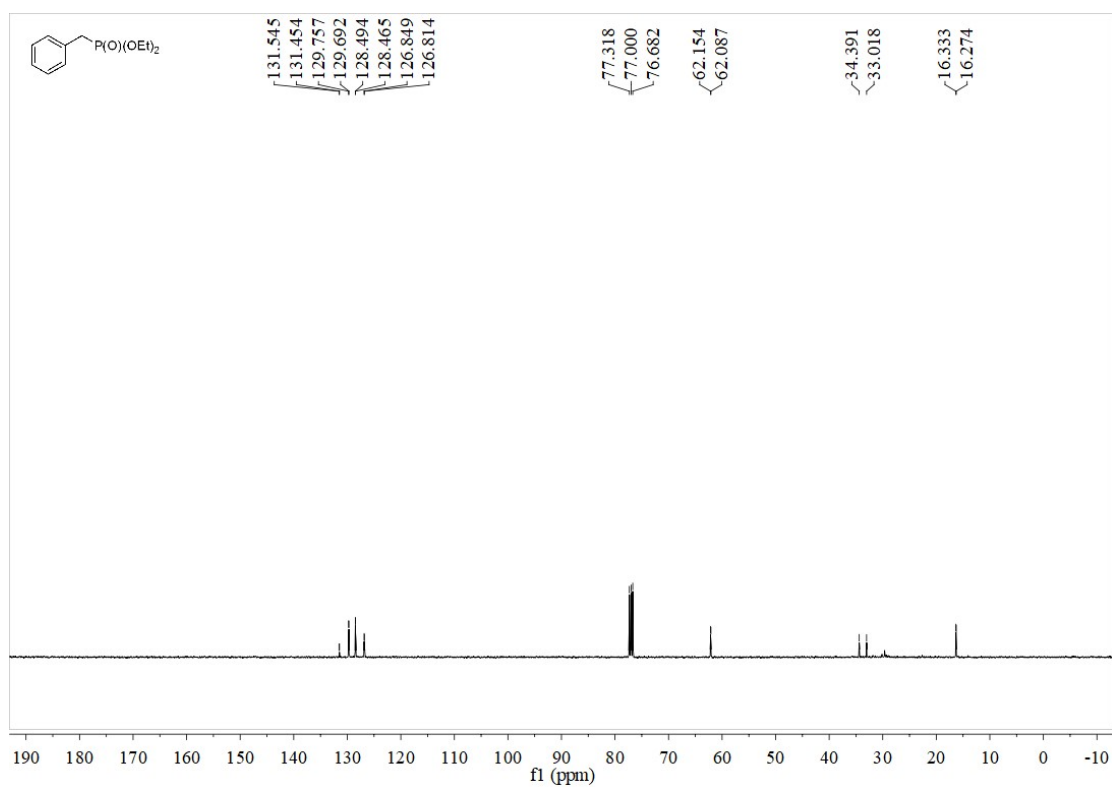
¹³C NMR spectra of benzylbis(4-(trifluoromethyl)phenyl)phosphine oxide (**23d**).



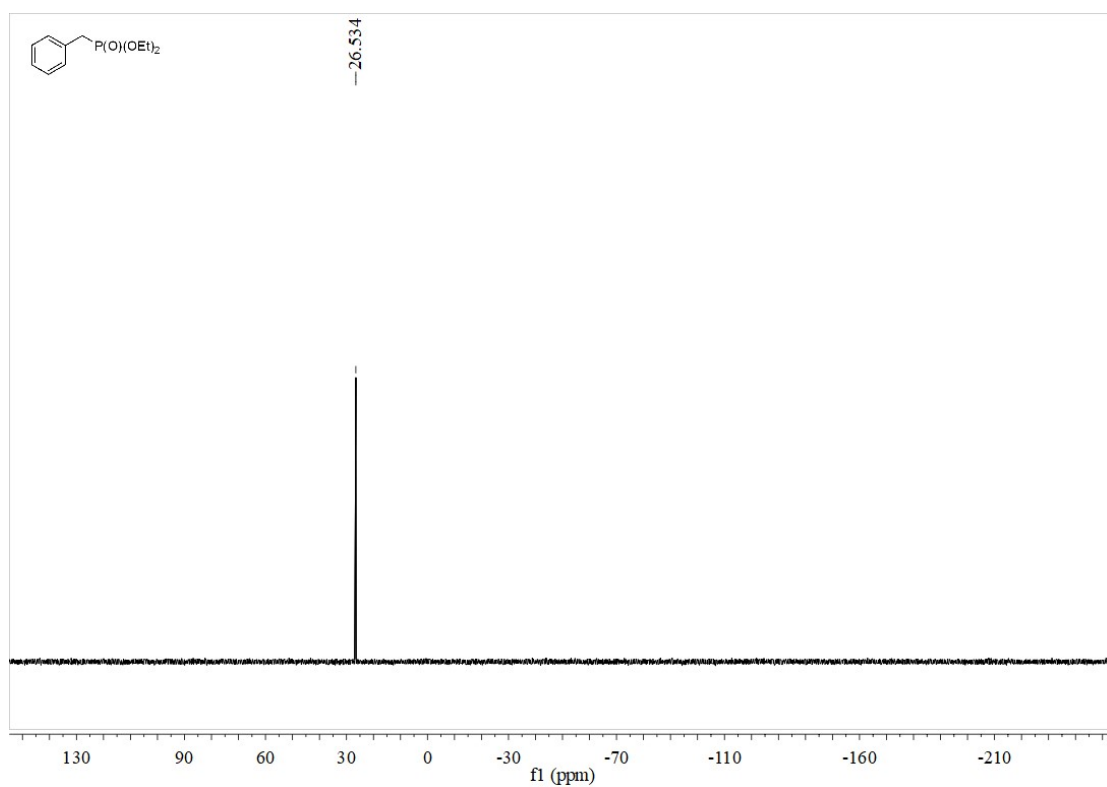
³¹P NMR spectra of benzylbis(4-(trifluoromethyl)phenyl)phosphine oxide (**23d**).



