

Electronic Supplementary Information For

Water mediated synthesis of dialkylphosphine oxides from white phosphorus and N-(acyloxy)phthalimides

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1. General information

General. ^1H , ^{13}C and ^{31}P NMR spectra were recorded on Bruker-500 or 600 spectrometer. ^1H and ^{13}C NMR chemical shifts were determined relative to internal standard TMS at 0.0, CDCl_3 ($\delta(^1\text{H})$, 7.26 ppm; $\delta(^{13}\text{C})$, 77.16 ppm). Chemical shifts (δ) are reported in ppm, and coupling constants (J) are in Hertz (Hz). The following abbreviations are used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Analytical thin layer chromatography (TLC) was performed on 0.25 mm silica gel 60 F254 plates and viewed by UV light (254 nm). Column chromatographic purification was performed using 200-300 mesh silica gel. All products were firstly examined by Bruker AmaZon SL ESI-IT-MS (Bruker Daltonics Inc., Germany) in positive ion mode, then further characterized by HRMS (ESI-qTOF MS, Bruker micrOTOF-Q II) in positive ion mode too.

2. Safety note for P_4

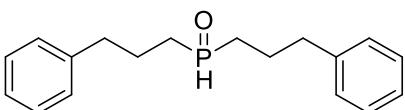
White phosphorus is spontaneously flammable; it should be stored in water or glove box. White phosphorus-toluene solution should be sealed in argon and stored away from light.

3. Preparation of P_4 -toluene solution

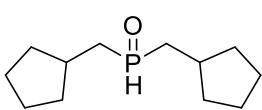
A piece of P_4 was taken out of water and then put in ethanol under argon. Two minutes later, P_4 was taken out and the ethanol on the surface of P_4 was blown away by the argon. Then, the dry P_4 was put in a conical flask containing toluene. The mixture was stirred intensely with a magnetic stirrer until P_4 was completely dissolved in the toluene. (the concentration of P_4 was determined by ^{31}P NMR analysis using triphenyl phosphate ($\text{C}_6\text{H}_5\text{O})_3\text{P}(\text{O})$ as an internal standard, D1 = 20 s, zg30, LB = 1).

4. Characterization of the target products

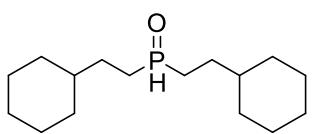
bis(3-phenylpropyl)phosphine oxide (3a)

 Pale yellow oil. $V_{\text{DCM}} : V_{\text{MeOH}} = 20:1$, Yield: 51.2 mg, 90%. ^1H NMR (600 MHz, CDCl_3) δ 7.28 (t, $J = 7.5$ Hz, 4H), 7.20 (t, $J = 7.4$ Hz, 2H), 7.15 (d, $J = 7.3$ Hz, 4H), 6.82 (d, $J = 442.5$ Hz, 1H), 2.72 (t, $J = 7.3$ Hz, 4H), 1.99-1.93 (m, 2H), 1.92-1.86 (m, 2H), 1.82-1.75 (m, 2H), 1.74-1.68 (m, 2H). ^{13}C NMR (150 MHz, CDCl_3) δ 140.6, 128.7, 128.6, 126.4, 36.6 (d, $J = 14.1$ Hz), 27.7 (d, $J = 65.5$ Hz), 23.5 (d, $J = 3.3$ Hz). ^{31}P NMR (242 MHz, CDCl_3) δ 34.07 (d, $J = 452.2$ Hz). HRMS (ESI-TOF) m/z: $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{18}\text{H}_{23}\text{OPNa}^+$ 309.1379, found 309.1375.

bis(cyclopentylmethyl)phosphine oxide (3b)

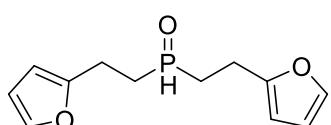
 Pale yellow oil. Eluent $V_{\text{DCM}} : V_{\text{MeOH}} = 20:1$, Yield: 31.1 mg, 73%. ^1H NMR (600 MHz, CDCl_3) δ 6.93 (d, $J = 445.9$ Hz, 1H), 2.22-2.14 (m, 2H), 1.99-1.91 (m, 6H), 1.76-1.71 (m, 2H), 1.68-1.62 (m, 4H), 1.58-1.53 (m, 4H), 1.24-1.18 (m, 4H). ^{13}C NMR (150 MHz, CDCl_3) δ 35.2 (d, $J = 64.4$ Hz), 34.4 (d, $J = 9.1$ Hz), 34.2 (d, $J = 4.3$ Hz), 33.8 (d, $J = 7.7$ Hz), 24.9, 24.8. ^{31}P NMR (242 MHz, CDCl_3) δ 31.36 (d, $J = 446.7$ Hz). HRMS (ESI-TOF) m/z: $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{12}\text{H}_{24}\text{OP}^+$ 215.1559, found 215.1559.

bis(2-cyclohexylethyl)phosphine oxide (3c)



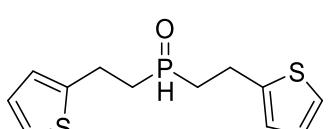
Colorless oil. Eluent $V_{\text{DCM}} : V_{\text{MeOH}} = 20:1$, Yield: 37.6 mg, 70%. ^1H NMR (600 MHz, CDCl_3) δ 6.81 (d, $J = 447.2$ Hz, 1H), 1.86-1.64 (m, 14H), 1.56-1.40 (m, 4H), 1.31-1.26 (m, 2H), 1.25-1.19 (m, 4H), 1.17-1.10 (m, 2H), 0.93-0.87 (m, 4H). ^{13}C NMR (150 MHz, CDCl_3) δ 38.6 (d, $J = 13.4$ Hz), 32.9 (d, $J = 14.2$ Hz), 29.0 (d, $J = 3.5$ Hz), 26.6, 26.3, 25.7 (d, $J = 65.2$ Hz). ^{31}P NMR (242 MHz, CDCl_3) δ 37.32 (d, $J = 450.7$ Hz). HRMS (ESI-TOF) m/z: $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{16}\text{H}_{32}\text{OP}^+$ 271.2185, found 271.2189.

bis(2-(furan-2-yl)ethyl)phosphine oxide (3d)



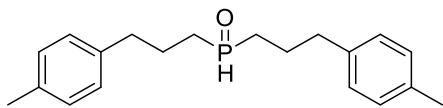
Colorless oil. Eluent $V_{\text{DCM}} : V_{\text{MeOH}} = 20:1$, Yield: 34.5 mg, 73%. ^1H NMR (600 MHz, CDCl_3) δ 7.30 (d, $J = 1.2$ Hz, 2H), 6.85 (d, $J = 459.7$ Hz, 1H), 6.27 (t, $J = 2.5$ Hz, 2H), 6.06 (d, $J = 3.1$ Hz, 2H), 3.04-2.92 (m, 4H), 2.17-2.04 (m, 4H). ^{13}C NMR (150 MHz, CDCl_3) δ 153.2 (d, $J = 12.2$ Hz), 141.7, 110.5, 106.2, 26.8 (d, $J = 65.4$ Hz), 20.5 (d, $J = 2.7$ Hz). ^{31}P NMR (242 MHz, CDCl_3) δ 30.69 (d, $J = 457.7$ Hz). HRMS (ESI-TOF) m/z: $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{12}\text{H}_{15}\text{O}_3\text{PNa}^+$ 261.0651, found 261.0651.

bis(2-(thiophen-2-yl)ethyl)phosphine oxide (3e)



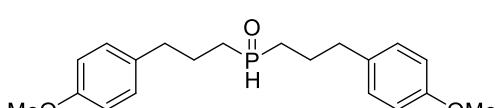
Colorless oil. $V_{\text{DCM}} : V_{\text{MeOH}} = 20:1$, Yield: 36.4 mg, 68%. ^1H NMR (600 MHz, CDCl_3) δ 7.15 (d, $J = 5.1$ Hz, 2H), 6.92 (dd, $J = 5.1, 3.5$ Hz, 2H), 6.89 (d, $J = 458.3$ Hz, 1H), 6.85 (d, $J = 3.1$ Hz, 2H), 3.27-3.14 (m, 4H), 2.25-2.16 (m, 2H), 2.12-2.03 (m, 2H). ^{13}C NMR (150 MHz, CDCl_3) δ 142.7 (d, $J = 13.3$ Hz), 127.2, 125.2, 124.1, 30.6 (d, $J = 64.1$ Hz), 22.4 (d, $J = 3.3$ Hz). ^{31}P NMR (242 MHz, CDCl_3) δ 29.39 (d, $J = 458.2$ Hz). HRMS (ESI-TOF) m/z: $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{12}\text{H}_{15}\text{OPS}_2\text{Na}^+$ 293.0194, found 293.0200.

bis(3-(p-tolyl)propyl)phosphine oxide (3f)



Pale yellow oil. $V_{\text{DCM}} : V_{\text{MeOH}} = 20:1$, Yield: 53.5 mg, 85%. ^1H NMR (600 MHz, CDCl_3) δ 7.06 (d, $J = 8.7$ Hz, 4H), 6.81 (d, $J = 449.3$ Hz, 1H), 6.82 (d, $J = 8.5$ Hz, 4H), 3.78 (s, 6H), 2.66 (t, $J = 7.3$ Hz, 4H), 1.97-1.89 (m, 2H), 1.88-1.82 (m, 2H), 1.78-1.74 (m, 2H), 1.73-1.70 (m, 2H). ^{13}C NMR (150 MHz, CDCl_3) δ 158.2, 132.7, 129.5, 114.1, 55.4, 35.7 (d, $J = 14.5$ Hz), 27.6 (d, $J = 65.4$ Hz), 23.7 (d, $J = 3.3$ Hz). ^{31}P NMR (242 MHz, CDCl_3) δ 33.85 (d, $J = 465.1$ Hz). HRMS (ESI-TOF) m/z: $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{20}\text{H}_{28}\text{OP}^+$ 315.1872, found 315.1872.

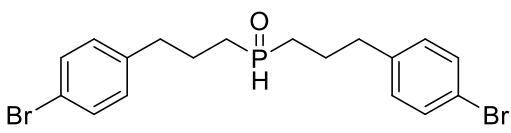
bis(3-(4-methoxyphenyl)propyl)phosphine oxide (3g)



White wax. $V_{\text{DCM}} : V_{\text{MeOH}} = 20:1$, Yield: 61.0 mg, 88%. ^1H NMR (600 MHz, CDCl_3) δ 7.06 (d, $J = 8.5$ Hz, 4H), 6.82 (t, $J = 8.6$ Hz, 4H), 6.81 (d, $J = 448.3$ Hz, 1H), 3.78 (s, 6H), 2.65 (t, $J = 7.3$ Hz, 4H), 1.97-1.88 (m, 2H), 1.87-1.80 (m, 2H), 1.80-1.73 (m, 2H), 1.73-1.65 (m, 2H). ^{13}C NMR (150 MHz, CDCl_3) δ 158.1, 132.7, 129.5, 114.0, 55.3, 35.7 (d, $J = 14.5$ Hz), 27.6 (d, $J = 65.3$ Hz), 23.7 (d, $J = 3.2$ Hz). ^{31}P NMR (242 MHz, CDCl_3) δ 34.42 (d, $J = 449.1$ Hz). HRMS (ESI-TOF) m/z: $[\text{M}+\text{H}]^+$

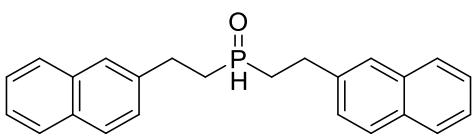
Calcd for C₂₀H₂₈O₃P⁺ 347.1771, found 347.1768.

bis(3-(4-bromophenyl)propyl)phosphine oxide (3h)



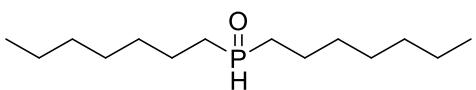
Pale yellow oil. $V_{DCM} : V_{MeOH} = 20:1$, Yield: 70.5 mg, 80%. ¹H NMR (600 MHz, CDCl₃) δ 7.41 (d, $J = 8.3$ Hz, 4H), 7.03 (d, $J = 8.5$ Hz, 4H), 6.85 (d, $J = 449.1$ Hz, 1H), 2.69 (t, $J = 7.4$ Hz, 4H), 2.01-1.84 (m, 4H), 1.83-1.69 (m, 4H). ¹³C NMR (150 MHz, CDCl₃) δ 139.6, 131.7, 130.3, 120.2, 36.0 (d, $J = 13.6$ Hz), 27.7 (d, $J = 65.7$ Hz), 23.5 (d, $J = 3.3$ Hz). ³¹P NMR (242 MHz, CDCl₃) δ 33.59 (d, $J = 456.3$ Hz). HRMS (ESI-TOF) m/z: [M+Na]⁺ Calcd for C₁₈H₂₁Br₂OPNa⁺ 464.9589, found 464.9588.

bis(2-(naphthalen-2-yl)ethyl)phosphine oxide (3i)



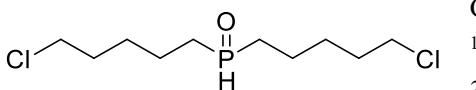
Pale yellow oil. $V_{DCM} : V_{MeOH} = 20:1$, Yield: 58.9 mg, 83%. ¹H NMR (600 MHz, CDCl₃) δ 7.94 (d, $J = 8.4$ Hz, 2H), 7.84 (d, $J = 7.9$ Hz, 2H), 7.72 (d, $J = 8.3$ Hz, 2H), 7.53-7.51 (m, 2H), 7.49-7.46 (m, 2H), 7.37 (t, $J = 7.6$ Hz, 2H), 7.31 (d, $J = 6.7$ Hz, 2H), 6.91 (d, $J = 454.6$ Hz, 1H), 3.48-3.41 (m, 2H), 3.34-3.27 (m, 2H), 2.25-2.17 (m, 2H), 2.09-2.02 (m, 2H). ¹³C NMR (150 MHz, CDCl₃) δ 136.0 (d, $J = 12.3$ Hz), 133.8, 131.1, 128.9, 127.5, 126.3, 126.0, 125.7, 125.5, 123.0, 29.2 (d, $J = 63.8$ Hz), 24.9 (d, $J = 2.9$ Hz). ³¹P NMR (242 MHz, CDCl₃) δ 31.54 (d, $J = 460.3$ Hz). HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for C₂₄H₂₄OP⁺ 359.1559, found 359.1558.

diheptylphosphine oxide (3j)



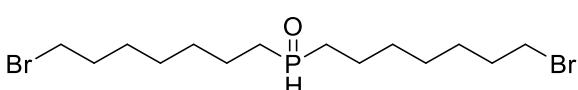
Colorless oil. $V_{DCM} : V_{MeOH} = 20:1$, Yield: 41.6 mg, 85%. ¹H NMR (600 MHz, CDCl₃) δ 6.84 (d, $J = 445.5$ Hz, 1H), 1.86-1.68 (m, 4H), 1.68-1.53 (m, 4H), 1.45-1.37 (m, 4H), 1.33-1.24 (m, 12H), 0.87 (t, $J = 7.1$ Hz, 6H). ¹³C NMR (150 MHz, CDCl₃) δ 31.6, 30.8 (d, $J = 13.9$ Hz), 28.9, 28.4 (d, $J = 65.1$ Hz), 22.7, 21.9 (d, $J = 4.1$ Hz), 14.1. ³¹P NMR (242 MHz, CDCl₃) δ 35.19 (d, $J = 446.9$ Hz). HRMS (ESI-TOF) m/z: [M+Na]⁺ Calcd for C₁₄H₃₁OPNa⁺ 269.2005, found 269.2004.

bis(5-chloropentyl)phosphine oxide (3k)



Colorless oil. $V_{DCM} : V_{MeOH} = 20:1$, Yield: 36.7 mg, 71%. ¹H NMR (600 MHz, CDCl₃) δ 6.87 (d, $J = 450.1$ Hz, 1H), 3.54 (t, $J = 6.5$ Hz, 4H), 1.89-1.75 (m, 8H), 1.73-1.57 (m, 8H). ¹³C NMR (150 MHz, CDCl₃) δ 44.7, 32.1, 28.3 (d, $J = 65.3$ Hz), 28.0 (d, $J = 13.7$ Hz), 21.4 (d, $J = 3.9$ Hz). ³¹P NMR (242 MHz, CDCl₃) δ 33.70 (d, $J = 450.5$ Hz). HRMS (ESI-TOF) m/z: [M+Na]⁺ Calcd for C₁₀H₂₁Cl₂OPNa⁺ 281.0599, found 281.0599.

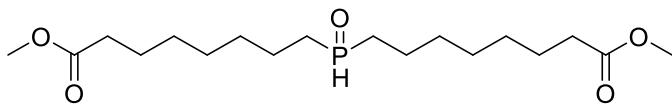
bis(7-bromoheptyl)phosphine oxide (3l)



Colorless oil. $V_{DCM} : V_{MeOH} = 20:1$, Yield: 67.2 mg, 84%. ¹H NMR (600 MHz, CDCl₃) δ 6.85 (d, $J = 446.6$ Hz, 1H), 3.39 (t, $J = 6.7$ Hz, 4H), 1.86-1.81 (m, 5H), 1.77-1.55 (m, 7H), 1.46-1.41 (m, 8H), 1.37-1.33 (m, 4H). ¹³C NMR

(150 MHz, CDCl₃) δ 33.9, 32.7, 30.6 (d, *J* = 13.7 Hz), 28.4, 28.3 (d, *J* = 65.5 Hz), 27.9, 21.8 (d, *J* = 3.4 Hz). ³¹P NMR (242 MHz, CDCl₃) δ 34.48 (d, *J* = 451.1 Hz). HRMS (ESI-TOF) m/z: [M+Na]⁺ Calcd for C₁₄H₂₉Br₂OPNa⁺ 425.0215, found 425.0222.

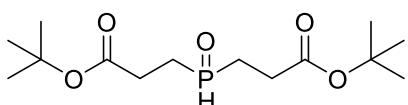
dimethyl 8,8'-(oxo-15-phosphanediyl)dioctanoate (3m)



Colorless oil. *V*_{DCM} : *V*_{MeOH} = 20:1, Yield: 58.3 mg, 81%. ¹H NMR (600 MHz, CDCl₃) δ 6.82 (d, *J* = 446.1

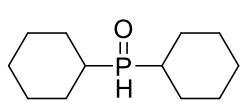
Hz, 1H), 3.63 (s, 6H), 2.27 (t, *J* = 7.5 Hz, 4H), 1.82-1.67 (m, 4H), 1.64-1.52 (m, 8H), 1.42-1.34 (m, 4H), 1.32-1.28 (m, 8H). ¹³C NMR (150 MHz, CDCl₃) δ 174.2, 51.5, 34.0, 30.5 (d, *J* = 13.6 Hz), 28.8 (d, *J* = 5.5 Hz), 28.3 (d, *J* = 65.2 Hz), 24.8, 21.8 (d, *J* = 3.5 Hz). ³¹P NMR (242 MHz, CDCl₃) δ 34.81 (d, *J* = 450.1 Hz). HRMS (ESI-TOF) m/z: [M+Na]⁺ Calcd for C₁₈H₃₅O₅PNa⁺ 385.2114, found 385.2119.

di-tert-butyl 3,3'-(oxo-15-phosphanediyl)dipropionate (3n)



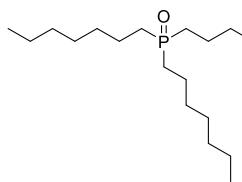
Colorless oil. *V*_{DCM} : *V*_{MeOH} = 20:1, Yield: 46.1 mg, 75%. ¹H NMR (600 MHz, CDCl₃) δ 7.01 (d, *J* = 467.2 Hz, 1H), 2.65-2.59 (m, 4H), 2.11-2.02 (m, 4H), 1.45 (s, 18H). ¹³C NMR (150 MHz, CDCl₃) δ 171.3 (d, *J* = 12.2 Hz), 81.6, 28.2, 27.5 (d, *J* = 3.3 Hz), 23.9 (d, *J* = 66.5 Hz). ³¹P NMR (242 MHz, CDCl₃) δ 31.87 (d, *J* = 466.1 Hz). HRMS (ESI-TOF) m/z: [M+Na]⁺ Calcd for C₁₄H₂₇O₅PNa⁺ 329.1488, found 329.1492.

dicyclohexylphosphine oxide (3o)



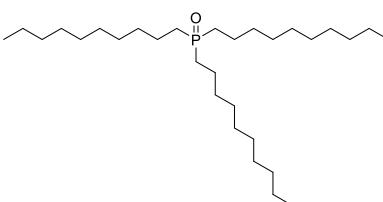
Colorless oil. *V*_{DCM} : *V*_{MeOH} = 20:1, Yield: 16.9 mg, 40%. ¹H NMR (600 MHz, CDCl₃) δ 6.29 (d, *J* = 435.1 Hz, 1H), 2.00-1.98 (m, 2H), 1.86-1.85 (m, 4H), 1.79-1.72 (m, 6H), 1.49-1.40 (m, 4H), 1.33-1.22 (m, 6H). ¹³C NMR (150 MHz, CDCl₃) δ 34.9 (d, *J* = 64.9 Hz), 26.3 (d, *J* = 14.3 Hz), 26.2, 26.1 (d, *J* = 12.4 Hz), 25.9, 25.1 (d, *J* = 3.4 Hz). ³¹P NMR (242 MHz, CDCl₃) δ 48.95 (d, *J* = 436.4 Hz). HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for C₁₂H₂₄OP⁺ 215.1559, found 215.1564.

triheptylphosphine oxide (3-2a)



White solid. Eluent *V*_{PE} : *V*_{EA} = 1:2, Yield: 49.6 mg, 72%. ¹H NMR (600 MHz, CDCl₃) δ 1.69-1.64 (m, 5H), 1.58-1.51 (m, 5H), 1.42-1.35 (m, 6H), 1.32-1.22 (m, 20H), 0.87 (t, *J* = 6.9 Hz, 9H). ¹³C NMR (150 MHz, CDCl₃) δ 31.7, 31.3 (d, *J* = 13.8 Hz), 28.9, 27.9 (d, *J* = 64.7 Hz), 22.7, 21.8 (d, *J* = 3.6 Hz), 14.2. ³¹P NMR (242 MHz, CDCl₃) δ 49.79. HRMS Calcd for C₂₁H₄₅OP[M+H]⁺ 345.3281, found 345.3280.

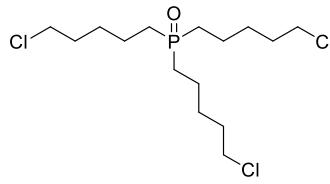
tris(decyl)phosphine oxide (3-2b)



Pale yellow oil. Eluent *V*_{PE} : *V*_{EA} = 1:2, Yield: 70.3 mg, 75%. ¹H NMR (600 MHz, CDCl₃) δ 1.67-1.62 (m, 6H), 1.57-1.50 (m, 6H), 1.40-1.35 (m, 6H), 1.31-1.22 (m, 36H), 0.87 (t, *J* = 7.2 Hz, 9H). ¹³C NMR (150 MHz, CDCl₃) δ 32.0, 31.3 (d, *J* = 13.6 Hz), 29.7, 29.5, 29.4, 29.3, 28.0 (d, *J* = 64.9 Hz), 22.8, 21.8 (d, *J* = 3.9 Hz), 14.2. ³¹P NMR (242 MHz, CDCl₃) δ 48.96.

HRMS Calcd for C₃₀H₆₃OP[M+H]⁺ 471.4689, found 471.4675.

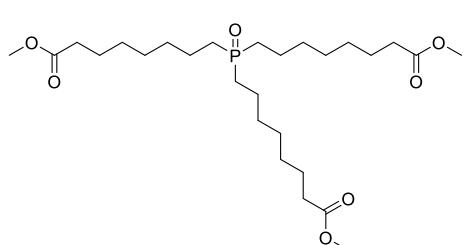
tris(5-chloropentyl)phosphine oxide (3-2c)



Pale yellow oil. Eluent V_{PE} : V_{EA} = 1:2, Yield: 48.4 mg, 67%. ¹H NMR (600 MHz, CDCl₃) δ 3.54 (t, J = 6.5 Hz, 6H), 1.80 (quint, J = 7.2 Hz, 6H), 1.71-1.66 (m, 6H), 1.63-1.53 (m, 12H). ¹³C NMR (150 MHz, CDCl₃) δ 44.8, 32.2, 28.5 (d, J = 13.9 Hz), 28.1 (d, J = 64.8 Hz), 21.2 (d, J = 3.5 Hz). ³¹P NMR (242 MHz, CDCl₃) δ 47.73.

HRMS Calcd for C₁₅H₃₀Cl₃OP[M+H]⁺ 363.1173, found 363.1175.

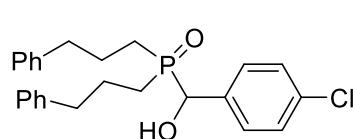
trimethyl 8,8',8''-(oxo-15-phosphanetriyl)trioctanoate (3-2d)



Pale yellow oil. Eluent V_{PE} : V_{EA} = 1:2, Yield: 42.4 mg, 41%. ¹H NMR (600 MHz, CDCl₃) δ 3.57 (s, 9H), 2.21 (t, J = 7.5 Hz, 6H), 1.59-1.54 (m, 6H), 1.53-1.50 (m, 6H), 1.49-1.42 (m, 6H), 1.34-1.27 (m, 6H), 1.26-1.20 (m, 12H). ¹³C NMR (150 MHz, CDCl₃) δ 174.1, 51.4, 33.9, 30.9 (d, J = 14.2 Hz), 28.8, 28.7, 27.8 (d, J = 64.6 Hz), 24.7, 21.6 (d, J = 4.1 Hz). ³¹P NMR (242 MHz, CDCl₃) δ 48.59.

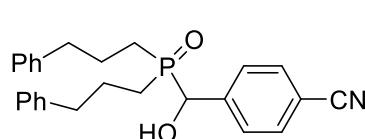
HRMS Calcd for C₂₇H₅₁O₇P[M+H]⁺ 519.3445, found 519.3446.

((4-chlorophenyl)(hydroxy)methyl)bis(3-phenylpropyl)phosphine oxide (4a)



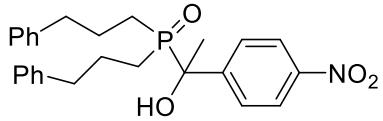
White solid. Eluent V_{DCM} : V_{MeOH} = 10:1, Yield: 61.3 mg, 72%. ¹H NMR (600 MHz, CDCl₃) δ 7.30-7.26 (m, 4H), 7.25-7.22 (m, 3H), 7.21 - 7.17 (m, 4H), 7.08 (dd, J = 16.3, 7.3 Hz, 4H), 4.91 (d, J = 6.6 Hz, 1H), 4.57 (br s, 1H), 2.67-2.56 (m, 4H), 1.78-1.72 (m, 4H), 1.67-1.57 (m, 4H). ¹³C NMR (150 MHz, CDCl₃) δ 140.8 (d, J = 8.4 Hz), 135.7, 134.0 (d, J = 3.1 Hz), 128.9, 128.7, 128.6, 127.8 (d, J = 3.8 Hz), 126.3 (d, J = 9.2 Hz), 71.6 (d, J = 72.8 Hz), 37.0 (d, J = 11.6 Hz), 36.9 (d, J = 11.2 Hz), 24.5 (d, J = 27.1 Hz), 24.1 (d, J = 28.6 Hz), 23.3 (d, J = 3.8 Hz), 23.2 (d, J = 3.4 Hz). ³¹P NMR (242 MHz, CDCl₃) δ 50.11. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for C₂₅H₂₉ClO₂P⁺ 427.1588, found 427.1588.

4-((bis(3-phenylpropyl)phosphoryl)(hydroxy)methyl)benzonitrile (4b)



White solid. Eluent V_{DCM} : V_{MeOH} = 10:1, Yield: 56.7 mg, 68%. ¹H NMR (600 MHz, CDCl₃) δ 7.52 (d, J = 8.2 Hz, 2H), 7.34 (d, J = 7.1 Hz, 2H), 7.30-7.27 (m, 4H), 7.24-7.20 (m, 2H), 7.08 (t, J = 7.5 Hz, 4H), 4.99 (d, J = 7.3 Hz, 1H), 4.53 (br s, 1H), 2.68-2.56 (m, 4H), 1.88-1.70 (m, 4H), 1.64-1.45 (m, 4H). ¹³C NMR (150 MHz, CDCl₃) δ 142.6, 140.7 (d, J = 9.1 Hz), 132.4, 128.7 (d, J = 6.9 Hz), 128.6 (d, J = 11.3 Hz), 127.0 (d, J = 3.4 Hz), 126.5 (d, J = 6.2 Hz), 118.6, 111.9 (d, J = 2.5 Hz), 71.8 (d, J = 70.9 Hz), 36.9 (d, J = 13.1 Hz), 36.8 (d, J = 12.7 Hz), 24.5 (d, J = 8.6 Hz), 24.1 (d, J = 7.4 Hz), 23.3 (d, J = 3.6 Hz), 23.1 (d, J = 3.6 Hz). ³¹P NMR (242 MHz, CDCl₃) δ 50.13. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for C₂₆H₂₉NO₂P⁺ 418.1930, found 418.1935.

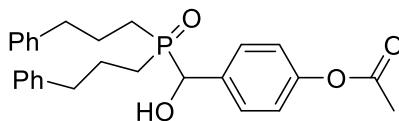
(1-hydroxy-1-(4-nitrophenyl)ethyl)bis(3-phenylpropyl)phosphine oxide (4c)



White solid. Eluent $V_{DCM} : V_{MeOH} = 10:1$, Yield: 57.5 mg, 64%.

1H NMR (600 MHz, CDCl₃) δ 8.10 (d, $J = 8.7$ Hz, 2H), 7.58 (dd, $J = 8.9, 1.7$ Hz, 2H), 7.27-7.23 (m, 4H), 7.21-7.18 (m, 2H), 7.09 (d, $J = 7.2$ Hz, 2H), 7.02 (d, $J = 7.2$ Hz, 2H), 4.31 (br s, 1H), 2.64-2.61 (m, 2H), 2.59-2.51 (m, 2H), 1.79 (d, $J = 12.1$ Hz, 3H), 1.74-1.61 (m, 4H), 1.59-1.40 (m, 4H). ^{13}C NMR (150 MHz, CDCl₃) δ 149.3 (d, $J = 3.2$ Hz), 147.2 (d, $J = 3.2$ Hz), 140.7 (d, $J = 24.3$ Hz), 128.62, 128.59 (d, $J = 2.5$ Hz), 126.7 (d, $J = 2.4$ Hz), 126.4, 123.5 (d, $J = 1.9$ Hz), 74.1 (d, $J = 74.3$ Hz), 37.1 (d, $J = 13.4$ Hz), 36.9 (d, $J = 13.4$ Hz), 24.9 (d, $J = 3.2$ Hz), 24.1, 23.8 (d, $J = 3.6$ Hz), 23.7 (d, $J = 4.7$ Hz), 23.6 (d, $J = 4.3$ Hz), 23.3. ^{31}P NMR (242 MHz, CDCl₃) δ 53.83. HRMS (ESI-TOF) m/z: [M+Na]⁺ Calcd for C₂₆H₃₀NO₄PNa⁺ 447.1805, found 447.1810.

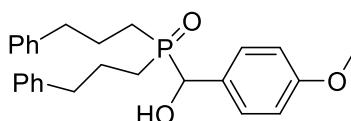
4-((bis(3-phenylpropyl)phosphoryl)(hydroxy)methyl)phenyl acetate (4d)



White solid. Eluent $V_{DCM} : V_{MeOH} = 10:1$, Yield: 68.4 mg,

76%. 1H NMR (600 MHz, CDCl₃) δ 7.28-7.25 (m, 6H), 7.21-7.17 (m, 2H), 7.09 (t, $J = 6.9$ Hz, 4H), 6.98 (t, $J = 8.4$ Hz, 2H), 4.84 (d, $J = 7.1$ Hz, 1H), 2.63-2.54 (m, 4H), 2.30 (s, 3H), 1.87-1.71 (m, 4H), 1.69-1.48 (m, 4H). ^{13}C NMR (150 MHz, CDCl₃) δ 169.5, 150.4, 141.0 (d, $J = 13.2$ Hz), 134.9, 128.6 (d, $J = 3.1$ Hz), 128.5 (d, $J = 3.3$ Hz), 127.6 (d, $J = 4.1$ Hz), 126.2 (d, $J = 4.2$ Hz), 121.8, 71.6 (d, $J = 75.3$ Hz), 37.1 (d, $J = 11.2$ Hz), 37.0 (d, $J = 10.8$ Hz), 24.4 (d, $J = 35.6$ Hz), 24.0 (d, $J = 34.6$ Hz), 23.3 (d, $J = 3.6$ Hz), 23.1 (d, $J = 4.1$ Hz), 21.3. ^{31}P NMR (242 MHz, CDCl₃) δ 50.57. HRMS (ESI-TOF) m/z: [M+Na]⁺ Calcd for C₂₇H₃₁O₄PNa⁺ 473.1852, found 473.1848.

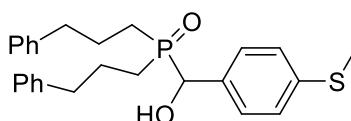
(hydroxy(4-methoxyphenyl)methyl)bis(3-phenylpropyl)phosphine oxide (4e)



White solid. Eluent $V_{DCM} : V_{MeOH} = 10:1$, Yield: 43.1 mg, 51%.

1H NMR (600 MHz, CDCl₃) δ 7.28-7.26 (m, 2H), 7.26-7.23 (m, 2H), 7.21-7.17 (m, 4H), 7.09 (dd, $J = 14.5, 7.4$ Hz, 4H), 6.80 (d, $J = 8.6$ Hz, 2H), 4.88 (d, $J = 5.5$ Hz, 1H), 4.73 (br s, 1H), 3.78 (s, 3H), 2.65-2.55 (m, 4H), 1.87-1.70 (m, 4H), 1.69-1.43 (m, 4H). ^{13}C NMR (150 MHz, CDCl₃) δ 159.5, 141.0 (d, $J = 10.6$ Hz), 129.1, 128.6 (d, $J = 8.7$ Hz), 128.5 (d, $J = 7.7$ Hz), 127.8 (d, $J = 4.3$ Hz), 126.2 (d, $J = 8.8$ Hz), 114.1, 72.0 (d, $J = 75.2$ Hz), 55.38, 37.1 (d, $J = 8.6$ Hz), 37.0 (d, $J = 8.2$ Hz), 24.5 (d, $J = 39.5$ Hz), 24.1 (d, $J = 40.3$ Hz), 23.3 (d, $J = 3.3$ Hz), 23.2 (d, $J = 4.1$ Hz). ^{31}P NMR (242 MHz, CDCl₃) δ 50.28. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for C₂₆H₃₂O₃P⁺ 423.2084, found 423.2089.

(hydroxy(4-(methylthio)phenyl)methyl)bis(3-phenylpropyl)phosphine oxide (4f)

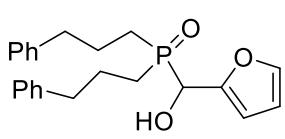


White solid. Eluent $V_{DCM} : V_{MeOH} = 10:1$, Yield: 48.1 mg, 55%.

1H NMR (600 MHz, CDCl₃) δ 7.28-7.27 (m, 2H), 7.26-7.24 (m, 2H), 7.21-7.17 (m, 4H), 7.14 (d, $J = 8.4$ Hz, 2H), 7.09 (dd, $J = 11.4, 7.4$ Hz, 4H), 4.90 (d, $J = 6.2$ Hz, 1H), 4.74 (br s, 1H), 2.65-2.55 (m, 4H), 2.46 (s, 3H), 1.84-1.71 (m, 4H), 1.69-1.44 (m, 4H). ^{13}C NMR (150 MHz, CDCl₃) δ 141.0 (d, $J = 11.5$ Hz), 138.6 (d, $J = 2.9$ Hz), 133.8, 128.6 (d, $J = 7.3$ Hz), 128.5 (d, $J = 7.0$ Hz), 127.0 (d, $J = 4.3$ Hz), 126.6 (d, $J = 2.0$ Hz), 126.3 (d, $J = 8.5$ Hz), 71.92 (d, $J = 74.1$ Hz), 37.1 (d, $J = 10.2$ Hz), 37.0 (d, $J = 10.3$ Hz), 24.5 (d, $J = 33.3$ Hz), 24.1 (d, $J = 34.3$ Hz), 23.3 (d, $J = 3.4$ Hz),

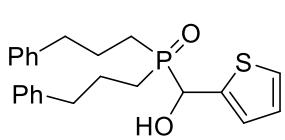
23.2 (d, $J = 3.9$ Hz), 15.8. ^{31}P NMR (242 MHz, CDCl_3) δ 50.44. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for $\text{C}_{26}\text{H}_{32}\text{O}_2\text{PS}^+$ 439.1855, found 439.1854.

(furan-2-yl(hydroxy)methyl)bis(3-phenylpropyl)phosphine oxide (4g)



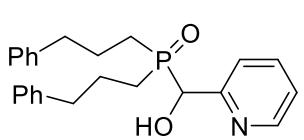
White solid. Eluent V_{DCM} : $V_{\text{MeOH}} = 10:1$, Yield: 46.7 mg, 61%. ^1H NMR (600 MHz, CDCl_3) δ 7.30-7.26 (m, 4H), 7.26-7.25 (m, 1H), 7.21-7.17 (m, 2H), 7.12 (t, $J = 8.5$ Hz, 4H), 6.35-6.32 (m, 2H), 5.74 (br s, 1H), 5.02 (d, $J = 6.3$ Hz, 1H), 2.69-2.58 (m, 4H), 1.93-1.79 (m, 4H), 1.78-1.53 (m, 4H). ^{13}C NMR (150 MHz, CDCl_3) δ 150.6, 142.6 (d, $J = 1.8$ Hz), 141.0 (d, $J = 19.1$ Hz), 128.6 (d, $J = 3.1$ Hz), 128.5 (d, $J = 7.6$ Hz), 126.2 (d, $J = 11.2$ Hz), 110.9, 108.9 (d, $J = 6.1$ Hz), 66.9 (d, $J = 76.3$ Hz), 37.1 (d, $J = 8.9$ Hz), 37.0 (d, $J = 8.8$ Hz), 25.6 (d, $J = 62.3$ Hz), 24.8 (d, $J = 63.7$ Hz), 23.1 (d, $J = 3.4$ Hz), 23.0 (d, $J = 3.7$ Hz). ^{31}P NMR (242 MHz, CDCl_3) δ 50.45. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for $\text{C}_{23}\text{H}_{28}\text{O}_3\text{P}^+$ 383.1771, found 383.1774.

