

Supporting Information

Excited-State Cobaloxime Catalysis Enabled Scalable Oxidant-Free Dehydrogenative C-H Phosphinoylation of Undirected Heterocycles

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General Information

Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were packed with 200-300 mesh silica gel in petroleum (boiling point is between 60-90 °C). Gradient flash chromatography was conducted eluting with a continuous gradient from petroleum to the indicated solvent, and they are listed as volume/volume ratios. NMR spectra were recorded on a Varian Mercury spectrometer on a Bruker spectrometer at 400 MHz (^1H NMR), 101 MHz (^{13}C NMR), 162 MHz (^{31}P NMR), 376 MHz (^{19}F NMR). Tetramethylsilane was used as an internal standard. All ^1H NMR spectra were reported in delta (δ) units, parts per million (ppm) downfield from the internal standard. Coupling constants are reported in Hertz (Hz). High resolution mass spectra (HRMS) were measured with a Agilent 6220 TOF mass spectrometer (ESI-Q-TOF), accurate masses are reported for the molecular ion ($[\text{M}+\text{H}]^+$). Blue LEDs (3 W, $\lambda = 450 \pm 10$ nm, 300mA) were used as the irradiation light.

General Experimental Procedures

1. General procedure for excited-state cobaloxime catalysis enables scalable oxidant-free dehydrogenative C-H phosphonylation of undirected bioactive molecules: in an oven-dried schlenk tube (25 mL) equipped with a stir bar, diphenylphosphine oxide (0.2 mmol) and Co(dmgh)₂pyCl (10 mol%) were added. After a process for removing oxygen rigorously and fill the schlenk tube with N₂, anhydrous CH₂Cl₂ (1.0 mL), benzofuran (0.4 mmol) and pyridine (0.2 mmol) was injected in the tube through a syringe. Then, the reaction mixture was irradiated with 3 W blue LEDs lights (3 W, $\lambda = 450 \pm 10$ nm) and stirred at room temperature for 24 h. After completion of the reaction, as indicated by TLC, the solvent was removed with a rotary evaporator. And the mixture was purified by column chromatography on silica gel using petroleum ether and ethyl acetate (PE/EA = 5/1 -1/2) as eluent to afford pure product.

2. Procedure for gram scale synthesis: in an oven-dried flask (100 mL) equipped with a stir bar, diphenylphosphine oxide (3 - 5 mmol), heterocycles or arene (6 – 10 mmol, if liquid, injected it through a syringe after protection of N₂) and Co(dmgh)₂pyCl (10 mol%) were added. After a process for removing oxygen rigorously and fill the flask with N₂, anhydrous CH₂Cl₂ (15 – 25 mL) and pyridine (3 – 5 mmol) was injected in the tube through a syringe. Then, the reaction mixture was irradiated with 3W blue LEDs lights and stirred at room temperature for 24 - 48 h. After completion of the reaction, as indicated by TLC, the solvent was removed with a rotary evaporator. And the mixture was purified by column chromatography on silica gel using petroleum ether

and ethyl acetate (PE/EA = 5/1 -1/1) as eluent to afford pure product.

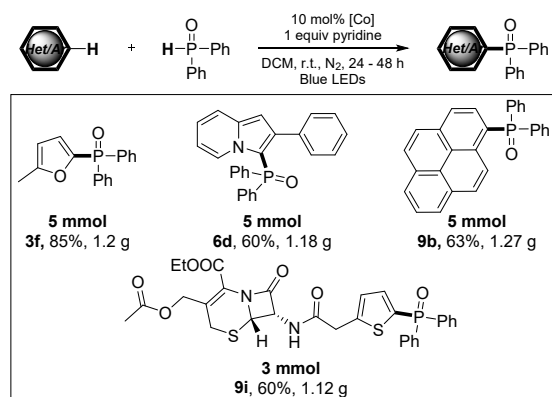


Figure S1. gram scale synthesis of **3f**, **6d**, **9b** and **9i**

3. Procedure for cyclic voltammetry (CV): cyclic voltammetry was performed in a three-electrode cell connected to a schlenk line under air at room temperature. The working electrode was a glassy-carbon disk electrode (diameter, 1 mm), the counter electrode a platinum wire. The reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution, and separated from reaction by a salt bridge. 5 mL of CH₂Cl₂ and ⁿBuN₄BF₄ was poured into the electrochemical cell in all experiments. The scan rate is 100 mVs⁻¹, ranging from 0 V to 4.0 V.

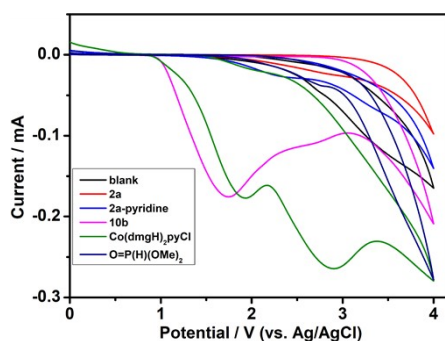


Figure S2. Cyclic voltammetry of **2a**, **2a-pyridine**, **10b**, O=P(H)(OMe)₂, Co(dmgh)₂pyCl in DCM with ⁿBu₄NBF₄ under N₂ atmosphere. A glassy carbon working electrode (diameter (d)= 2 mm), Ag/AgCl reference electrode and a platinum wire counter electrode were used. The scan rate was 100 mV/s.

3. Detection of hydrogen gas by GC-TCD analysis: in an oven-dried Schlenk tube (25 mL) equipped with a stir bar, diphenylphosphine oxide (0.2 mmol) and Co(dmgH)₂pyCl (10 mol%) were added. After a process for removing oxygen rigorously and fill the Schlenk tube with N₂, anhydrous CH₂Cl₂ (1.0 mL), benzofuran (0.4 mmol) and pyridine (0.2 mmol) was injected in the tube through a syringe. Then, the reaction mixture was irradiated with 3 W blue LEDs lights and stirred at room temperature for 24 h. After completion of the reaction, the extracted 1000 μ L gas from the reaction system was analyzed by GC-TCD. According to the spectra (Figure S3), the only peak stands for the generation of hydrogen gas (nitrogen cannot be detected due to the instrument parameter setting).

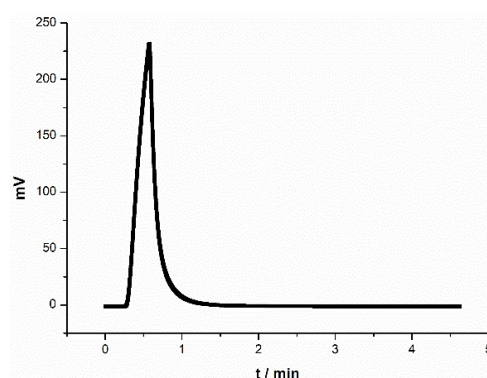
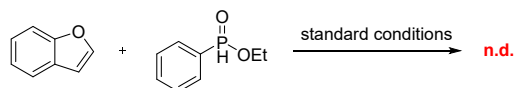


Figure S3. Detection of hydrogen gas by GC-TCD analysis

4. Investigate the reactivity of phosphite reagents, indoles and thiazoles

(a) Failed phosphite reagents



(b) Failed heteroarenes

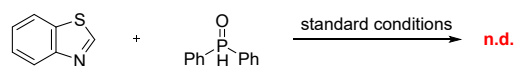
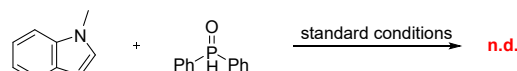


Figure S4. Failed phosphite reagents and heteroarenes

5. Crystal Data and Refinement Results

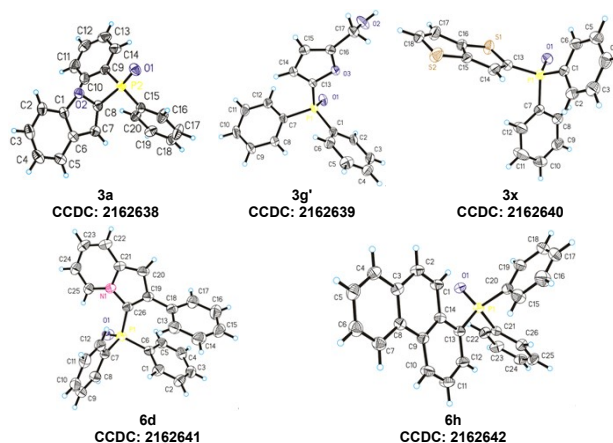


Figure S5. Crystal structure of **3a**, **3g'**, **3x**, **6d** and **6h**

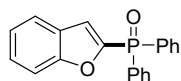
Table 1. Crystal Data and Refinement Results for complexes **3a**, **3g'**, **3x**, **6d** and **6g**

Compound	3a	3g'	3x
Empirical formula	C ₂₀ H ₁₅ O ₂ P	C ₁₇ H ₁₅ O ₃ P	C ₁₈ H ₁₃ OPS ₂
Formula weight	318.29	298.26	340.37
Temperature / K	296(2)	293(2)	293(2)
Crystal system	Triclinic	Monoclinic	Orthorhombic
Space group	<i>P</i> - 1	<i>P</i> 21/ <i>n</i>	<i>P</i> 21 21 21
<i>a</i> / Å	9.4992(2)	10.9372(5)	8.5680(6)
<i>b</i> / Å	10.3406(3)	9.5238(3)	9.8972(5)
<i>c</i> / Å	18.4801(5)	14.1492(6)	19.3273(12)
β / °	95.138(2)	94.251(4)	90.00
<i>V</i> (Å ³)	1632.71(7)	1469.78(10)	2957.4(6)
<i>Z</i>	4	4	4
D _c (g cm ⁻³)	1.295	1.348	1.379
F(000)	664	624	704
θ range / °	2.12 – 26.00	2.27 – 25.00	2.11 – 24.99
Reflns. Collected	19106	6798	4801
Independent reflns.	6417	2589	2715
Goodness-of-fit	1.074	1.077	1.049
<i>R</i> ₁ ^a (<i>I</i> > 2σ (<i>I</i>))	0.0410	0.0374	0.0367

wR_2^b ($I > 2\sigma(I)$)	0.1219	0.0999	0.0900
6d (the crystal cell with			
Compound	water molecular)	6h	
Empirical formula	C ₂₆ H ₂₀ NOP·H ₂ O	C ₂₆ H ₁₉ OP	
Formula weight	411.42	378.38	
Temperature / K	293(2)	293(2)	
Crystal system	Monoclinic	Monoclinic	
Space group	<i>P</i> 21/ <i>n</i>	P 21/ <i>c</i>	
<i>a</i> / Å	9.9038(3)	12.6908(4)	
<i>b</i> / Å	14.8253(5)	9.4451(3)	
<i>c</i> / Å	14.9817(5)	16.5679(6)	
β / °	103.847(3)	100.759(4)	
<i>V</i> (Å ³)	2135.79	1951.02(11)	
<i>Z</i>	4	4	
D _c (g cm ⁻³)	1.279	1.288	
F(000)	864	792	
θ range / °	1.96 – 24.99	2.49 – 25.00	
Reflns. Collected	13743	9222	
Independent reflns.	3761	3419	
Goodness-of-fit	1.023	1.095	
R_1^a ($I > 2\sigma(I)$)	0.0415	0.0472	
wR_2^b ($I > 2\sigma(I)$)	0.1244	0.1343	

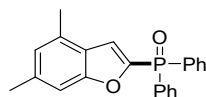
^a $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. ^b $wR_2 = |\Sigma w(|F_o|^2 - |F_c|^2)|/\Sigma[w(F_o)^2]^{1/2}$, where $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$. $P = (F_o^2 + 2F_c^2)/3$.

Detailed descriptions for products



Benzofuran-2-ylidiphenylphosphine oxide (3a)^[1]

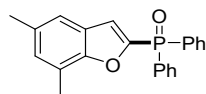
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (54 mg, 85%). ¹H NMR (400 MHz, CDCl₃) δ 7.82 (dd, *J* = 12.9, 7.0 Hz, 4H), 7.65 (d, *J* = 7.8 Hz, 1H), 7.58 (td, *J* = 7.3, 1.6 Hz, 2H), 7.54 – 7.46 (m, 5H), 7.42 (d, *J* = 2.5 Hz, 1H), 7.39 (t, *J* = 7.7 Hz, 1H), 7.27 (d, *J* = 6.0 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 157.91 (d, *J*_{C-P} = 8.1 Hz), 150.33 (d, *J*_{C-P} = 130.4 Hz), 132.50 (d, *J*_{C-P} = 2.9 Hz), 131.74 (d, *J*_{C-P} = 10.6 Hz), 131.00 (d, *J*_{C-P} = 111.1 Hz), 128.63 (d, *J*_{C-P} = 12.8 Hz), 126.74, 126.57 (d, *J*_{C-P} = 9.1 Hz), 123.56, 122.38, 119.50 (d, *J*_{C-P} = 17.5 Hz), 112.14. ³¹P NMR (162 MHz, CDCl₃) δ 17.53. IR (KBr): 1110 cm⁻¹ (P=O).



(4,6-Dimethylbenzofuran-2-yl)diphenylphosphine oxide (3b)

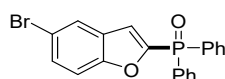
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (42.9 mg, 62%), melting point: 160 – 162 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.81 (dd, *J* = 12.9, 7.6 Hz, 4H), 7.57 (t, *J* = 7.5 Hz, 2H), 7.53 – 7.43 (m, 5H), 7.15 (s, 1H), 6.91 (s, 1H), 2.47 (s, 3H), 2.42 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 158.50 (d, *J*_{C-P} = 8.0 Hz), 148.75 (d, *J*_{C-P} = 132.8 Hz), 137.45, 132.44 (d, *J*_{C-P} = 2.9 Hz), 132.04, 131.78 (d, *J*_{C-P} = 10.6 Hz), 131.34 (d, *J*_{C-P} = 111.1), 128.63 (d, *J*_{C-P} = 12.8 Hz), 125.48, 124.28 (d, *J*_{C-P} = 9.0 Hz), 118.43 (d, *J*_{C-P} = 17.5 Hz), 109.59, 21.79, 18.52. ³¹P NMR

(162 MHz, CDCl₃) δ 17.74. HRMS (ESI) calcd for C₂₂H₁₉O₂P [M+H]⁺: 347.1195; found: 347.1201. IR (KBr): 1102 cm⁻¹ (P=O).



(5,7-Dimethylbenzofuran-2-yl)diphenylphosphine oxide (3c)

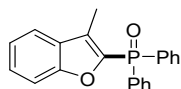
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (45 mg, 65%), melting point: 130 – 132 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.80 (dd, *J* = 12.8, 7.6 Hz, 4H), 7.57 (t, *J* = 7.4 Hz, 2H), 7.49 (td, *J* = 7.9, 2.7 Hz, 4H), 7.28 (t, *J* = 1.7 Hz, 1H), 7.24 (s, 1H), 7.01 (s, 1H), 2.44 (s, 3H), 2.39 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 155.83 (d, *J*_{C-P} = 7.9 Hz), 149.77 (d, *J*_{C-P} = 131.7 Hz), 133.25, 132.44 (d, *J*_{C-P} = 2.9 Hz), 131.3 (d, *J*_{C-P} = 111.1 Hz), 131.80 (d, *J*_{C-P} = 10.6 Hz), 129.14, 128.61 (d, *J*_{C-P} = 12.8 Hz), 126.22 (d, *J*_{C-P} = 9.1 Hz), 121.85, 119.75 (d, *J*_{C-P} = 18.1 Hz), 119.29, 21.22, 15.05. ³¹P NMR (162 MHz, CDCl₃) δ 17.78. HRMS (ESI) calcd for C₂₂H₁₉O₂P [M+H]⁺: 347.1195; found: 347.1199. IR (KBr): 1102 cm⁻¹ (P=O).



(5-Bromobenzofuran-2-yl)diphenylphosphine oxide (3d)

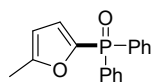
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a colorless oil (43.6 mg, 55%). ¹H NMR (400 MHz, CDCl₃) δ 7.86 – 7.74 (m, 5H), 7.61 – 7.56 (m, 2H), 7.54 – 7.43 (m, 5H), 7.42 – 7.31 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 156.56 (d, *J*_{C-P} = 8.1 Hz), 151.99 (d, *J*_{C-P} = 127.8 Hz), 132.65 (d, *J*_{C-P} = 2.9 Hz), 131.68 (d, *J*_{C-P} = 10.6 Hz), 130.55 (d, *J*_{C-P} = 111.4 Hz), 129.66, 128.69 (d, *J*_{C-P} = 12.8

Hz), 128.49 (d, J_{C-P} = 9.1 Hz), 124.87, 118.54 (d, J_{C-P} = 17.4 Hz), 116.60 (d, J_{C-P} = 1.4 Hz), 113.56. ^{31}P NMR (162 MHz, CDCl_3) δ 17.19. HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{14}\text{BrO}_2\text{P}$ $[\text{M}+\text{H}]^+$: 396.9988; found: 396.9991. IR (KBr): 1114 cm^{-1} (P=O).



(3-Methylbenzofuran-2-yl)diphenylphosphine oxide (3e)

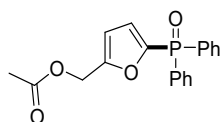
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (46.5 mg, 70%), melting point: $178 - 180\text{ }^\circ\text{C}$. ^1H NMR (400 MHz, CDCl_3) δ 7.88 – 7.72 (m, 4H), 7.61 (d, J = 7.7 Hz, 1H), 7.58 – 7.51 (m, 2H), 7.50 – 7.32 (m, 6H), 7.31 – 7.25 (m, 1H), 2.57 (d, J = 2.0 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 156.50 (d, J_{C-P} = 8.5 Hz), 143.26 (d, J_{C-P} = 133.3 Hz), 132.72, 132.23 (d, J_{C-P} = 2.8 Hz), 131.67 (d, J_{C-P} = 10.4 Hz), 130.19 (d, J_{C-P} = 17.0 Hz), 129.08 (d, J_{C-P} = 9.1 Hz), 128.53 (d, J_{C-P} = 12.7 Hz), 126.68, 122.97, 120.58, 111.86, 8.66. ^{31}P NMR (162 MHz, CDCl_3) δ 19.51. HRMS (ESI) calcd for $\text{C}_{21}\text{H}_{17}\text{O}_2\text{P}$ $[\text{M}+\text{H}]^+$: 333.1039; found: 333.1038. IR (KBr): 1120 cm^{-1} (P=O).



(5-Methylfuran-2-yl)diphenylphosphine oxide (3f)

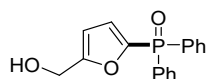
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (50.8 mg, 90%), melting point: $104 - 106\text{ }^\circ\text{C}$. ^1H NMR (400 MHz, CDCl_3) δ 7.74 (dd, J = 12.5, 7.4 Hz, 4H), 7.54 (t, J = 7.2 Hz, 2H), 7.46 (dt, J = 7.0, 3.7 Hz, 4H), 6.84 (s, 1H), 6.10 (s, 1H), 2.33 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.32 (d, J_{C-P}

= 7.1 Hz), 145.85 (d, J_{C-P} = 139.5 Hz), 132.10 (d, J_{C-P} = 2.6 Hz), 131.69 (d, J_{C-P} = 111.2 Hz), 131.62 (d, J_{C-P} = 10.4 Hz), 128.41 (d, J_{C-P} = 12.5 Hz), 124.71 (d, J_{C-P} = 18.9 Hz), 107.16 (d, J_{C-P} = 8.0 Hz), 13.85. ^{31}P NMR (162 MHz, CDCl_3) δ 16.27. HRMS (ESI) calcd for $\text{C}_{17}\text{H}_{15}\text{O}_2\text{P}$ $[\text{M}+\text{H}]^+$: 283.0882; found: 283.0878. IR (KBr): 1118 cm^{-1} (P=O).



(5-(Diphenylphosphoryl)furan-2-yl)methyl acetate (3g)

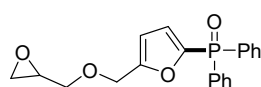
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a colorless oil (44.2 mg, 65%). ^1H NMR (400 MHz, CDCl_3) δ 7.80 – 7.68 (m, 4H), 7.57 (td, J = 7.3, 1.5 Hz, 2H), 7.48 (td, J = 7.4, 3.1 Hz, 4H), 6.94 (dd, J = 3.4, 1.9 Hz, 1H), 6.50 (dd, J = 3.5, 1.5 Hz, 1H), 5.07 (s, 2H), 2.05 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 170.33, 155.92 (d, J_{C-P} = 7.1 Hz), 148.73 (d, J_{C-P} = 134.2 Hz), 132.36 (d, J_{C-P} = 2.9 Hz), 131.67 (d, J_{C-P} = 10.5 Hz), 131.33 (d, J_{C-P} = 111.5 Hz), 128.56 (d, J_{C-P} = 12.7 Hz), 123.98 (d, J_{C-P} = 18.3 Hz), 111.07 (d, J_{C-P} = 8.0 Hz), 57.79, 20.72. ^{31}P NMR (162 MHz, CDCl_3) δ 15.92. HRMS (ESI) calcd for $\text{C}_{19}\text{H}_{17}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$: 341.0937; found: 341.0935. IR (KBr): 2923 (C-H), 1120 cm^{-1} (P=O).



(5-(Hydroxymethyl)furan-2-yl)diphenylphosphine oxide (3g')

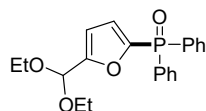
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (11.9 mg, 20%), melting point: 188 – 191 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.73 (ddt, J = 12.8, 6.9, 1.3 Hz, 4H), 7.59 – 7.54 (m, 2H), 7.48 (ddd, J = 10.2, 5.1, 2.2

Hz, 4H), 6.87 (dd, $J = 3.3, 1.8$ Hz, 1H), 6.43 – 6.35 (m, 1H), 4.64 (s, 2H), 1.74 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 161.10 (d, $J_{\text{C-P}} = 6.6$ Hz), 147.68 (d, $J_{\text{C-P}} = 135.7$ Hz), 132.41 (d, $J_{\text{C-P}} = 2.9$ Hz), 131.76 (d, $J_{\text{C-P}} = 10.5$ Hz), 131.39 (d, $J_{\text{C-P}} = 111.5$ Hz), 128.65 (d, $J_{\text{C-P}} = 12.8$ Hz), 124.29 (d, $J_{\text{C-P}} = 18.8$ Hz), 108.39 (d, $J_{\text{C-P}} = 8.2$ Hz), 57.62. ^{31}P NMR (162 MHz, CDCl_3) δ 16.36. HRMS (ESI) calcd for $\text{C}_{17}\text{H}_{15}\text{O}_3\text{P}$ $[\text{M}+\text{H}]^+$: 299.0832; found: 299.0835. IR (KBr): 2921 (OH), 1121 cm^{-1} (P=O).



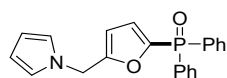
(5-((Oxiran-2-ylmethoxy)methyl)furan-2-yl)diphenylphosphine oxide (3h)

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a colorless oil (46 mg, 65%). Colorless oil, 46 mg, 65% yield, PE/EA = 5/1 as eluent. ^1H NMR (400 MHz, CDCl_3) δ 7.80 – 7.68 (m, 4H), 7.60 – 7.45 (m, 6H), 6.95 (dd, $J = 3.2, 1.9$ Hz, 1H), 6.54 – 6.39 (m, 1H), 4.67 – 4.46 (m, 2H), 3.76 (dd, $J = 11.5, 2.8$ Hz, 1H), 3.38 (dd, $J = 11.5, 5.9$ Hz, 1H), 3.11 (ddt, $J = 5.8, 4.1, 2.8$ Hz, 1H), 2.94 – 2.62 (m, 1H), 2.55 (dd, $J = 5.0, 2.7$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 158.14 (d, $J_{\text{C-P}} = 6.9$ Hz), 148.14 (d, $J_{\text{C-P}} = 135.3$ Hz), 132.29 (d, $J_{\text{C-P}} = 2.9$ Hz), 131.63 (d, $J_{\text{C-P}} = 10.5$ Hz), 131.39 (d, $J_{\text{C-P}} = 111.4$ Hz), 128.52 (d, $J_{\text{C-P}} = 12.8$ Hz), 123.99 (d, $J_{\text{C-P}} = 18.4$ Hz), 110.07 (d, $J_{\text{C-P}} = 8.1$ Hz), 70.87, 65.02, 50.54, 44.05. ^{31}P NMR (162 MHz, CDCl_3) δ 16.15. HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{19}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$: 355.1094; found: 355.1086. IR (KBr): 1119 cm^{-1} (P=O).



(5-(Diethoxymethyl)furan-2-yl)diphenylphosphine oxide (3i)

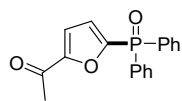
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a colorless oil (40.7 mg, 55%). ^1H NMR (400 MHz, CDCl_3) δ 7.79 – 7.70 (m, 4H), 7.58 – 7.52 (m, 2H), 7.50 – 7.44 (m, 4H), 7.02 (dd, J = 3.4, 1.9 Hz, 1H), 6.59 – 6.50 (m, 1H), 5.54 (s, 1H), 3.61 – 3.52 (m, 4H), 1.18 (t, J = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 158.52 (d, $J_{\text{C-P}}$ = 6.6 Hz), 147.70 (d, $J_{\text{C-P}}$ = 135.7 Hz), 132.30 (d, $J_{\text{C-P}}$ = 2.9 Hz), 131.67 (d, $J_{\text{C-P}}$ = 10.5 Hz), 131.55 (d, $J_{\text{C-P}}$ = 111.3 Hz), 128.52 (d, $J_{\text{C-P}}$ = 12.8 Hz), 123.80 (d, $J_{\text{C-P}}$ = 18.3 Hz), 109.17 (d, $J_{\text{C-P}}$ = 8.1 Hz), 96.02, 61.44, 15.10. ^{31}P NMR (162 MHz, CDCl_3) δ 16.15. HRMS (ESI) calcd for $\text{C}_{21}\text{H}_{23}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$: 371.1407; found: 371.1402. IR (KBr): 3053 (C-H), 1119 cm^{-1} (P=O).



(5-((1H-Pyrrol-1-yl)methyl)furan-2-yl)diphenylphosphine oxide (3j)

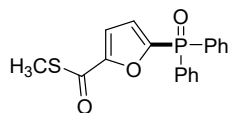
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a brown oil (34.7 mg, 50%). ^1H NMR (400 MHz, CDCl_3) δ 7.70 (ddd, J = 12.8, 8.2, 1.2 Hz, 4H), 7.60 – 7.51 (m, 2H), 7.49 – 7.42 (m, 4H), 6.88 (dd, J = 3.3, 1.9 Hz, 1H), 6.65 (t, J = 2.1 Hz, 2H), 6.31 – 6.23 (m, 1H), 6.15 (t, J = 2.1 Hz, 2H), 5.05 (s, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.64 (d, $J_{\text{C-P}}$ = 6.7 Hz), 148.17 (d, $J_{\text{C-P}}$ = 134.9 Hz), 132.34 (d, $J_{\text{C-P}}$ = 2.9 Hz), 131.62 (d, $J_{\text{C-P}}$ = 10.5 Hz), 131.31 (d, $J_{\text{C-P}}$ = 111.5 Hz), 128.55 (d, $J_{\text{C-P}}$ = 12.8 Hz), 124.07 (d, $J_{\text{C-P}}$ = 18.3 Hz), 120.85, 108.89, 108.72 (d, $J_{\text{C-P}}$ = 8.0 Hz), 46.21. ^{31}P NMR (162 MHz, CDCl_3) δ 16.14. HRMS (ESI) calcd for $\text{C}_{21}\text{H}_{18}\text{NO}_2\text{P}$ $[\text{M}+\text{H}]^+$:

348.1148; found: 348.1141. IR (KBr): 1118 cm^{-1} (P=O).



1-(5-(Diphenylphosphoryl)furan-2-yl)ethan-1-one (3k)^[1]

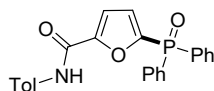
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 3/1) and obtained as a white solid (46.5 mg, 75%). ^1H NMR (400 MHz, CDCl_3) δ 7.76 (dd, J = 13.0, 7.6 Hz, 4H), 7.60 (t, J = 7.5 Hz, 2H), 7.51 (td, J = 7.7, 2.8 Hz, 4H), 7.22 (d, J = 3.4 Hz, 1H), 7.12 (d, J = 3.5 Hz, 1H), 2.47 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 187.21, 157.10 (d, $J_{\text{C-P}}$ = 5.9 Hz), 152.08 (d, $J_{\text{C-P}}$ = 126.8 Hz), 132.78 (d, $J_{\text{C-P}}$ = 2.9 Hz), 131.65 (d, $J_{\text{C-P}}$ = 10.6 Hz), 130.51 (d, $J_{\text{C-P}}$ = 111.8 Hz), 128.80 (d, $J_{\text{C-P}}$ = 12.8 Hz), 124.03 (d, $J_{\text{C-P}}$ = 17.6 Hz), 116.20 (d, $J_{\text{C-P}}$ = 7.9 Hz), 26.45. ^{31}P NMR (162 MHz, CDCl_3) δ 16.43. IR (KBr): 2919 (C-H), 1119 cm^{-1} (P=O).



S-Methyl 5-(diphenylphosphoryl)furan-2-carbothioate (3l)

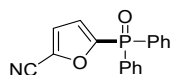
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (47.9 mg, 70%), melting point: 138 – 141 $^{\circ}\text{C}$. ^1H NMR (400 MHz, CDCl_3) δ 7.86 – 7.74 (m, 4H), 7.63 – 7.56 (m, 2H), 7.51 (td, J = 7.6, 3.2 Hz, 4H), 7.27 – 7.14 (m, 2H), 2.42 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 181.15, 155.05 (d, $J_{\text{C-P}}$ = 6.3 Hz), 151.94 (d, $J_{\text{C-P}}$ = 126.7 Hz), 132.65 (d, $J_{\text{C-P}}$ = 2.9 Hz), 131.56 (d, $J_{\text{C-P}}$ = 10.6 Hz), 130.61 (d, $J_{\text{C-P}}$ = 111.6 Hz), 128.70 (d, $J_{\text{C-P}}$ = 12.9 Hz), 123.73 (d, $J_{\text{C-P}}$ = 17.0 Hz), 114.56 (d,

$J_{C-P} = 7.8$ Hz), 10.91. ^{31}P NMR (162 MHz, CDCl_3) δ 16.01. HRMS (ESI) calcd for $\text{C}_{18}\text{H}_{15}\text{O}_3\text{PS}$ $[\text{M}+\text{H}]^+$: 343.0552; found: 343.0558. IR (KBr): 3090 (C-H), 1122 cm^{-1} (P=O).



5-(Diphenylphosphoryl)-N-(p-tolyl)furan-2-carboxamide (3m)

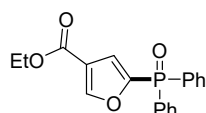
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 1/1) and obtained as a white solid (48.1 mg, 60%), melting point: 185 – 187 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.27 (s, 1H), 7.74 (dd, $J = 12.8, 7.5$ Hz, 4H), 7.62 (t, $J = 7.4$ Hz, 2H), 7.58 – 7.46 (m, 6H), 7.26 (dd, $J = 3.6, 1.4$ Hz, 1H), 7.13 (d, $J = 8.4$ Hz, 2H), 6.79 (dd, $J = 3.6, 1.4$ Hz, 1H), 2.31 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 155.19, 153.22 (d, $J_{C-P} = 5.9$ Hz), 150.25 (d, $J_{C-P} = 127.5$ Hz), 134.56, 134.40, 132.91 (d, $J_{C-P} = 2.7$ Hz), 131.76 (d, $J_{C-P} = 10.6$ Hz), 130.29 (d, $J_{C-P} = 112.4$ Hz), 129.57, 128.89 (d, $J_{C-P} = 12.8$ Hz), 124.65 (d, $J_{C-P} = 18.2$ Hz), 120.19, 114.92 (d, $J_{C-P} = 7.8$ Hz), 20.92. ^{31}P NMR (162 MHz, CDCl_3) δ 16.94. HRMS (ESI) calcd for $\text{C}_{24}\text{H}_{20}\text{NO}_3\text{P}$ $[\text{M}+\text{H}]^+$: 402.1254; found: 402.1256. IR (KBr): 3114 (C-H), 1119 cm^{-1} (P=O).



5-(Diphenylphosphoryl)furan-2-carbonitrile (3n)

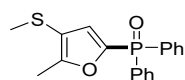
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a colorless oil (32.2 mg, 55%). ^1H NMR (400 MHz, CDCl_3) δ 7.74 (ddd, $J = 13.0, 5.2, 3.2$ Hz, 4H), 7.66 – 7.59 (m, 2H), 7.53 (td, $J = 7.5, 3.3$ Hz, 4H), 7.20 (d, $J = 1.3$ Hz,

2H). ^{13}C NMR (101 MHz, CDCl_3) δ 154.57 (d, $J_{\text{C-P}} = 123.7$ Hz), 133.00 (d, $J_{\text{C-P}} = 2.9$ Hz), 131.56 (d, $J_{\text{C-P}} = 10.7$ Hz), 130.90 (d, $J_{\text{C-P}} = 8.6$ Hz), 129.86 (d, $J_{\text{C-P}} = 112.4$ Hz), 128.87 (d, $J_{\text{C-P}} = 13.0$ Hz), 122.88 (d, $J_{\text{C-P}} = 16.2$ Hz), 122.00 (d, $J_{\text{C-P}} = 7.4$ Hz), 110.50. ^{31}P NMR (162 MHz, CDCl_3) δ 16.09. HRMS (ESI) calcd for $\text{C}_{17}\text{H}_{12}\text{NO}_2\text{P}$ $[\text{M}+\text{H}]^+$: 294.0678; found: 294.0675. IR (KBr): 2234 (C-N), 1119 cm^{-1} (P=O).



Ethyl 5-(diphenylphosphoryl)furan-3-carboxylate (3o)

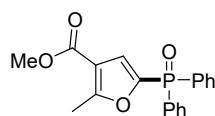
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (54.4 mg, 80%), melting point: 108 – 110 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.79 – 7.70 (m, 4H), 7.58 – 7.52 (m, 3H), 7.47 (td, $J = 7.4, 3.2$ Hz, 4H), 6.92 (t, $J = 1.5$ Hz, 1H), 4.02 (q, $J = 7.2$ Hz, 2H), 0.98 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 161.44, 151.60 (d, $J_{\text{C-P}} = 124.6$ Hz), 146.90 (d, $J_{\text{C-P}} = 7.7$ Hz), 131.90 (d, $J_{\text{C-P}} = 2.9$ Hz), 131.25 (d, $J_{\text{C-P}} = 10.3$ Hz), 131.18 (d, $J_{\text{C-P}} = 114.3$ Hz), 128.41 (d, $J_{\text{C-P}} = 14.3$ Hz), 128.15 (d, $J_{\text{C-P}} = 13.0$ Hz), 112.47 (d, $J_{\text{C-P}} = 5.9$ Hz), 60.83, 13.36. ^{31}P NMR (162 MHz, CDCl_3) δ 17.22. HRMS (ESI) calcd for $\text{C}_{19}\text{H}_{17}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$: 341.0937; found: 341.0931. IR (KBr): 3057 (C-H), 1116 cm^{-1} (P=O).



(5-Methyl-4-(methylthio)furan-2-yl)diphenylphosphine oxide (3p)

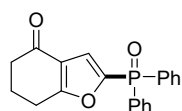
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a colorless oil (49.2 mg, 75%). ^1H NMR (400 MHz, CDCl_3) δ 7.74 (ddd, $J = 12.7, 8.2,$

1.2 Hz, 4H), 7.57 (td, $J = 6.5, 5.9, 1.4$ Hz, 2H), 7.51 – 7.45 (m, 4H), 6.92 (d, $J = 1.9$ Hz, 1H), 2.37 (s, 3H), 2.25 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.83 (d, $J_{\text{C-P}} = 5.8$ Hz), 146.04 (d, $J_{\text{C-P}} = 136.3$ Hz), 132.33 (d, $J_{\text{C-P}} = 2.9$ Hz), 131.68 (d, $J_{\text{C-P}} = 10.5$ Hz), 131.30 (d, $J_{\text{C-P}} = 111.4$ Hz), 128.56 (d, $J_{\text{C-P}} = 12.7$ Hz), 127.41 (d, $J_{\text{C-P}} = 17.4$ Hz), 114.51 (d, $J_{\text{C-P}} = 8.6$ Hz), 19.13, 12.40. ^{31}P NMR (162 MHz, CDCl_3) δ 15.85. HRMS (ESI) calcd for $\text{C}_{18}\text{H}_{17}\text{O}_2\text{PS}$ $[\text{M}+\text{H}]^+$: 329.0760; found: 329.0761. IR (KBr): 2920 (C-H), 1105 cm^{-1} (P=O).



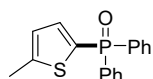
Methyl 5-(diphenylphosphoryl)-2-methylfuran-3-carboxylate (3q)

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a colorless oil (44.2 mg, 65%). ^1H NMR (400 MHz, CDCl_3) δ 7.74 (dd, $J = 12.7, 8.1$ Hz, 4H), 7.59 (dd, $J = 10.5, 4.3$ Hz, 2H), 7.56 – 7.42 (m, 4H), 7.09 (d, $J = 1.8$ Hz, 1H), 3.81 (s, 3H), 2.63 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 165.37 (d, $J_{\text{C-P}} = 6.3$ Hz), 163.48, 146.45 (d, $J_{\text{C-P}} = 134.3$ Hz), 132.53 (d, $J_{\text{C-P}} = 2.9$ Hz), 131.71 (d, $J_{\text{C-P}} = 10.5$ Hz), 130.81 (d, $J_{\text{C-P}} = 112.1$ Hz), 128.67 (d, $J_{\text{C-P}} = 12.8$ Hz), 124.00 (d, $J_{\text{C-P}} = 18.1$ Hz), 114.62 (d, $J_{\text{C-P}} = 8.1$ Hz), 51.63, 14.29. ^{31}P NMR (162 MHz, CDCl_3) δ 15.85. HRMS (ESI) calcd for $\text{C}_{19}\text{H}_{17}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$: 341.0937; found: 341.0938. IR (KBr): 2925 (C-H), 1118 cm^{-1} (P=O).



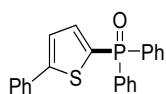
2-(Diphenylphosphoryl)-6,7-dihydrobenzofuran-4(5H)-one (3r)

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 3/1) and obtained as a white solid (47 mg, 70%), melting point: 113 – 115 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.86 – 7.66 (m, 4H), 7.65 – 7.56 (m, 2H), 7.51 (td, *J* = 7.5, 3.0 Hz, 4H), 7.01 (d, *J* = 2.0 Hz, 1H), 2.94 (t, *J* = 6.2 Hz, 2H), 2.60 – 2.48 (m, 2H), 2.26 – 2.13 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 193.82, 172.38 (d, *J*_{C-P} = 7.1 Hz), 149.07 (d, *J*_{C-P} = 131.0 Hz), 132.68 (d, *J*_{C-P} = 2.9 Hz), 131.71 (d, *J*_{C-P} = 10.6 Hz), 130.26 (d, *J*_{C-P} = 112.4 Hz), 128.73 (d, *J*_{C-P} = 12.8 Hz), 121.59 (d, *J*_{C-P} = 8.3 Hz), 119.59 (d, *J*_{C-P} = 18.1 Hz), 37.59, 23.77, 22.24. ³¹P NMR (162 MHz, CDCl₃) δ 16.35. HRMS (ESI) calcd for C₂₀H₁₇O₃P [M+H]⁺: 337.0988; found: 337.0992. IR (KBr): 3061 (C-H), 1114 cm⁻¹ (P=O).



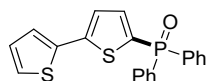
(5-Methylthiophen-2-yl)diphenylphosphine oxide (3s)^[1]

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (37 mg, 62%). ¹H NMR (400 MHz, CDCl₃) δ 7.73 (dd, *J* = 12.6, 7.5 Hz, 4H), 7.55 (t, *J* = 7.5 Hz, 2H), 7.47 (td, *J* = 7.7, 2.7 Hz, 4H), 7.27 (s, 1H), 6.85 (s, 1H), 2.53 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 149.64 (d, *J*_{C-P} = 4.7 Hz), 137.52 (d, *J*_{C-P} = 9.3 Hz), 133.04 (d, *J*_{C-P} = 109.8 Hz), 132.11 (d, *J*_{C-P} = 2.9 Hz), 131.82 (d, *J*_{C-P} = 10.3 Hz), 130.91 (d, *J*_{C-P} = 114.6 Hz), 128.48 (d, *J*_{C-P} = 12.6 Hz), 126.88 (d, *J*_{C-P} = 12.9 Hz), 15.47. ³¹P NMR (162 MHz, CDCl₃) δ 21.65. IR (KBr): 2960 (C-H), 1015 cm⁻¹ (P=O).



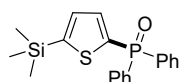
Diphenyl(5-phenylthiophen-2-yl)phosphine oxide (3t)^[1]

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (46.8 mg, 65%). ^1H NMR (400 MHz, CDCl_3) δ 7.78 (ddt, J = 12.6, 6.9, 1.4 Hz, 4H), 7.58 (dddd, J = 10.6, 8.8, 3.4, 1.6 Hz, 4H), 7.49 (ddd, J = 8.5, 6.6, 3.0 Hz, 4H), 7.45 – 7.29 (m, 5H). ^{13}C NMR (101 MHz, CDCl_3) δ 153.33 (d, $J_{\text{C-P}}$ = 4.9 Hz), 137.92 (d, $J_{\text{C-P}}$ = 9.1 Hz), 133.35, 133.20 (d, $J_{\text{C-P}}$ = 1.4 Hz), 132.54 (d, $J_{\text{C-P}}$ = 113.1 Hz), 132.27 (d, $J_{\text{C-P}}$ = 2.8 Hz), 131.86 (d, $J_{\text{C-P}}$ = 10.4 Hz), 129.09, 128.71, 128.58 (d, $J_{\text{C-P}}$ = 12.6 Hz), 126.27, 124.15 (d, $J_{\text{C-P}}$ = 12.8 Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 21.67. IR (KBr): 1014 cm^{-1} (P=O).



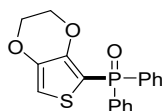
[2,2'-Bithiophen]-5-ylidiphenylphosphine oxide (3u)

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (47.6 mg, 65%), melting point: 110 – 112 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.87 – 7.69 (m, 4H), 7.62 – 7.45 (m, 6H), 7.35 (dd, J = 7.6, 3.7 Hz, 1H), 7.24 (dt, J = 20.1, 3.6 Hz, 3H), 7.00 (dd, J = 5.0, 3.7 Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 146.06 (d, $J_{\text{C-P}}$ = 5.3 Hz), 137.64 (d, $J_{\text{C-P}}$ = 9.0 Hz), 135.74 (d, $J_{\text{C-P}}$ = 1.7 Hz), 132.56 (d, $J_{\text{C-P}}$ = 110.1 Hz), 132.26 (d, $J_{\text{C-P}}$ = 2.9 Hz), 131.88 (d, $J_{\text{C-P}}$ = 111.7 Hz), 131.75 (d, $J_{\text{C-P}}$ = 10.5 Hz), 128.54 (d, $J_{\text{C-P}}$ = 12.6 Hz), 128.05, 125.96, 125.20, 124.46 (d, $J_{\text{C-P}}$ = 12.9 Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 21.56. HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{15}\text{OPS}_2$ $[\text{M}+\text{H}]^+$: 367.0375; found: 367.0373. IR (KBr): 1097 cm^{-1} (P=O).



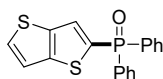
Diphenyl(5-(trimethylsilyl)thiophen-2-yl)phosphine oxide (3v)^[1]

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 3/1) and obtained as a white solid (46.3 mg, 65%). ¹H NMR (400 MHz, CDCl₃) δ 7.75 (ddt, *J* = 12.5, 6.9, 1.4 Hz, 4H), 7.60 – 7.53 (m, 2H), 7.53 – 7.44 (m, 5H), 7.29 (dd, *J* = 3.5, 1.6 Hz, 1H), 0.32 (s, 9H). ¹³C NMR (101 MHz, CDCl₃) δ 150.74, 138.90, 137.83 (d, *J*_{C-P} = 11.0 Hz), 134.72 (d, *J*_{C-P} = 15.3 Hz), 133.10 (d, *J*_{C-P} = 109.6 Hz), 132.14 (d, *J*_{C-P} = 2.9 Hz), 131.80 (d, *J*_{C-P} = 10.4 Hz), 128.51 (d, *J*_{C-P} = 12.6 Hz), -0.13. ³¹P NMR (162 MHz, CDCl₃) δ 21.64. IR (KBr): 2954 (C-H), 1016 cm⁻¹ (P=O).



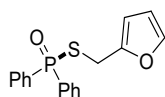
(2,3-Dihydrothieno[3,4-b][1,4]dioxin-5-yl)diphenylphosphine oxide (3w)^[1]

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (51.3 mg, 75%). ¹H NMR (400 MHz, CDCl₃) δ 7.81 – 7.71 (m, 4H), 7.57 – 7.51 (m, 2H), 7.49 – 7.41 (m, 4H), 6.72 (d, *J* = 5.0 Hz, 1H), 4.16 (s, 4H). ¹³C NMR (101 MHz, CDCl₃) δ 147.12 (d, *J*_{C-P} = 4.6 Hz), 142.36 (d, *J*_{C-P} = 11.2 Hz), 132.41 (d, *J*_{C-P} = 111.6 Hz), 132.01 (d, *J*_{C-P} = 2.9 Hz), 131.65 (d, *J*_{C-P} = 10.7 Hz), 128.28 (d, *J*_{C-P} = 12.7 Hz), 108.51 (d, *J*_{C-P} = 4.0 Hz), 105.90 (d, *J*_{C-P} = 114.4 Hz), 64.78, 64.06. ³¹P NMR (162 MHz, CDCl₃) δ 20.53. IR (KBr): 2921 (C-H), 1024 cm⁻¹ (P=O).



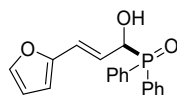
Diphenyl(thieno[3,2-b]thiophen-2-yl)phosphine oxide (3x)

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (49 mg, 72%), melting point: 183 – 186 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.82 – 7.74 (m, 4H), 7.63 – 7.43 (m, 8H), 7.26 (d, $J = 5.5$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 146.00 (d, $J_{\text{C-P}} = 6.0$ Hz), 139.99 (d, $J_{\text{C-P}} = 15.6$ Hz), 135.75 (d, $J_{\text{C-P}} = 108.2$ Hz), 133.02, 132.35 (d, $J_{\text{C-P}} = 2.9$ Hz), 131.89 (d, $J_{\text{C-P}} = 10.3$ Hz), 131.15, 128.76 (d, $J_{\text{C-P}} = 10.0$ Hz), 128.58 (d, $J_{\text{C-P}} = 12.6$ Hz), 119.44 (d, $J_{\text{C-P}} = 1.7$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 22.27. HRMS (ESI) calcd for $\text{C}_{18}\text{H}_{13}\text{OPS}_2$ $[\text{M}+\text{H}]^+$: 341.0218; found: 341.0225. IR (KBr): 1012 cm^{-1} (P=O).



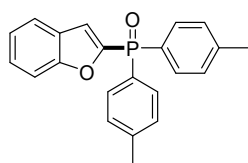
Furan-2-ylmethyl diphenylphosphinothioate (3y)

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a colorless oil (47.1 mg, 75%). ^1H NMR (400 MHz, CDCl_3) δ 7.89 – 7.79 (m, 4H), 7.54 – 7.48 (m, 2H), 7.44 (td, $J = 7.4, 3.6$ Hz, 4H), 7.28 – 7.12 (m, 1H), 6.12 (dd, $J = 3.2, 2.0$ Hz, 1H), 5.99 (d, $J = 3.2$ Hz, 1H), 4.09 (d, $J = 10.0$ Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 149.41 (d, $J_{\text{C-P}} = 4.8$ Hz), 142.28, 132.70 (d, $J_{\text{C-P}} = 107.4$ Hz), 132.26 (d, $J_{\text{C-P}} = 3.0$ Hz), 131.39 (d, $J_{\text{C-P}} = 10.6$ Hz), 128.55 (d, $J_{\text{C-P}} = 13.1$ Hz), 110.45, 108.64, 25.33 (d, $J_{\text{C-P}} = 2.2$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 42.98. HRMS (ESI) calcd for $\text{C}_{17}\text{H}_{15}\text{O}_2\text{PS}$ $[\text{M}+\text{H}]^+$: 315.0609; found: 315.0603. IR (KBr): 1113 cm^{-1} (P=O).



(3-(furan-2-yl)-1-hydroxyallyl)diphenylphosphine oxide (3z)

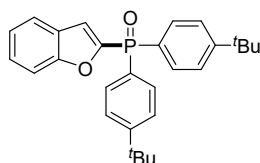
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 2/1) and obtained as a pale yellow solid (35.6 mg, 55%), melting point: 157 – 159 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.89 – 7.77 (m, 4H), 7.49 (t, *J* = 7.4 Hz, 2H), 7.44 – 7.36 (m, 4H), 7.25 (s, 1H), 6.44 (ddd, *J* = 15.7, 4.4, 1.7 Hz, 1H), 6.33 – 6.26 (m, 1H), 6.21 (dt, *J* = 15.8, 5.2 Hz, 1H), 6.08 (d, *J* = 3.3 Hz, 1H), 5.57 (s, 1H), 5.15 (dd, *J* = 9.4, 5.9 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 152.27 (d, *J*_{C-P} = 3.6 Hz), 142.12, 132.06 (d, *J*_{C-P} = 20.7 Hz), 132.06 (d, *J*_{C-P} = 3.0 Hz), 130.47 (d, *J*_{C-P} = 39.8 Hz), 129.53 (d, *J*_{C-P} = 39.8 Hz), 128.42 (d, *J*_{C-P} = 11.4 Hz), 122.54 (d, *J*_{C-P} = 1.8 Hz), 121.07 (d, *J*_{C-P} = 11.1 Hz), 111.26, 108.56 (d, *J*_{C-P} = 2.5 Hz), 72.48 (d, *J*_{C-P} = 83.6 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 31.06. HRMS (ESI) calcd for C₁₉H₁₇O₃P [M+H]⁺: 325.0988; found: 325.0979. IR (KBr): 3112 (OH), 1150 cm⁻¹ (P=O).



Benzofuran-2-yl-di-p-tolylphosphine oxide (4a)

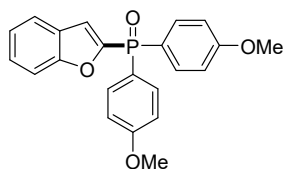
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (34.6 mg, 50%), melting point: 128 – 130 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.73 – 7.61 (m, 5H), 7.51 (d, *J* = 8.4 Hz, 1H), 7.41 – 7.35 (m, 2H), 7.34 – 7.26 (m, 5H), 2.41 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 157.93 (d, *J*_{C-P} = 8.1 Hz), 150.90 (d, *J*_{C-P} = 130.3 Hz), 143.11 (d, *J*_{C-P} = 2.9 Hz), 131.85 (d, *J*_{C-P} = 11.0 Hz), 129.40 (d, *J*_{C-P}

= 13.3 Hz), 127.88 (d, J_{C-P} = 113.6 Hz), 126.73 (d, J_{C-P} = 8.9 Hz), 126.62, 123.51, 122.38, 119.21 (d, J_{C-P} = 17.5 Hz), 112.19, 21.74 (d, J_{C-P} = 1.4 Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 17.90. HRMS (ESI) calcd for $\text{C}_{22}\text{H}_{19}\text{O}_2\text{P}$ $[\text{M}+\text{H}]^+$: 347.1195; found: 347.1194. IR (KBr): 2918 (C-H), 1101 cm^{-1} (P=O).



Benzofuran-2-ylbis(4-(tert-butyl)phenyl)phosphine oxide (4b)

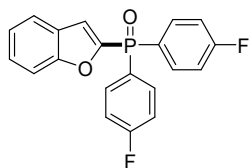
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (60.2 mg, 70%), melting point: 200 – 202 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.74 (dd, J = 12.4, 8.0 Hz, 4H), 7.65 (d, J = 7.8 Hz, 1H), 7.56 – 7.47 (m, 5H), 7.44 – 7.34 (m, 2H), 7.29 (d, J = 7.6 Hz, 1H), 1.33 (s, 18H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.95 (d, J_{C-P} = 8.0 Hz), 155.99 (d, J_{C-P} = 2.9 Hz), 151.09 (d, J_{C-P} = 129.6 Hz), 131.71 (d, J_{C-P} = 10.9 Hz), 127.96 (d, J_{C-P} = 113.5 Hz), 126.79 (d, J_{C-P} = 9.0 Hz), 126.59, 125.69 (d, J_{C-P} = 13.0 Hz), 123.50, 122.39, 119.14 (d, J_{C-P} = 17.5 Hz), 112.21, 35.14, 31.15. ^{31}P NMR (162 MHz, CDCl_3) δ 17.46. HRMS (ESI) calcd for $\text{C}_{28}\text{H}_{31}\text{O}_2\text{P}$ $[\text{M}+\text{H}]^+$: 431.2134; found: 431.2135. IR (KBr): 2960 (C-H), 1091 cm^{-1} (P=O).



Benzofuran-2-ylbis(4-methoxyphenyl)phosphine oxide (4c)

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as

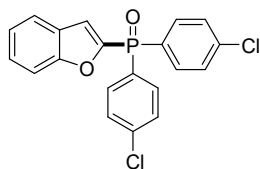
a white solid (55.2 mg, 73%), melting point: 138 – 140 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.71 (dd, $J = 12.3, 8.3$ Hz, 4H), 7.64 (d, $J = 7.8$ Hz, 1H), 7.51 (d, $J = 8.3$ Hz, 1H), 7.42 – 7.33 (m, 2H), 7.26 (d, $J = 7.1$ Hz, 1H), 6.99 (d, $J = 8.1$ Hz, 4H), 3.85 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 162.88 (d, $J_{\text{C-P}} = 2.9$ Hz), 157.87 (d, $J_{\text{C-P}} = 8.0$ Hz), 151.22 (d, $J_{\text{C-P}} = 130.7$ Hz), 133.74 (d, $J_{\text{C-P}} = 12.0$ Hz), 126.73 (d, $J_{\text{C-P}} = 9.0$ Hz), 126.56, 123.48, 122.40 (d, $J_{\text{C-P}} = 118.0$ Hz), 122.34, 118.95 (d, $J_{\text{C-P}} = 17.7$ Hz), 114.22 (d, $J_{\text{C-P}} = 13.8$ Hz), 112.14, 55.39. ^{31}P NMR (162 MHz, CDCl_3) δ 17.65. HRMS (ESI) calcd for $\text{C}_{22}\text{H}_{19}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$: 379.1094; found: 379.1089. IR (KBr): 3058 (C-H), 1115 cm^{-1} (P=O).



Benzofuran-2-ylbis(4-fluorophenyl)phosphine oxide (4d)

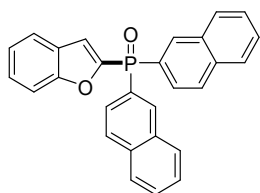
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (38.9 mg, 55%), melting point: 116 – 117 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.81 (ddd, $J = 13.0, 8.4, 5.6$ Hz, 4H), 7.68 (d, $J = 7.8$ Hz, 1H), 7.53 (d, $J = 8.4$ Hz, 1H), 7.47 (s, 1H), 7.42 (t, $J = 7.8$ Hz, 1H), 7.31 (t, $J = 7.5$ Hz, 1H), 7.20 (t, $J = 7.7$ Hz, 4H). ^{13}C NMR (101 MHz, CDCl_3) δ 165.54 (dd, $J_{\text{C-F/C-P}} = 254.7, 3.3$ Hz), 158.02 (d, $J_{\text{C-P}} = 8.3$ Hz), 149.74 (d, $J_{\text{C-P}} = 133.1$ Hz), 134.37 (dd, $J_{\text{C-F/C-P}} = 12.2, 9.0$ Hz), 127.09, 126.94 (dd, $J_{\text{C-F/C-P}} = 114.7, 3.4$ Hz), 126.55 (d, $J_{\text{C-P}} = 9.1$ Hz), 123.83, 122.59, 119.87 (d, $J_{\text{C-P}} = 17.8$ Hz), 116.29 (dd, $J_{\text{C-F/C-P}} = 21.5, 14.1$ Hz), 112.21. ^{19}F NMR (376 MHz,

CDCl_3) δ -105.24. ^{31}P NMR (162 MHz, CDCl_3) δ 15.88. HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{13}\text{F}_2\text{O}_2\text{P}$ $[\text{M}+\text{H}]^+$: 355.0694; found: 355.0691. IR (KBr): 1118 cm^{-1} ($\text{P}=\text{O}$).



Benzofuran-2-ylbis(4-chlorophenyl)phosphine oxide (4e)

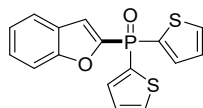
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (47.1 mg, 61%), melting point: $118 - 120\text{ }^\circ\text{C}$. ^1H NMR (400 MHz, CDCl_3) δ 7.74 (dd, $J = 12.4, 8.2\text{ Hz}$, 4H), 7.67 (d, $J = 7.8\text{ Hz}$, 1H), 7.56 – 7.46 (m, 6H), 7.41 (t, $J = 7.7\text{ Hz}$, 1H), 7.30 (t, $J = 7.5\text{ Hz}$, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.98 (d, $J_{\text{C-P}} = 8.1\text{ Hz}$), 149.25 (d, $J_{\text{C-P}} = 133.7\text{ Hz}$), 139.45 (d, $J_{\text{C-P}} = 3.6\text{ Hz}$), 133.09 (d, $J_{\text{C-P}} = 11.5\text{ Hz}$), 129.26 (d, $J_{\text{C-P}} = 113.0\text{ Hz}$), 129.17 (d, $J_{\text{C-P}} = 13.5\text{ Hz}$), 127.13, 126.44 (d, $J_{\text{C-P}} = 9.3\text{ Hz}$), 123.83, 122.56, 120.06 (d, $J_{\text{C-P}} = 17.9\text{ Hz}$), 112.16. ^{31}P NMR (162 MHz, CDCl_3) δ 15.83. HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{13}\text{Cl}_2\text{O}_2\text{P}$ $[\text{M}+\text{H}]^+$: 387.0103; found: 387.0106. IR (KBr): 1086 cm^{-1} ($\text{P}=\text{O}$).



Benzofuran-2-ylidene(naphthalen-2-yl)phosphine oxide (4f)

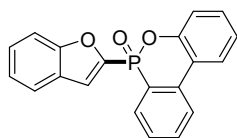
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (46 mg, 55%), melting point: $160 - 162\text{ }^\circ\text{C}$. ^1H NMR (400 MHz, CDCl_3)

δ 8.47 (d, $J = 14.6$ Hz, 2H), 7.96 – 7.86 (m, 6H), 7.84 – 7.78 (m, 2H), 7.66 (d, $J = 7.8$ Hz, 1H), 7.63 – 7.50 (m, 6H), 7.39 (t, $J = 7.8$ Hz, 1H), 7.29 (t, $J = 7.5$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 158.05 (d, $J_{\text{C-P}} = 8.0$ Hz), 150.42 (d, $J_{\text{C-P}} = 131.1$ Hz), 135.06 (d, $J_{\text{C-P}} = 2.5$ Hz), 134.05 (d, $J_{\text{C-P}} = 9.9$ Hz), 132.49 (d, $J_{\text{C-P}} = 14.0$ Hz), 129.10, 128.59 (d, $J_{\text{C-P}} = 12.7$ Hz), 128.56, 127.90, 127.55, 127.09, 126.85, 126.70 (d, $J_{\text{C-P}} = 9.1$ Hz), 126.39 (d, $J_{\text{C-P}} = 11.5$ Hz), 123.64, 122.48, 119.79 (d, $J_{\text{C-P}} = 17.7$ Hz), 112.24. ^{31}P NMR (162 MHz, CDCl_3) δ 17.82. HRMS (ESI) calcd for $\text{C}_{28}\text{H}_{19}\text{O}_2\text{P}$ $[\text{M}+\text{H}]^+$: 419.1195; found: 419.1187. IR (KBr): 1087 cm^{-1} (P=O).



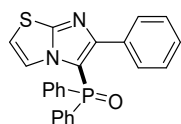
Benzofuran-2-ylidenebis(thiophen-2-yl)phosphine oxide (4g)

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (36.3 mg, 55%), melting point: $130 - 133\text{ }^\circ\text{C}$. ^1H NMR (400 MHz, CDCl_3) δ 7.81 (t, $J = 4.8$ Hz, 2H), 7.72 (dd, $J = 8.2, 3.6$ Hz, 2H), 7.68 (d, $J = 7.9$ Hz, 1H), 7.55 (d, $J = 8.4$ Hz, 1H), 7.50 (s, 1H), 7.42 (t, $J = 7.8$ Hz, 1H), 7.30 (t, $J = 7.5$ Hz, 1H), 7.27 – 7.20 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.87 (d, $J_{\text{C-P}} = 9.2$ Hz), 150.36 (d, $J_{\text{C-P}} = 148.2$ Hz), 137.18 (d, $J_{\text{C-P}} = 11.5$ Hz), 134.74 (d, $J_{\text{C-P}} = 5.9$ Hz), 132.31 (d, $J_{\text{C-P}} = 129.8$ Hz), 128.47 (d, $J_{\text{C-P}} = 15.4$ Hz), 127.12, 126.59 (d, $J_{\text{C-P}} = 9.8$ Hz), 123.73, 122.65, 118.99 (d, $J_{\text{C-P}} = 20.0$ Hz), 112.26. ^{31}P NMR (162 MHz, CDCl_3) δ 1.97. HRMS (ESI) calcd for $\text{C}_{16}\text{H}_{11}\text{O}_2\text{PS}_2$ $[\text{M}+\text{H}]^+$: 331.0011; found: 331.0005. IR (KBr): 1094 cm^{-1} (P=O).