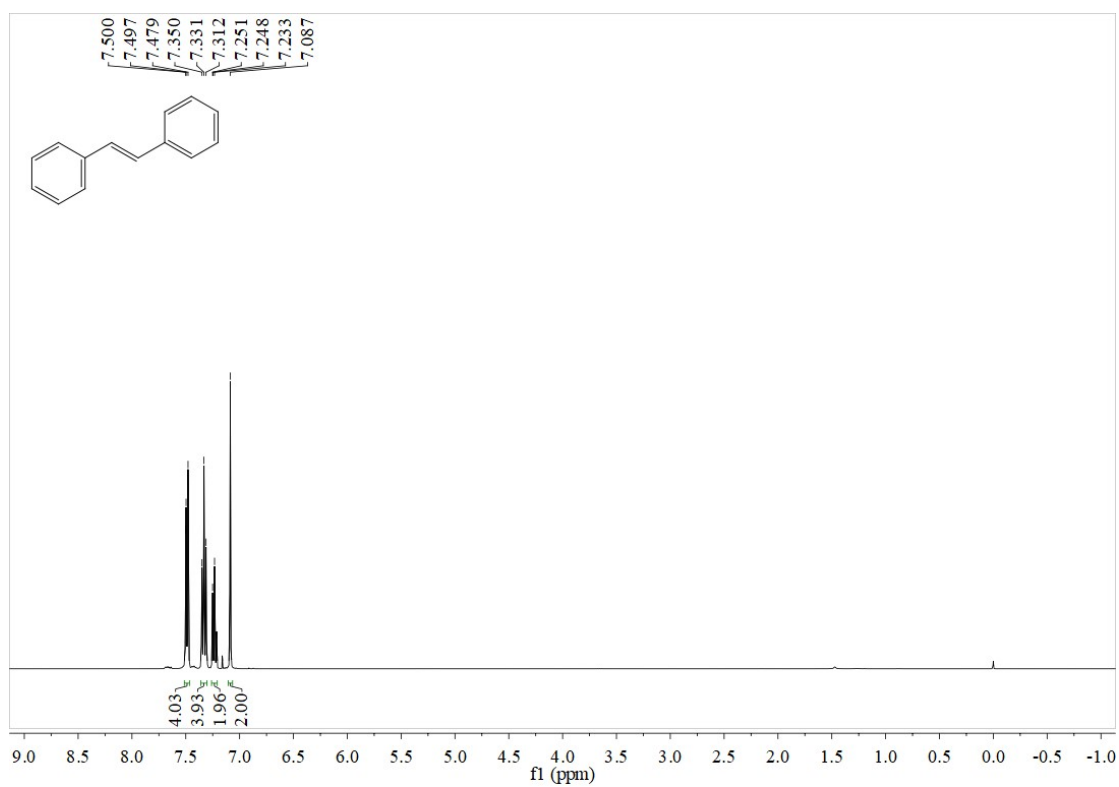
^1H NMR spectra of Diethyl benzylphosphonate (**24d**).



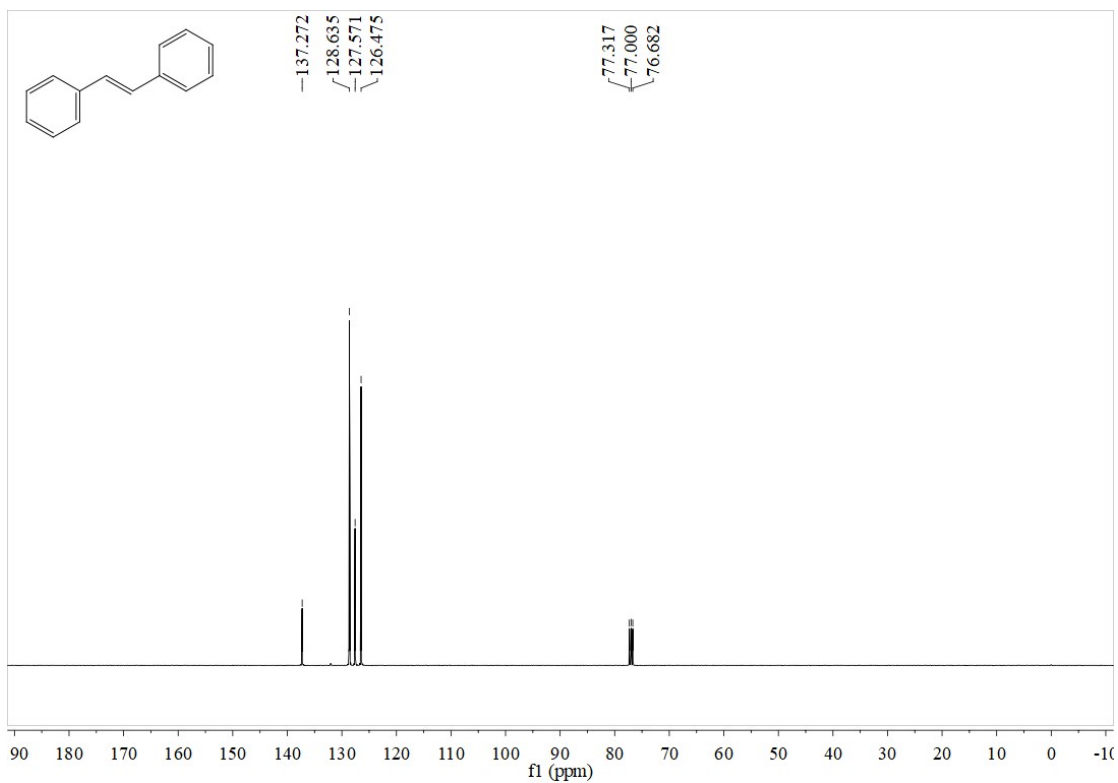
^{13}C NMR spectra of Diethyl benzylphosphonate (**24d**).



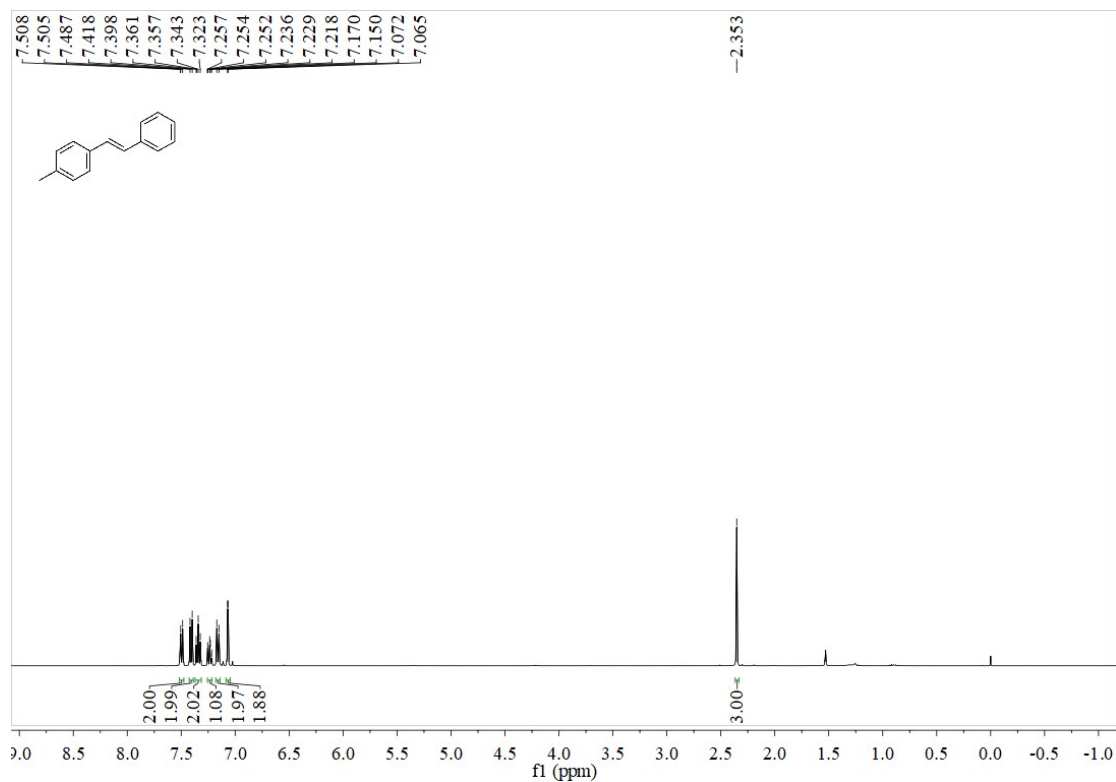
^{31}P NMR spectra of Diethyl benzylphosphonate (**24d**).