(hydroxy(thiophen-2-yl)methyl)bis(3-phenylpropyl)phosphine oxide (4h)



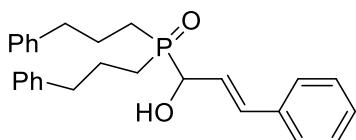
White solid. Eluent V_{DCM} : $V_{\text{MeOH}} = 10:1$, Yield: 51.0 mg, 64%. ^1H NMR (600 MHz, CDCl_3) δ 7.28-7.27 (m, 4H), 7.26-7.23 (m, 3H), 7.21-7.16 (m, 2H), 7.10 (dd, $J = 13.1, 7.3$ Hz, 4H), 6.94 (d, $J = 3.7$ Hz, 2H), 5.28 (s, 1H), 5.19 (br s, 1H), 2.67-2.56 (m, 4H), 1.87-1.75 (m, 4H), 1.74-1.53 (m, 4H). ^{13}C NMR (150 MHz, CDCl_3) δ 140.9 (d, $J = 10.3$ Hz), 140.2, 128.6 (d, $J = 5.8$ Hz), 128.5 (d, $J = 7.8$ Hz), 127.3, 126.3 (d, $J = 10.1$ Hz), 125.5 (d, $J = 2.3$ Hz), 125.4 (d, $J = 5.7$ Hz), 68.9 (d, $J = 77.2$ Hz), 37.1 (d, $J = 6.2$ Hz), 37.0 (d, $J = 5.7$ Hz), 24.6 (d, $J = 44.8$ Hz), 24.2 (d, $J = 45.4$ Hz), 23.3 (d, $J = 3.7$ Hz), 23.2 (d, $J = 3.6$ Hz). ^{31}P NMR (242 MHz, CDCl_3) δ 50.42. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for $\text{C}_{23}\text{H}_{28}\text{O}_2\text{PS}^+$ 399.1542, found 399.1542.

(hydroxy(pyridin-2-yl)methyl)bis(3-phenylpropyl)phosphine oxide (4i)



White wax. Eluent V_{DCM} : $V_{\text{MeOH}} = 10:1$, Yield: 40.7 mg, 52%. ^1H NMR (600 MHz, CDCl_3) δ 8.50 (d, $J = 4.7$ Hz, 1H), 7.72-7.69 (m, 1H), 7.62 (d, $J = 7.8$ Hz, 1H), 7.29-7.26 (m, 2H), 7.24-7.14 (m, 7H), 7.00 (d, $J = 7.3$ Hz, 2H), 5.38 (br s, 1H), 5.08 (d, $J = 7.7$ Hz, 1H), 2.74 (d, $J = 7.5$ Hz, 2H), 2.48 (t, $J = 7.5$ Hz, 2H), 2.08-1.81 (m, 4H), 1.69-1.34 (m, 4H). ^{13}C NMR (150 MHz, CDCl_3) δ 154.2, 147.9, 141.0 (d, $J = 27.1$ Hz), 137.1, 128.6 (d, $J = 14.2$ Hz), 128.4 (d, $J = 7.2$ Hz), 126.1 (d, $J = 13.5$ Hz), 123.2 (d, $J = 1.5$ Hz), 122.5 (d, $J = 2.7$ Hz), 70.3 (d, $J = 74.9$ Hz), 37.1 (d, $J = 14.3$ Hz), 37.0 (d, $J = 14.1$ Hz), 26.0 (d, $J = 63.1$ Hz), 23.2 (d, $J = 66.6$ Hz), 23.1, 22.8 (d, $J = 3.6$ Hz). ^{31}P NMR (242 MHz, CDCl_3) δ 51.57. HRMS (ESI-TOF) m/z: [M+Na]⁺ Calcd for $\text{C}_{24}\text{H}_{28}\text{NO}_2\text{PNa}^+$ 416.1750, found 416.1756.

(E)-(1-hydroxy-3-phenylallyl)bis(3-phenylpropyl)phosphine oxide (4j)



White solid. Eluent V_{DCM} : $V_{\text{MeOH}} = 10:1$, Yield: 43.4 mg, 52%. ^1H NMR (600 MHz, CDCl_3) δ 7.31-7.27 (m, 5H), 7.25-7.21 (m, 4H), 7.20-7.15 (m, 2H), 7.11-7.09 (m, 4H), 6.70-6.67 (m, 1H), 6.23-6.18 (m, 1H), 5.21 (br s, 1H), 4.66 (t, $J = 7.5$ Hz, 1H), 2.67-2.60 (m, 4H), 2.01-1.87 (m, 4H), 1.84-1.61 (m, 4H). ^{13}C NMR (150 MHz, CDCl_3) δ 140.9 (d, $J = 14.4$ Hz), 136.3 (d, $J = 2.1$ Hz), 132.3 (d, $J = 10.8$ Hz), 128.7, 128.6, 128.5 (d, $J = 4.5$ Hz), 128.1,

126.7, 126.2 (d, J = 9.5 Hz), 124.3, 71.2 (d, J = 75.8 Hz), 37.0 (d, J = 6.8 Hz), 36.9 (d, J = 6.8 Hz), 24.7 (d, J = 61.4 Hz), 24.4 (d, J = 62.1 Hz), 23.2 (d, J = 3.7 Hz), 23.1 (d, J = 3.7 Hz). ^{31}P NMR (242 MHz, CDCl_3) δ 50.62. HRMS (ESI-TOF) m/z: [M+Na]⁺ Calcd for $\text{C}_{27}\text{H}_{31}\text{O}_2\text{PNa}^+$ 441.1954, found 441.1952.

((4-chlorophenyl)(hydroxy)methyl)diethylphosphine oxide (4k)

White wax. Eluent $V_{\text{DCM}} : V_{\text{MeOH}} = 10:1$, Yield: 20.2 mg, 41%. ^1H NMR (600 MHz, CDCl_3) δ 7.36 (d, J = 8.1 Hz, 2H), 7.31 (d, J = 8.4 Hz, 2H), 5.21 (br s, 1H), 5.01 (d, J = 7.2 Hz, 1H), 1.64 (m, 4H), 1.08 (m, 6H). ^{13}C NMR (150 MHz, CDCl_3) δ 136.1, 133.9 (d, J = 2.8 Hz), 128.8 (d, J = 2.1 Hz), 128.1 (d, J = 4.2 Hz), 71.1 (d, J = 74.1 Hz), 17.5 (d, J = 13.9 Hz), 17.1 (d, J = 15.1 Hz), 5.6 (d, J = 4.7 Hz), 5.5 (d, J = 5.4 Hz). ^{31}P NMR (242 MHz, CDCl_3) δ 53.38. HRMS Calcd for $\text{C}_{11}\text{H}_{16}\text{ClO}_2\text{P}$ [M+Na]⁺ 269.0496, found 269.0498.

([1,1'-biphenyl]-4-yl(hydroxy)methyl)diheptylphosphine oxide (4l)

White wax. Eluent $V_{\text{DCM}} : V_{\text{MeOH}} = 10:1$, Yield: 43.5 mg, 51%. ^1H NMR (600 MHz, CDCl_3) δ 7.61-7.58 (m, 4H), 7.48 (d, J = 7.3 Hz, 2H), 7.44 (t, J = 7.6 Hz, 2H), 7.35 (t, J = 7.4 Hz, 1H), 5.34 (br s, 1H), 5.05 (t, J = 4.8 Hz, 1H), 1.74-1.66 (m, 4H), 1.61-1.53 (m, 4H), 1.40-1.33 (m, 4H), 1.30-1.23 (m, 12H), 0.86 (m, 6H). ^{13}C NMR (150 MHz, CDCl_3) δ 141.2, 140.6, 136.2, 128.9, 127.6, 127.5, 127.2, 127.0 (d, J = 4.2 Hz), 72.1 (d, J = 70.8 Hz), 31.7, 31.6, 31.3 (d, J = 3.8 Hz), 31.2 (d, J = 3.9 Hz), 29.8, 28.93, 28.91, 25.2, 22.72, 22.71, 21.6 (d, J = 3.8 Hz), 21.4 (d, J = 4.3 Hz), 14.19, 14.17. ^{31}P NMR (242 MHz, CDCl_3) δ 50.18. HRMS (ESI-TOF) m/z: [M+H]⁺ Calcd for $\text{C}_{27}\text{H}_{42}\text{O}_2\text{P}^+$ 429.2917, found 429.2918.

5. In situ $^{31}\text{P}\{\text{H}\}$ NMR spectra for the synthesis of $\text{R}_2\text{P(O)H}$

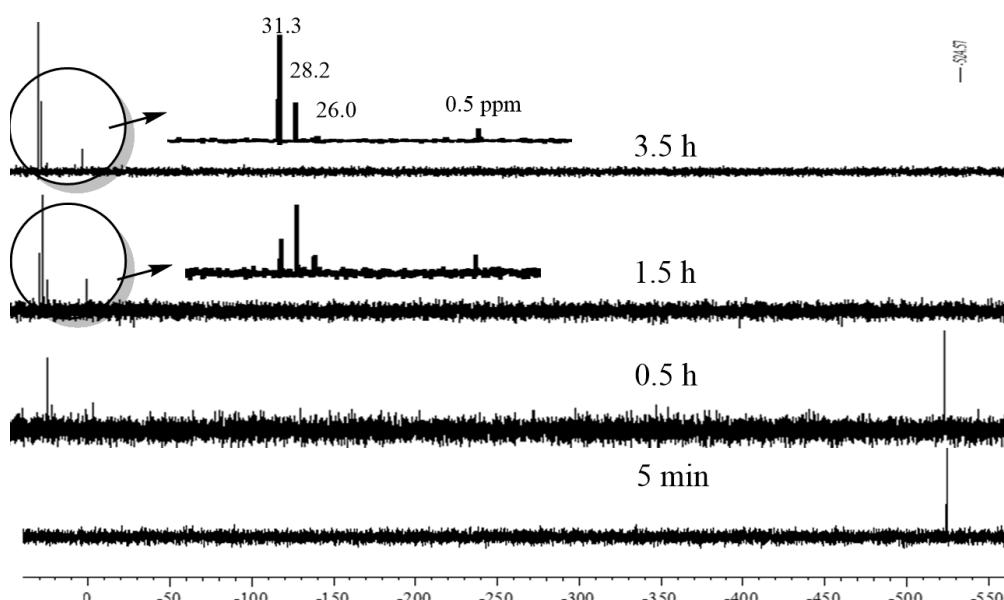


Fig 1

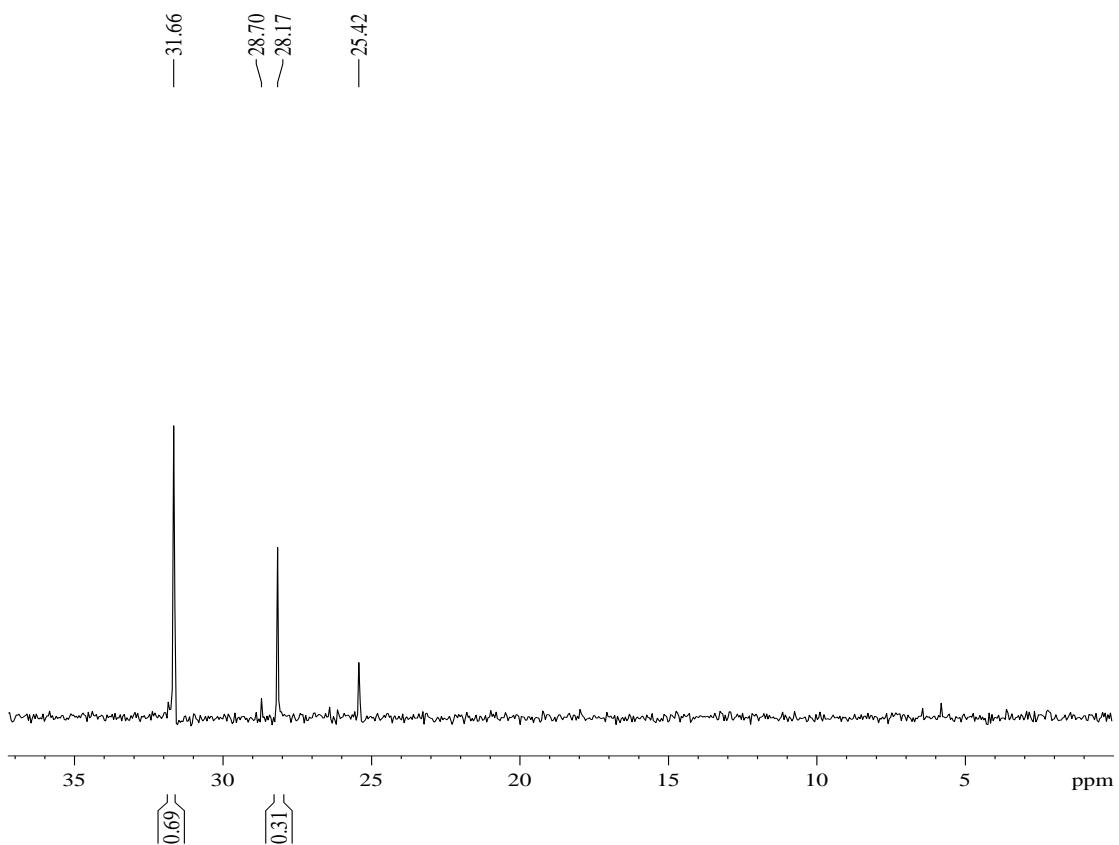


Fig 2 In situ ${}^1\text{H}$ NMR spectra for the synthesis of R2P(O)H (reaction time: 3.5 hours)

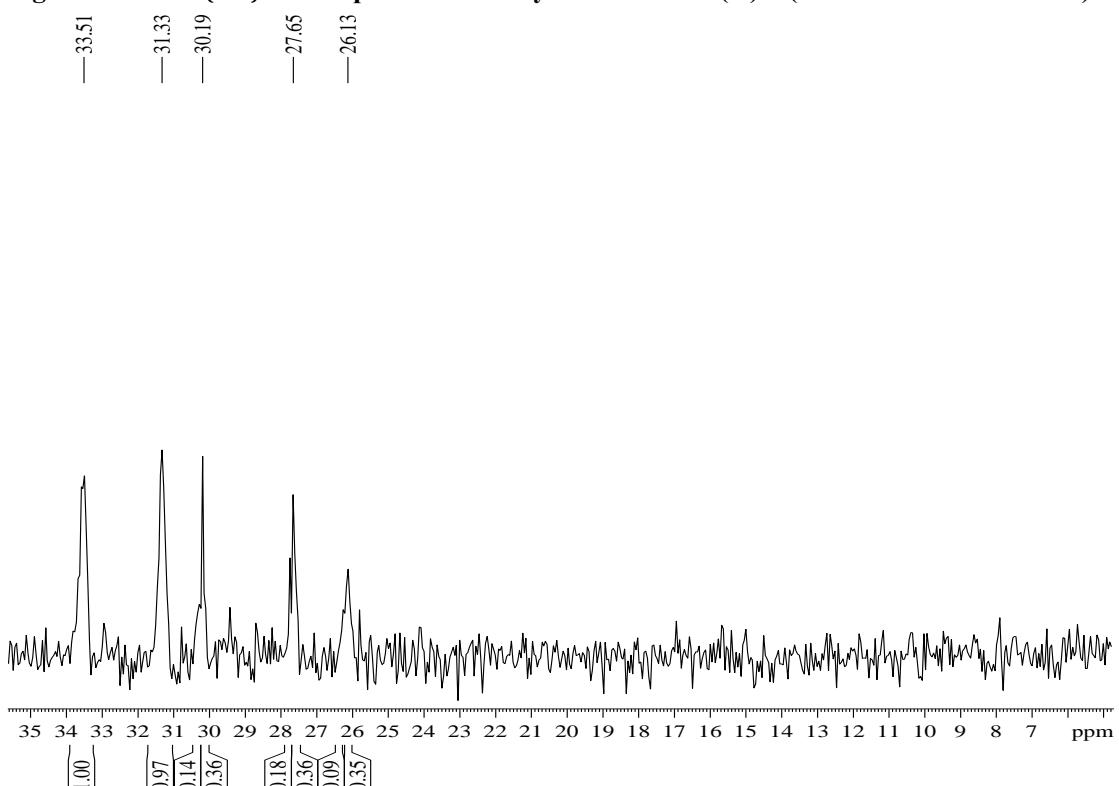
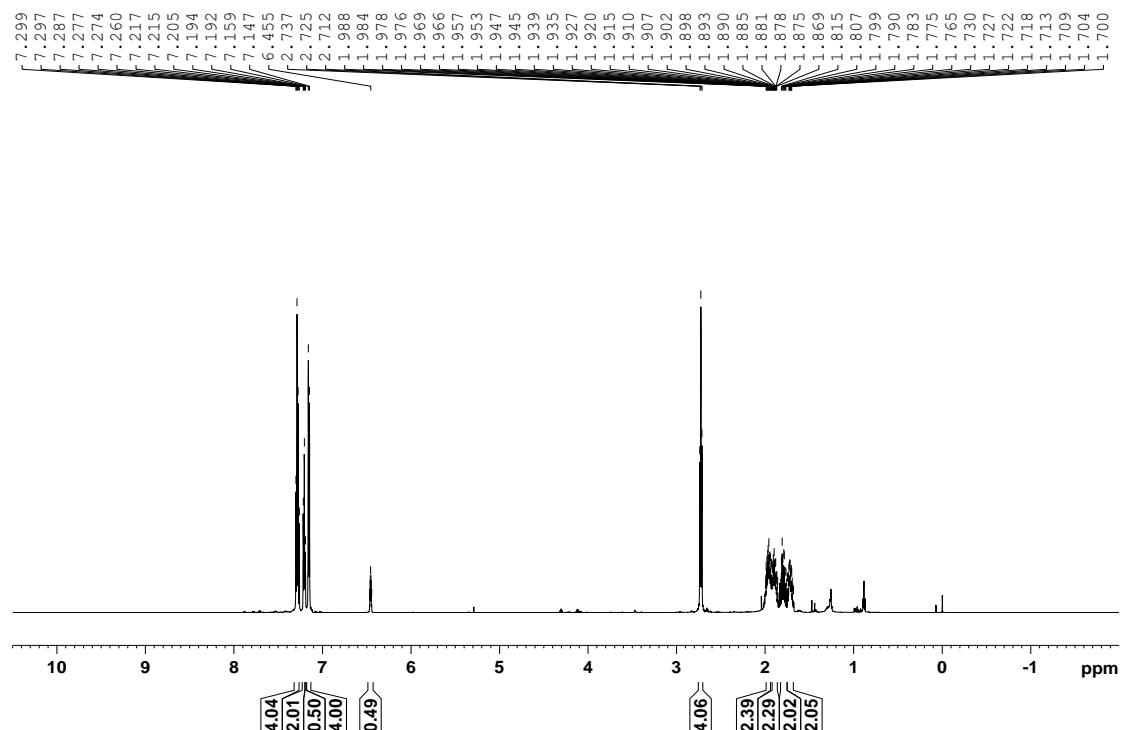
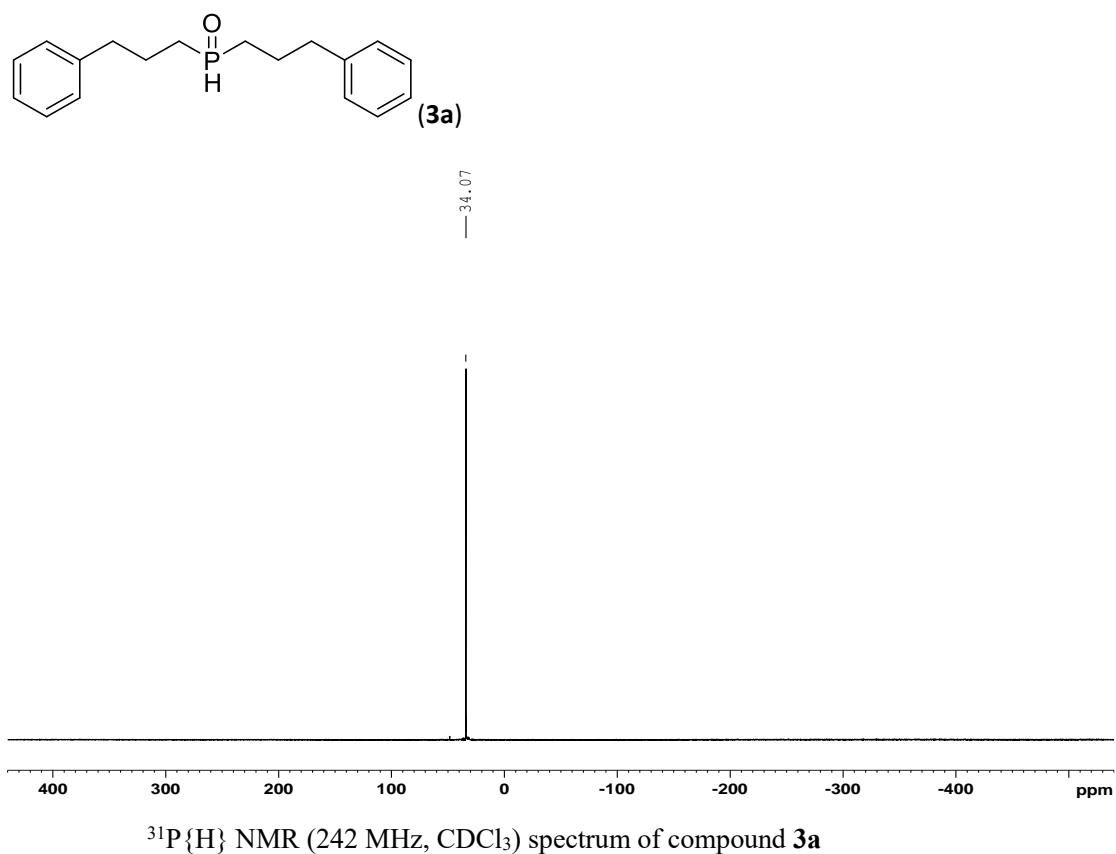
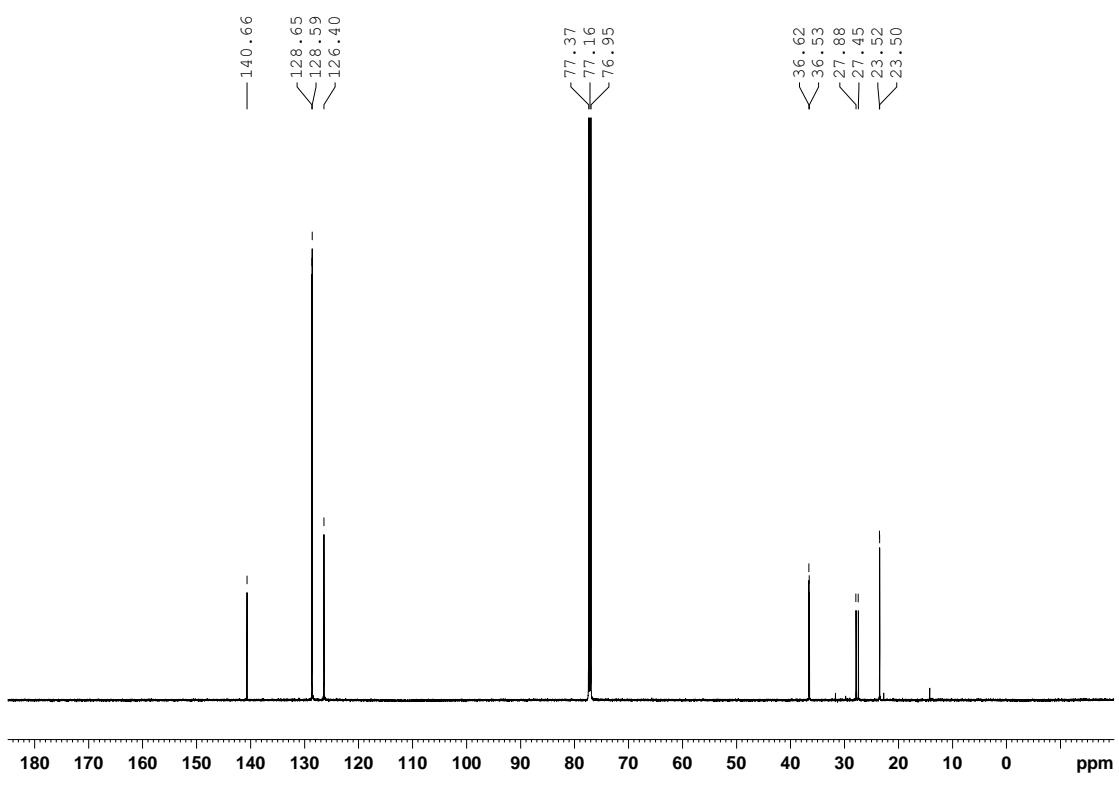


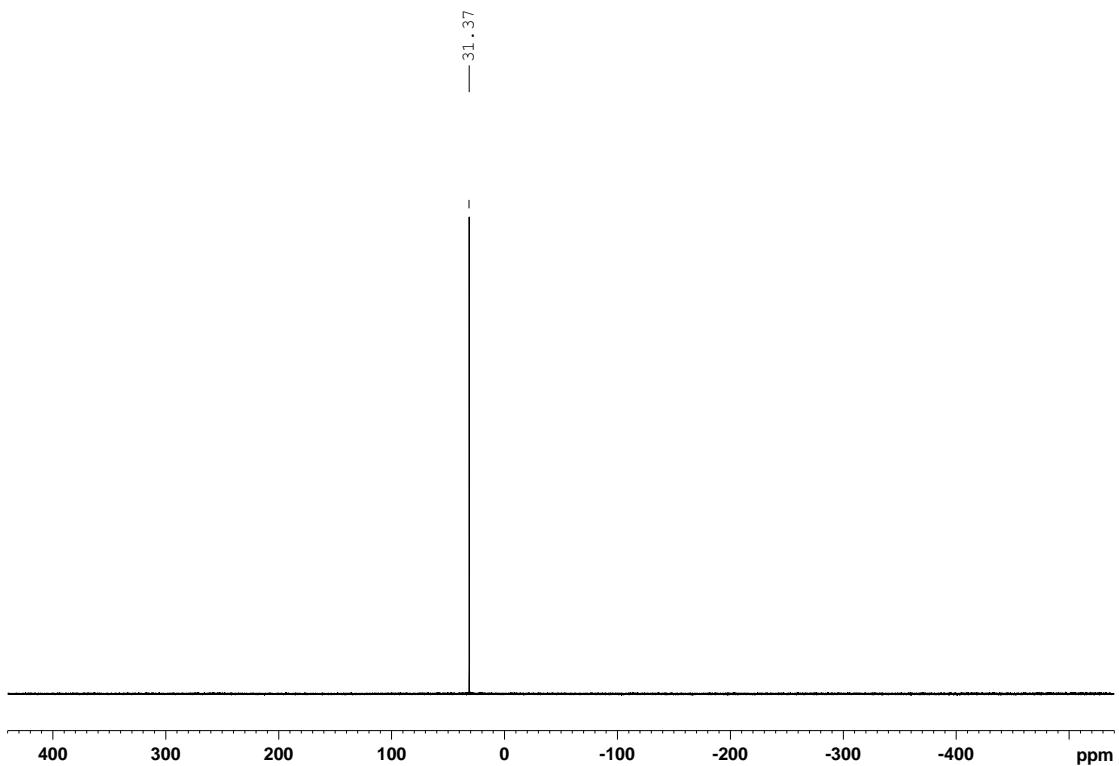
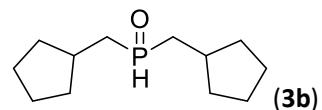
Fig 3 In situ ${}^{31}\text{P}$ NMR spectra for the synthesis of R2P(O)H (reaction time: 3.5 hours)

6. Copies of NMR spectra for products

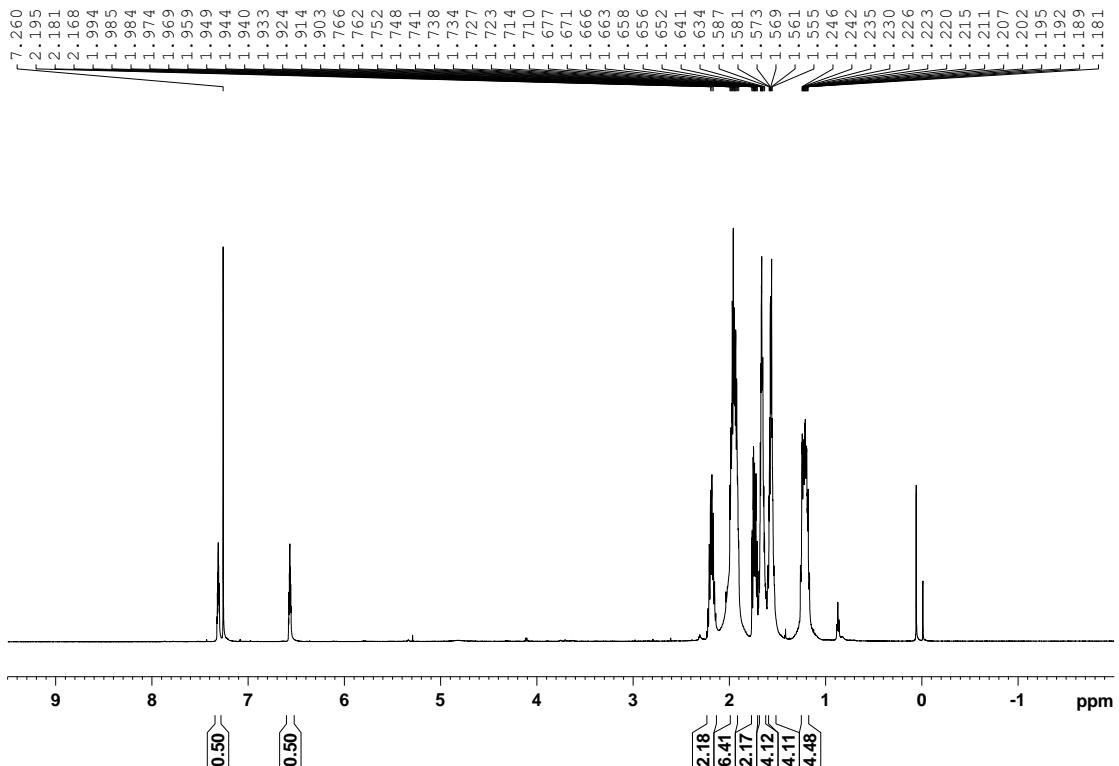




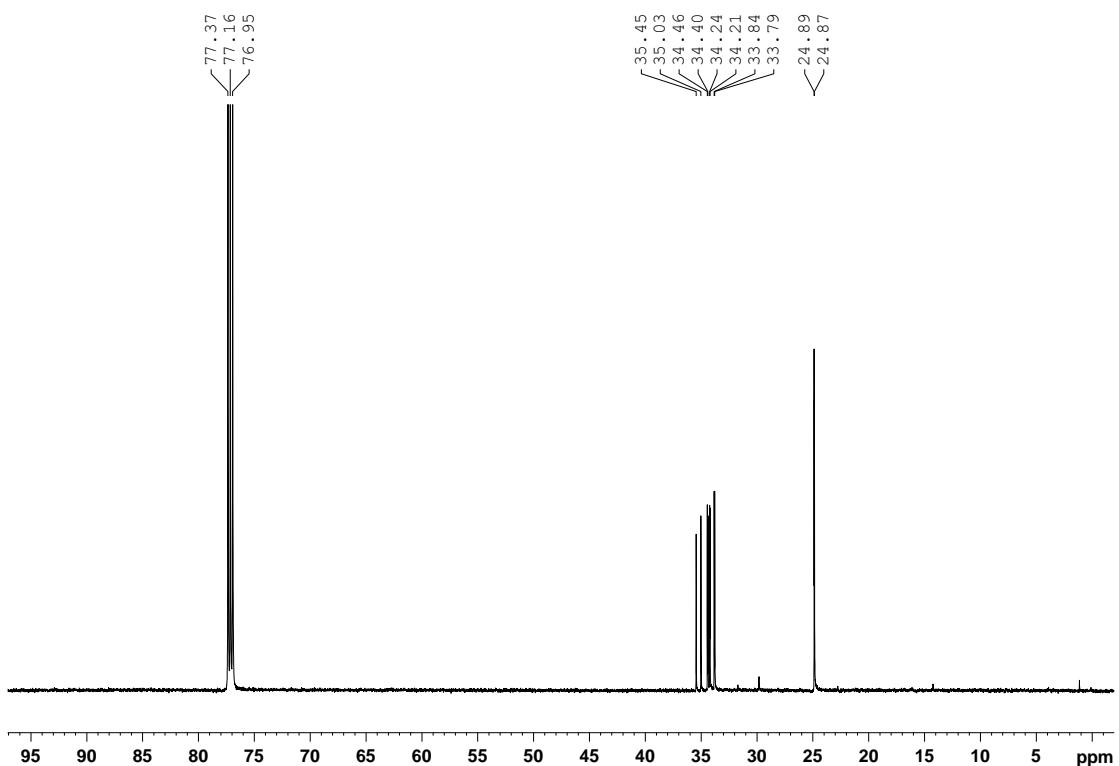
$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **3a**



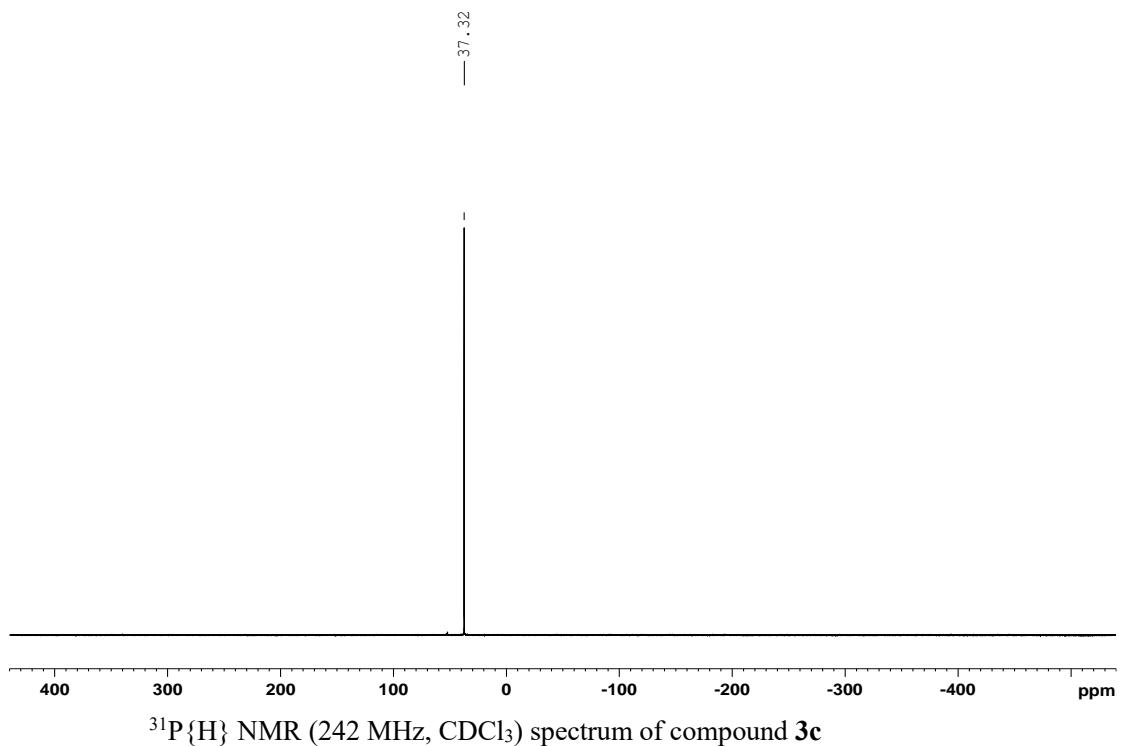
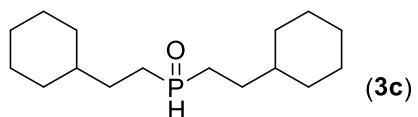
$^{31}\text{P}\{\text{H}\}$ NMR (242 MHz, CDCl_3) spectrum of compound **3b**



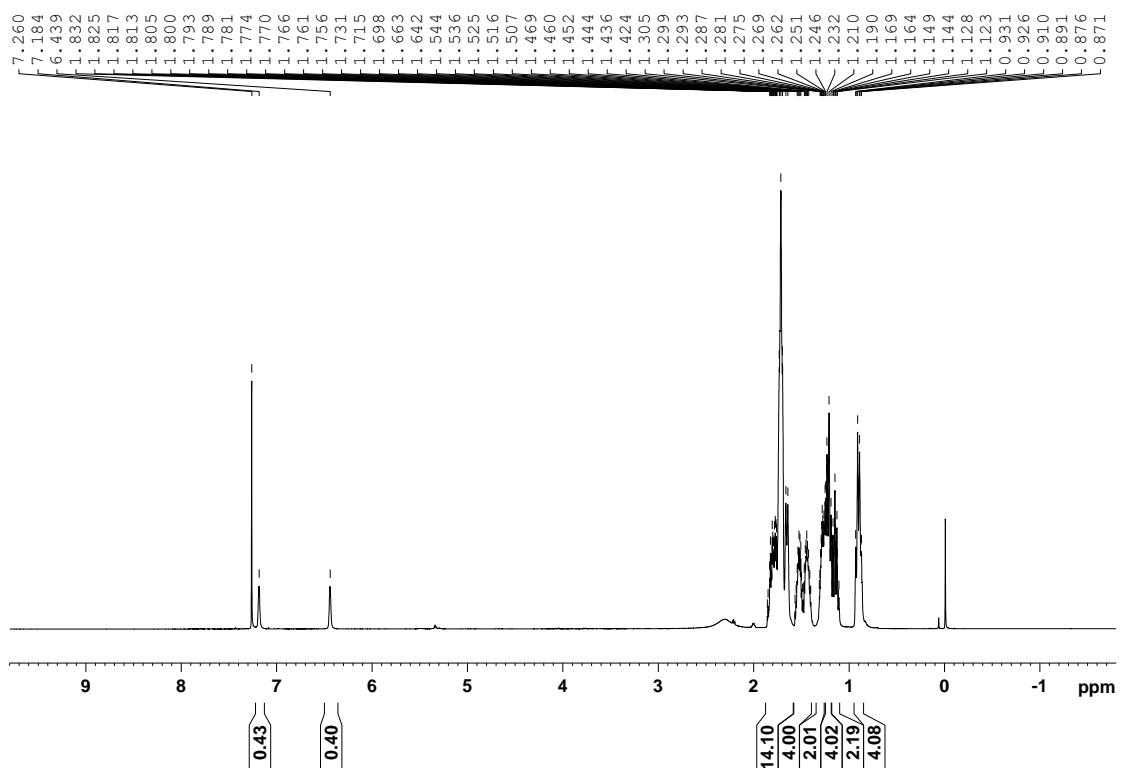
^1H NMR (600 MHz, CDCl_3) spectrum of compound **3b**