6-(benzofuran-2-yl)dibenzo[c,e][1,2]oxaphosphinine 6-oxide (4h)

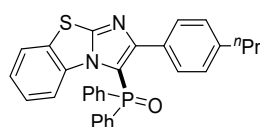
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (39.8 mg, 60%), melting point: 140 – 142 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.07 (dd, $J = 8.2, 5.5$ Hz, 1H), 8.02 (dd, $J = 8.2, 1.6$ Hz, 1H), 7.83 (dd, $J = 14.8, 7.6$ Hz, 1H), 7.74 (t, $J = 7.8$ Hz, 1H), 7.68 (d, $J = 7.8$ Hz, 1H), 7.65 (d, $J = 2.6$ Hz, 1H), 7.49 (td, $J = 7.5, 3.1$ Hz, 1H), 7.46 – 7.36 (m, 3H), 7.34 – 7.25 (m, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.87 (d, $J_{\text{C-P}} = 10.5$ Hz), 149.15 (d, $J_{\text{C-P}} = 8.2$ Hz), 148.07, 146.23, 136.10 (d, $J_{\text{C-P}} = 6.6$ Hz), 133.84 (d, $J_{\text{C-P}} = 2.5$ Hz), 131.20 (d, $J_{\text{C-P}} = 11.8$ Hz), 130.76, 128.60 (d, $J_{\text{C-P}} = 14.6$ Hz), 127.38, 126.39 (d, $J_{\text{C-P}} = 10.9$ Hz), 124.97 (d, $J_{\text{C-P}} = 16.1$ Hz), 123.74 (d, $J_{\text{C-P}} = 10.5$ Hz), 123.74 (d, $J_{\text{C-P}} = 1.0$ Hz), 123.25 (d, $J_{\text{C-P}} = 137.8$ Hz), 122.73, 121.63 (d, $J_{\text{C-P}} = 11.7$ Hz), 120.60 (d, $J_{\text{C-P}} = 6.7$ Hz), 120.13 (d, $J_{\text{C-P}} = 22.5$ Hz), 112.31. ^{31}P NMR (162 MHz, CDCl_3) δ 11.32. HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{13}\text{O}_3\text{P}$ $[\text{M}+\text{H}]^+$: 333.0675; found: 333.0682. IR (KBr): 1115 cm^{-1} (P=O).



Diphenyl(6-phenylimidazo[2,1-b]thiazol-5-yl)phosphine oxide (6a)

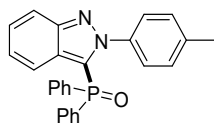
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1 – 3/1) and obtained as a white solid (60 mg, 75%), melting point: 210 – 213 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.63 (dd, $J = 12.7, 7.4$ Hz, 4H), 7.48 – 7.39 (m, 2H), 7.39 – 7.28 (m,

5H), 7.26 (d, $J = 7.3$ Hz, 2H), 7.07 (t, $J = 7.3$ Hz, 1H), 6.98 (t, $J = 7.4$ Hz, 2H), 6.78 (d, $J = 4.5$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.02 (d, $J_{\text{C-P}} = 13.4$ Hz), 153.96 (d, $J_{\text{C-P}} = 10.8$ Hz), 133.44, 132.24 (d, $J_{\text{C-P}} = 2.9$ Hz), 131.76 (d, $J_{\text{C-P}} = 10.3$ Hz), 131.46 (d, $J_{\text{C-P}} = 112.1$ Hz), 129.26, 128.59 (d, $J_{\text{C-P}} = 12.8$ Hz), 127.91, 127.48, 120.71, 113.24 (d, $J_{\text{C-P}} = 123.5$ Hz), 112.92. ^{31}P NMR (162 MHz, CDCl_3) δ 17.35. HRMS (ESI) calcd for $\text{C}_{23}\text{H}_{17}\text{N}_2\text{OPS}$ $[\text{M}+\text{H}]^+$: 401.0872; found: 401.0871. IR (KBr): 1095 cm^{-1} (P=O).



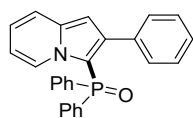
Diphenyl(2-(4-propylphenyl)benzo[d]imidazo[2,1-b]thiazol-3-yl)phosphine oxide (6b)

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1 – 3/1) and obtained as a white solid (65.9 mg, 67%), melting point: 222 – 224 $^{\circ}\text{C}$. ^1H NMR (400 MHz, CDCl_3) δ 8.52 – 8.39 (m, 1H), 7.59 – 7.49 (m, 4H), 7.31 (t, $J = 7.4$ Hz, 3H), 7.26 – 7.15 (m, 6H), 6.99 (d, $J = 8.0$ Hz, 2H), 6.71 (d, $J = 7.9$ Hz, 2H), 2.41 (t, $J = 7.6$ Hz, 2H), 1.59 – 1.47 (m, 2H), 0.91 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 158.35 (d, $J_{\text{C-P}} = 14.2$ Hz), 152.92 (d, $J_{\text{C-P}} = 10.7$ Hz), 142.13, 132.84 (d, $J_{\text{C-P}} = 95.8$ Hz), 131.95, 131.92, 131.82, 131.06 (d, $J_{\text{C-P}} = 37.2$ Hz), 129.60, 129.38, 128.36 (d, $J_{\text{C-P}} = 13.0$ Hz), 127.44, 126.28, 124.89, 123.54, 117.43, 115.27 (d, $J_{\text{C-P}} = 121.0$ Hz), 37.68, 24.32, 13.79. ^{31}P NMR (162 MHz, CDCl_3) δ 18.24. HRMS (ESI) calcd for $\text{C}_{30}\text{H}_{25}\text{N}_2\text{OPS}$ $[\text{M}+\text{H}]^+$: 493.1498; found: 493.1504. IR (KBr): 2927 (C-H), 1193 cm^{-1} (P=O).



Diphenyl(2-(p-tolyl)-2H-indazol-3-yl)phosphine oxide (6c)

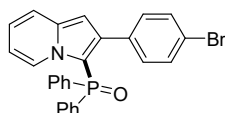
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (53 mg, 65%), melting point: 174 – 176 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.85 (d, *J* = 8.7 Hz, 1H), 7.66 (dd, *J* = 12.7, 7.7 Hz, 4H), 7.51 (t, *J* = 7.5 Hz, 2H), 7.44 – 7.33 (m, 6H), 7.30 – 7.24 (m, 1H), 7.01 (d, *J* = 7.9 Hz, 2H), 6.92 (t, *J* = 7.7 Hz, 1H), 6.33 (d, *J* = 8.7 Hz, 1H), 2.28 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 148.73 (d, *J*_{C-P} = 12.4 Hz), 139.25, 138.23, 132.19 (d, *J*_{C-P} = 2.9 Hz), 132.00 (d, *J*_{C-P} = 112.1 Hz), 131.82 (d, *J*_{C-P} = 10.0 Hz), 128.98, 128.59 (d, *J*_{C-P} = 12.7 Hz), 127.72 (d, *J*_{C-P} = 14.7 Hz), 127.62 (d, *J*_{C-P} = 114.1 Hz), 126.60, 126.36, 124.09, 120.41, 118.47, 21.17. ³¹P NMR (162 MHz, CDCl₃) δ 12.89. HRMS (ESI) calcd for C₂₆H₂₁N₂OP [M+H]⁺: 409.1464; found: 409.1462. IR (KBr): 3063 (C-H), 1118 cm⁻¹ (P=O).



Diphenyl(2-phenylindolizin-3-yl)phosphine oxide (6d)

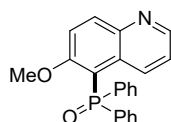
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 3/1) and obtained as a white solid (53.4 mg, 68%), melting point: 131 – 133 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.76 (d, *J* = 7.1 Hz, 1H), 7.51 – 7.41 (m, 5H), 7.38 – 7.29 (m, 2H), 7.24 – 7.13 (m, 4H), 6.99 – 6.89 (m, 4H), 6.86 (ddd, *J* = 8.5, 6.9, 1.6 Hz, 2H), 6.57 – 6.46 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 140.70 (d, *J*_{C-P} = 14.1 Hz), 137.22 (d, *J*_{C-P} = 7.2 Hz), 135.43,

132.97, 131.84 (d, J_{C-P} = 10.5 Hz), 131.67 (d, J_{C-P} = 2.9 Hz), 129.65, 128.31 (d, J_{C-P} = 12.7 Hz), 127.83 (d, J_{C-P} = 2.0 Hz), 127.13, 126.37, 121.47, 118.54, 111.63, 108.99 (d, J_{C-P} = 129.7 Hz), 103.61 (d, J_{C-P} = 9.3 Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 19.94. HRMS (ESI) calcd for $\text{C}_{26}\text{H}_{20}\text{NOP}$ $[\text{M}+\text{H}]^+$: 394.1355; found: 394.1356. IR (KBr): 1118 cm^{-1} (P=O).



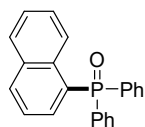
(2-(4-Bromophenyl)indolizin-3-yl)diphenylphosphine oxide (6e)

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (70.7 mg, 75%), melting point: $194 - 196\text{ }^{\circ}\text{C}$. ^1H NMR (400 MHz, CDCl_3) δ 8.73 (d, J = 7.1 Hz, 1H), 7.50 – 7.35 (m, 7H), 7.29 – 7.18 (m, 4H), 7.03 – 6.89 (m, 3H), 6.83 – 6.72 (m, 2H), 6.54 (td, J = 7.0, 1.4 Hz, 1H), 6.47 (d, J = 3.3 Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 139.19 (d, J_{C-P} = 13.8 Hz), 137.25 (d, J_{C-P} = 7.1 Hz), 134.40, 132.81, 131.84 (d, J_{C-P} = 10.4 Hz), 131.69 (d, J_{C-P} = 3.2 Hz), 131.12, 130.12, 128.44 (d, J_{C-P} = 12.6 Hz), 127.77 (d, J_{C-P} = 1.9 Hz), 121.69, 120.81, 118.58, 111.88, 109.45 (d, J_{C-P} = 129.0 Hz), 103.25 (d, J_{C-P} = 9.1 Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 19.54. HRMS (ESI) calcd for $\text{C}_{26}\text{H}_{19}\text{BrNOP}$ $[\text{M}+\text{H}]^+$: 472.0460; found: 472.0463. IR (KBr): 1112 cm^{-1} (P=O).



(6-Methoxyquinolin-5-yl)diphenylphosphine oxide (6f)

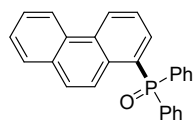
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (41.6 mg, 58%), melting point: 126 – 128 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.75 (t, *J* = 4.1 Hz, 1H), 8.05 (dd, *J* = 9.2, 1.6 Hz, 1H), 7.81 (d, *J* = 2.8 Hz, 1H), 7.78 – 7.66 (m, 4H), 7.64 – 7.57 (m, 2H), 7.51 (tdd, *J* = 8.3, 2.9, 1.3 Hz, 4H), 7.36 (dd, *J* = 9.3, 2.8 Hz, 1H), 7.12 (dd, *J* = 15.0, 4.3 Hz, 1H), 3.67 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 158.37, 146.34 (d, *J*_{C-P} = 11.8 Hz), 144.89 (d, *J*_{C-P} = 6.9 Hz), 136.60 (d, *J*_{C-P} = 95.9 Hz), 132.46 (d, *J*_{C-P} = 2.9 Hz), 131.96 (d, *J*_{C-P} = 10.0 Hz), 131.61 (d, *J*_{C-P} = 2.0 Hz), 131.33 (d, *J*_{C-P} = 105.1 Hz), 129.08 (d, *J*_{C-P} = 6.2 Hz), 128.87 (d, *J*_{C-P} = 12.4 Hz), 126.18 (d, *J*_{C-P} = 9.5 Hz), 123.07, 105.07 (d, *J*_{C-P} = 5.8 Hz), 55.52. ³¹P NMR (162 MHz, CDCl₃) δ 30.16. HRMS (ESI) calcd for C₂₂H₁₈NO₂P [M+H]⁺: 360.1148; found: 360.1157. IR (KBr): 2923 (C-H), 1114 cm⁻¹ (P=O).



Naphthalen-1-ylidiphenylphosphine oxide (6g)^[1]

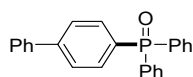
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (49.2 mg, 75%). ¹H NMR (400 MHz, CDCl₃) δ 8.59 (d, *J* = 8.5 Hz, 1H), 8.00 (d, *J* = 8.2 Hz, 1H), 7.88 (d, *J* = 8.1 Hz, 1H), 7.69 (dd, *J* = 12.1, 7.6 Hz, 4H), 7.54 (t, *J* = 7.5 Hz, 2H), 7.51 – 7.40 (m, 6H), 7.37 (dt, *J* = 7.8, 3.9 Hz, 1H), 7.33 – 7.25 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 133.88 (d, *J*_{C-P} = 9.0 Hz), 133.76 (d, *J*_{C-P} = 12.1 Hz), 133.7 (d, *J*_{C-P} = 8.1 Hz), 133.28 (d, *J*_{C-P} = 2.4 Hz), 132.25, 132.05 (d, *J*_{C-P} = 9.8 Hz), 131.89 (d, *J*_{C-P} = 2.8 Hz), 128.87 (d, *J*_{C-P} = 103.0 Hz), 128.76 (d, *J*_{C-P} = 1.3 Hz),

128.58 (d, J_{C-P} = 12.2 Hz), 127.58 (d, J_{C-P} = 5.8 Hz), 127.34, 126.49, 124.13 (d, J_{C-P} = 14.4 Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 32.43. IR (KBr): 1096 cm^{-1} (P=O).



Phenanthren-9-ylidiphenylphosphine oxide (6h) (main product)

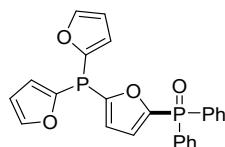
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (52.9 mg, 70%), melting point: 195 – 198 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.94 (d, J = 8.4 Hz, 1H), 8.71 (d, J = 8.3 Hz, 1H), 8.58 (d, J = 9.2 Hz, 1H), 7.87 (d, J = 7.8 Hz, 1H), 7.82 – 7.66 (m, 7H), 7.64 (d, J = 7.4 Hz, 1H), 7.57 (t, J = 7.4 Hz, 3H), 7.48 (dt, J = 7.9, 4.0 Hz, 4H). ^{13}C NMR (101 MHz, CDCl_3) δ 133.61 (d, J_{C-P} = 12.0 Hz), 133.42, 132.38, 132.15 (d, J_{C-P} = 9.8 Hz), 131.98 (d, J_{C-P} = 2.8 Hz), 131.66, 131.19 (d, J_{C-P} = 9.4 Hz), 130.19 (d, J_{C-P} = 1.2 Hz), 129.41 (d, J_{C-P} = 102.0 Hz), 128.69, 128.66 (d, J_{C-P} = 12.1 Hz), 128.44, 127.44 (d, J_{C-P} = 2.8 Hz), 127.23, 126.94, 125.45 (d, J_{C-P} = 6.7 Hz), 124.99 (d, J_{C-P} = 14.2 Hz), 122.76. ^{31}P NMR (162 MHz, CDCl_3) δ 32.98. HRMS (ESI) calcd for $\text{C}_{26}\text{H}_{19}\text{OP}$ $[\text{M}+\text{H}]^+$: 379.1246; found: 379.1254. IR (KBr): 1116 cm^{-1} (P=O).



[1,1'-Biphenyl]-4-ylidiphenylphosphine oxide (6i) (single product)^[2]

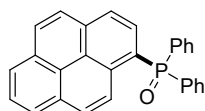
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a pale yellow solid (24.8 mg, 35%). ^1H NMR (400 MHz, CDCl_3) δ 7.78 – 7.65 (m, 8H), 7.58 (dd, J = 20.5, 7.5 Hz, 4H), 7.53 – 7.42 (m, 6H), 7.39 (t, J = 7.3 Hz, 1H). ^{13}C NMR

(101 MHz, CDCl_3) δ 144.72 (d, $J_{\text{C-P}} = 2.8$ Hz), 139.89, 132.61 (d, $J_{\text{C-P}} = 10.2$ Hz), 132.55 (d, $J_{\text{C-P}} = 104.5$ Hz), 132.11 (d, $J_{\text{C-P}} = 10.0$ Hz), 132.00 (d, $J_{\text{C-P}} = 2.7$ Hz), 131.07 (d, $J_{\text{C-P}} = 105.3$ Hz), 128.98, 128.56 (d, $J_{\text{C-P}} = 12.2$ Hz), 128.19, 127.27 (d, $J_{\text{C-P}} = 2.1$ Hz), 127.14. ^{31}P NMR (162 MHz, CDCl_3) δ 29.13. IR (KBr): 1116 cm^{-1} (P=O).



(5-(Di(furan-2-yl)phosphanyl)furan-2-yl)diphenylphosphine oxide (9a)

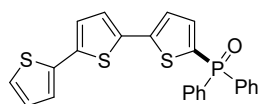
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (64.8 mg, 75%), melting point: $151 - 153\text{ }^\circ\text{C}$. ^1H NMR (400 MHz, CDCl_3) δ 7.78 – 7.68 (m, 4H), 7.61 (s, 2H), 7.57 – 7.50 (m, 2H), 7.43 (td, $J = 7.5, 3.0$ Hz, 4H), 7.12 – 7.06 (m, 1H), 6.87 – 6.63 (m, 3H), 6.39 (dt, $J = 3.1, 1.5$ Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 156.55 (dd, $J_{\text{C-P}} = 6.9, 5.4$ Hz), 152.56 (dd, $J_{\text{C-P}} = 132.2, 2.2$ Hz), 147.69 (d, $J_{\text{C-P}} = 2.8$ Hz), 147.62 (d, $J_{\text{C-P}} = 4.1$ Hz), 132.20 (d, $J_{\text{C-P}} = 2.7$ Hz), 131.65 (d, $J_{\text{C-P}} = 111.0$ Hz), 131.58 (d, $J_{\text{C-P}} = 10.5$ Hz), 128.45 (d, $J_{\text{C-P}} = 12.8$ Hz), 123.52 (dd, $J_{\text{C-P}} = 18.3, 4.8$ Hz), 122.02 (d, $J_{\text{C-P}} = 26.1$ Hz), 120.32 (dd, $J_{\text{C-P}} = 20.7, 8.4$ Hz), 110.90 (d, $J_{\text{C-P}} = 6.7$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 15.84, -76.40. HRMS (ESI) calcd for $\text{C}_{24}\text{H}_{18}\text{O}_4\text{P}_2$ $[\text{M}+\text{H}]^+$: 433.0753; found: 433.0752. IR (KBr): 1118 cm^{-1} (P=O).



Diphenyl(pyren-1-yl)phosphine oxide (9b) (main product)

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as

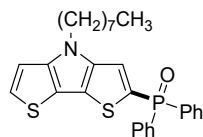
a white solid (56.3 mg, 70%), melting point: 246 – 248 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.94 (d, $J = 9.4$ Hz, 1H), 8.20 (t, $J = 7.1$ Hz, 2H), 8.15 (d, $J = 9.0$ Hz, 1H), 8.10 – 8.00 (m, 4H), 7.79 – 7.66 (m, 5H), 7.57 – 7.51 (m, 2H), 7.45 (td, $J = 7.4, 2.8$ Hz, 4H). ^{13}C NMR (101 MHz, CDCl_3) δ 134.22 (d, $J_{\text{C-P}} = 2.5$ Hz), 134.14 (d, $J_{\text{C-P}} = 8.0$ Hz), 133.85, 132.81, 132.21 (d, $J_{\text{C-P}} = 9.8$ Hz), 131.90 (d, $J_{\text{C-P}} = 2.8$ Hz), 131.18 (d, $J_{\text{C-P}} = 12.2$ Hz), 131.00, 130.40, 129.86, 128.91, 128.62 (d, $J_{\text{C-P}} = 12.2$ Hz), 127.11, 126.48, 126.31 (d, $J_{\text{C-P}} = 6.6$ Hz), 126.19, 125.62, 125.09 (d, $J_{\text{C-P}} = 10.4$ Hz), 124.38 (d, $J_{\text{C-P}} = 42.8$ Hz), 123.55 (d, $J_{\text{C-P}} = 13.7$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 32.83. HRMS (ESI) calcd for $\text{C}_{28}\text{H}_{19}\text{OP}$ $[\text{M}+\text{H}]^+$: 403.1246; found: 403.1240. IR (KBr): 1103 cm^{-1} (P=O).



[2,2':5',2''-Terthiophen]-5-ylidiphenylphosphine oxide (9c)

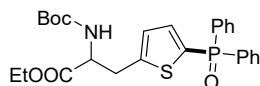
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a yellow solid (53.8 mg, 60%), melting point: 161 – 163 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.83 – 7.73 (m, 4H), 7.61 – 7.55 (m, 2H), 7.50 (td, $J = 7.4, 3.0$ Hz, 4H), 7.35 (dd, $J = 7.5, 3.7$ Hz, 1H), 7.26 – 7.22 (m, 1H), 7.21 (dd, $J = 3.8, 1.9$ Hz, 1H), 7.18 (dd, $J = 3.6, 1.1$ Hz, 1H), 7.12 (d, $J = 3.8$ Hz, 1H), 7.07 (d, $J = 3.8$ Hz, 1H), 7.02 (dd, $J = 5.1, 3.6$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 145.74 (d, $J_{\text{C-P}} = 5.3$ Hz), 137.93, 137.75 (d, $J_{\text{C-P}} = 8.9$ Hz), 136.64, 134.40 (d, $J_{\text{C-P}} = 1.7$ Hz), 133.17, 132.34 (d, $J_{\text{C-P}} = 2.8$ Hz), 132.08, 132.02 (d, $J_{\text{C-P}} = 111.2$ Hz), 131.83 (d, $J_{\text{C-P}} = 10.4$ Hz), 128.62 (d, $J_{\text{C-P}} = 12.6$ Hz), 128.01, 125.88, 125.04, 124.35 (d, $J_{\text{C-P}} = 12.8$ Hz), 124.32 (d, $J_{\text{C-P}} = 29.9$

Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 21.46. HRMS (ESI) calcd for $\text{C}_{24}\text{H}_{17}\text{OPS}_3$ $[\text{M}+\text{H}]^+$: 449.0252; found: 449.0251. IR (KBr): 1113 cm^{-1} ($\text{P}=\text{O}$).



(4-Octyl-4H-dithieno[3,2-b:2',3'-d]pyrrol-2-yl)diphenylphosphine oxide (9d)

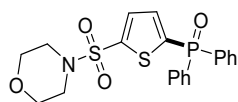
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a brown solid (66.8 mg, 68%), melting point: $113 - 115\text{ }^\circ\text{C}$. ^1H NMR (400 MHz, CDCl_3) δ 7.87 – 7.71 (m, 4H), 7.61 – 7.52 (m, 3H), 7.47 (td, $J = 7.6, 3.0\text{ Hz}$, 4H), 7.22 (d, $J = 5.3\text{ Hz}$, 1H), 6.99 (d, $J = 5.4\text{ Hz}$, 1H), 4.15 (t, $J = 7.1\text{ Hz}$, 2H), 1.82 (t, $J = 7.2\text{ Hz}$, 2H), 1.31 – 1.14 (m, 10H), 0.85 (t, $J = 6.8\text{ Hz}$, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 146.77, 145.12 (d, $J_{\text{C-P}} = 16.8\text{ Hz}$), 133.04 (d, $J_{\text{C-P}} = 109.9\text{ Hz}$), 132.11 (d, $J_{\text{C-P}} = 2.8\text{ Hz}$), 131.85 (d, $J_{\text{C-P}} = 10.4\text{ Hz}$), 128.47 (d, $J_{\text{C-P}} = 113.1\text{ Hz}$), 128.40 (d, $J_{\text{C-P}} = 12.5\text{ Hz}$), 125.79, 122.19 (d, $J_{\text{C-P}} = 6.5\text{ Hz}$), 120.18 (d, $J_{\text{C-P}} = 9.8\text{ Hz}$), 114.17 (d, $J_{\text{C-P}} = 2.3\text{ Hz}$), 110.92, 47.47, 31.67, 30.25, 29.09, 29.06, 26.93, 22.54, 14.05. ^{31}P NMR (162 MHz, CDCl_3) δ 23.05. HRMS (ESI) calcd for $\text{C}_{28}\text{H}_{30}\text{NOPS}_2$ $[\text{M}+\text{H}]^+$: 492.1579; found: 492.1577. IR (KBr): 2915 (C-H) , 1104 cm^{-1} ($\text{P}=\text{O}$).



Ethyl 2-((tert-butoxycarbonyl)amino)-3-(5-(diphenylphosphoryl)thiophen-2-yl)propanoate (9e)

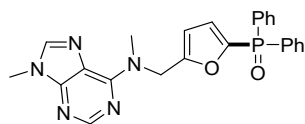
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 3/1) and obtained as

a colorless oil (69.9 mg, 70%). ^1H NMR (400 MHz, CDCl_3) δ 7.75 – 7.67 (m, 4H), 7.60 – 7.52 (m, 2H), 7.47 (td, $J = 7.5, 3.0$ Hz, 4H), 7.32 – 7.23 (m, 1H), 6.90 (s, 1H), 5.20 (d, $J = 7.8$ Hz, 1H), 4.56 (q, $J = 5.2$ Hz, 1H), 4.15 (q, $J = 7.1$ Hz, 2H), 3.56 – 3.11 (m, 2H), 1.41 (s, 9H), 1.22 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 170.73, 154.97, 147.42 (d, $J_{\text{C-P}} = 3.2$ Hz), 137.09 (d, $J_{\text{C-P}} = 9.4$ Hz), 132.91 (d, $J_{\text{C-P}} = 113.1$ Hz), 132.76 (d, $J_{\text{C-P}} = 110.1$ Hz), 132.20 (d, $J_{\text{C-P}} = 2.7$ Hz), 131.77 (d, $J_{\text{C-P}} = 10.5$ Hz), 128.50 (d, $J_{\text{C-P}} = 12.6$ Hz), 128.05 (d, $J_{\text{C-P}} = 13.0$ Hz), 80.17, 61.83, 54.11, 32.65, 28.29, 14.14. ^{31}P NMR (162 MHz, CDCl_3) δ 21.63. HRMS (ESI) calcd for $\text{C}_{26}\text{H}_{30}\text{NO}_5\text{PS}$ $[\text{M}+\text{H}]^+$: 500.1655; found: 500.1659. IR (KBr): 2977 (C-H), 1157 cm^{-1} (P=O).



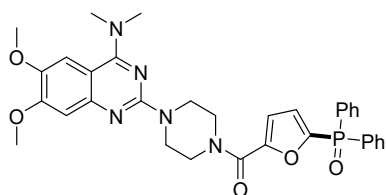
(5-(Morpholinosulfonyl)thiophen-2-yl)diphenylphosphine oxide (9f)

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 1/1) and obtained as a pale yellow solid (47.6 mg, 55%), melting point: 110 – 113 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.79 – 7.69 (m, 4H), 7.66 – 7.59 (m, 2H), 7.59 – 7.49 (m, 5H), 7.47 (dd, $J = 6.9, 3.8$ Hz, 1H), 3.84 – 3.71 (m, 4H), 3.13 – 2.98 (m, 4H). ^{13}C NMR (101 MHz, CDCl_3) δ 143.05 (d, $J_{\text{C-P}} = 4.5$ Hz), 142.34 (d, $J_{\text{C-P}} = 100.7$ Hz), 136.10 (d, $J_{\text{C-P}} = 9.0$ Hz), 132.89 (d, $J_{\text{C-P}} = 2.9$ Hz), 132.32 (d, $J_{\text{C-P}} = 85.3$ Hz), 131.72 (d, $J_{\text{C-P}} = 10.5$ Hz), 130.80, 128.89 (d, $J_{\text{C-P}} = 12.8$ Hz), 65.92, 45.93. ^{31}P NMR (162 MHz, CDCl_3) δ 20.91. HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{20}\text{NO}_4\text{PS}_2$ $[\text{M}+\text{H}]^+$: 434.0644; found: 434.0652. IR (KBr): 1112 cm^{-1} (P=O).



(5-((Methyl(9-methyl-9H-purin-6-yl)amino)methyl)furan-2-yl)diphenylphosphine oxide (9g)

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 1/2) and obtained as a white solid (48.7 mg, 55%), melting point: 126 – 128 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.39 (s, 1H), 7.72 (s, 1H), 7.68 (dd, J = 12.7, 7.4 Hz, 4H), 7.57 – 7.49 (m, 2H), 7.43 (td, J = 7.5, 3.0 Hz, 4H), 6.91 (dd, J = 3.4, 1.9 Hz, 1H), 6.53 – 6.00 (m, 1H), 5.35 (s, 2H), 3.81 (s, 3H), 3.44 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 158.60 (d, $J_{\text{C-P}}$ = 6.2 Hz), 154.37, 152.31, 147.32 (d, $J_{\text{C-P}}$ = 136.4 Hz), 139.15, 132.22 (d, $J_{\text{C-P}}$ = 2.9 Hz), 131.59 (d, $J_{\text{C-P}}$ = 10.4 Hz), 131.49 (d, $J_{\text{C-P}}$ = 111.4 Hz), 128.47 (d, $J_{\text{C-P}}$ = 12.7 Hz), 124.22 (d, $J_{\text{C-P}}$ = 18.5 Hz), 119.99, 108.89 (d, $J_{\text{C-P}}$ = 8.2 Hz), 29.78, 29.69, 19.16. ^{31}P NMR (162 MHz, CDCl_3) δ 15.96. HRMS (ESI) calcd for $\text{C}_{24}\text{H}_{22}\text{N}_5\text{O}_2\text{P}$ $[\text{M}+\text{H}]^+$: 444.1584; found: 444.1590. IR (KBr): 2922 (C-H), 1118 cm^{-1} (P=O).



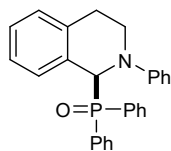
(4-(4-(Dimethylamino)-6,7-dimethoxyquinazolin-2-yl)piperazin-1-yl)(5-(diphenylphosphoryl)furan-2-yl)methanone (9h)

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 1/2) and obtained as a white solid (73.3 mg, 60%), melting point: 163 – 168 °C. ^1H NMR (400 MHz, CDCl_3)

$p = 13.0$ Hz), 122.14, 118.79, 65.56, 62.38, 60.60, 53.38, 50.02, 36.77, 20.80, 14.00.

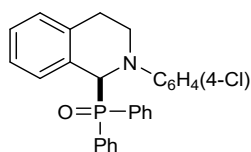
^{31}P NMR (162 MHz, CDCl_3) δ 21.70. HRMS (ESI) calcd for $\text{C}_{30}\text{H}_{29}\text{N}_2\text{O}_7\text{PS}_2$ $[\text{M}+\text{H}]^+$:

625.1227; found: 625.1230. IR (KBr): 2920 (C-H), 1104 cm^{-1} (P=O).



Diphenyl(2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphine oxide (11a)^[3]

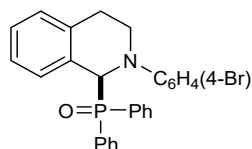
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (62.2 mg, 76%). ^1H NMR (400 MHz, CDCl_3) δ 7.80 (ddt, $J = 10.3$, 6.9, 1.4 Hz, 2H), 7.70 (ddt, $J = 10.6$, 7.0, 1.4 Hz, 2H), 7.56 – 7.50 (m, 1H), 7.44 (qd, $J = 7.5$, 2.2 Hz, 3H), 7.33 (td, $J = 7.7$, 3.2 Hz, 2H), 7.14 (ddd, $J = 8.8$, 7.2, 2.3 Hz, 3H), 7.06 (d, $J = 7.5$ Hz, 1H), 6.93 (t, $J = 7.5$ Hz, 1H), 6.78 (dd, $J = 16.7$, 7.9 Hz, 3H), 6.64 (d, $J = 7.8$, 1.5 Hz, 1H), 5.56 (d, $J = 10.6$ Hz, 1H), 4.03 (ddd, $J = 13.4$, 10.1, 4.7 Hz, 1H), 3.64 – 3.51 (m, 1H), 2.88 – 2.77(m, 1H), 2.72 – 2.59 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 149.93 (d, $J_{\text{C-P}} = 7.9$ Hz), 136.84 (d, $J_{\text{C-P}} = 4.3$ Hz), 132.22 (d, $J_{\text{C-P}} = 8.5$ Hz), 131.21 (d, $J_{\text{C-P}} = 85.6$ Hz), 132.16 (d, $J_{\text{C-P}} = 99.3$ Hz), 131.91 (d, $J_{\text{C-P}} = 2.8$ Hz), 131.69, 131.60, 129.86, 129.21 (d, $J_{\text{C-P}} = 2.2$ Hz), 129.09, 128.41 (d, $J_{\text{C-P}} = 11.2$ Hz), 128.25 (d, $J_{\text{C-P}} = 11.3$ Hz), 127.73 (d, $J_{\text{C-P}} = 3.2$ Hz), 127.38 (d, $J_{\text{C-P}} = 2.9$ Hz), 125.49 (d, $J_{\text{C-P}} = 2.6$ Hz), 119.50, 116.70, 61.89 (d, $J_{\text{C-P}} = 79.8$ Hz), 45.10, 25.56. ^{31}P NMR (162 MHz, CDCl_3) δ 30.74. IR (KBr): 2921 (C-H), 1064 cm^{-1} (P=O).



(2-(4-chlorophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)diphenylphosphine oxide

(11b)^[3]

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (48,7 mg, 55%). ¹H NMR (400 MHz, CDCl₃) δ 7.83 – 7.72 (m, 2H), 7.73 – 7.62 (m, 2H), 7.55 (t, *J* = 7.3 Hz, 1H), 7.46 (dt, *J* = 7.3, 3.7 Hz, 3H), 7.35 (td, *J* = 7.6, 2.9 Hz, 2H), 7.16 (t, *J* = 7.4 Hz, 1H), 7.11 – 7.01 (m, 3H), 6.94 (t, *J* = 7.4 Hz, 1H), 6.74 – 6.67 (m, 2H), 6.61 (d, *J* = 7.8 Hz, 1H), 5.47 (d, *J* = 10.0 Hz, 1H), 4.04 (ddd, *J* = 14.1, 9.7, 4.9 Hz, 1H), 3.50 (dt, *J* = 13.1, 4.9 Hz, 1H), 2.91 – 2.56 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 148.57 (d, *J*_{C-P} = 7.2 Hz), 136.70 (d, *J*_{C-P} = 4.2 Hz), 132.24 (d, *J*_{C-P} = 8.5 Hz), 132.07 (d, *J*_{C-P} = 2.8 Hz), 132.00 (d, *J*_{C-P} = 95.0 Hz), 131.82 (d, *J*_{C-P} = 2.8 Hz), 131.62 (d, *J*_{C-P} = 8.8 Hz), 130.92 (d, *J*_{C-P} = 90.9 Hz), 129.65, 129.23 (d, *J*_{C-P} = 2.2 Hz), 128.98, 128.43 (d, *J*_{C-P} = 23.9 Hz), 128.43 (d, *J*_{C-P} = 1.3 Hz), 127.75 (d, *J*_{C-P} = 3.3 Hz), 127.61 (d, *J*_{C-P} = 2.9 Hz), 125.67 (d, *J*_{C-P} = 2.5 Hz), 124.26, 117.73, 62.15 (d, *J*_{C-P} = 78.9 Hz), 45.25, 25.77. ³¹P NMR (162 MHz, CDCl₃) δ 30.53. IR (KBr): 2919 (C-H), 1086 cm⁻¹ (P=O).

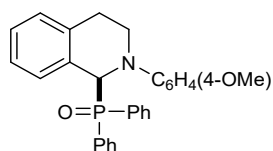


(2-(4-Bromophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)diphenylphosphine oxide

(11c)^[3]

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as

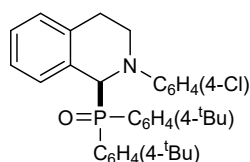
a white solid (58.4 mg, 60%). ^1H NMR (400 MHz, CDCl_3) δ 7.76 (t, $J = 9.1$ Hz, 2H), 7.73 – 7.63 (m, 2H), 7.55 (t, $J = 7.5$ Hz, 1H), 7.46 (t, $J = 7.5$ Hz, 3H), 7.35 (t, $J = 6.7$ Hz, 2H), 7.20 (d, $J = 8.4$ Hz, 2H), 7.15 (d, $J = 7.5$ Hz, 1H), 7.08 (d, $J = 7.6$ Hz, 1H), 6.94 (t, $J = 7.6$ Hz, 1H), 6.66 (d, $J = 8.5$ Hz, 2H), 6.61 (d, $J = 7.9$ Hz, 1H), 5.48 (d, $J = 9.9$ Hz, 1H), 4.03 (ddd, $J = 13.8, 9.5, 4.9$ Hz, 1H), 3.59 – 3.38 (m, 1H), 2.81 (dt, $J = 15.4, 7.3$ Hz, 1H), 2.75 – 2.65 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 148.97 (d, $J_{\text{C-P}} = 7.1$ Hz), 136.67 (d, $J_{\text{C-P}} = 4.1$ Hz), 132.24 (d, $J_{\text{C-P}} = 8.4$ Hz), 132.09 (d, $J_{\text{C-P}} = 2.7$ Hz), 131.89 (d, $J_{\text{C-P}} = 111.1$), 131.88, 131.85 (d, $J_{\text{C-P}} = 2.7$ Hz), 131.61 (d, $J_{\text{C-P}} = 8.8$ Hz), 130.96 (d, $J_{\text{C-P}} = 107.1$), 129.65, 129.22 (d, $J_{\text{C-P}} = 2.2$ Hz), 128.51 (d, $J_{\text{C-P}} = 12.1$), 128.39 (d, $J_{\text{C-P}} = 12.1$), 127.75 (d, $J_{\text{C-P}} = 3.3$ Hz), 127.64 (d, $J_{\text{C-P}} = 2.9$ Hz), 125.68 (d, $J_{\text{C-P}} = 2.5$ Hz), 118.04, 111.51, 62.13 (d, $J_{\text{C-P}} = 78.7$ Hz), 45.10, 25.83. ^{31}P NMR (162 MHz, CDCl_3) δ 30.71. IR (KBr): 2915 (C-H), 1090 cm^{-1} (P=O).



(2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)diphenylphosphine oxide (11d)^[3]

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (61.5 mg, 70%). ^1H NMR (400 MHz, CDCl_3) δ 7.77 (t, $J = 9.1$ Hz, 2H), 7.71 (t, $J = 9.2$ Hz, 2H), 7.52 (t, $J = 7.4$ Hz, 1H), 7.44 (p, $J = 6.8, 5.8$ Hz, 3H), 7.35 (dt, $J = 8.2, 4.1$ Hz, 2H), 7.14 (t, $J = 7.5$ Hz, 1H), 7.07 (d, $J = 7.6$ Hz, 1H), 6.93 (t, $J = 7.5$ Hz, 1H), 6.76 (d, $J = 8.8$ Hz, 2H), 6.71 (d, $J = 8.8$ Hz, 2H), 6.61 (d, $J = 7.8$ Hz, 1H),

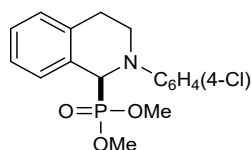
5.36 (d, $J = 11.8$ Hz, 1H), 3.98 (ddd, $J = 14.5, 11.0, 4.4$ Hz, 1H), 3.71 (s, 3H), 3.47 – 3.31 (m, 1H), 2.75 (dq, $J = 16.0, 5.5, 4.9$ Hz, 1H), 2.55 (dd, $J = 16.4, 3.8$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 154.02, 144.60 (d, $J_{\text{C-P}} = 11.1$ Hz), 137.01 (d, $J_{\text{C-P}} = 4.0$ Hz), 132.45 (d, $J_{\text{C-P}} = 96.0$ Hz), 132.15 (d, $J_{\text{C-P}} = 8.0$ Hz), 131.85 (d, $J_{\text{C-P}} = 90.9$ Hz), 131.80, 131.74 (d, $J_{\text{C-P}} = 7.1$ Hz), 131.53 (d, $J_{\text{C-P}} = 3.0$ Hz), 129.69, 129.36 (d, $J_{\text{C-P}} = 2.0$ Hz), 128.38 (d, $J_{\text{C-P}} = 11.1$ Hz), 128.21 (d, $J_{\text{C-P}} = 11.1$ Hz), 127.77 (d, $J_{\text{C-P}} = 3.0$ Hz), 127.21 (d, $J_{\text{C-P}} = 3.0$ Hz), 125.44 (d, $J_{\text{C-P}} = 3.0$ Hz), 120.35, 114.41, 62.09 (d, $J_{\text{C-P}} = 81.8$ Hz), 55.49, 46.79, 24.76. ^{31}P NMR (162 MHz, CDCl_3) δ 31.06. IR (KBr): 3048 (C-H), 1109 cm^{-1} (P=O).



Bis(4-(tert-butyl)phenyl)(2-(4-chlorophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphine oxide (11e)

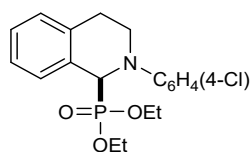
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (75.5 mg, 68%), melting point: 233 – 235 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.69 – 7.64 (m, 2H), 7.61 – 7.56 (m, 2H), 7.46 (d, $J = 7.9$ Hz, 2H), 7.36 – 7.30 (m, 2H), 7.15 (t, $J = 7.3$ Hz, 1H), 7.08 (d, $J = 7.5$ Hz, 1H), 7.03 (d, $J = 8.7$ Hz, 2H), 6.92 (t, $J = 7.4$ Hz, 1H), 6.66 (d, $J = 8.7$ Hz, 2H), 6.61 (d, $J = 7.6$ Hz, 1H), 5.45 (d, $J = 9.2$ Hz, 1H), 4.05 (ddd, $J = 13.7, 8.7, 5.5$ Hz, 1H), 3.50 (dt, $J = 12.2, 5.3$ Hz, 1H), 2.90 – 2.68 (m, 2H), 1.34 (s, 9H), 1.27 (s, 9H). ^{13}C NMR (101 MHz, CDCl_3) δ 155.58 (d, $J_{\text{C-P}} = 2.9$ Hz), 155.25 (d, $J_{\text{C-P}} = 2.7$ Hz), 148.46 (d, $J_{\text{C-P}} = 6.2$ Hz), 136.70 (d, $J_{\text{C-P}} = 4.0$ Hz),

132.09 (d, J_{C-P} = 8.8 Hz), 131.53 (d, J_{C-P} = 9.1 Hz), 130.06, 129.08 (d, J_{C-P} = 2.2 Hz), 128.83, 127.85 (d, J_{C-P} = 3.4 Hz), 127.48 (d, J_{C-P} = 2.9 Hz), 125.53 (d, J_{C-P} = 3.0 Hz), 125.50, 125.38, 125.26, 123.75, 117.33, 62.16 (d, J_{C-P} = 77.6 Hz), 44.95, 35.06, 34.95, 31.18, 31.11, 26.08. ^{31}P NMR (162 MHz, CDCl_3) δ 31.00. HRMS (ESI) calcd for $\text{C}_{35}\text{H}_{39}\text{ClNOP}$ $[\text{M}+\text{H}]^+$: 556.2531; found: 556.2536. IR (KBr): 3048 (C-H), 1089 cm^{-1} (P=O).



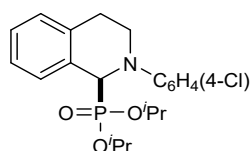
Dimethyl (2-(4-chlorophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (11f)^[4]

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 20/1) and obtained as a white solid (52 mg, 75%). ^1H NMR (400 MHz, CDCl_3) δ 7.34 (d, J = 7.2 Hz, 1H), 7.27 – 7.11 (m, J = 6.5 Hz, 5H), 6.89 (d, J = 9.0 Hz, 2H), 5.12 (d, J = 19.2 Hz, 1H), 3.97 (ddd, J = 12.7, 8.2, 4.7 Hz, 1H), 3.63 (dd, J = 10.5, 8.0 Hz, 6H), 3.54 (dt, J = 11.9, 5.7 Hz, 1H), 3.19 – 3.07 (m, 1H), 3.04 – 2.91 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 147.73 (d, J_{C-P} = 5.3 Hz), 136.25 (d, J_{C-P} = 5.5 Hz), 130.12, 129.01, 128.77 (d, J_{C-P} = 2.7 Hz), 127.94 (d, J_{C-P} = 4.8 Hz), 127.74 (d, J_{C-P} = 3.5 Hz), 126.20 (d, J_{C-P} = 2.9 Hz), 123.34, 115.68, 58.72 (d, J_{C-P} = 159.9 Hz), 53.87 (d, J_{C-P} = 7.2 Hz), 53.04 (d, J_{C-P} = 7.7 Hz), 43.77, 26.81. ^{31}P NMR (162 MHz, CDCl_3) δ 24.06. IR (KBr): 1061 cm^{-1} (P=O).



Diethyl (2-(4-chlorophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (11g)^[4]

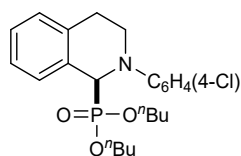
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 20/1) and obtained as a white solid (46.2 mg, 61%). ¹H NMR (400 MHz, CDCl₃) δ 7.38 – 7.32 (m, 1H), 7.25 – 7.13 (m, 5H), 6.95 – 6.84 (m, 2H), 5.10 (d, *J* = 19.2 Hz, 1H), 4.11 – 3.84 (m, 5H), 3.58 – 3.50 (m, 1H), 3.19 – 3.09 (m, 1H), 3.03 – 2.90 (m, 1H), 1.24 (t, *J* = 7.1 Hz, 3H), 1.14 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 147.92 (d, *J*_{C-P} = 5.1 Hz), 136.31 (d, *J*_{C-P} = 5.5 Hz), 130.41, 128.90, 128.69 (d, *J*_{C-P} = 2.8 Hz), 128.13 (d, *J*_{C-P} = 4.8 Hz), 127.65 (d, *J*_{C-P} = 3.5 Hz), 126.02 (d, *J*_{C-P} = 2.9 Hz), 123.14, 115.73, 63.30 (d, *J*_{C-P} = 7.4 Hz), 62.47 (d, *J*_{C-P} = 7.7 Hz), 58.84 (d, *J*_{C-P} = 159.7 Hz), 43.74, 26.92, 16.48 (d, *J*_{C-P} = 5.4 Hz), 16.39 (d, *J*_{C-P} = 5.8 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 21.86. IR (KBr): 2979 (C-H), 1020 cm⁻¹ (P=O).



Diisopropyl (2-(4-chlorophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (11h)^[5]

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 20/1) and obtained as a white solid (44.8 mg, 55%). ¹H NMR (400 MHz, CDCl₃) δ 7.38 (d, *J* = 6.7 Hz, 1H), 7.24 – 7.05 (m, 5H), 6.87 (d, *J* = 8.7 Hz, 2H), 5.06 (d, *J* = 20.3 Hz, 1H), 4.72 – 4.46 (m, 2H), 4.09 – 3.93 (m, 1H), 3.56 (dt, *J* = 12.0, 5.5 Hz, 1H), 3.16 – 3.04 (m, 1H),

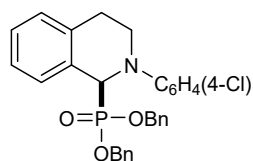
3.01 – 2.88 (m, 1H), 1.28 (t, $J = 5.6$ Hz, 6H), 1.15 (d, $J = 6.2$ Hz, 3H), 0.94 (d, $J = 6.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 148.05 (d, $J_{\text{C-P}} = 5.8$ Hz), 136.24 (d, $J_{\text{C-P}} = 5.4$ Hz), 130.61, 128.74, 128.63 (d, $J_{\text{C-P}} = 2.7$ Hz), 128.39 (d, $J_{\text{C-P}} = 4.7$ Hz), 127.45 (d, $J_{\text{C-P}} = 3.6$ Hz), 125.75 (d, $J_{\text{C-P}} = 2.8$ Hz), 122.84, 115.92, 72.24 (d, $J_{\text{C-P}} = 7.7$ Hz), 70.98 (d, $J_{\text{C-P}} = 8.1$ Hz), 58.81 (d, $J_{\text{C-P}} = 161.4$ Hz), 43.66, 26.71, 24.55 (d, $J_{\text{C-P}} = 3.1$ Hz), 24.14 (d, $J_{\text{C-P}} = 3.2$ Hz), 23.72 (d, $J_{\text{C-P}} = 5.7$ Hz), 23.34 (d, $J_{\text{C-P}} = 5.4$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 20.56. IR (KBr): 2976 (C-H), 1104 cm^{-1} (P=O).



Dibutyl (2-(4-chlorophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (11i)

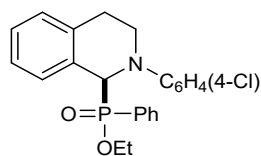
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 20/1) and obtained as a white solid (39.2 mg, 45%), melting point: 155 – 157 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.35 (d, $J = 6.8$ Hz, 1H), 7.19 (t, $J = 8.5$ Hz, 5H), 6.88 (d, $J = 8.7$ Hz, 2H), 5.12 (d, $J = 19.1$ Hz, 1H), 4.05 – 3.91 (m, 3H), 3.90 – 3.81 (m, 1H), 3.81 – 3.72 (m, 1H), 3.54 (dt, $J = 11.9, 5.7$ Hz, 1H), 3.18 – 3.08 (m, 1H), 2.96 (dt, $J = 14.9, 6.6$ Hz, 1H), 1.59 – 1.50 (m, 2H), 1.49 – 1.40 (m, 2H), 1.40 – 1.07 (m, 5H), 0.88 (t, $J = 7.4$ Hz, 3H), 0.82 (t, $J = 7.4$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 147.81 (d, $J_{\text{C-P}} = 4.9$ Hz), 136.19 (d, $J_{\text{C-P}} = 5.4$ Hz), 130.43, 128.82, 128.62 (d, $J_{\text{C-P}} = 2.8$ Hz), 128.06 (d, $J_{\text{C-P}} = 4.8$ Hz), 127.56 (d, $J_{\text{C-P}} = 3.5$ Hz), 125.95 (d, $J_{\text{C-P}} = 2.9$ Hz), 123.00, 115.63, 66.82 (d, $J_{\text{C-P}} = 7.6$ Hz), 66.02 (d, $J_{\text{C-P}} = 7.8$ Hz), 58.70 (d, $J_{\text{C-P}} = 158.8$ Hz), 43.60, 32.54 (d, $J_{\text{C-P}} = 6.2$ Hz), 32.47 (d, $J_{\text{C-P}} = 6.0$ Hz), 26.92, 18.65, 18.60, 13.56, 13.52. ^{31}P NMR (162

MHz, CDCl₃) δ 22.03. HRMS (ESI) calcd for C₂₃H₃₁ClNO₃P [M+H]⁺: 436.1803; found: 436.1807. IR (KBr): 2958 (C-H), 1058 cm⁻¹ (P=O).



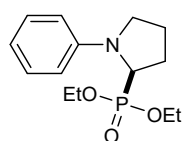
Dibenzyl (2-(4-chlorophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (11j)^[6]

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 20/1) and obtained as a white solid (50.3 mg, 50%). ¹H NMR (400 MHz, CDCl₃) δ 7.41 – 7.04 (m, 16H), 6.89 – 6.79 (m, 2H), 5.18 (d, *J* = 18.8 Hz, 1H), 5.00 – 4.82 (m, 3H), 4.75 (dd, *J* = 11.6, 7.9 Hz, 1H), 3.99 – 3.89 (m, 1H), 3.51 (dt, *J* = 12.0, 5.8 Hz, 1H), 3.17 – 3.06 (m, 1H), 3.01 – 2.89 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 147.73 (d, *J*_{C-P} = 4.8 Hz), 136.35 (d, *J*_{C-P} = 5.5 Hz), 136.11, 136.11 (d, *J*_{C-P} = 11.3 Hz), 130.10, 128.93, 128.78 (d, *J*_{C-P} = 2.8 Hz), 128.51, 128.43, 128.39, 128.32, 128.19 (d, *J*_{C-P} = 4.8 Hz), 128.08, 128.06, 127.78 (d, *J*_{C-P} = 3.6 Hz), 126.14 (d, *J*_{C-P} = 2.9 Hz), 123.28, 115.83, 68.67 (d, *J*_{C-P} = 7.3 Hz), 67.87 (d, *J*_{C-P} = 7.9 Hz), 59.01 (d, *J*_{C-P} = 158.1 Hz), 43.73, 26.98. ³¹P NMR (162 MHz, CDCl₃) δ 22.71. IR (KBr): 1046 cm⁻¹ (P=O).



Ethyl (2-(4-chlorophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)(phenyl)phosphinate (11k)

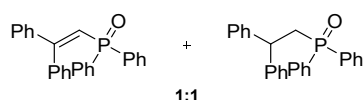
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 20/1) and obtained as a white solid (32.9 mg, 40%), melting point: 136 –138 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.77 – 7.65 (m, 2H), 7.52 – 7.45 (m, 1H), 7.44 – 7.31 (m, 3H), 7.25 – 7.15 (m, 2H), 7.12 (q, *J* = 5.8 Hz, 1H), 7.08 – 6.87 (m, 2H), 6.75 – 6.45 (m, 2H), 5.11 (d, *J* = 14.9 Hz, 1H), 4.17 – 3.90 (m, 3H), 3.49 (dt, *J* = 12.9, 5.1 Hz, 1H), 2.92 – 2.82 (m, 1H), 2.82 – 2.70 (m, 1H), 1.31 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 148.07 (d, *J*_{C-P} = 6.8 Hz), 136.29 (d, *J*_{C-P} = 4.7 Hz), 132.37, 132.28 (d, *J*_{C-P} = 9.5 Hz), 130.19 (d, *J*_{C-P} = 121.3 Hz), 129.89, 128.98 (d, *J*_{C-P} = 2.3 Hz), 128.70, 128.51 (d, *J*_{C-P} = 3.9 Hz), 128.39 (d, *J*_{C-P} = 12.2 Hz), 127.51 (d, *J*_{C-P} = 3.1 Hz), 125.86 (d, *J*_{C-P} = 2.6 Hz), 123.41, 116.50, 61.41 (d, *J*_{C-P} = 7.3 Hz), 61.11 (d, *J*_{C-P} = 113.7 Hz), 43.89, 26.06, 16.54 (d, *J*_{C-P} = 6.0 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 37.51. HRMS (ESI) calcd for C₂₃H₂₃ClNO₂P [M+H]⁺: 412.1228; found: 412.1228. IR (KBr): 2920 (C-H), 1024 cm⁻¹ (P=O).



Diethyl (1-phenylpyrrolidin-2-yl)phosphonate (11l)^[7]

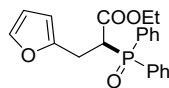
According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 20/1) and obtained as a pale yellow oil (32.9 mg, 40%). ¹H NMR (400 MHz, CDCl₃) δ 7.29 – 7.15 (m, 2H), 6.81 (d, *J* = 8.2 Hz, 2H), 6.74 (t, *J* = 7.3 Hz, 1H), 4.17 – 4.01 (m, 4H), 3.97 – 3.86 (m, 1H), 3.59 (t, *J* = 8.4 Hz, 1H), 3.24 – 3.11 (m, 1H), 2.44 – 2.31 (m, 2H), 2.14 – 1.99

(m, 2H), 1.31 (t, $J = 7.1$ Hz, 3H), 1.18 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 147.73, 128.77, 116.95, 113.08, 62.44 (d, $J_{\text{C-P}} = 7.0$ Hz), 61.86 (d, $J_{\text{C-P}} = 7.6$ Hz), 56.38 (d, $J_{\text{C-P}} = 169.1$ Hz), 49.65 (d, $J_{\text{C-P}} = 2.3$ Hz), 27.76, 24.21, 16.46 (d, $J_{\text{C-P}} = 4.7$ Hz), 16.41 (d, $J_{\text{C-P}} = 4.8$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 25.63. IR (KBr): 2977 (C-H), 1020 cm^{-1} (P=O).



(2,2-diphenylvinyl)diphenylphosphine oxide (13) and (2,2-diphenylethyl)diphenylphosphine oxide (13')^[8]

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 5/1) and obtained as a white solid (68.4 mg, 90%). ^1H NMR (400 MHz, CDCl_3) δ 7.73 – 7.64 (m, 4H), 7.61 – 7.52 (m, 4H), 7.32 (dtd, $J = 20.2, 11.9, 10.1, 6.9$ Hz, 19H), 7.26 – 7.19 (m, 4H), 7.19 (d, $J = 7.3$ Hz, 5H), 7.14 – 6.99 (m, 9H), 6.79 (d, $J = 18.2$ Hz, 1H), 4.71 (dt, $J = 11.7, 7.1$ Hz, 1H), 3.10 (dd, $J = 11.0, 7.2$ Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 161.87, 143.77 (d, $J_{\text{C-P}} = 7.6$ Hz), 141.76 (d, $J_{\text{C-P}} = 16.1$ Hz), 137.90 (d, $J_{\text{C-P}} = 6.7$ Hz), 134.26 (d, $J_{\text{C-P}} = 105.9$ Hz), 133.15 (d, $J_{\text{C-P}} = 98.8$ Hz), 131.20 (d, $J_{\text{C-P}} = 2.8$ Hz), 130.97 (d, $J_{\text{C-P}} = 2.8$ Hz), 130.74 (d, $J_{\text{C-P}} = 9.5$ Hz), 130.52 (d, $J_{\text{C-P}} = 9.3$ Hz), 130.20, 128.98 (d, $J_{\text{C-P}} = 90.1$ Hz), 128.29, 128.23 (d, $J_{\text{C-P}} = 10.0$ Hz), 128.16 (d, $J_{\text{C-P}} = 12.0$ Hz), 127.72, 126.89 (d, $J_{\text{C-P}} = 117.3$ Hz), 120.44 (d, $J_{\text{C-P}} = 103.5$ Hz), 44.37 (d, $J_{\text{C-P}} = 2.6$ Hz), 36.41 (d, $J_{\text{C-P}} = 70.5$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 29.49, 18.70.



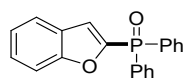
Ethyl 2-(diphenylphosphoryl)-3-(furan-2-yl)propanoate (15)

According to the general procedure, the product was purified by chromatography on silica gel eluting with petroleum ether and ethylacetate (PE/EA = 10/1) and obtained as a white solid (44.9 mg, 61%), melting point: 117 – 119 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.93 – 7.83 (m, 4H), 7.58 – 7.46 (m, 6H), 7.25 (dd, *J* = 1.9, 0.8 Hz, 1H), 6.20 (dd, *J* = 3.2, 1.9 Hz, 1H), 6.00 (dd, *J* = 3.2, 0.9 Hz, 1H), 3.93 – 3.77 (m, 3H), 3.42 (ddd, *J* = 15.7, 11.6, 6.0 Hz, 1H), 3.13 (ddd, *J* = 15.5, 8.9, 3.1 Hz, 1H), 0.87 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 168.69 (d, *J*_{C-P} = 2.9 Hz), 151.93 (d, *J*_{C-P} = 14.9 Hz), 141.61, 132.26, 132.23, 131.59 (d, *J*_{C-P} = 9.4 Hz), 131.20 (d, *J*_{C-P} = 9.4 Hz), 131.13 (d, *J*_{C-P} = 67.0 Hz), 130.12 (d, *J*_{C-P} = 67.2 Hz), 128.74 (d, *J*_{C-P} = 12.0 Hz), 128.44 (d, *J*_{C-P} = 12.2 Hz), 110.27, 106.65, 61.35, 47.90 (d, *J*_{C-P} = 57.6 Hz), 25.18 (d, *J*_{C-P} = 1.5 Hz), 13.59. ³¹P NMR (162 MHz, CDCl₃) δ 28.87. HRMS (ESI) calcd for C₂₁H₂₁O₄P [M+H]⁺: 369.1250; found: 369.1258. IR (KBr): 2918 (C-H), 1187 cm⁻¹ (P=O).