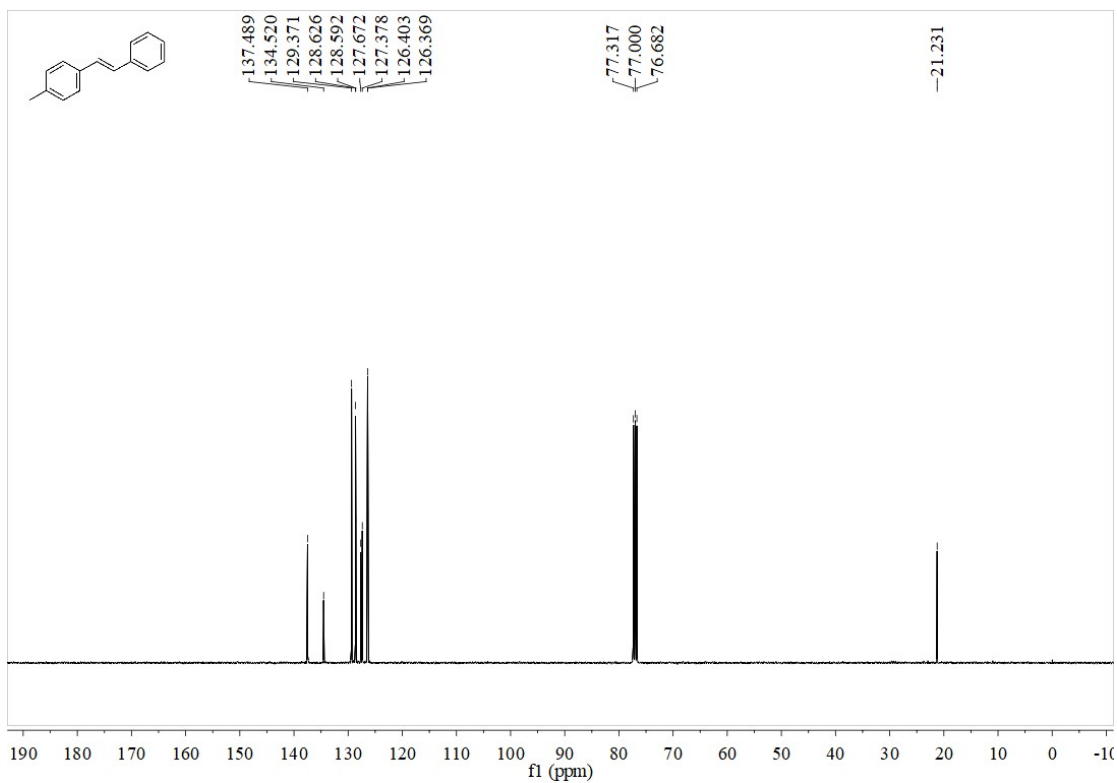
^1H NMR spectra of (E)-1,2-diphenylethene (**1e**).



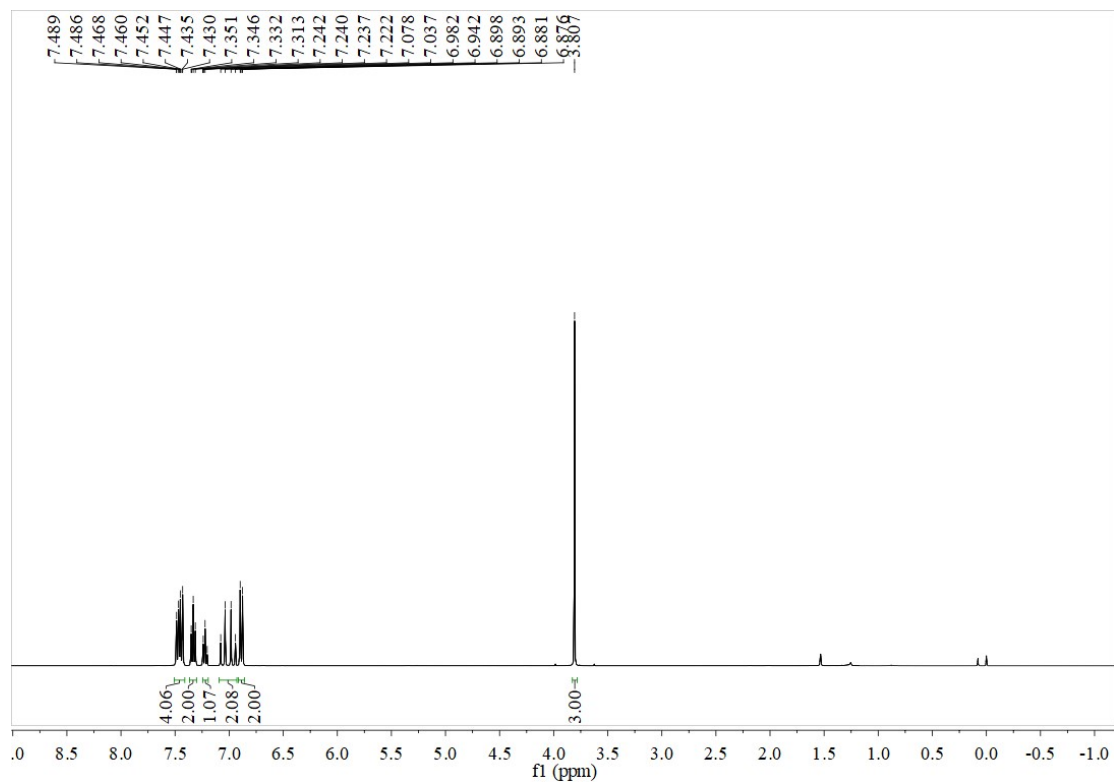
¹³C NMR spectra of (E)-1,2-diphenylethene (**1e**).



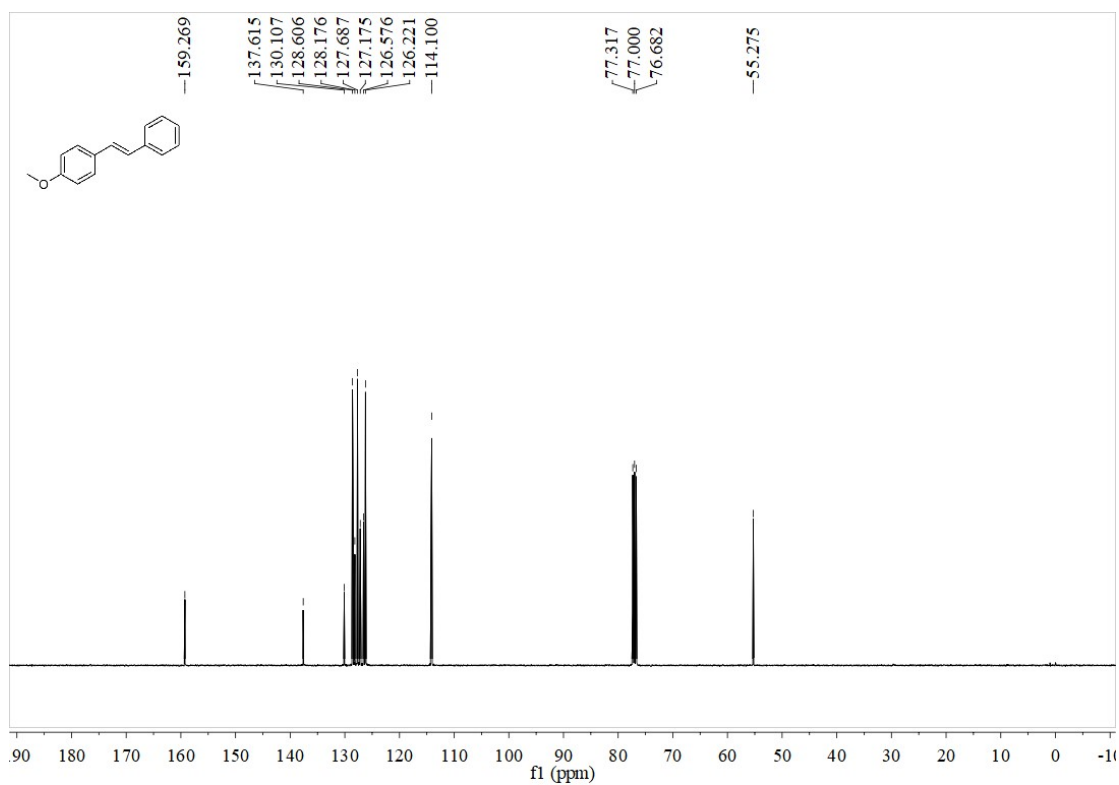
¹H NMR spectra of (E)-1-methyl-4-styrylbenzene (**2e**)