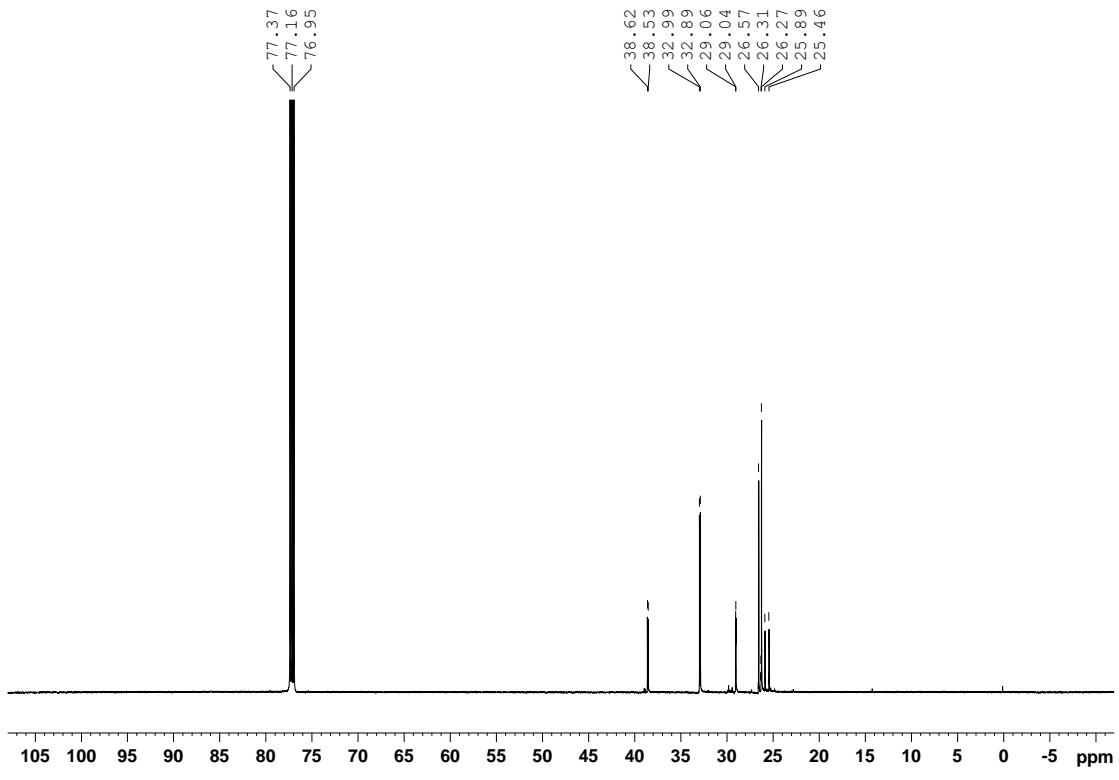
$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **3b**



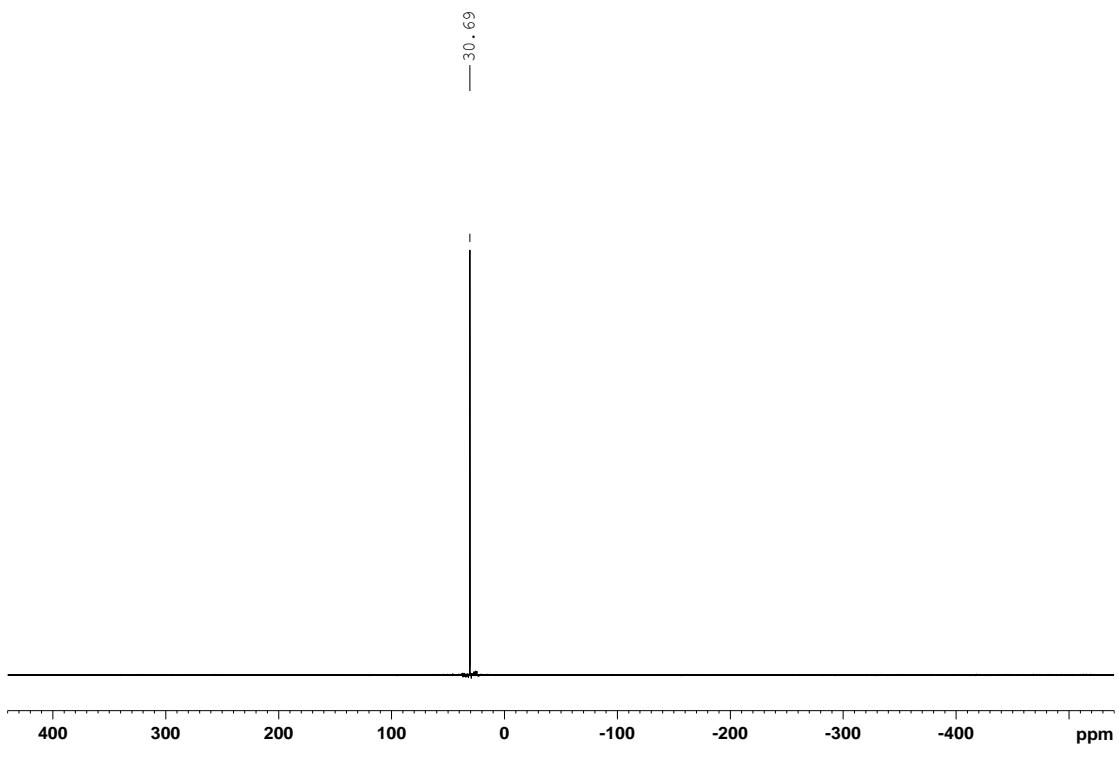
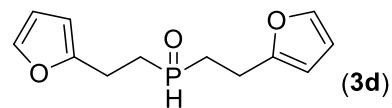
$^{31}\text{P}\{\text{H}\}$ NMR (242 MHz, CDCl_3) spectrum of compound **3c**



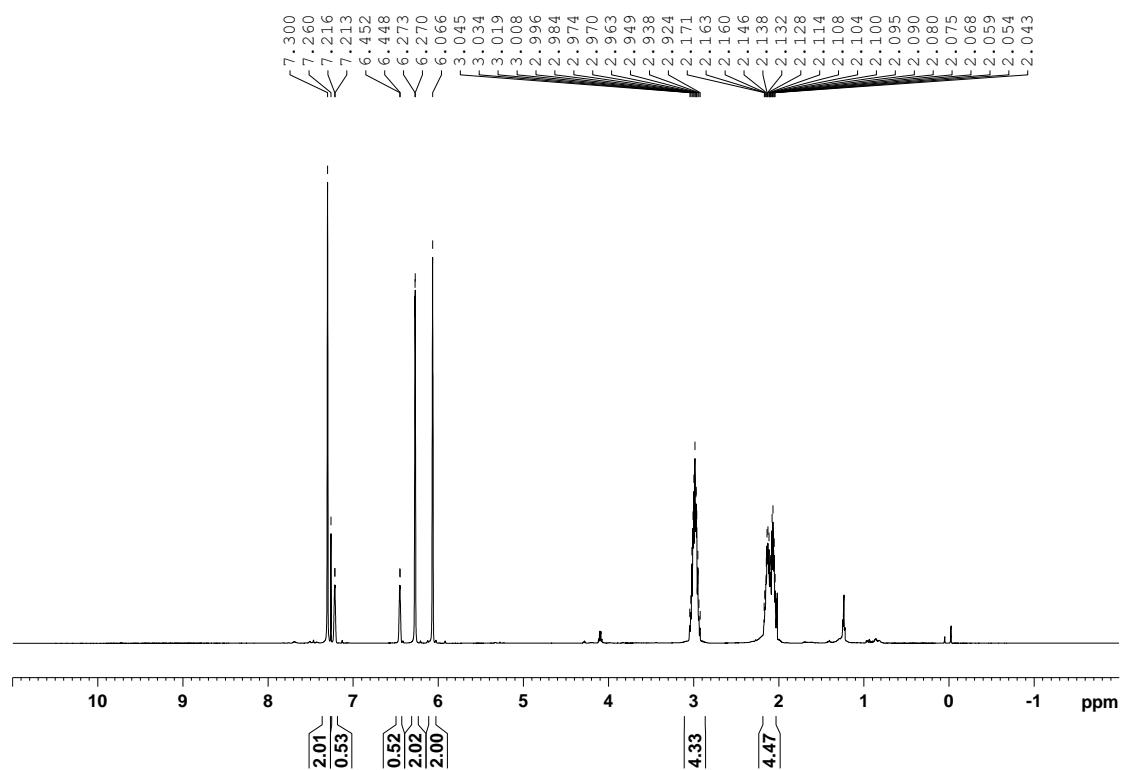
¹H NMR (600 MHz, CDCl₃) spectrum of compound 3c



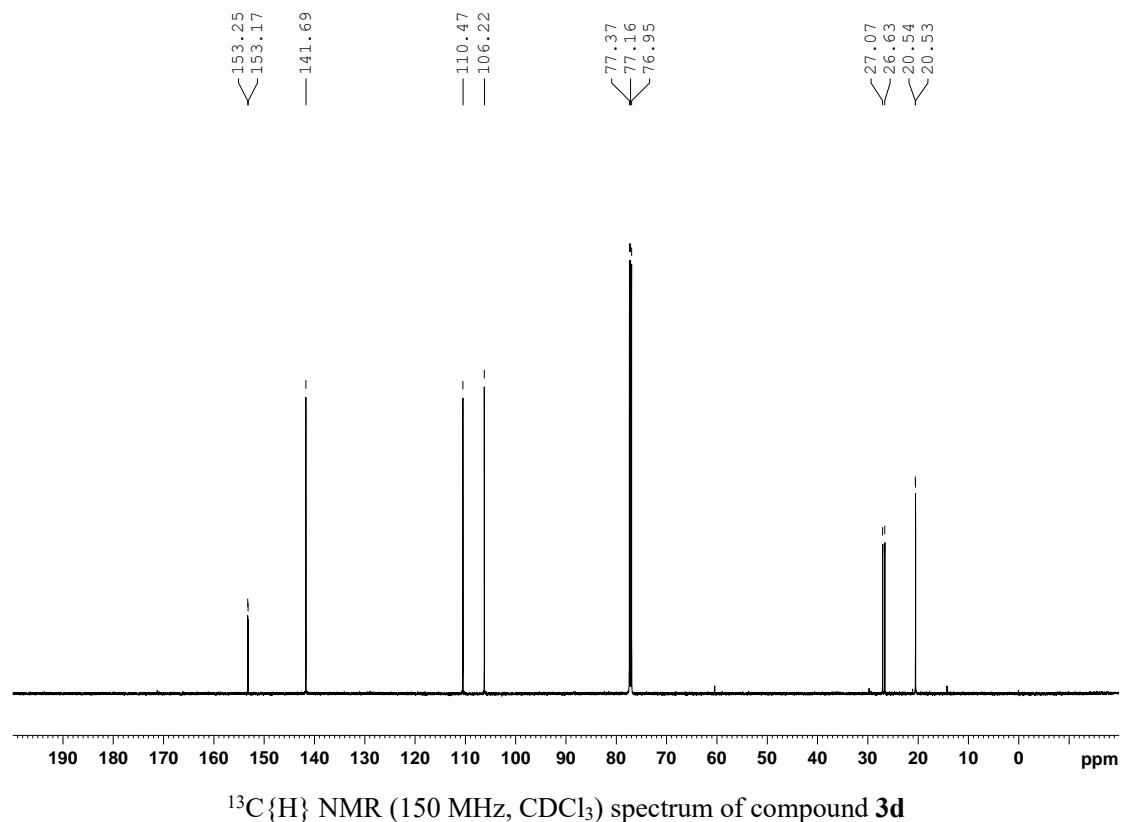
$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **3c**



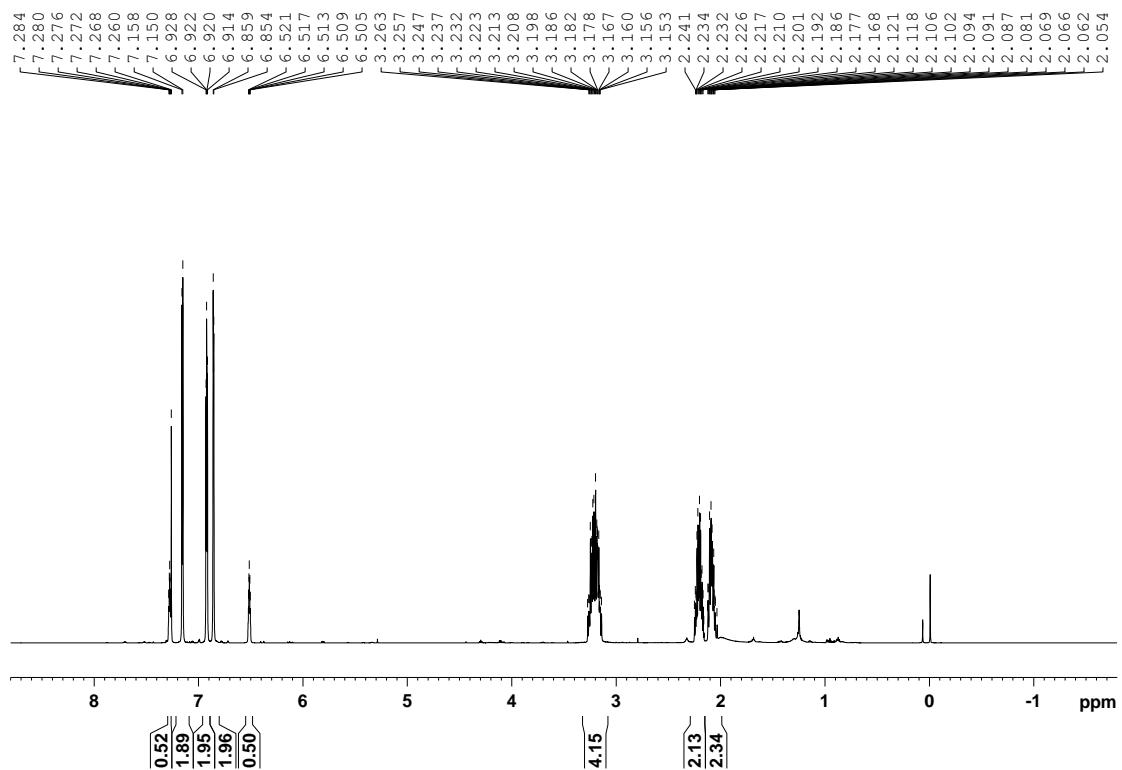
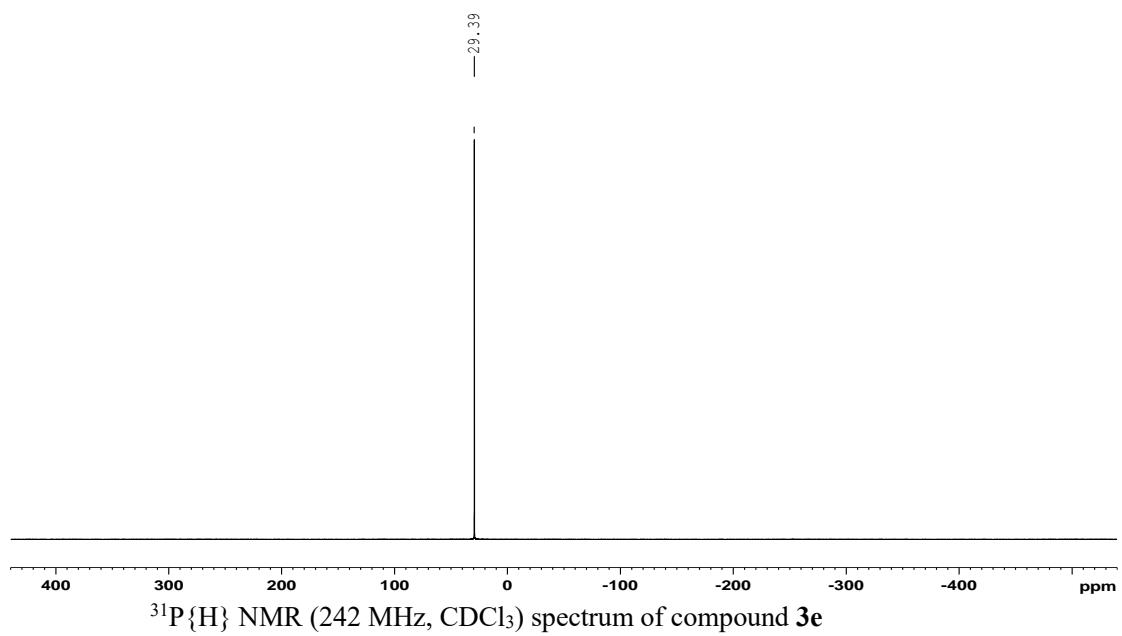
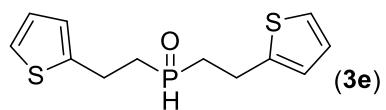
$^{31}\text{P}\{\text{H}\}$ NMR (242 MHz, CDCl_3) spectrum of compound **3d**



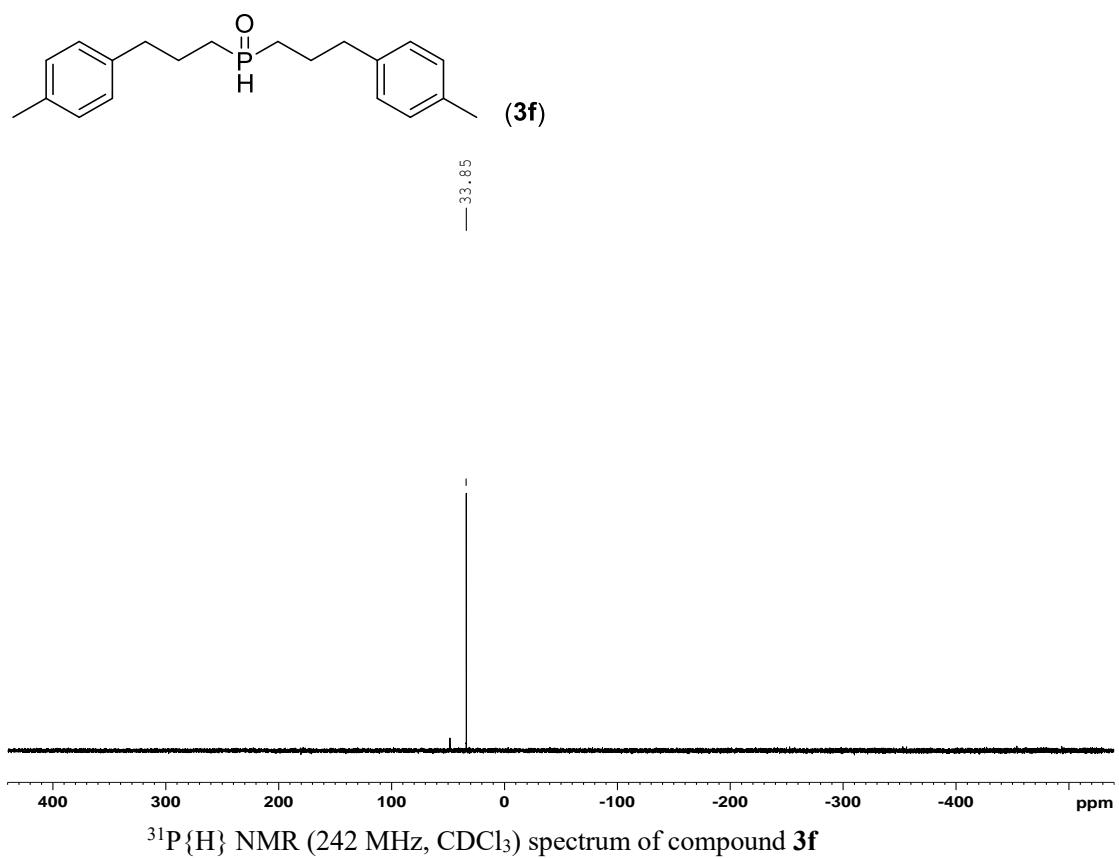
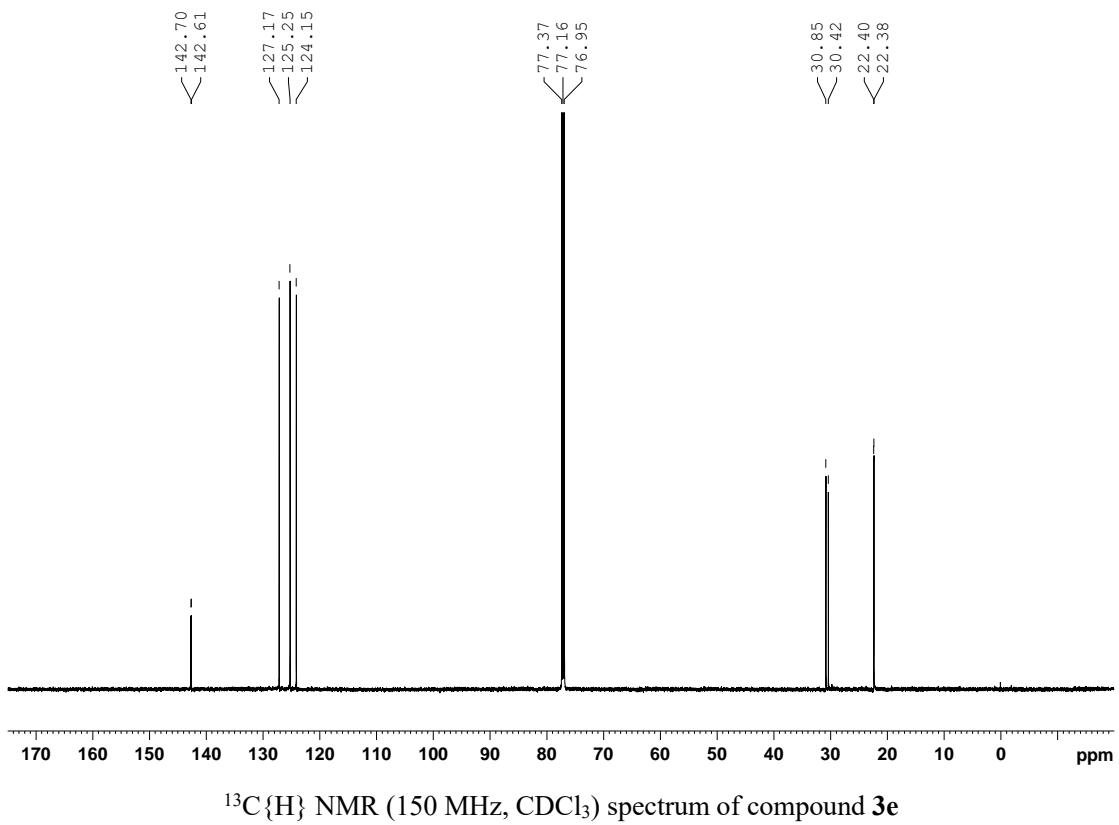
^1H NMR (600 MHz, CDCl_3) spectrum of compound **3d**

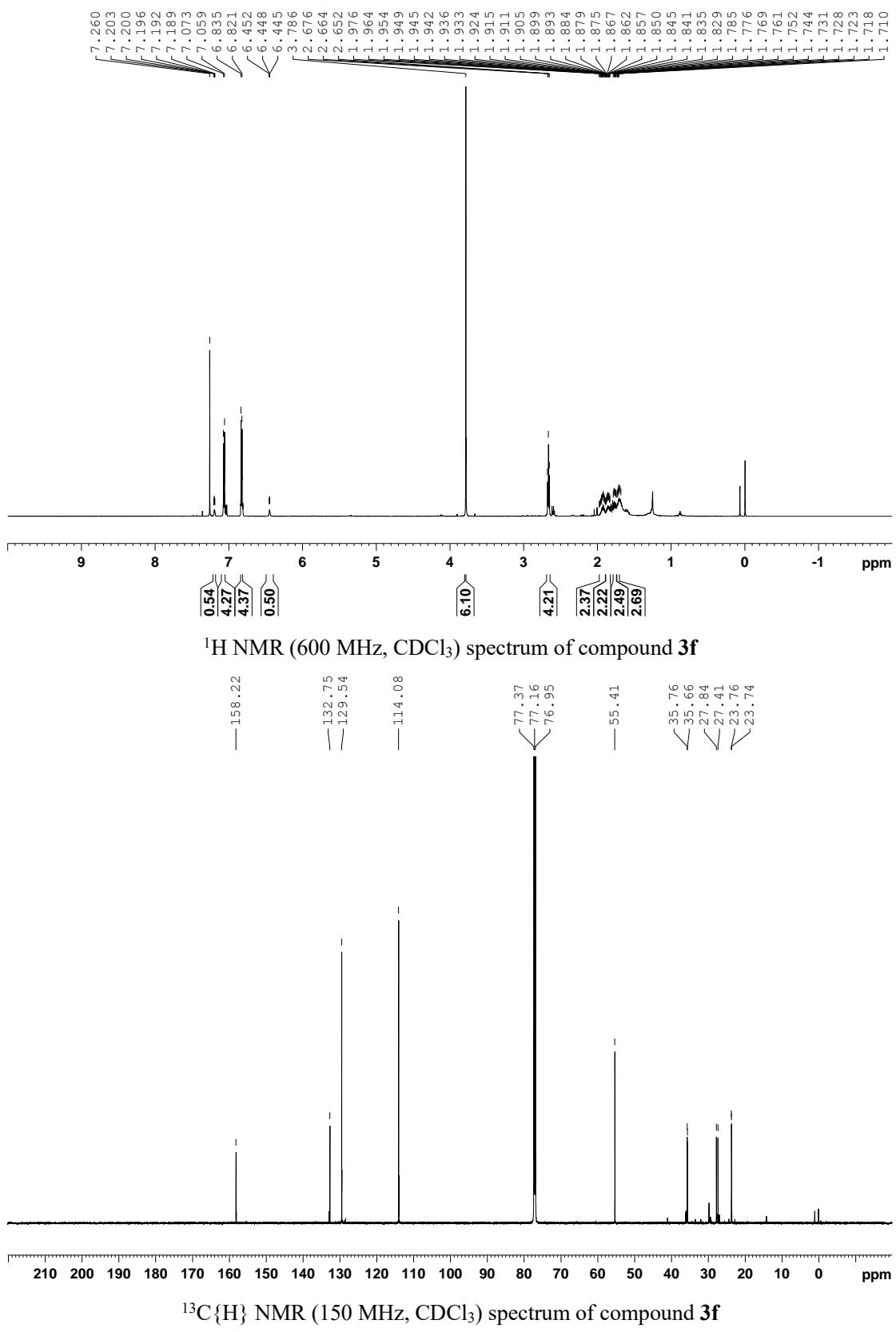


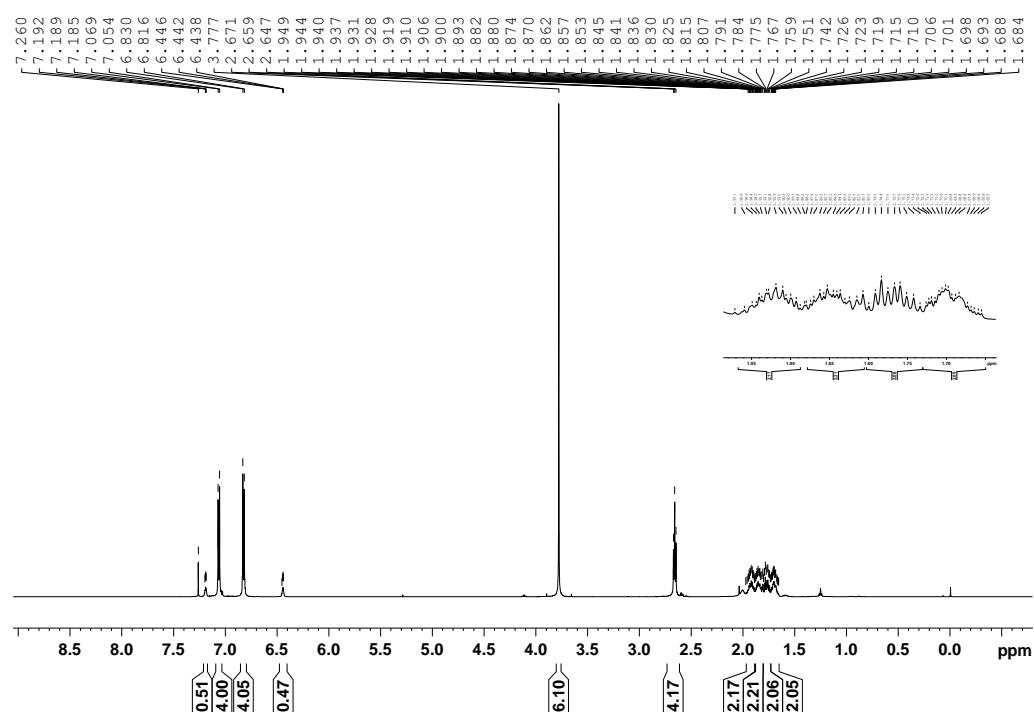
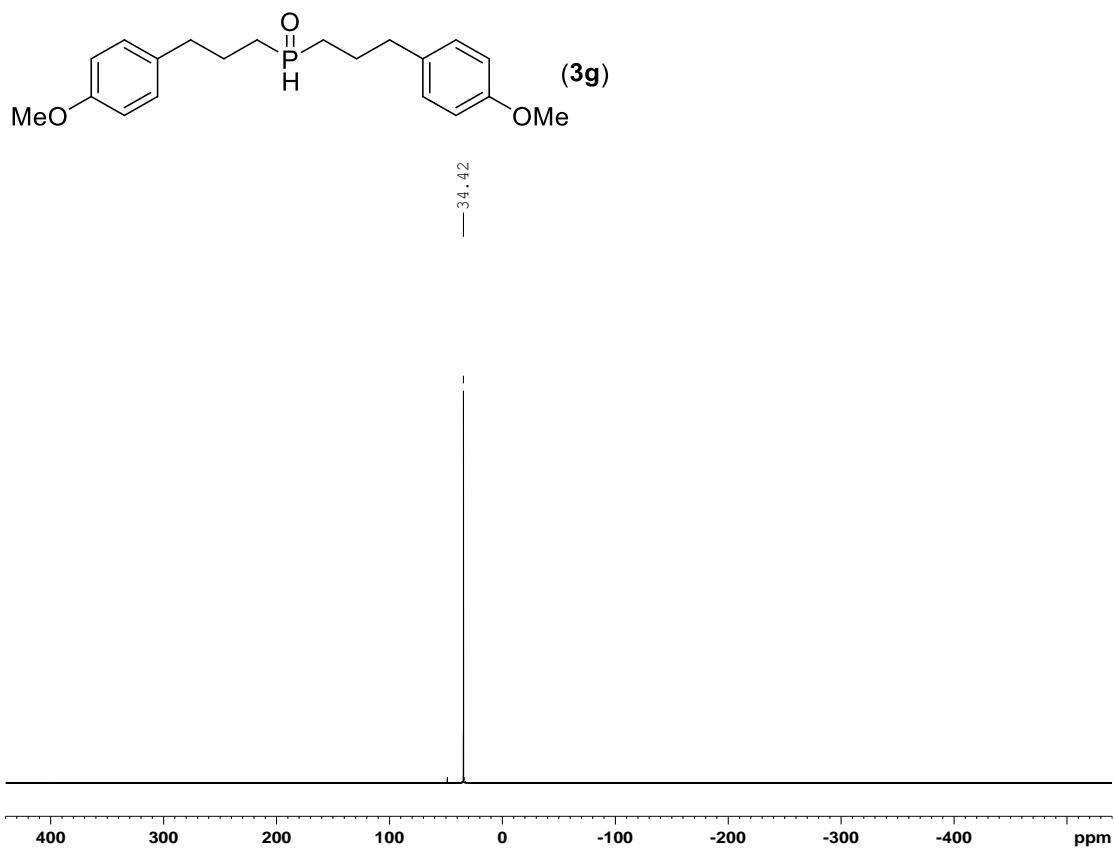
$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **3d**



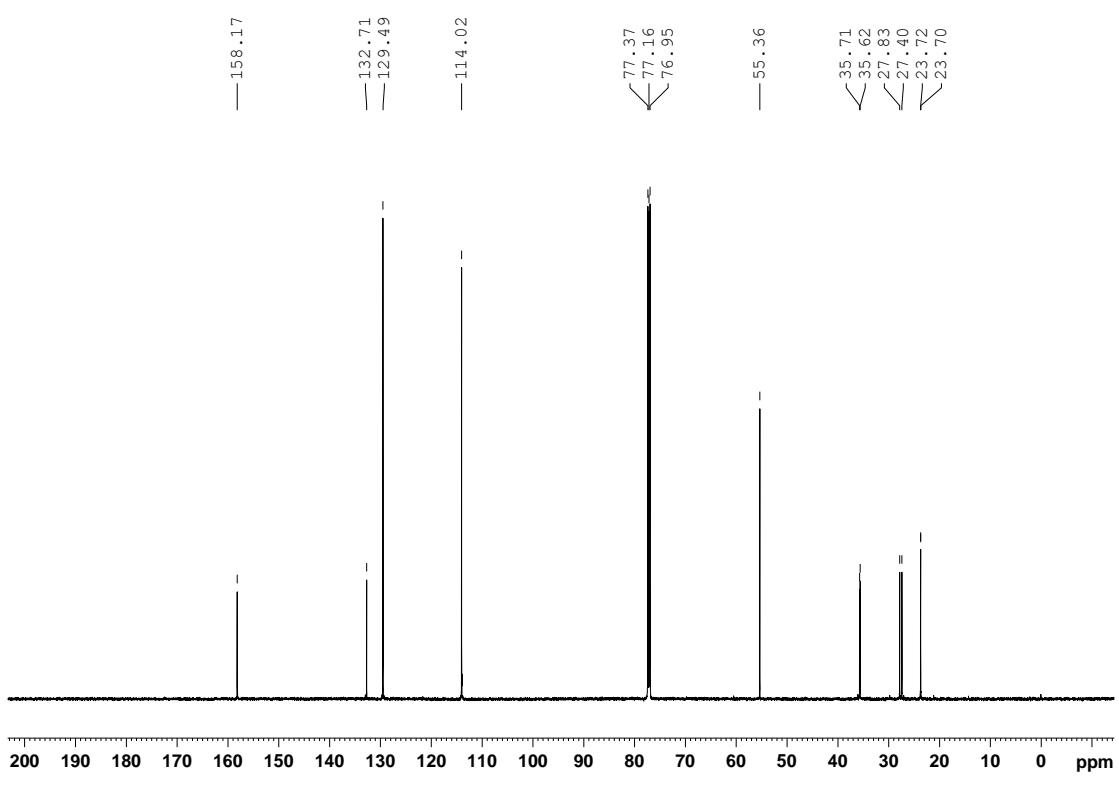
¹H NMR (600 MHz, CDCl₃) spectrum of compound 3e



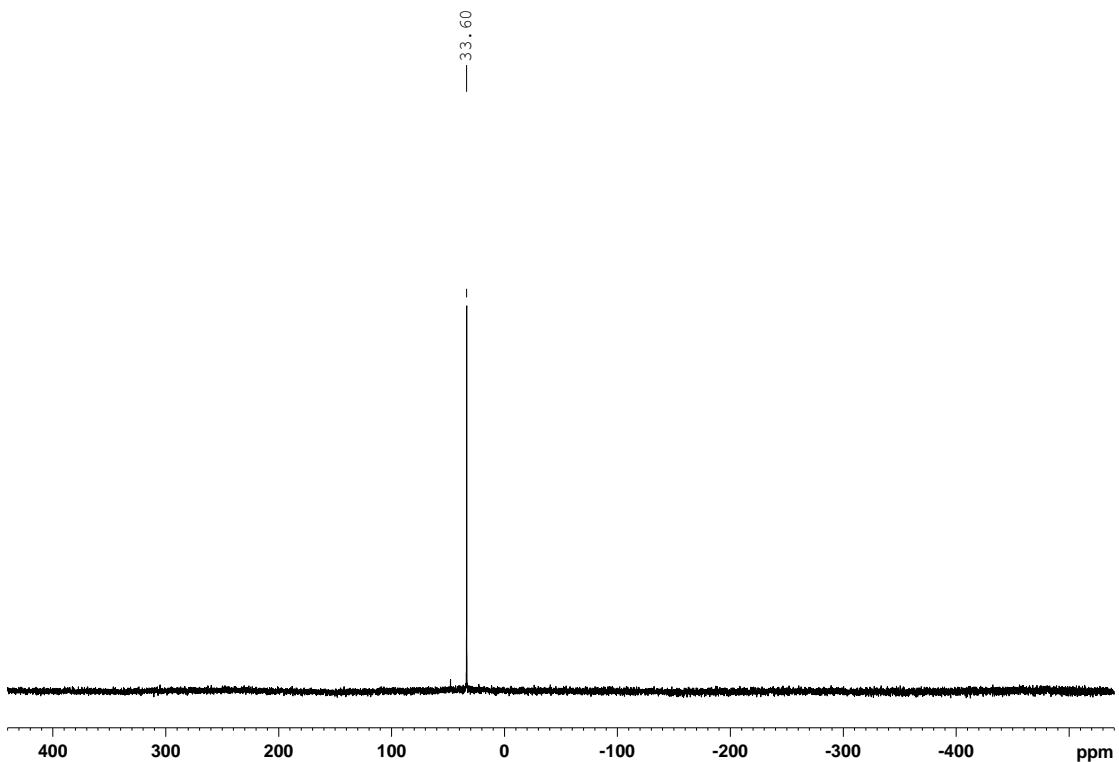
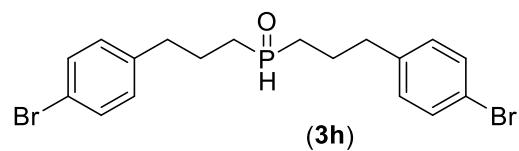




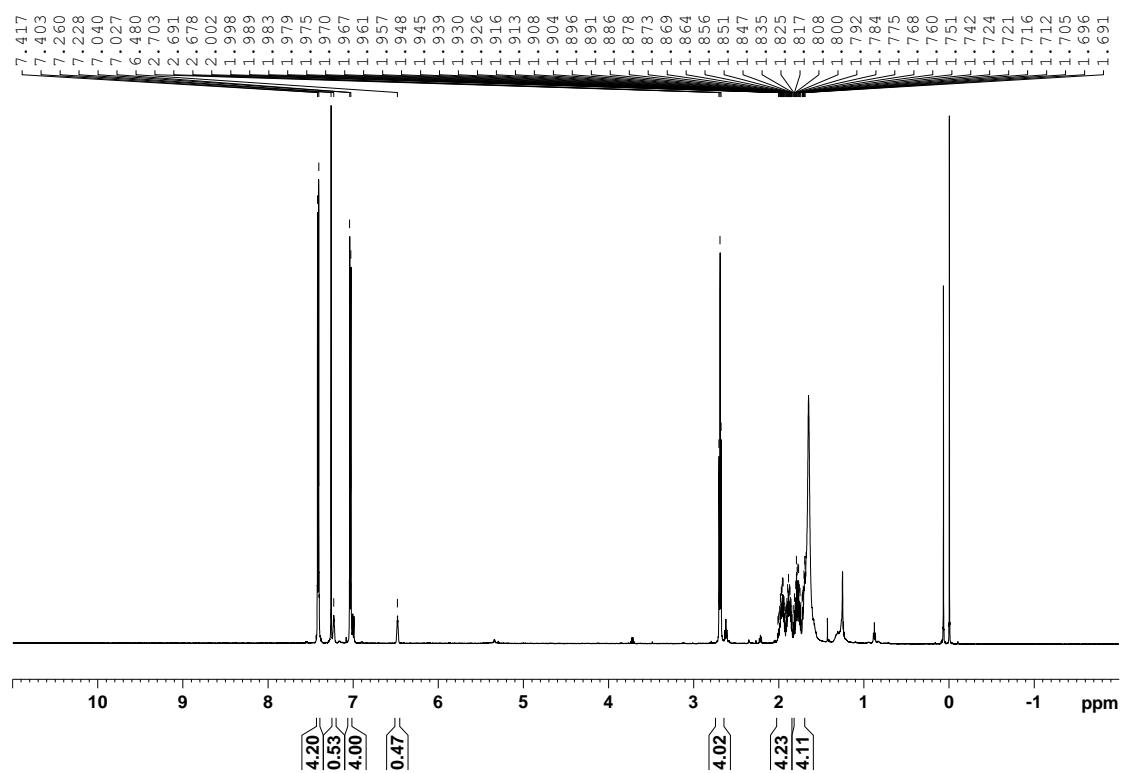
^1H NMR (600 MHz, CDCl_3) spectrum of compound **3g**