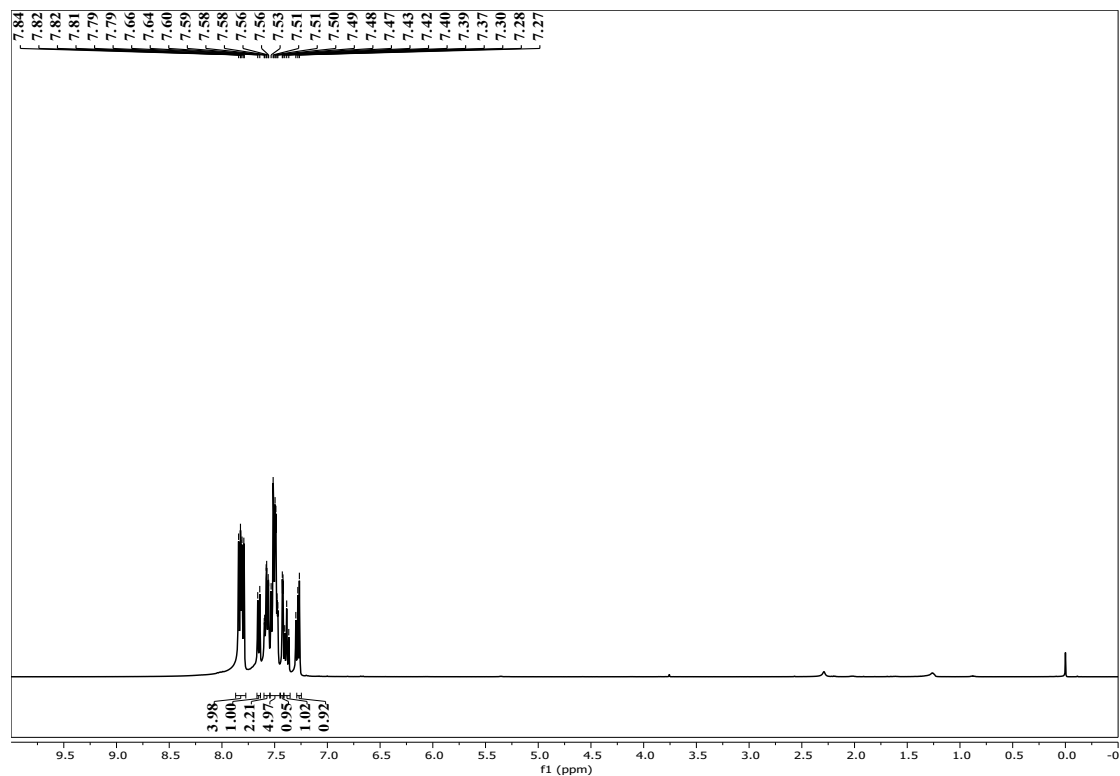
References

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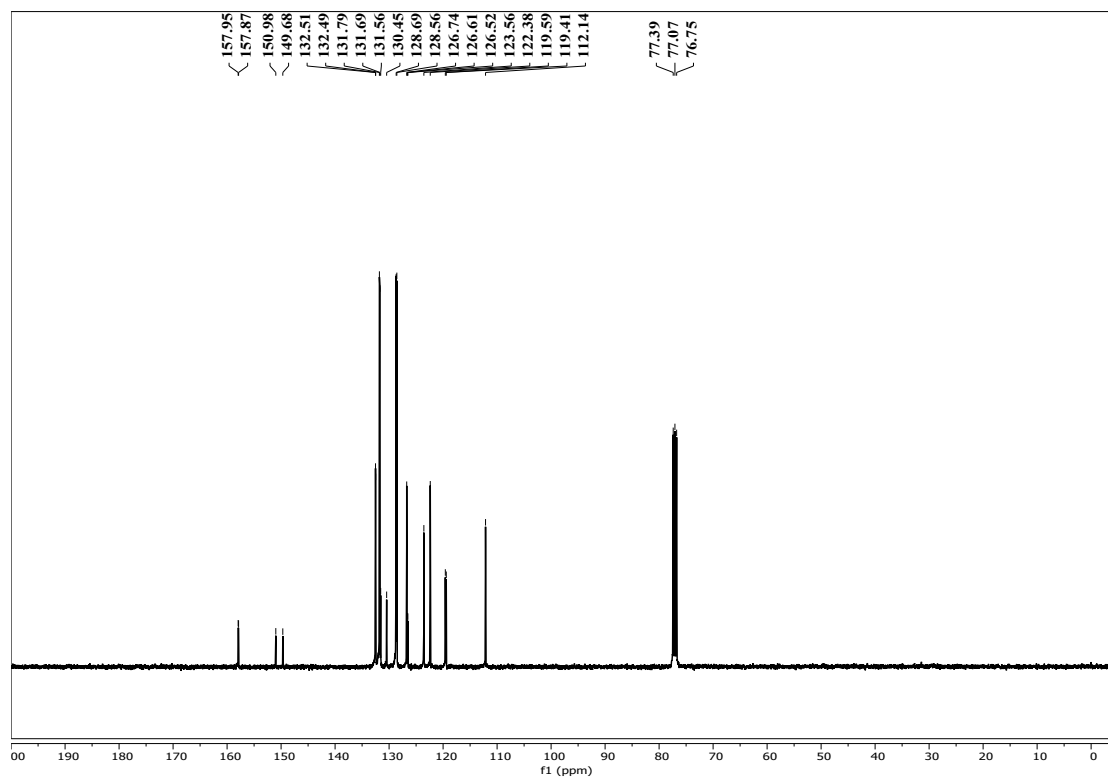
NMR spectra of products



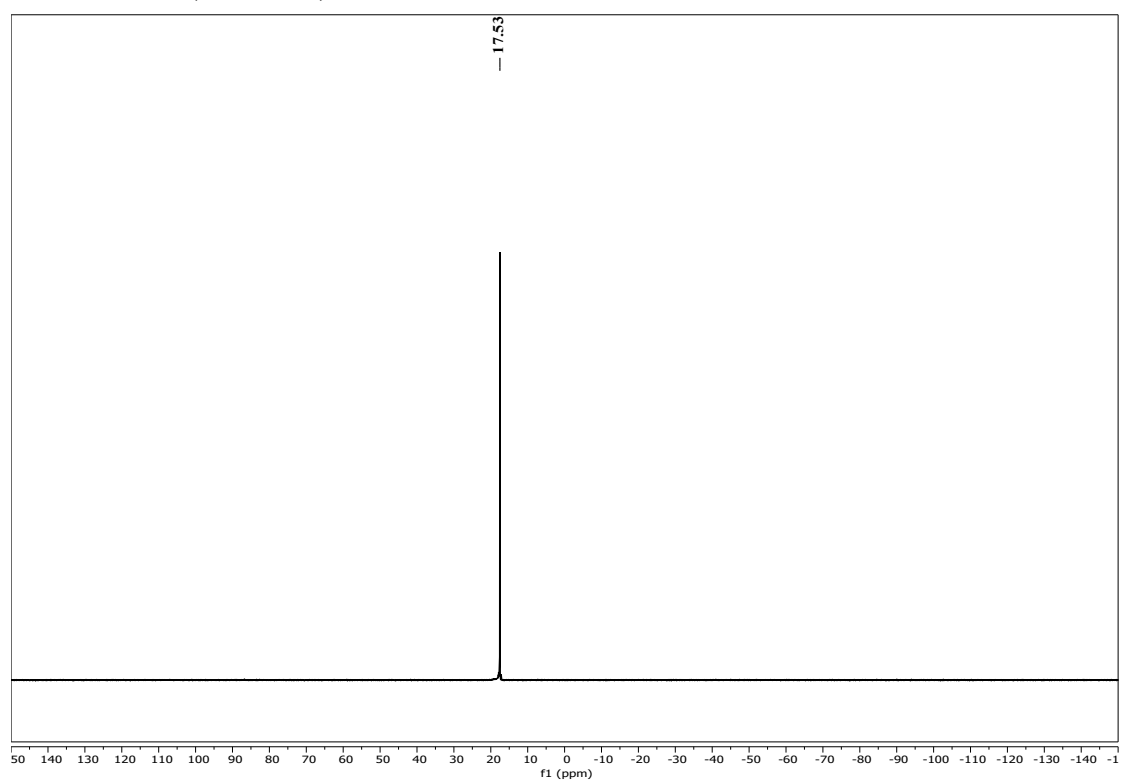
3a, ^1H NMR (400 MHz), CDCl_3

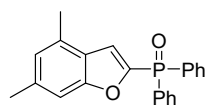


3a, ^{13}C NMR (101 MHz), CDCl_3

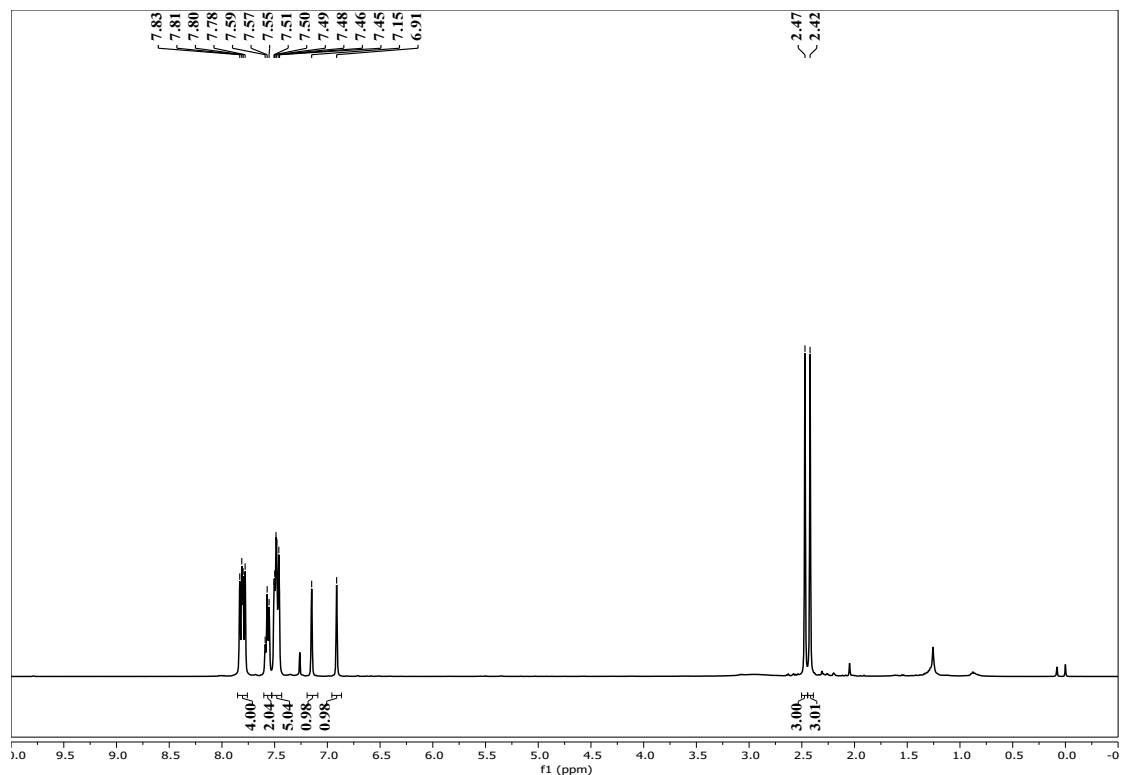


3a, ^{31}P NMR (162 MHz), CDCl_3

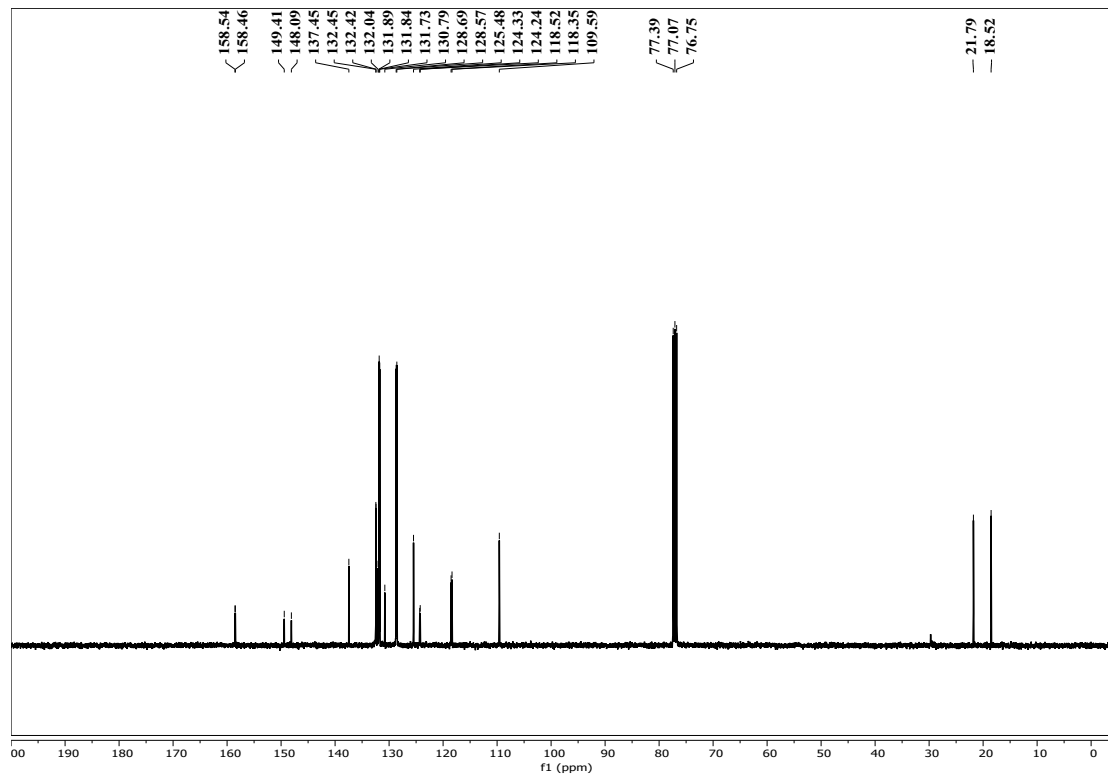




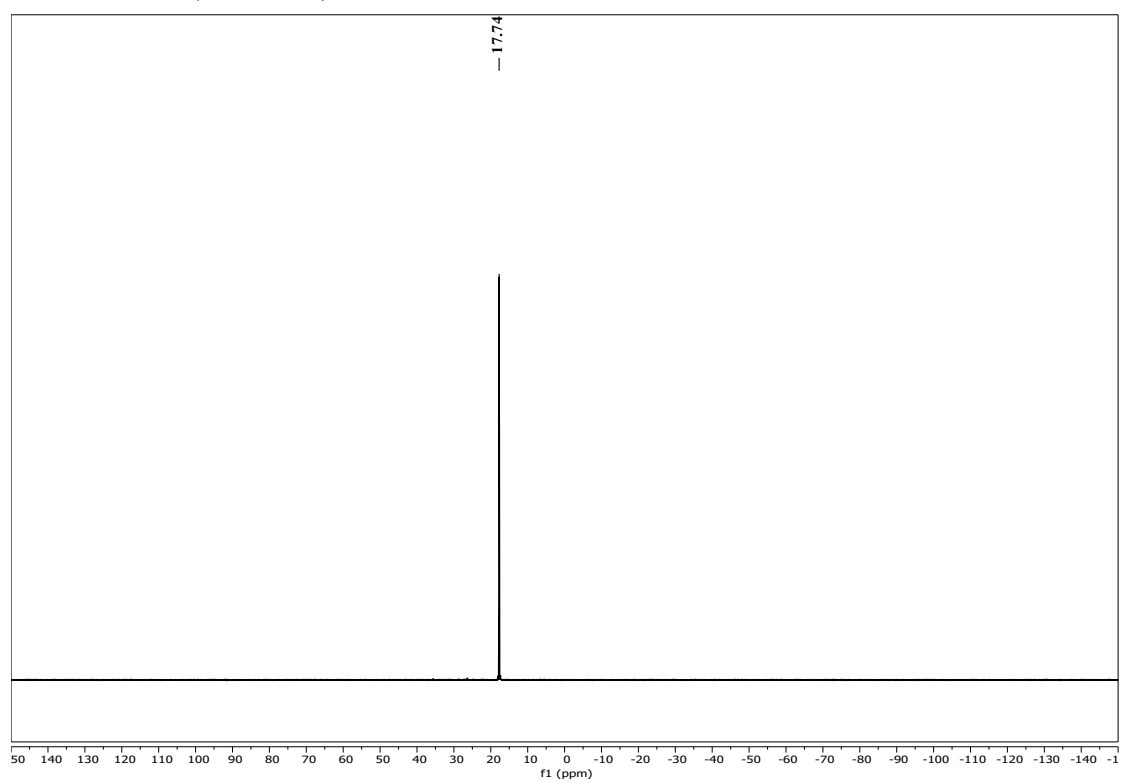
3b, ^1H NMR (400 MHz), CDCl_3

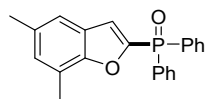


3b, ^{13}C NMR (101 MHz), CDCl_3

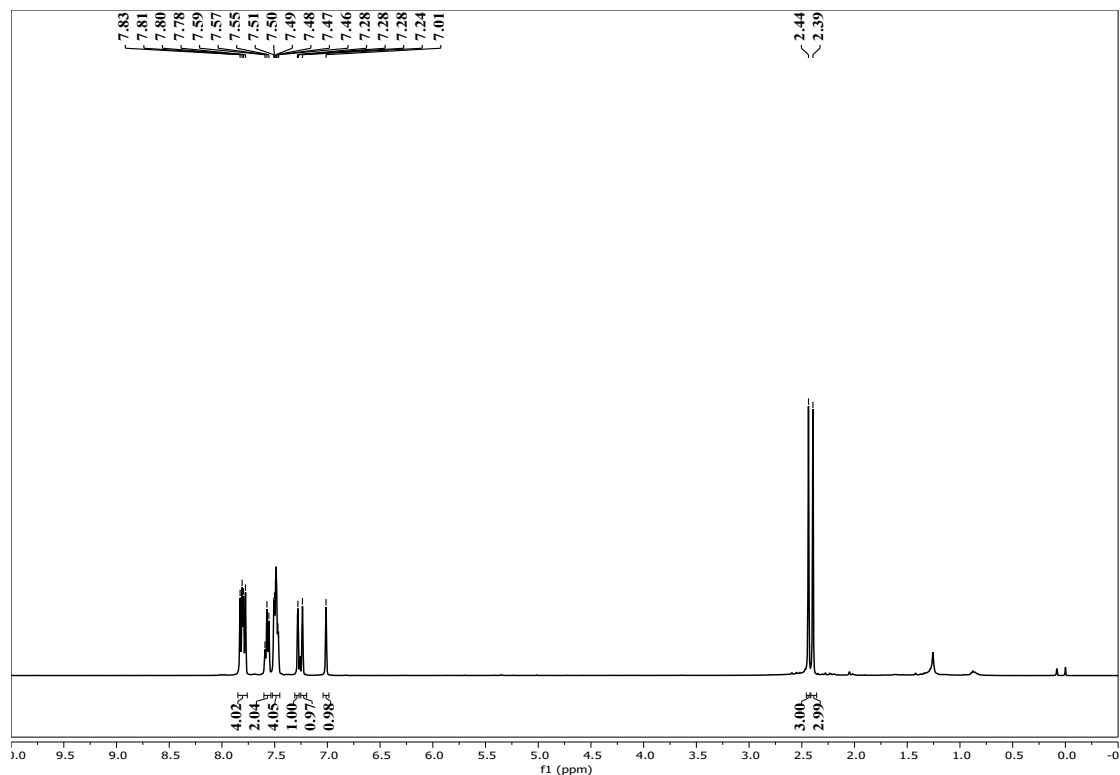


3b, ^{31}P NMR (162 MHz), CDCl_3

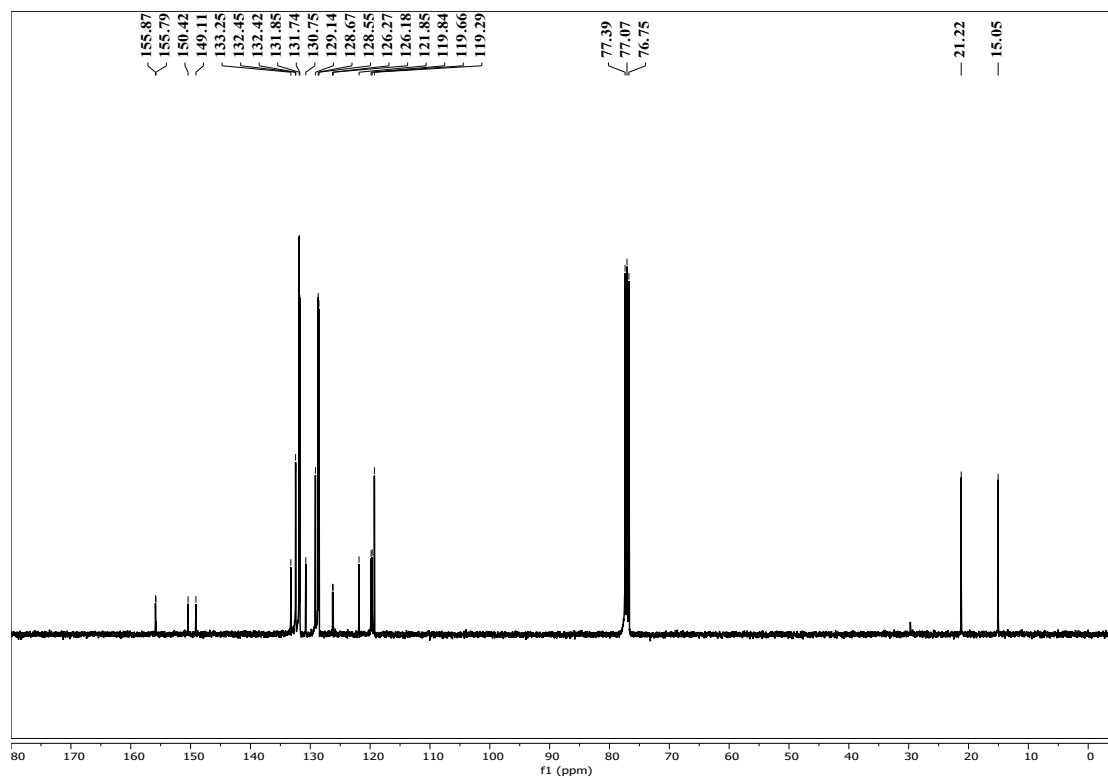




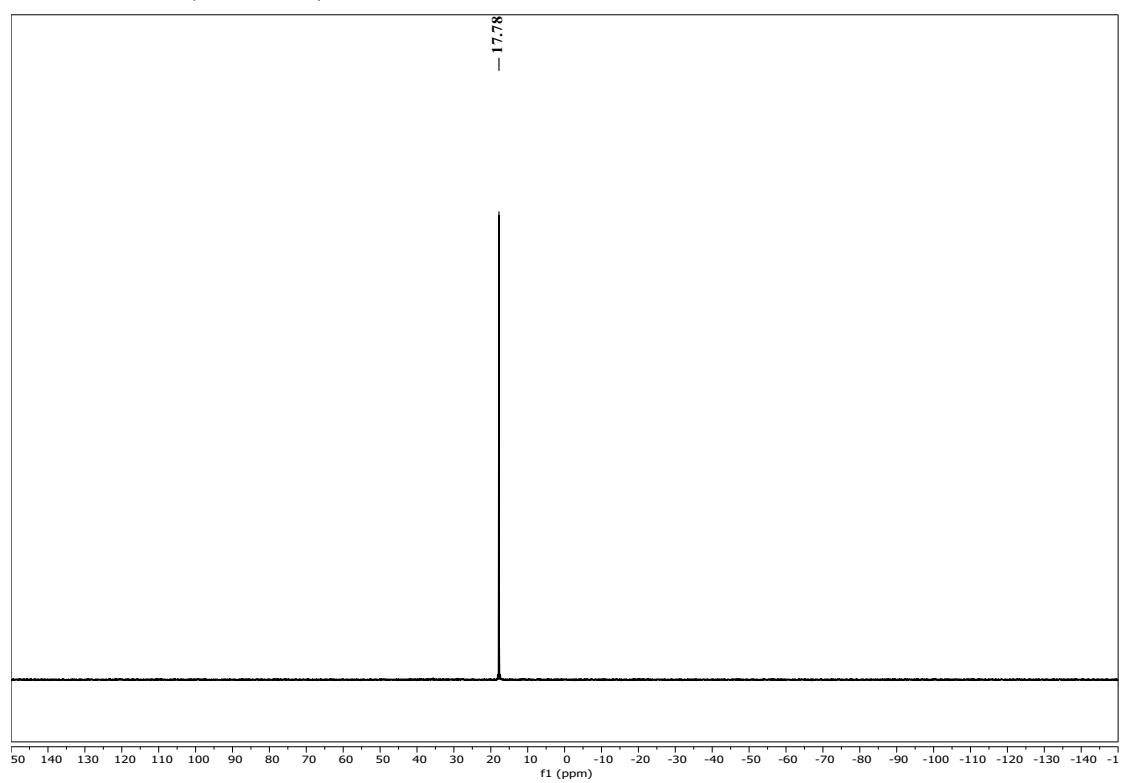
3c, ^1H NMR (400 MHz), CDCl_3

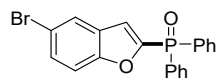


3c, ^{13}C NMR (101 MHz), CDCl_3

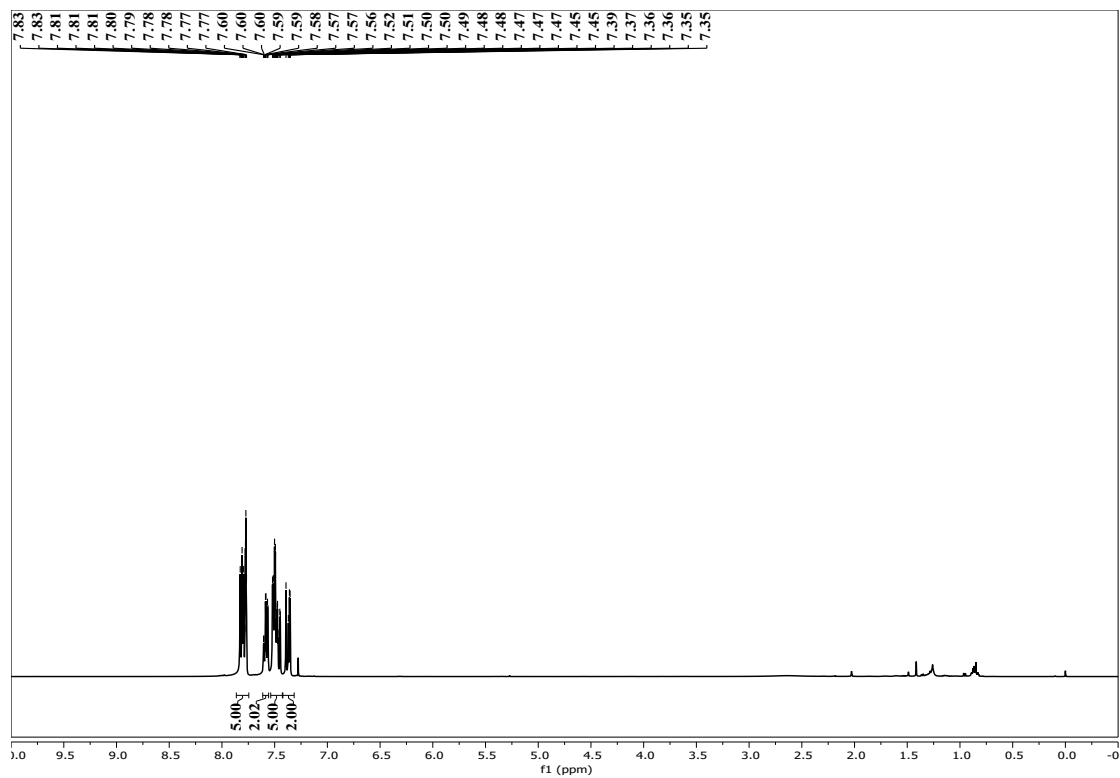


3c, ^{31}P NMR (162 MHz), CDCl_3

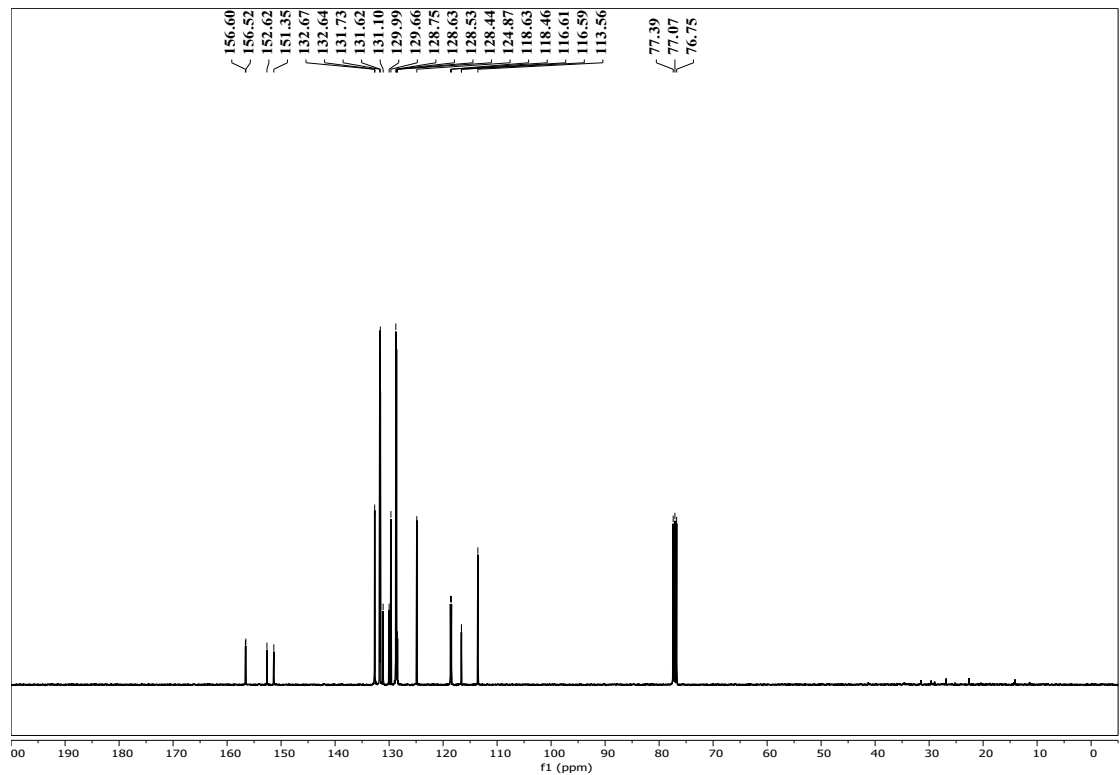




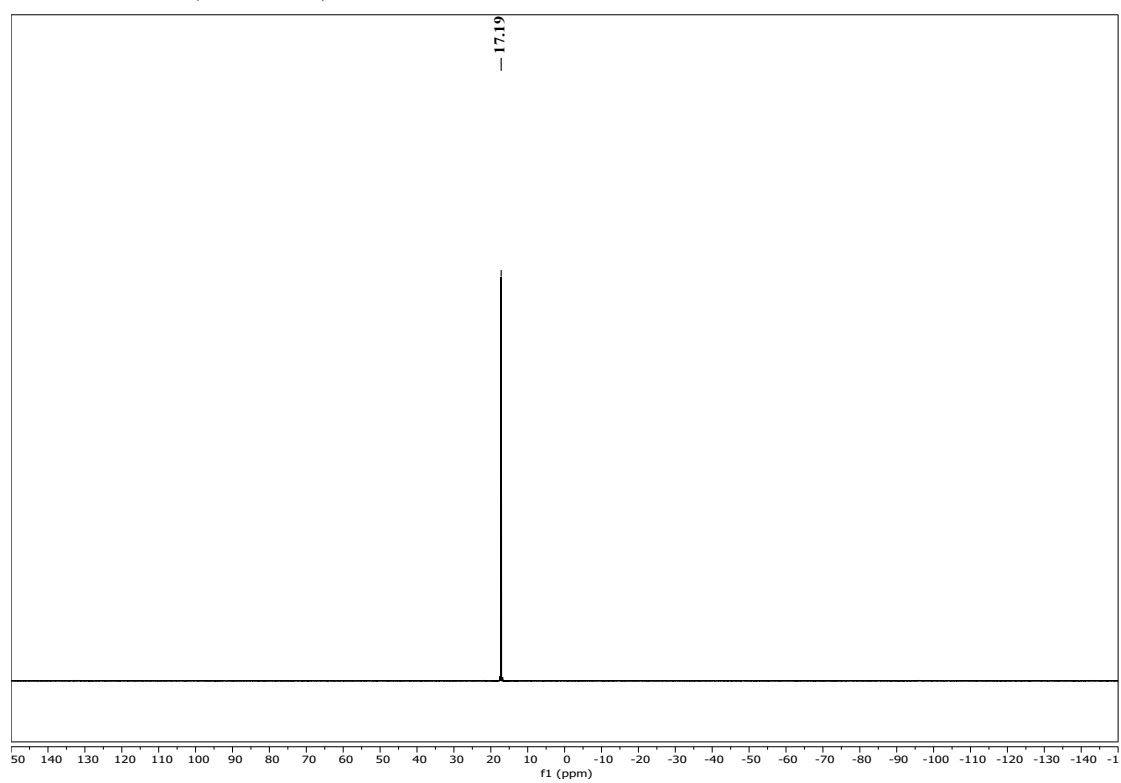
3d, ^1H NMR (400 MHz), CDCl_3

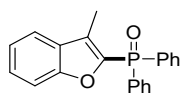


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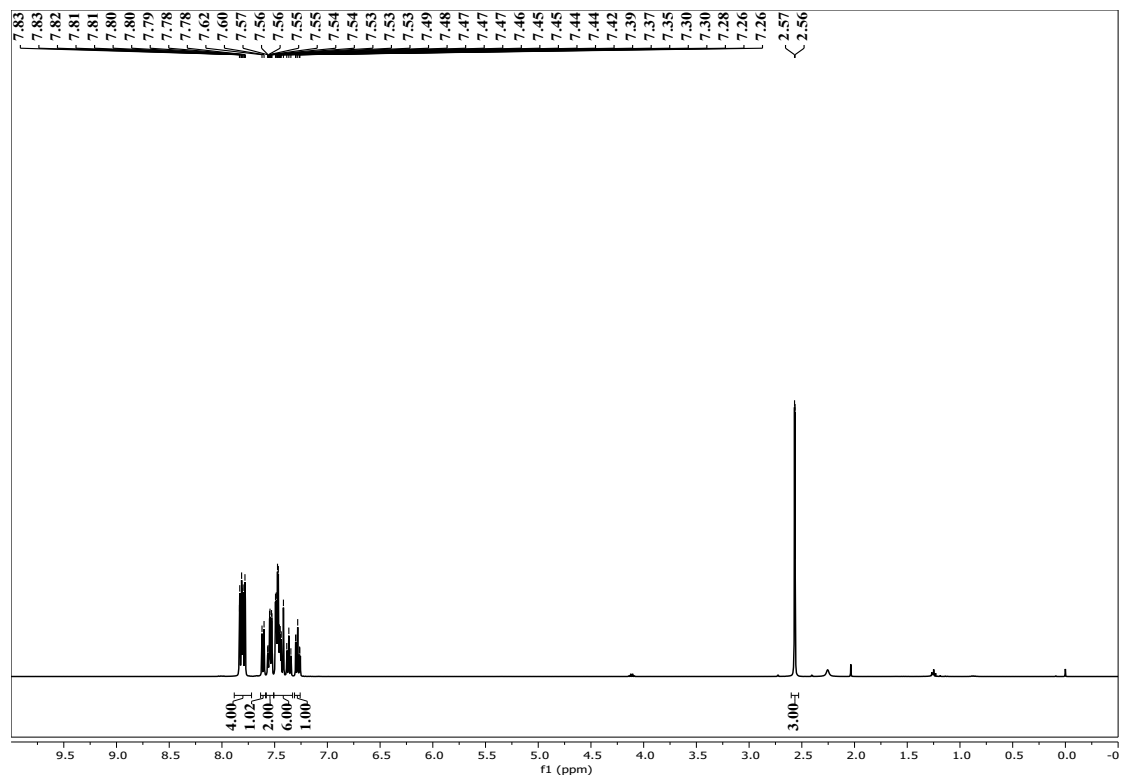


3d, ^{31}P NMR (162 MHz), CDCl_3

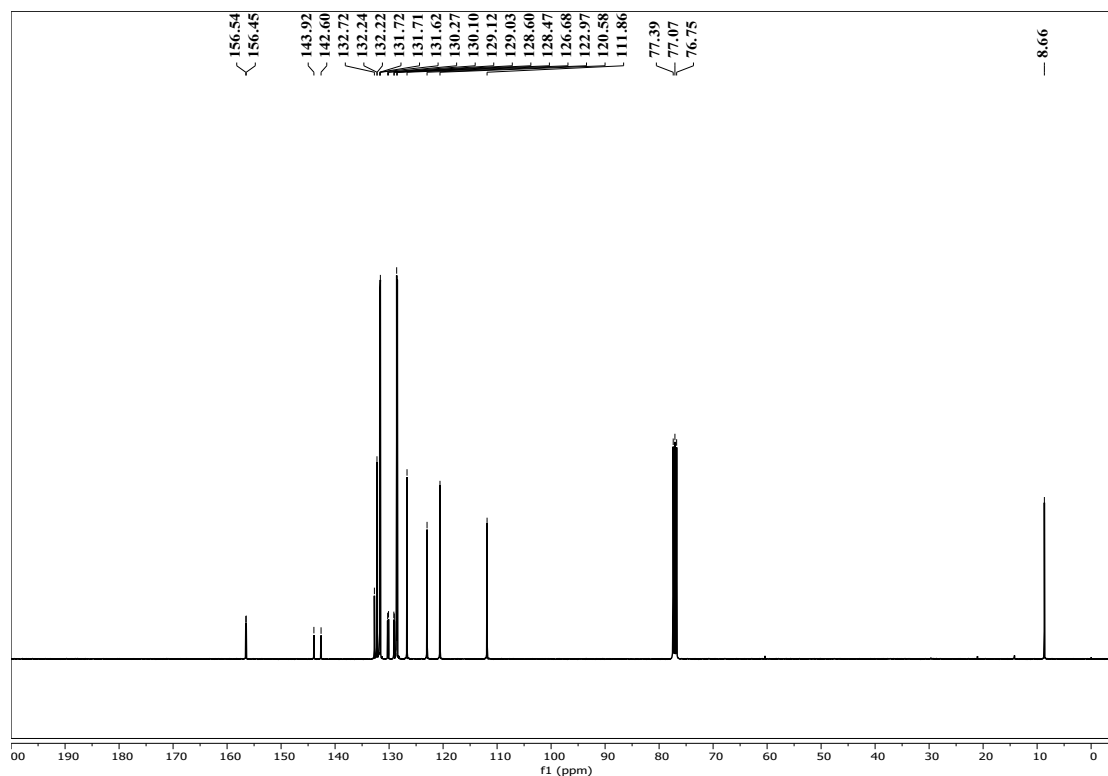




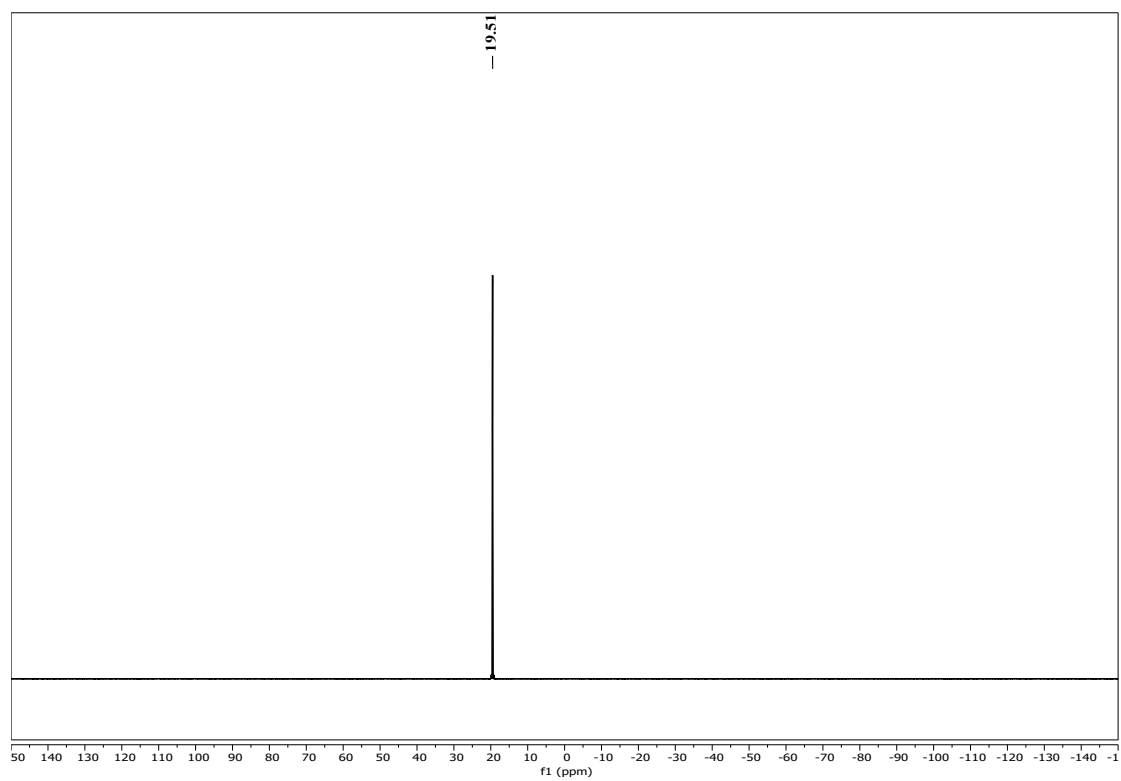
3e, ^1H NMR (400 MHz), CDCl_3

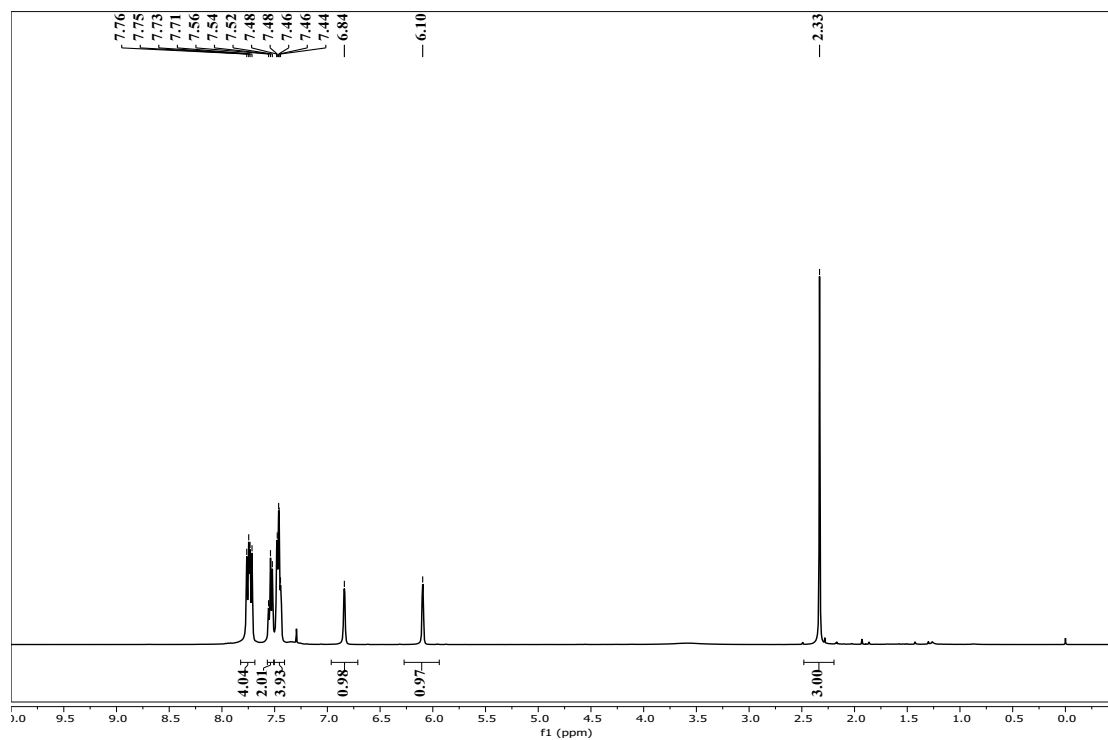


3e, ^{13}C NMR (101 MHz), CDCl_3

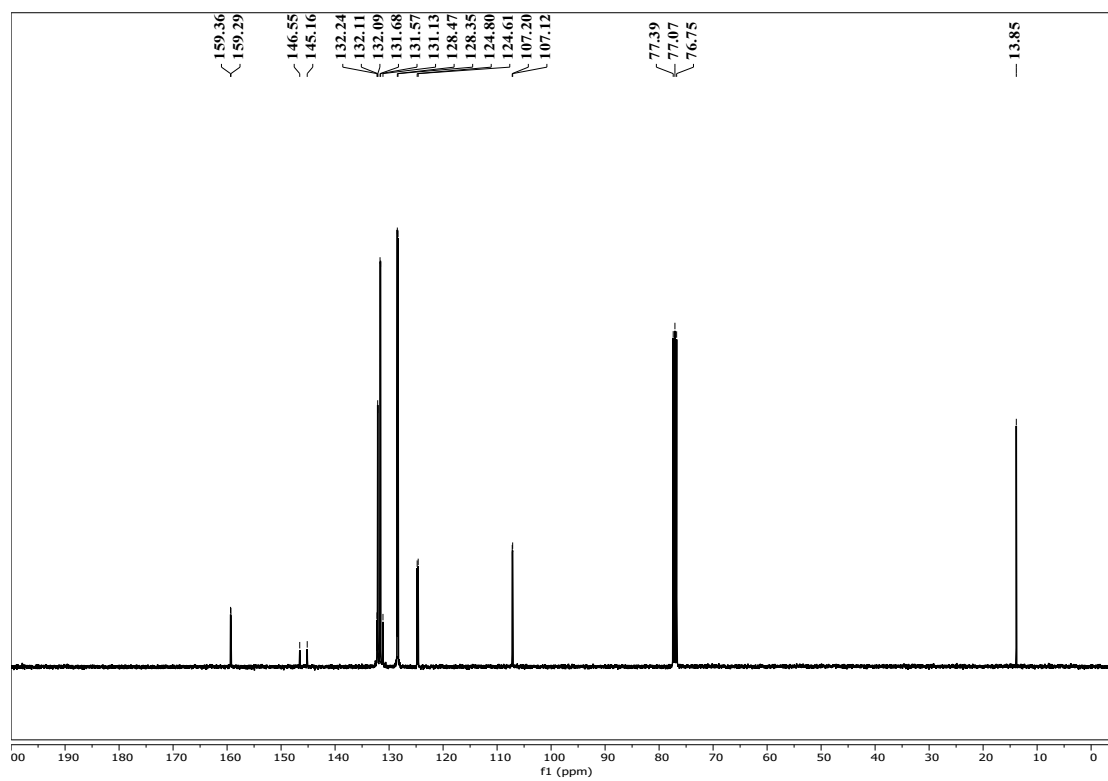


3e, ^{31}P NMR (162 MHz), CDCl_3

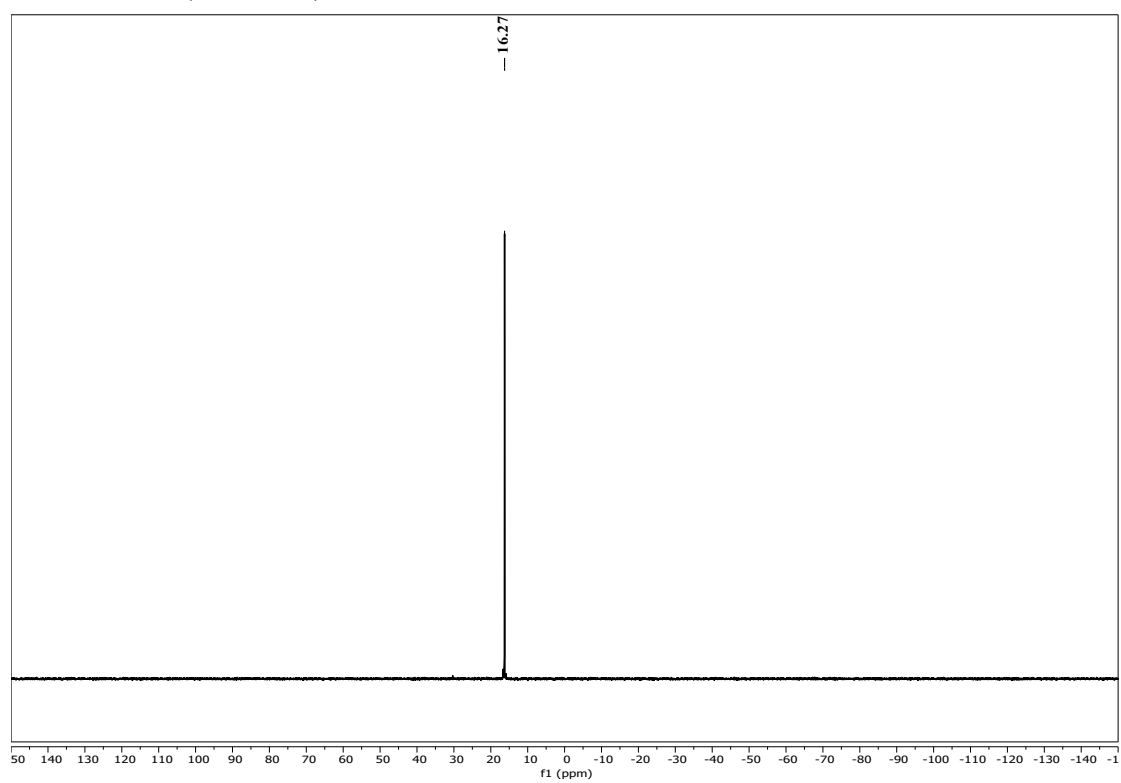


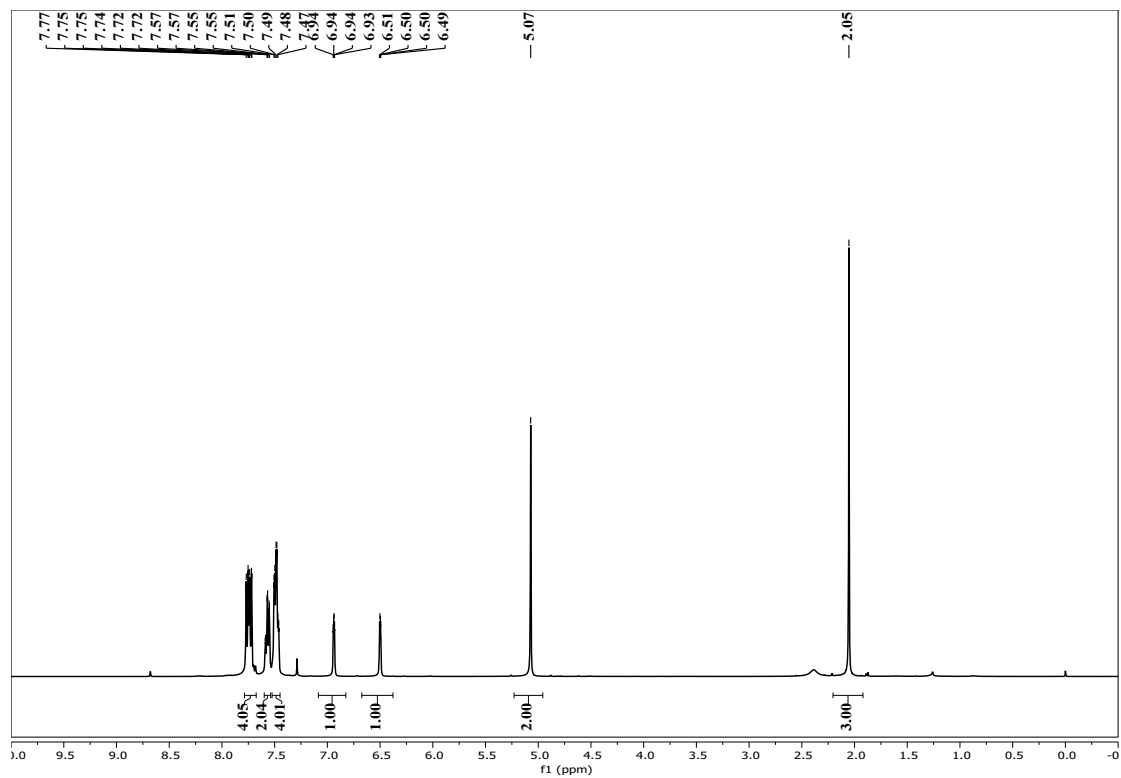


3f, ^{13}C NMR (101 MHz), CDCl_3

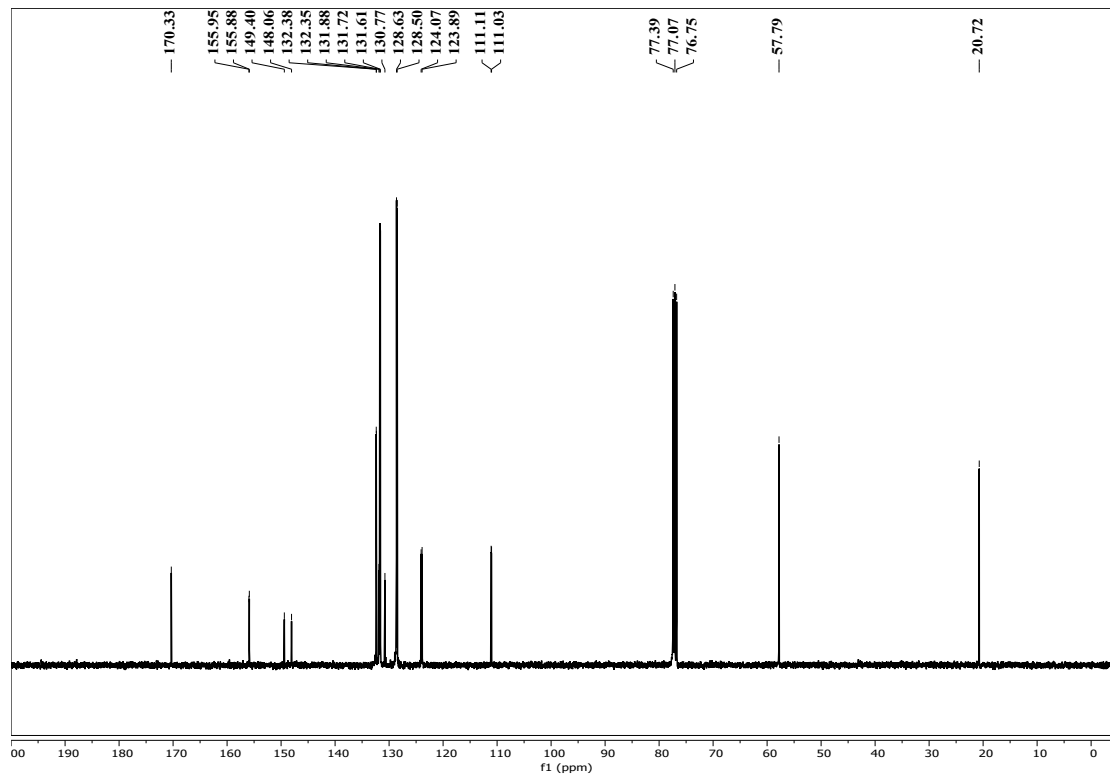


3f, ^{31}P NMR (162 MHz), CDCl_3

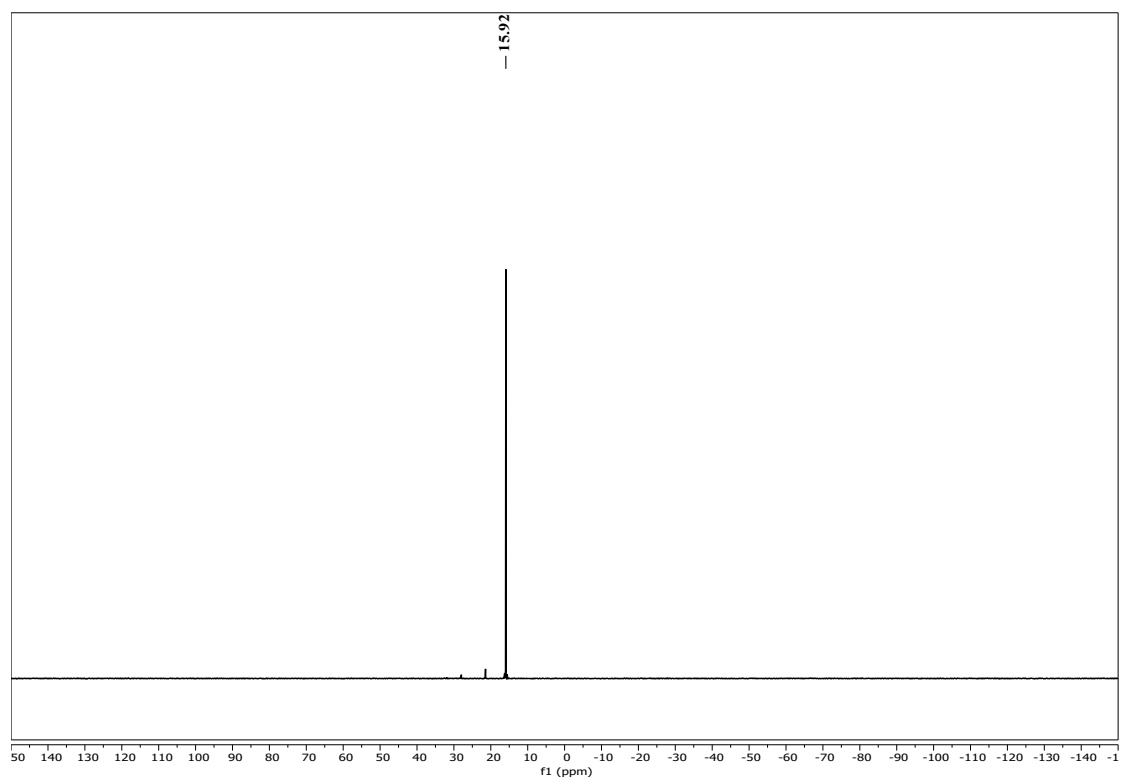


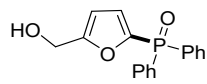


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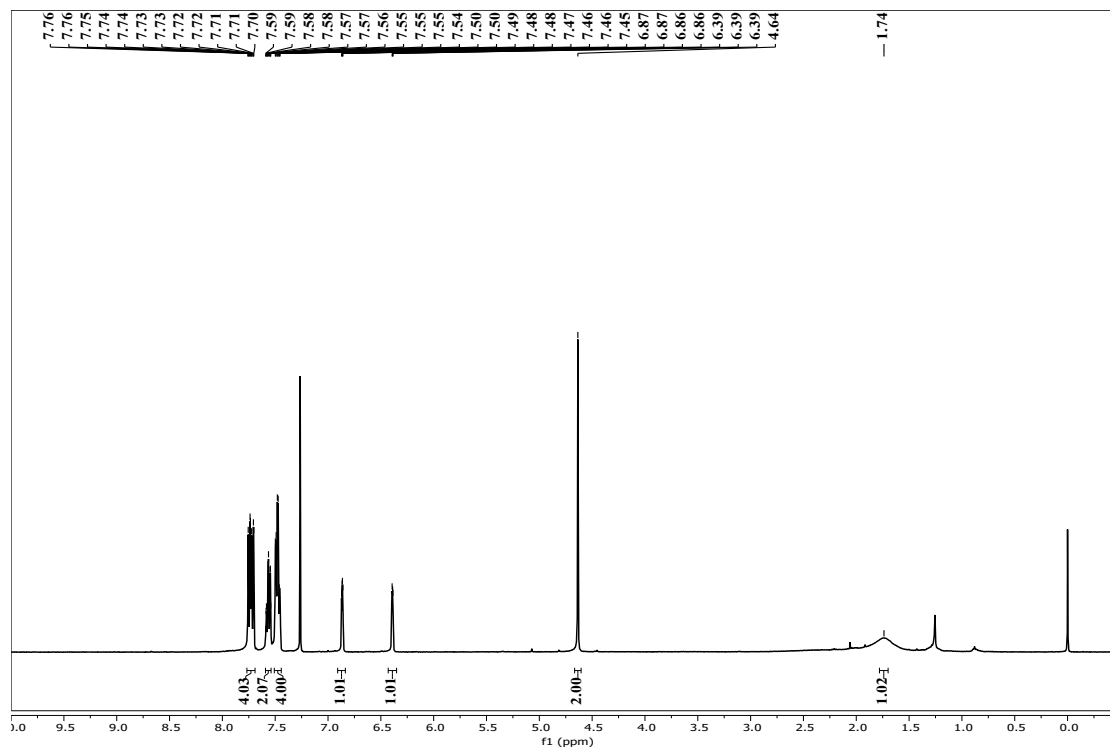


3g, ^{31}P NMR (162 MHz), CDCl_3

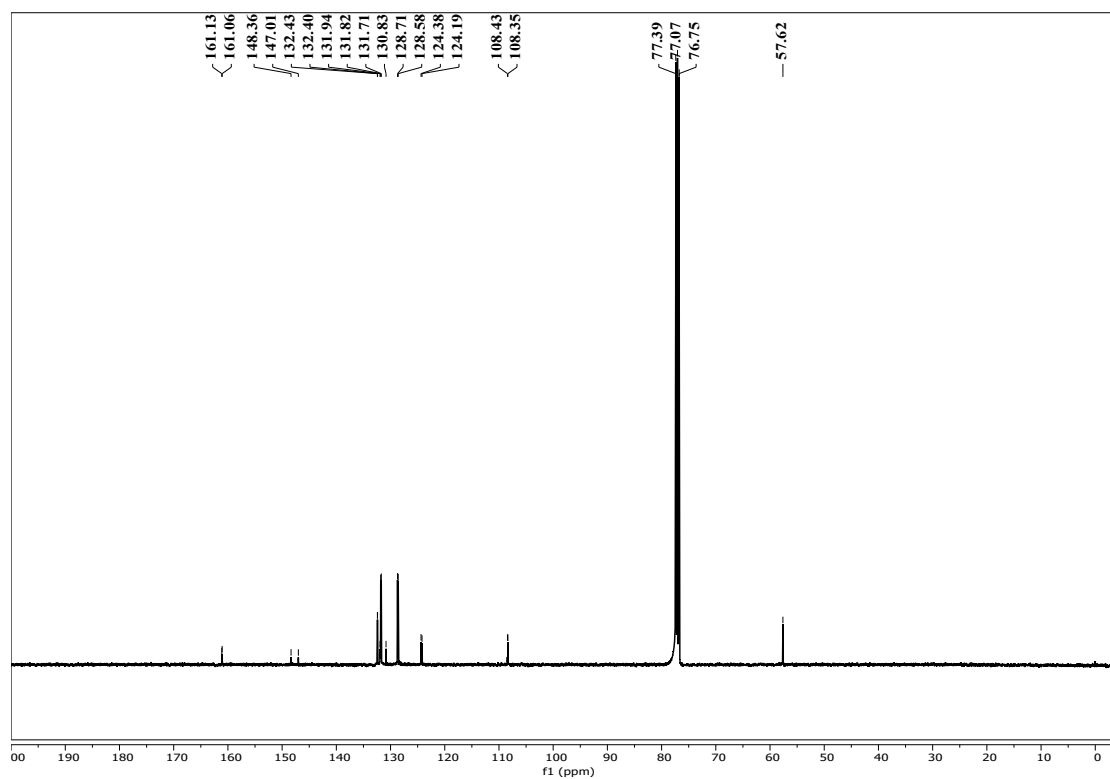




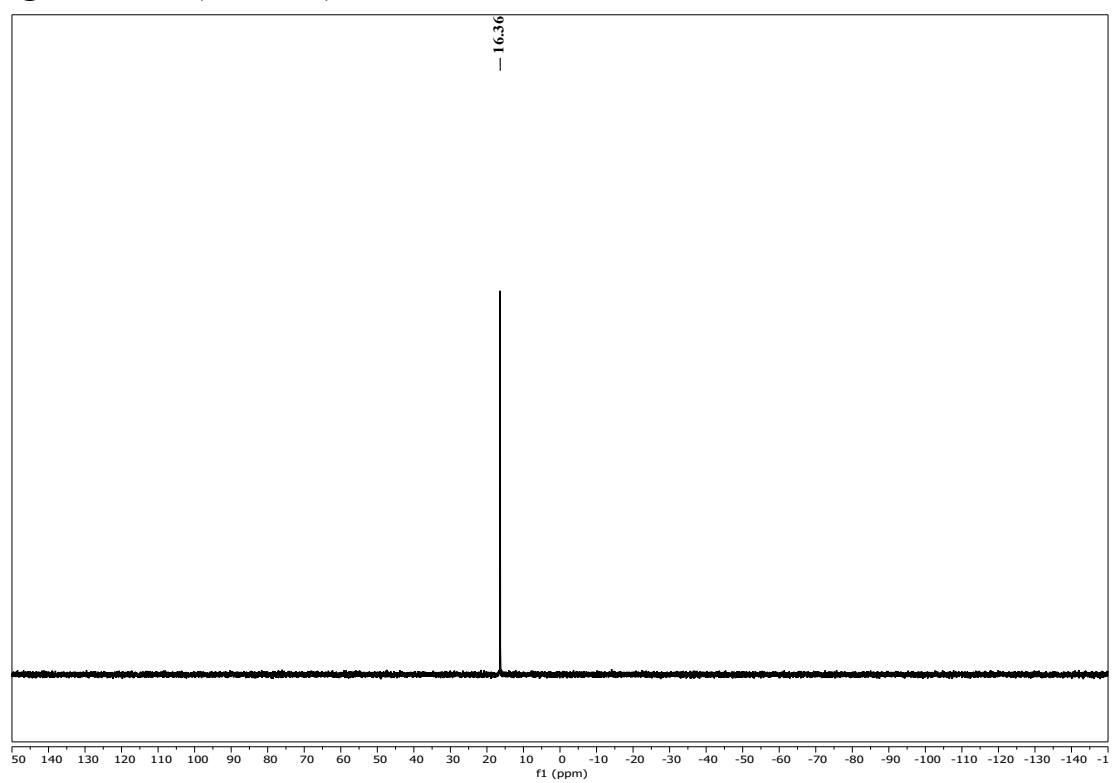
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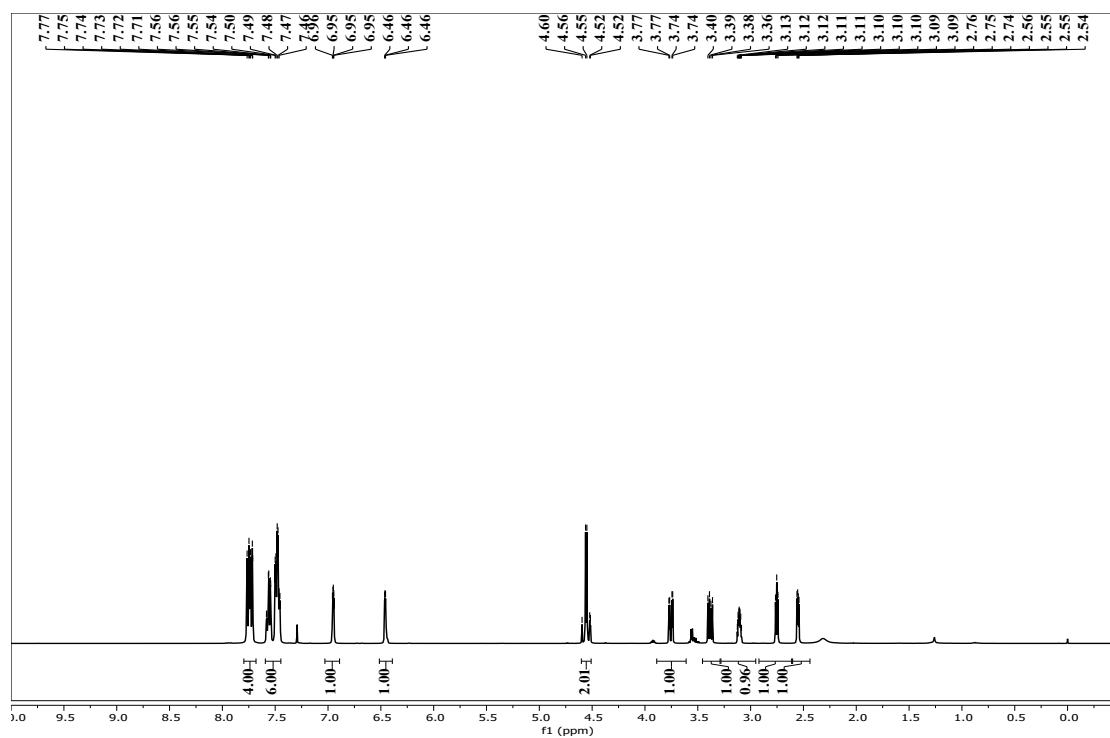
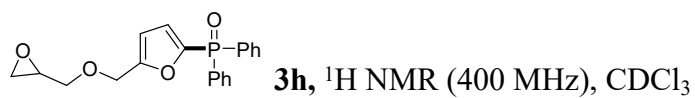