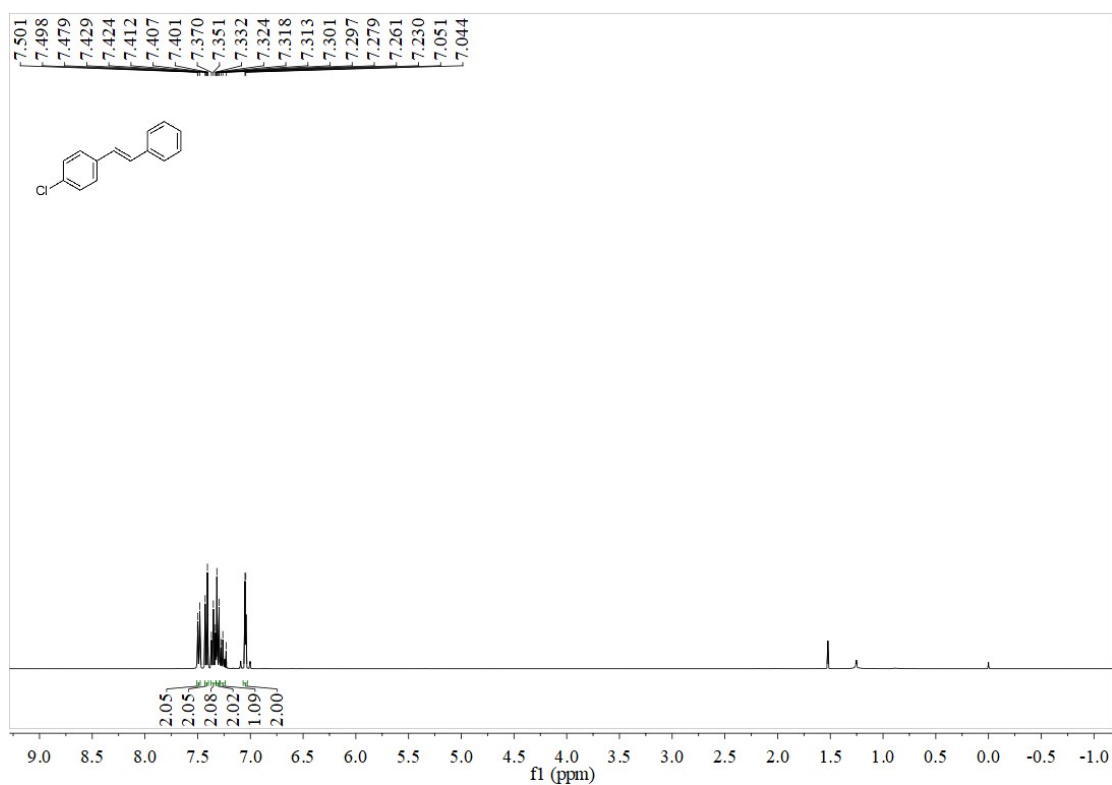
¹³C NMR spectra of (E)-1-methyl-4-styrylbenzene (**2e**)



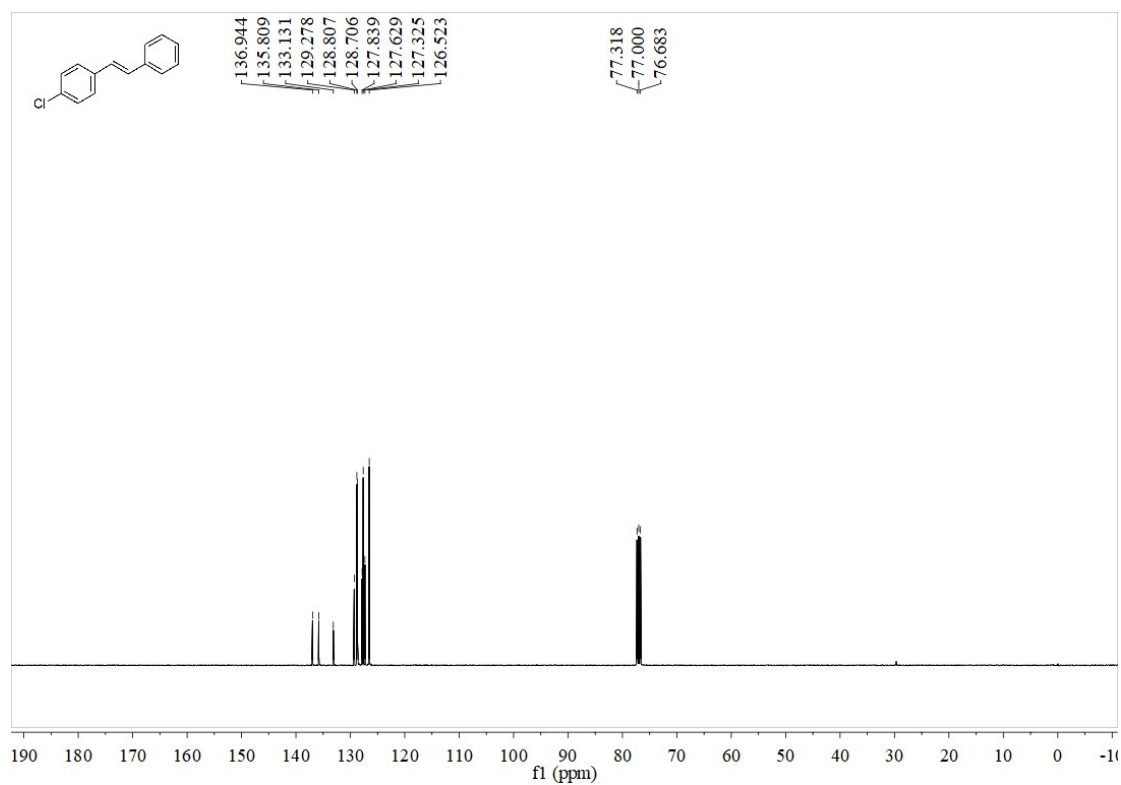
¹H NMR spectra of (E)-1-methoxy-4-styrylbenzene (**3e**)