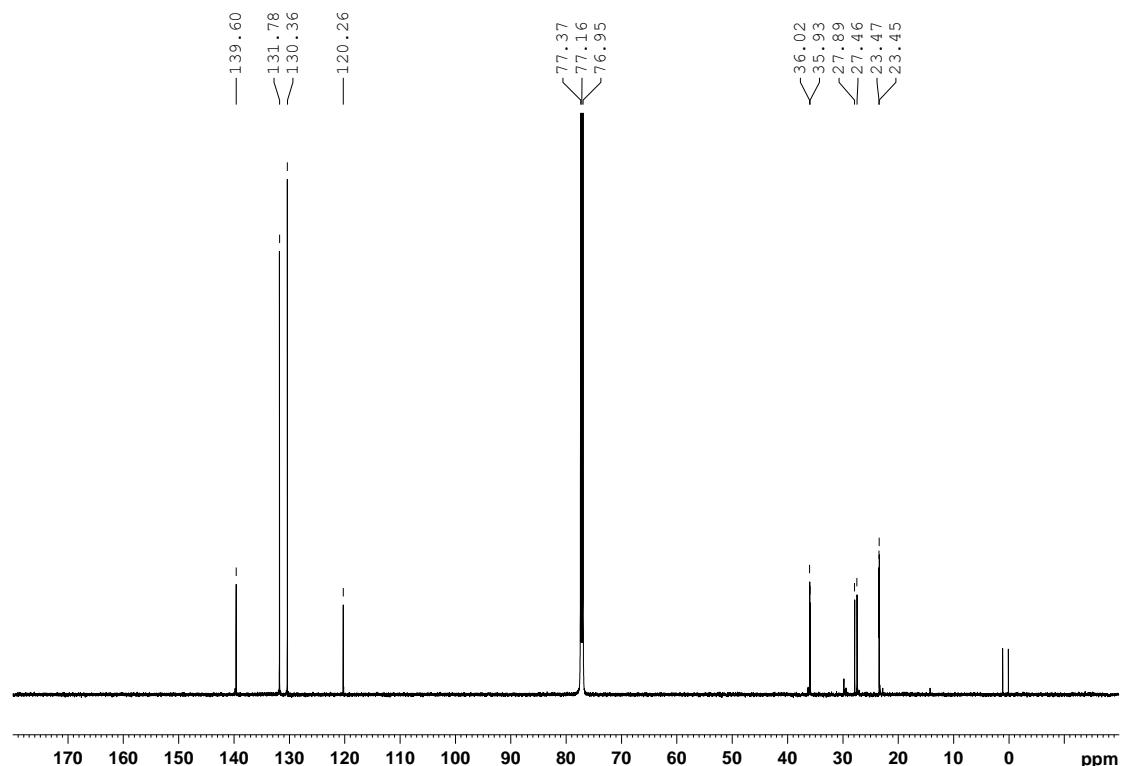
$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **3g**



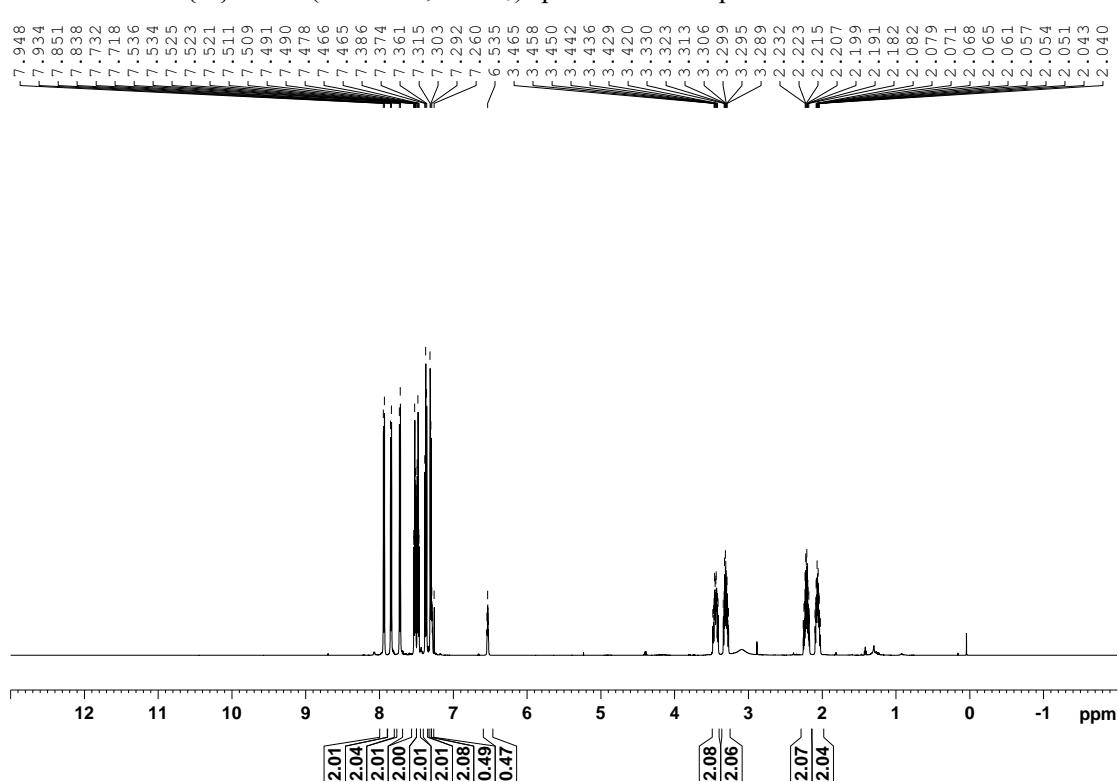
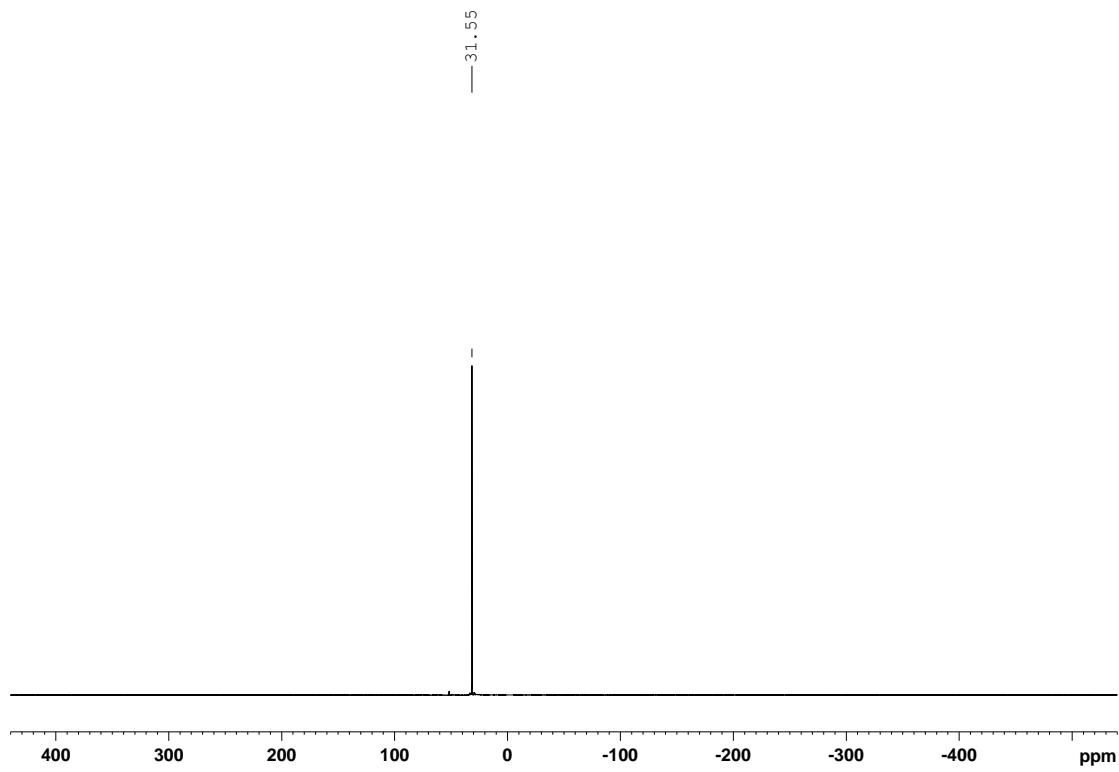
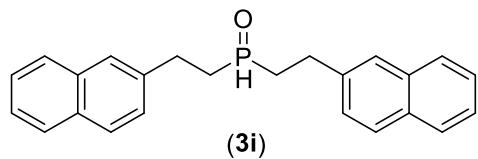
$^{31}\text{P}\{\text{H}\}$ NMR (242 MHz, CDCl_3) spectrum of compound **3h**

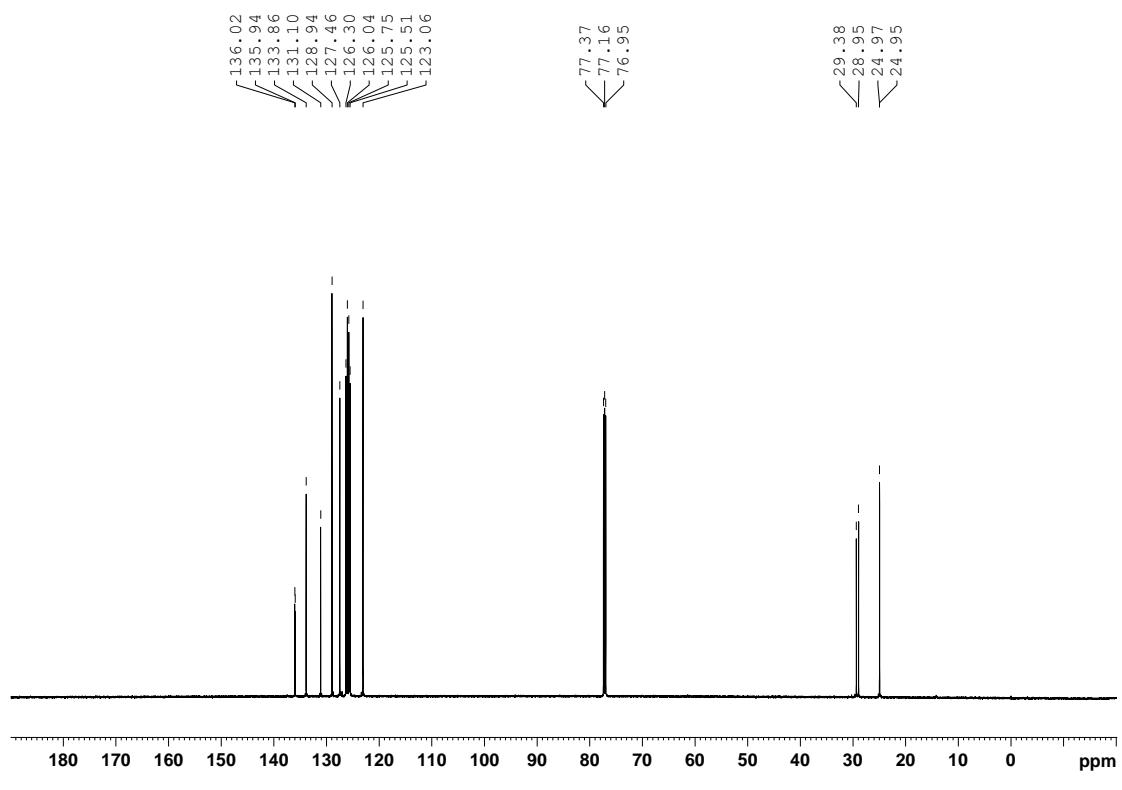


^1H NMR (600 MHz, CDCl_3) spectrum of compound **3h**

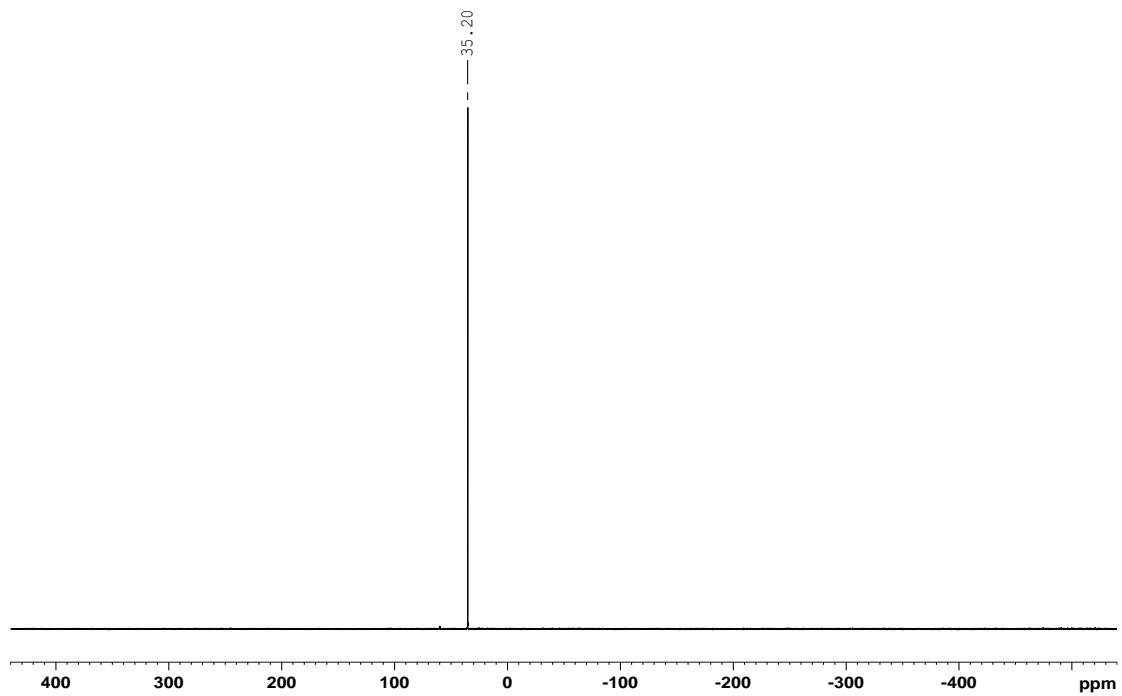
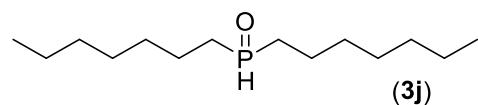


$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **3h**

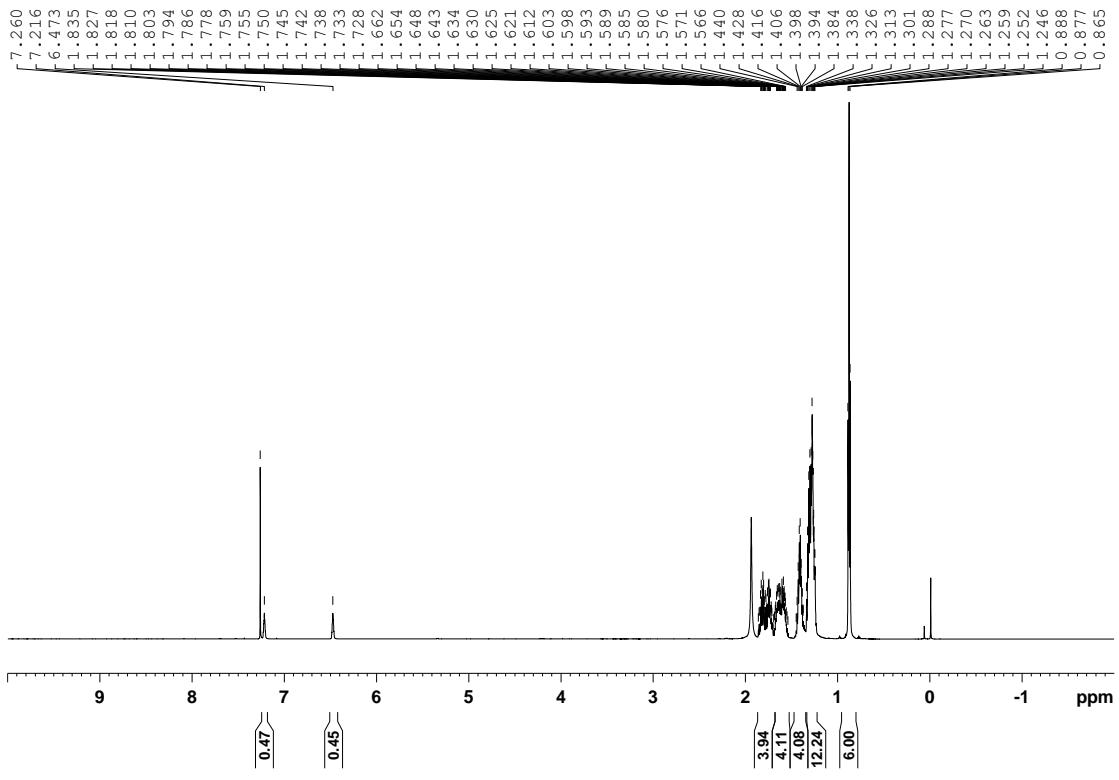




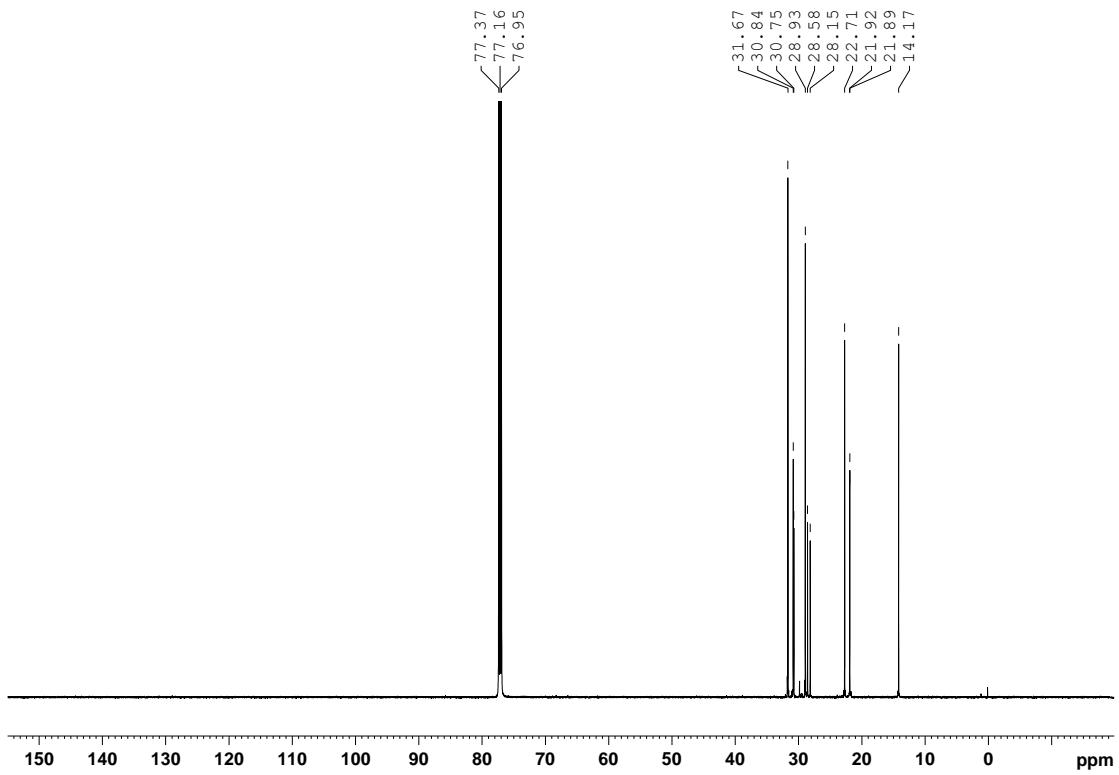
¹³C{H} NMR (150 MHz, CDCl₃) spectrum of compound **3i**



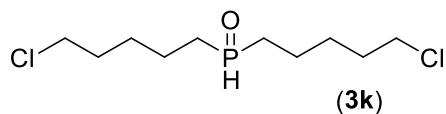
^{31}P {H} NMR (242 MHz, CDCl_3) spectrum of compound **3j**



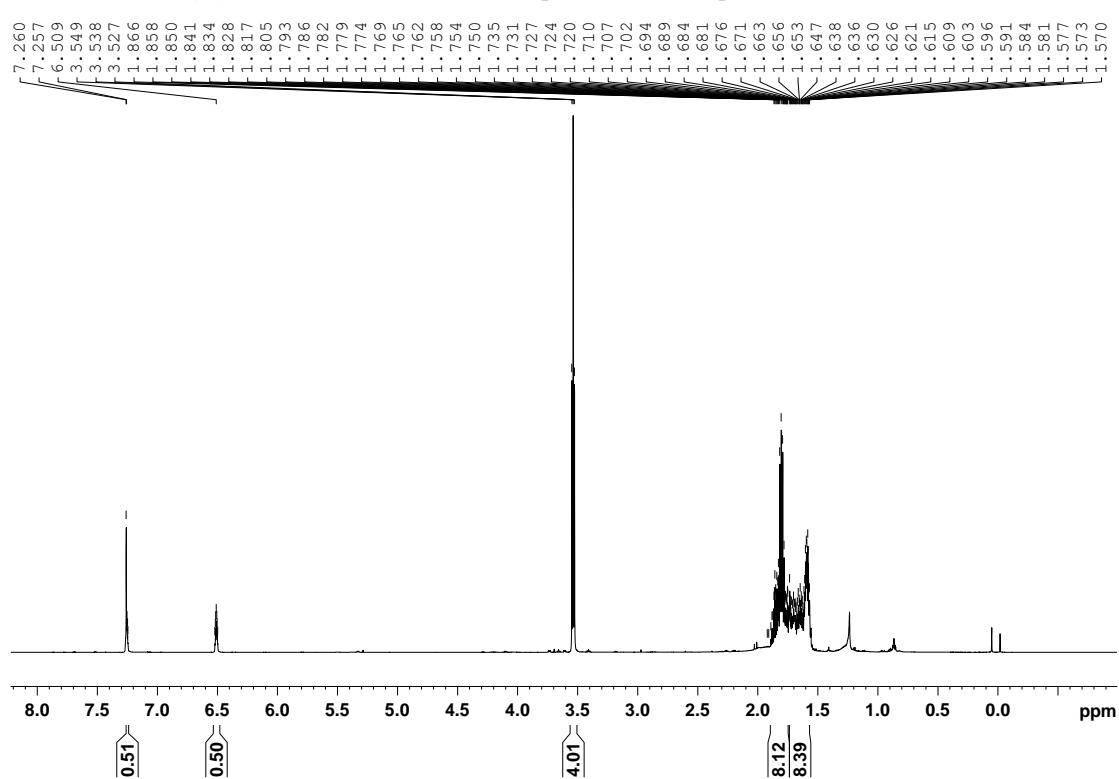
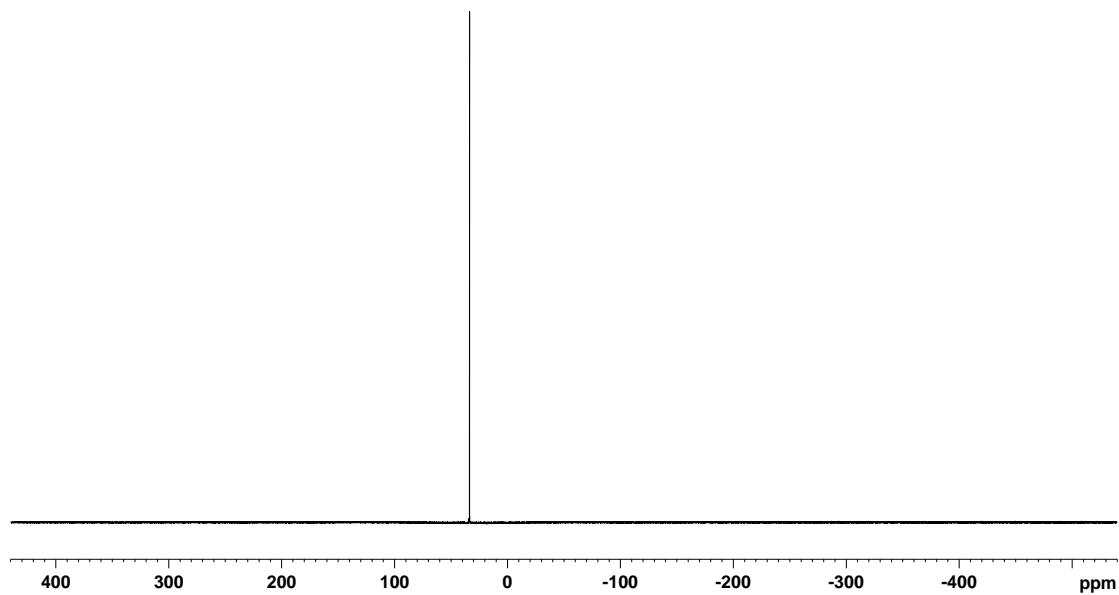
^1H NMR (600 MHz, CDCl_3) spectrum of compound **3j**

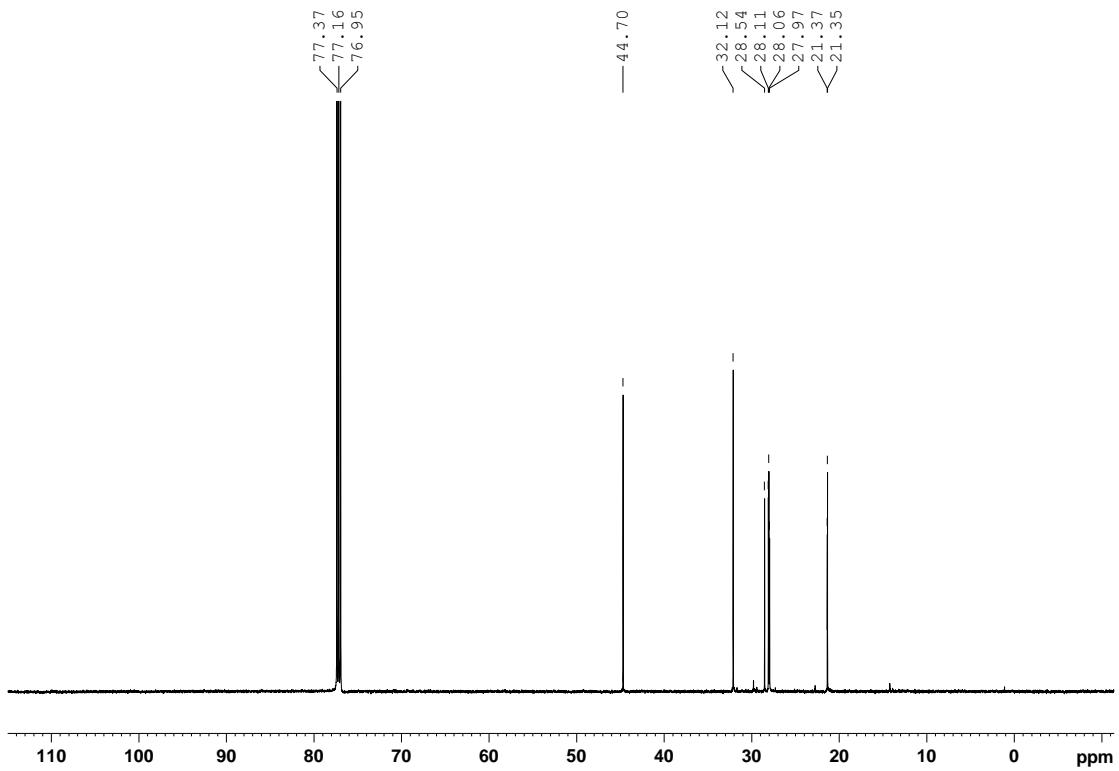


$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **3j**

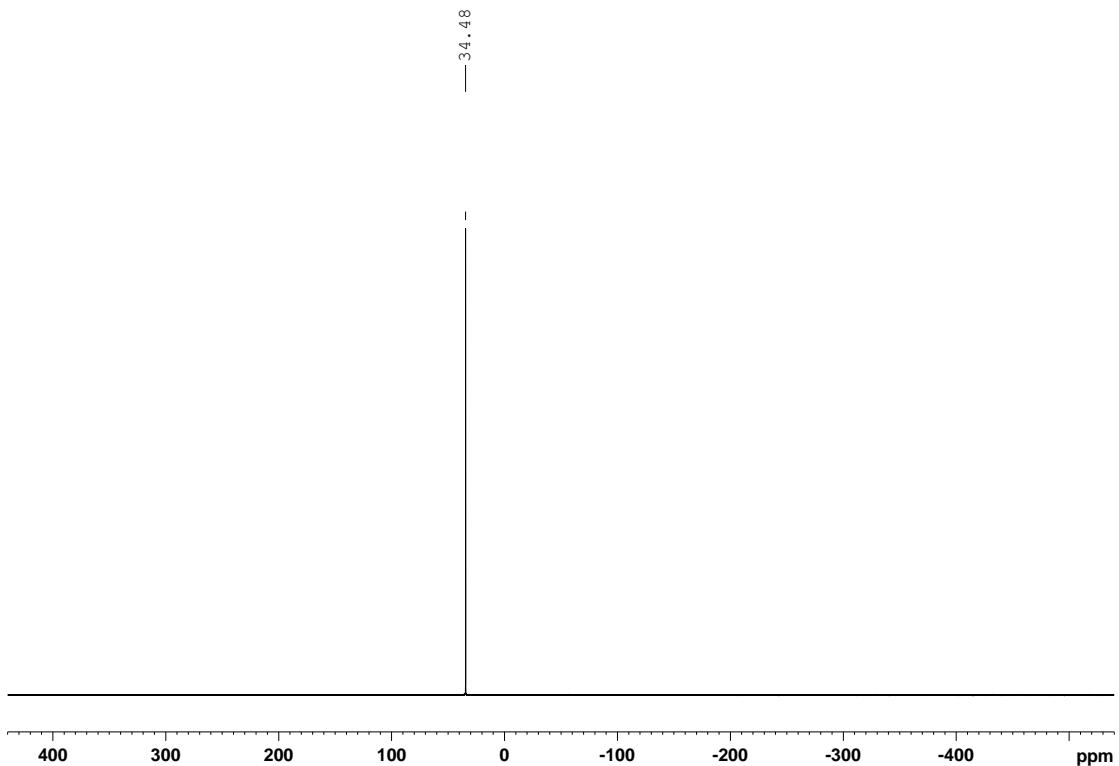
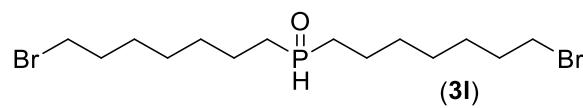


— 33.70

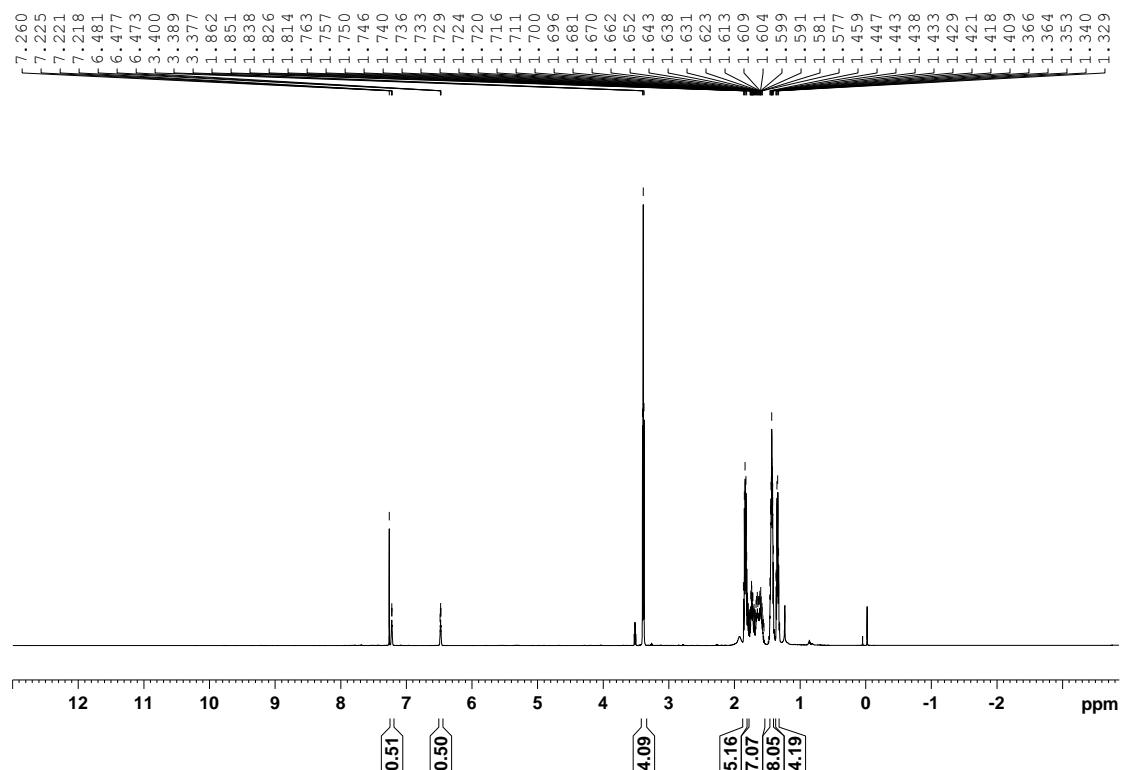




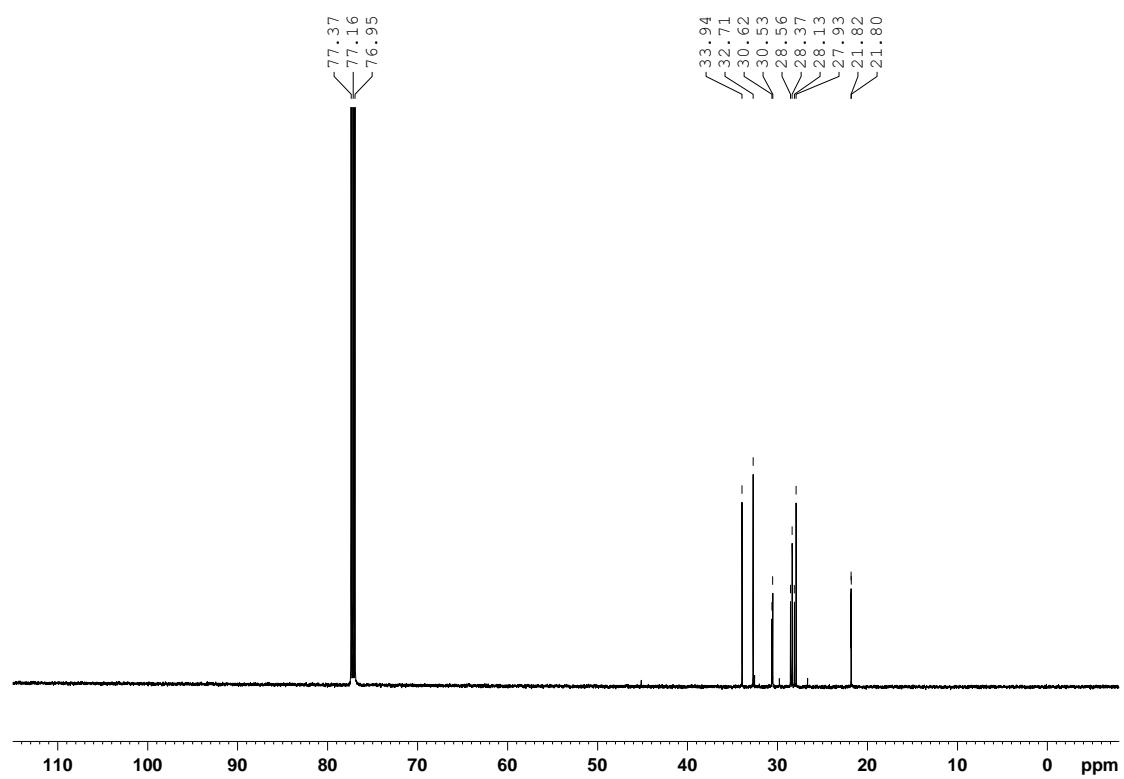
$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **3k**