3g', ^{13}C NMR (101 MHz), CDCl_3

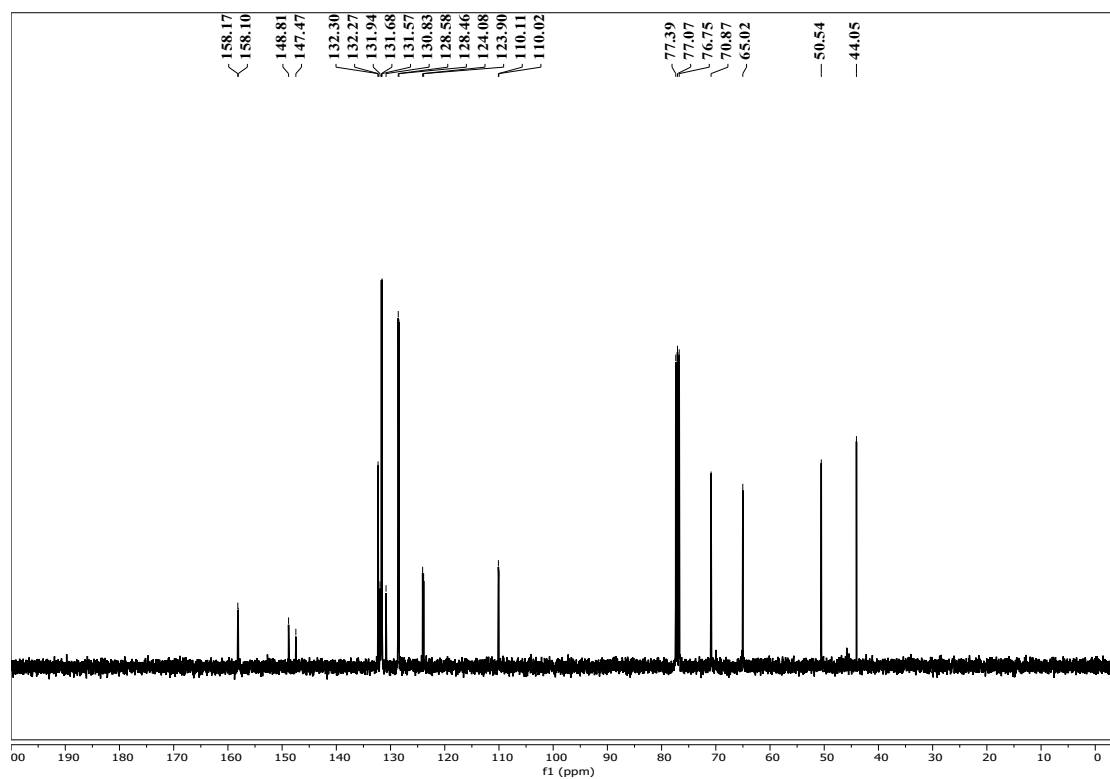


3g', ^{31}P NMR (162 MHz), CDCl_3

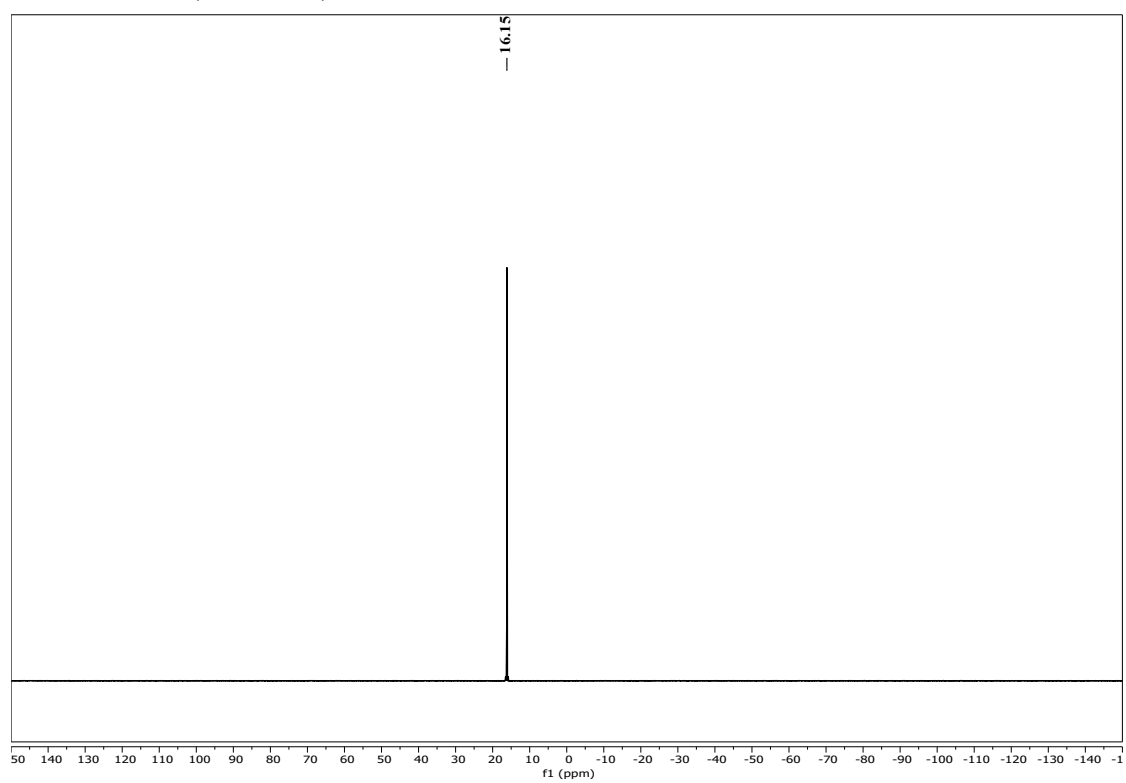


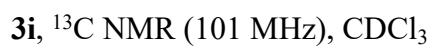


3h, ^{13}C NMR (101 MHz), CDCl_3

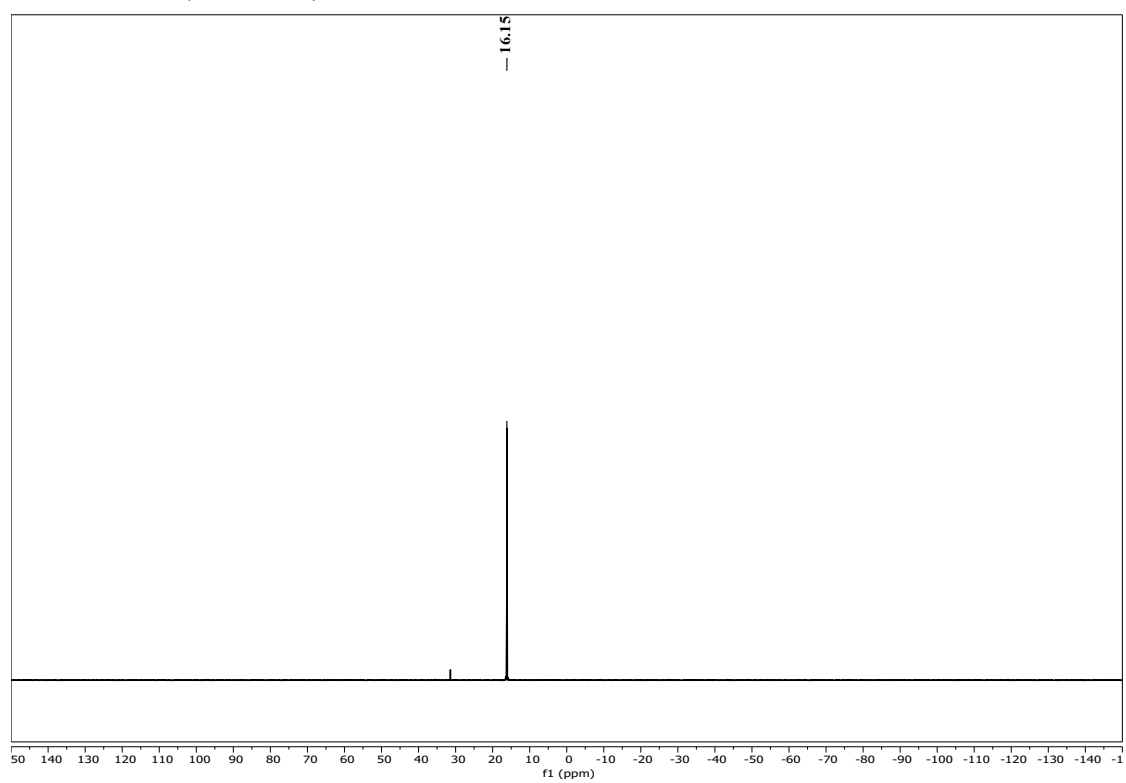


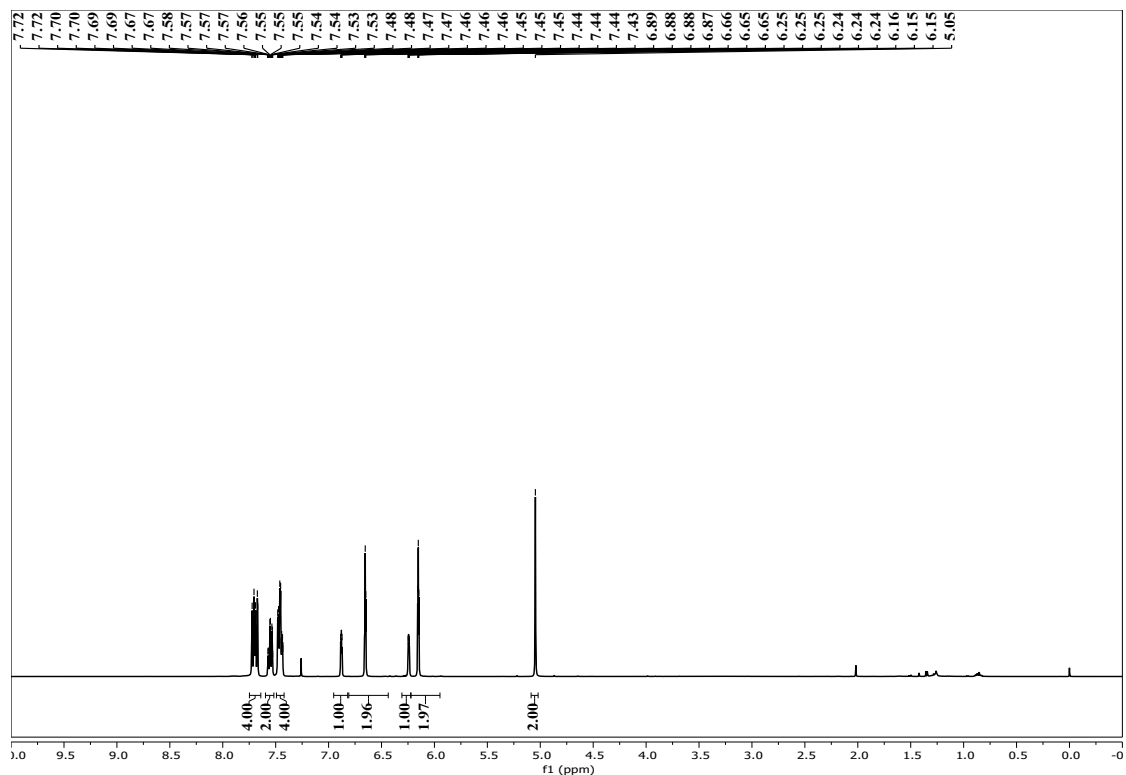
3h, ^{31}P NMR (162 MHz), CDCl_3



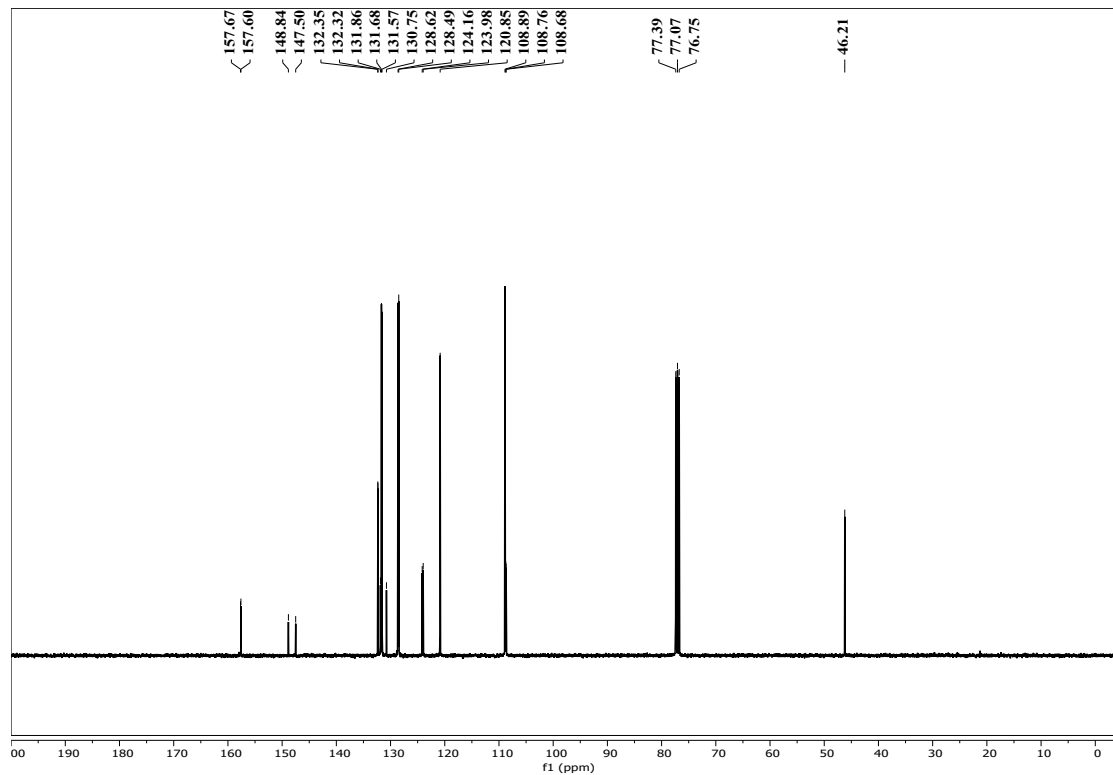


3i, ^{31}P NMR (162 MHz), CDCl_3

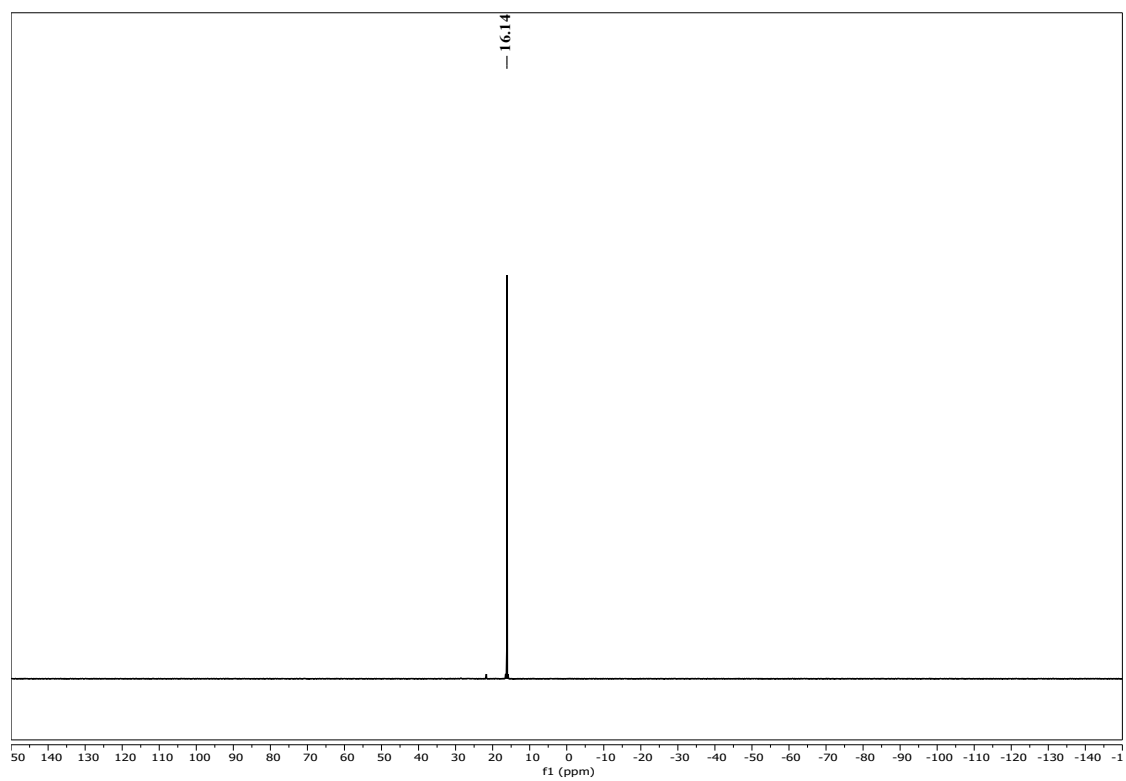


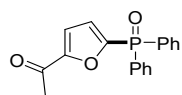


3j, ^{13}C NMR (101 MHz), CDCl_3

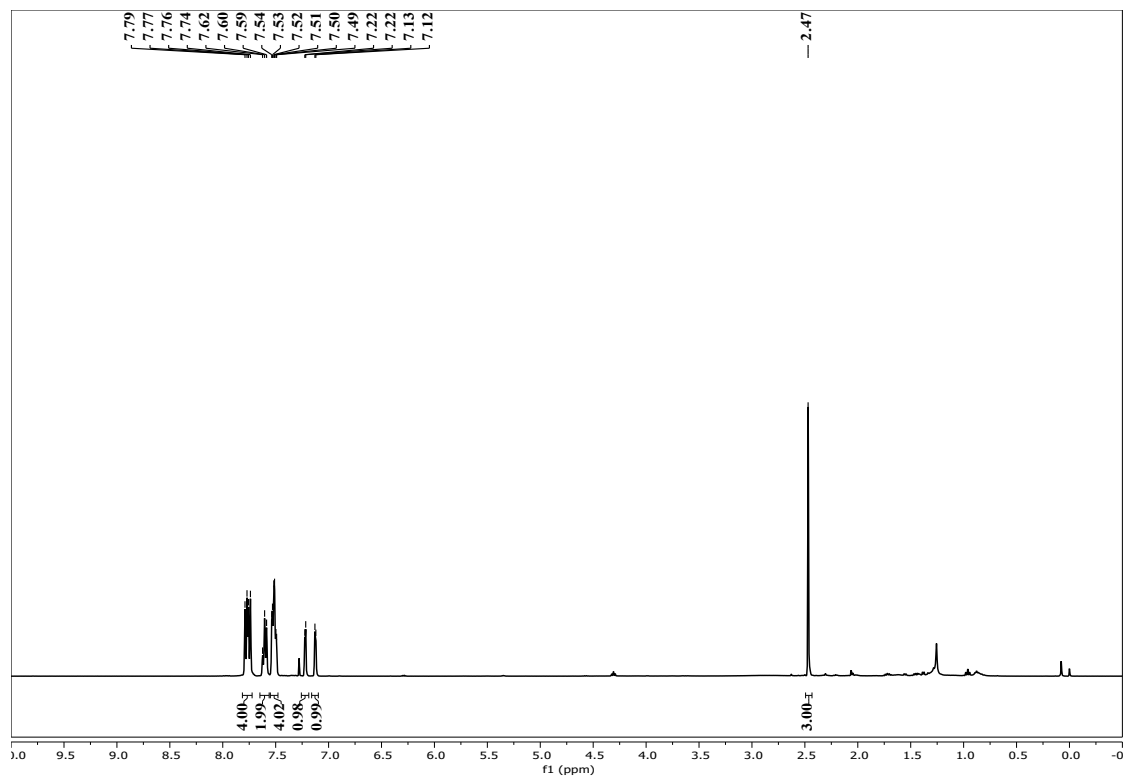


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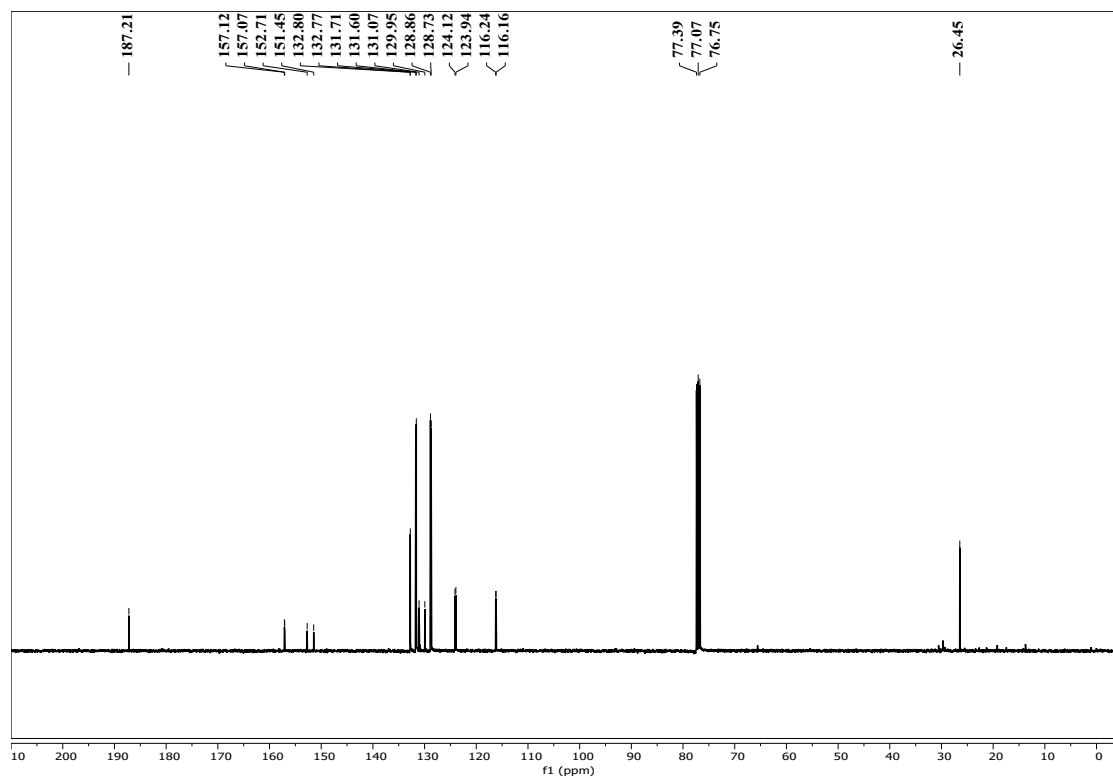




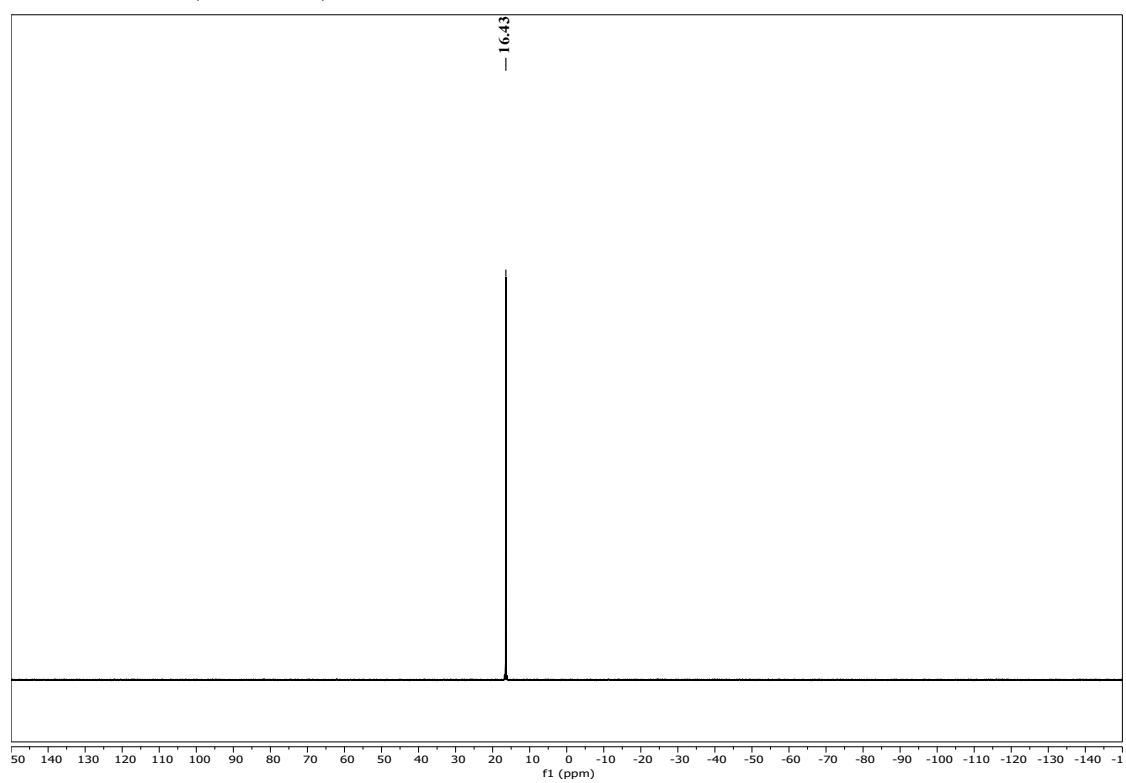
3k, ^1H NMR (400 MHz), CDCl_3

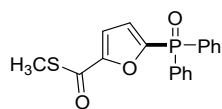


3k, ^{13}C NMR (101 MHz), CDCl_3

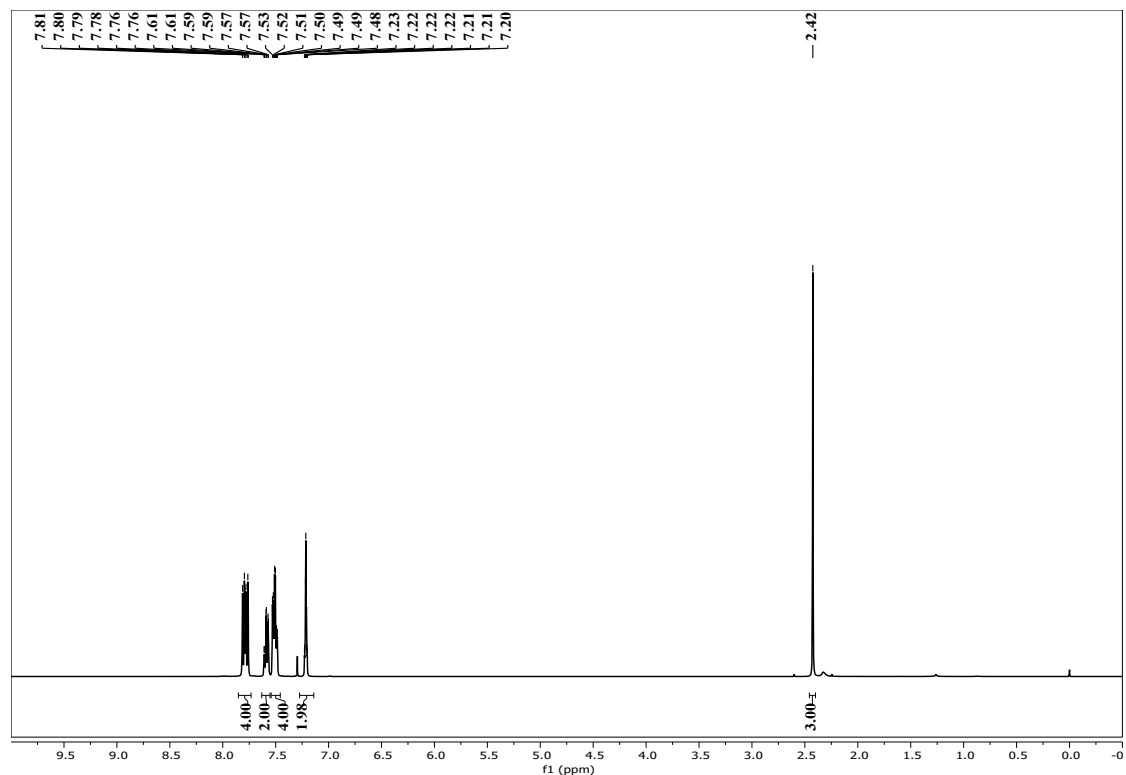


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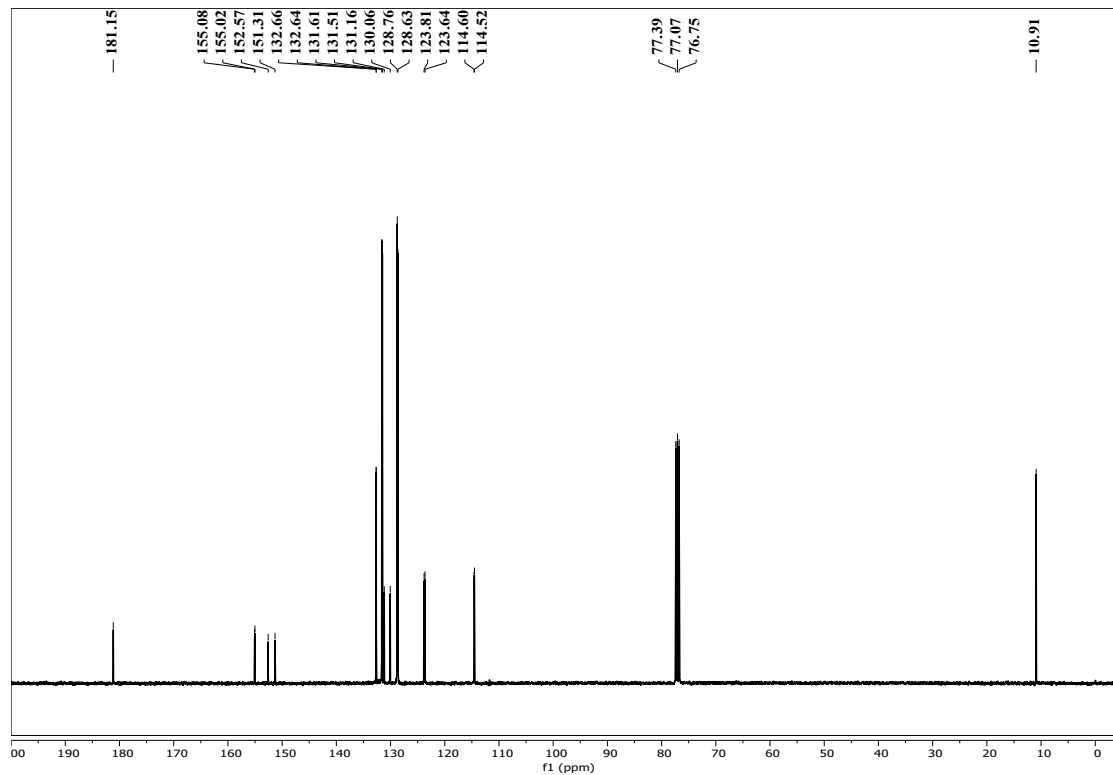




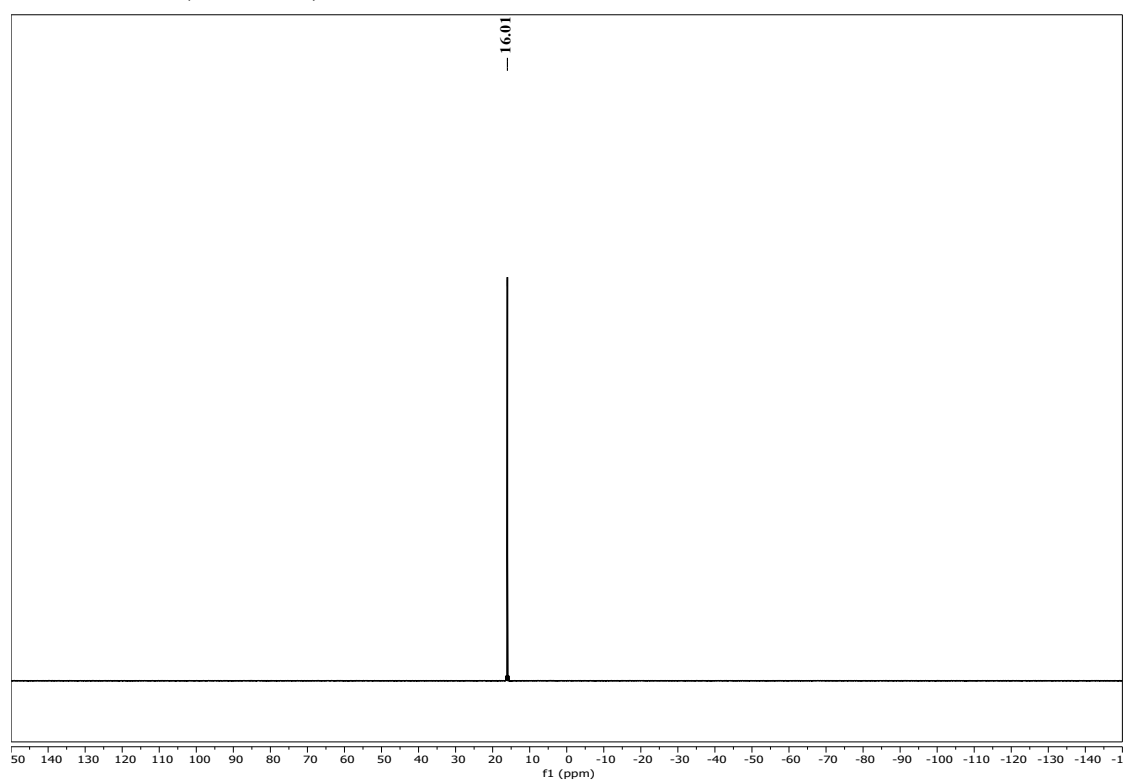
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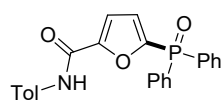


3l, ^{13}C NMR (101 MHz), CDCl_3

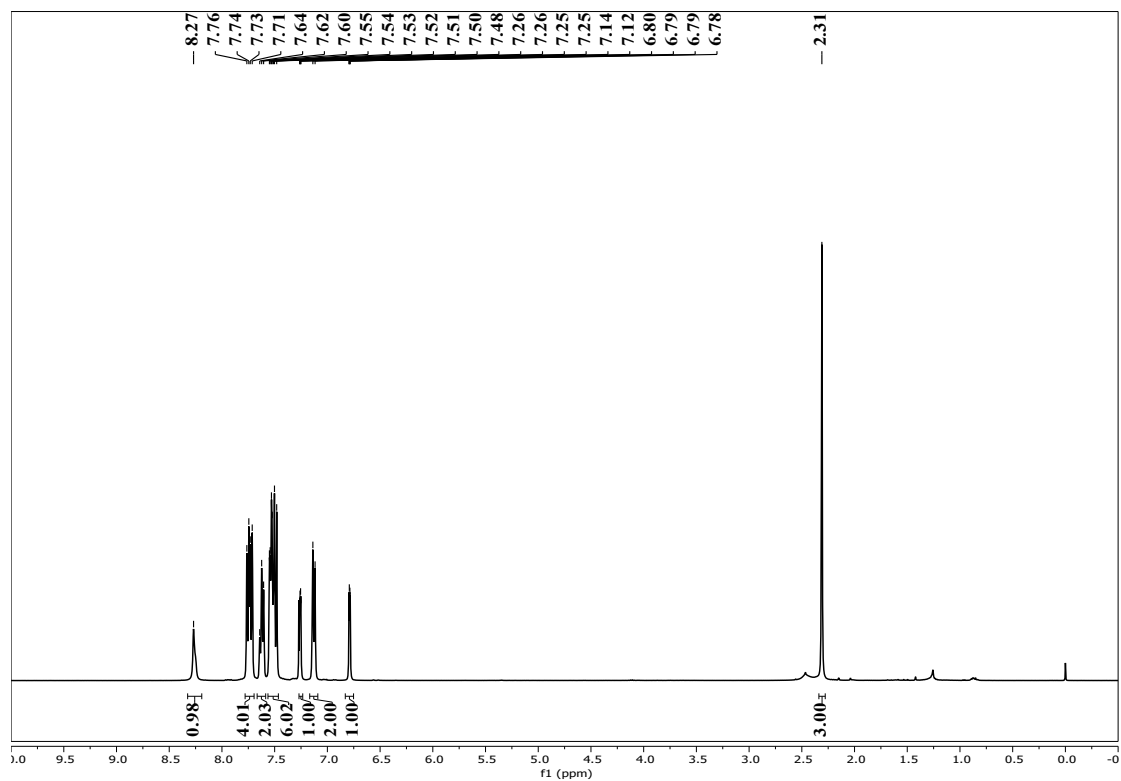


31, ^{31}P NMR (162 MHz), CDCl_3

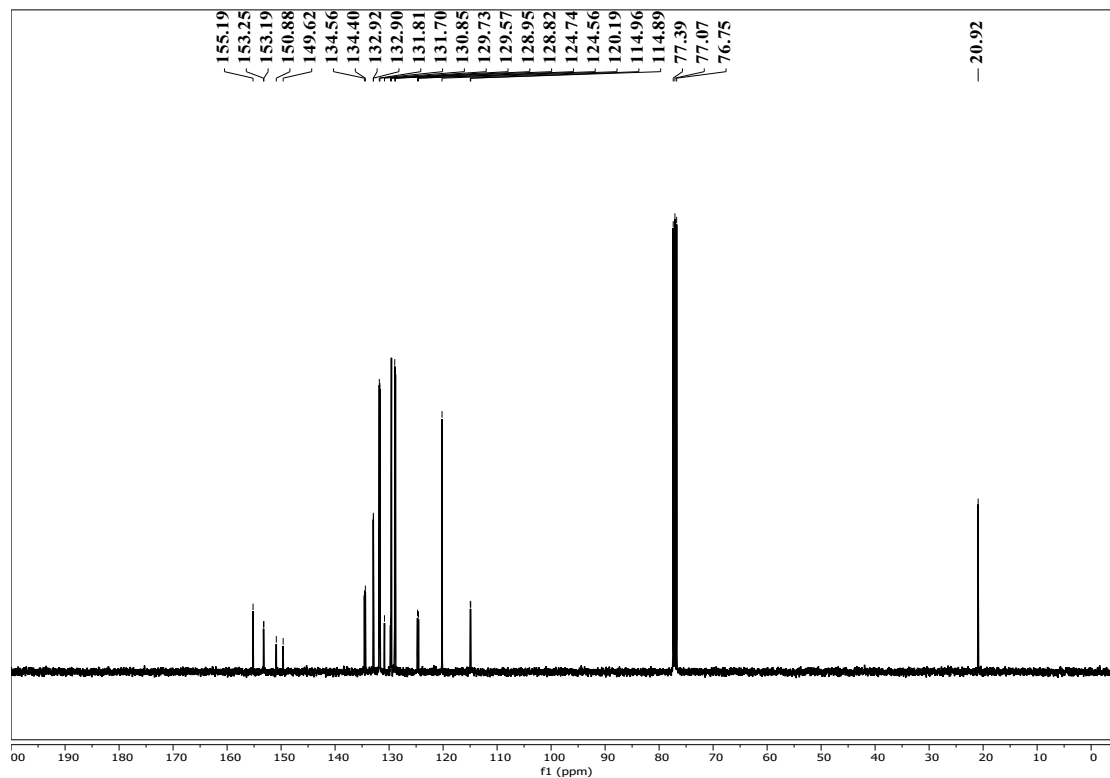




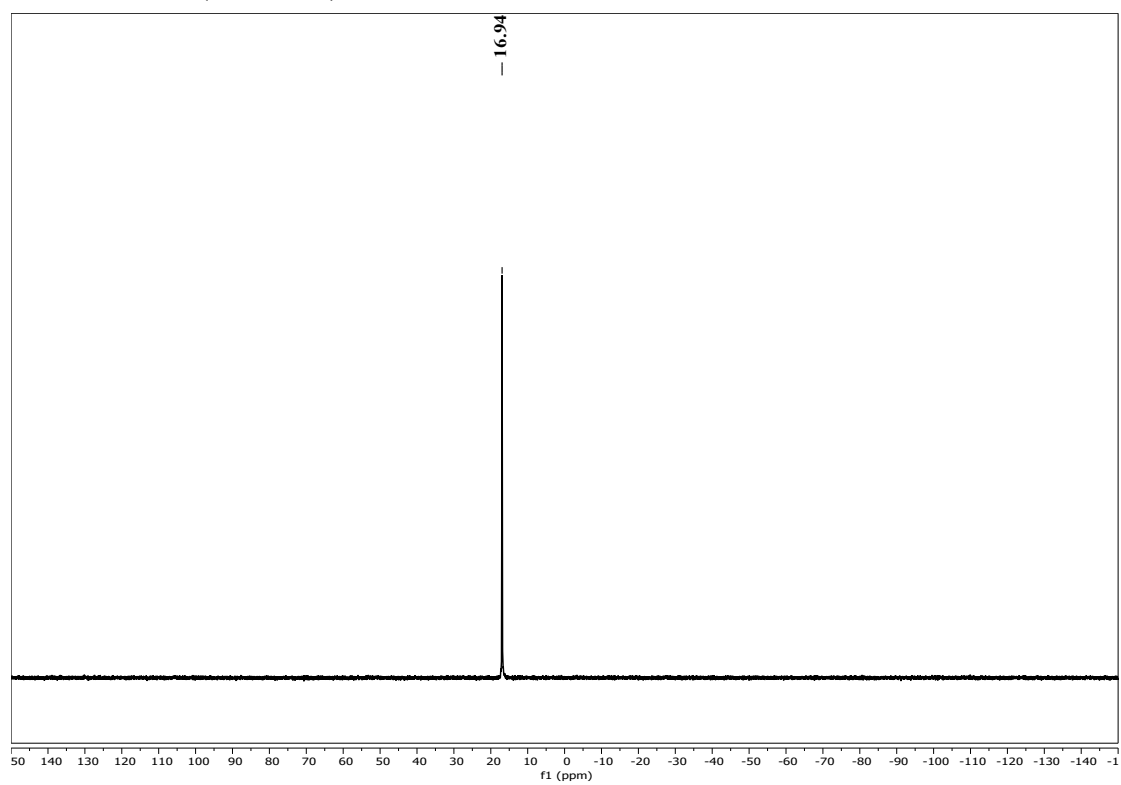
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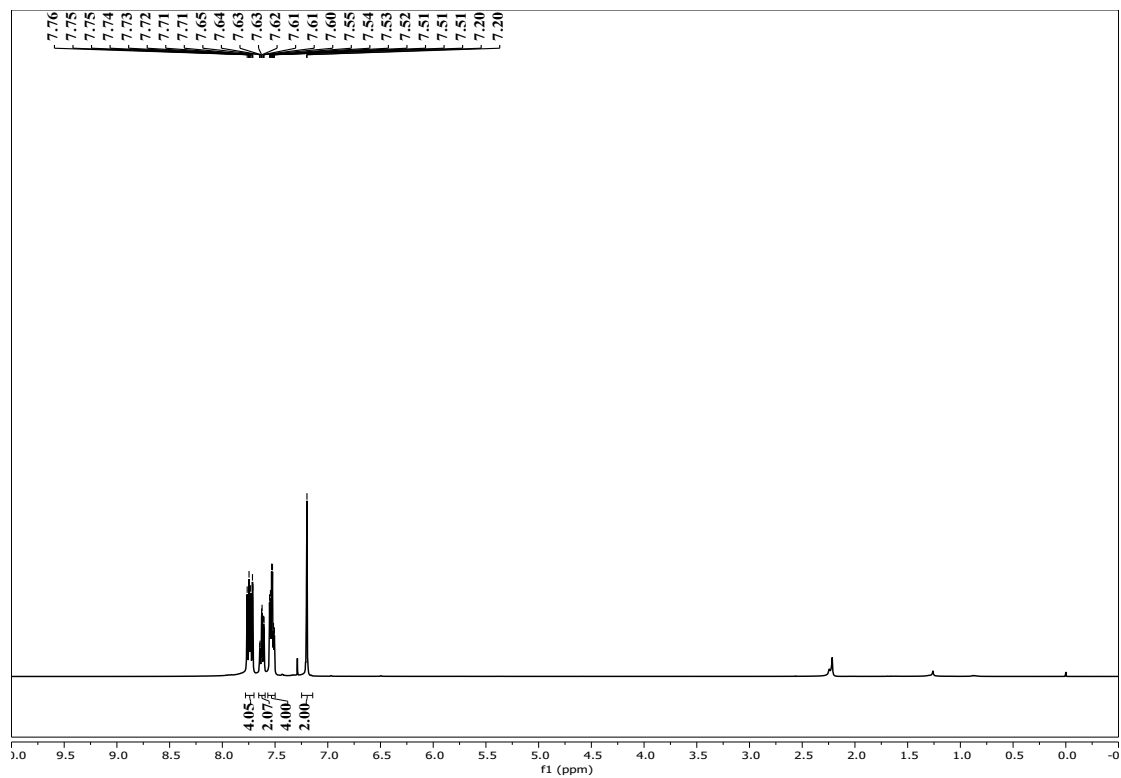


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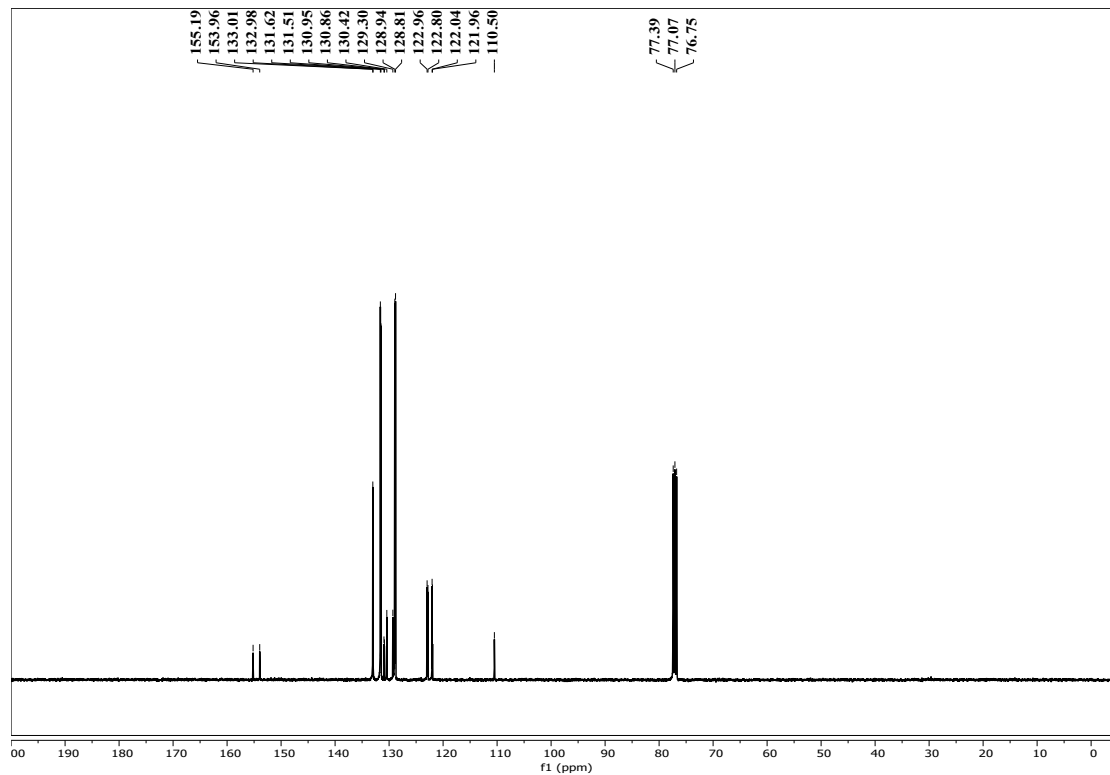


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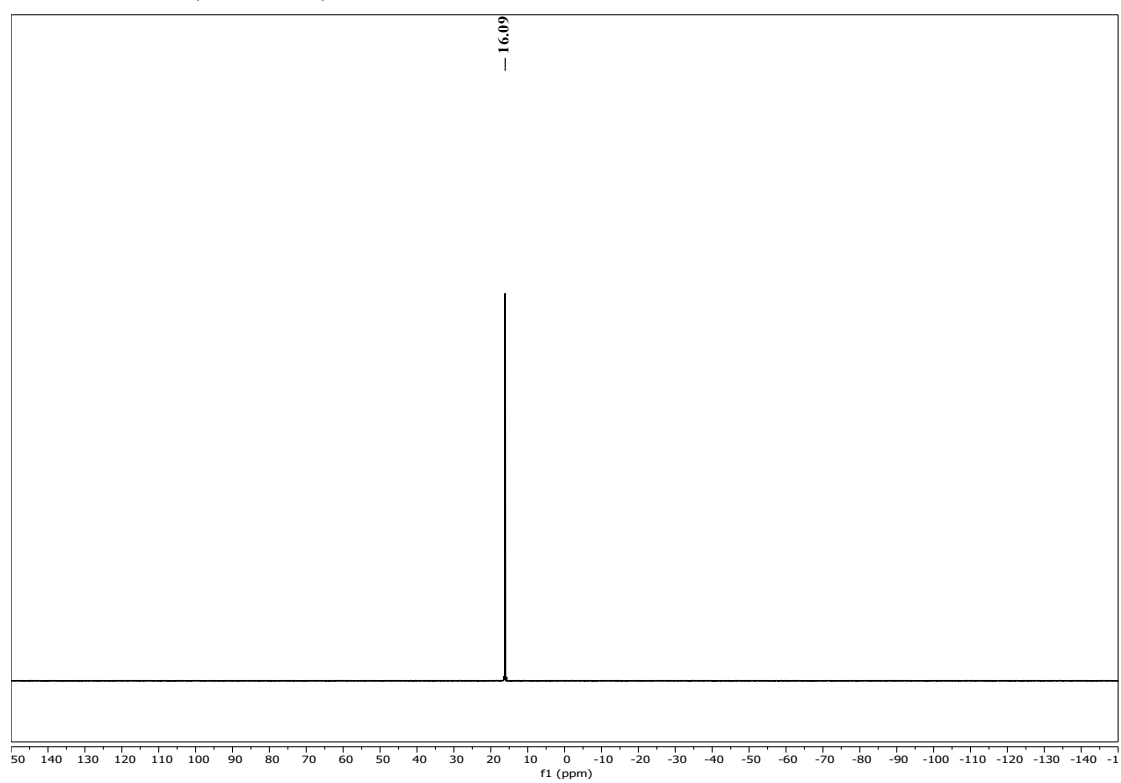


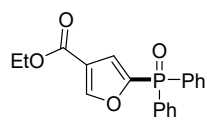


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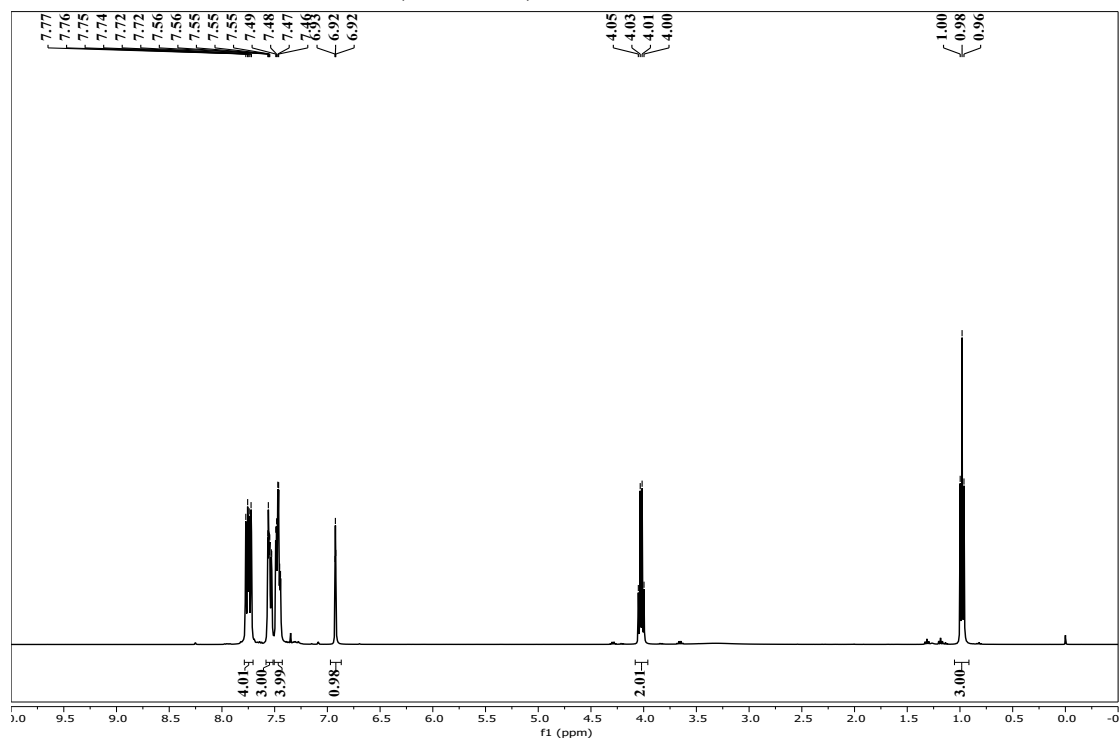


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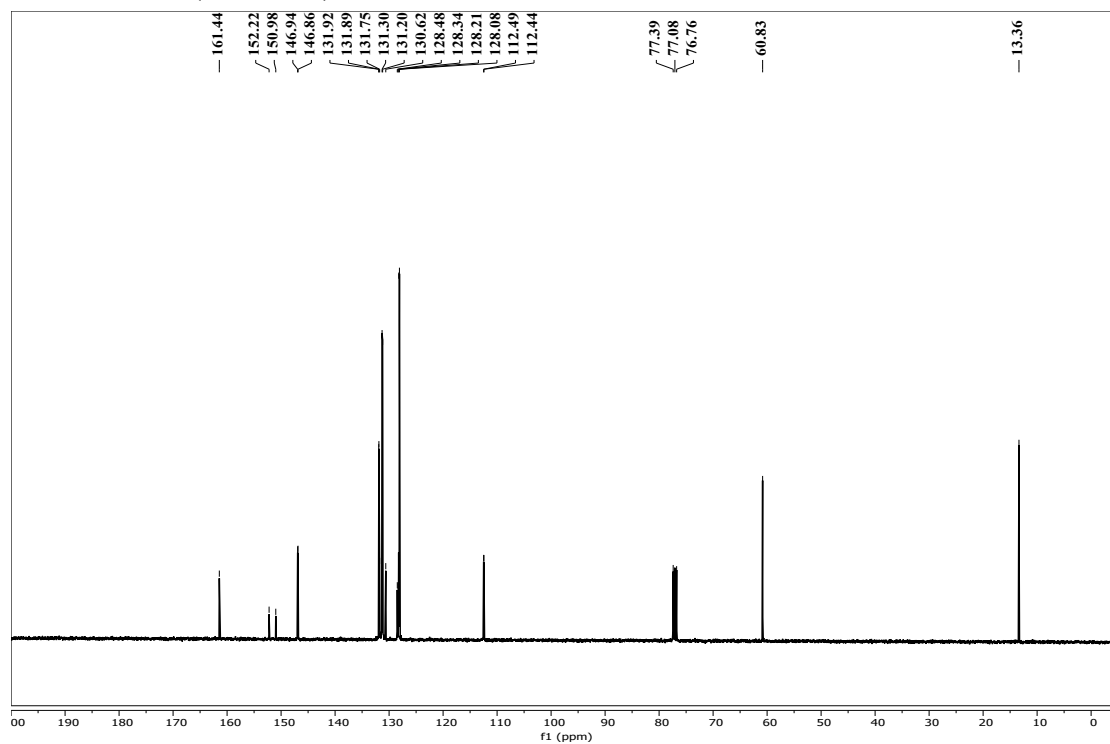