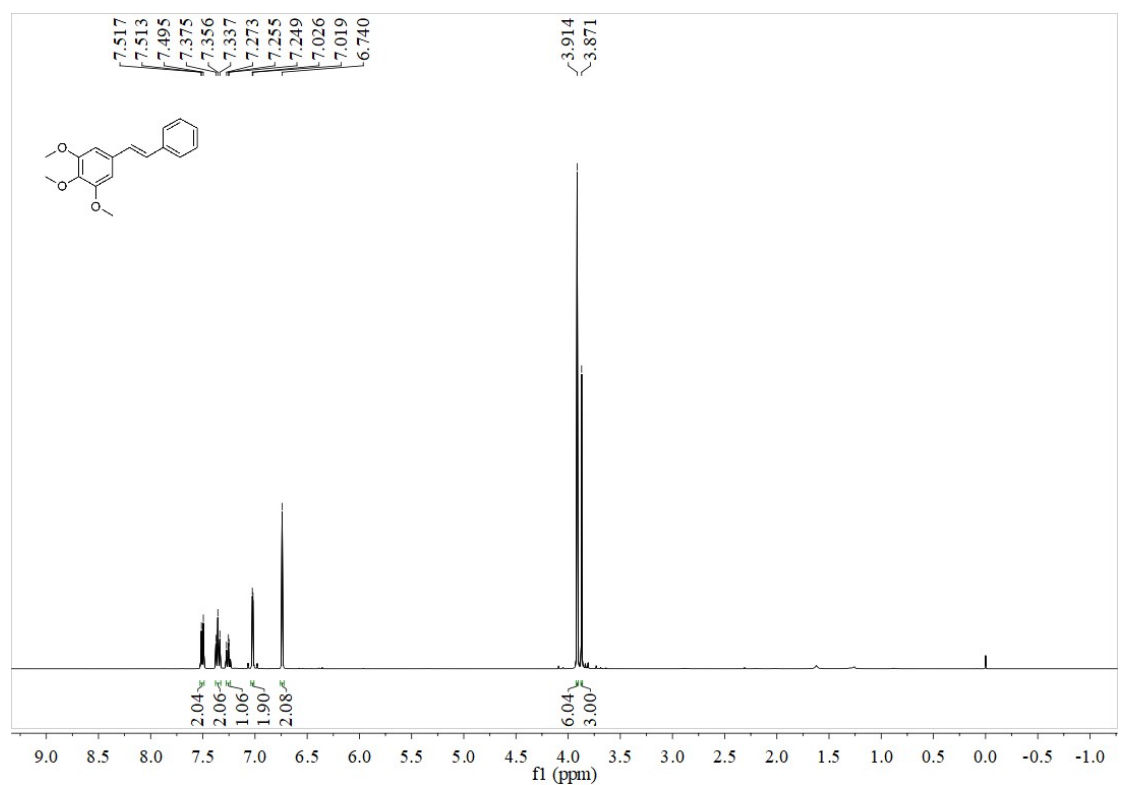
¹³C NMR spectra of (E)-1-methoxy-4-styrylbenzene (**3e**)



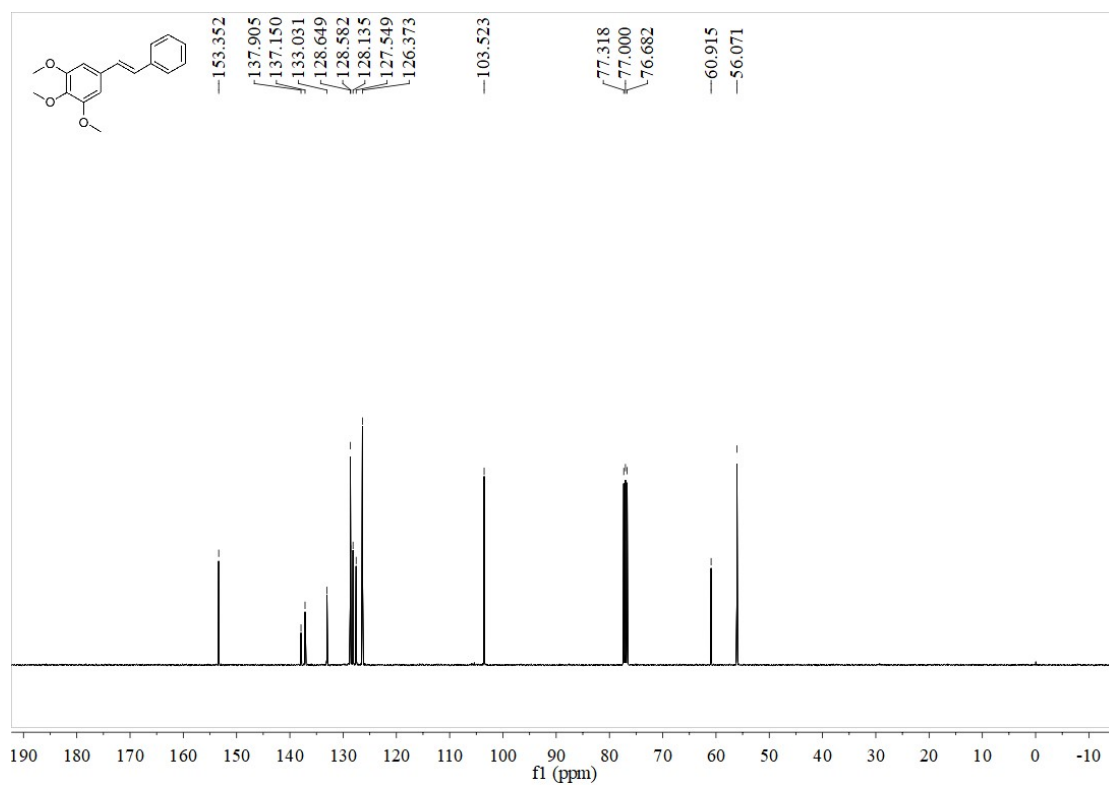
¹H NMR spectra of (E)-1-chloro-4-styrylbenzene (**4e**)



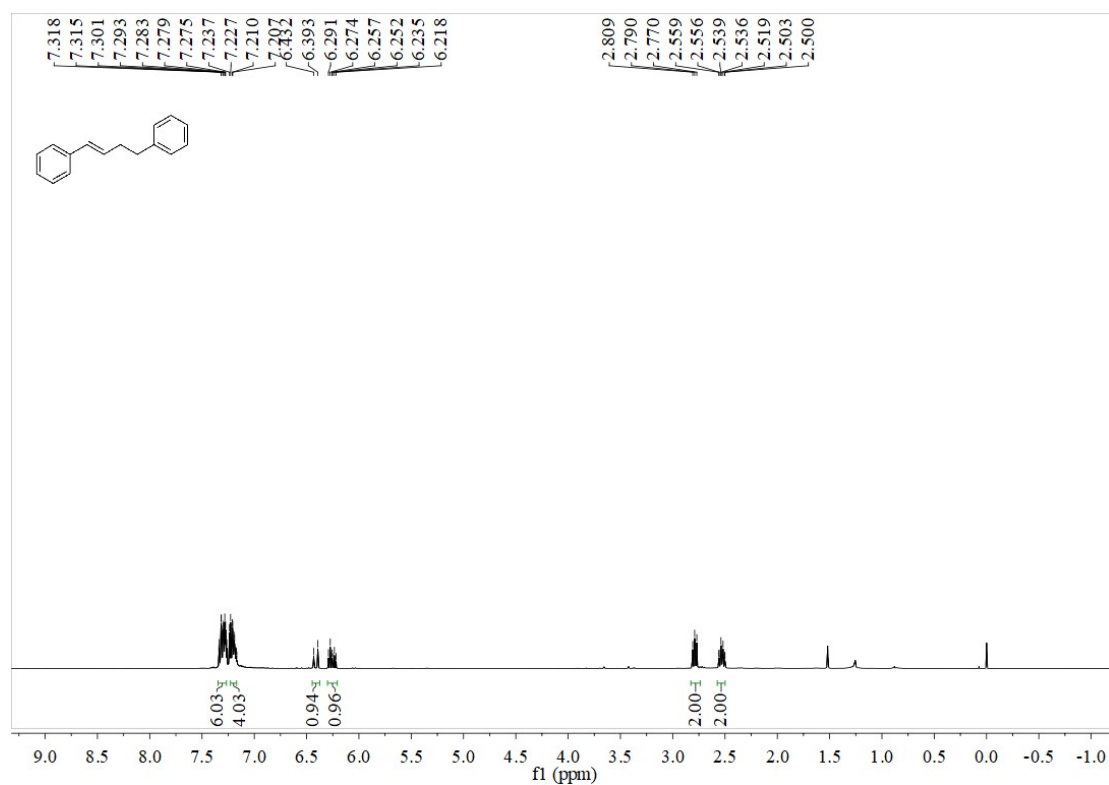
¹³C NMR spectra of (E)-1-chloro-4-styrylbenzene (**4e**)