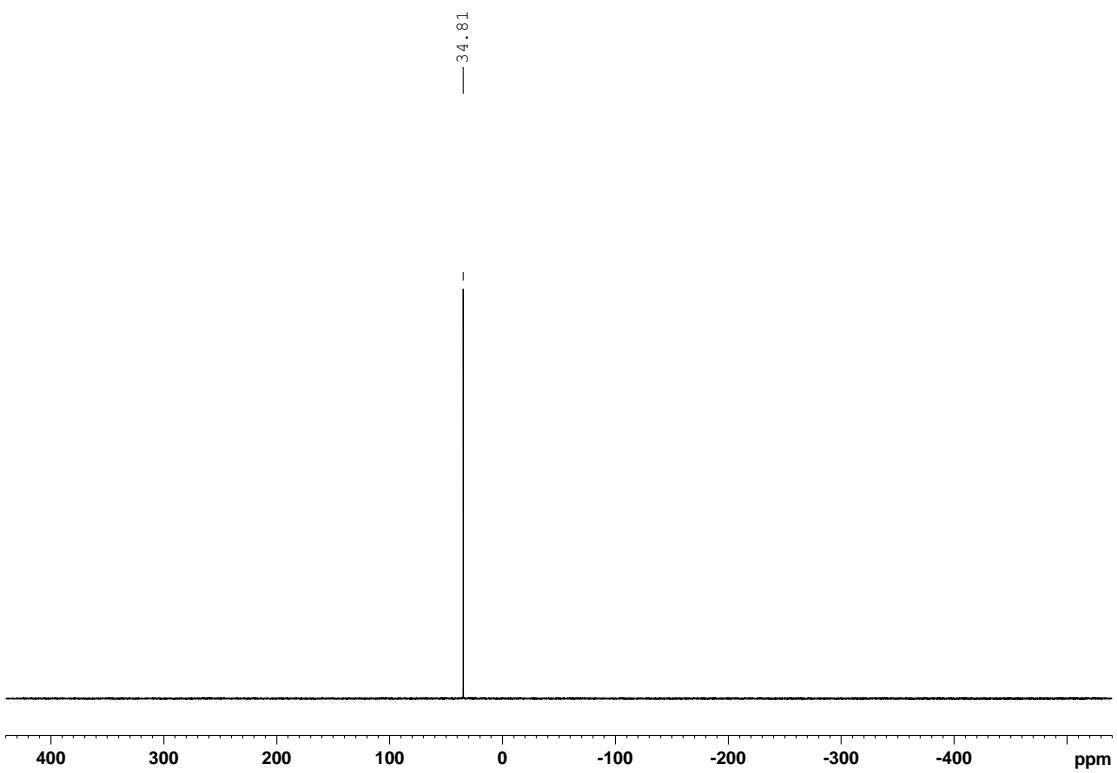
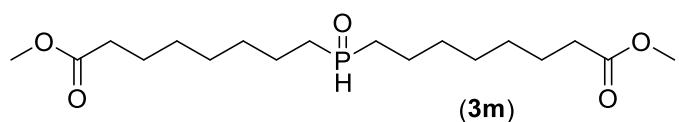
$^{31}\text{P}\{\text{H}\}$ NMR (242 MHz, CDCl_3) spectrum of compound **3l**



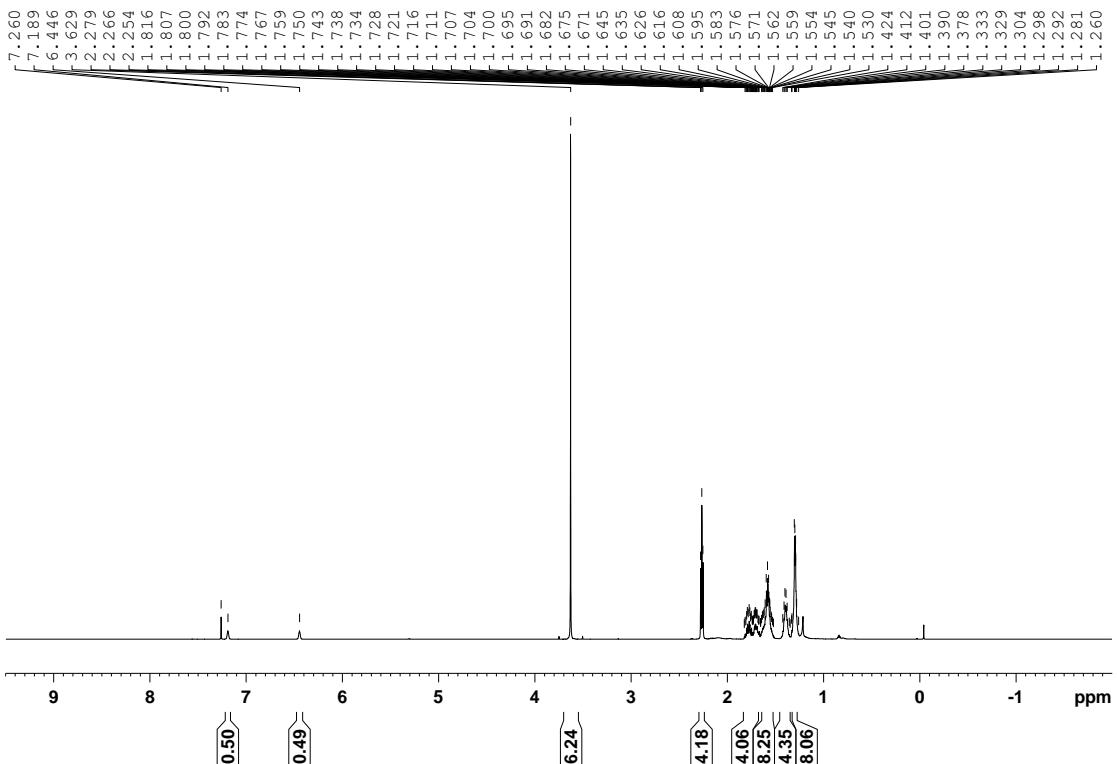
^1H NMR (600 MHz, CDCl_3) spectrum of compound **3l**



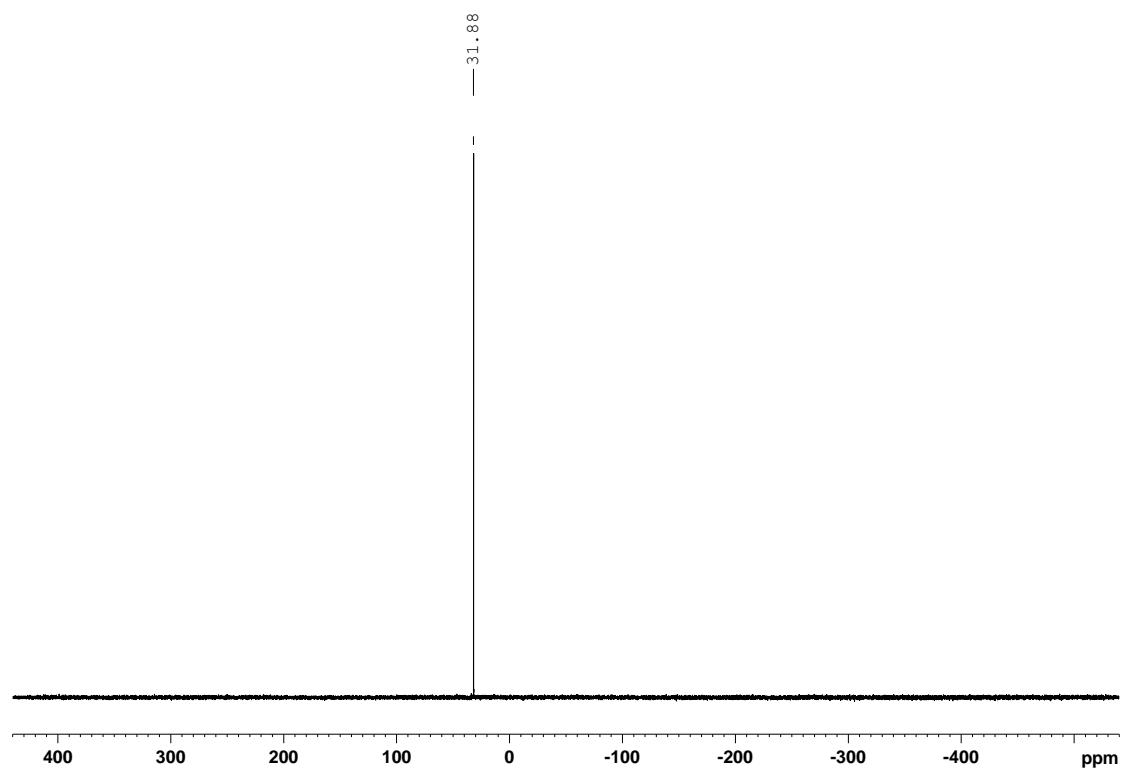
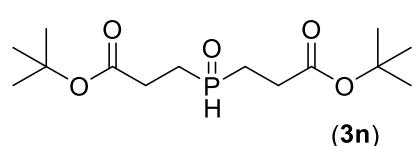
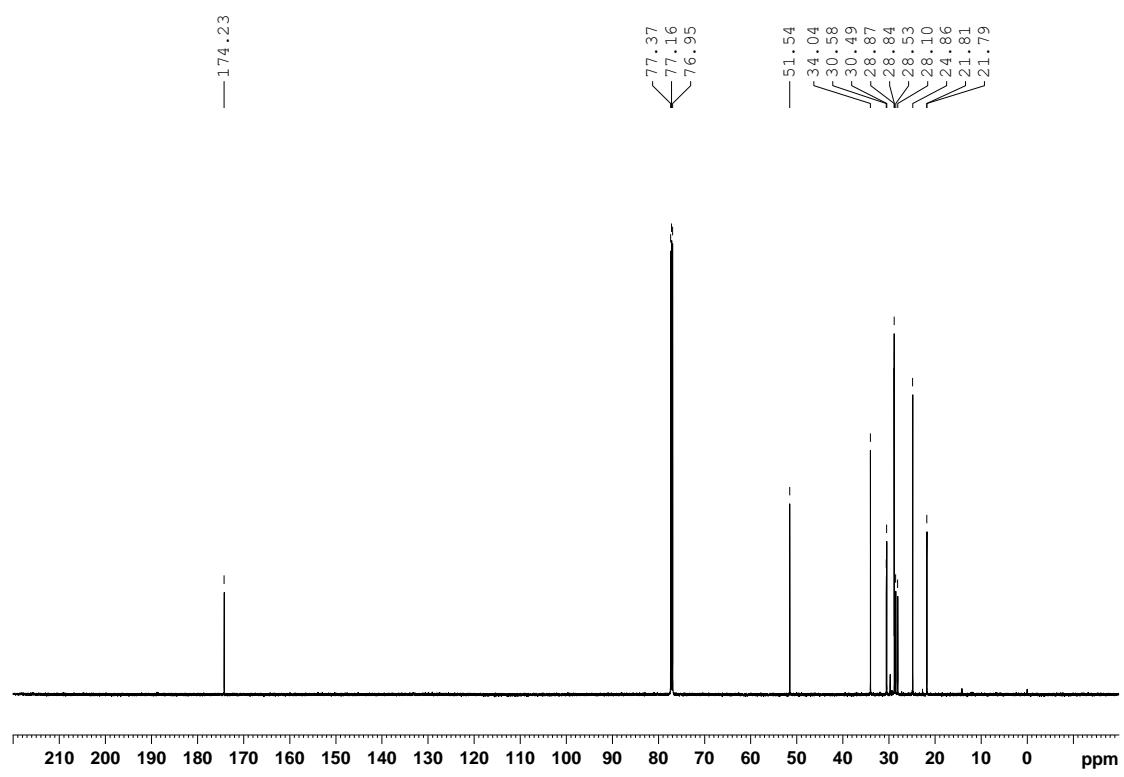
$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **3l**



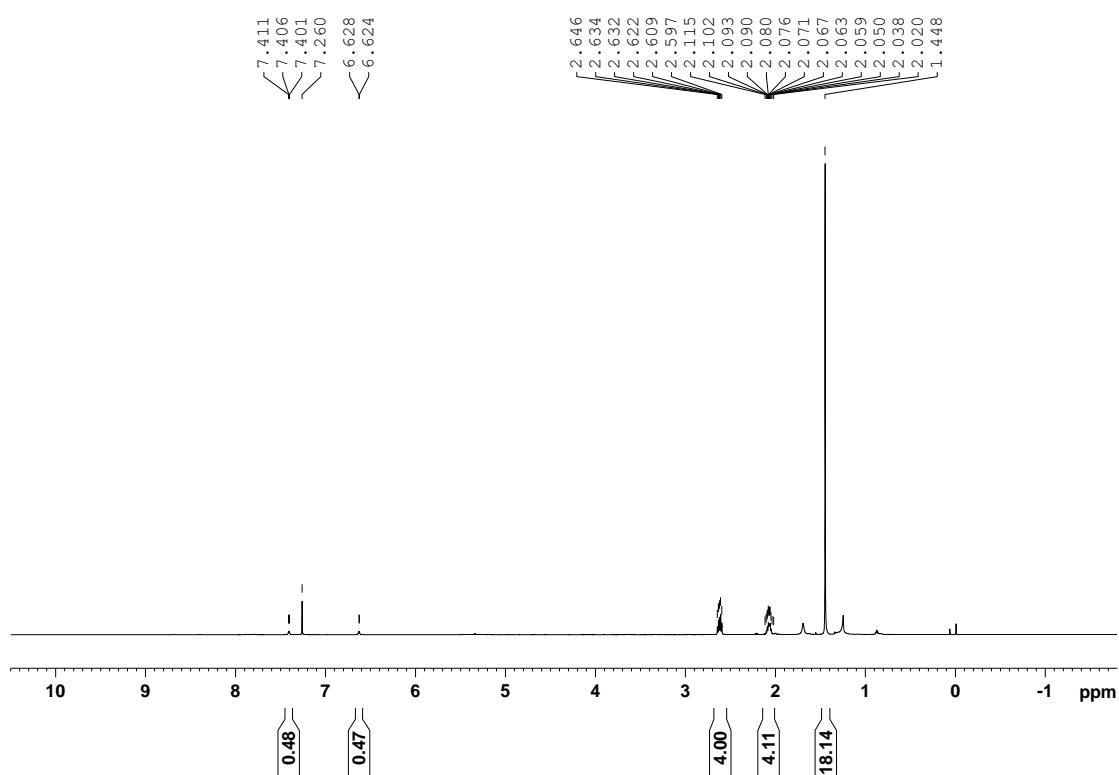
$^{31}\text{P}\{\text{H}\}$ NMR (242 MHz, CDCl_3) spectrum of compound **3m**



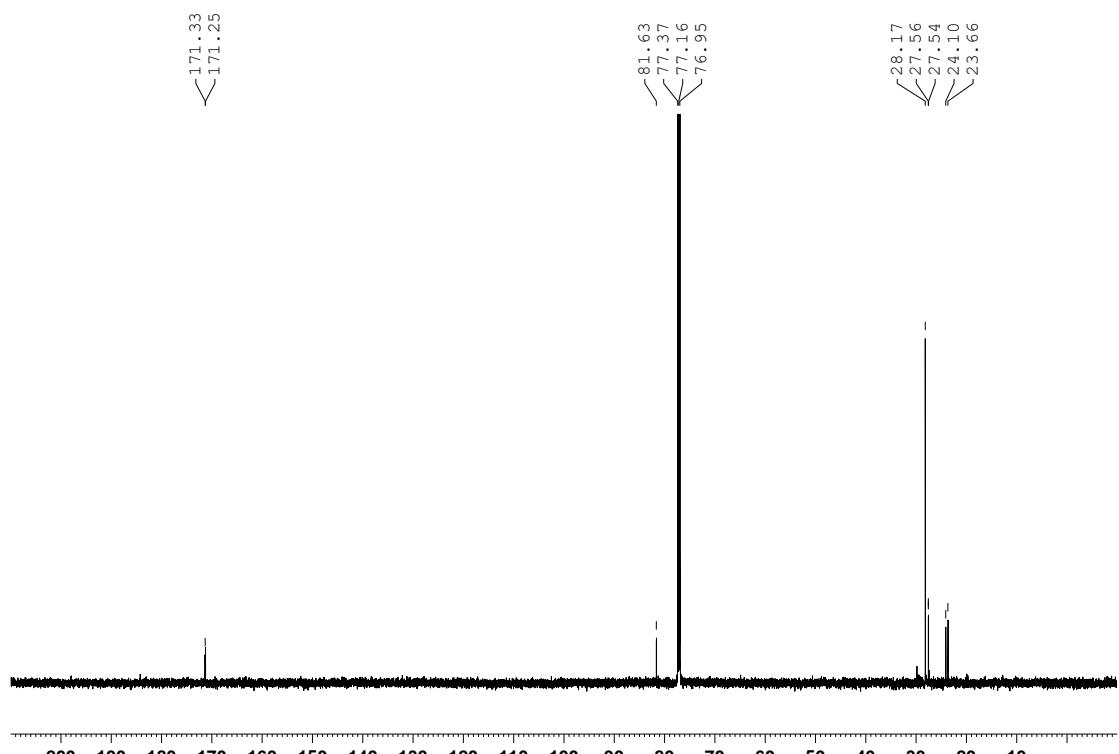
¹H NMR (600 MHz, CDCl₃) spectrum of compound 3m



$^{31}\text{P}\{\text{H}\}$ NMR (242 MHz, CDCl_3) spectrum of compound **3n**

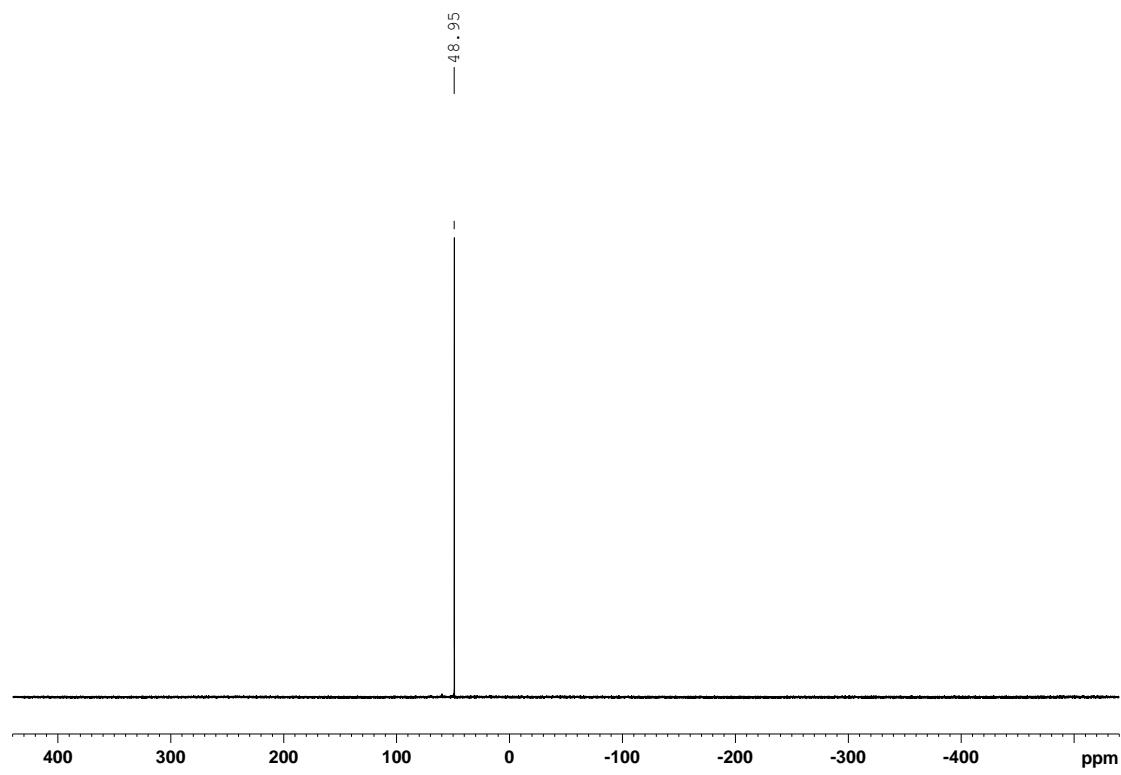
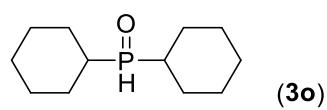


^1H NMR (600 MHz, CDCl_3) spectrum of compound **3n**



^1H NMR (600 MHz, CDCl_3) spectrum of compound **3n**

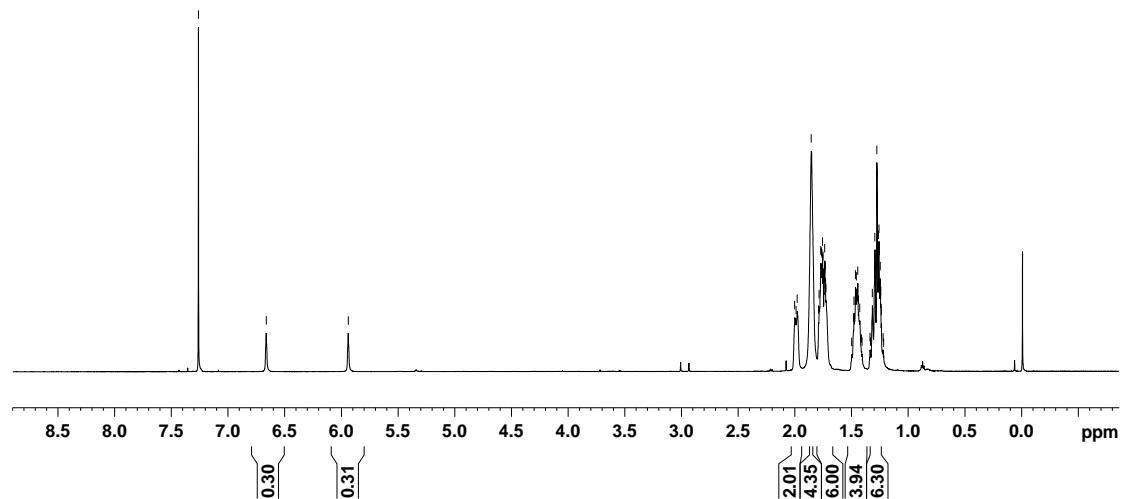
$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **3n**



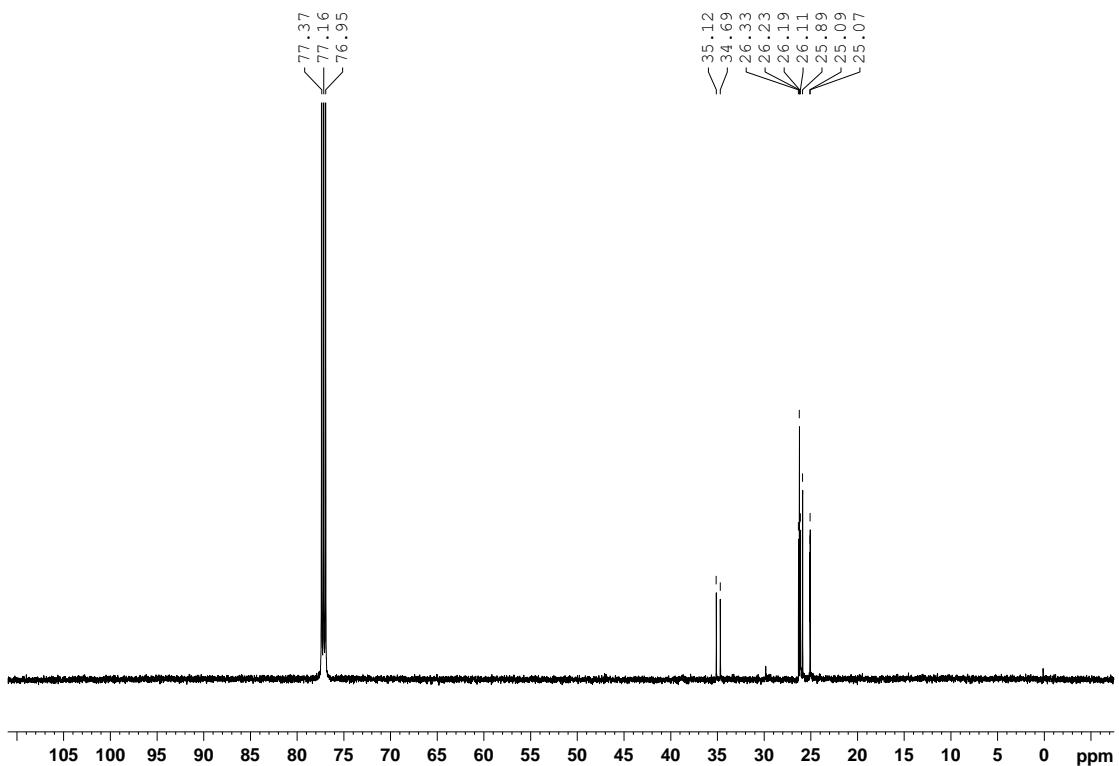
³¹P{H} NMR (242 MHz, CDCl₃) spectrum of compound **3o**

— 7.260
 — 6.663
 — 5.939

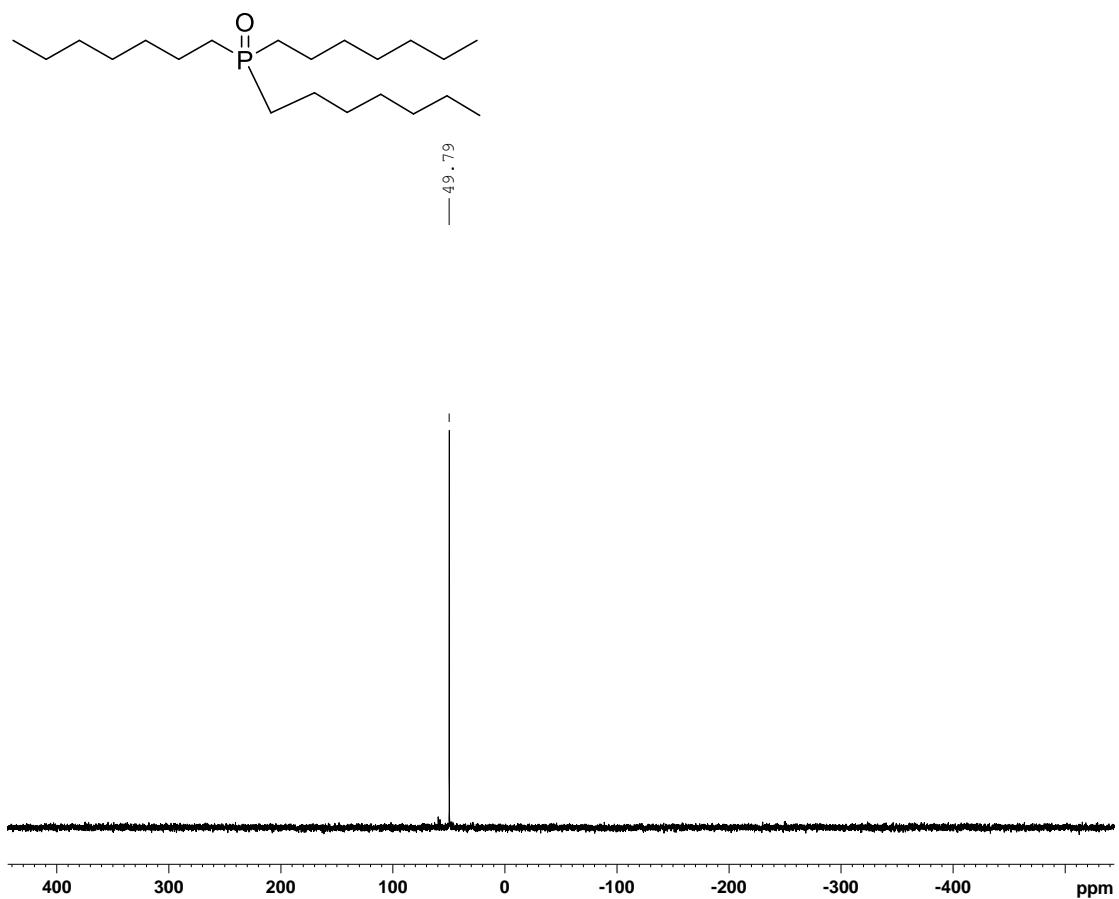
2.001
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 1.771
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 1.761
 1.756
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 1.736
 1.731
 1.725
 1.7198
 1.71483
 1.71478
 1.71465
 1.71457
 1.71445
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 1.71262
 1.71258



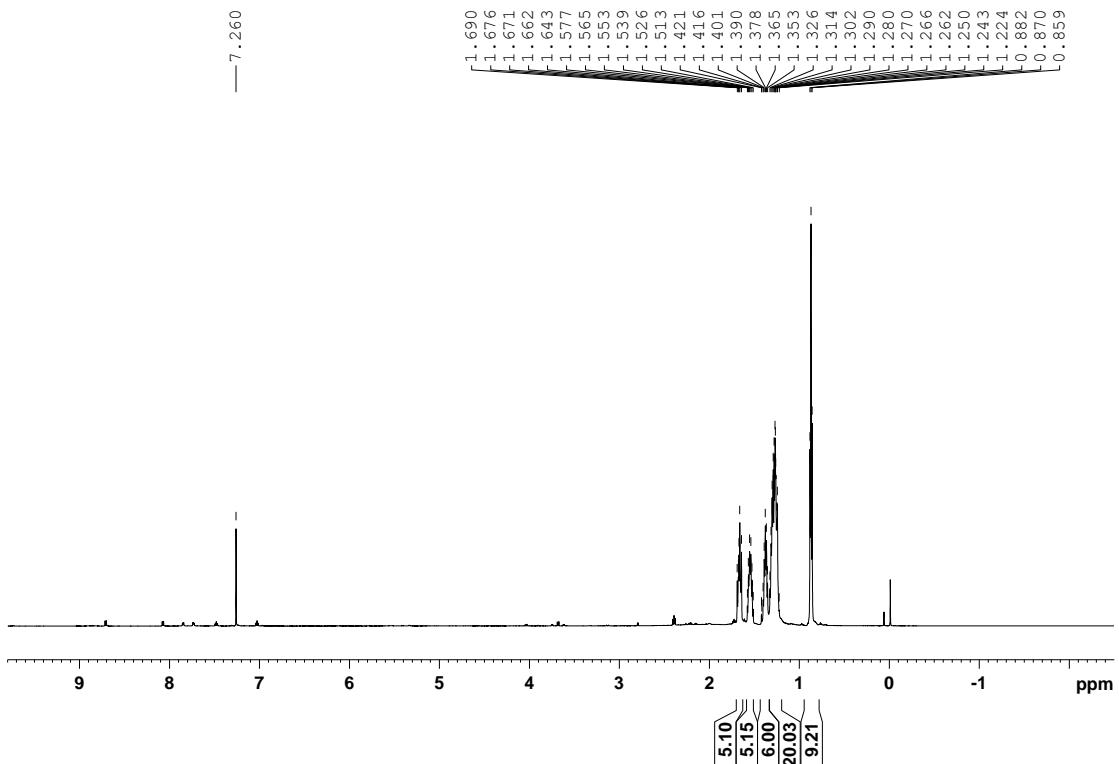
¹H NMR (600 MHz, CDCl₃) spectrum of compound **3o**



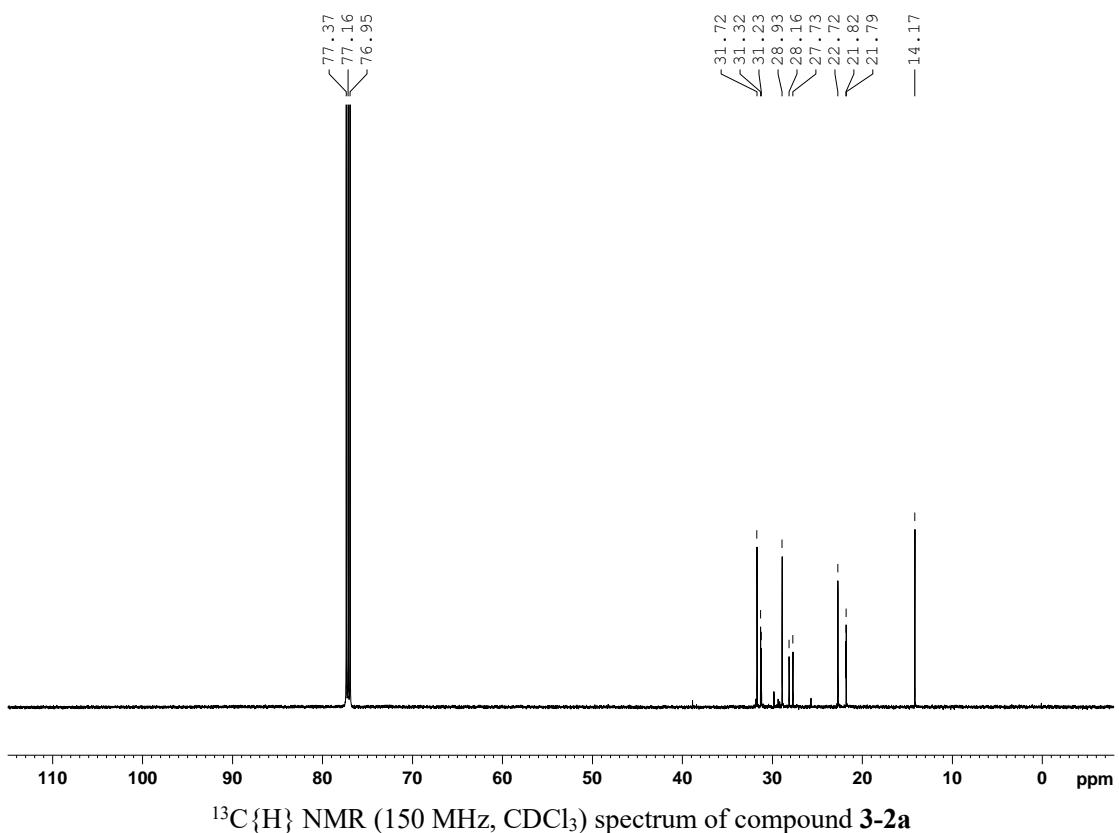
$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **3o**



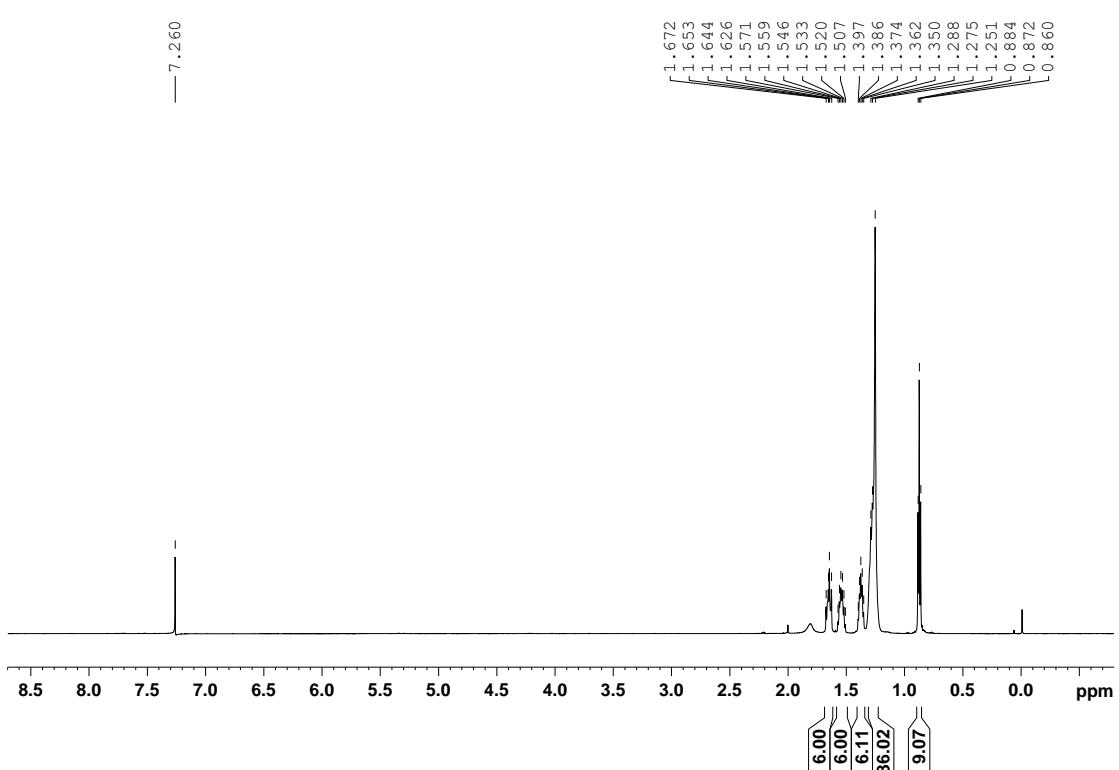
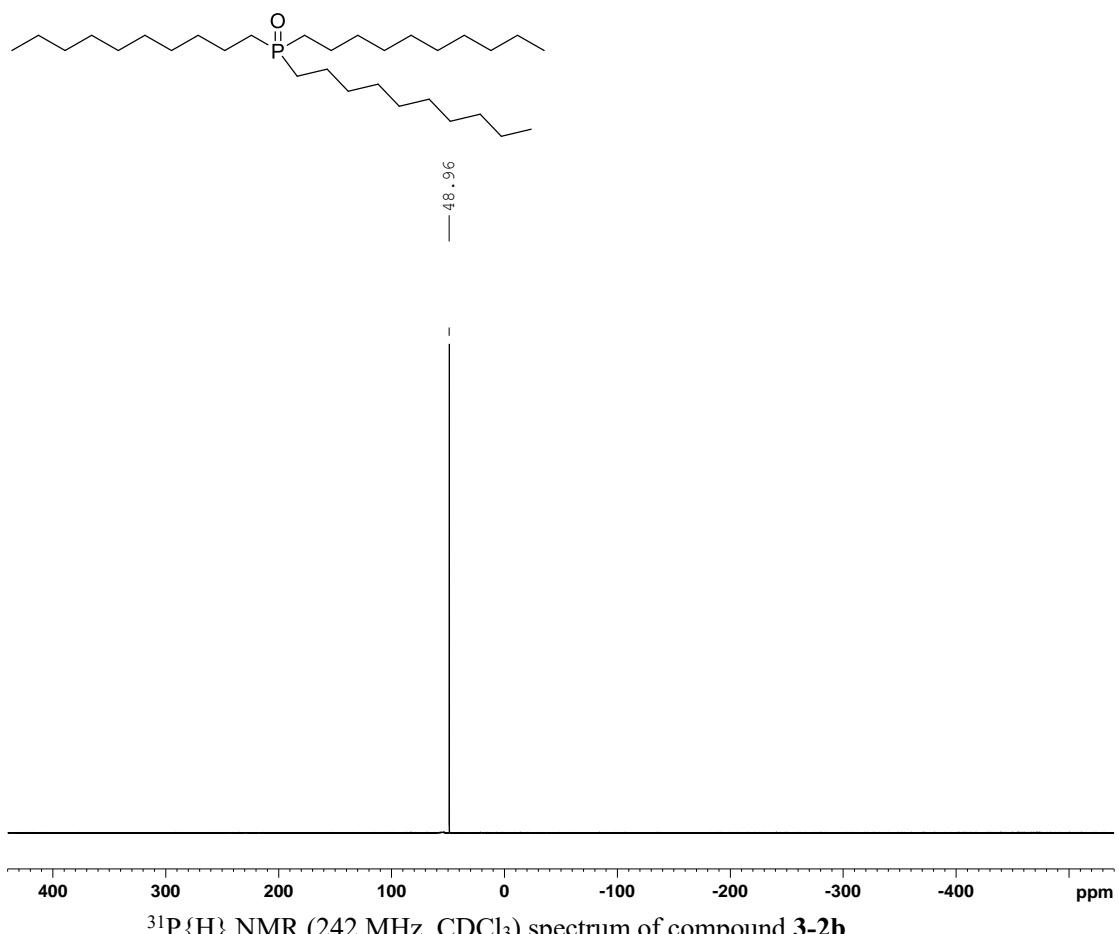
$^{31}\text{P}\{\text{H}\}$ NMR (242 MHz, CDCl_3) spectrum of compound **3-2a**