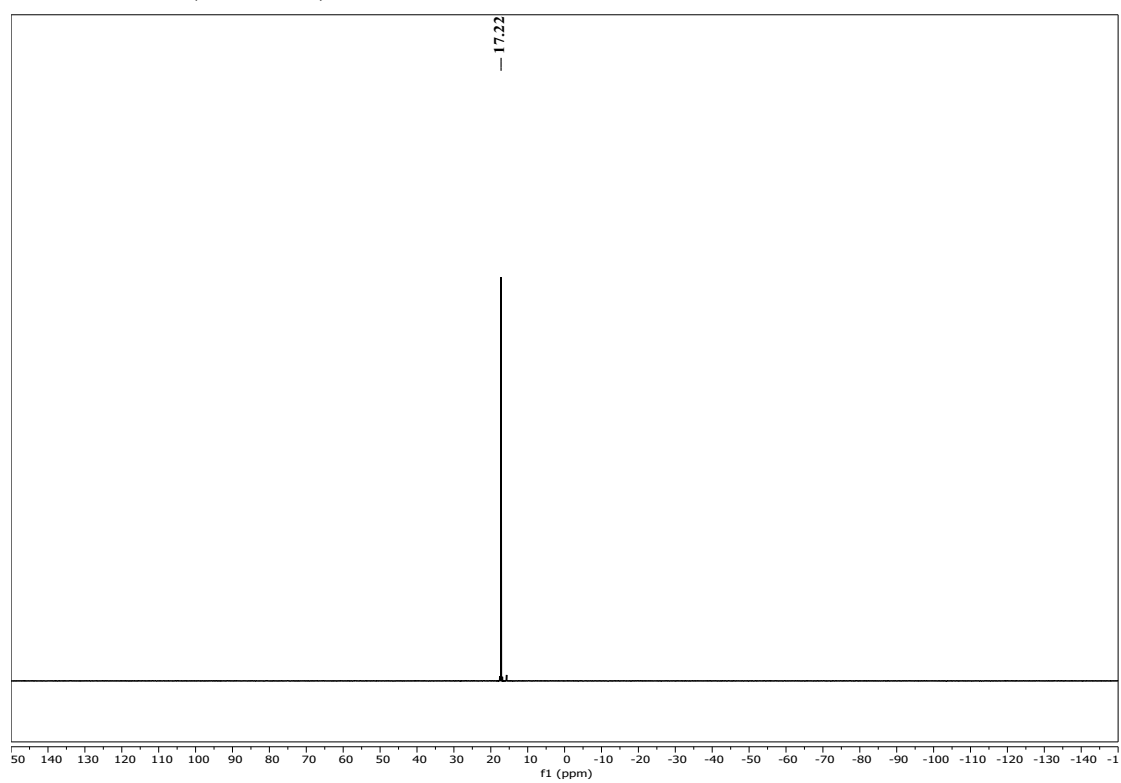
30, ^1H NMR (400 MHz), CDCl_3

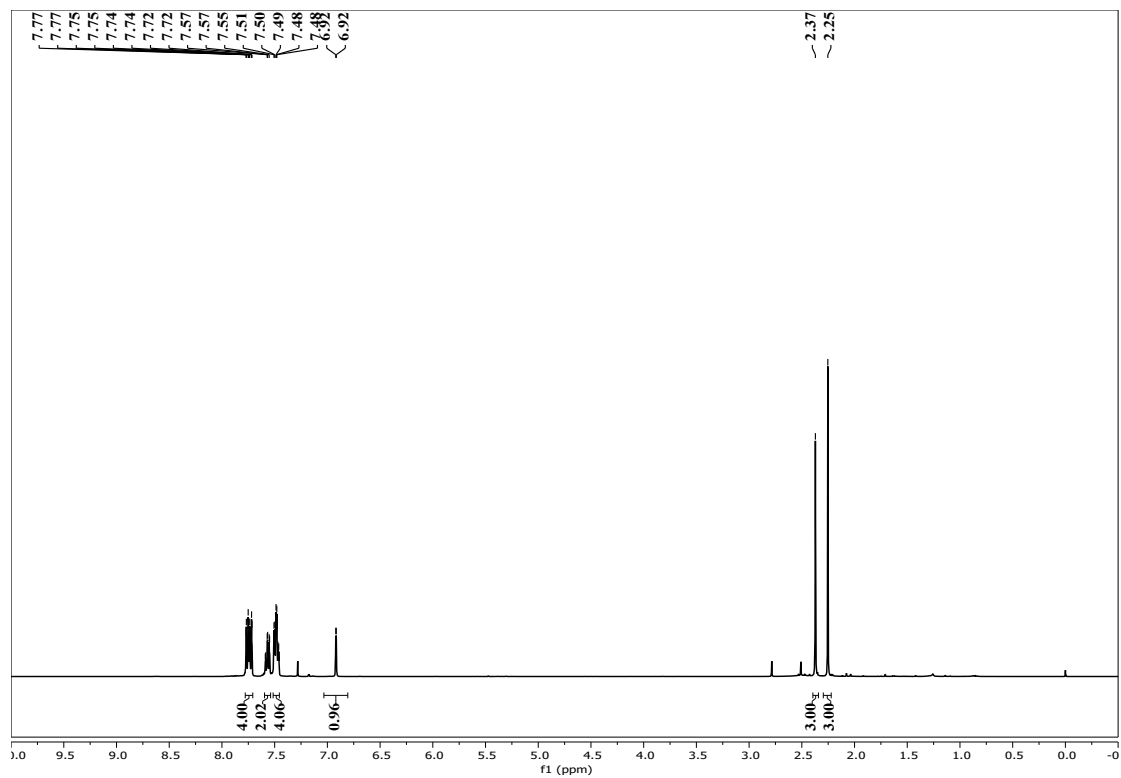


30, ^{13}C NMR (101 MHz), CDCl_3

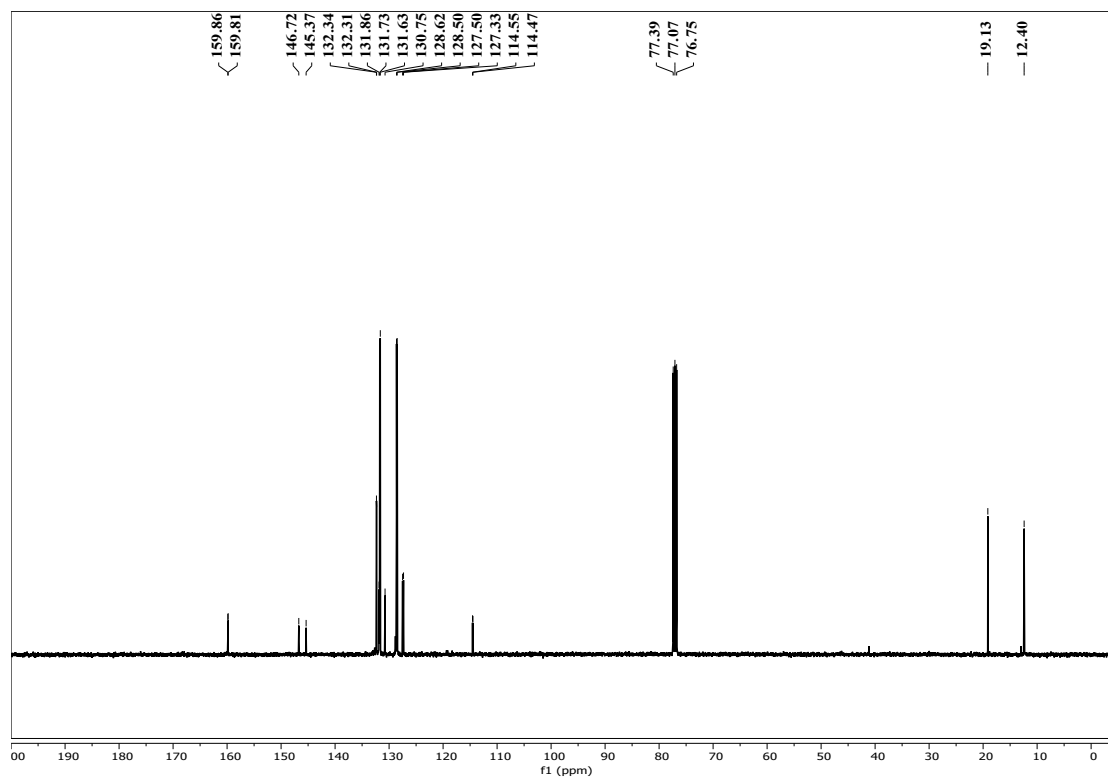


3o, ^{31}P NMR (162 MHz), CDCl_3

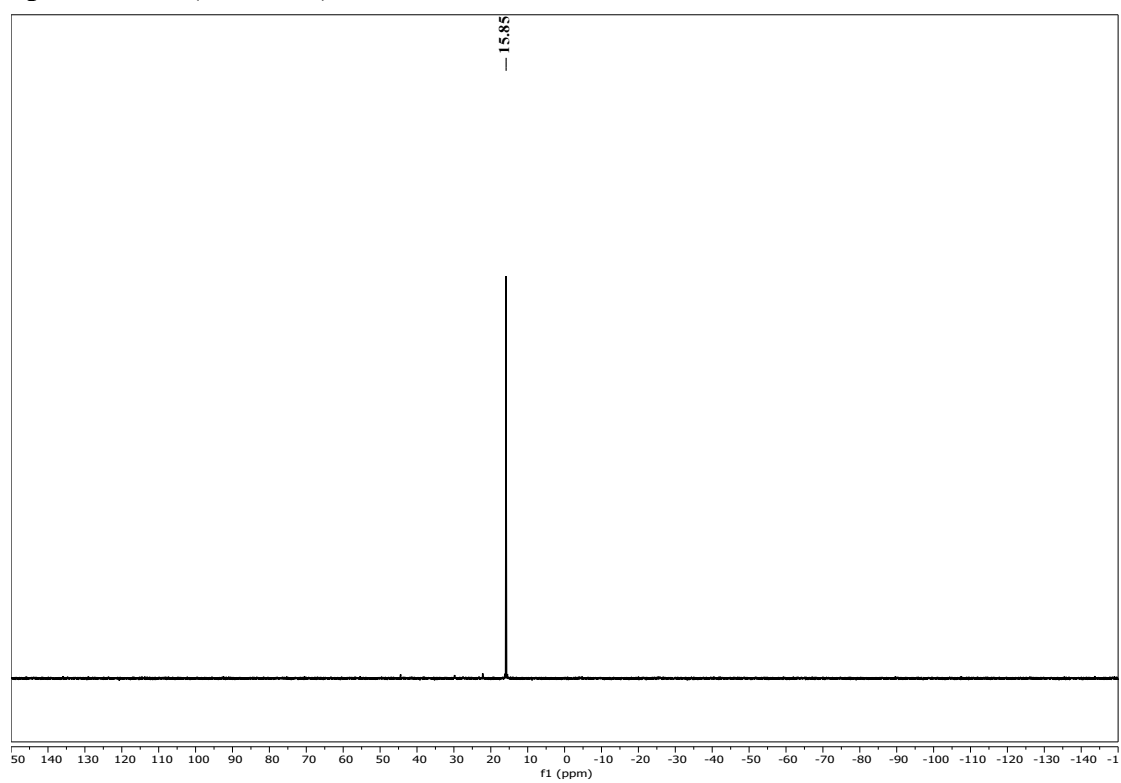


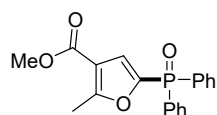


3p, ^{13}C NMR (101 MHz), CDCl_3

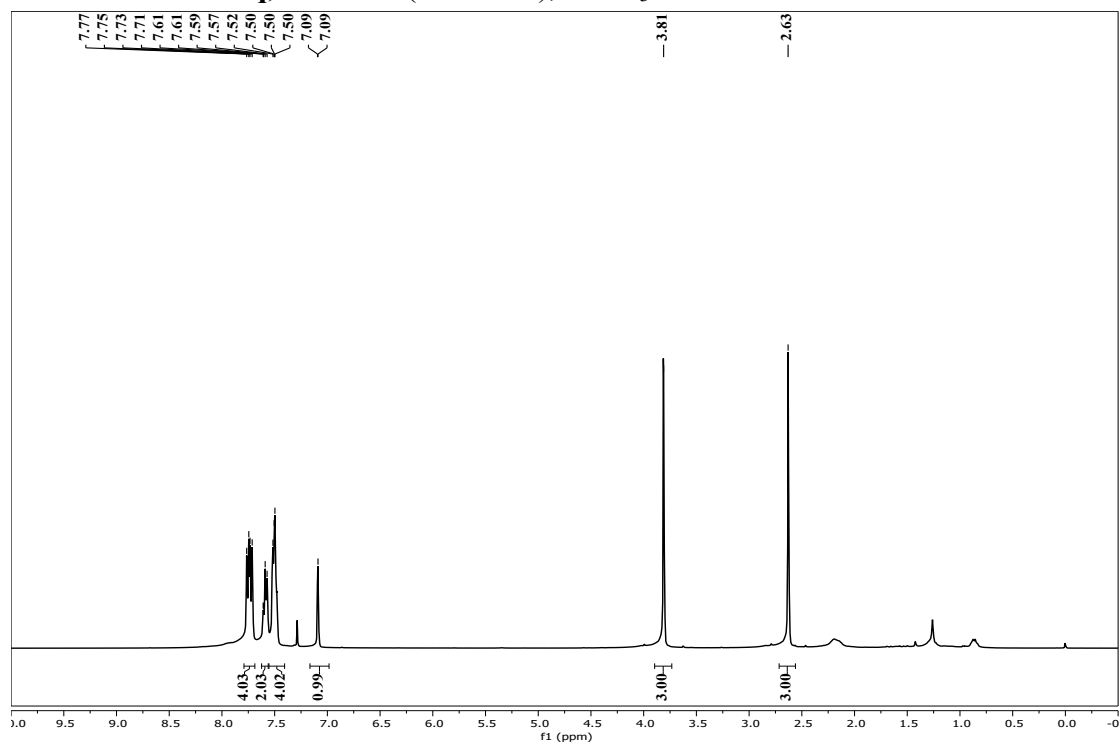


3p, ^{31}P NMR (162 MHz), CDCl_3

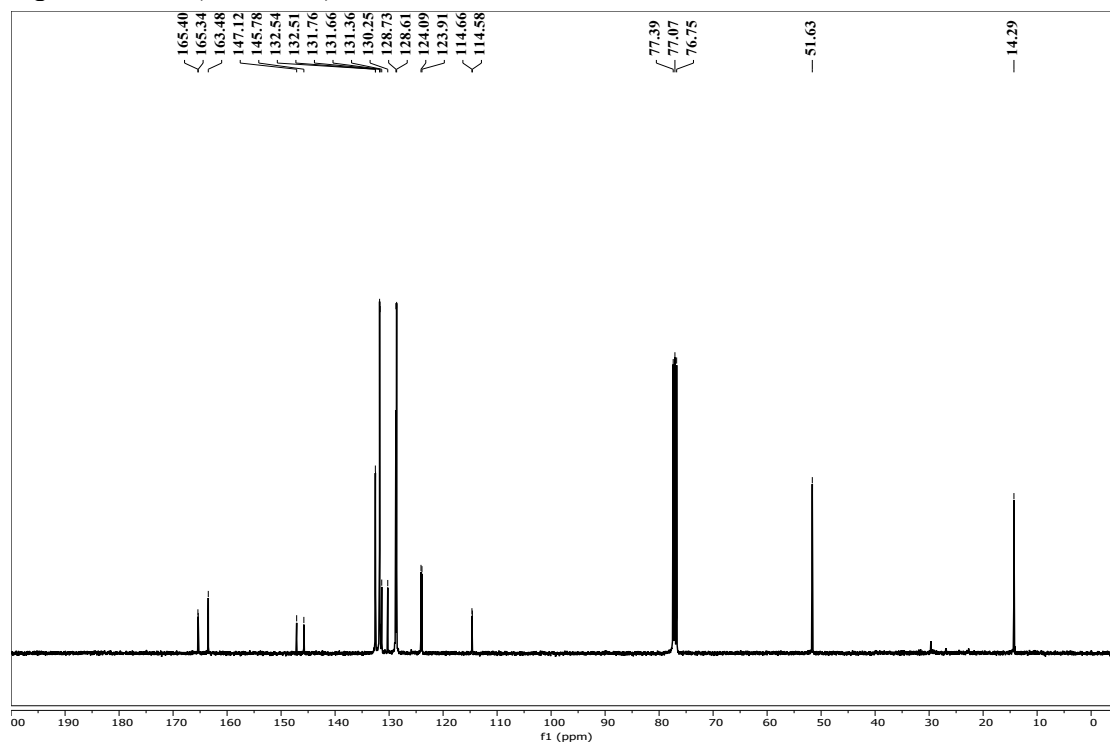




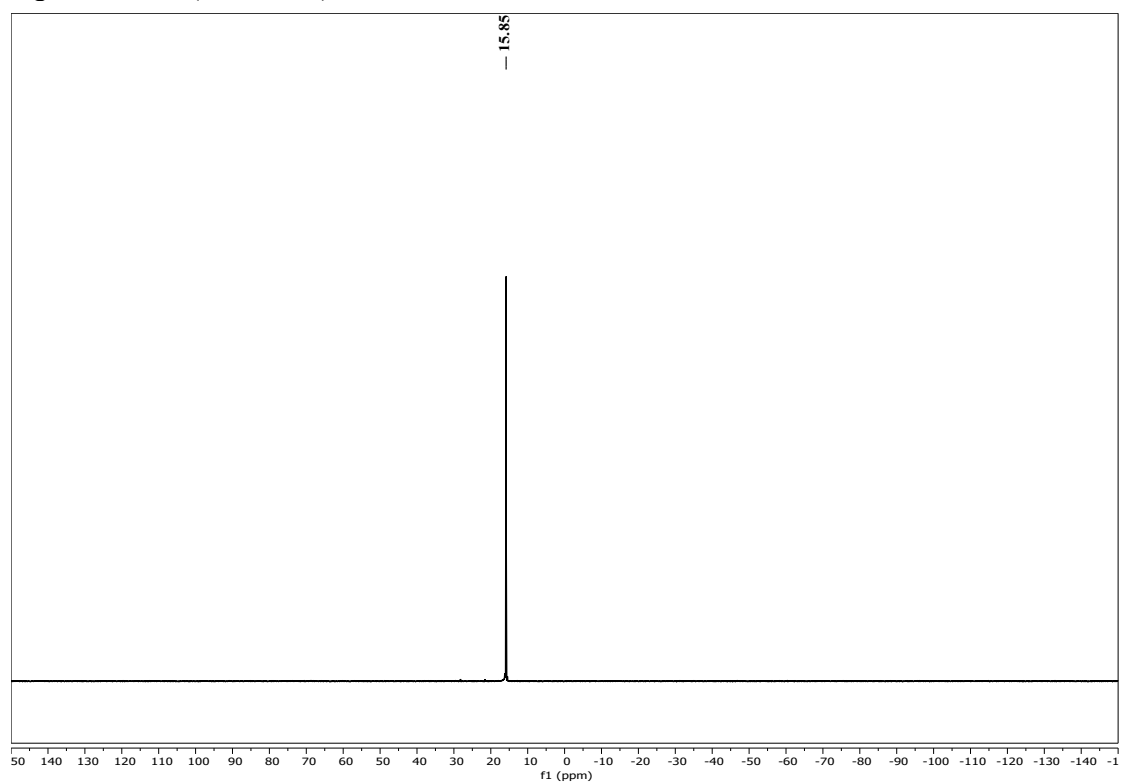
3q, ^1H NMR (400 MHz), CDCl_3

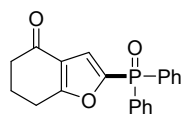


3q, ^{13}C NMR (101 MHz), CDCl_3

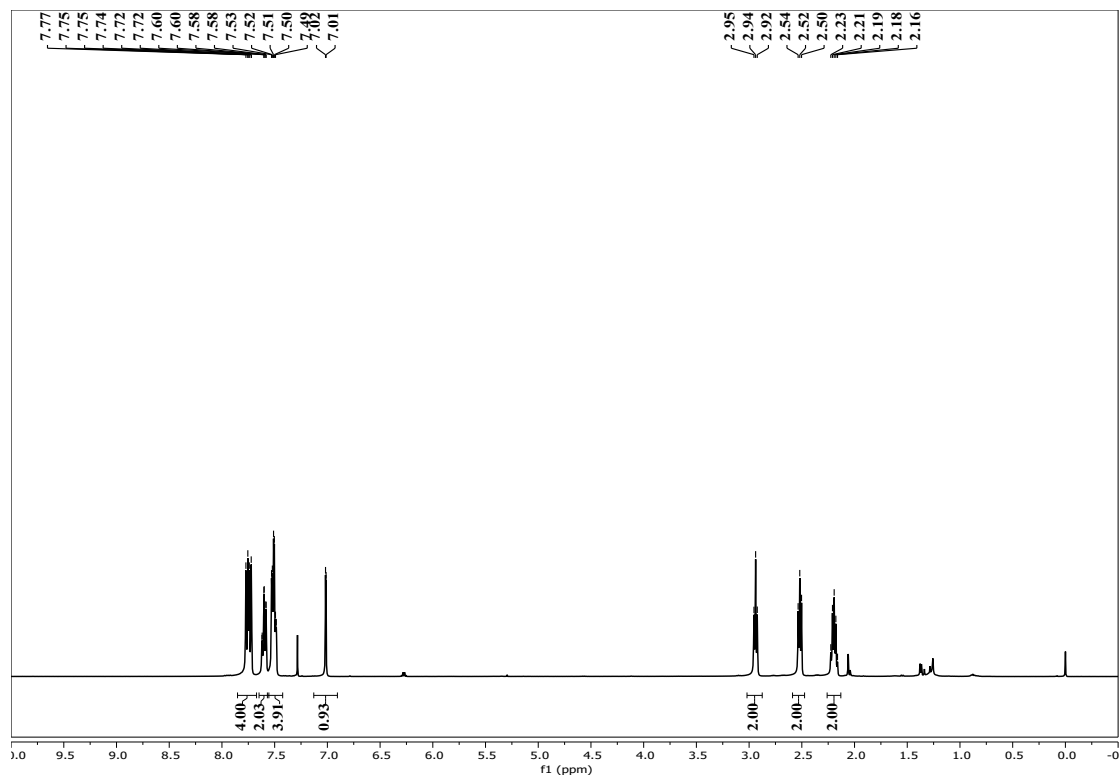


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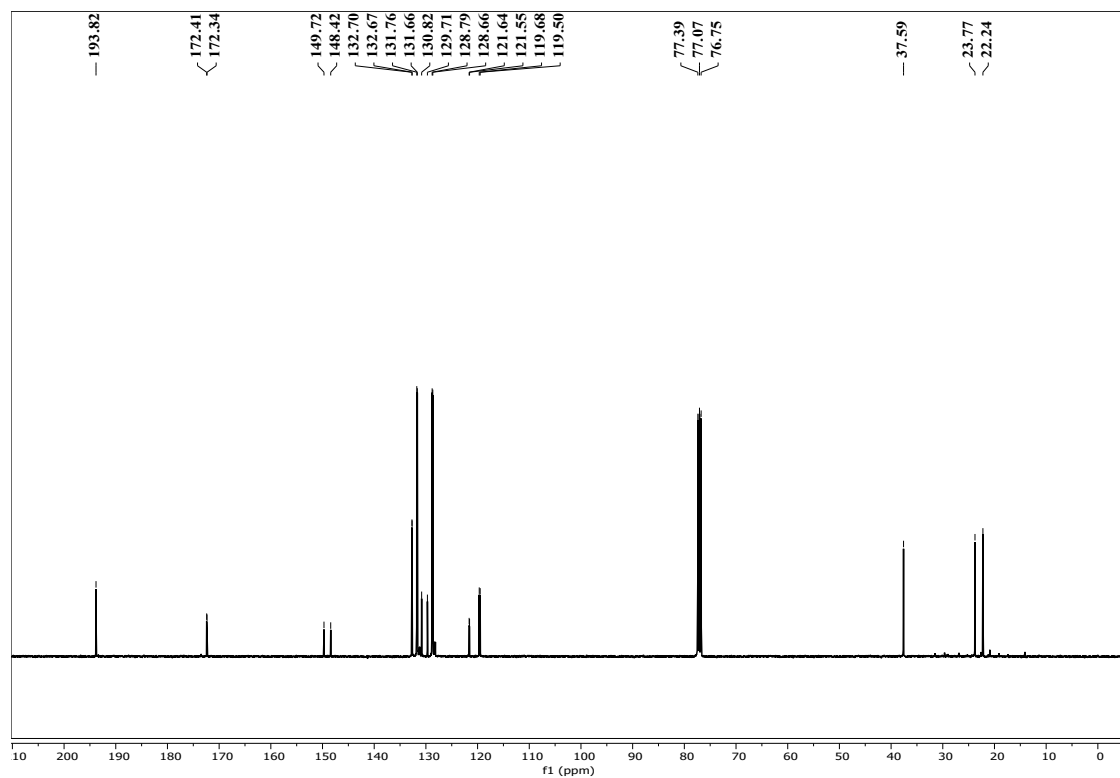




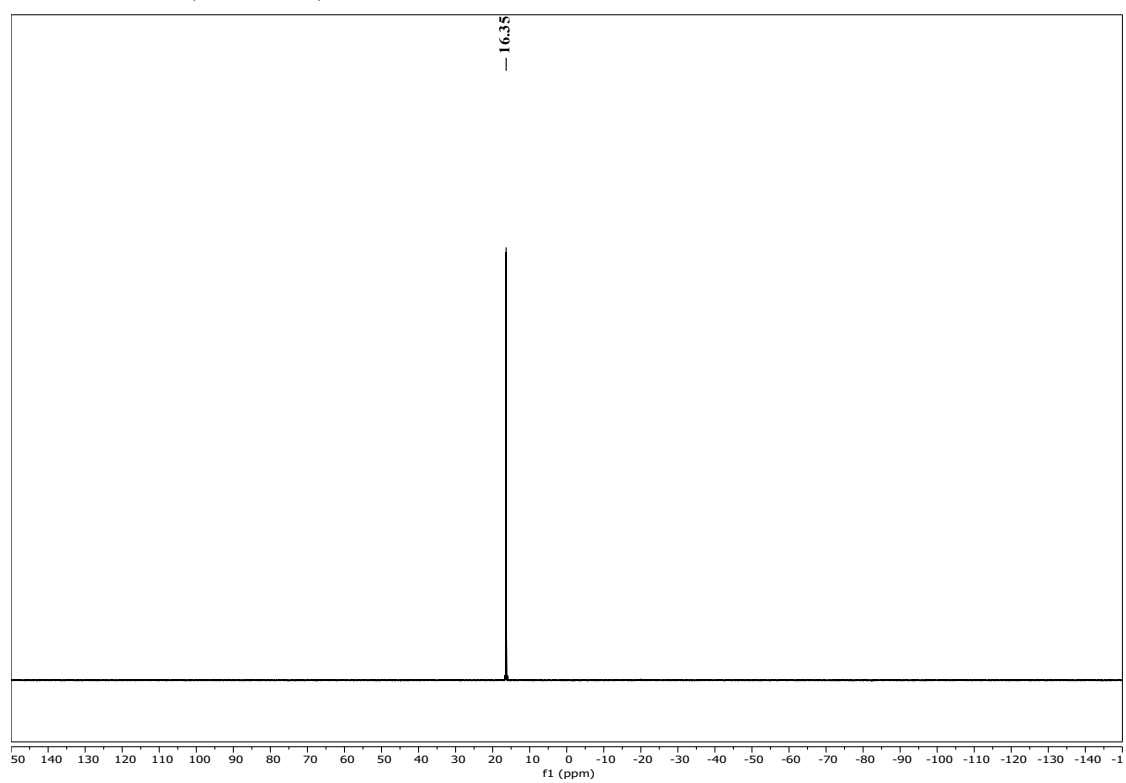
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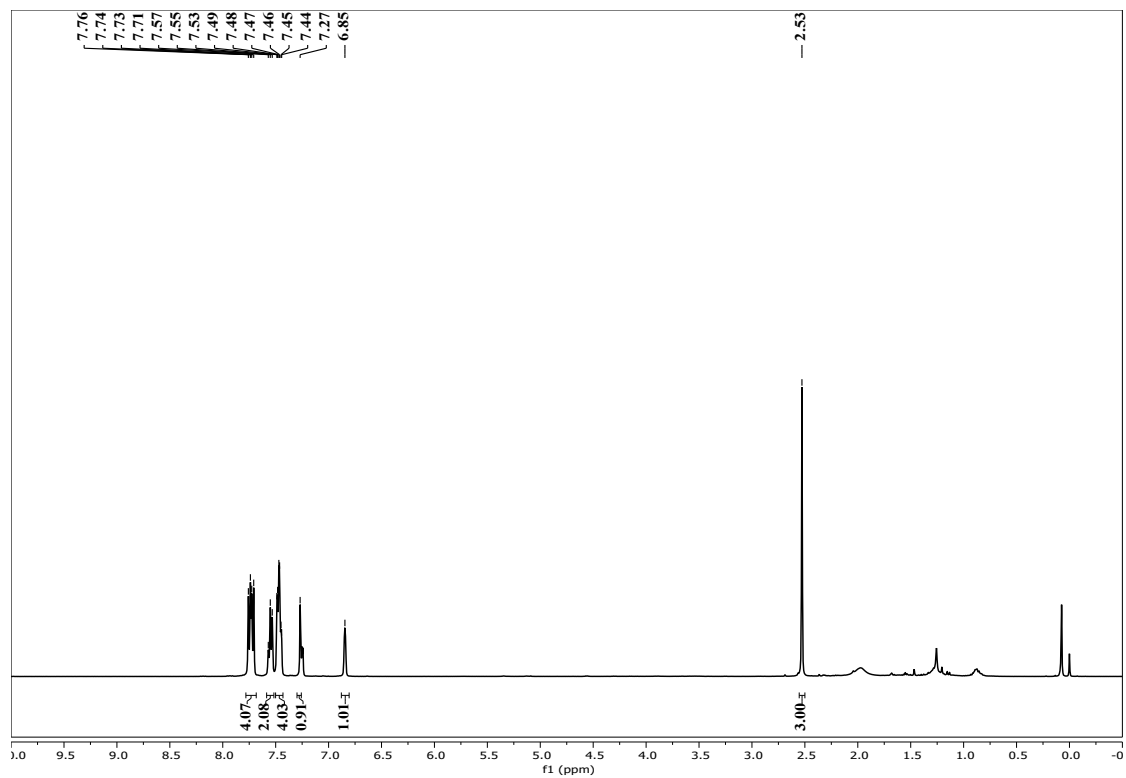


3r, ^{13}C NMR (101 MHz), CDCl_3

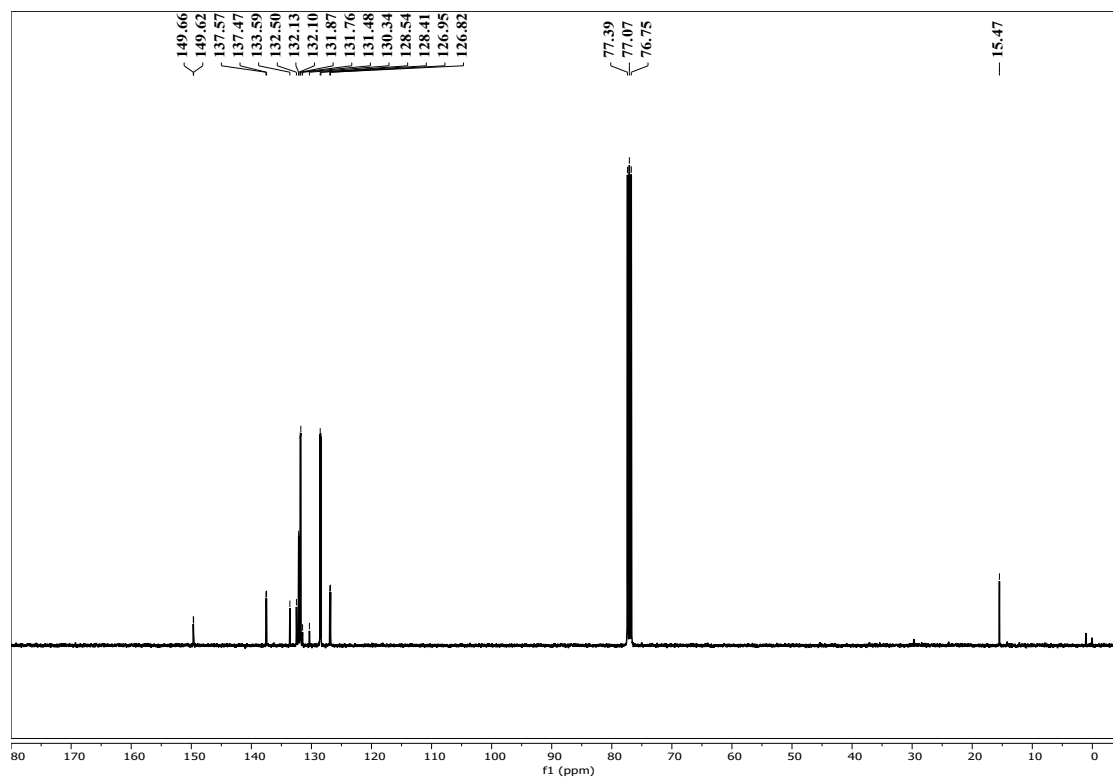


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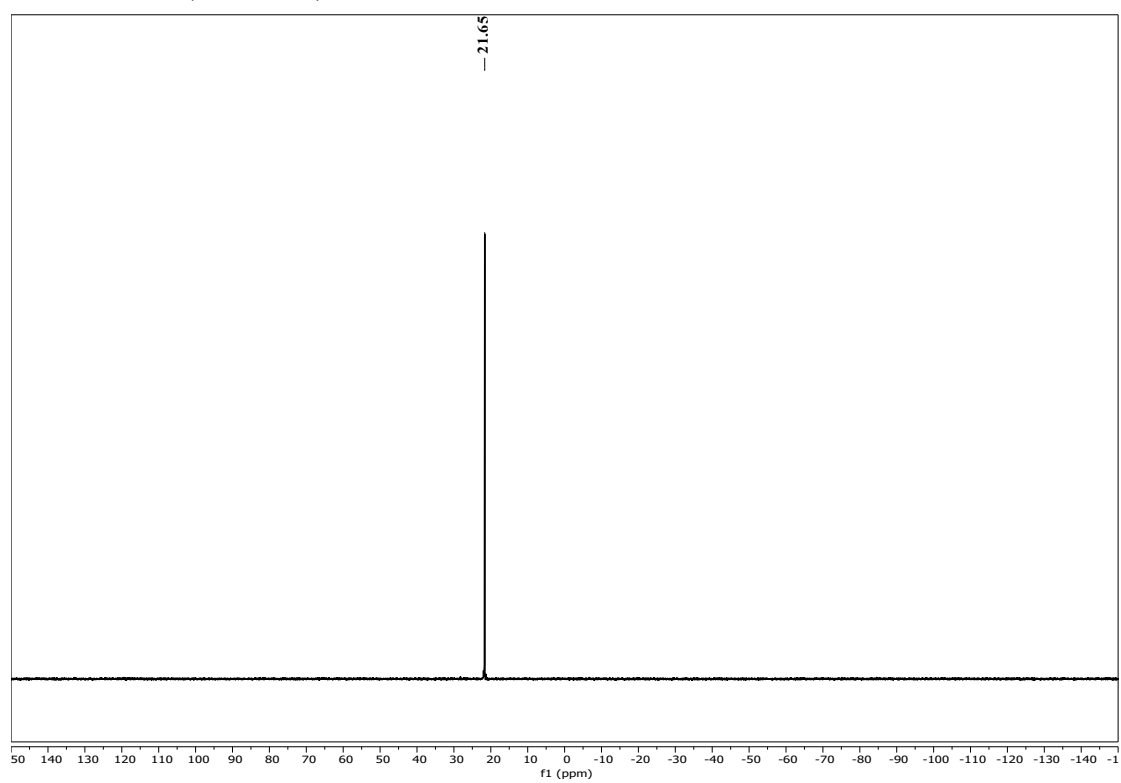


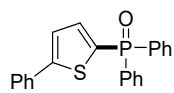


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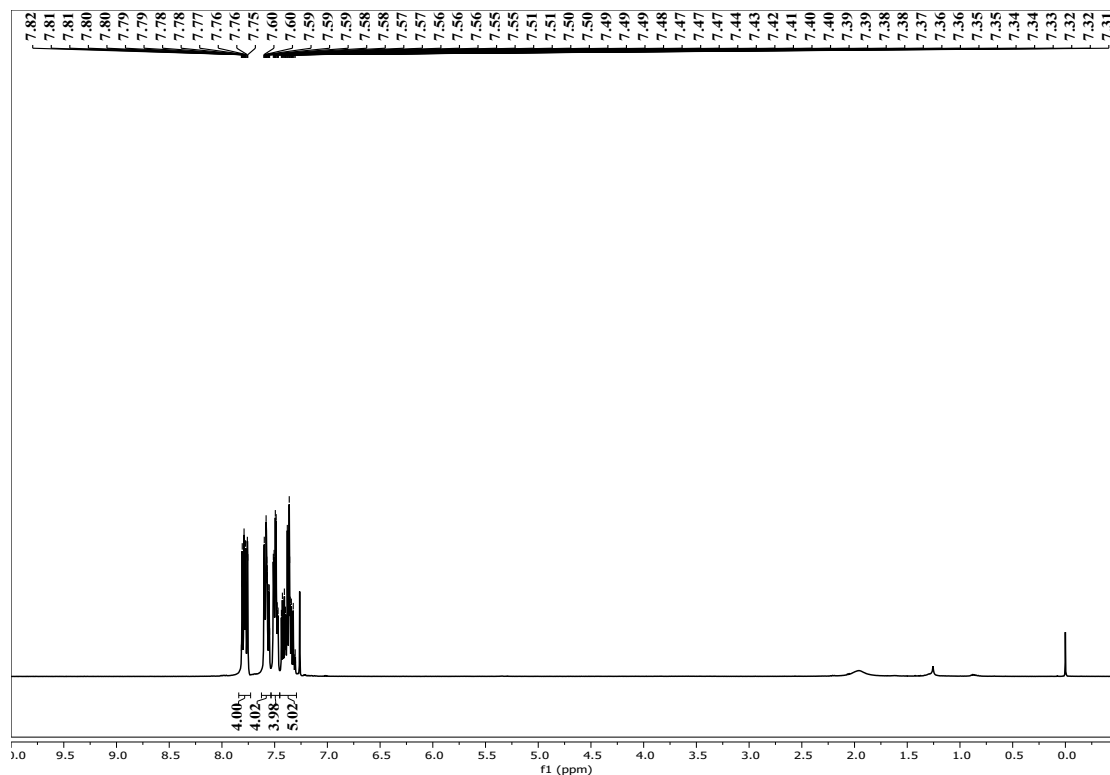


3s, ^{31}P NMR (162 MHz), CDCl_3

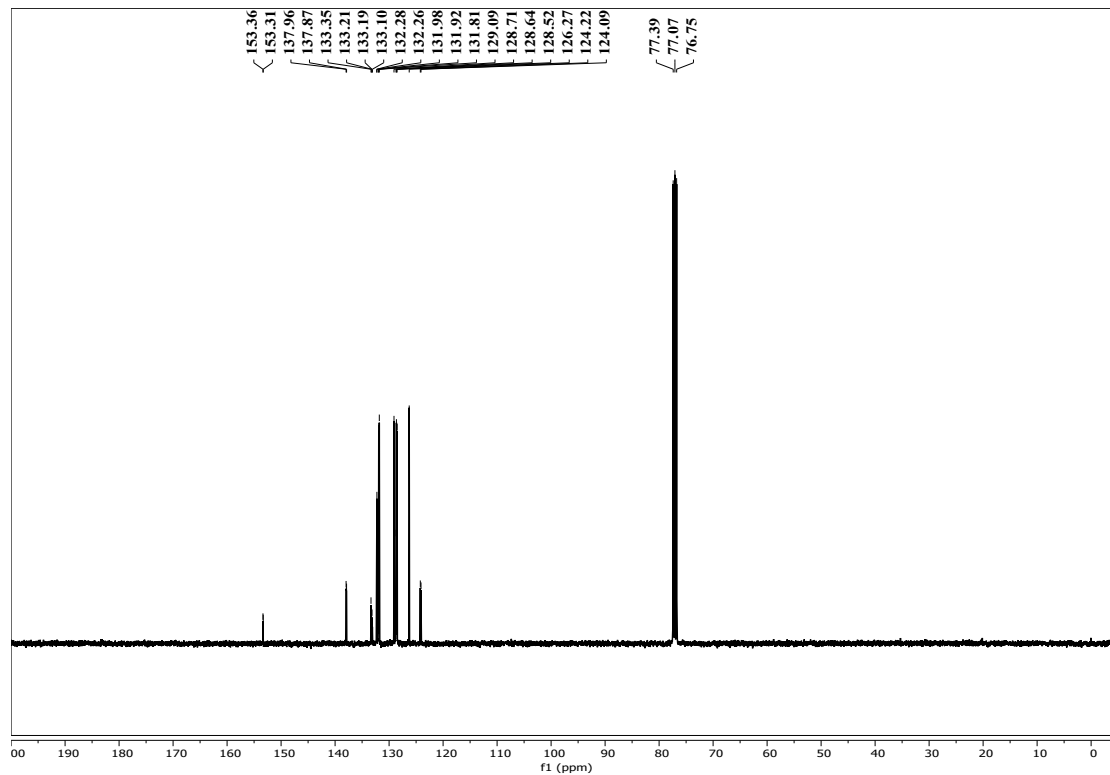




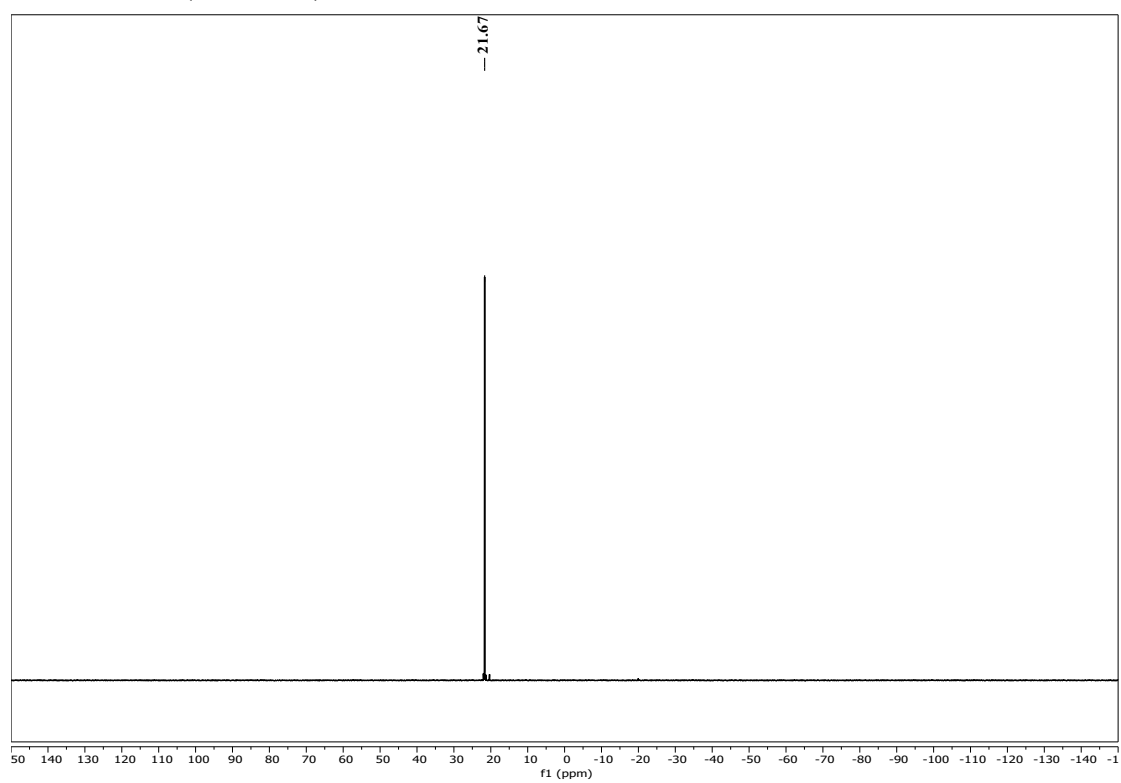
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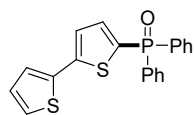


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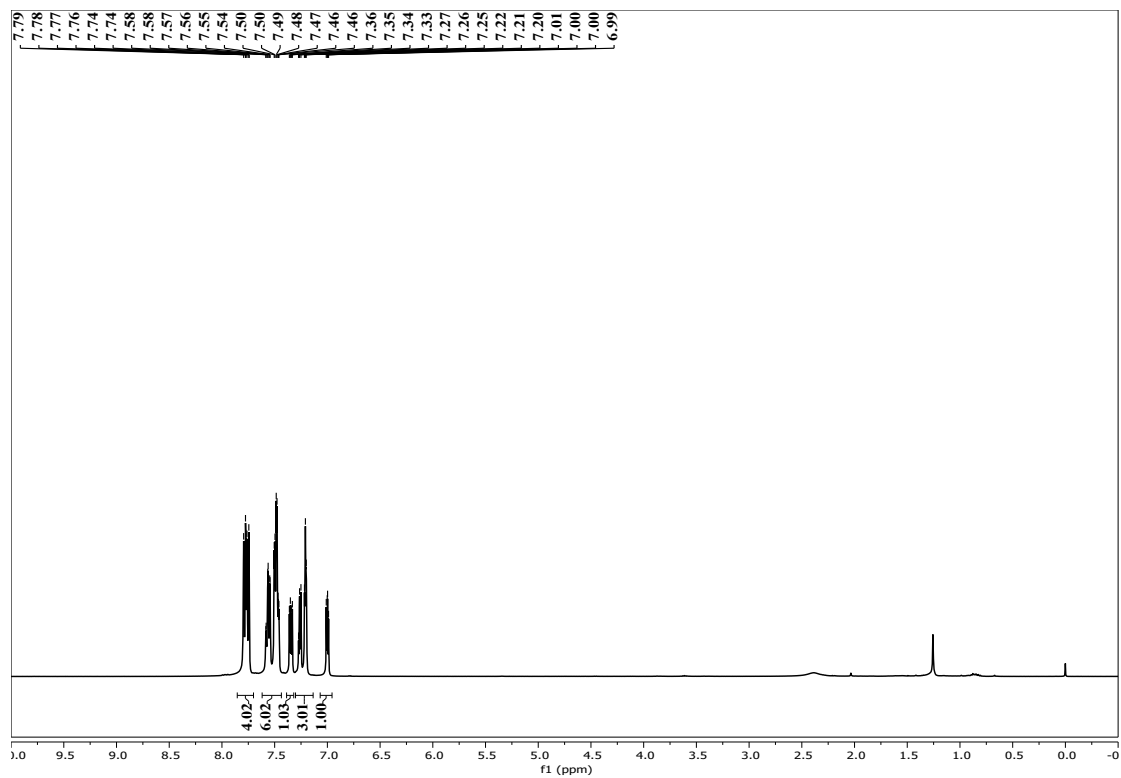


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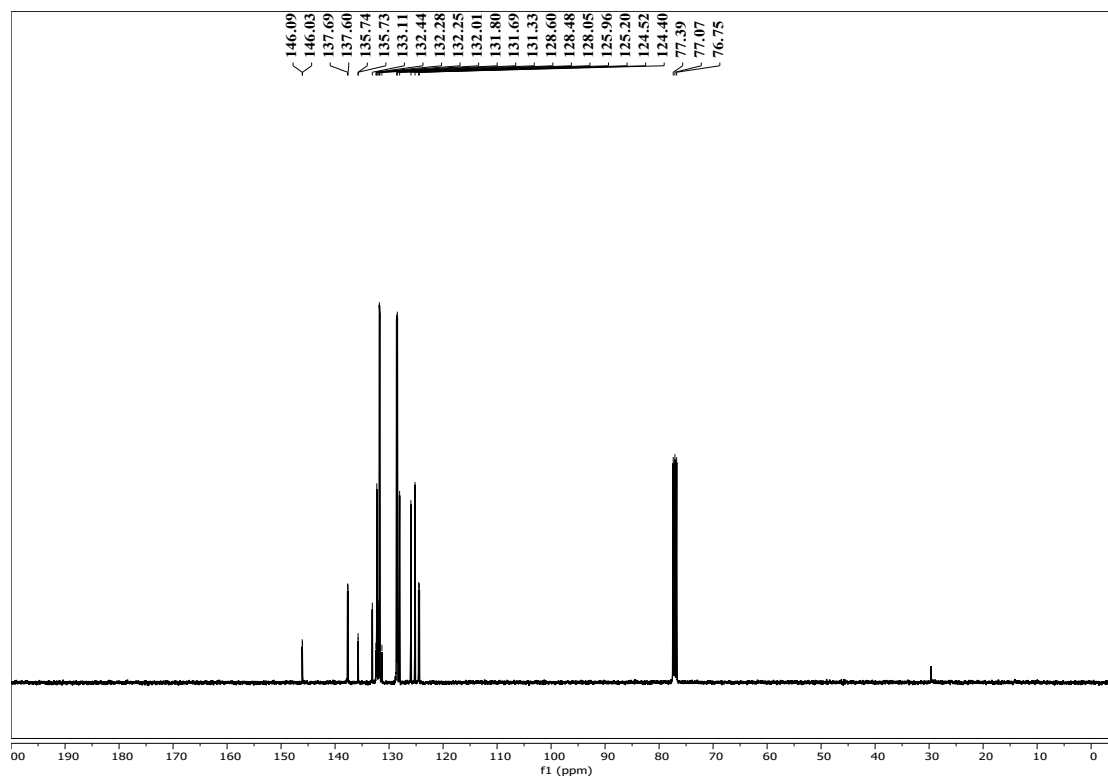




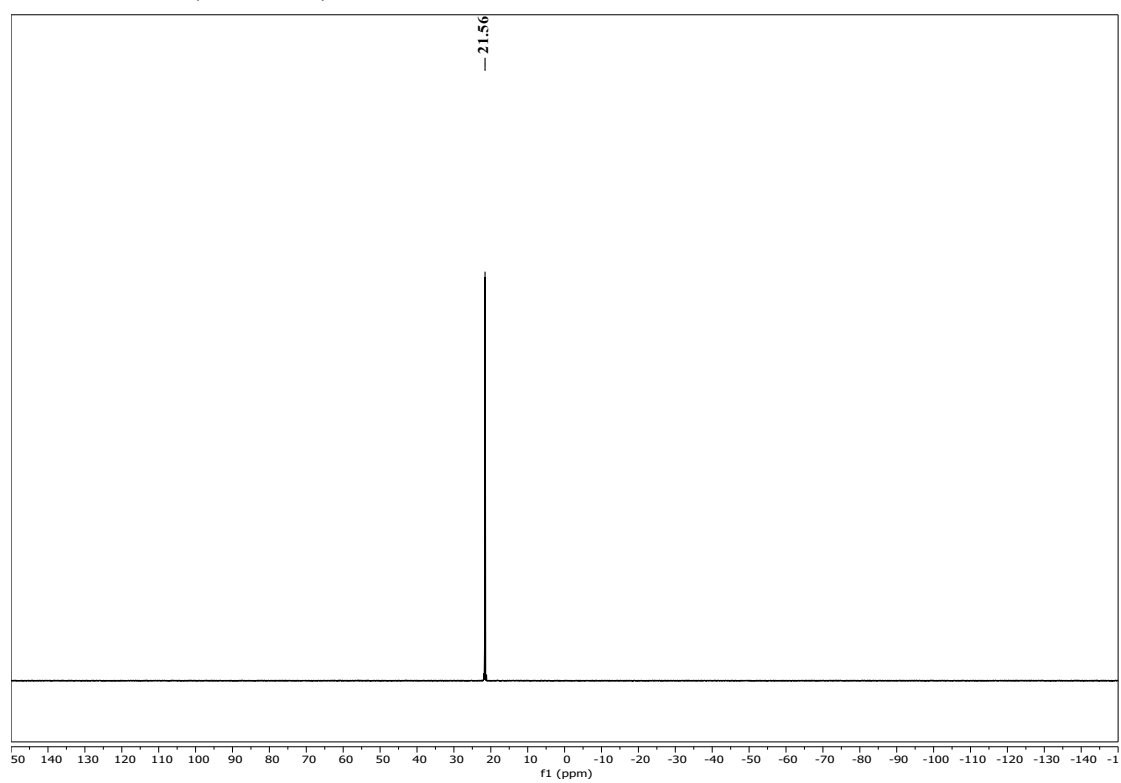
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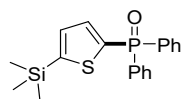


3u, ^{13}C NMR (101 MHz), CDCl_3

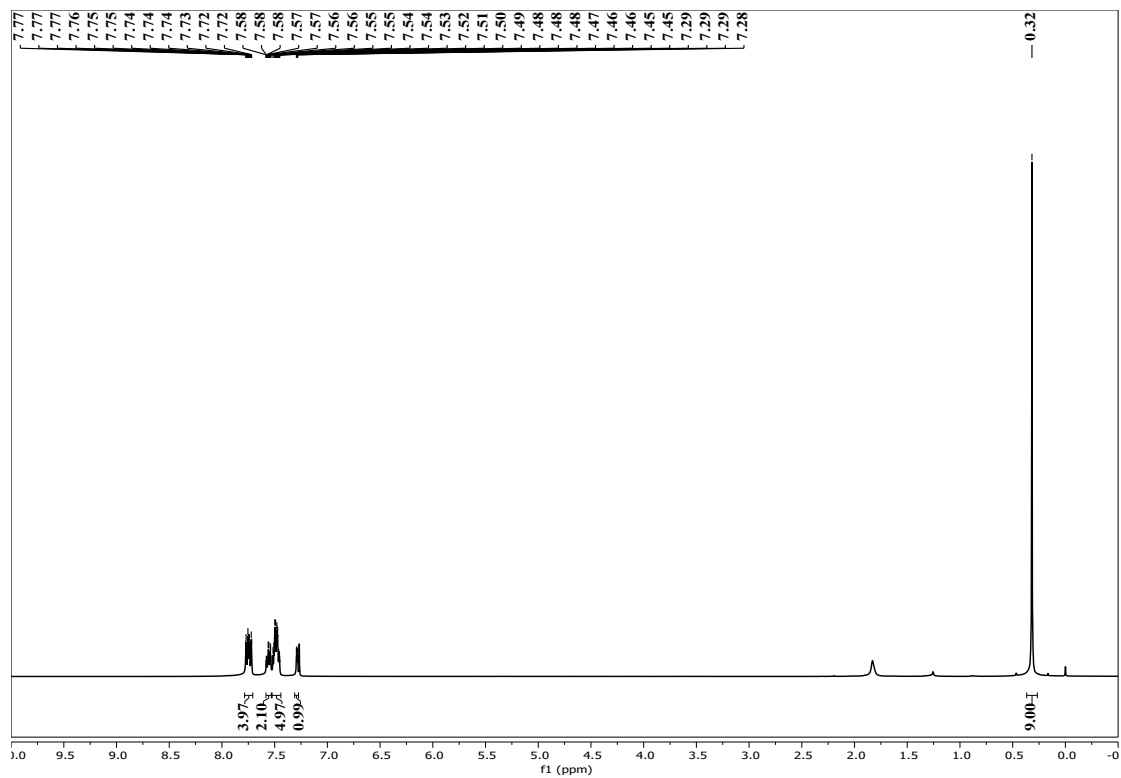


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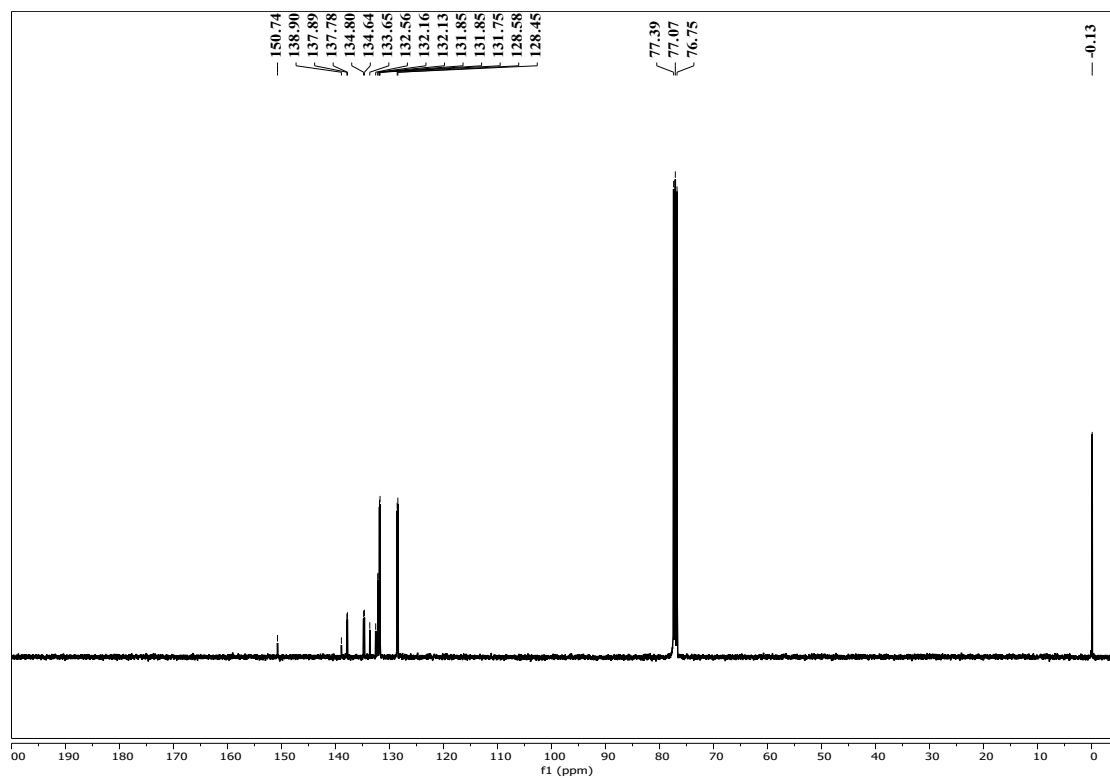




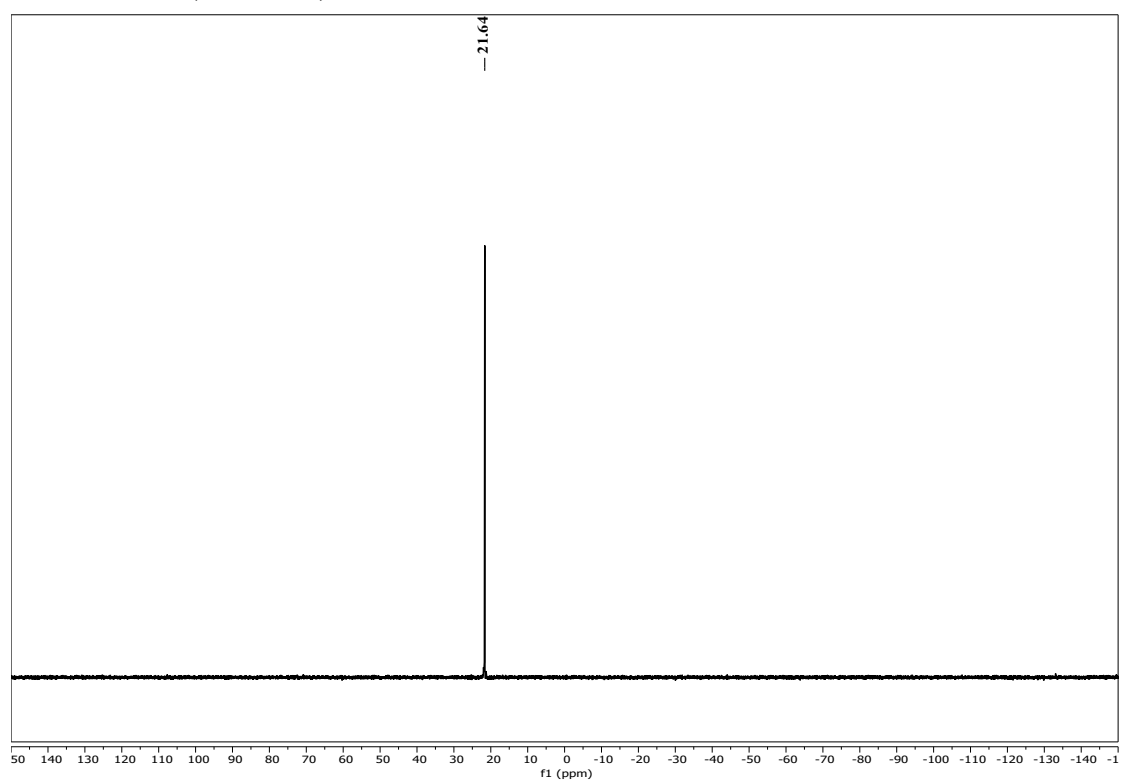
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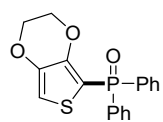


3v, ^{13}C NMR (101 MHz), CDCl_3

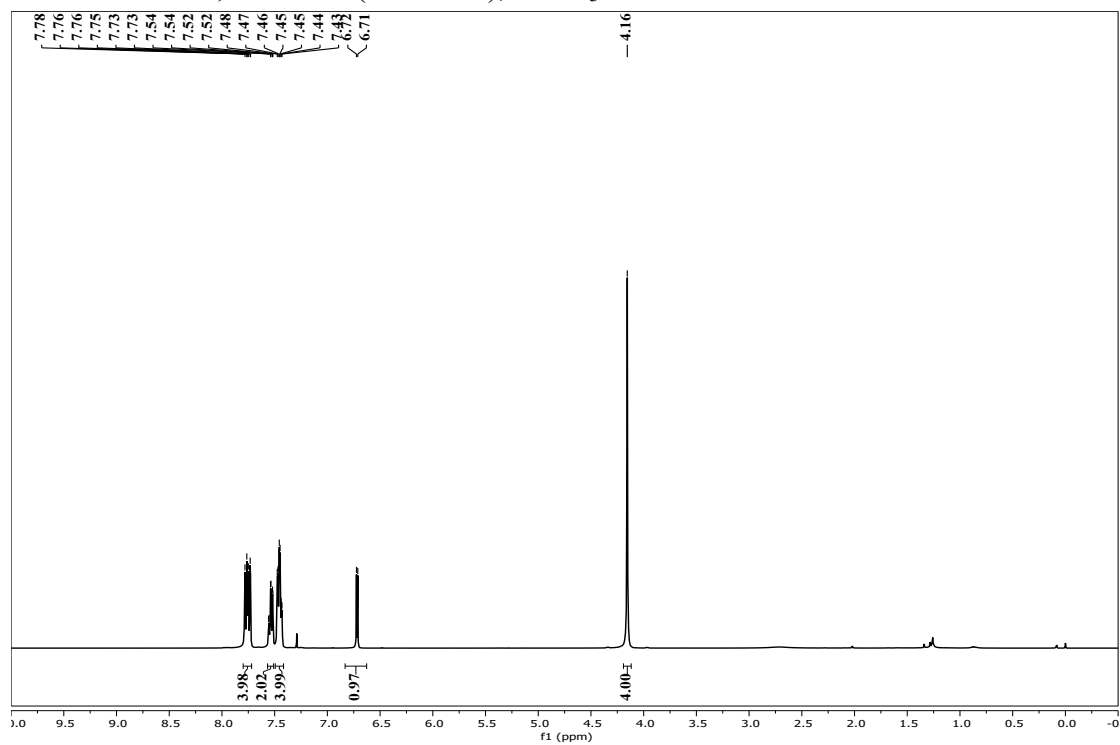


3v, ^{31}P NMR (162 MHz), CDCl_3

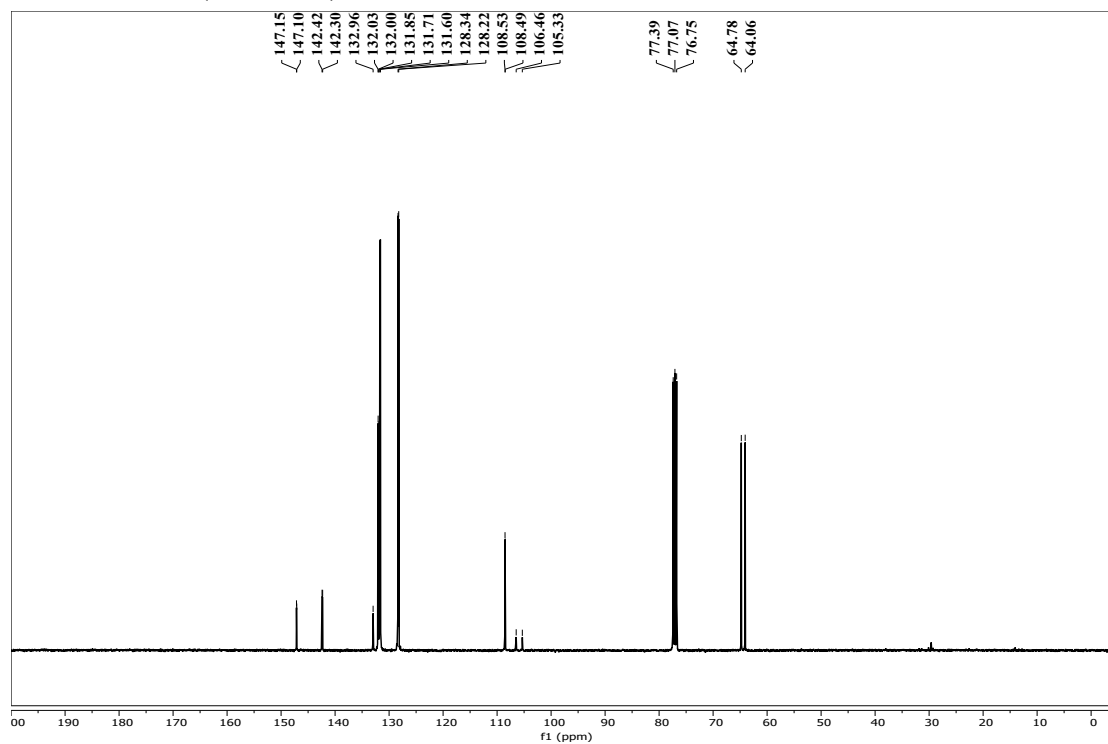




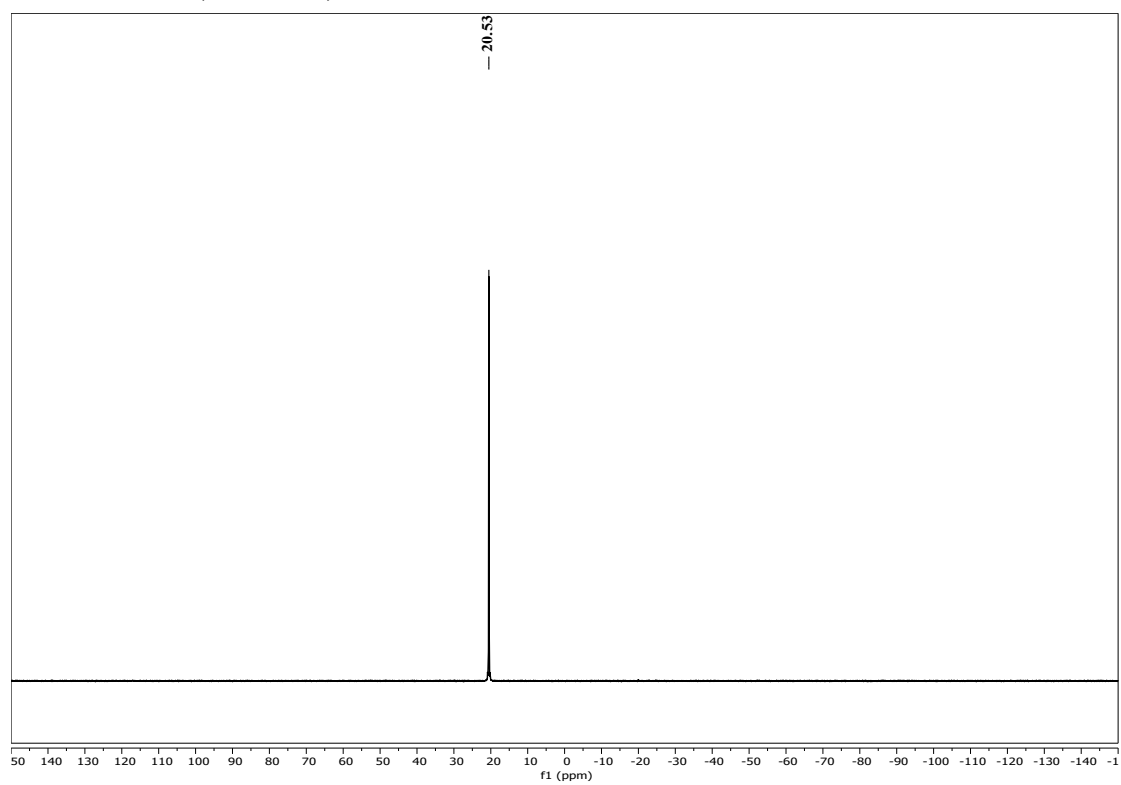
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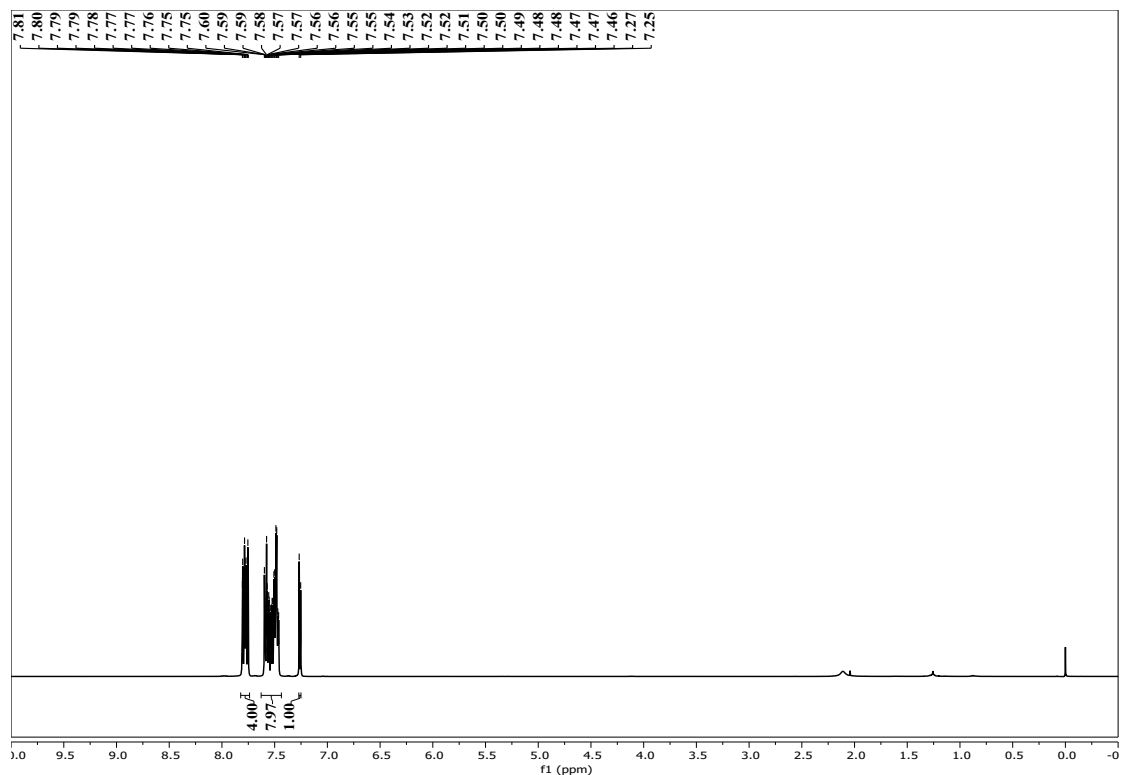