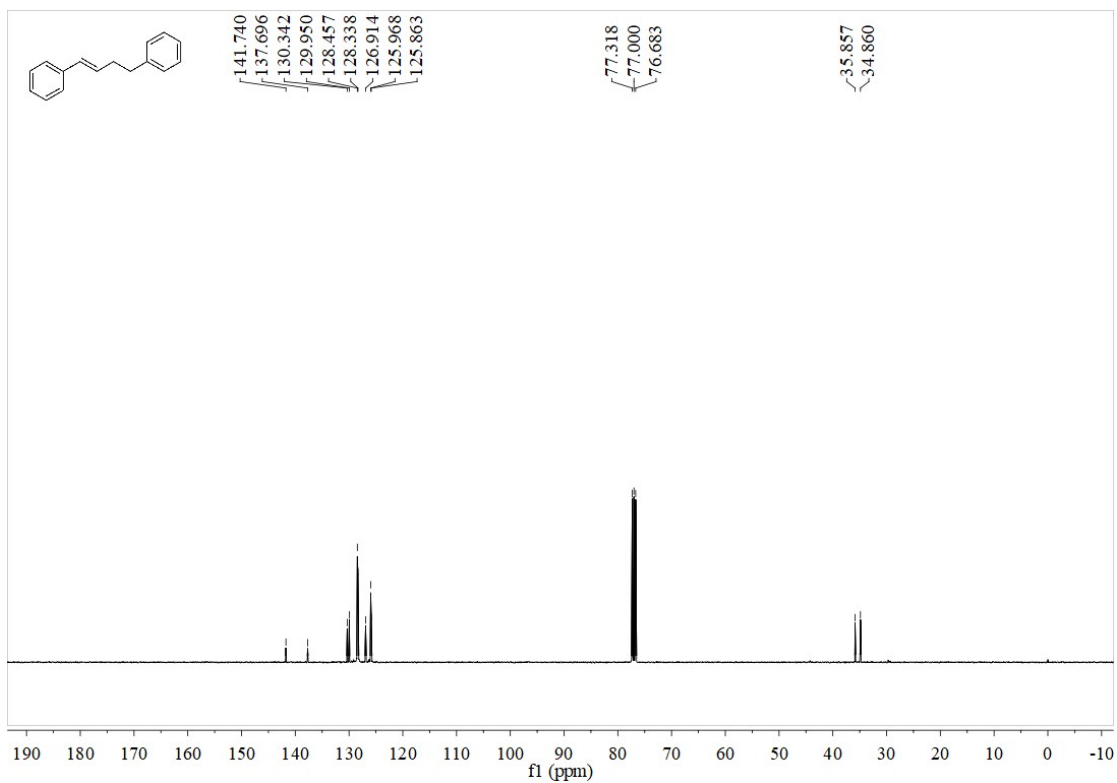
¹H NMR spectra of (E)-1,2,3-trimethoxy-5-styrylbenzene (**5e**)



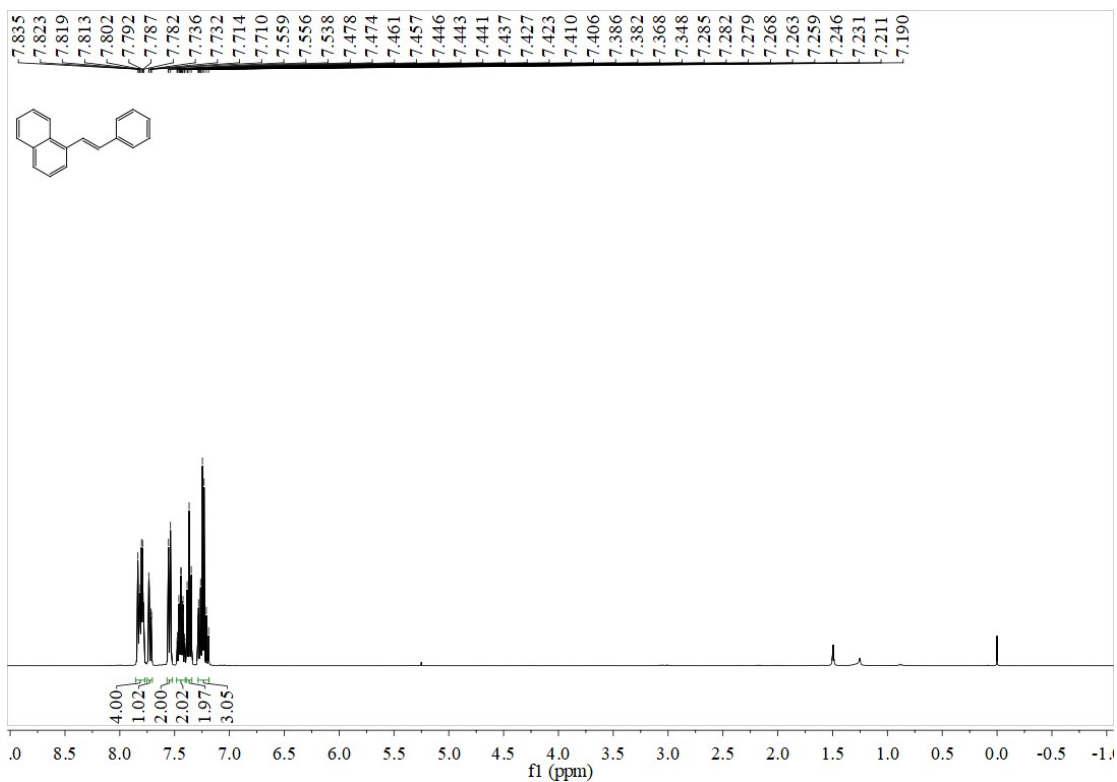
¹³C NMR spectra of (E)-1,2,3-trimethoxy-5-styrylbenzene (**5e**)