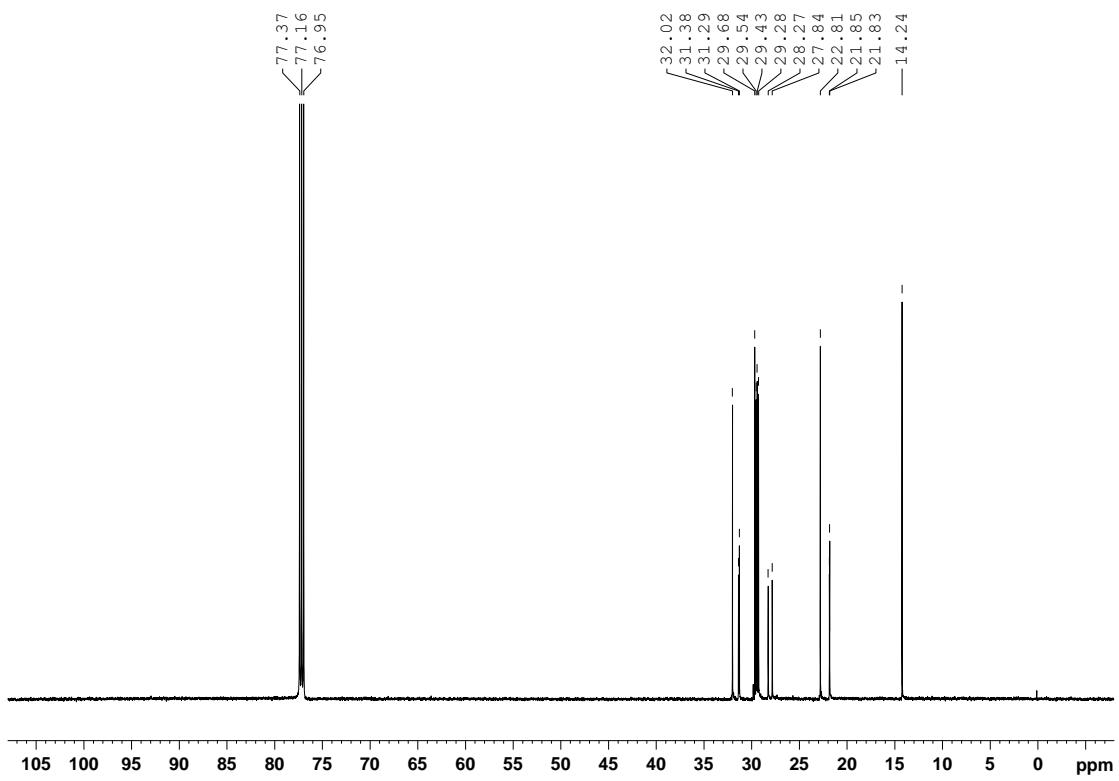
^1H NMR (600 MHz, CDCl_3) spectrum of compound **3-2a**



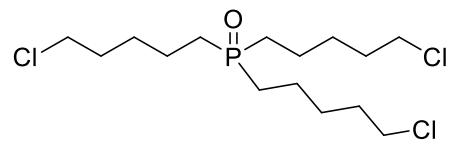
$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **3-2a**



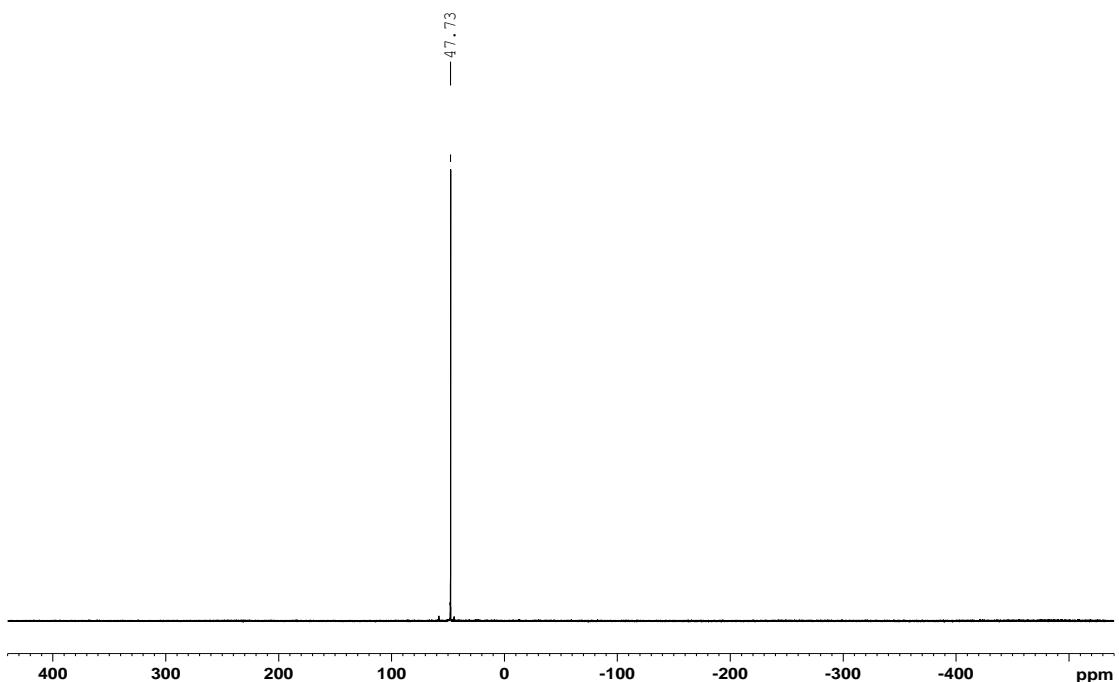
¹H NMR (600 MHz, CDCl₃) spectrum of compound **3-2b**



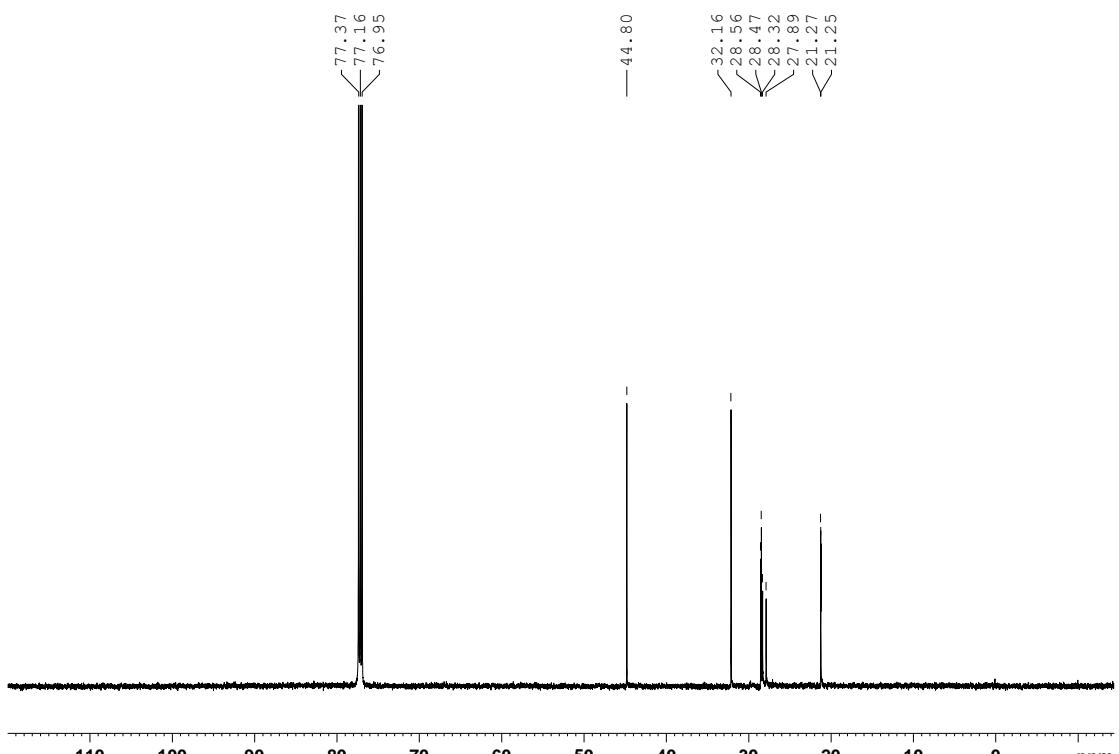
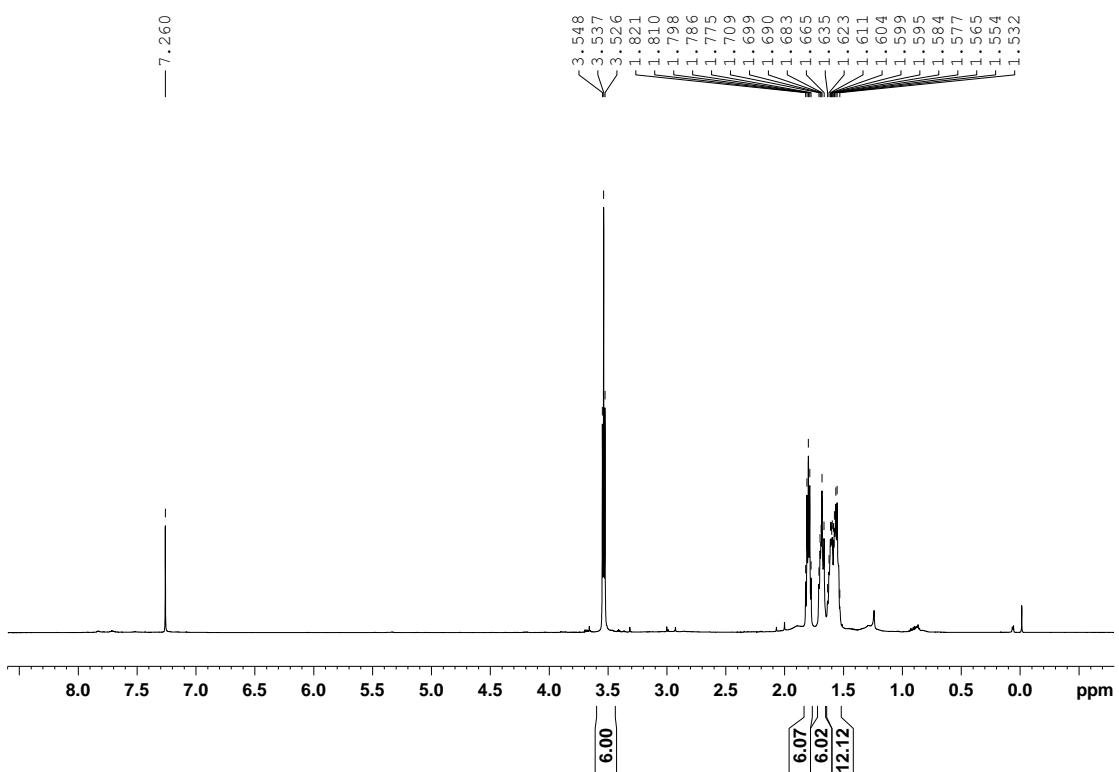
$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **3-2b**



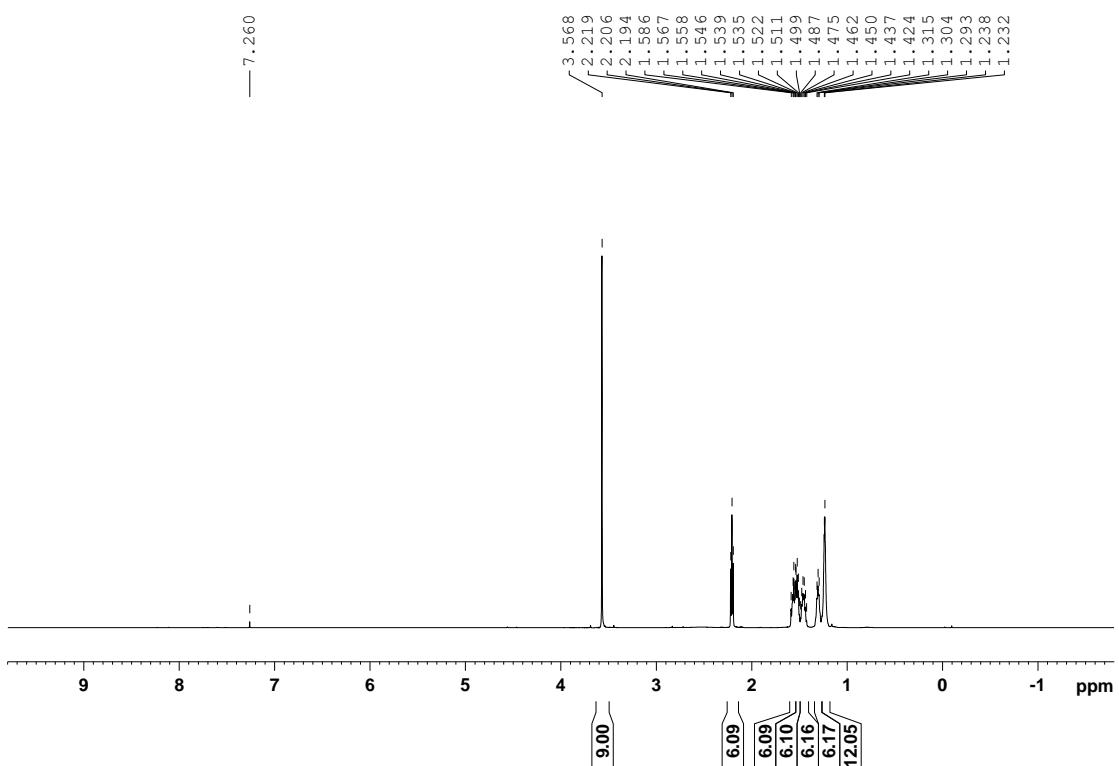
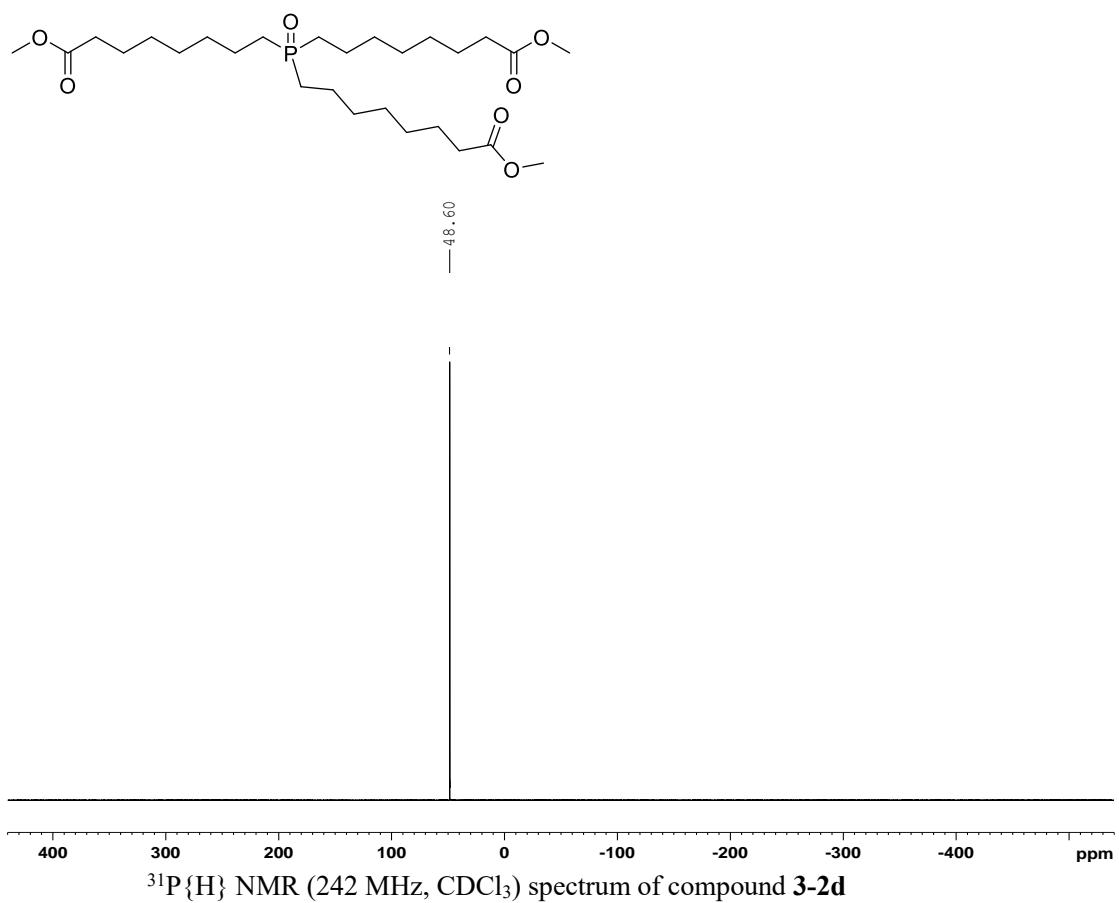
—47.73



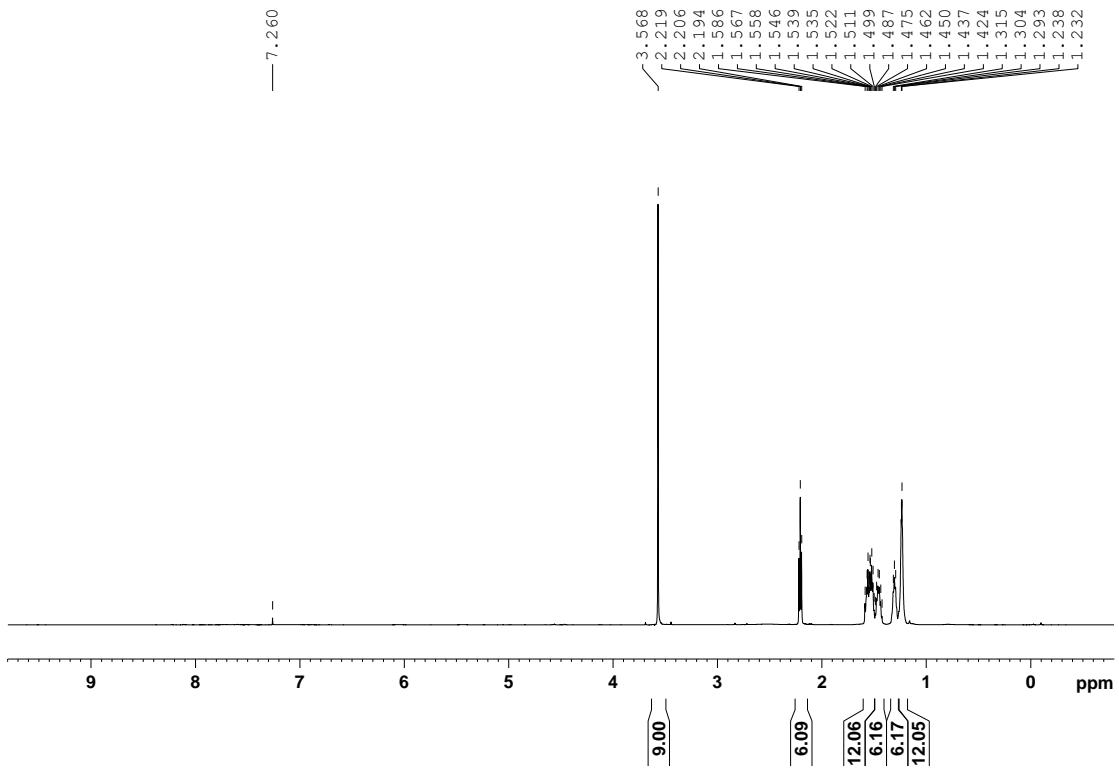
$^{31}\text{P}\{\text{H}\}$ NMR (242 MHz, CDCl_3) spectrum of compound **3-2c**



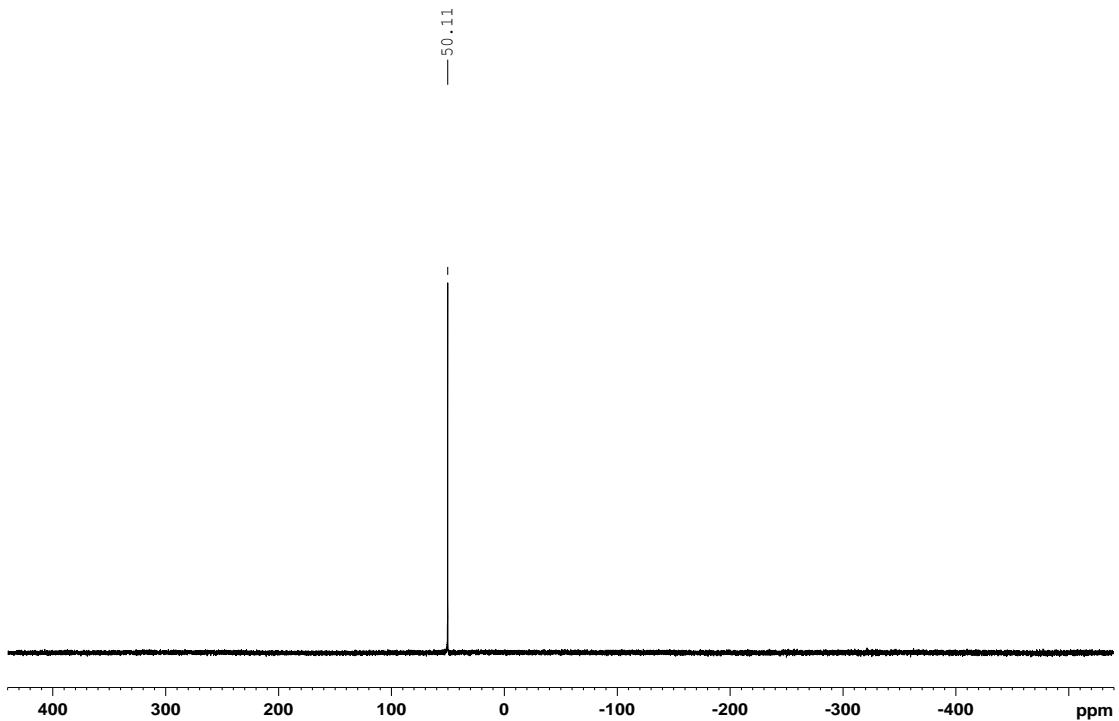
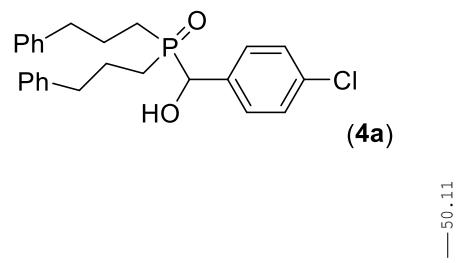
¹³C{H} NMR (150 MHz, CDCl₃) spectrum of compound 3-2c



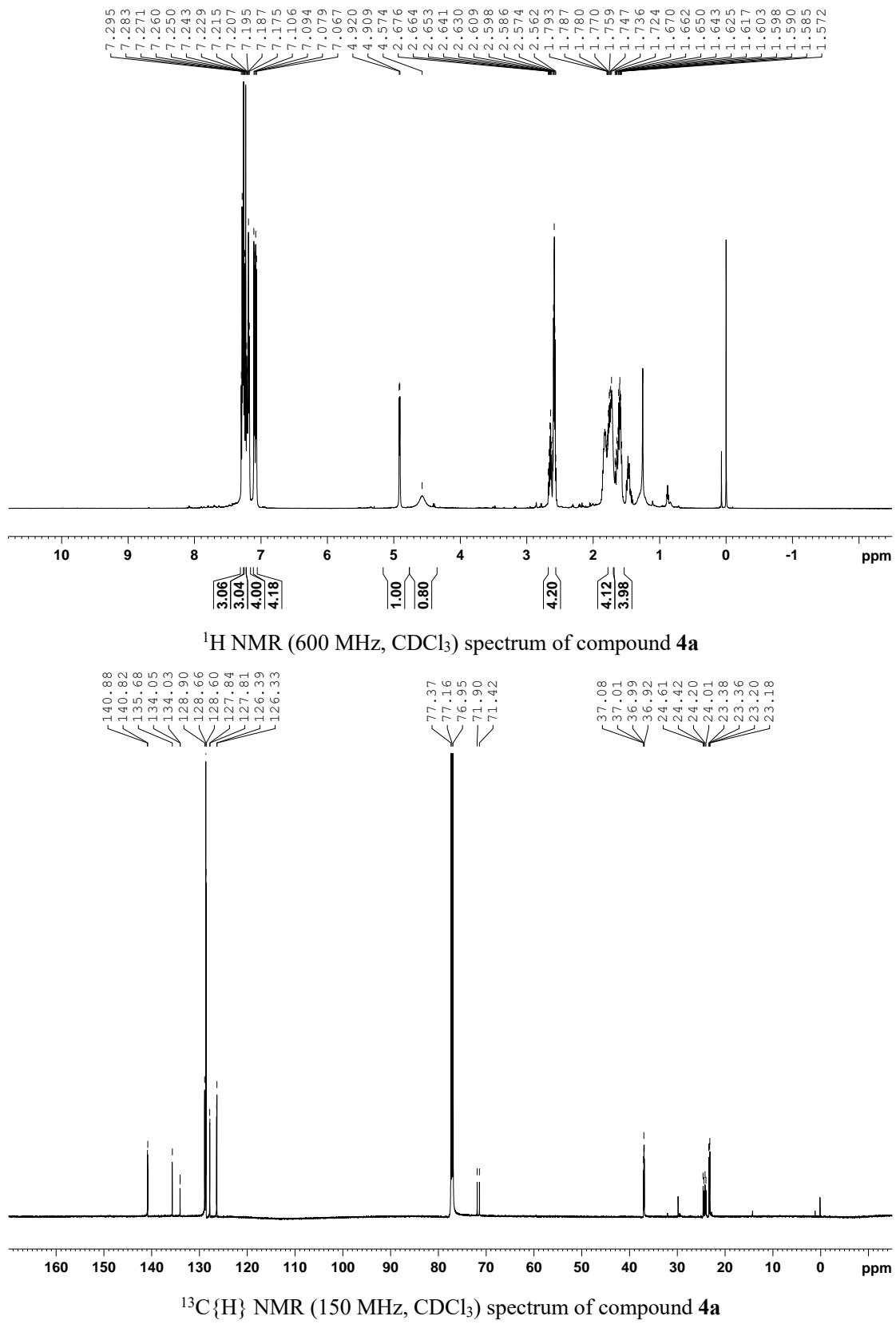
¹H NMR (600 MHz, CDCl₃) spectrum of compound **3-2d**

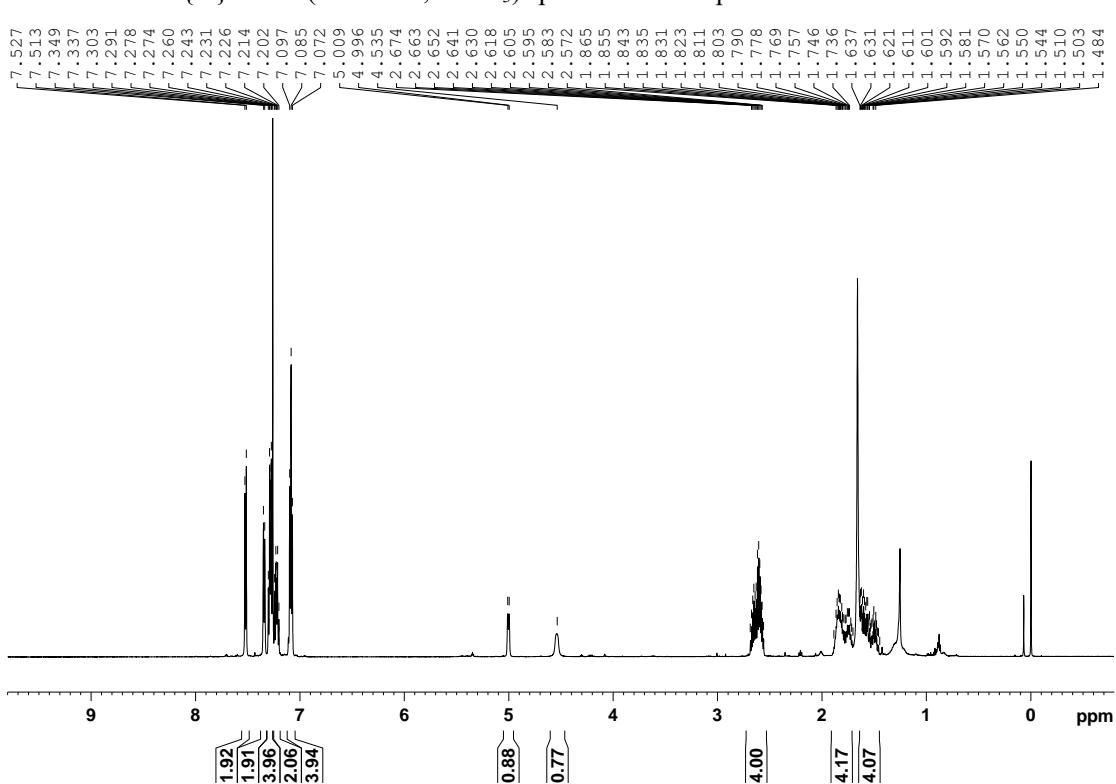
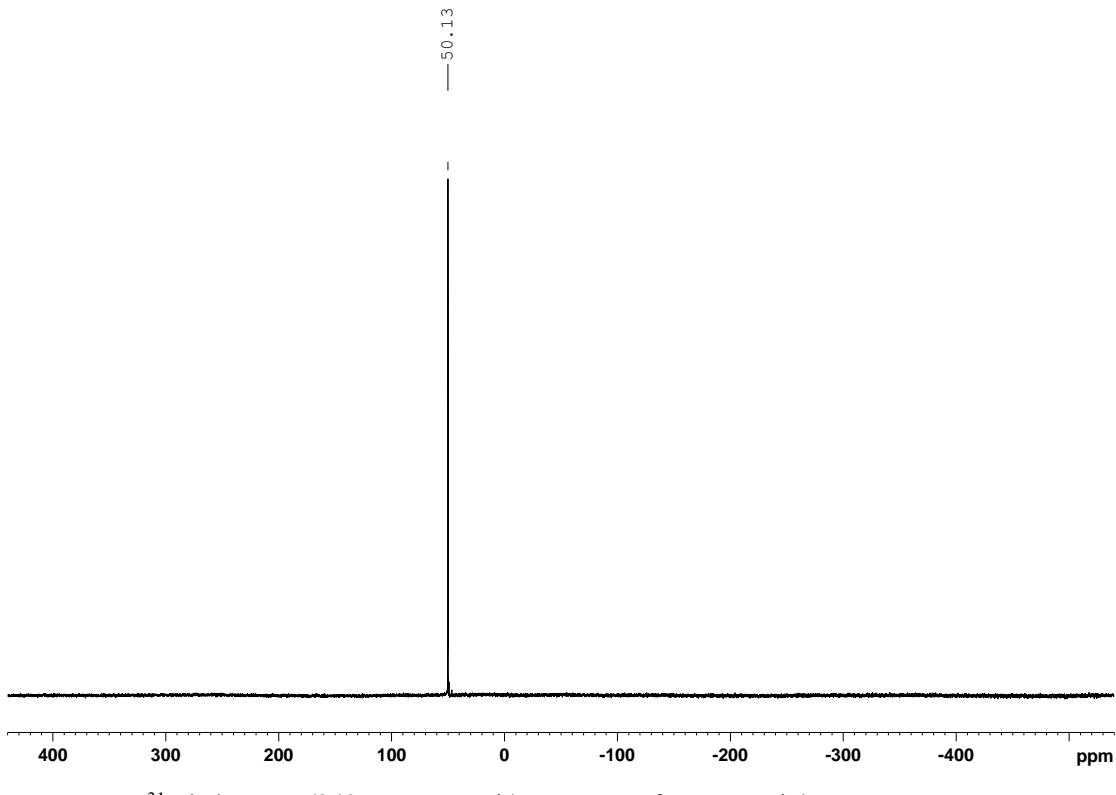
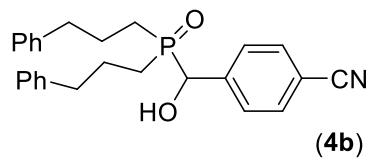


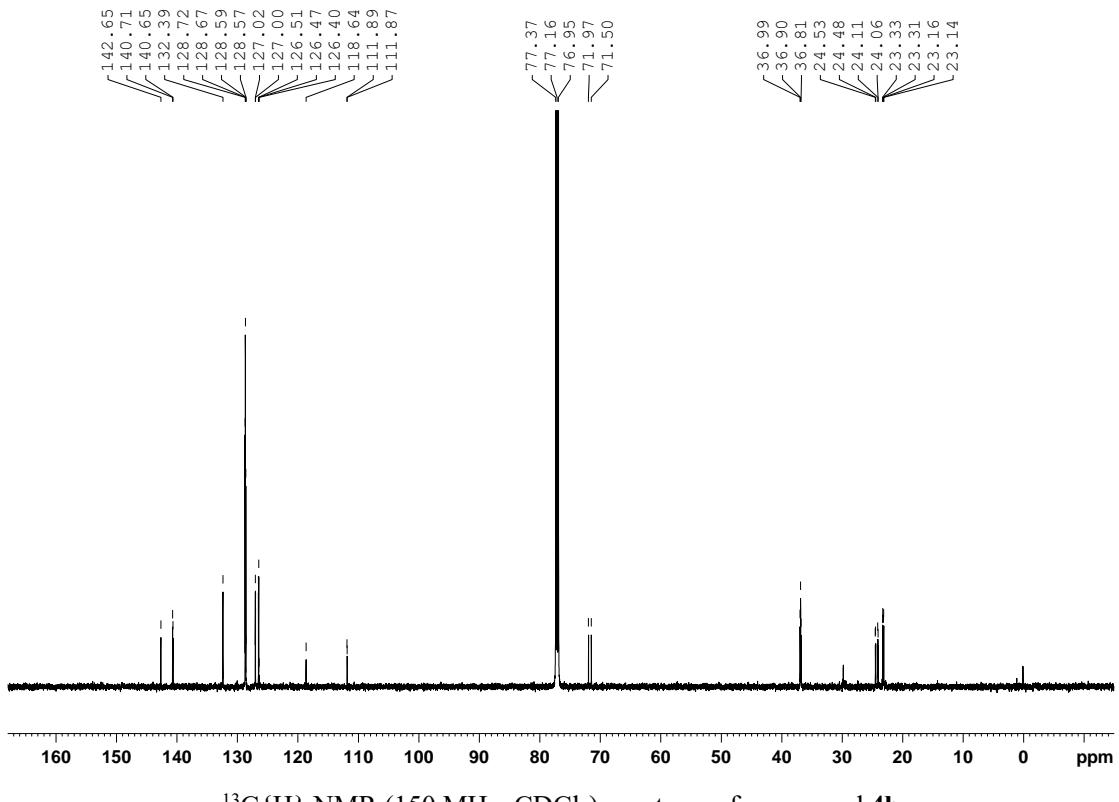
$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **3-2d**



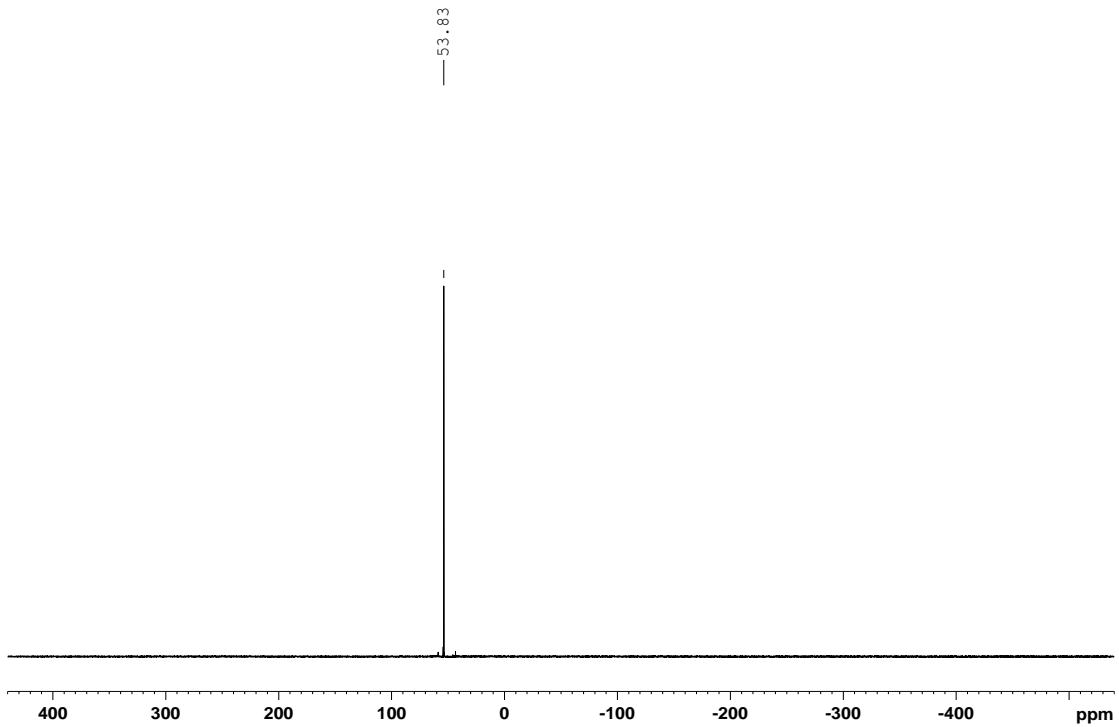
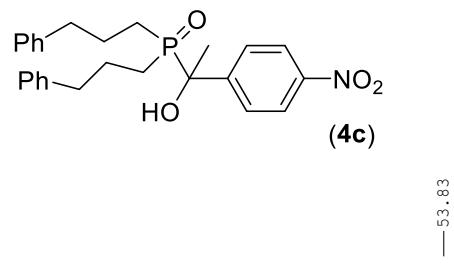
$^{31}\text{P}\{\text{H}\}$ NMR (242 MHz, CDCl_3) spectrum of compound **4a**