3w, ^{13}C NMR (101 MHz), CDCl_3

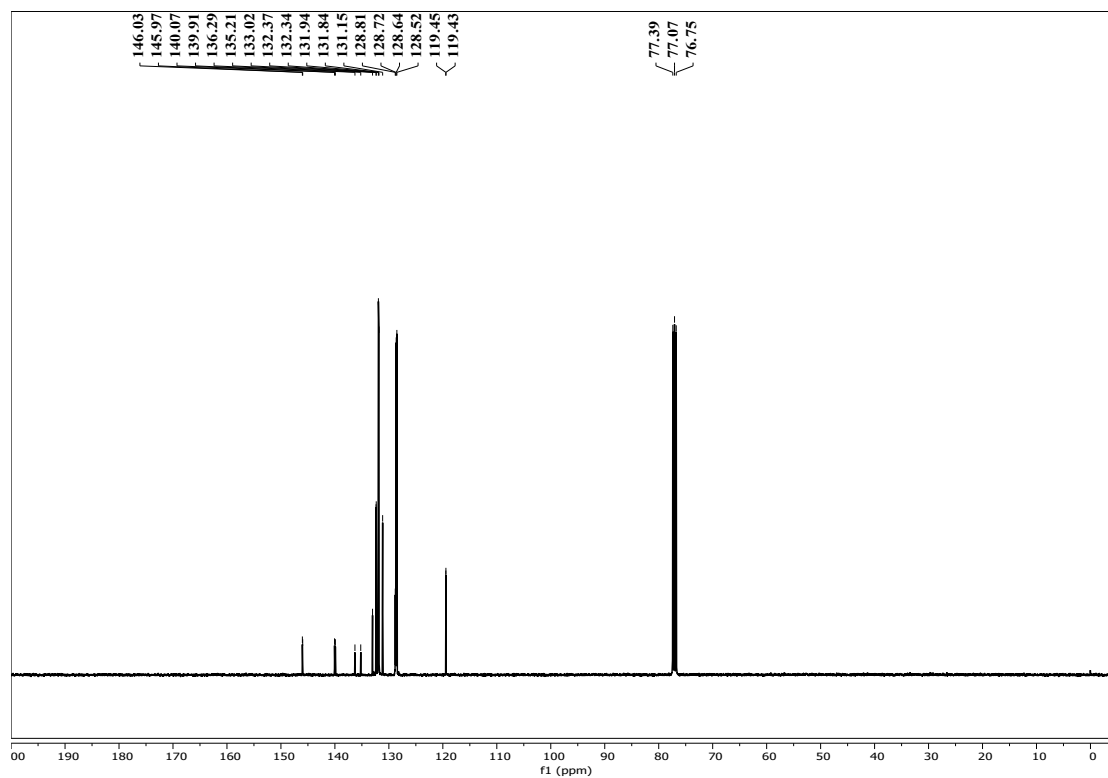


3w, ^{31}P NMR (162 MHz), CDCl_3

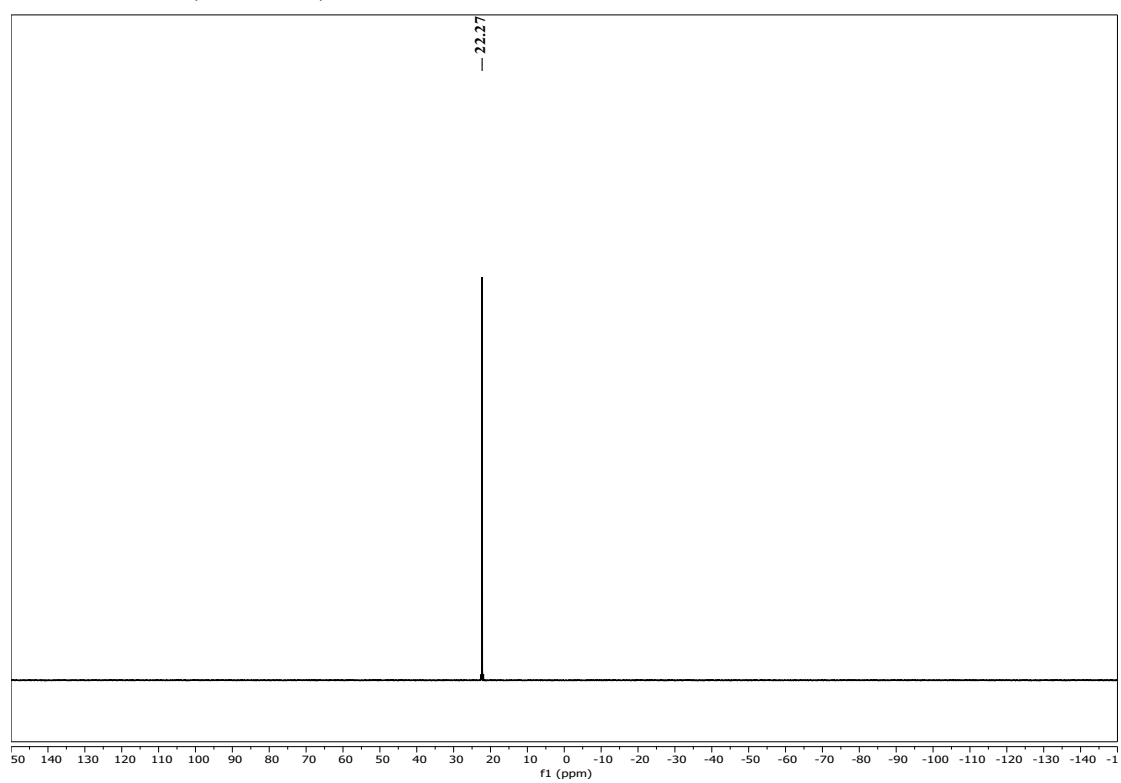


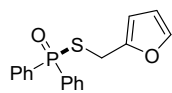


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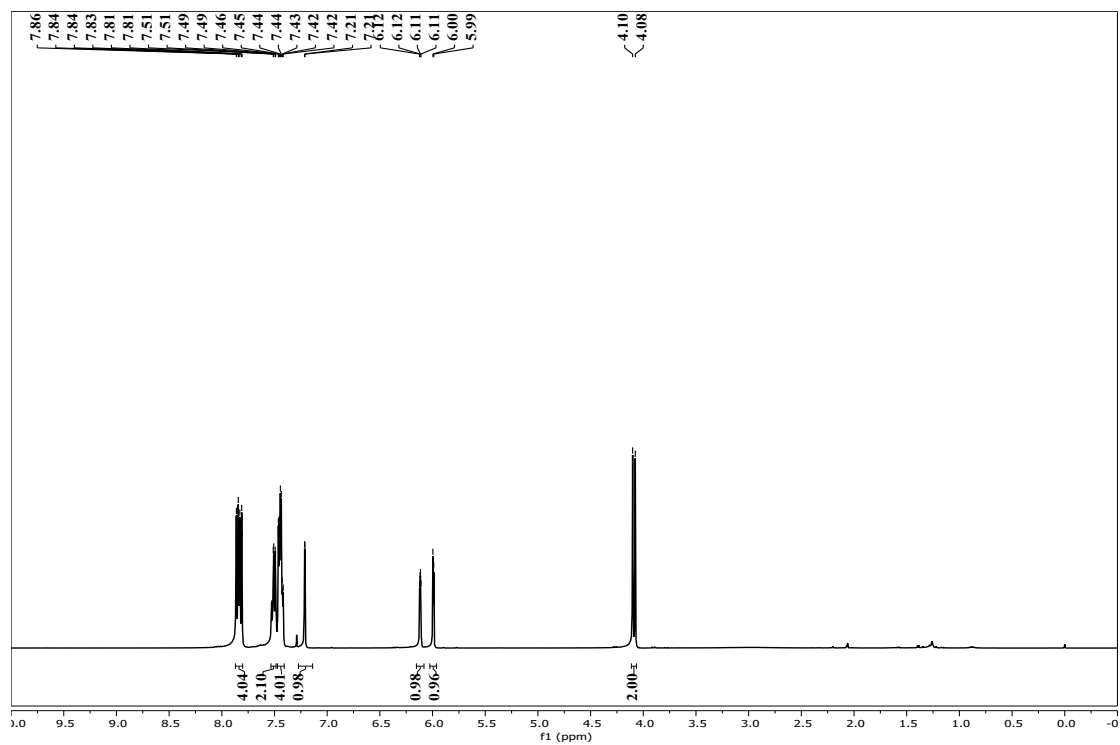


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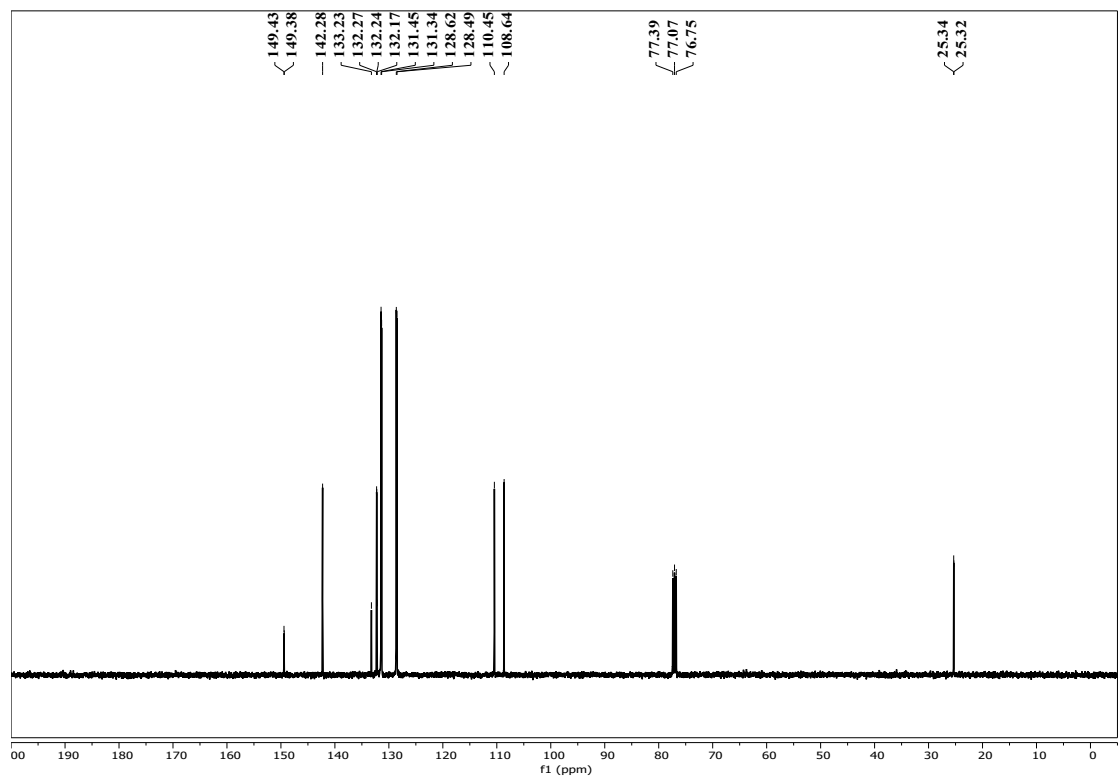




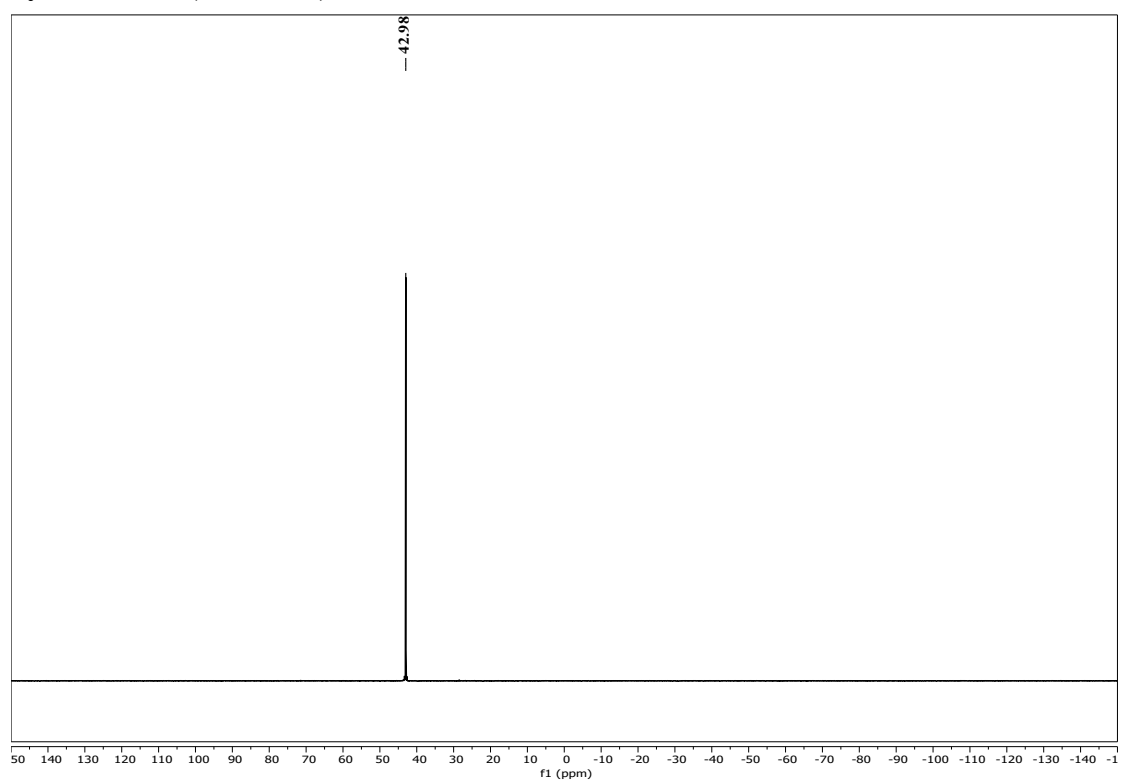
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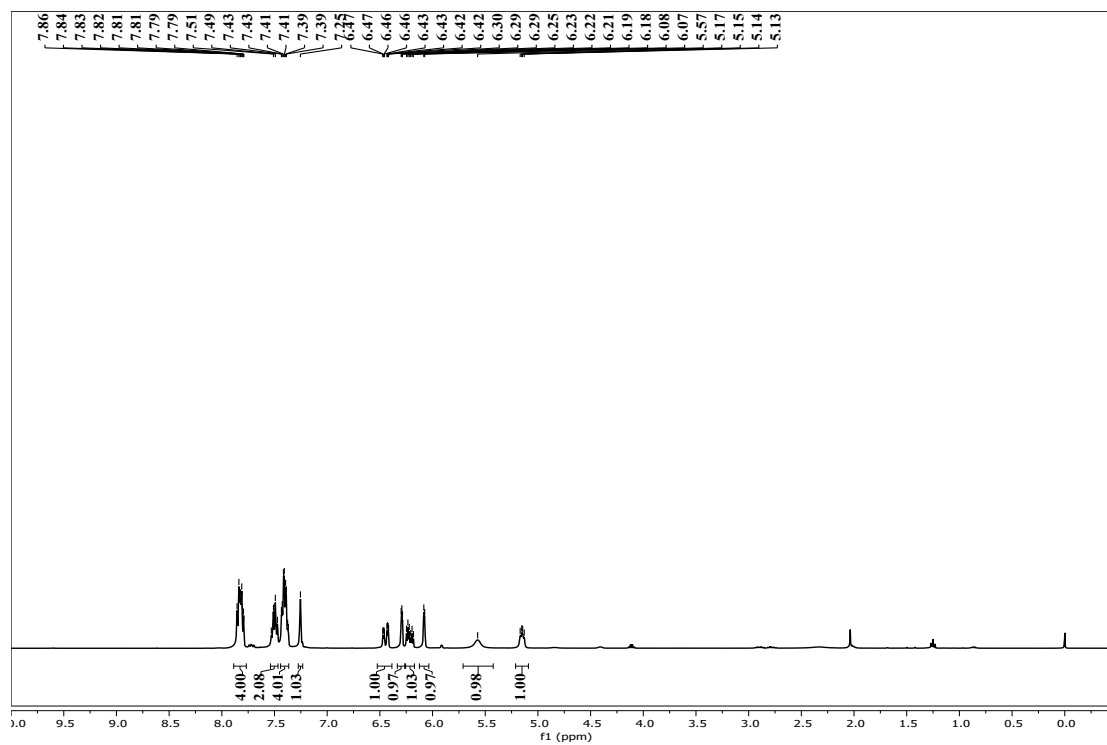


3y, ^{13}C NMR (101 MHz), CDCl_3

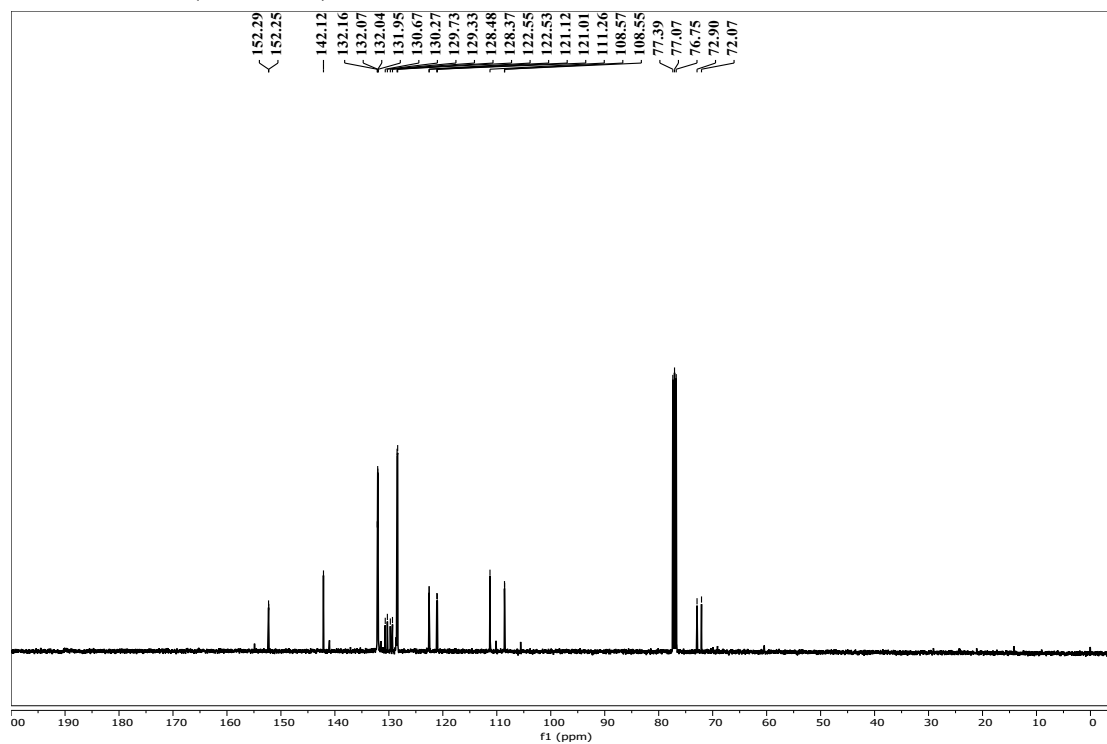


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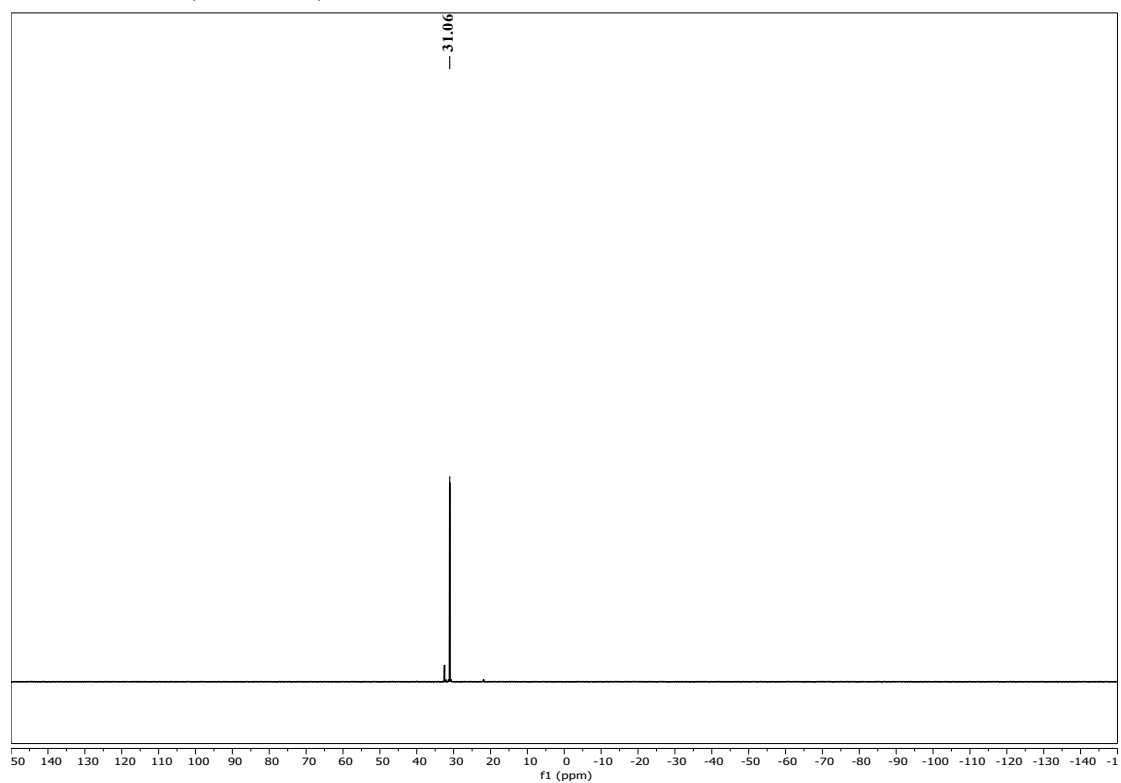


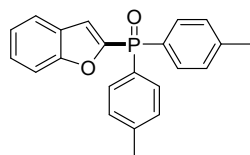


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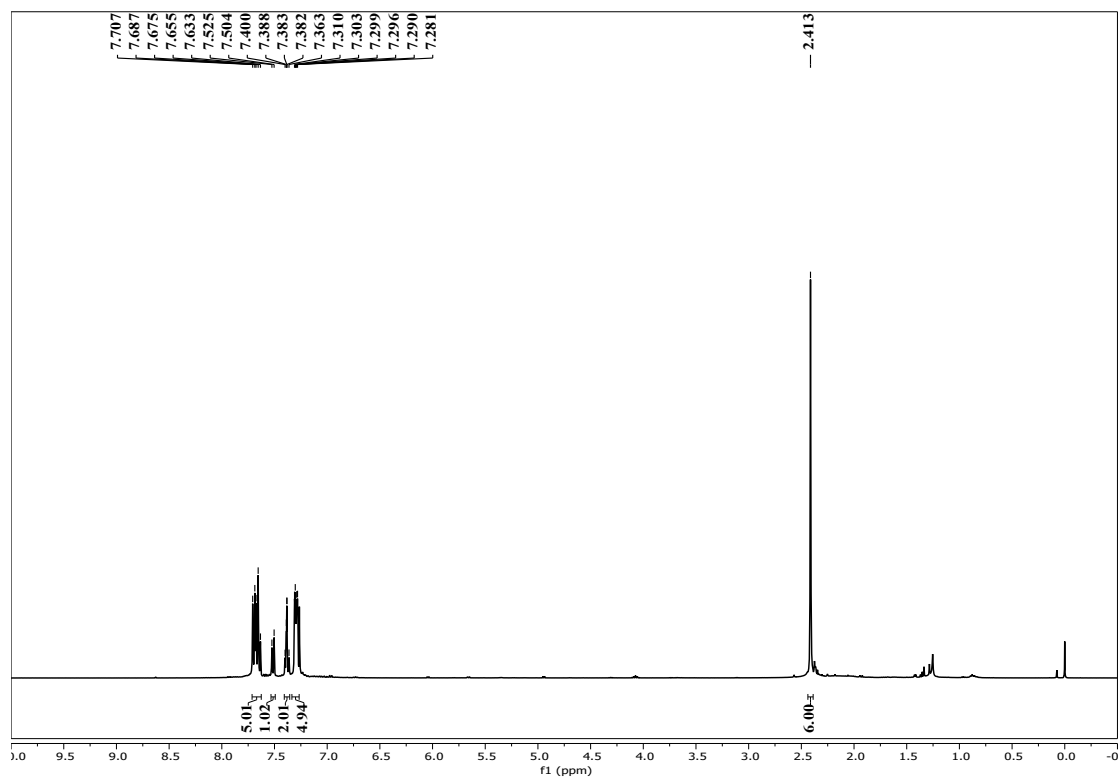


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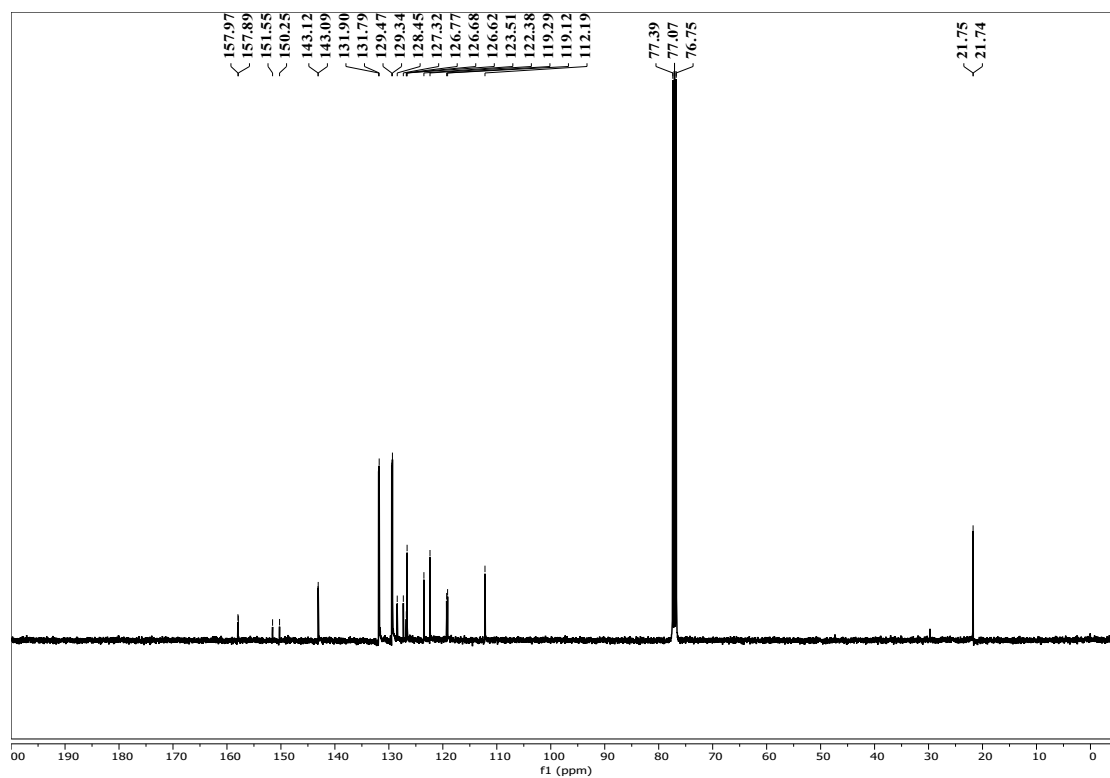




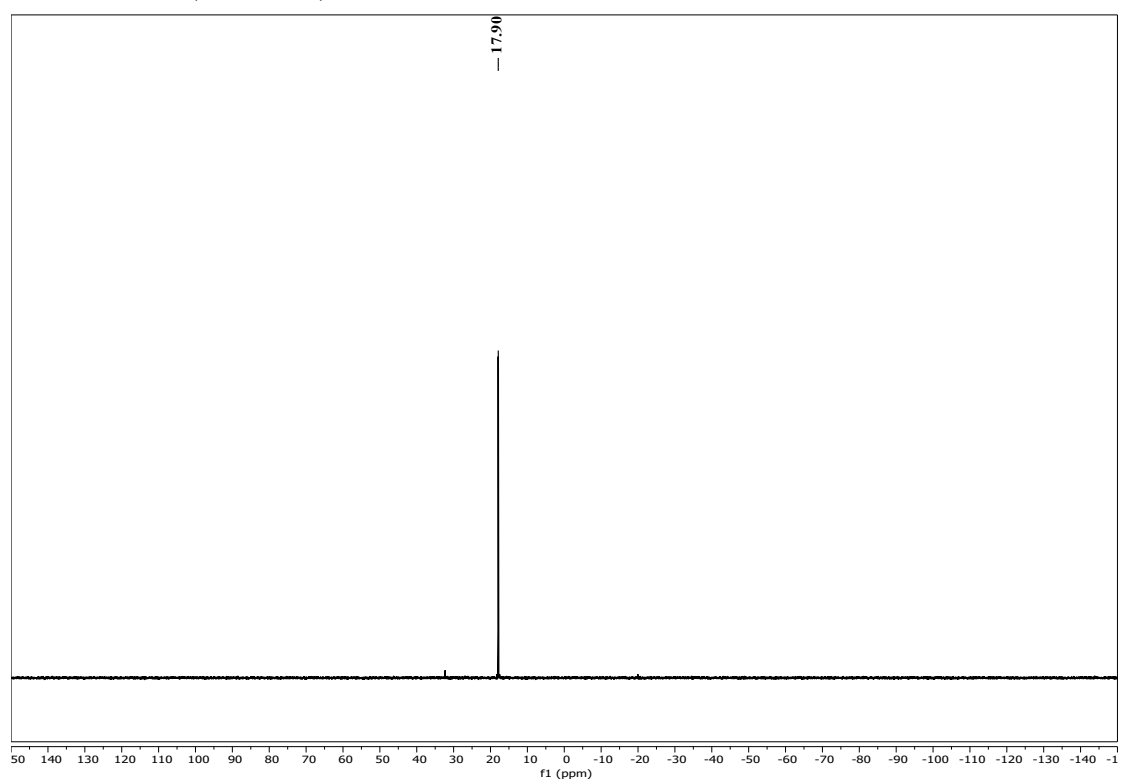
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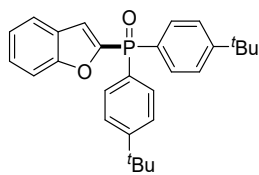


4a, ^{13}C NMR (101 MHz), CDCl_3

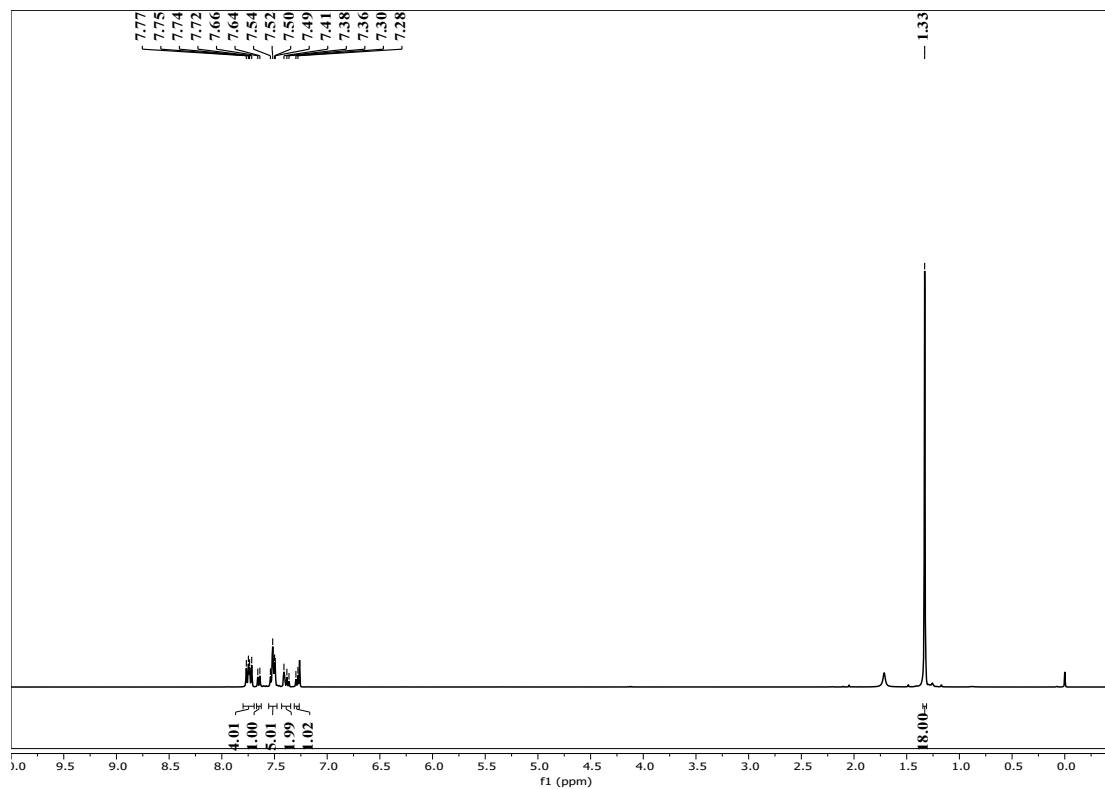


4a, ^{31}P NMR (162 MHz), CDCl_3

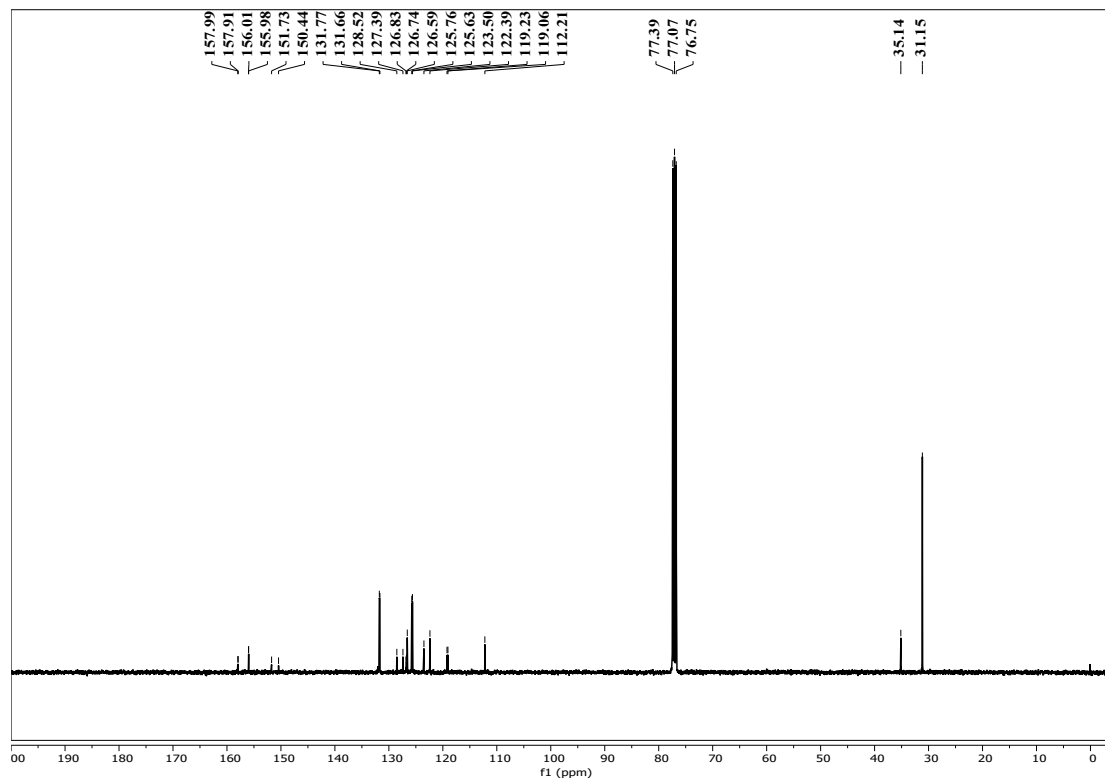




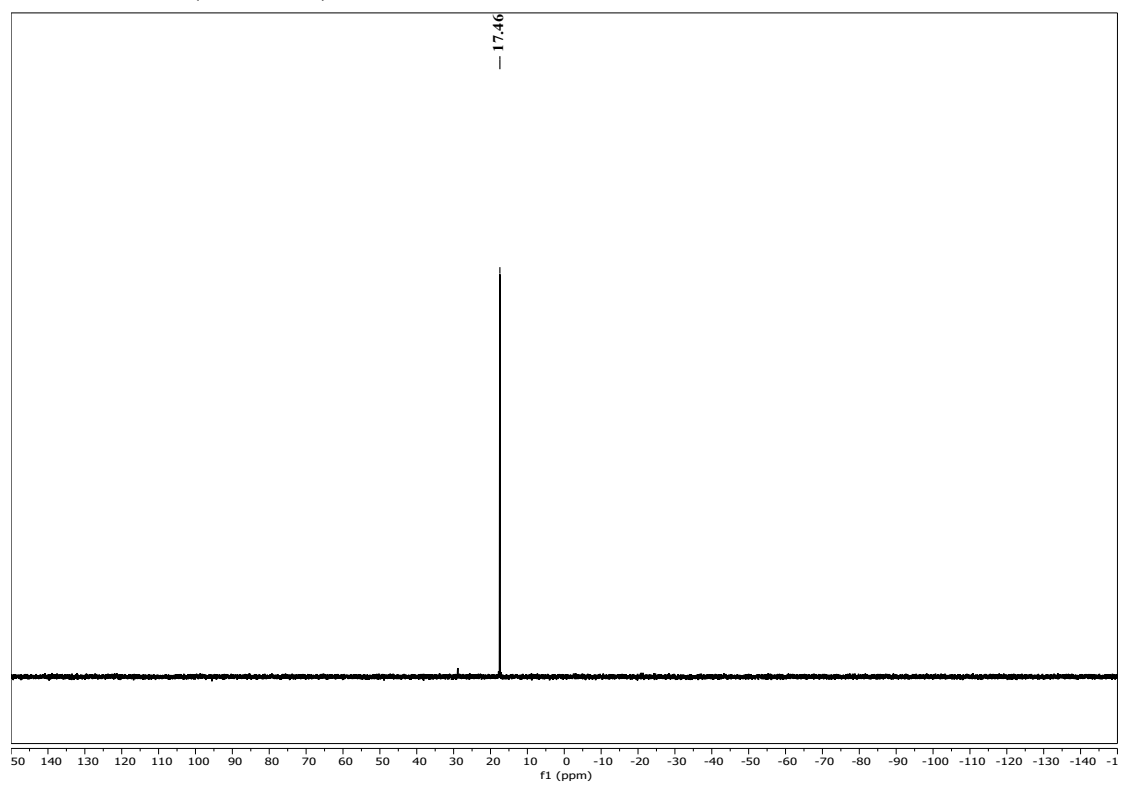
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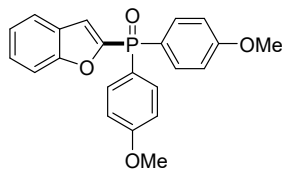