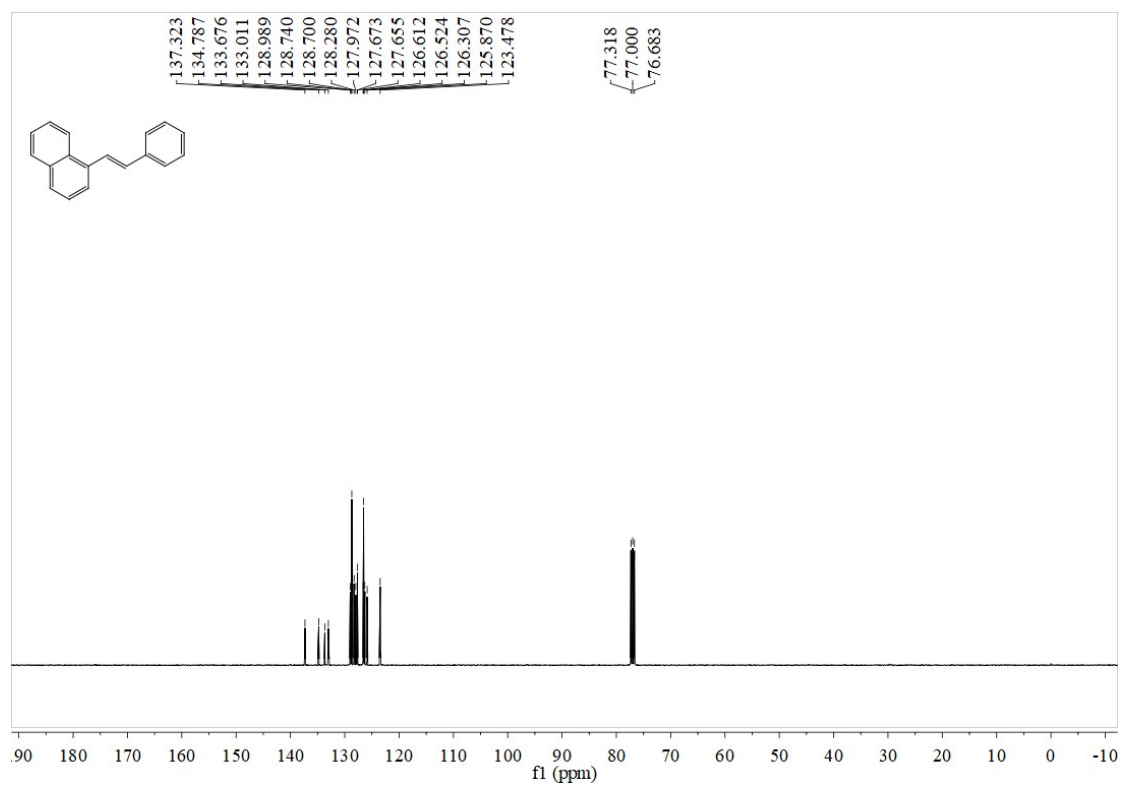
¹H NMR spectra of (E)-but-1-ene-1,4-diylidibenzene (**6e**)



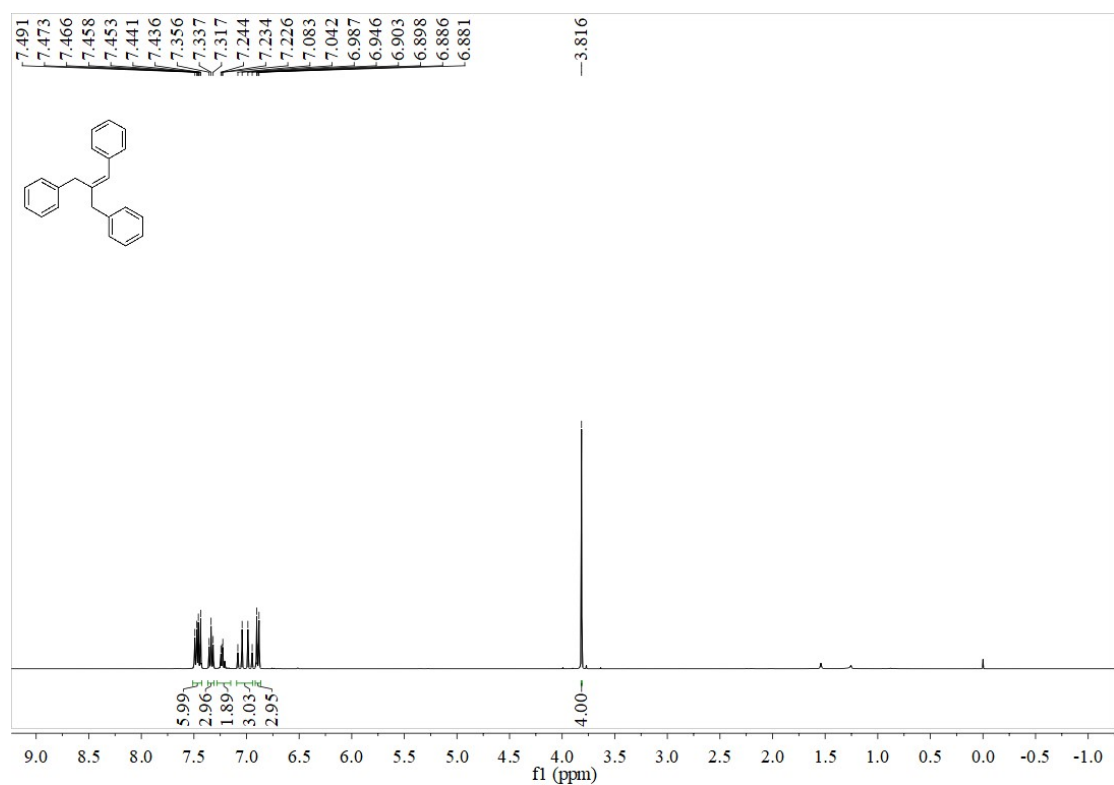
¹³C NMR spectra of (E)-but-1-ene-1,4-diylidibenzene (**6e**)



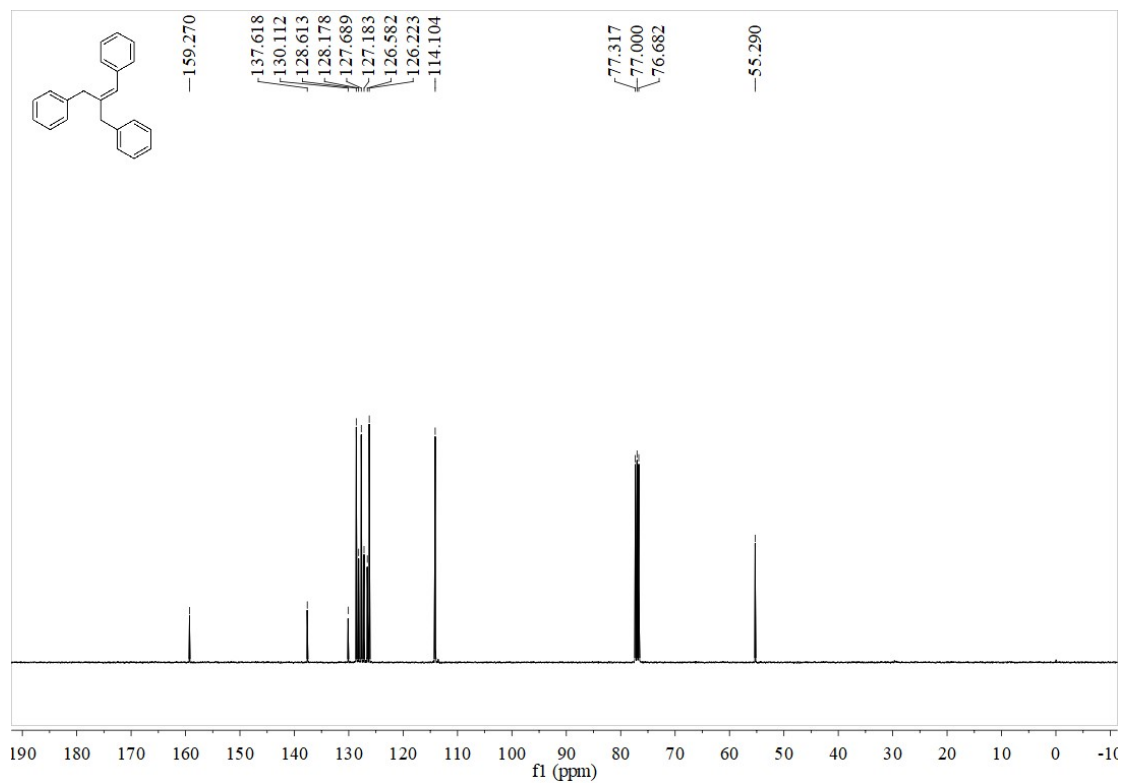
¹H NMR spectra of (E)-1-styrylnaphthalene (**7e**)