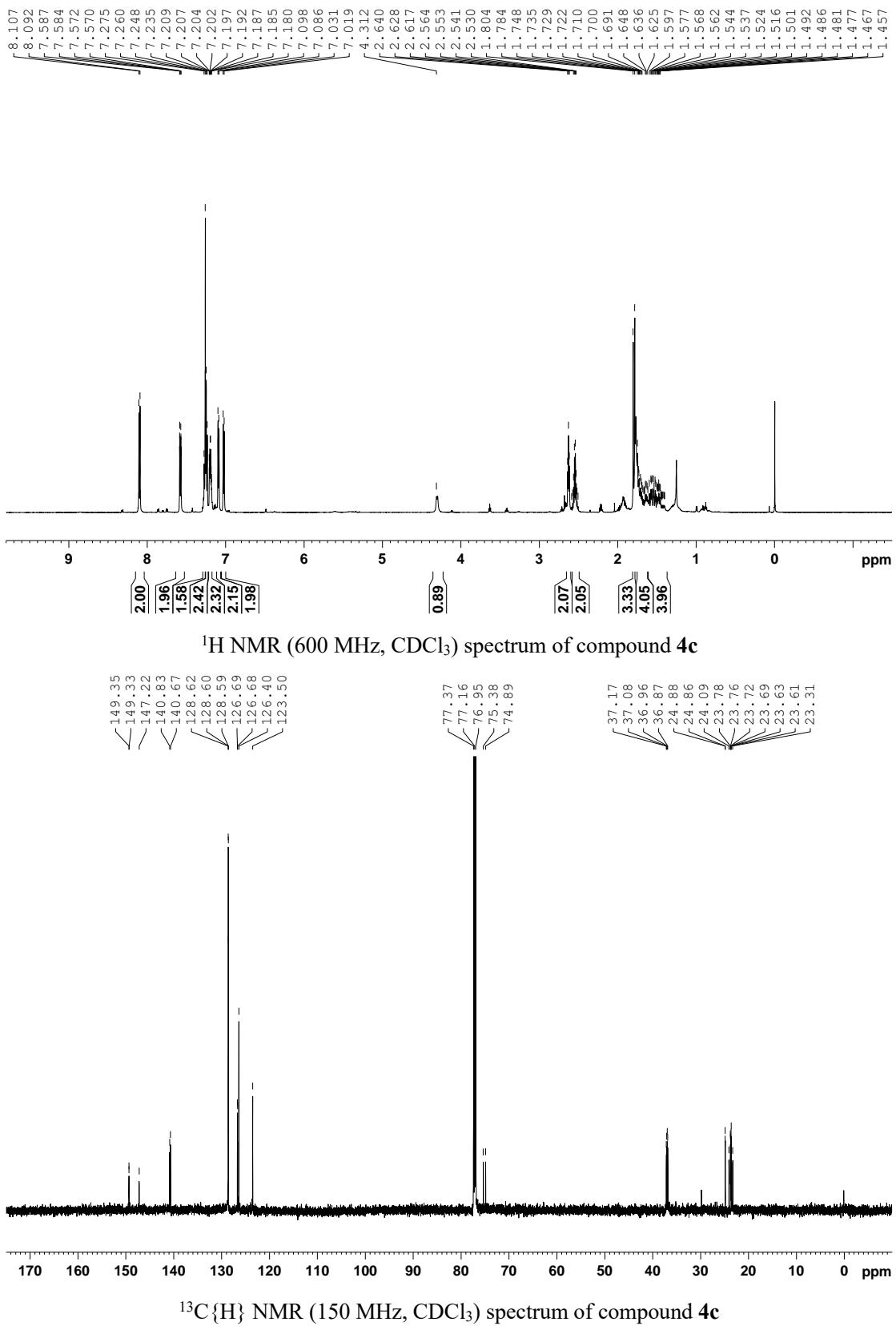


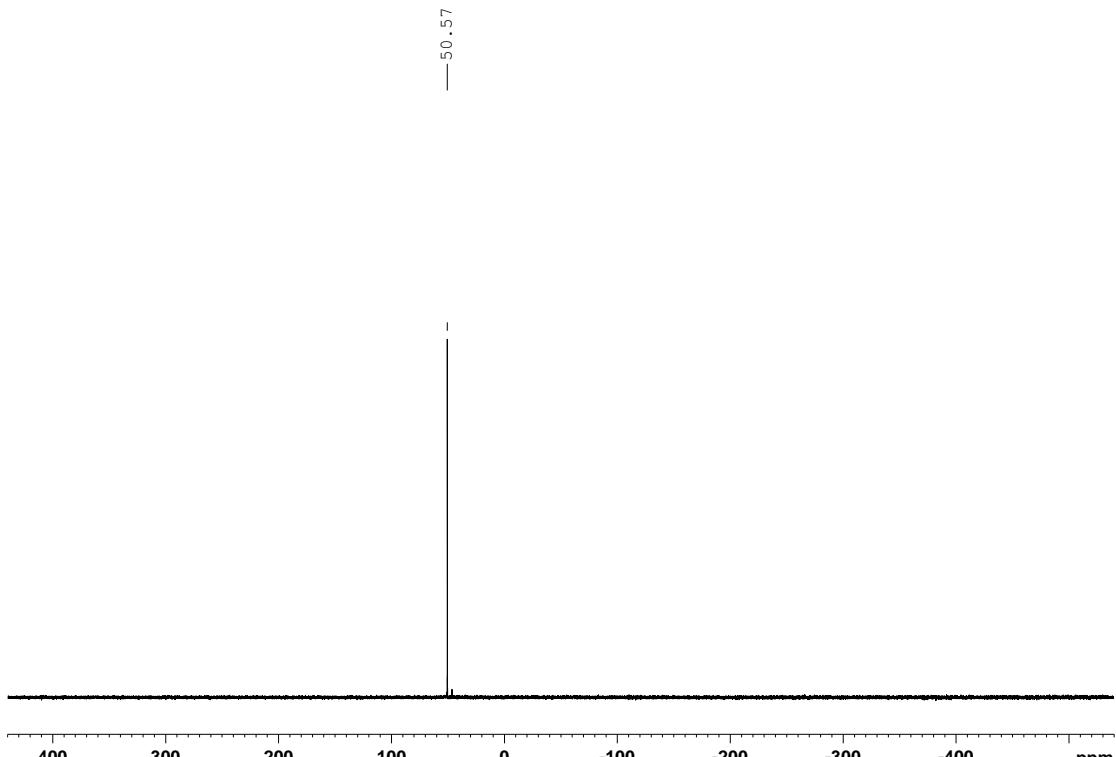
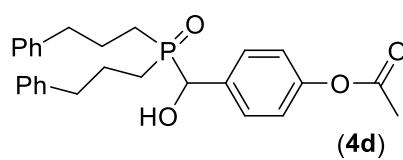


$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl₃) spectrum of compound **4b**

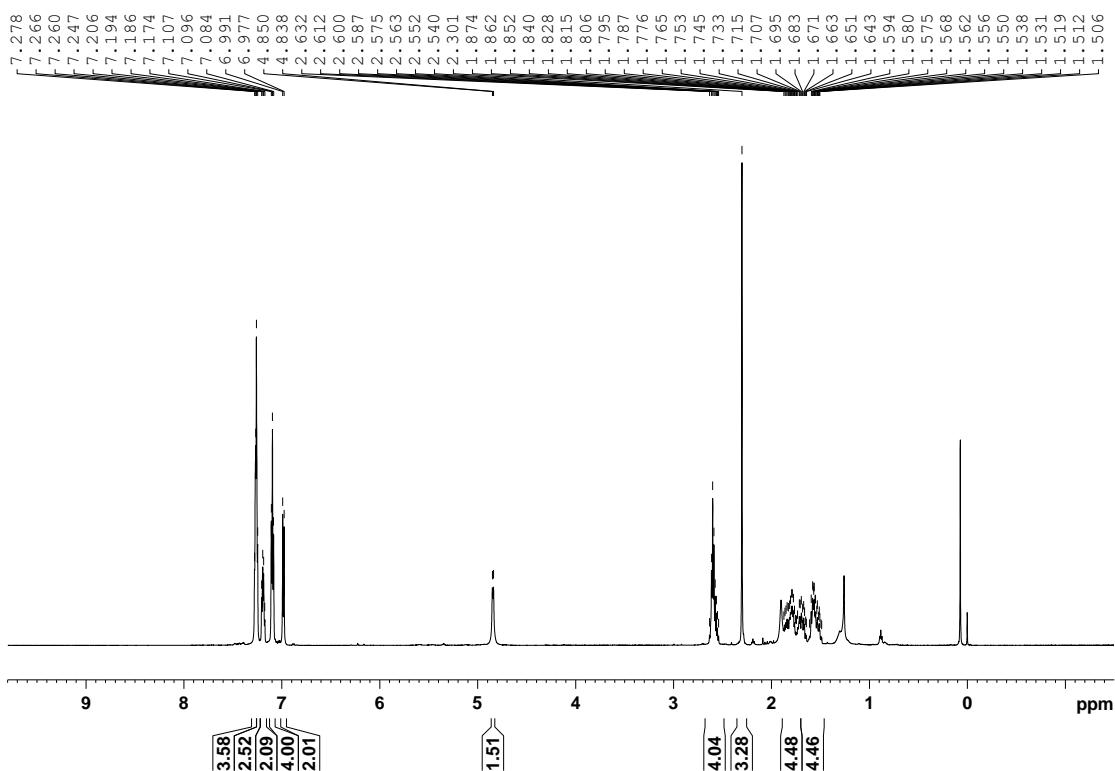


$^{31}\text{P}\{\text{H}\}$ NMR (242 MHz, CDCl₃) spectrum of compound **4c**

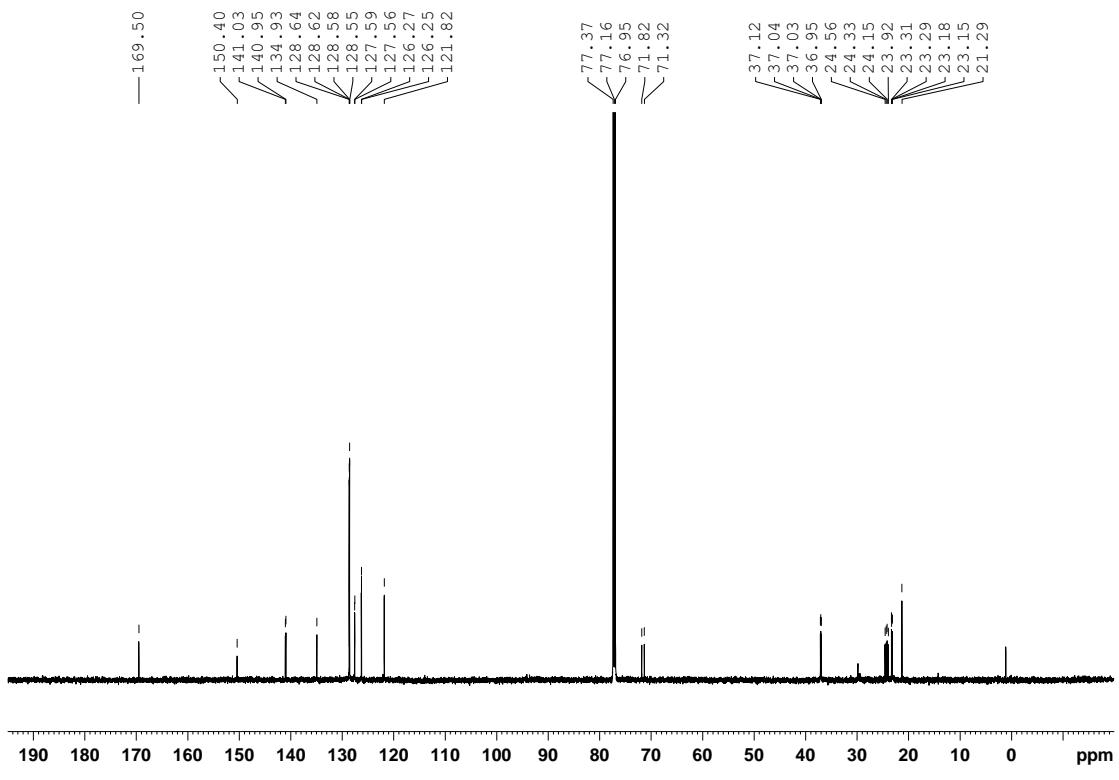




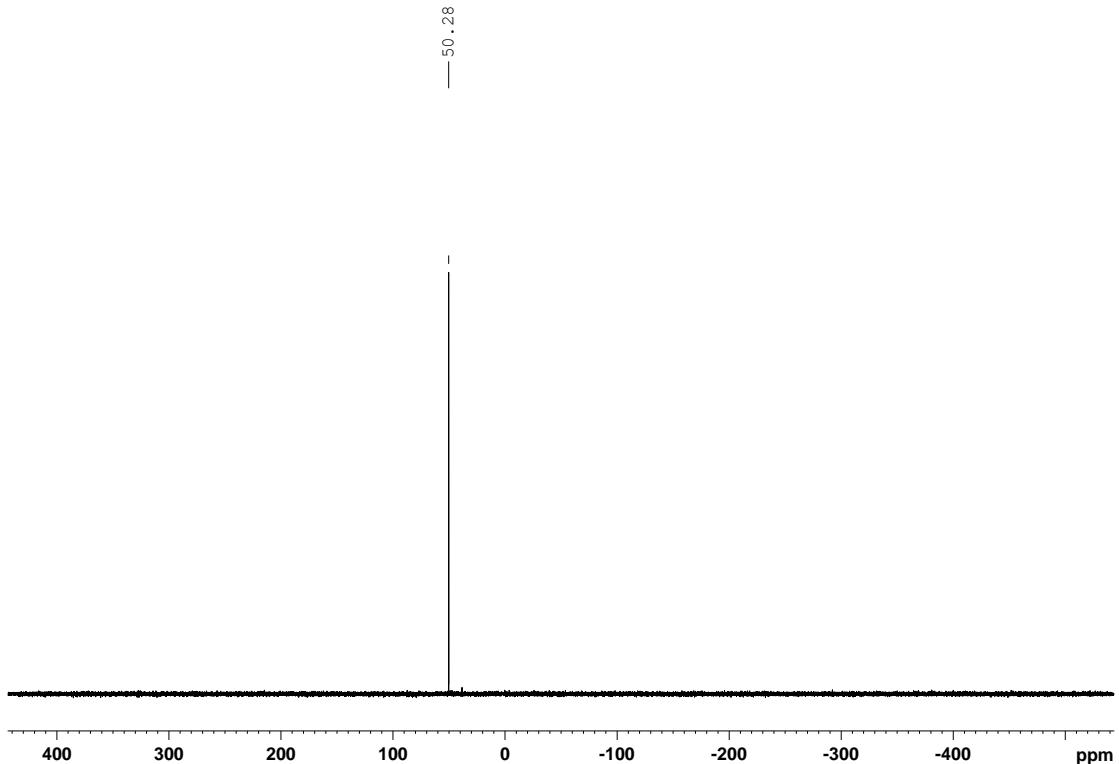
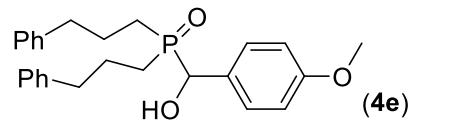
$^{31}\text{P}\{\text{H}\}$ NMR (242 MHz, CDCl_3) spectrum of compound **4d**



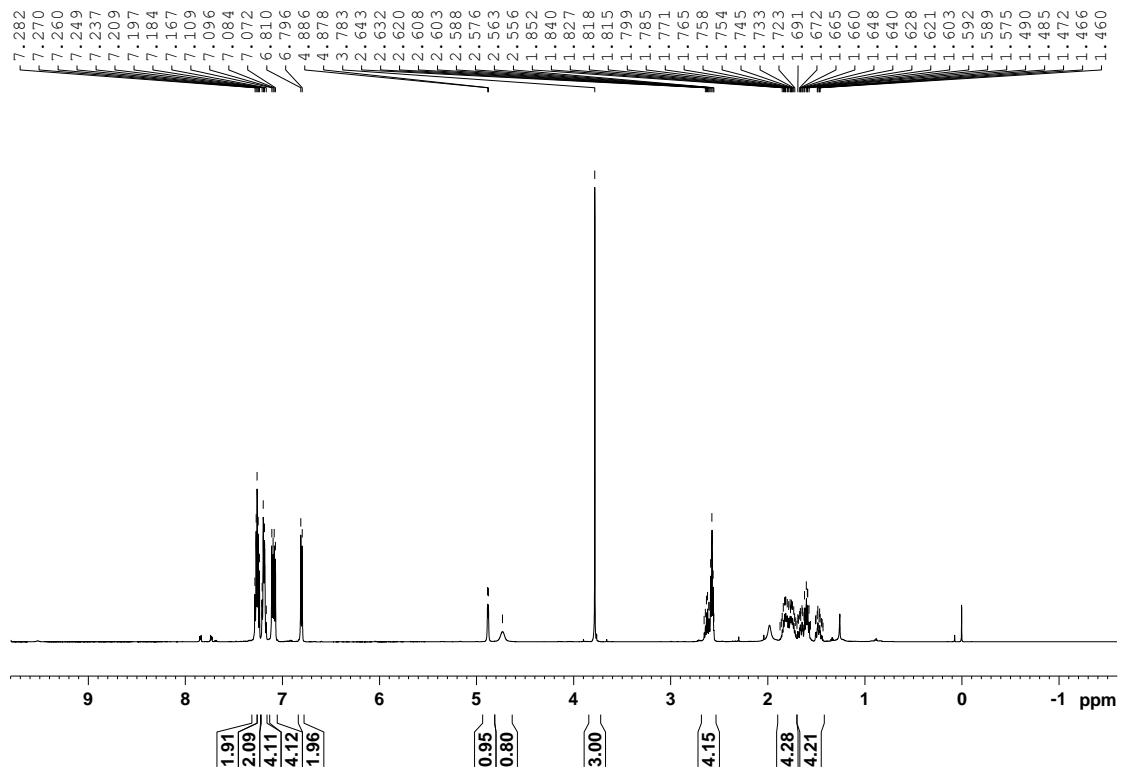
¹H NMR (600 MHz, CDCl₃) spectrum of compound 4d



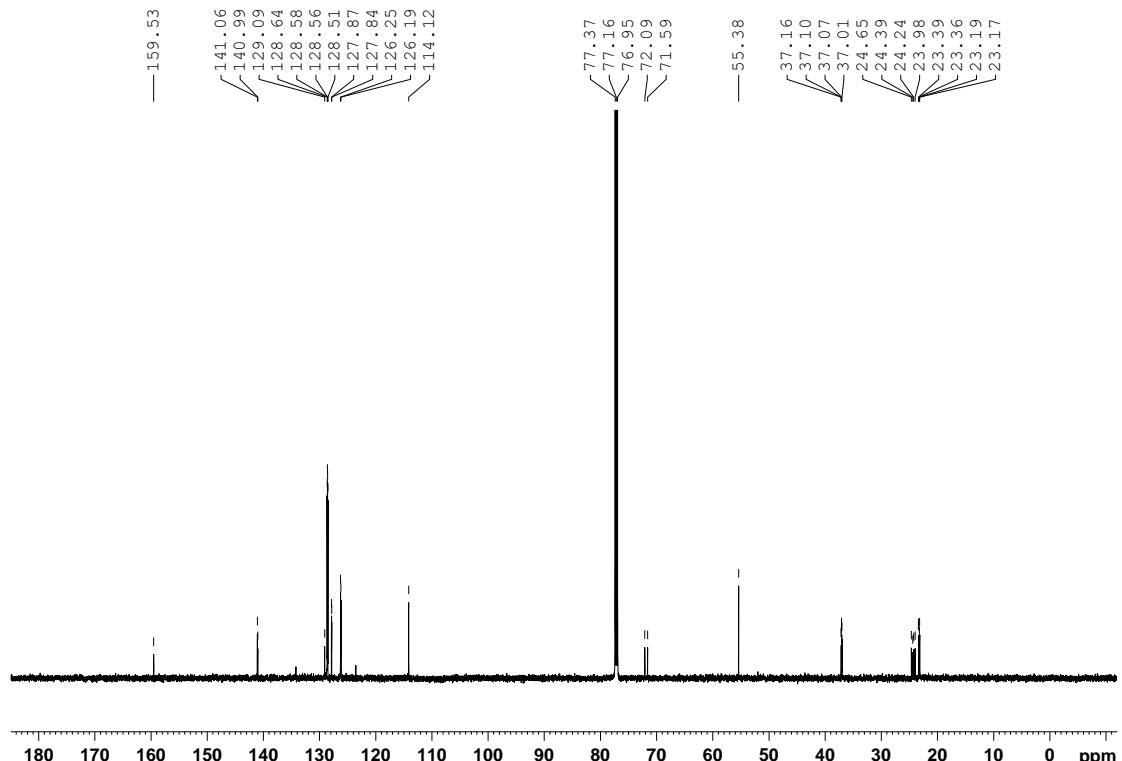
$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **4d**



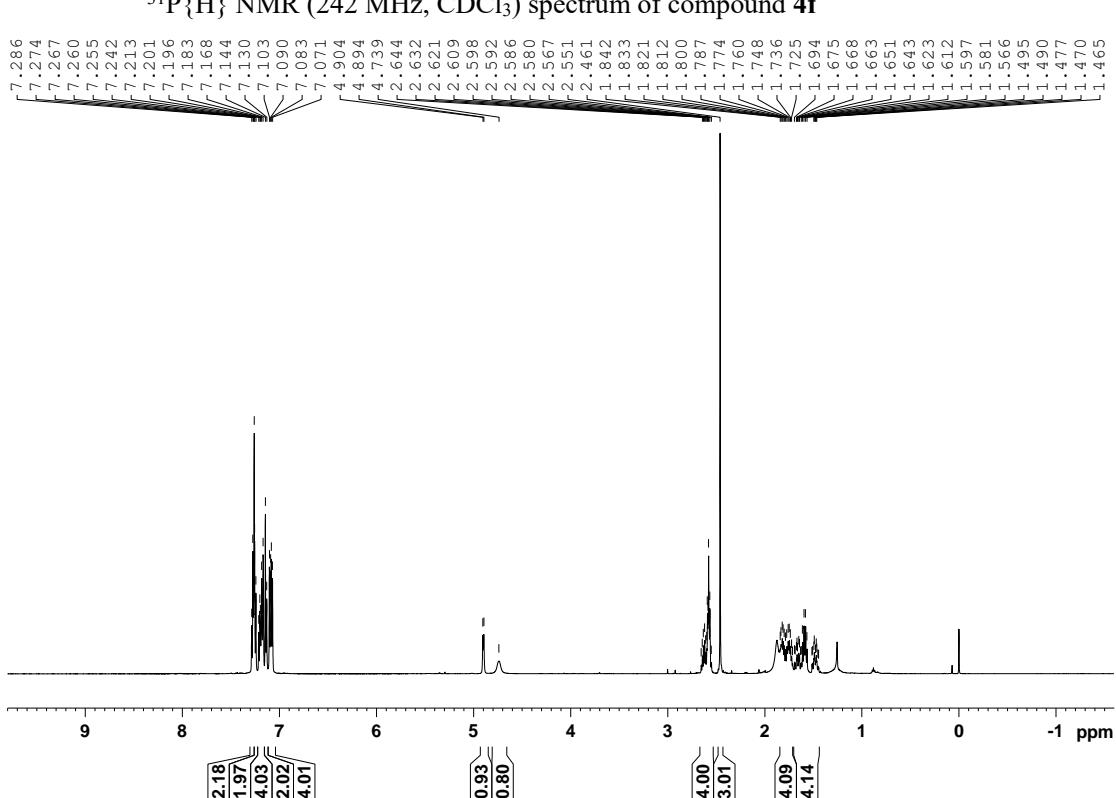
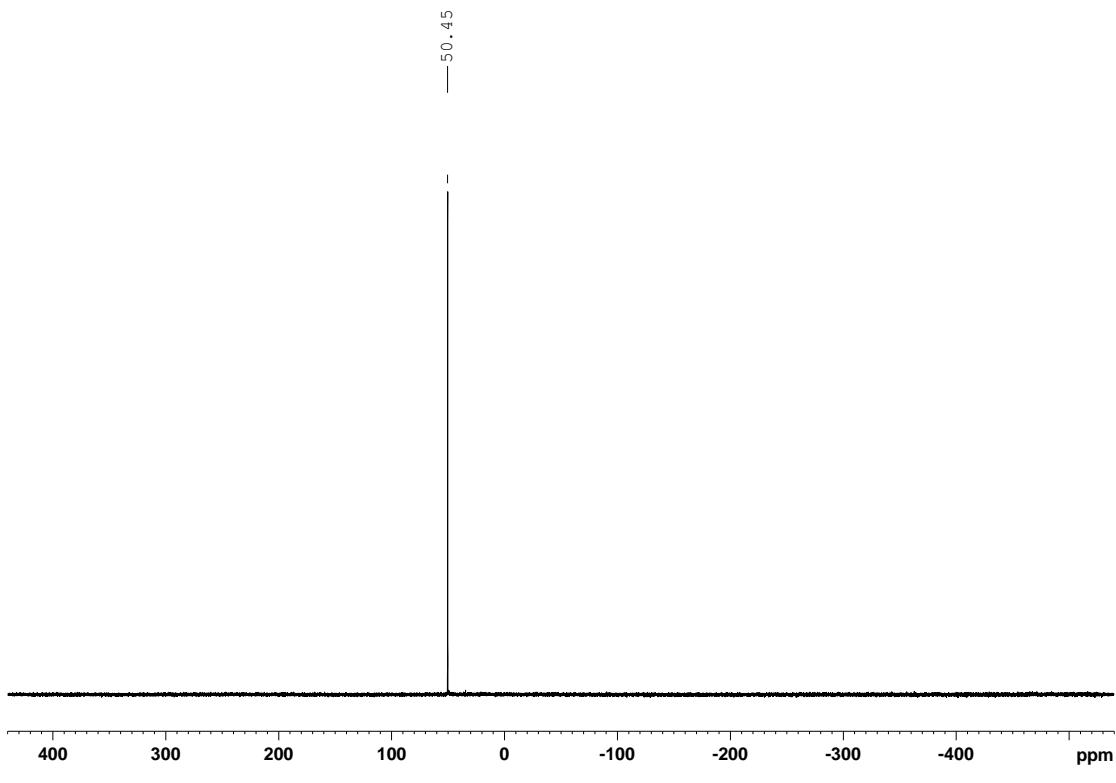
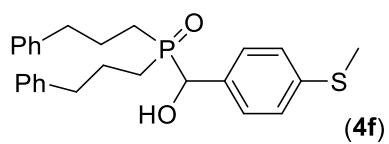
$^{31}\text{P}\{\text{H}\}$ NMR (242 MHz, CDCl_3) spectrum of compound **4e**

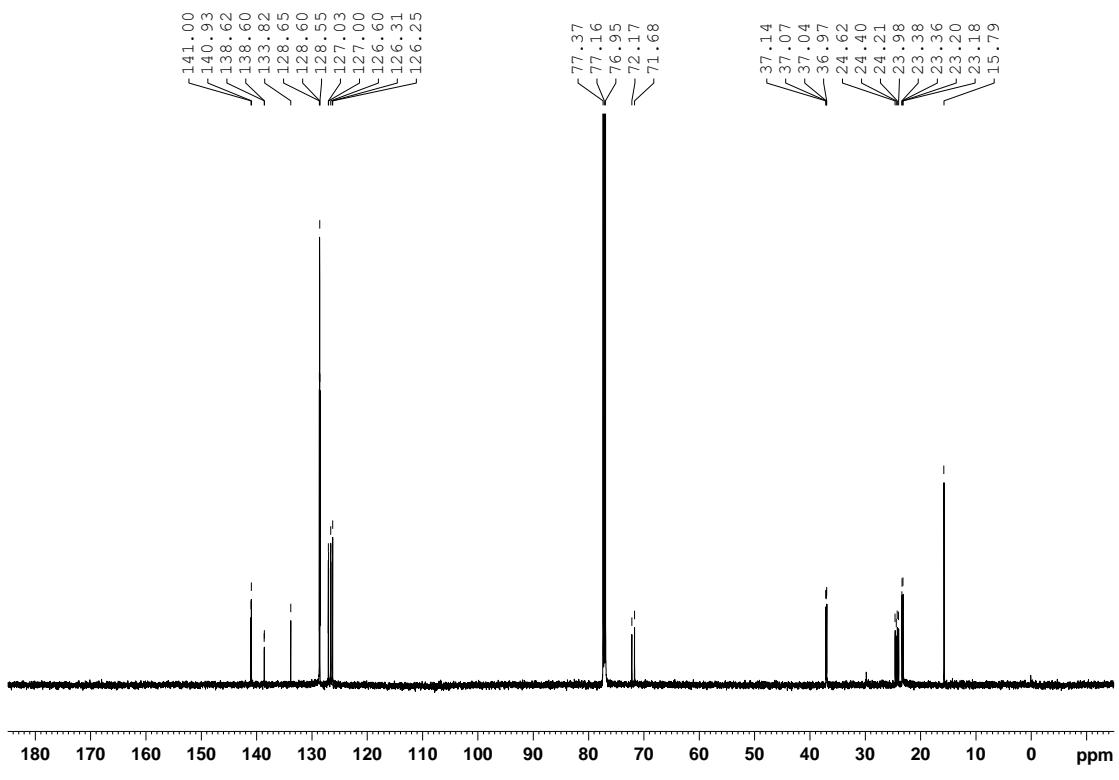


¹H NMR (600 MHz, CDCl₃) spectrum of compound 4e

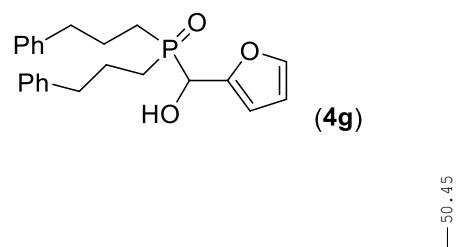


¹³C{H} NMR (150 MHz, CDCl₃) spectrum of compound **4e**

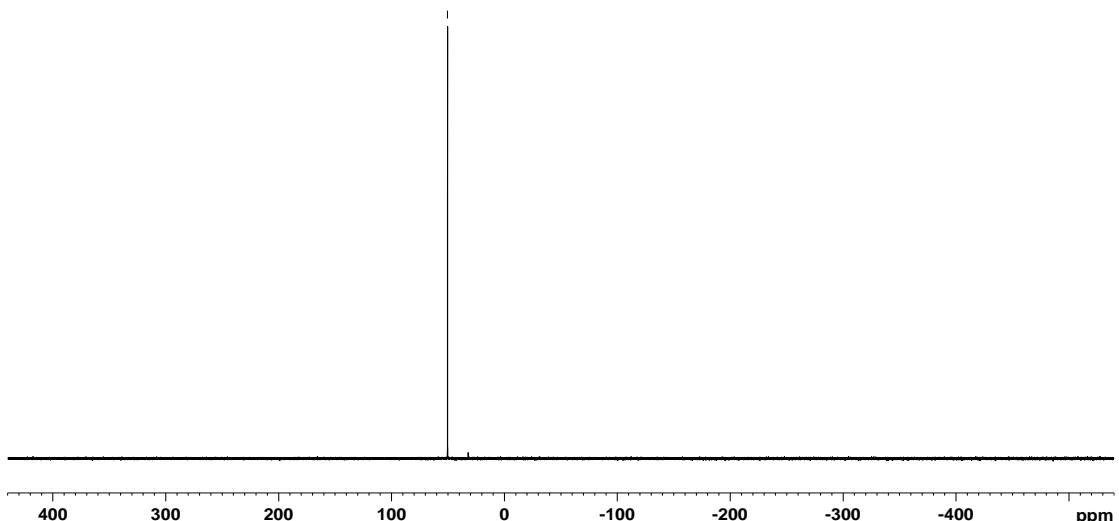




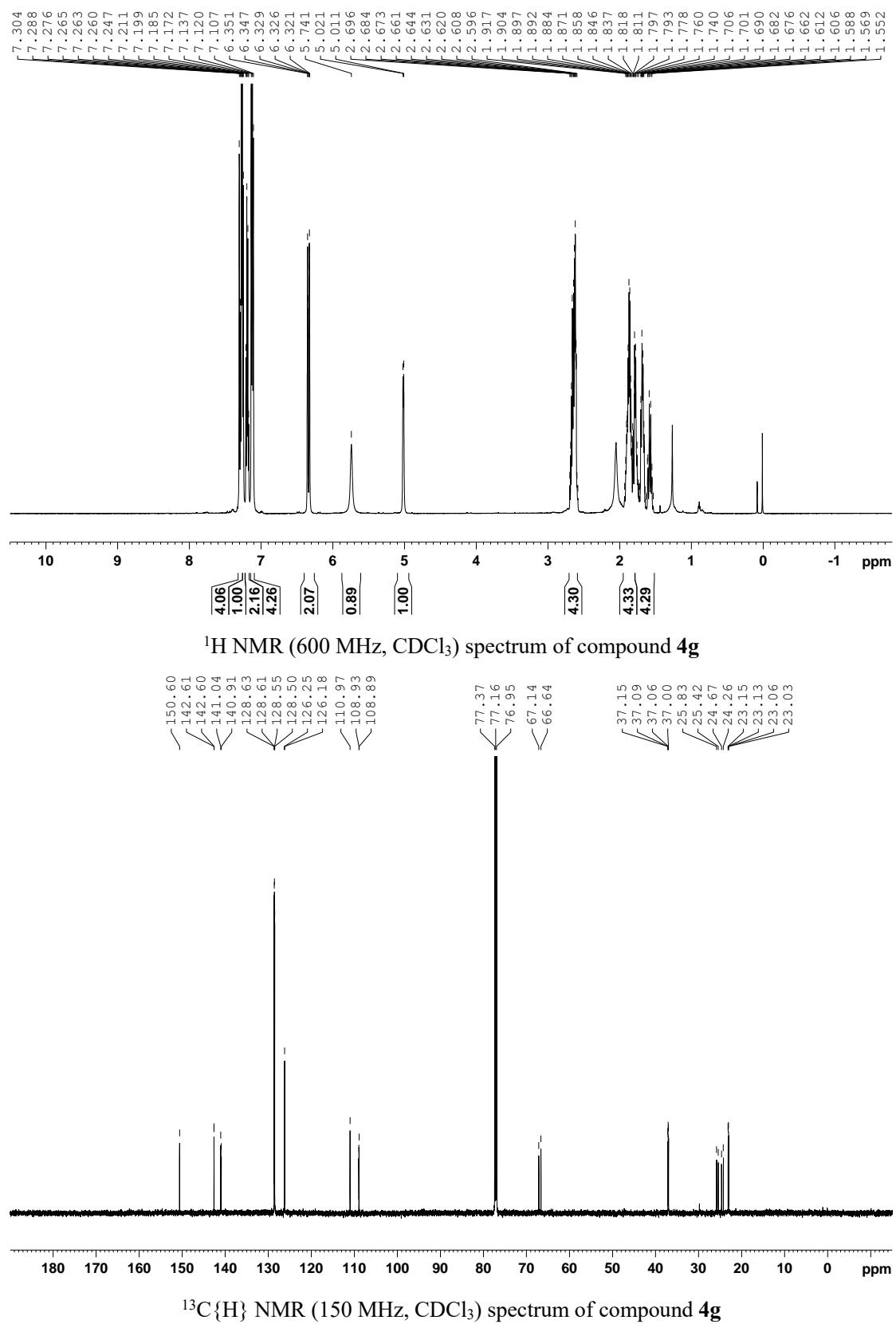
$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **4f**

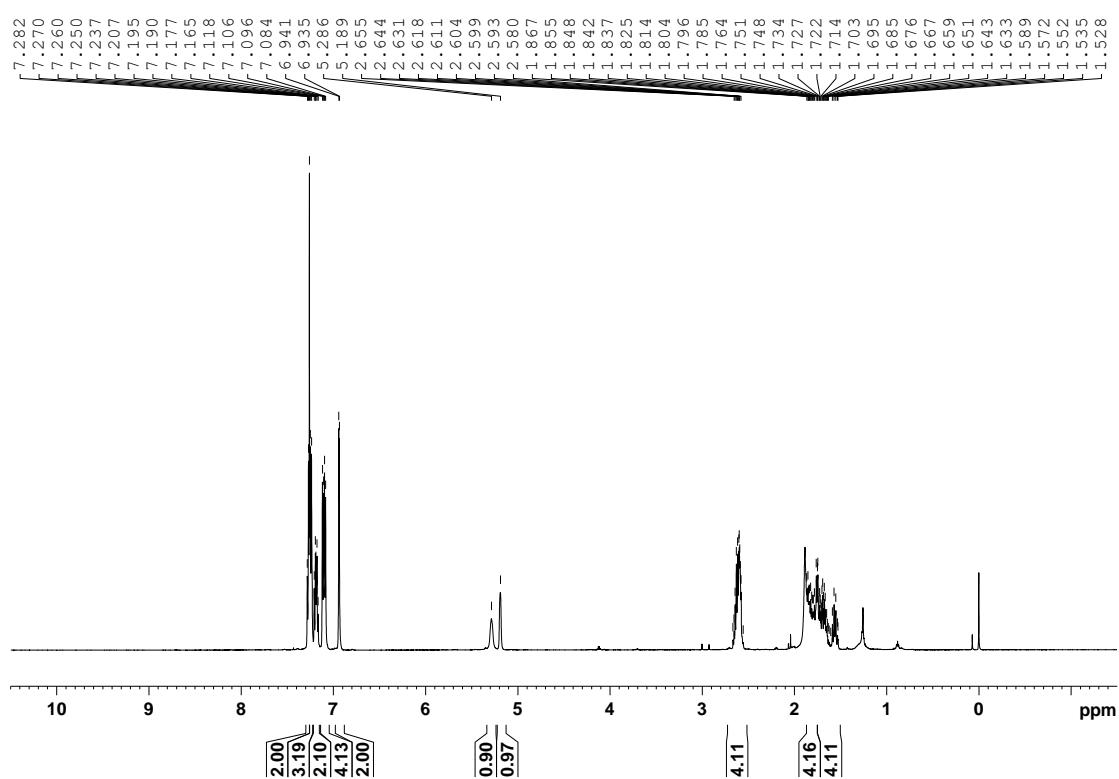
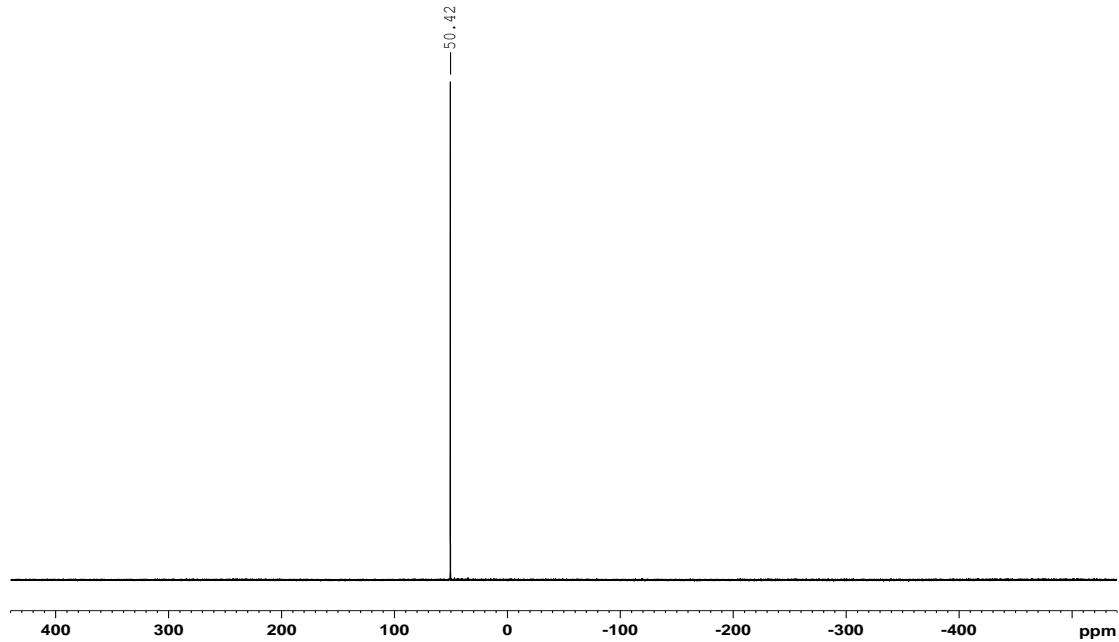
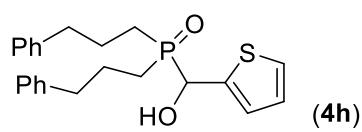