4b, ^{13}C NMR (101 MHz), CDCl_3

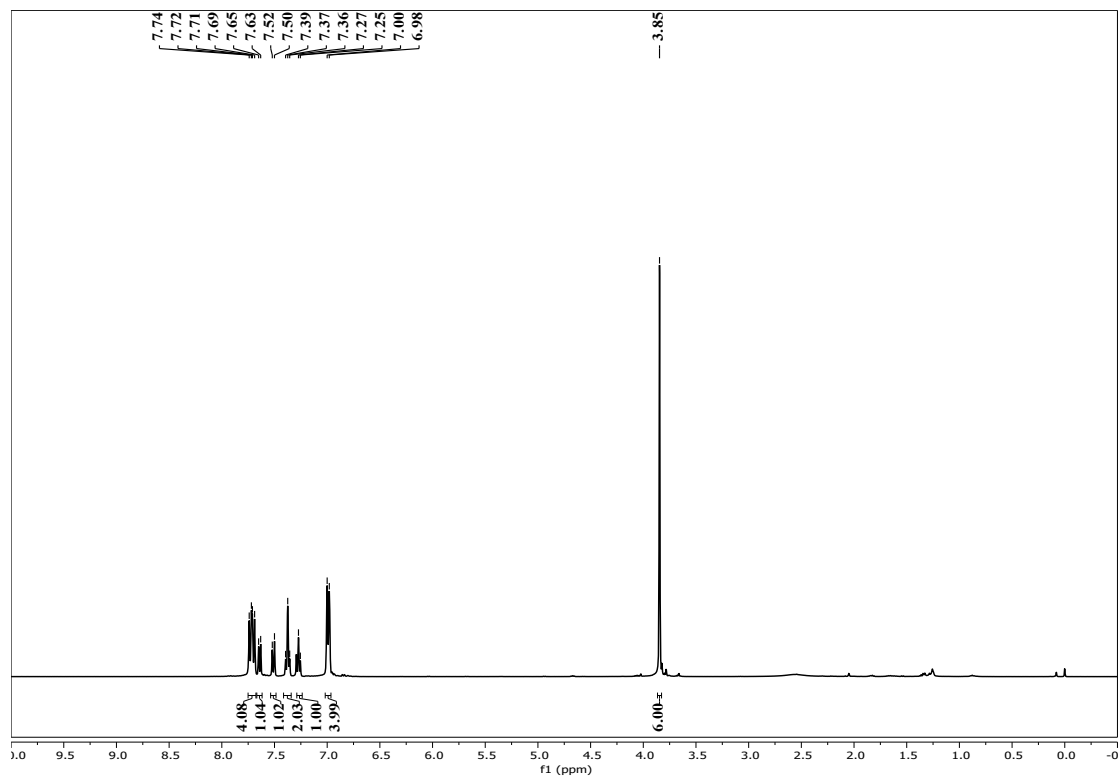


4b, ^{31}P NMR (162 MHz), CDCl_3

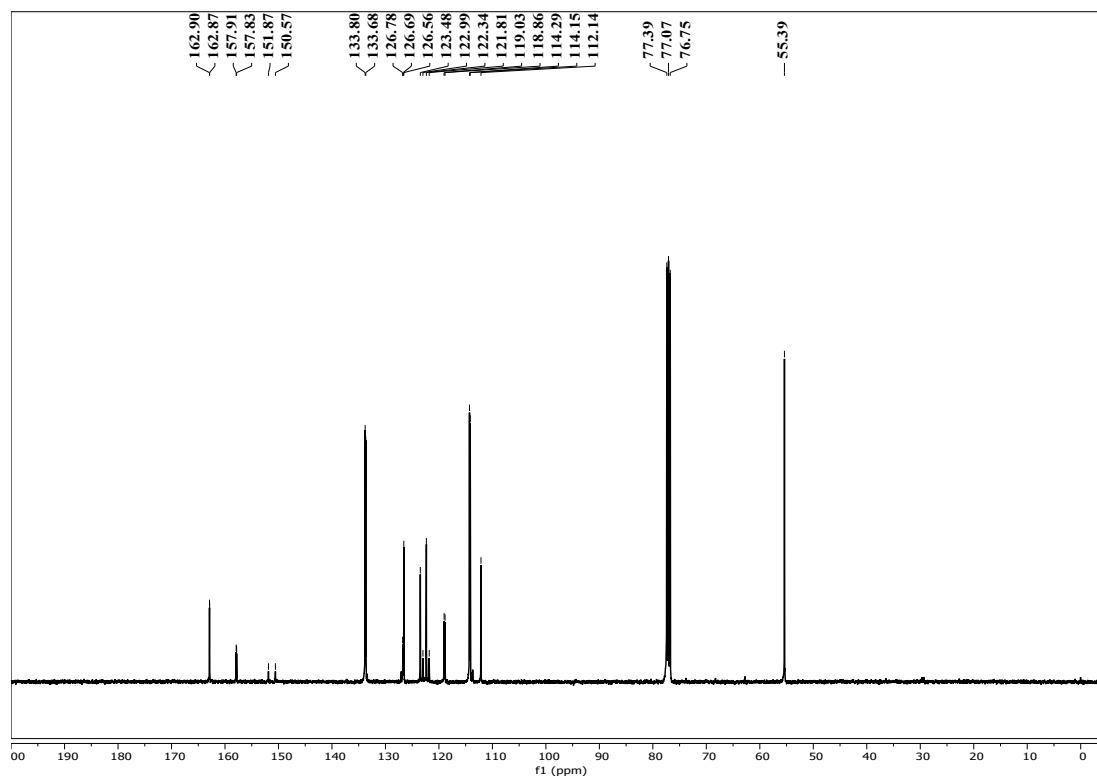




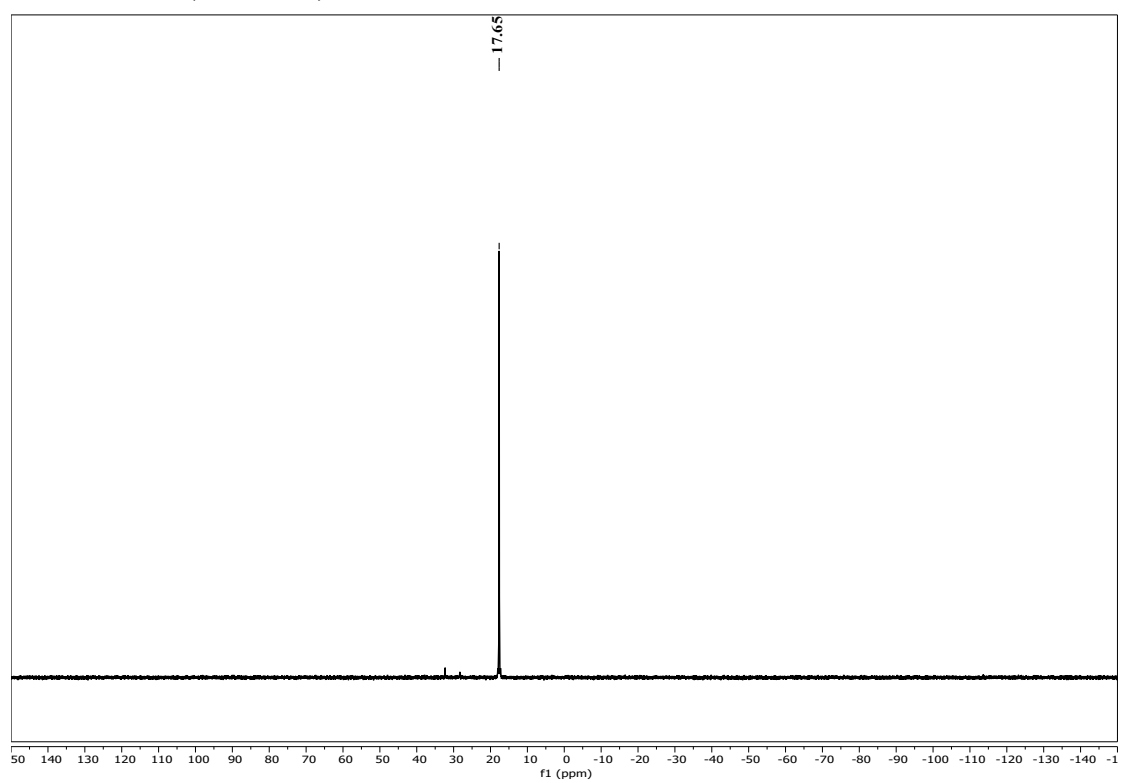
4c, ^1H NMR (400 MHz), CDCl_3

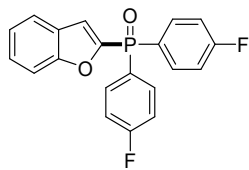


4c, ^{13}C NMR (101 MHz), CDCl_3

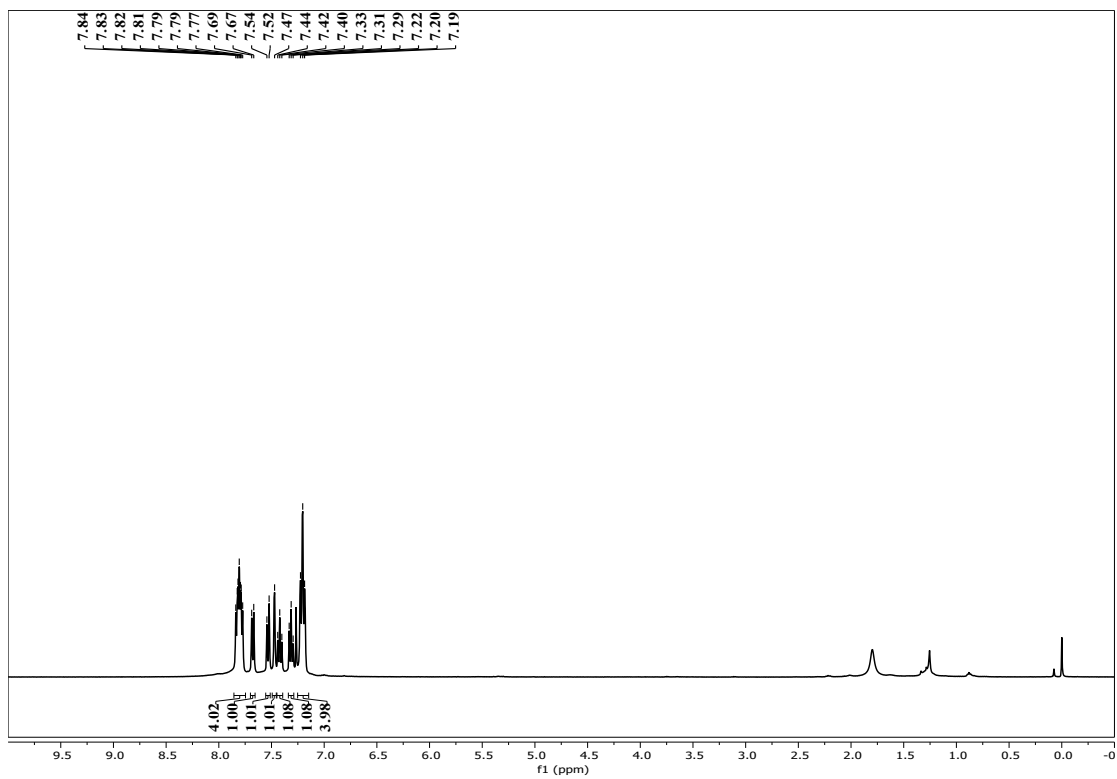


4c, ^{31}P NMR (162 MHz), CDCl_3

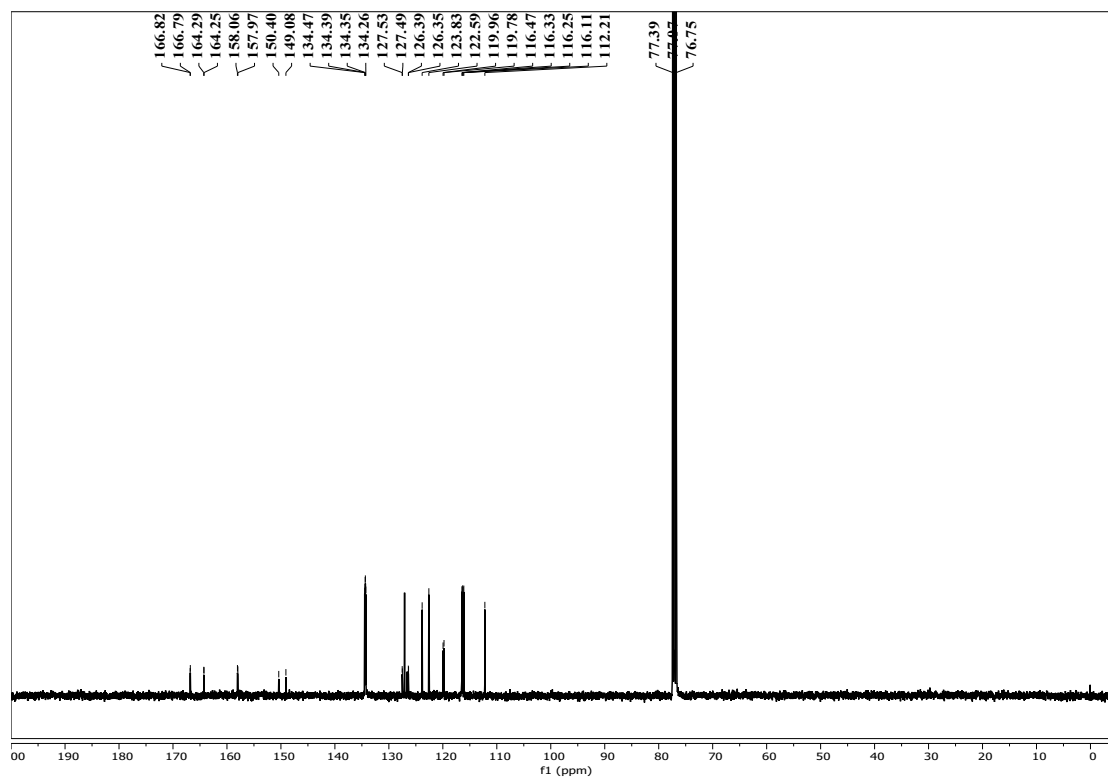




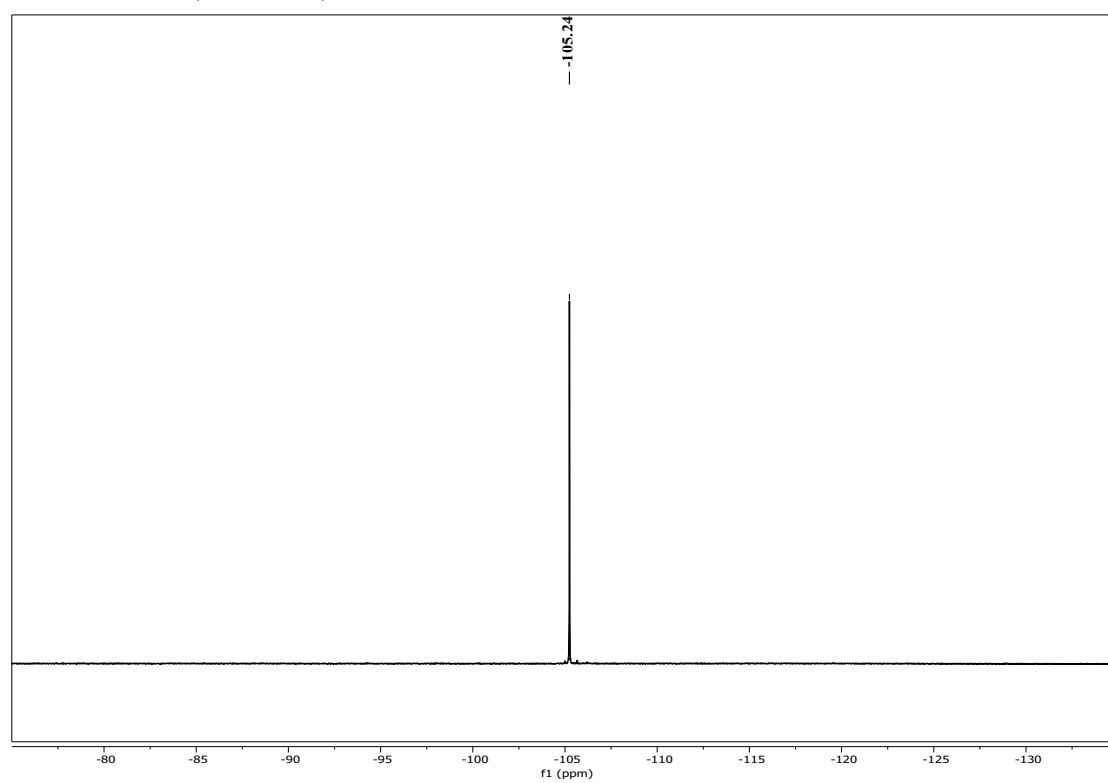
4d, ^1H NMR (400 MHz), CDCl_3



4d, ^{13}C NMR (101 MHz), CDCl_3

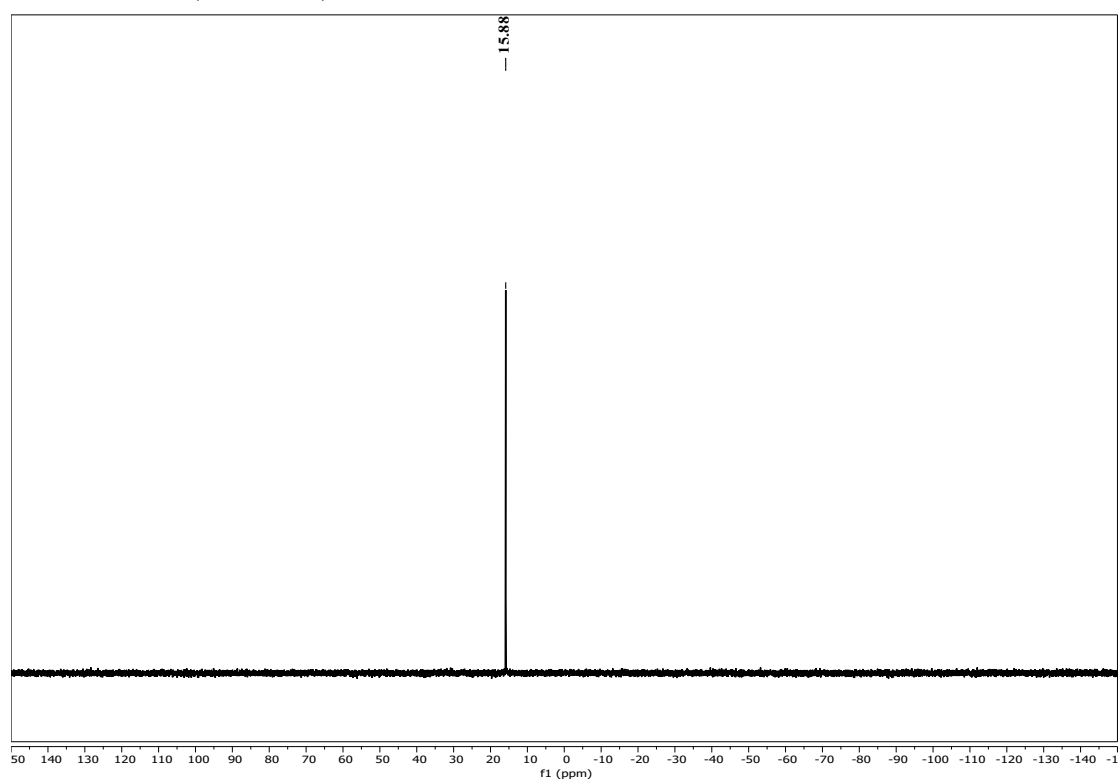


4d, ^{19}F NMR (376 MHz), CDCl_3

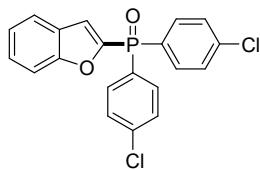


^{19}F NMR spectra

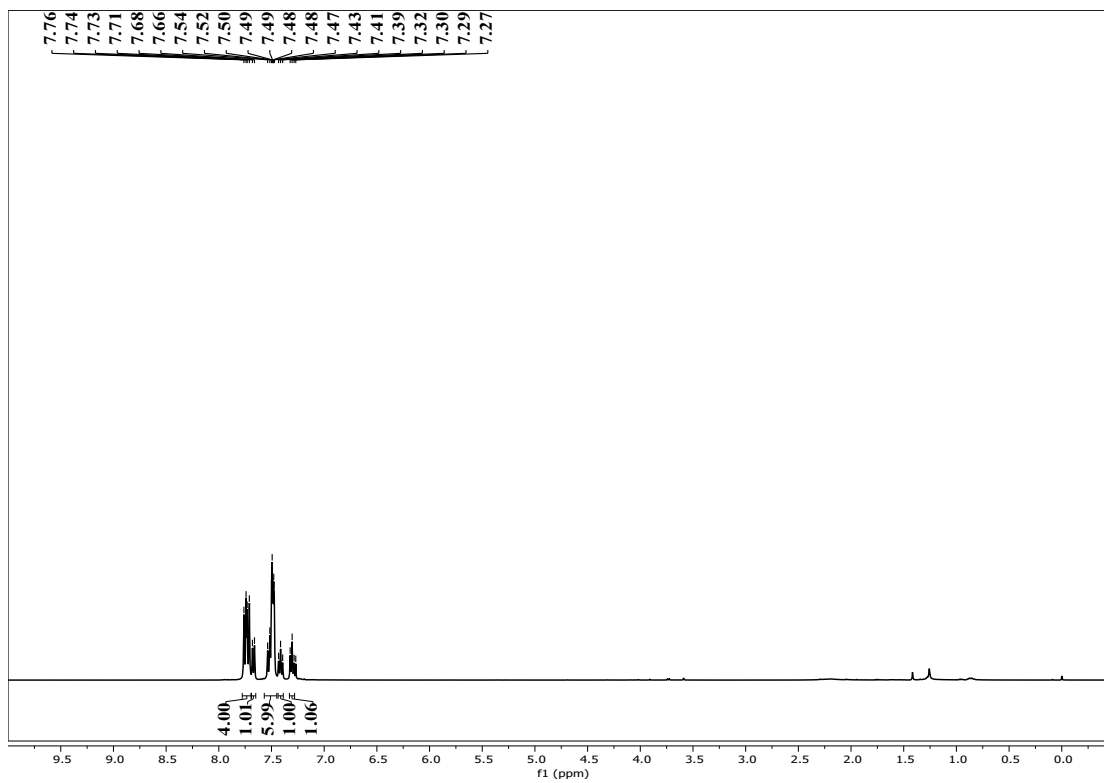
4d, ^{31}P NMR (162 MHz), CDCl_3



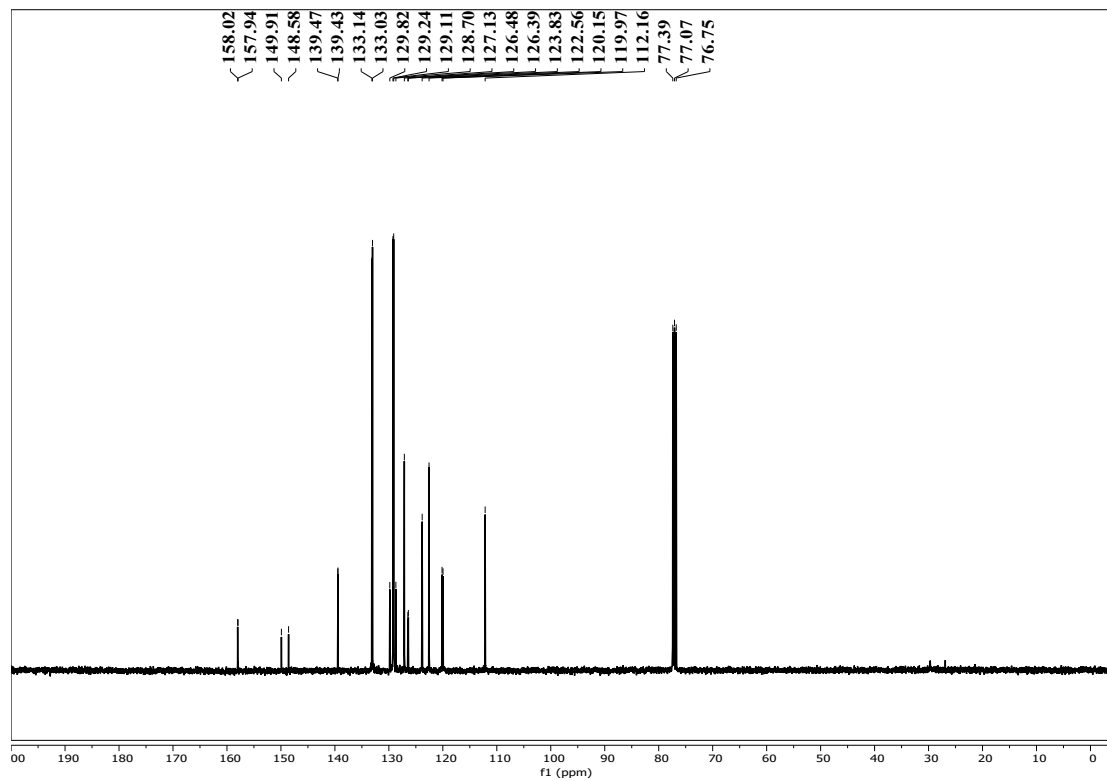
^{31}P NMR spectra



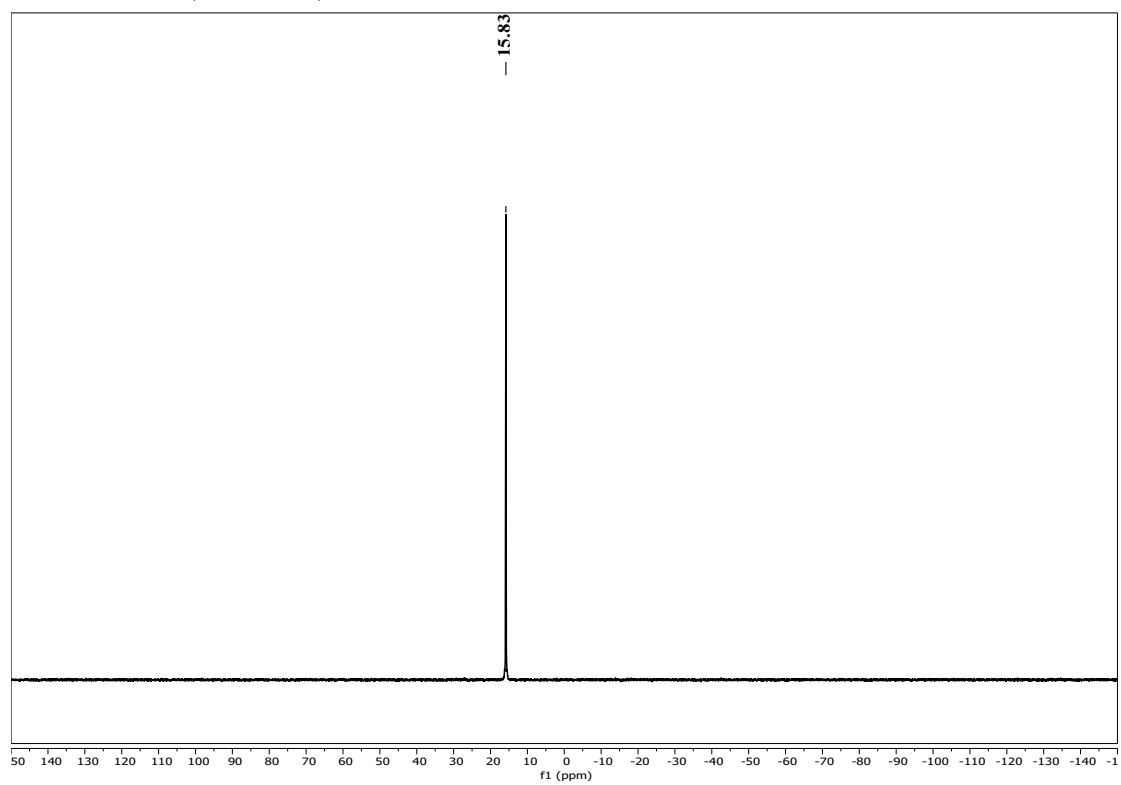
4e, ^1H NMR (400 MHz), CDCl_3

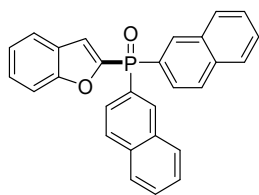


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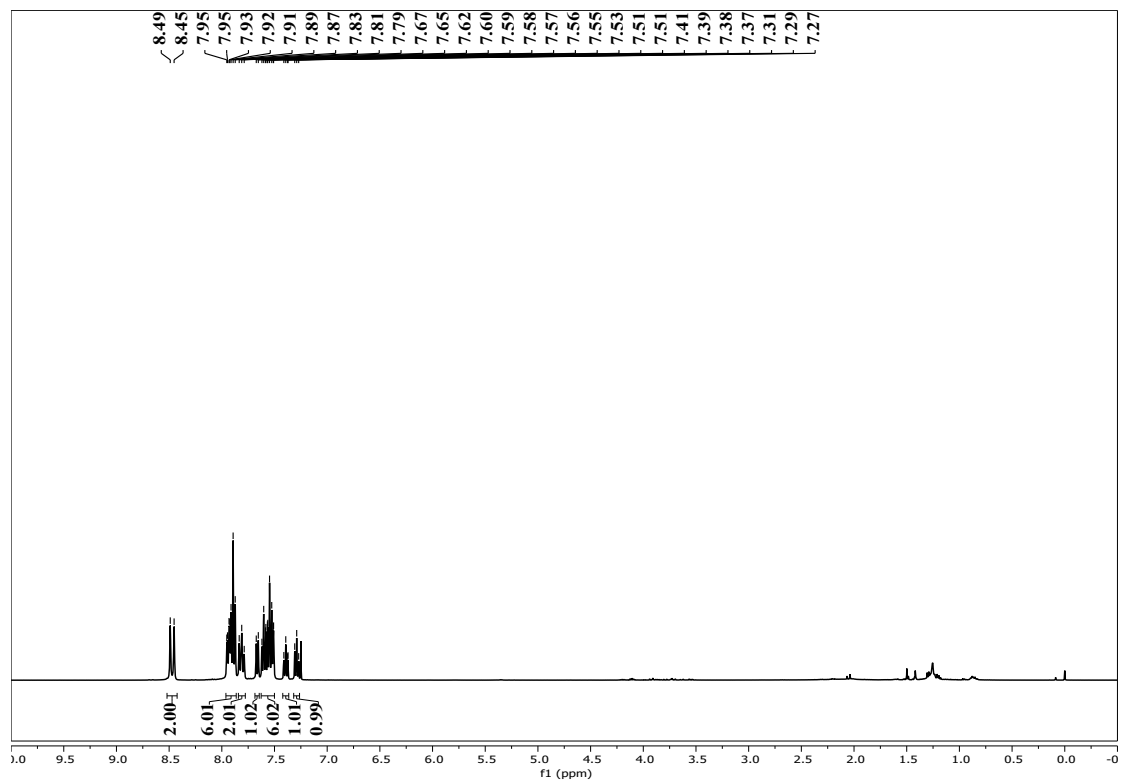


4e, ^{31}P NMR (162 MHz), CDCl_3

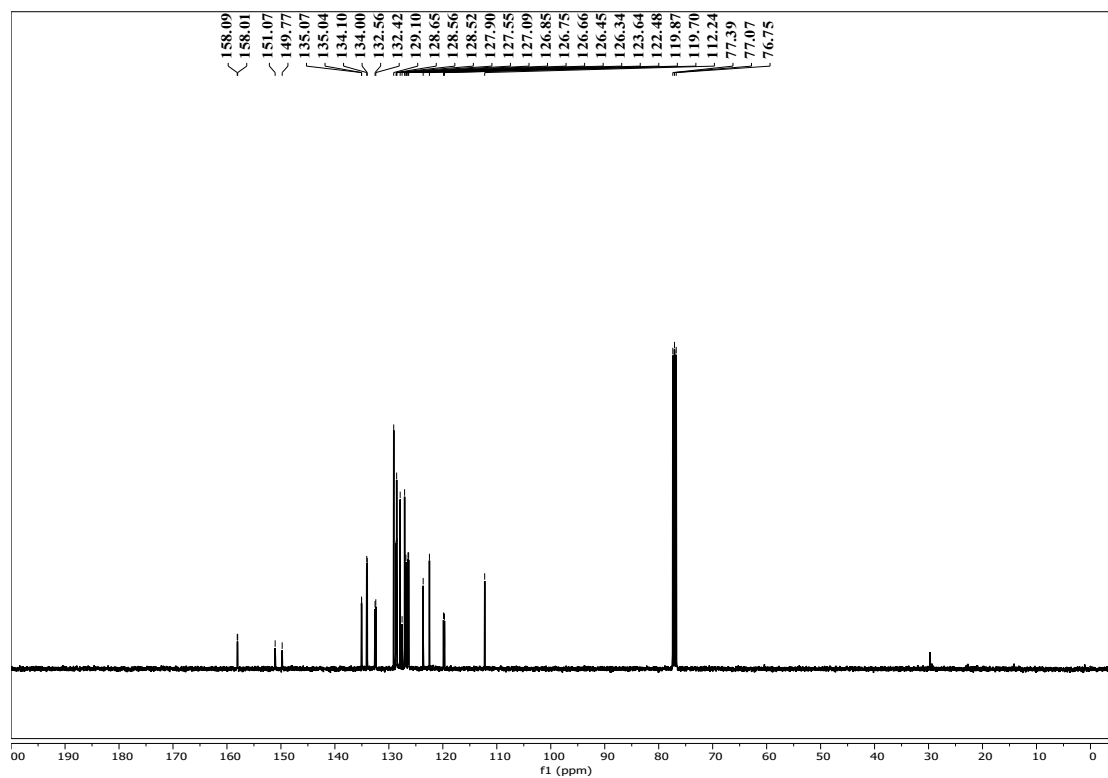




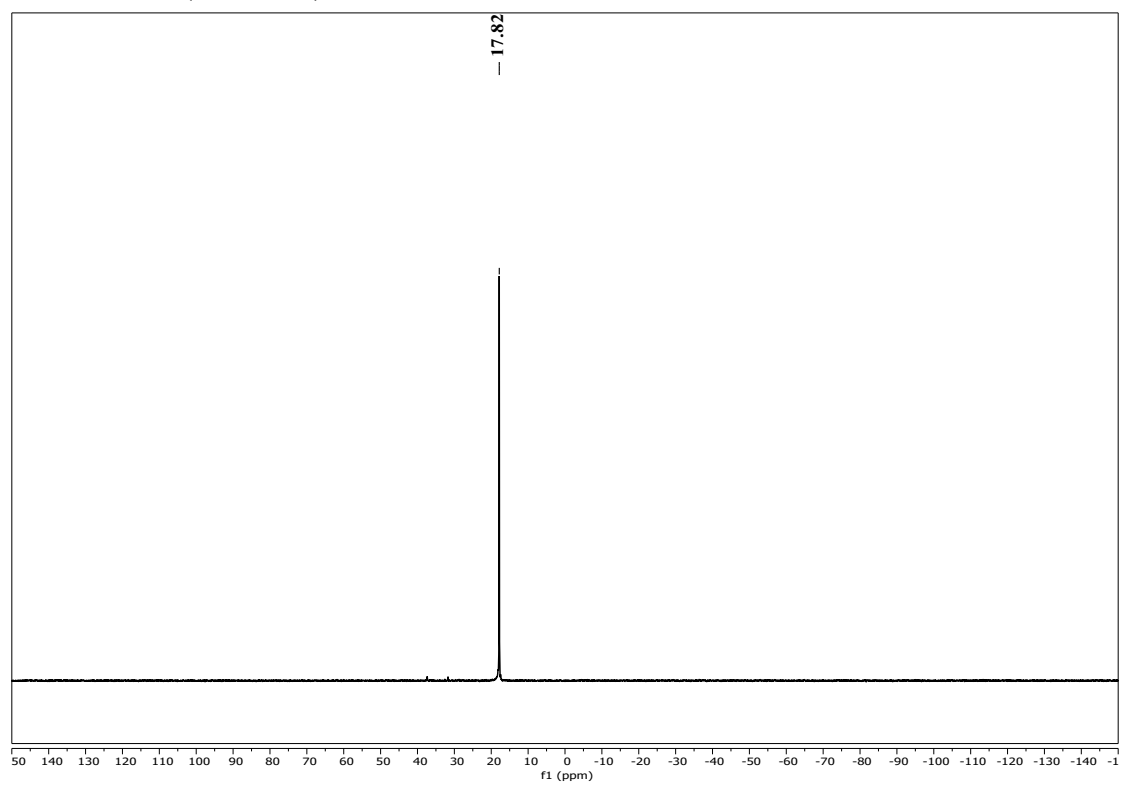
4f, ^1H NMR (400 MHz), CDCl_3

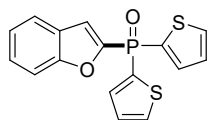


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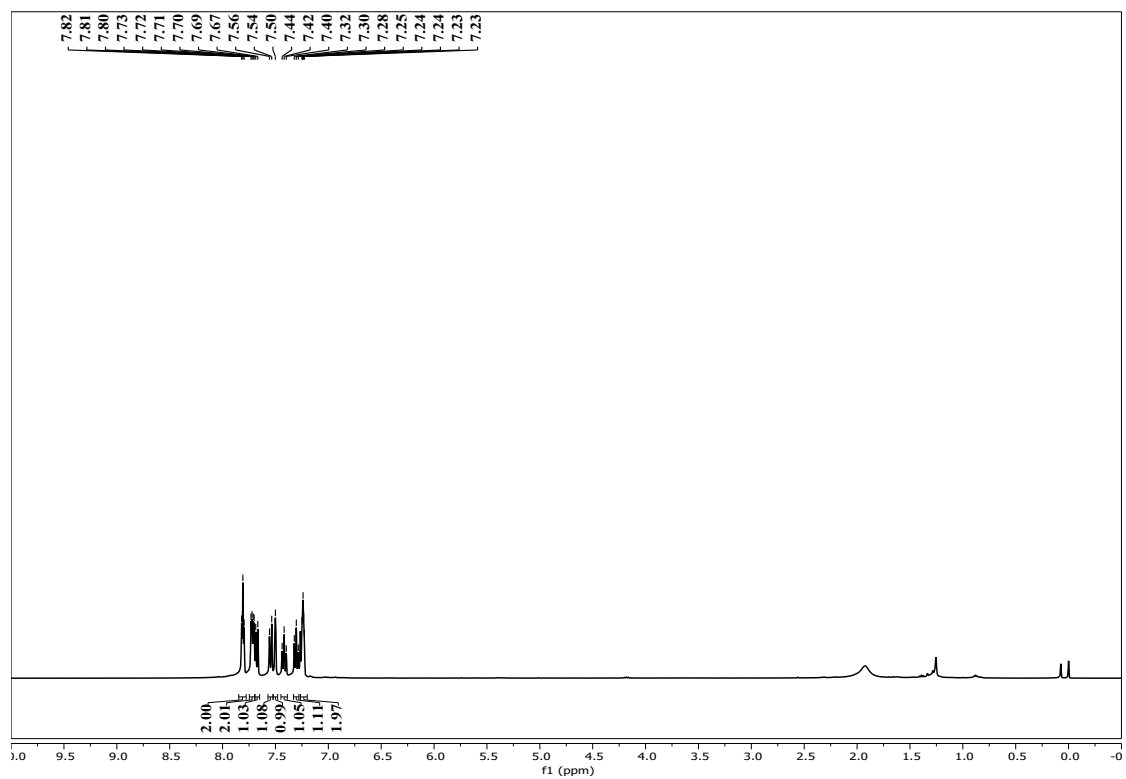


4f, ^{31}P NMR (162 MHz), CDCl_3

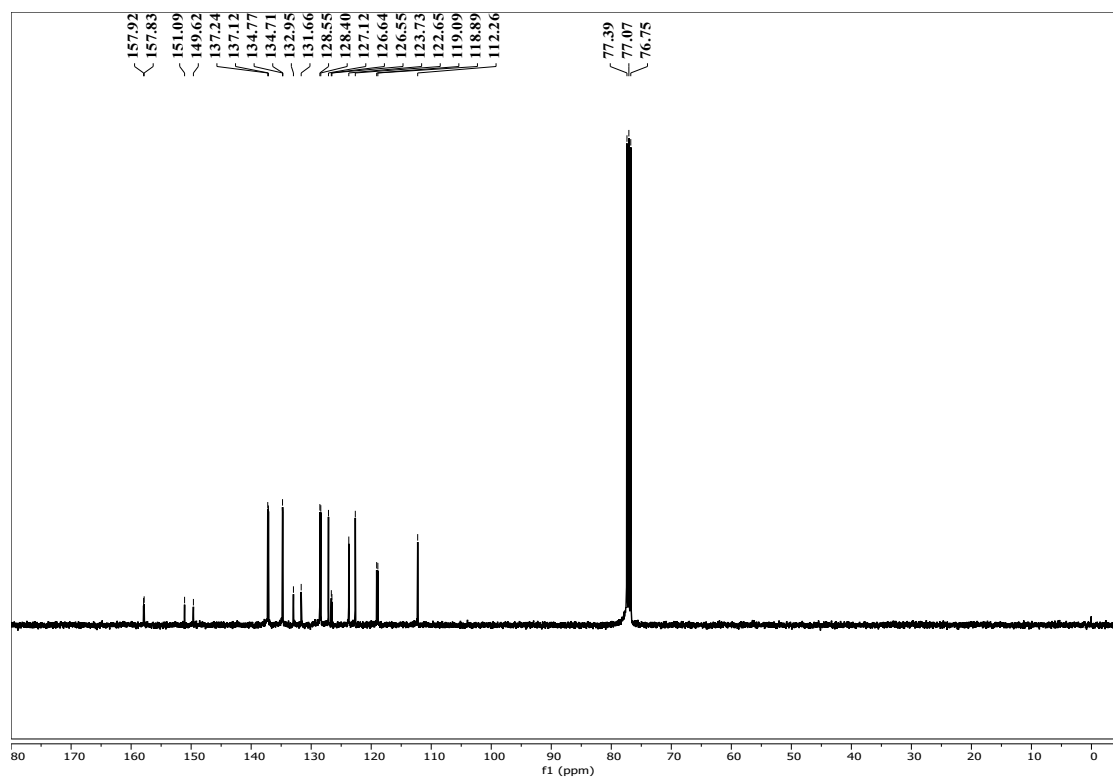




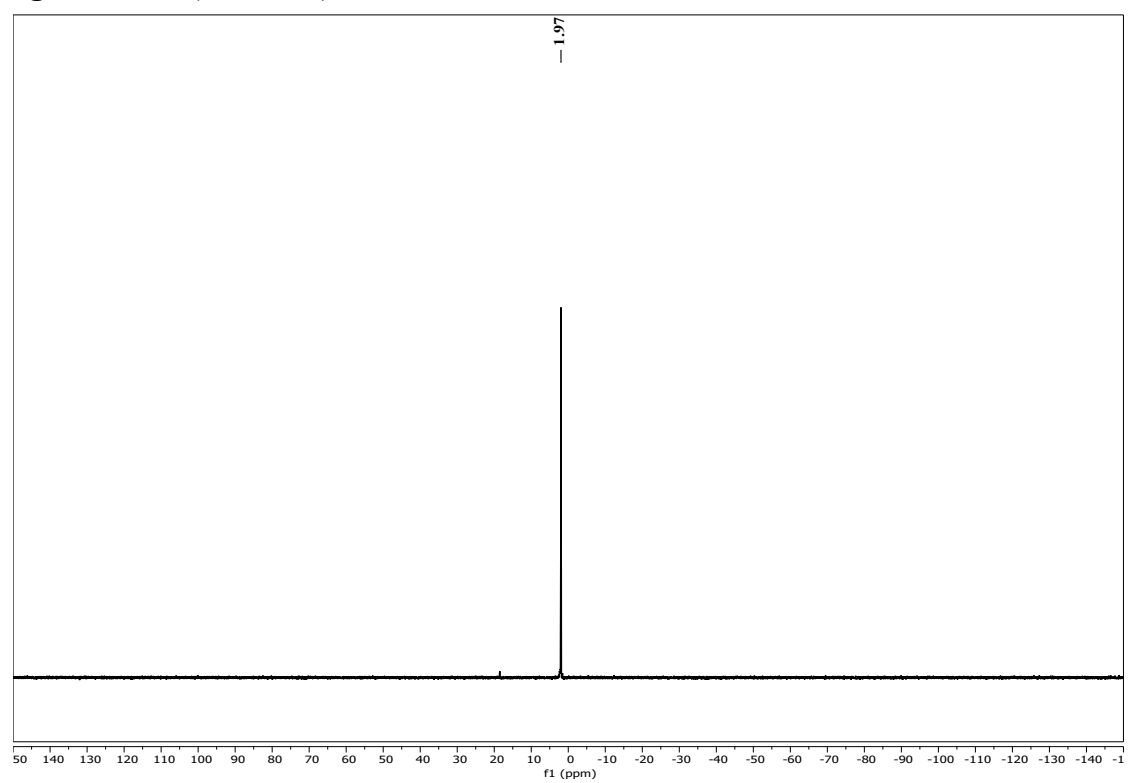
4g, ^1H NMR (400 MHz), CDCl_3

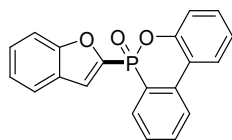


4g, ^{13}C NMR (101 MHz), CDCl_3

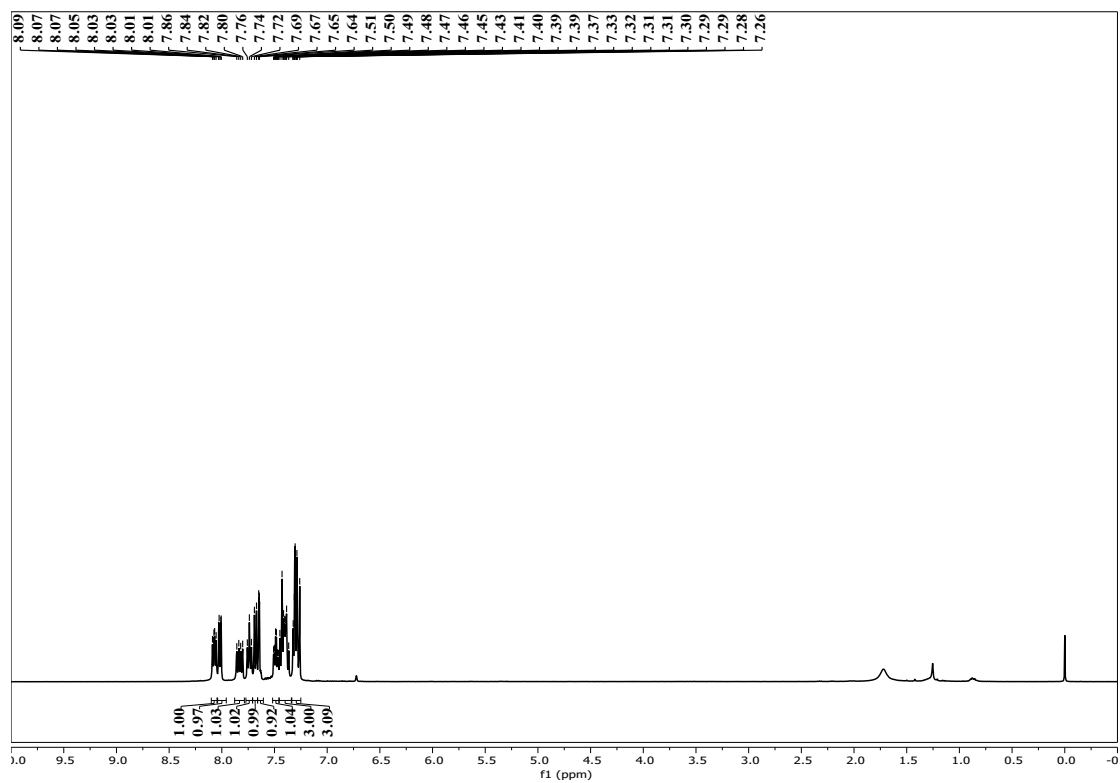


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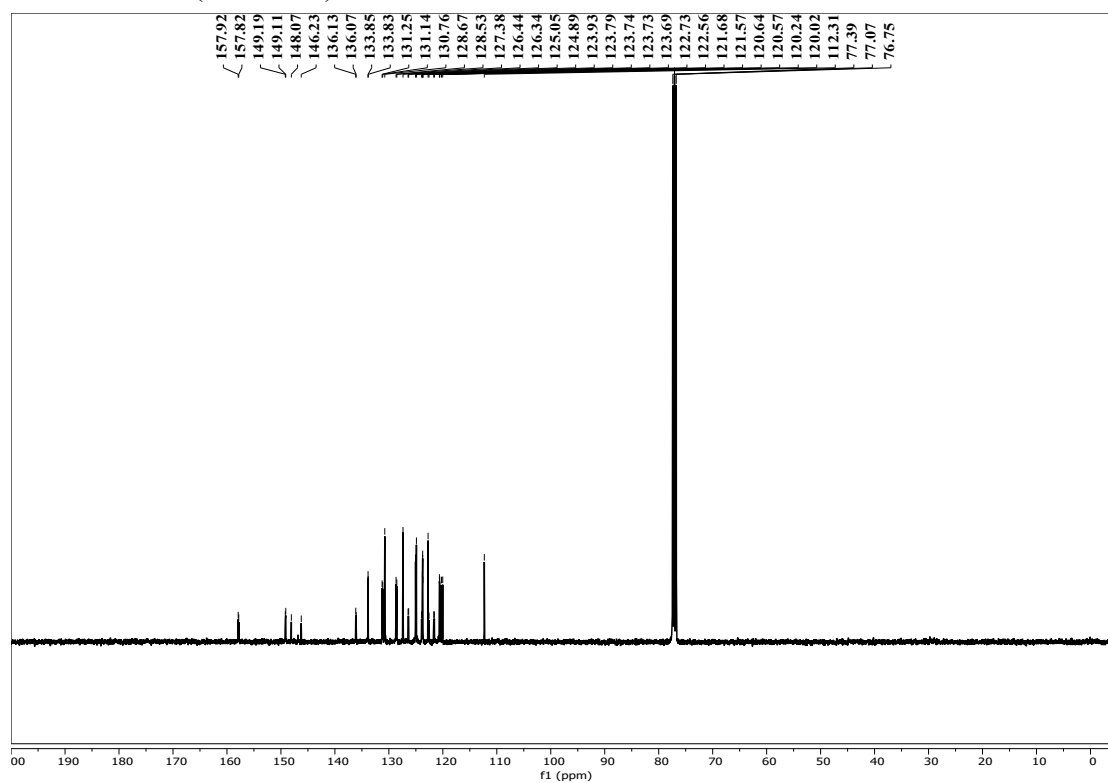




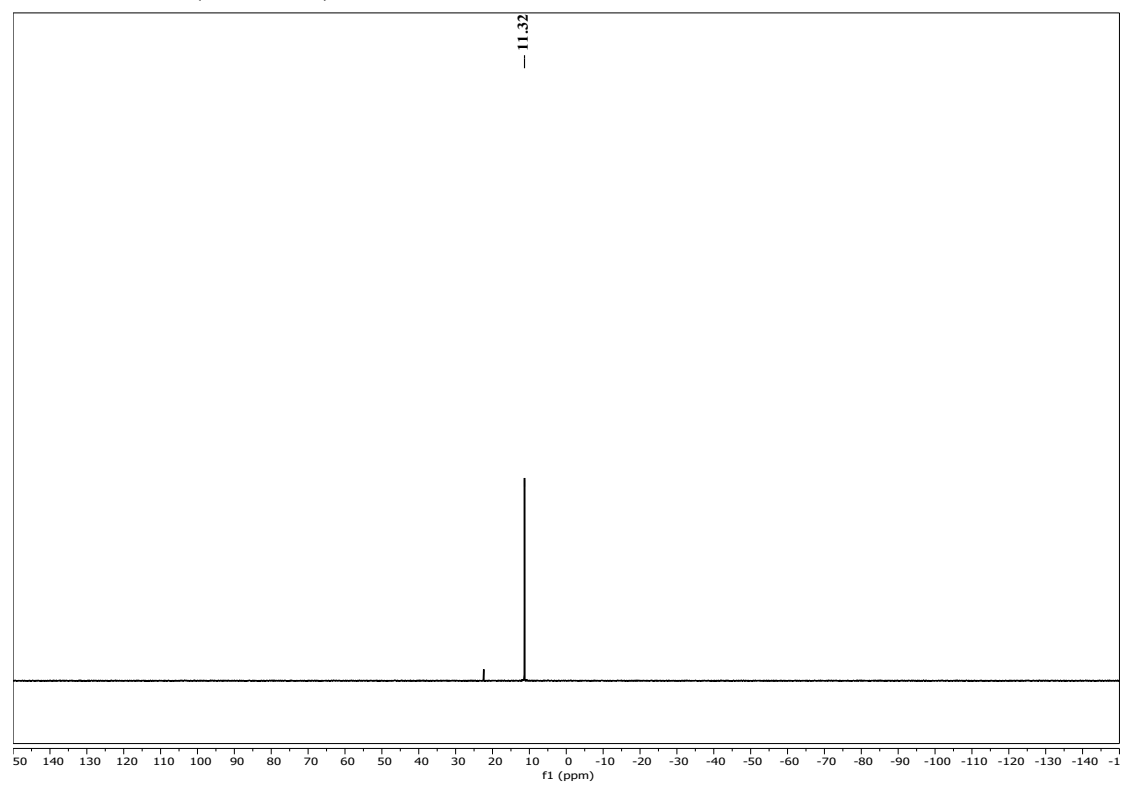
4h, ^1H NMR (400 MHz), CDCl_3

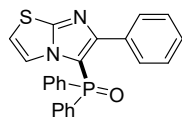


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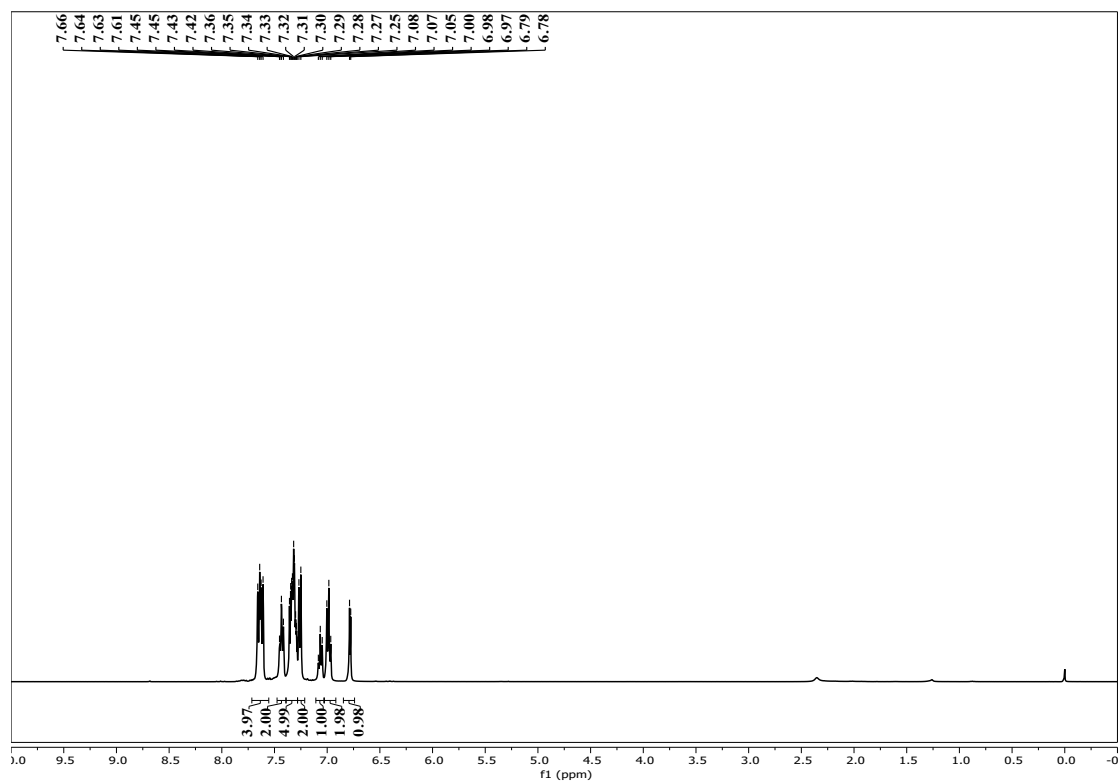


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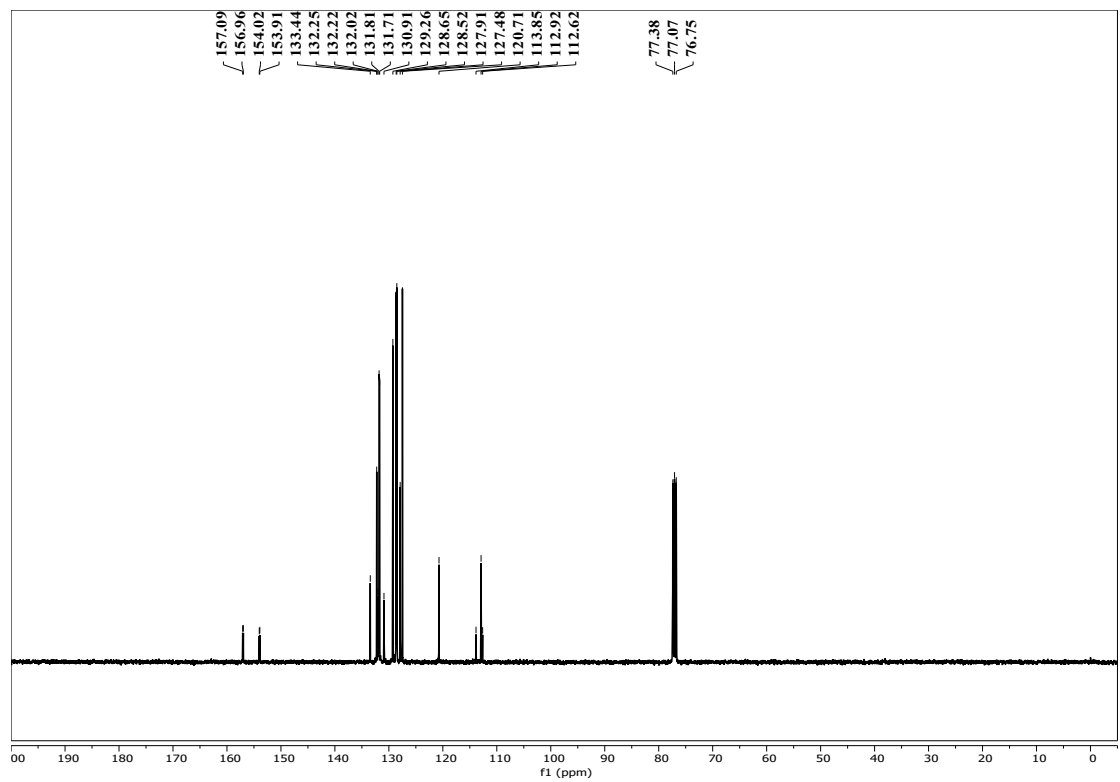




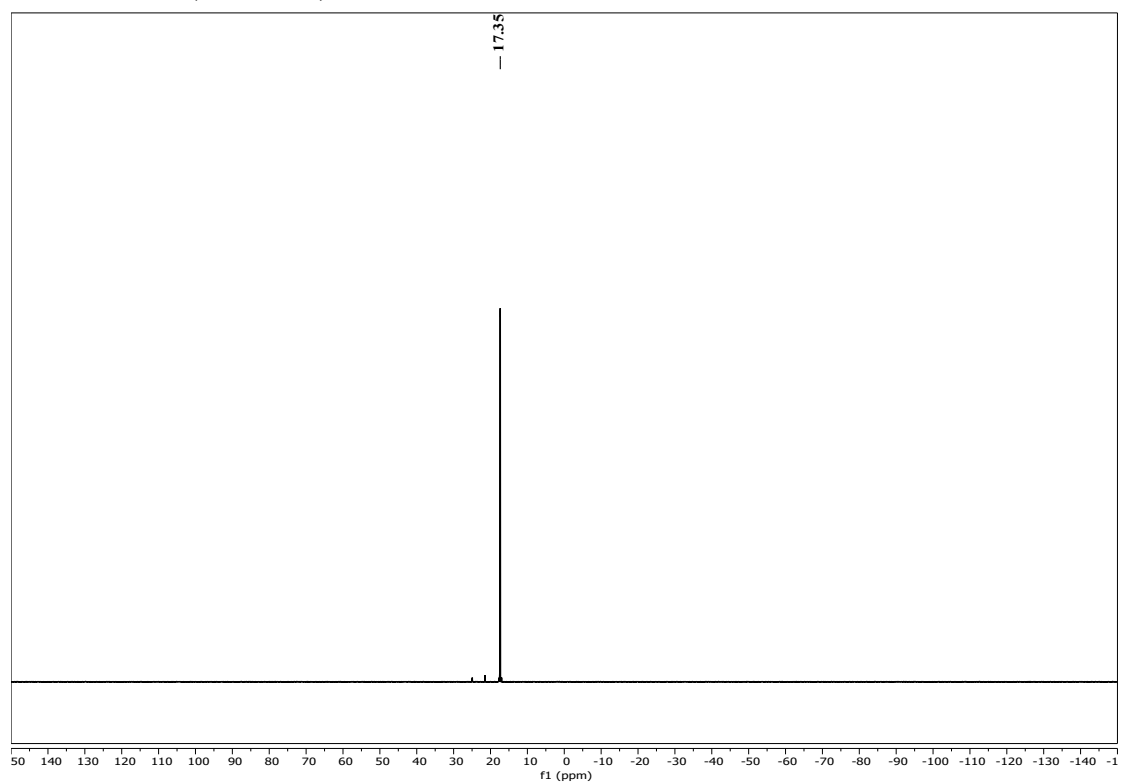
6a, ^1H NMR (400 MHz), CDCl_3

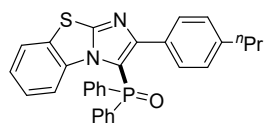


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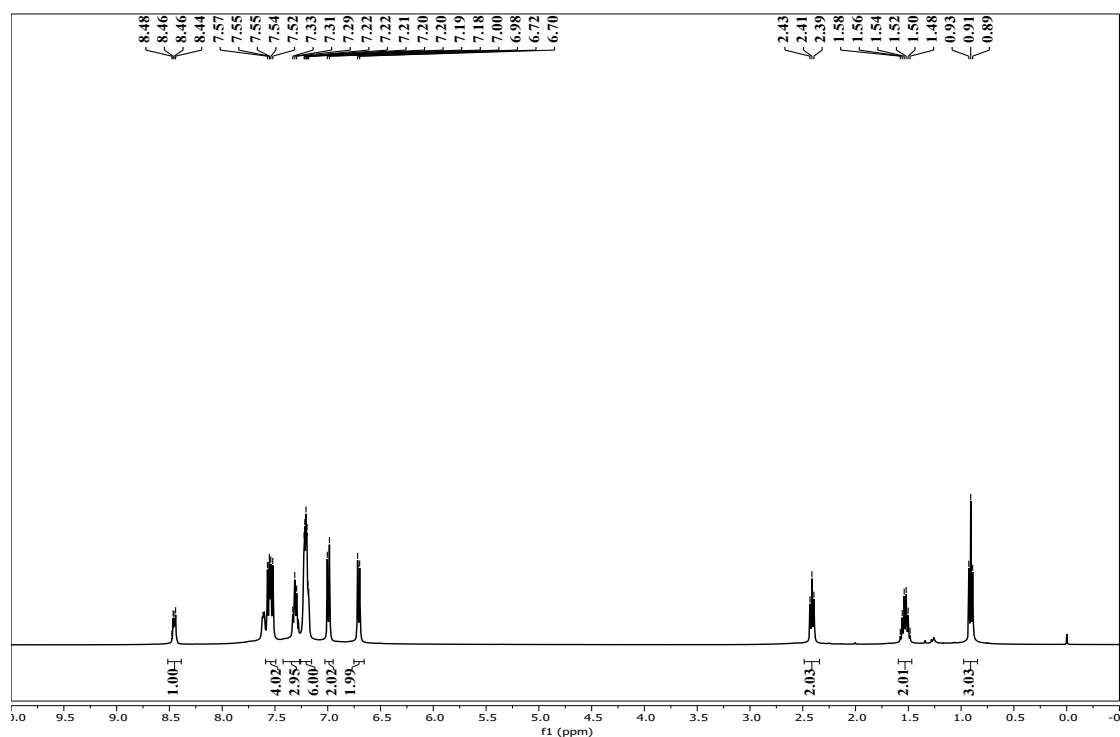


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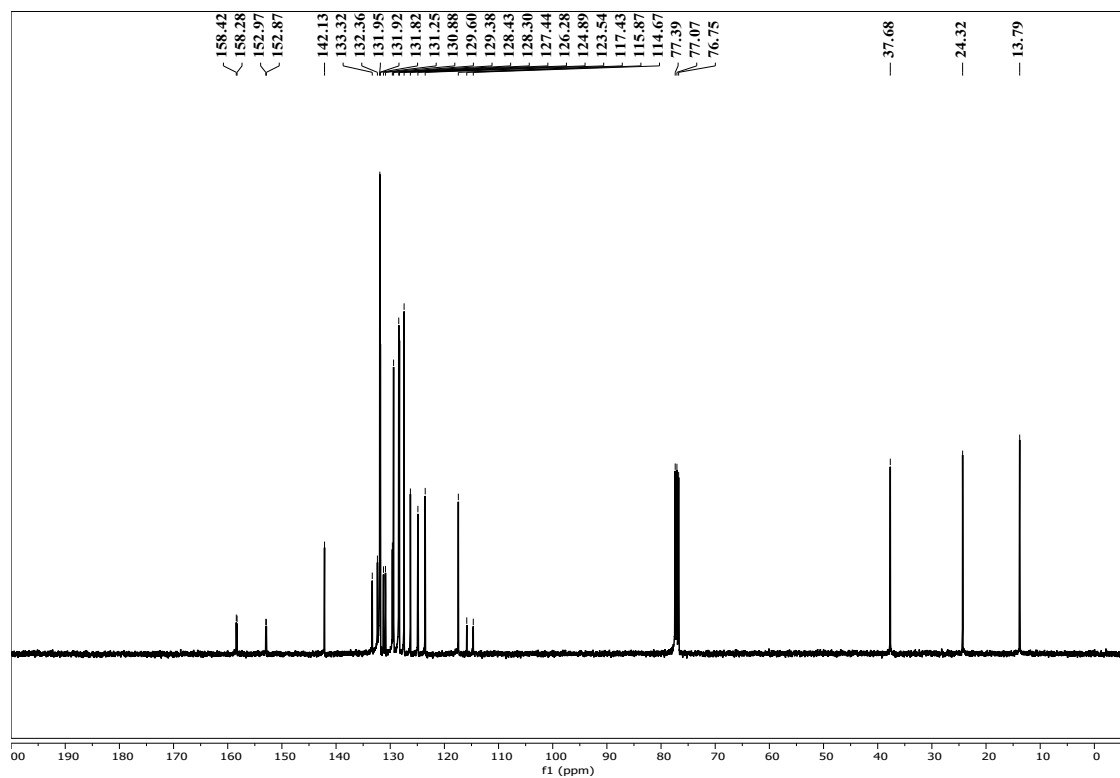




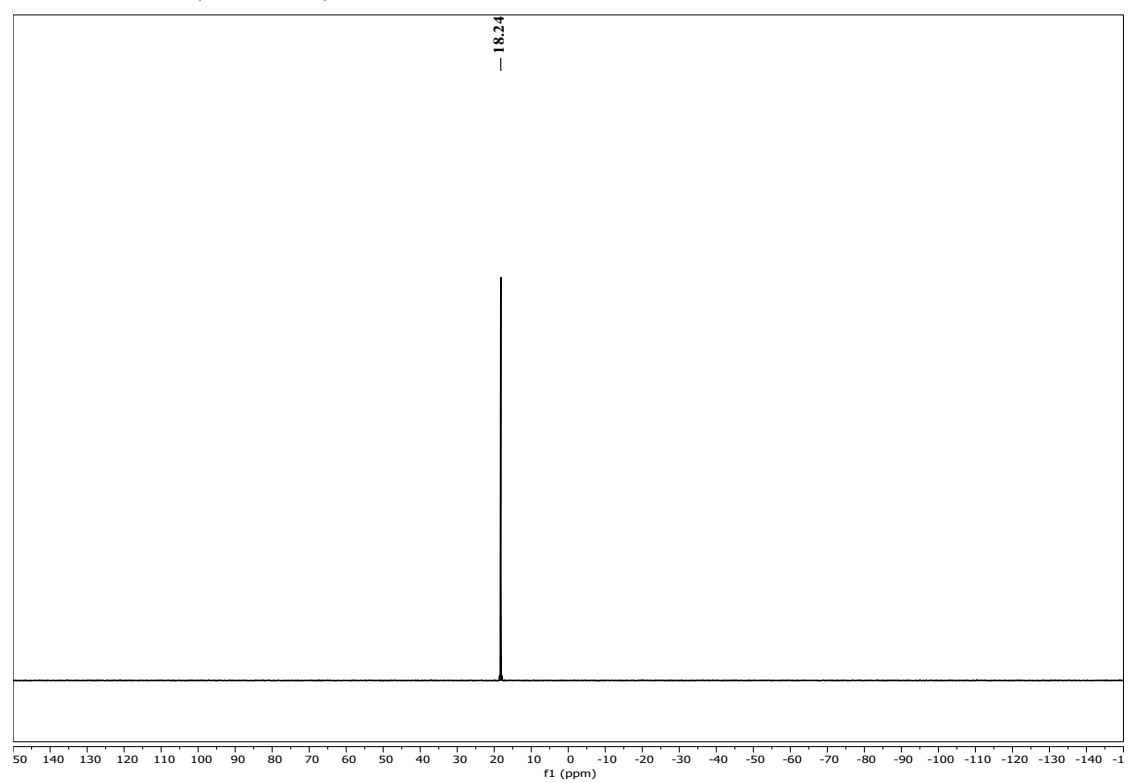
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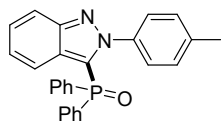


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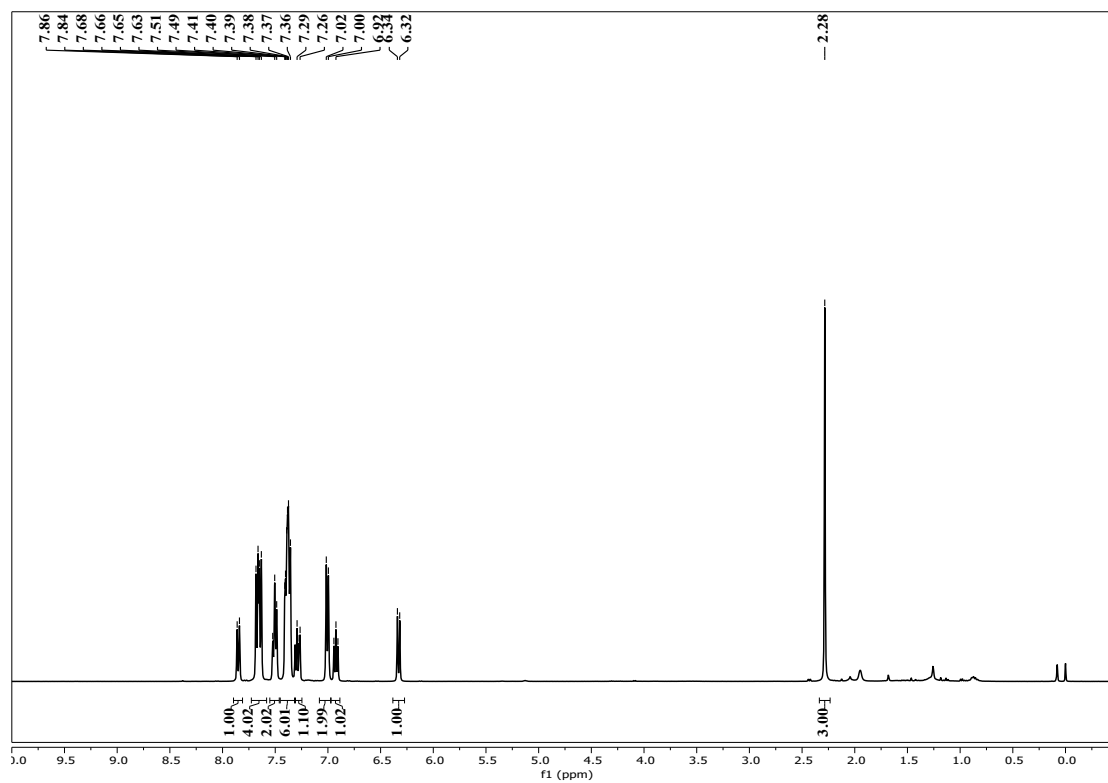


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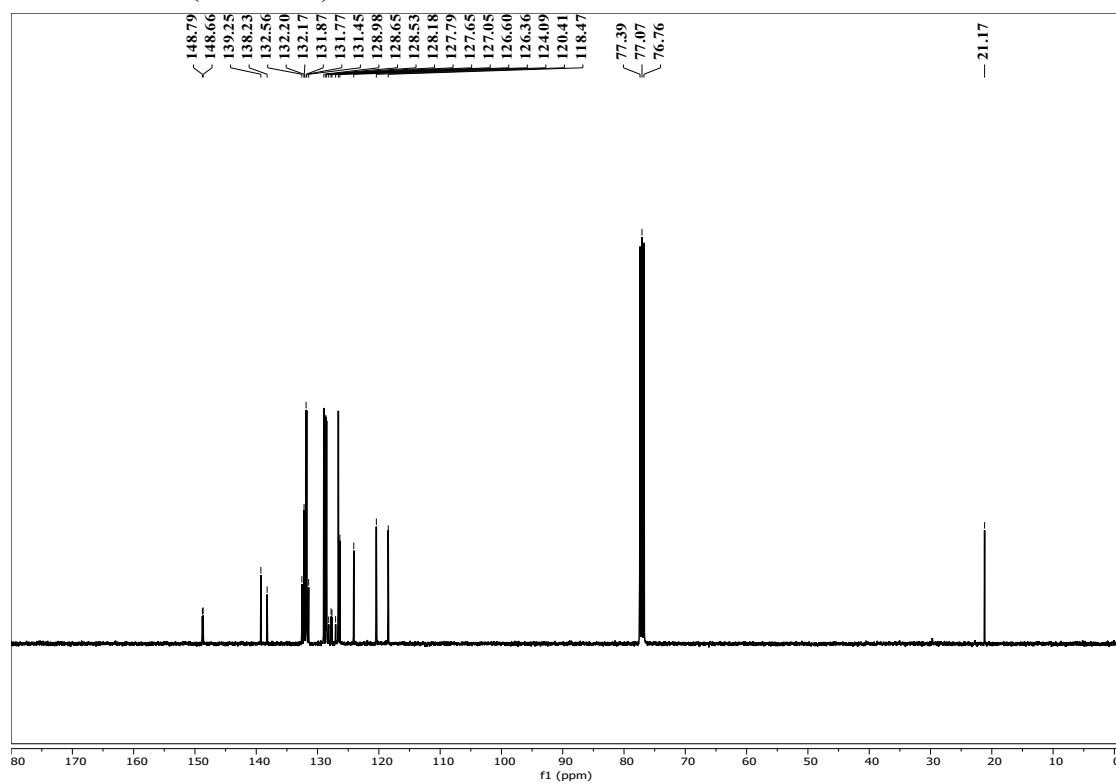




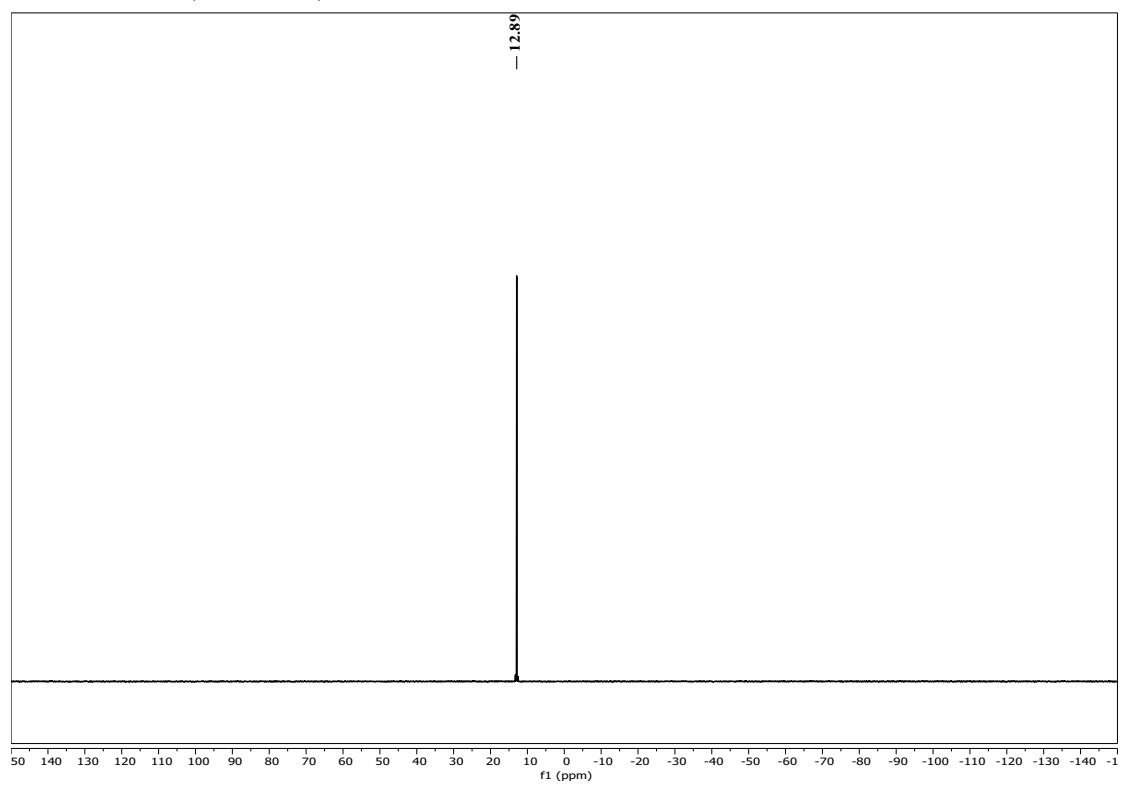
6c, ^1H NMR (400 MHz), CDCl_3

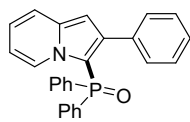


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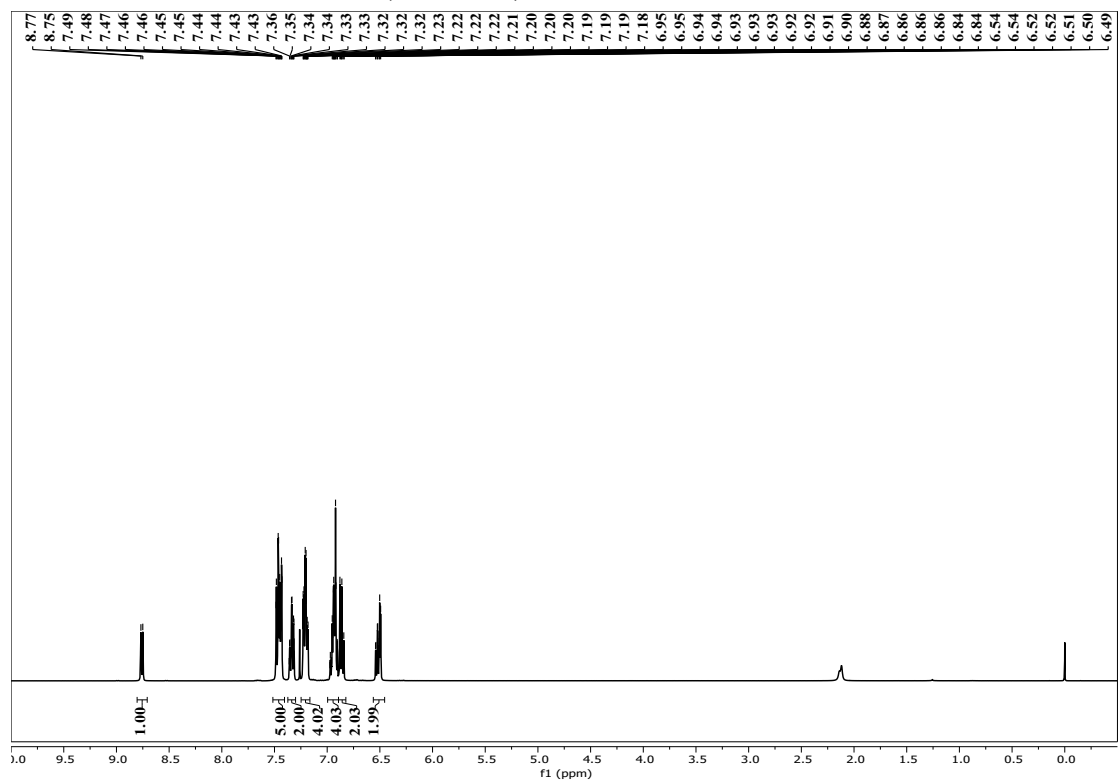