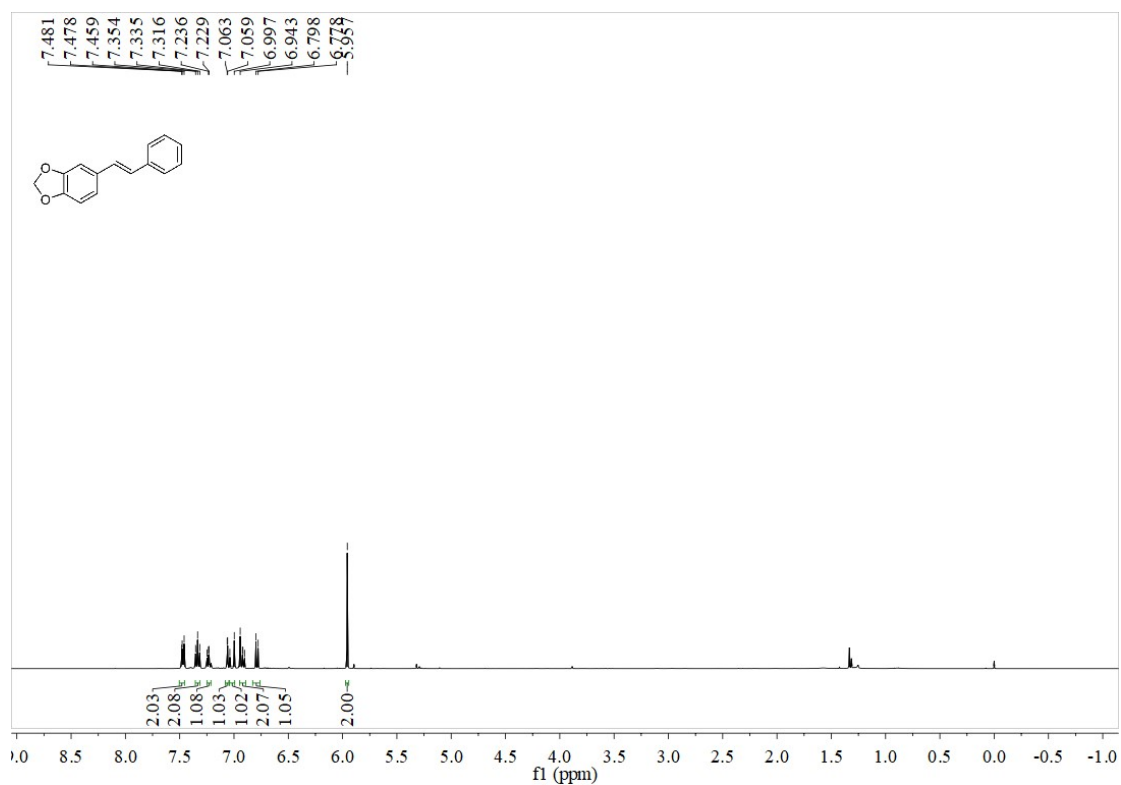
¹³C NMR spectra of (E)-1-styrylnaphthalene (**7e**)



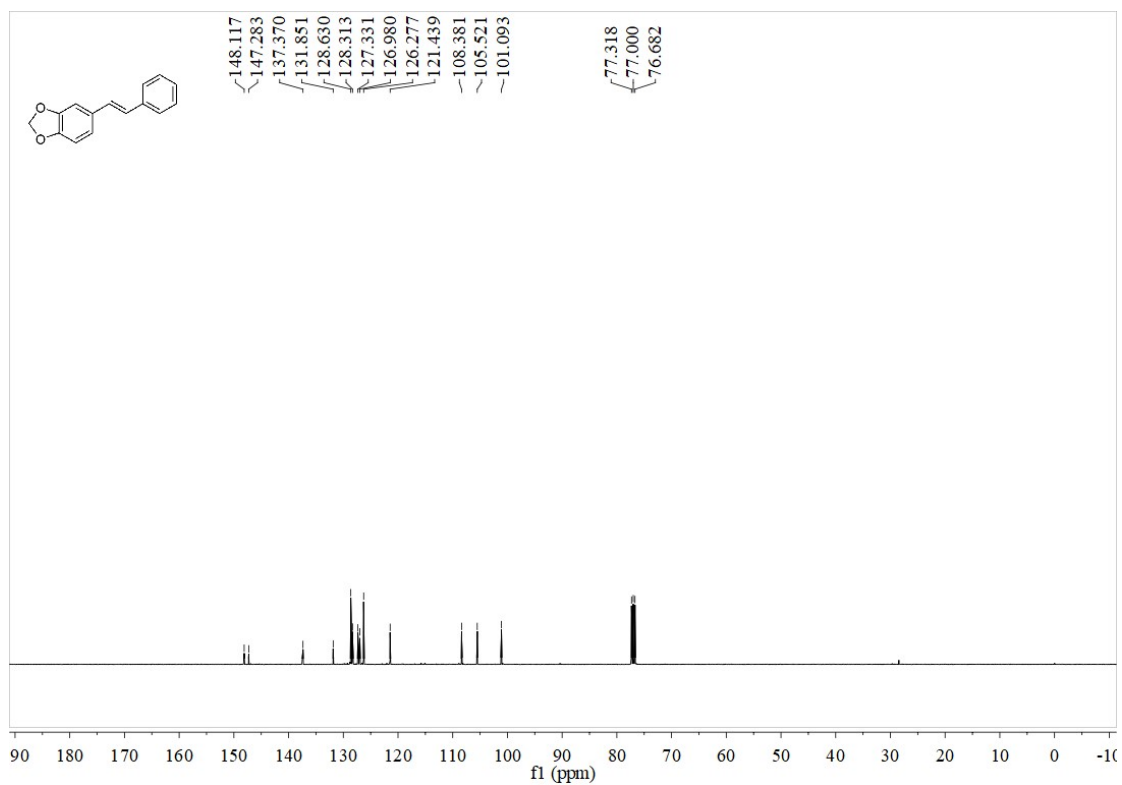
¹H NMR spectra of (2-benzylprop-1-ene-1,3-diyl)dibenzene (**8e**)



¹³C NMR spectra of (2-benzylprop-1-ene-1,3-diyl)dibenzene (**8e**)



¹H NMR spectra of (E)-5-styrylbenzo[d][1,3]dioxole (**9e**)



^{13}C NMR spectra of (E)-5-styrylbenzo[d][1,3]dioxole (**9e**)