— 50.45 —

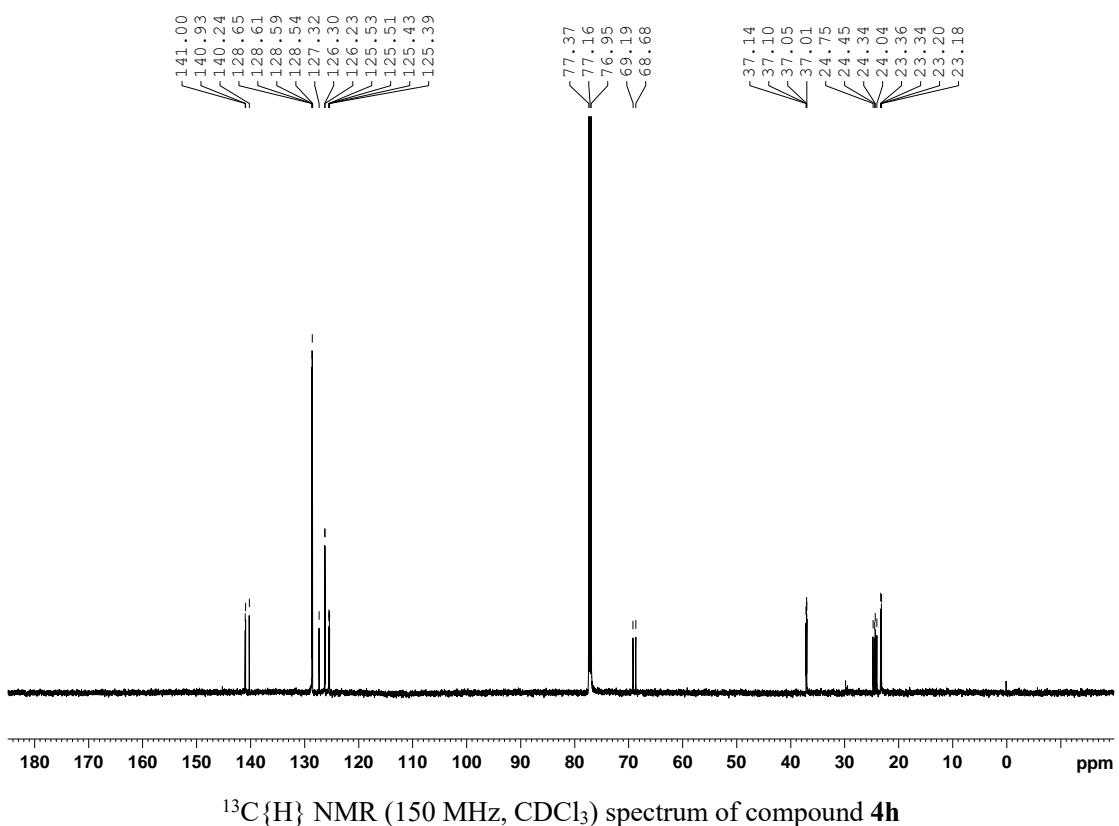


$^{31}\text{P}\{\text{H}\}$ NMR (242 MHz, CDCl_3) spectrum of compound **4g**

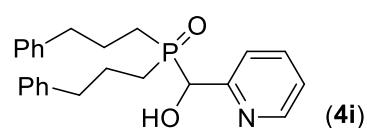




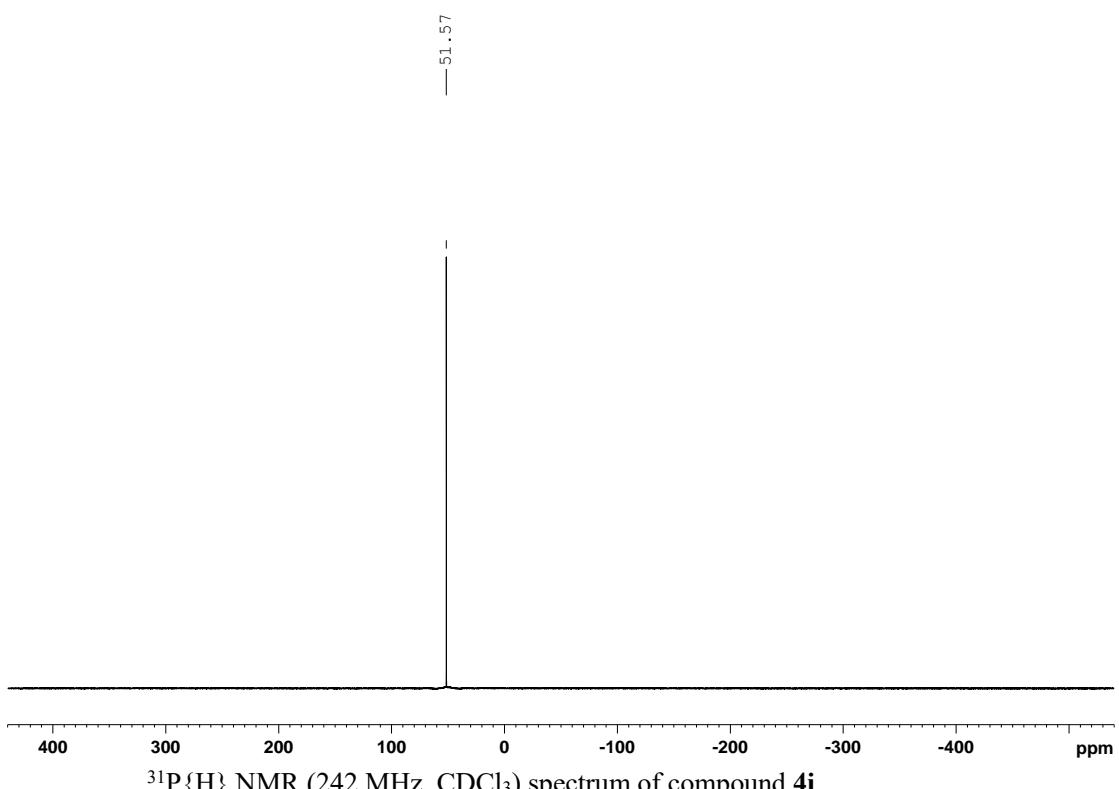
^1H NMR (600 MHz, CDCl_3) spectrum of compound **4h**



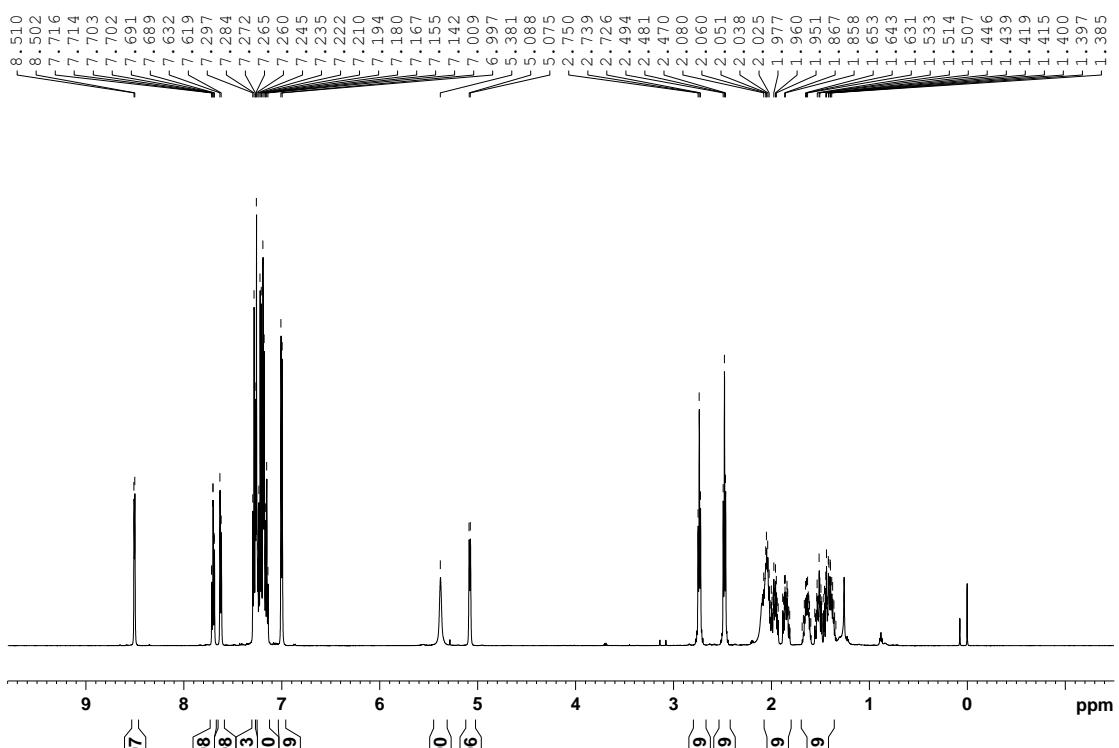
$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **4h**



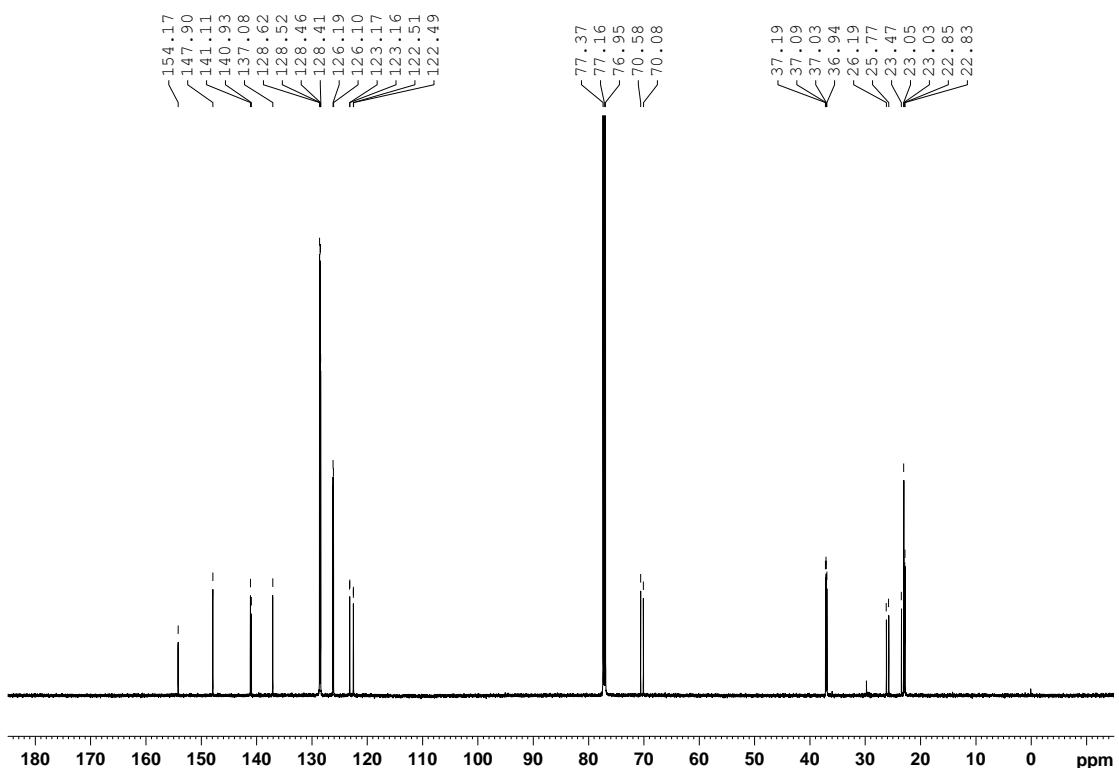
— 51.57



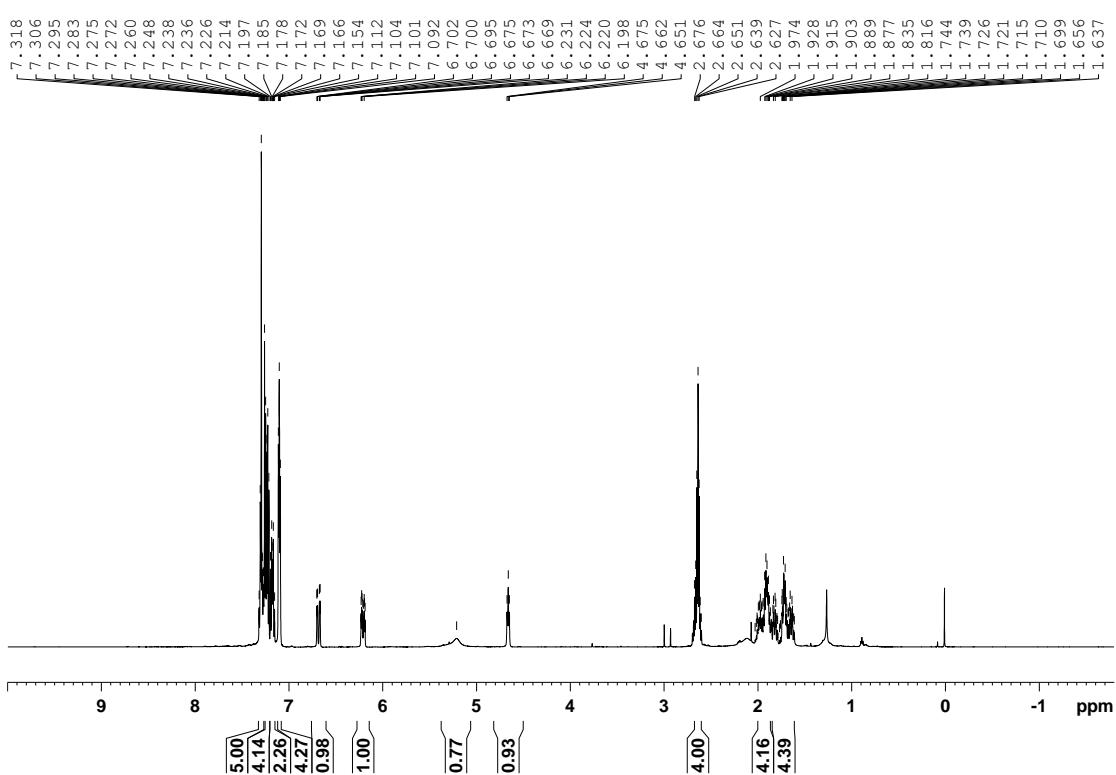
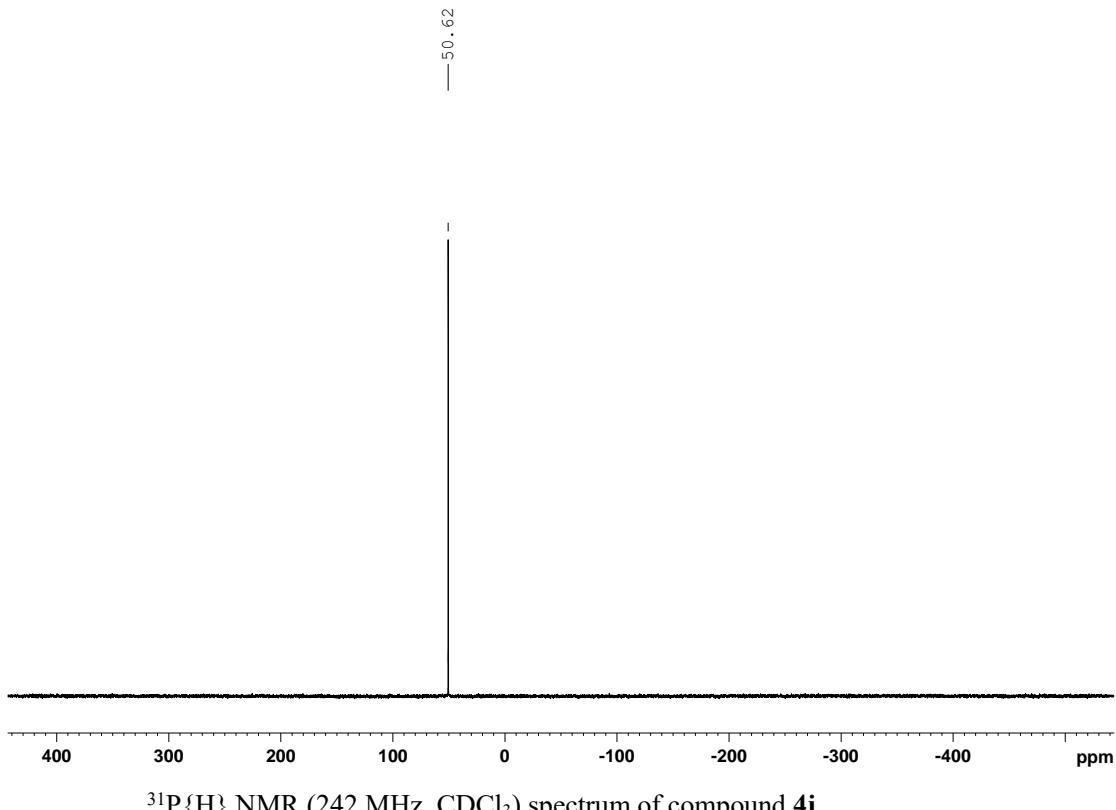
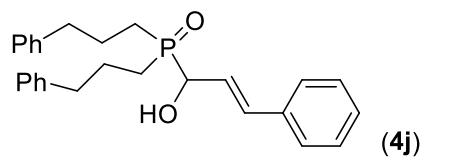
$^{31}\text{P}\{\text{H}\}$ NMR (242 MHz, CDCl_3) spectrum of compound **4i**

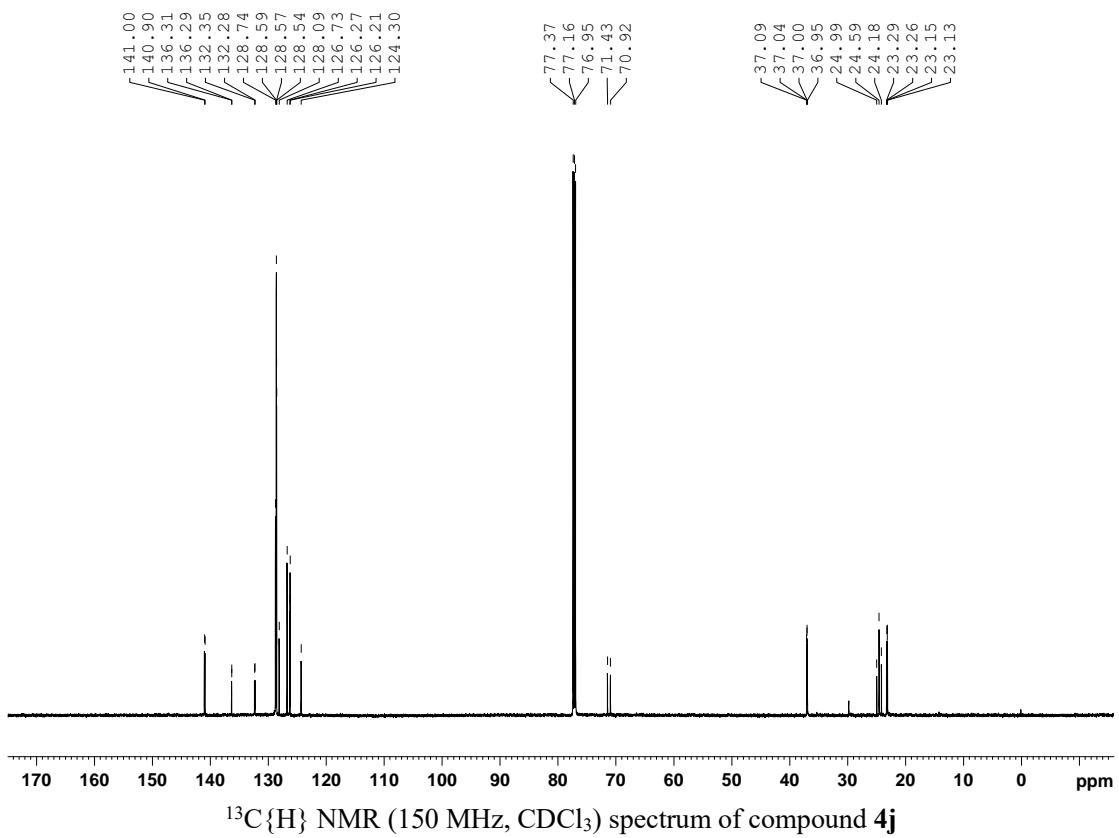


^1H NMR (600 MHz, CDCl_3) spectrum of compound **4i**

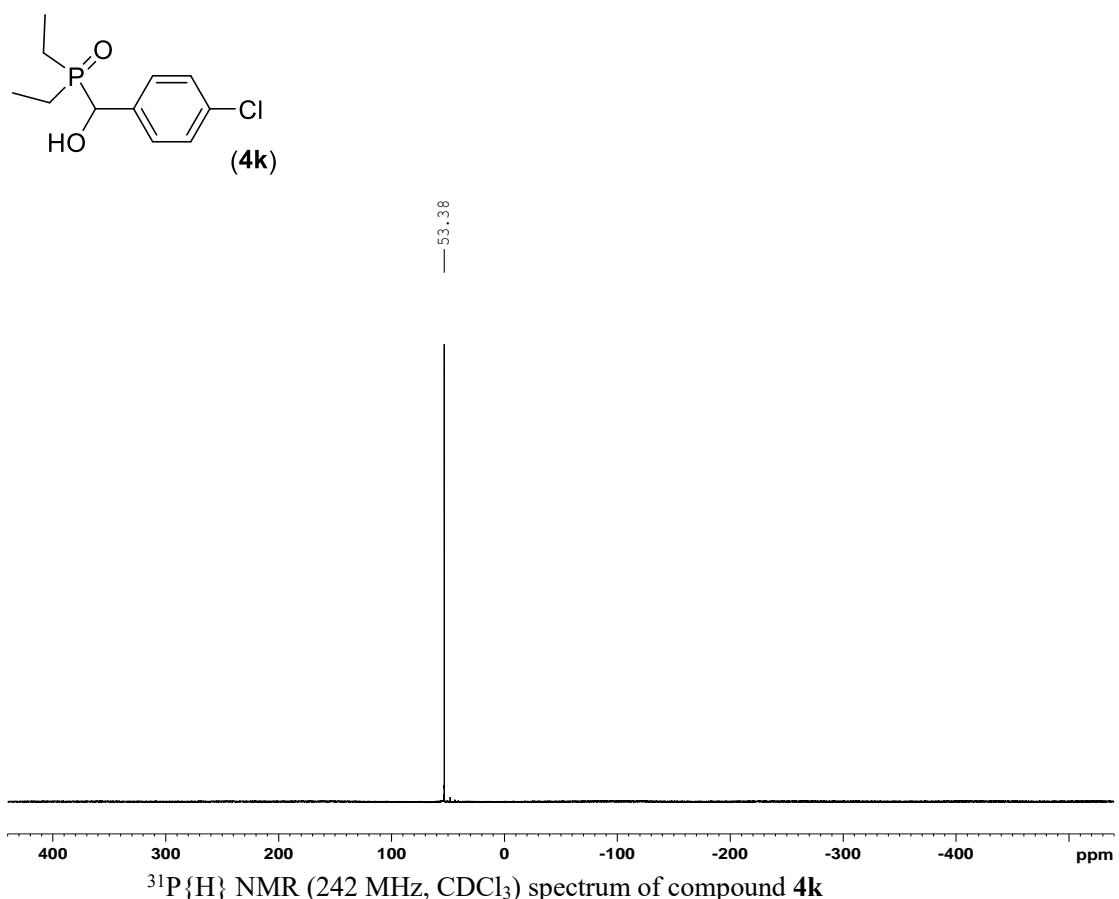


$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **4i**

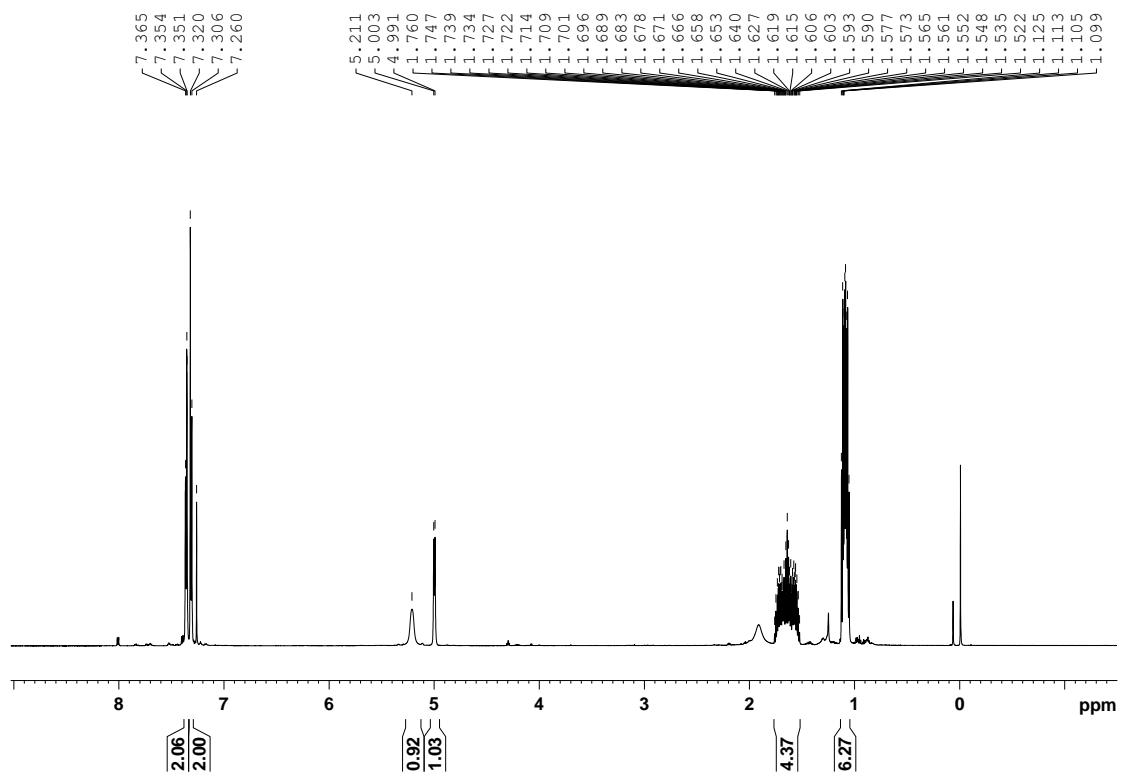




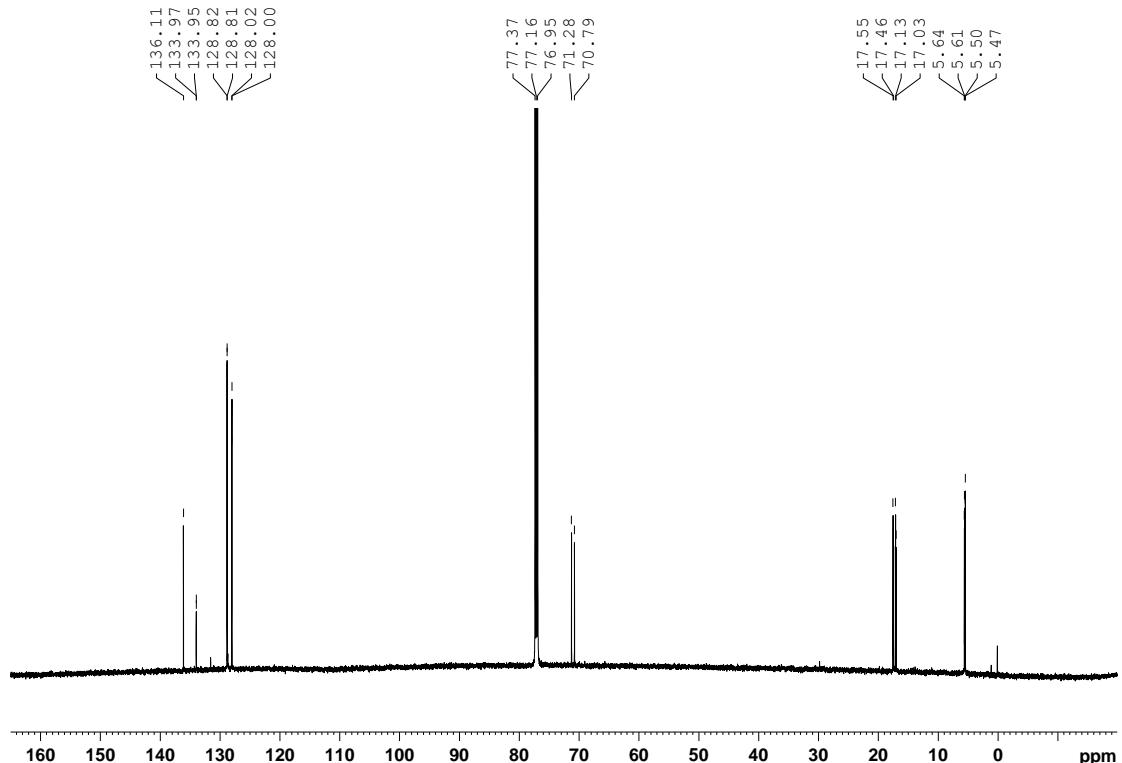
$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound **4j**



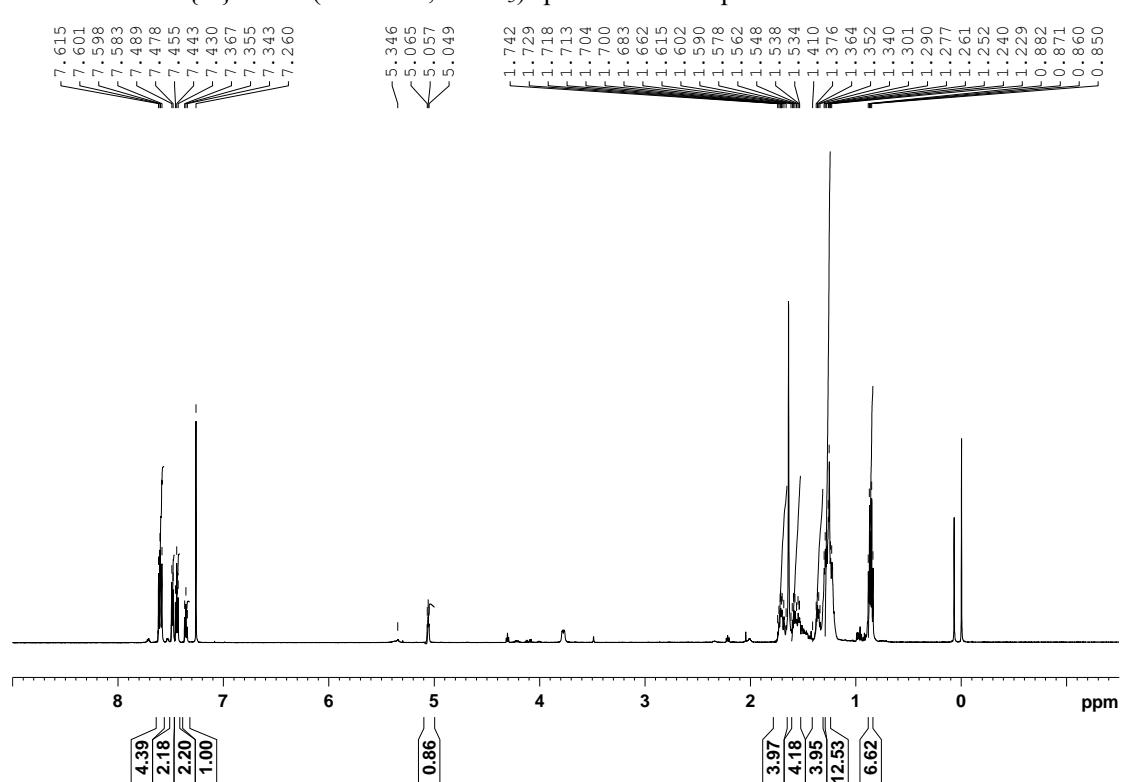
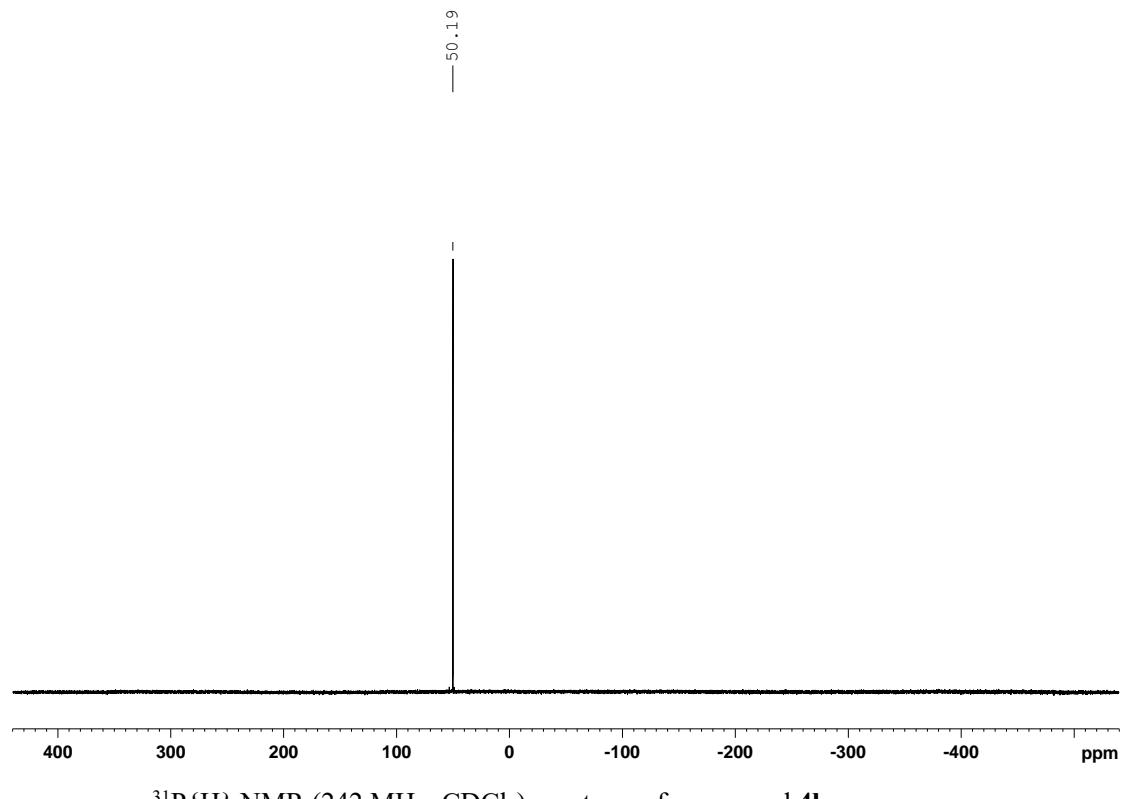
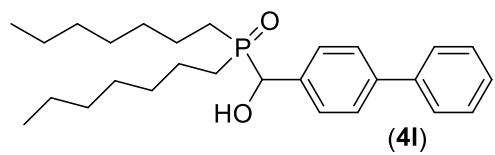
$^{31}\text{P}\{\text{H}\}$ NMR (242 MHz, CDCl_3) spectrum of compound **4k**

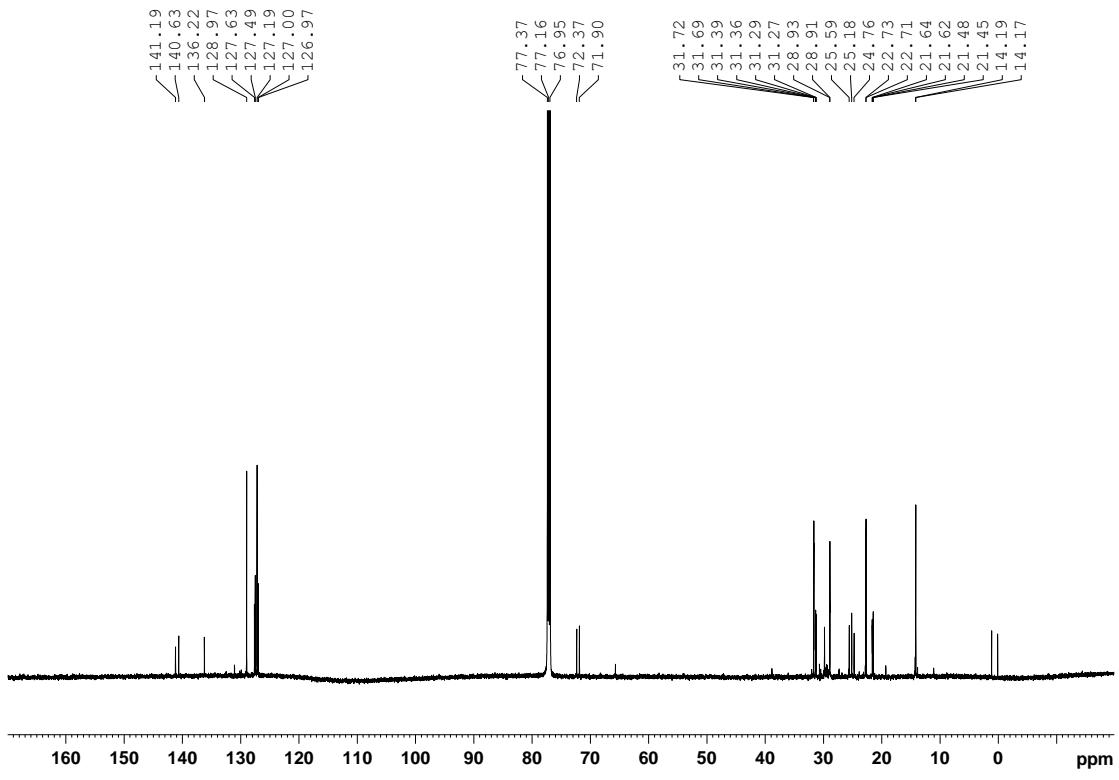


¹H NMR (600 MHz, CDCl₃) spectrum of compound 4k



¹³C{H} NMR (150 MHz, CDCl₃) spectrum of compound **4k**





$^{13}\text{C}\{\text{H}\}$ NMR (150 MHz, CDCl_3) spectrum of compound 4l