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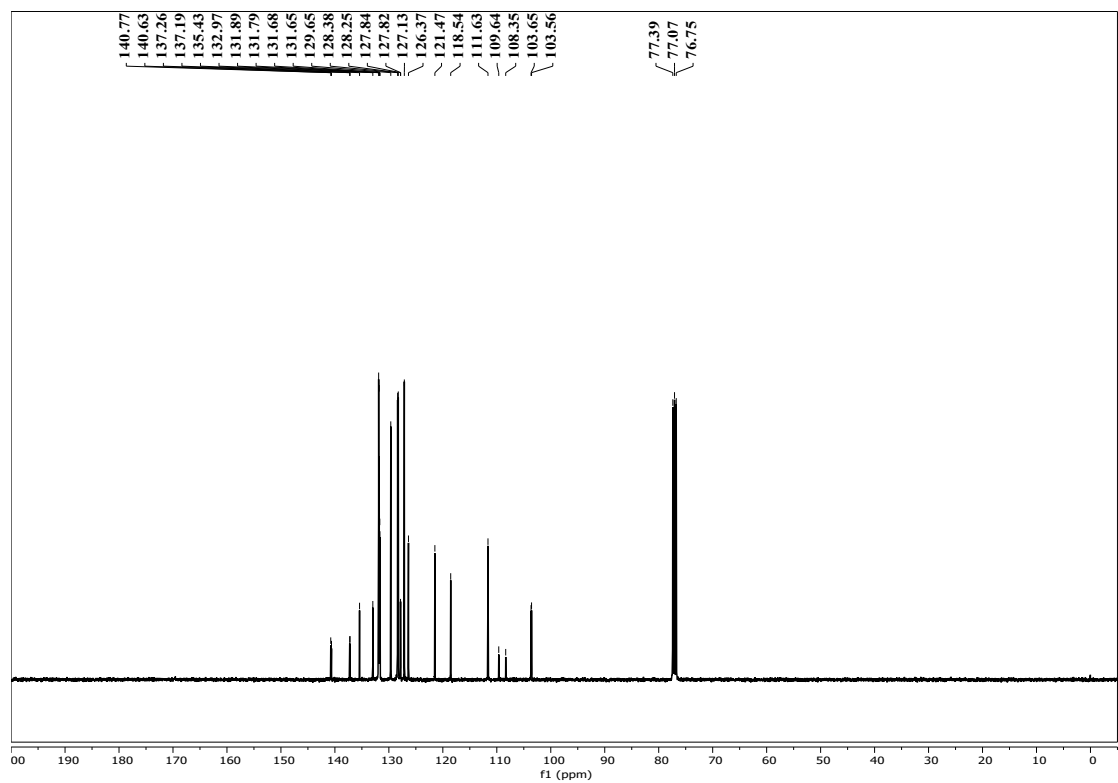




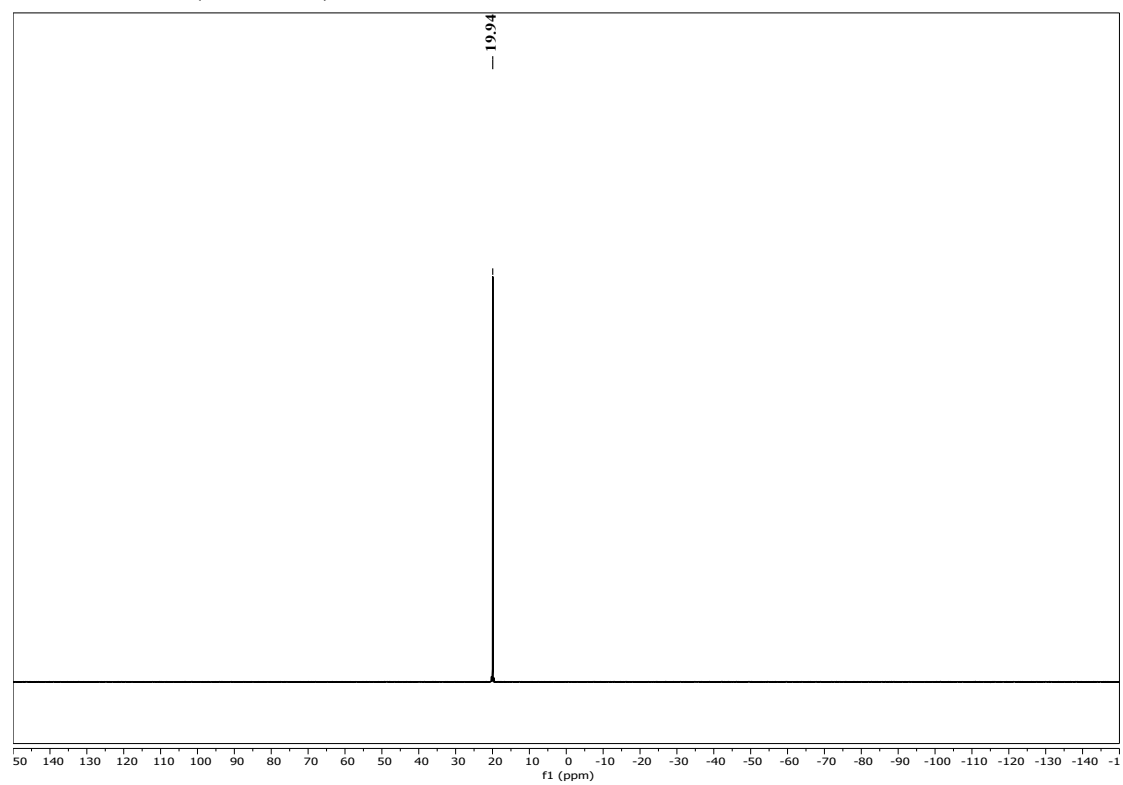
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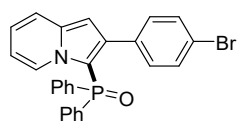


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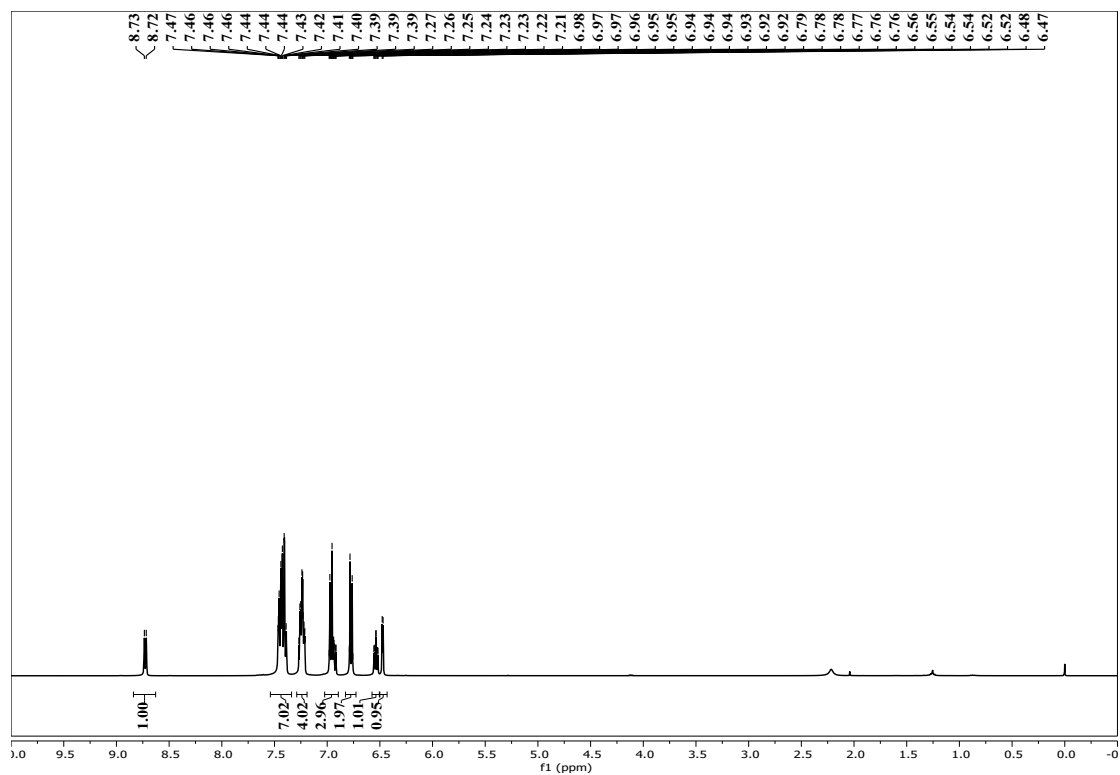


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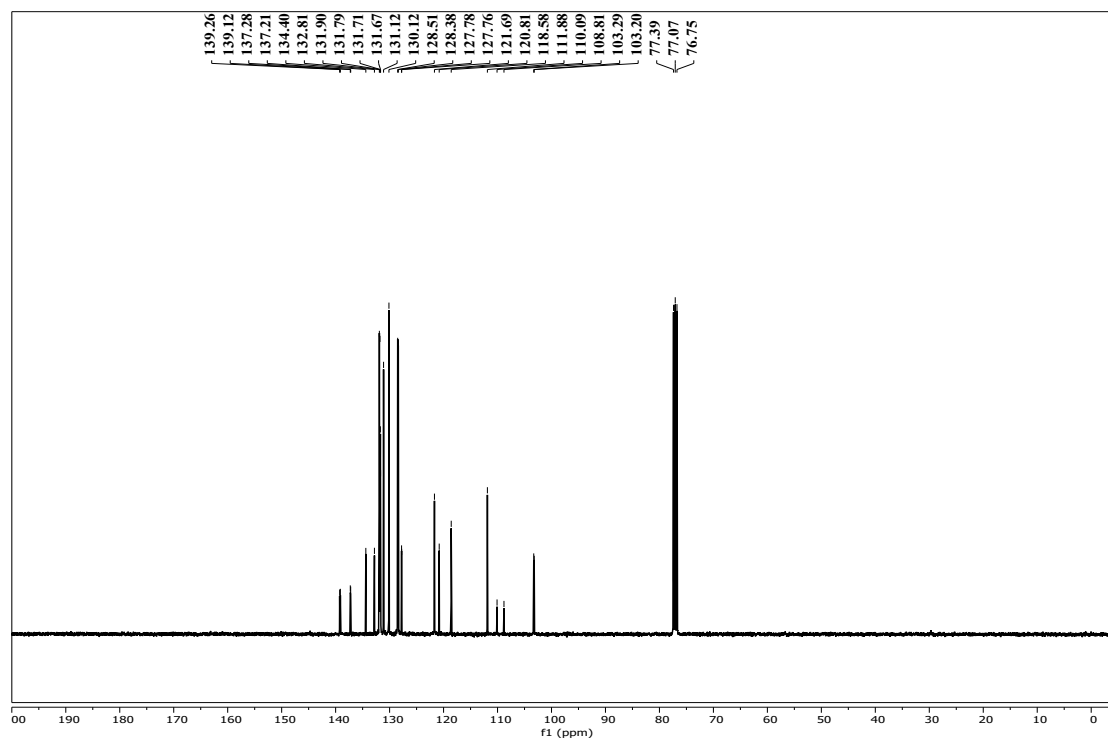




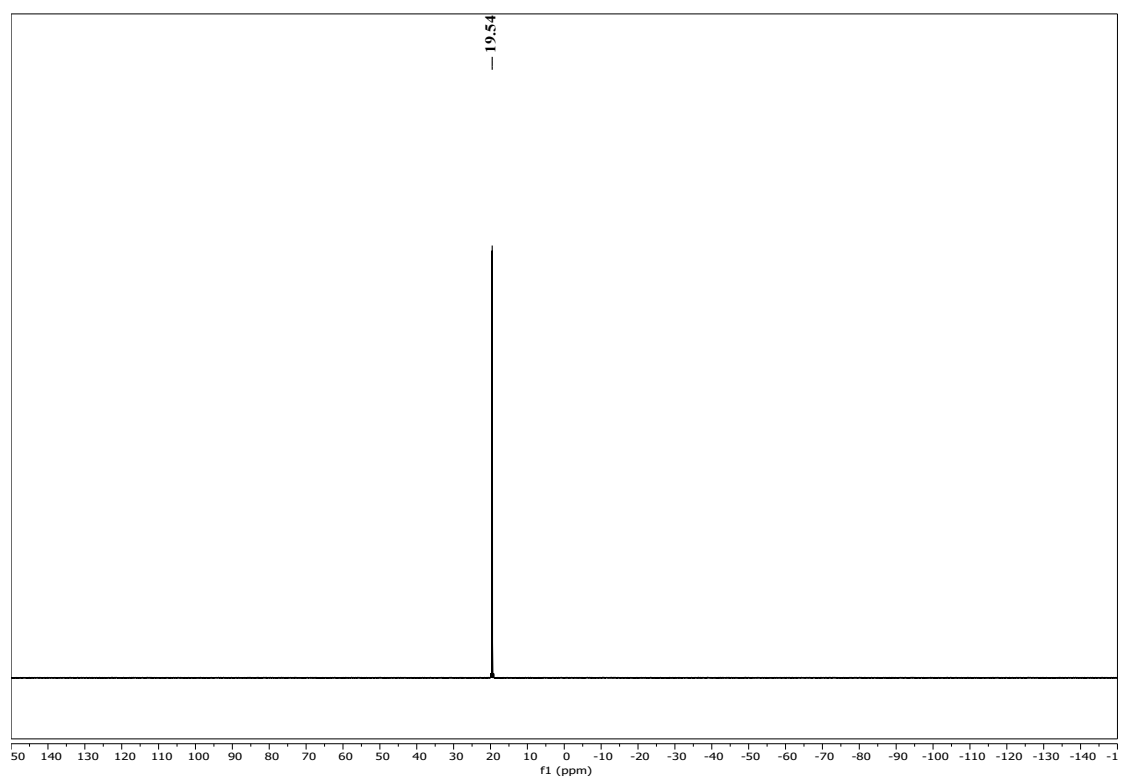
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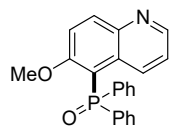


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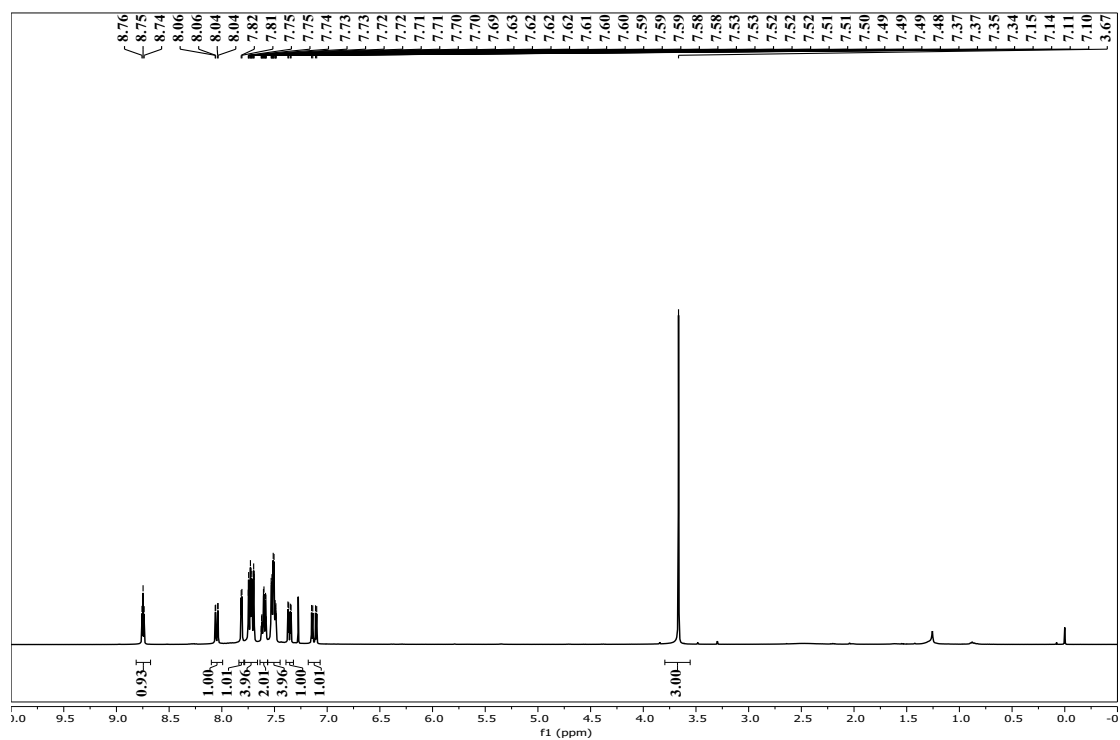


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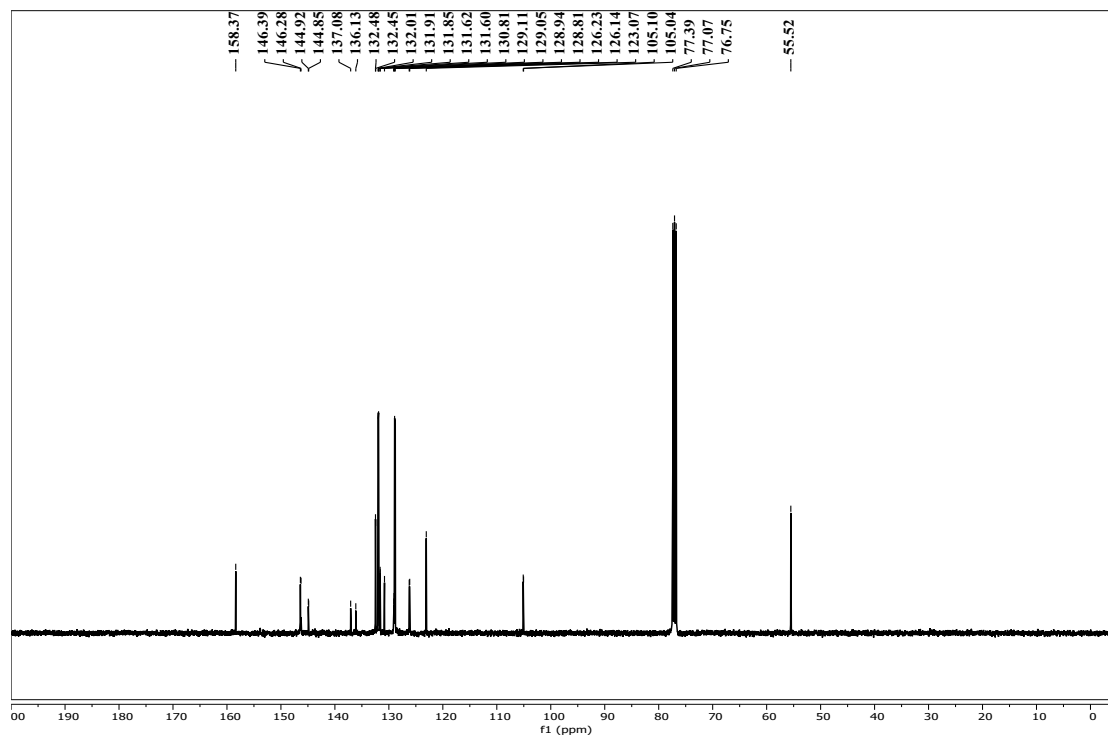




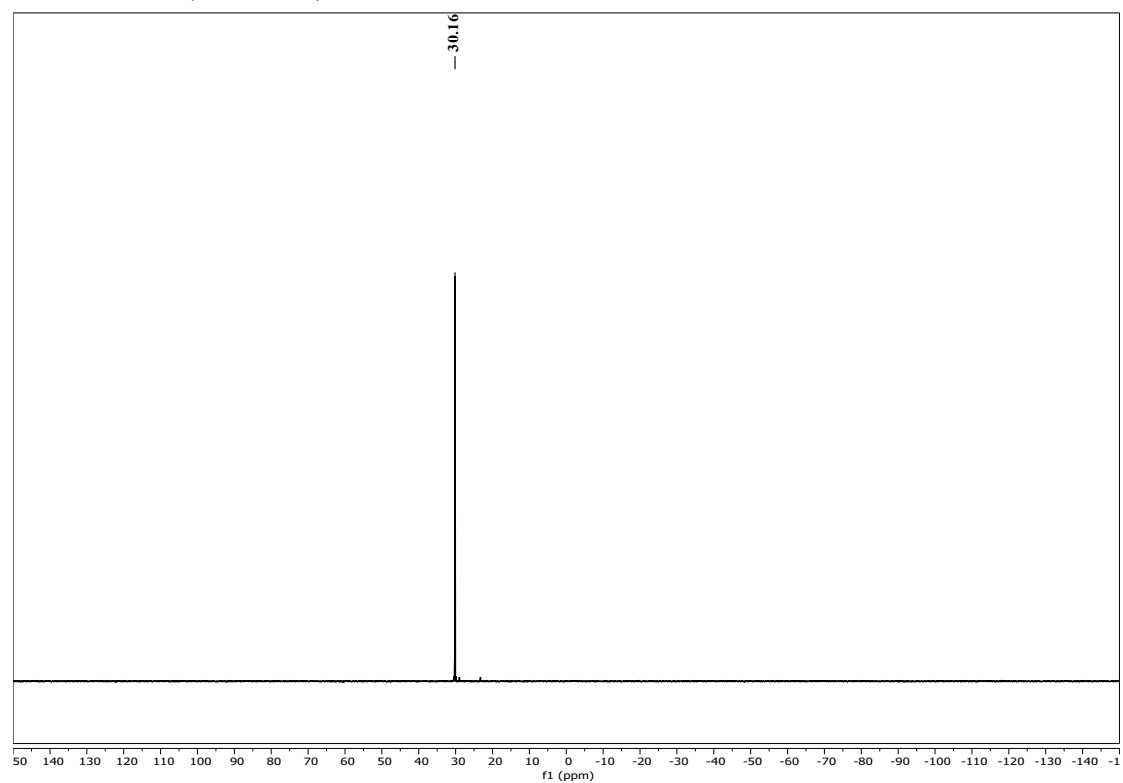
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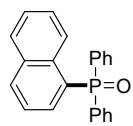


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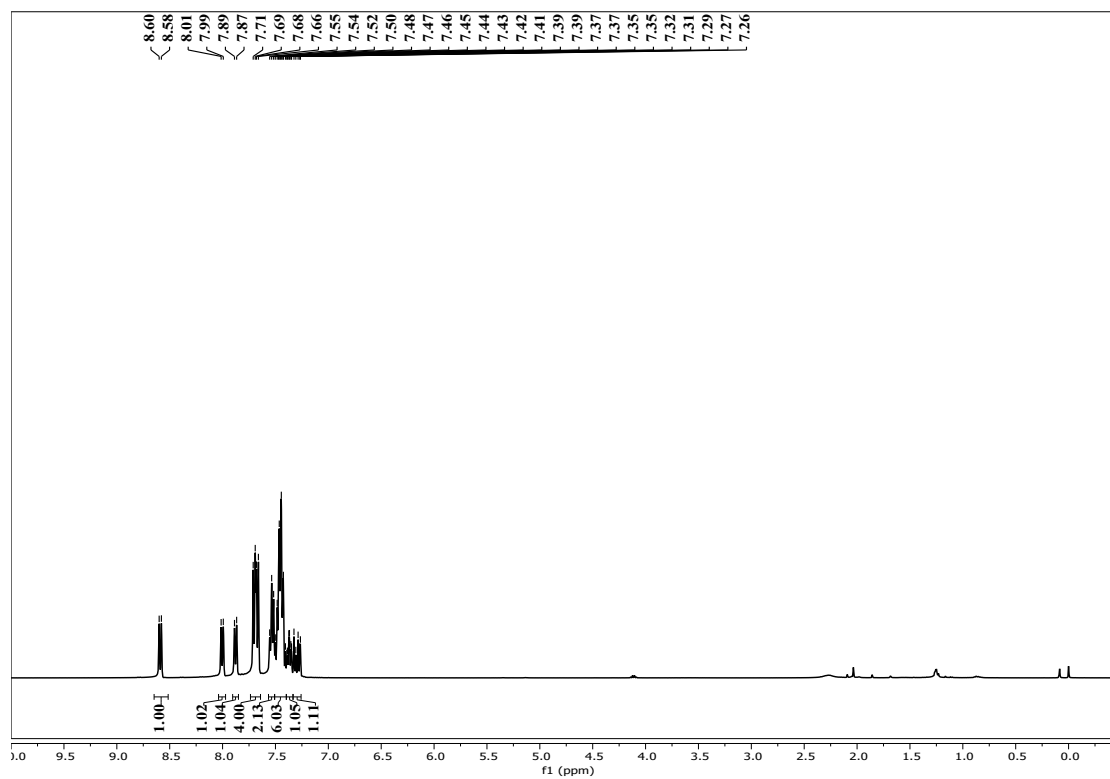


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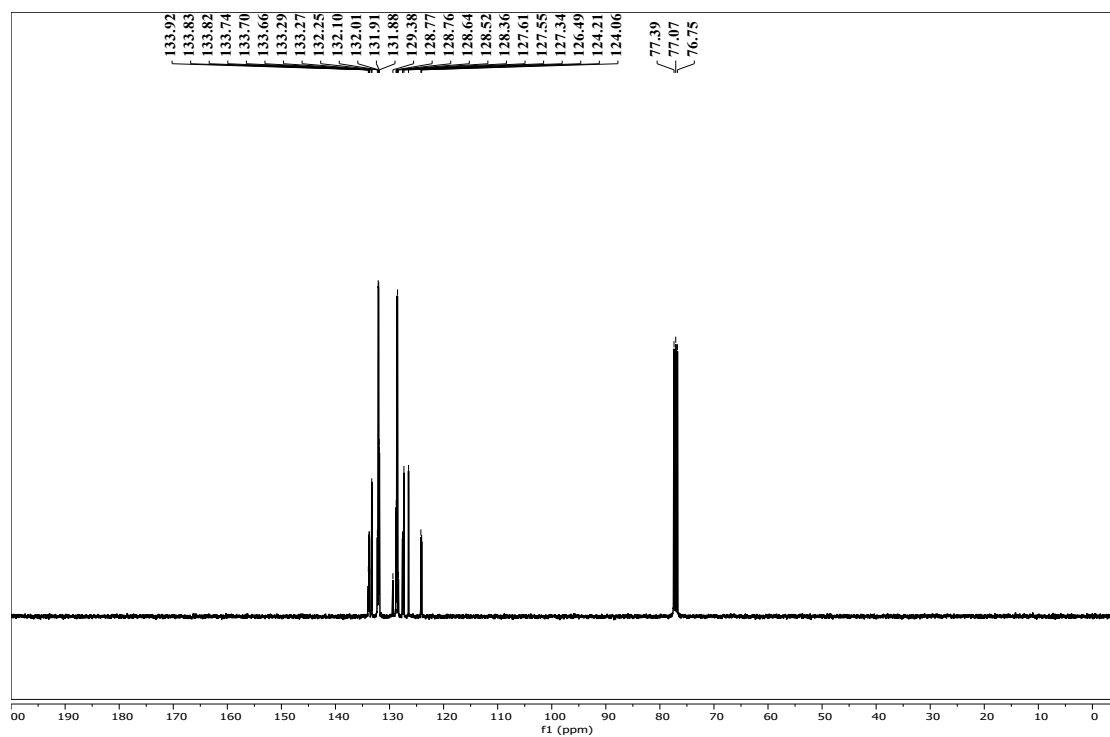




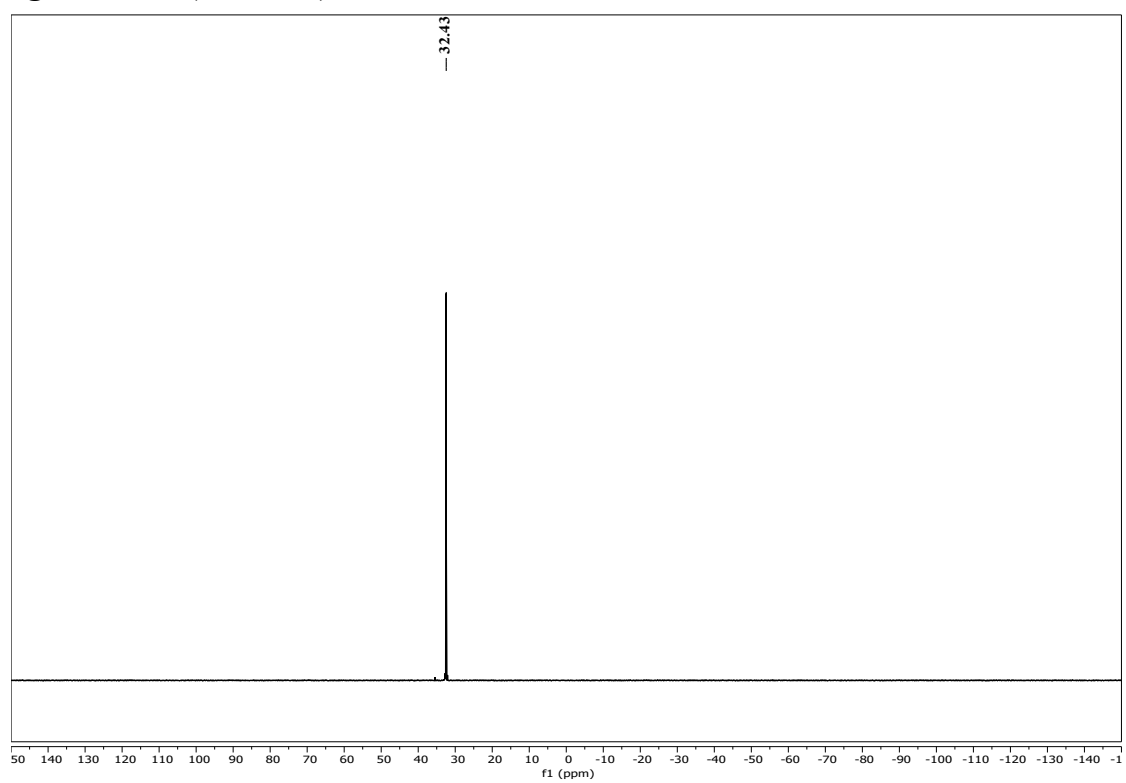
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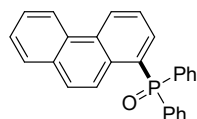


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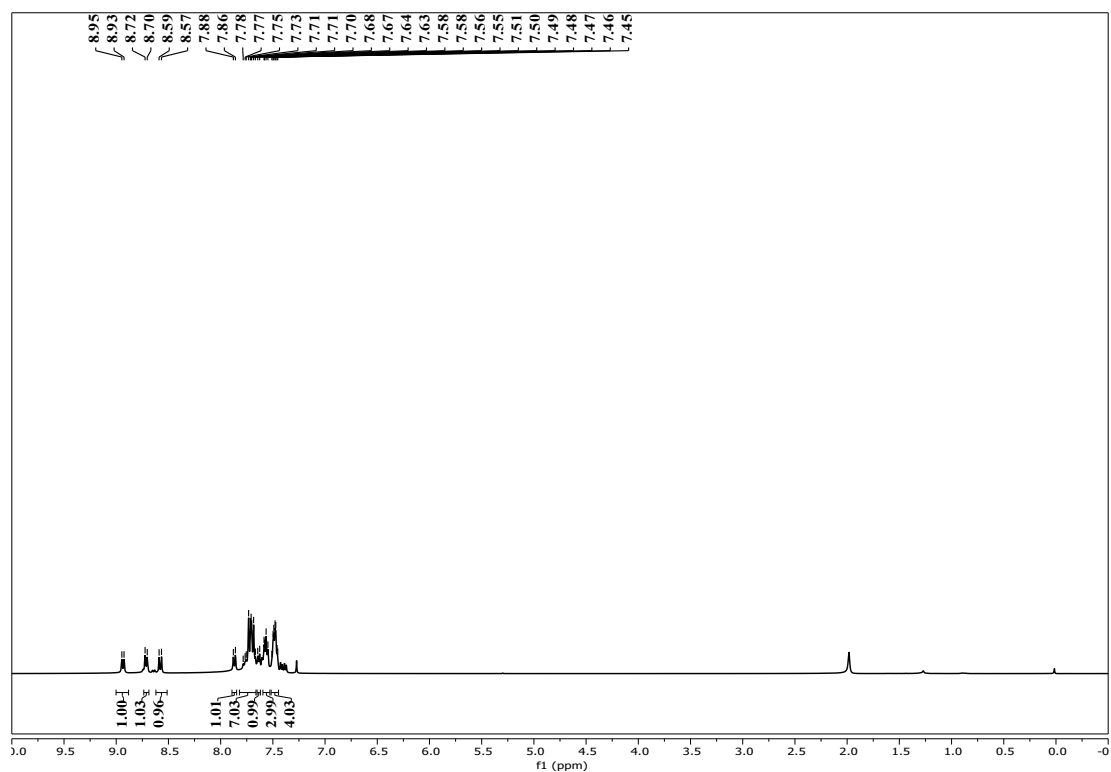


6g, ^{31}P NMR (162 MHz), CDCl_3

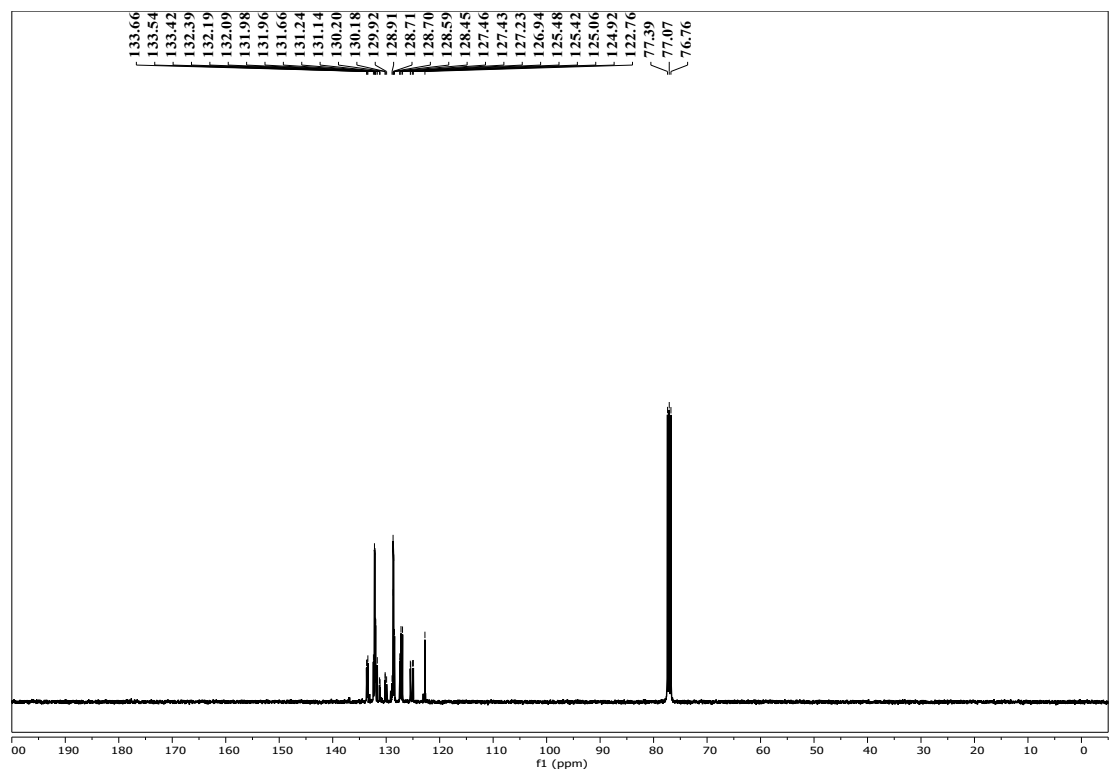




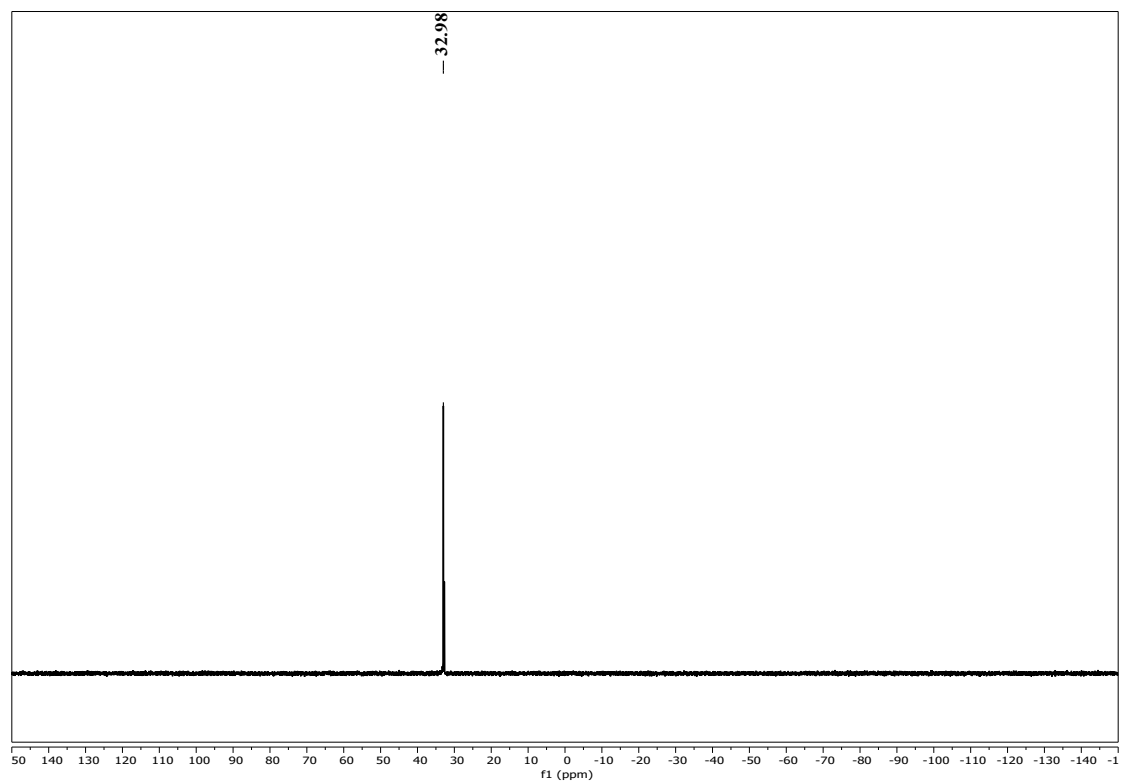
(main product) **6h**, ^1H NMR (400 MHz), CDCl_3

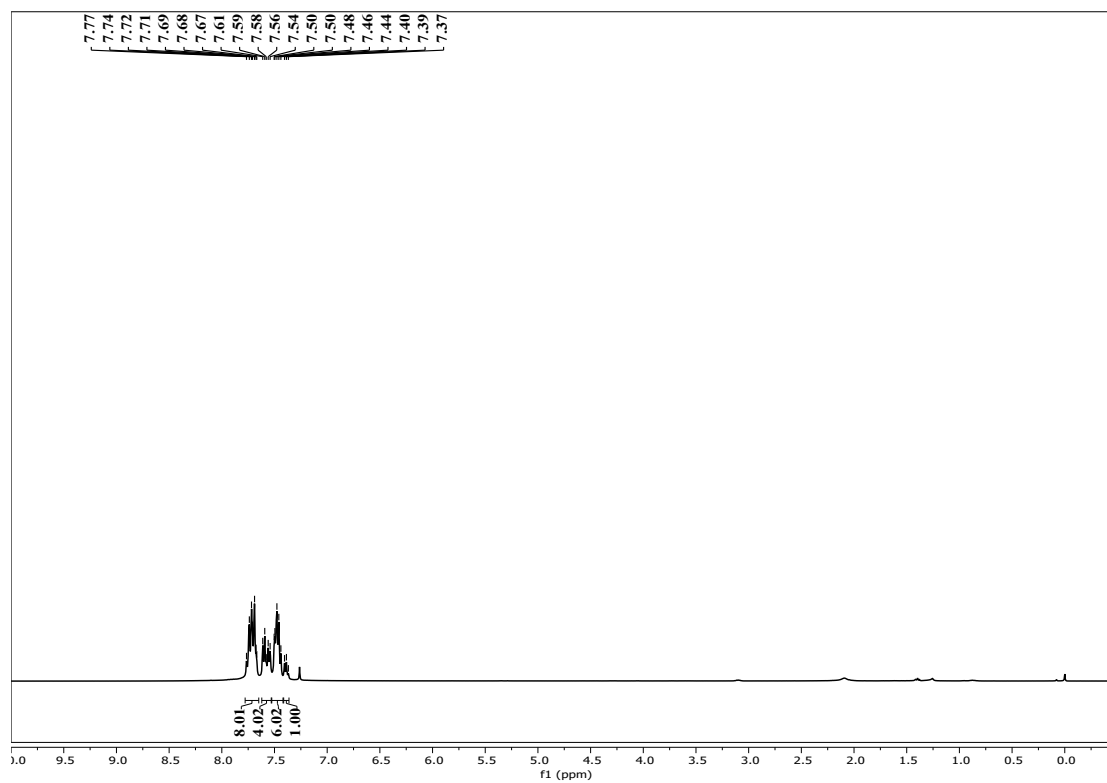


6h, ^{13}C NMR (101 MHz), CDCl_3

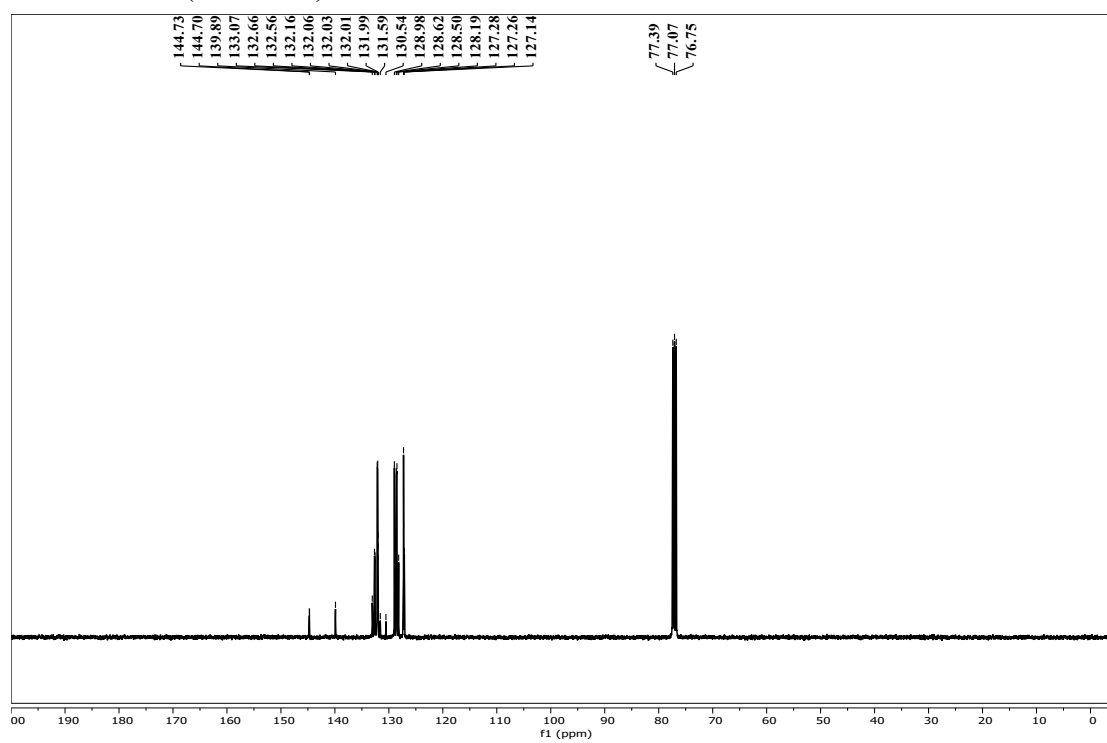


6h, ^{31}P NMR (162 MHz), CDCl_3

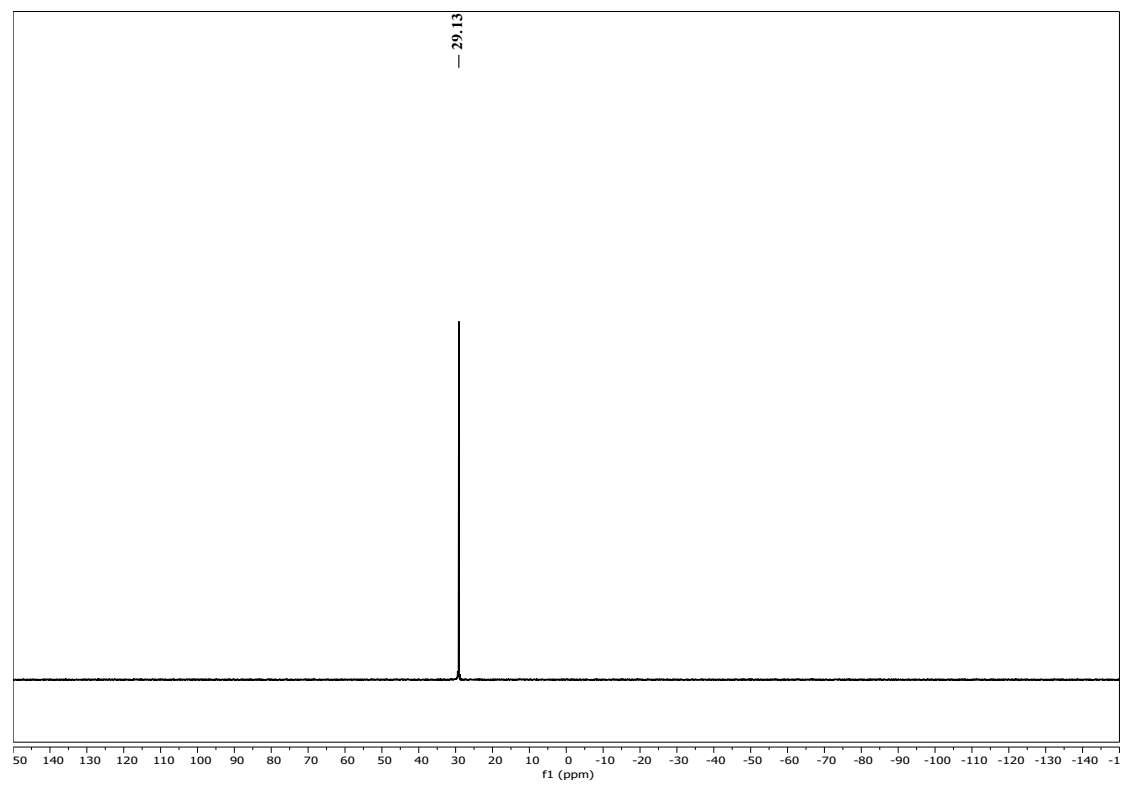


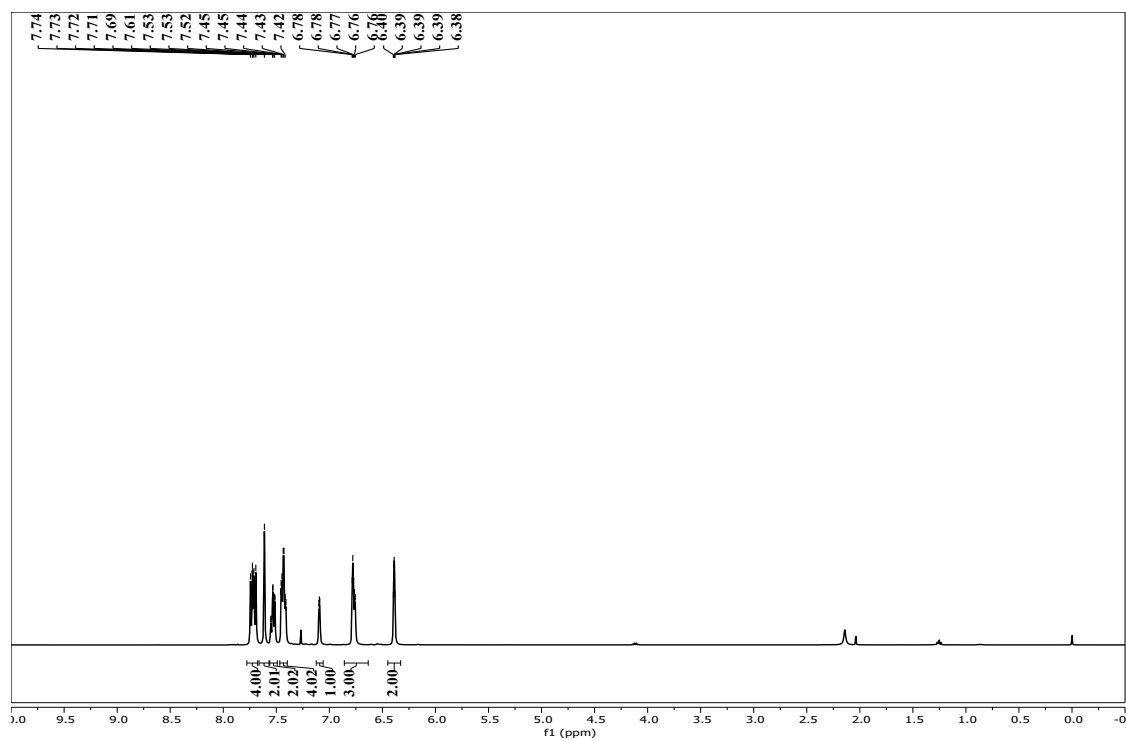
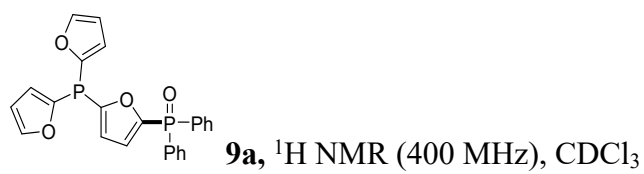


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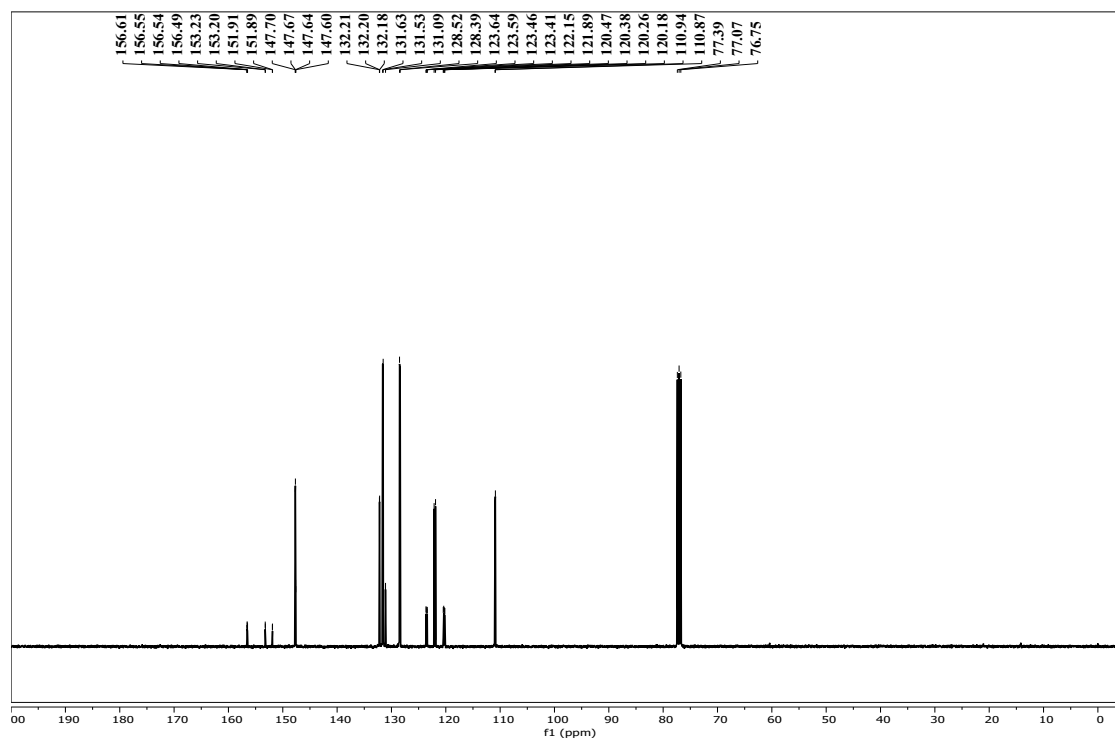


6i, ^{31}P NMR (162 MHz), CDCl_3

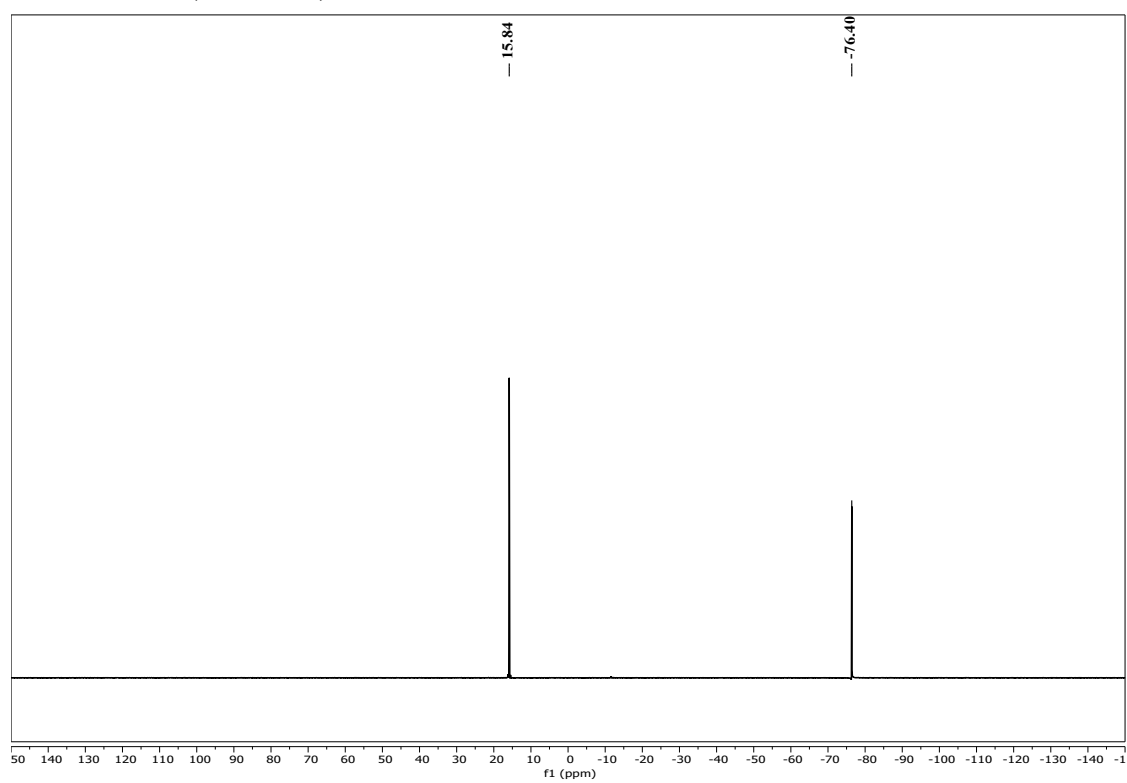


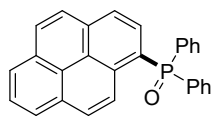


9a, ^{13}C NMR (101 MHz), CDCl_3

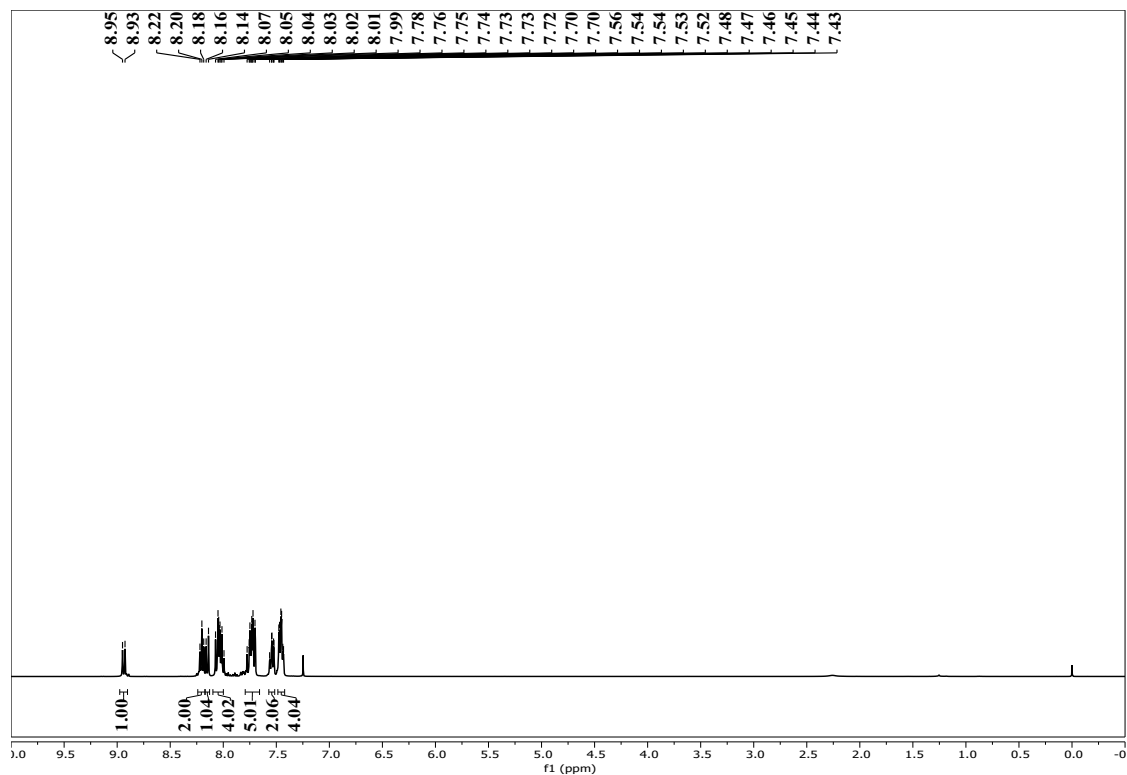


9a, ^{31}P NMR (162 MHz), CDCl_3

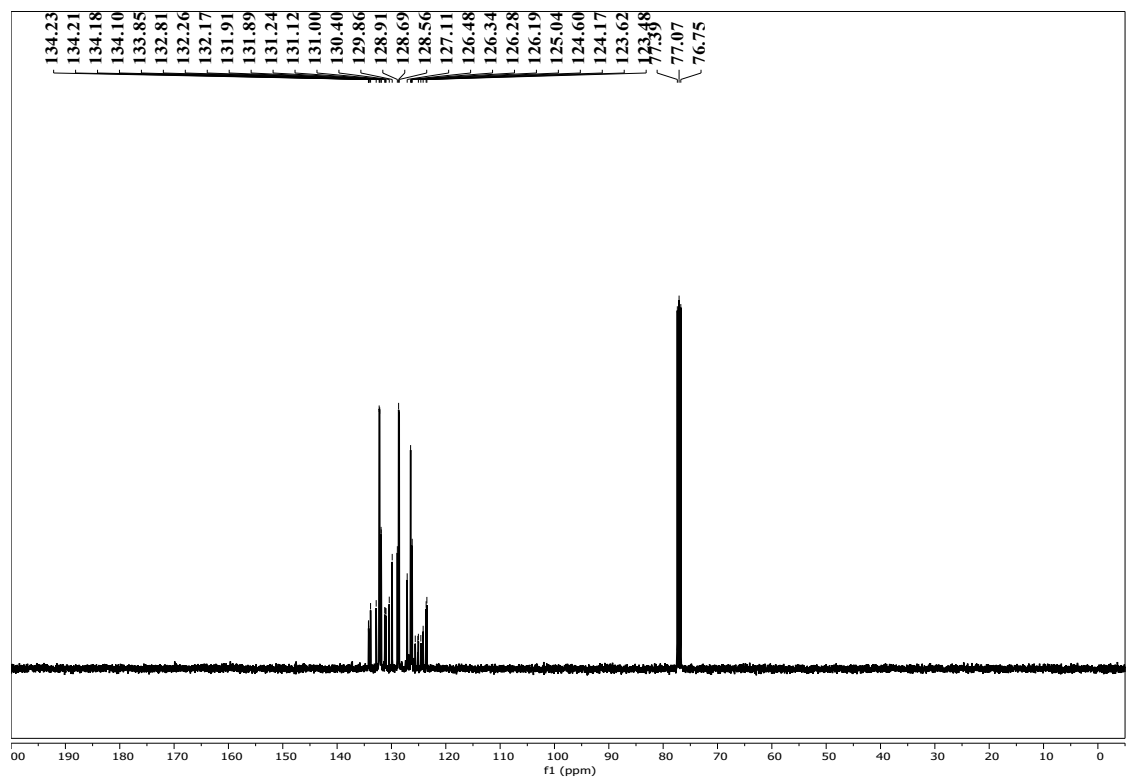




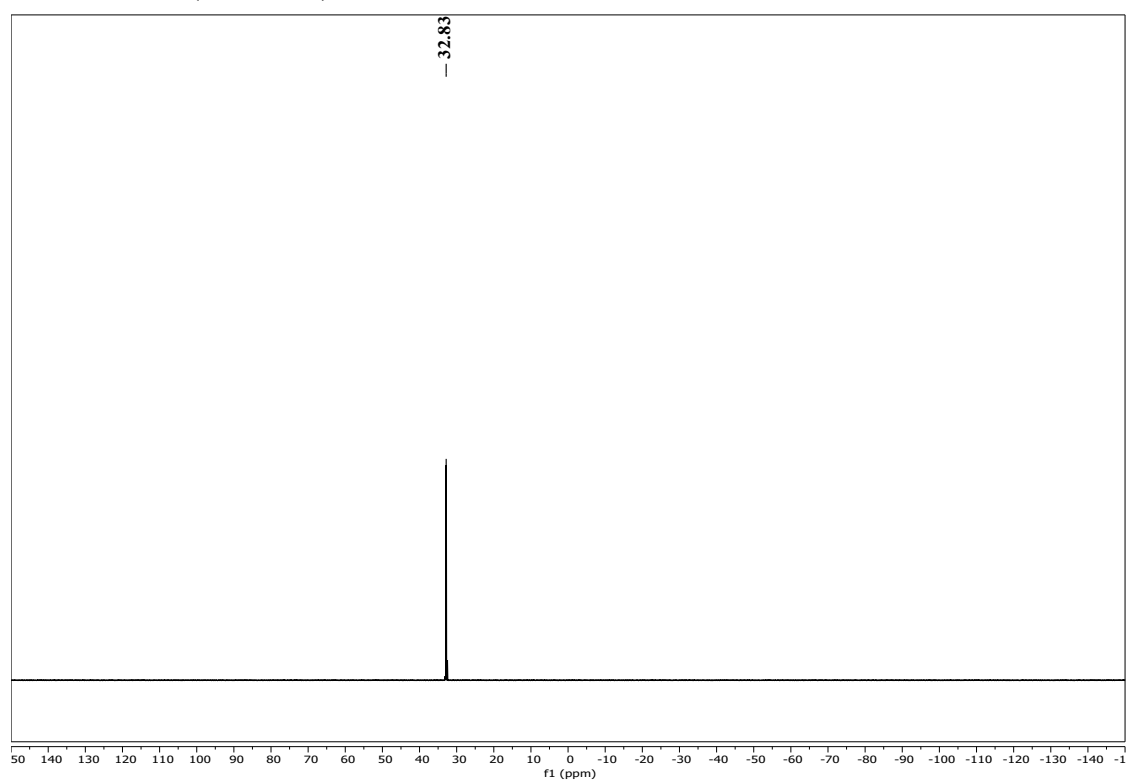
9b, ^1H NMR (400 MHz), CDCl_3 (main product)

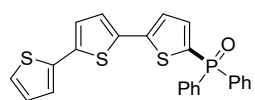


9b, ^{13}C NMR (101 MHz), CDCl_3

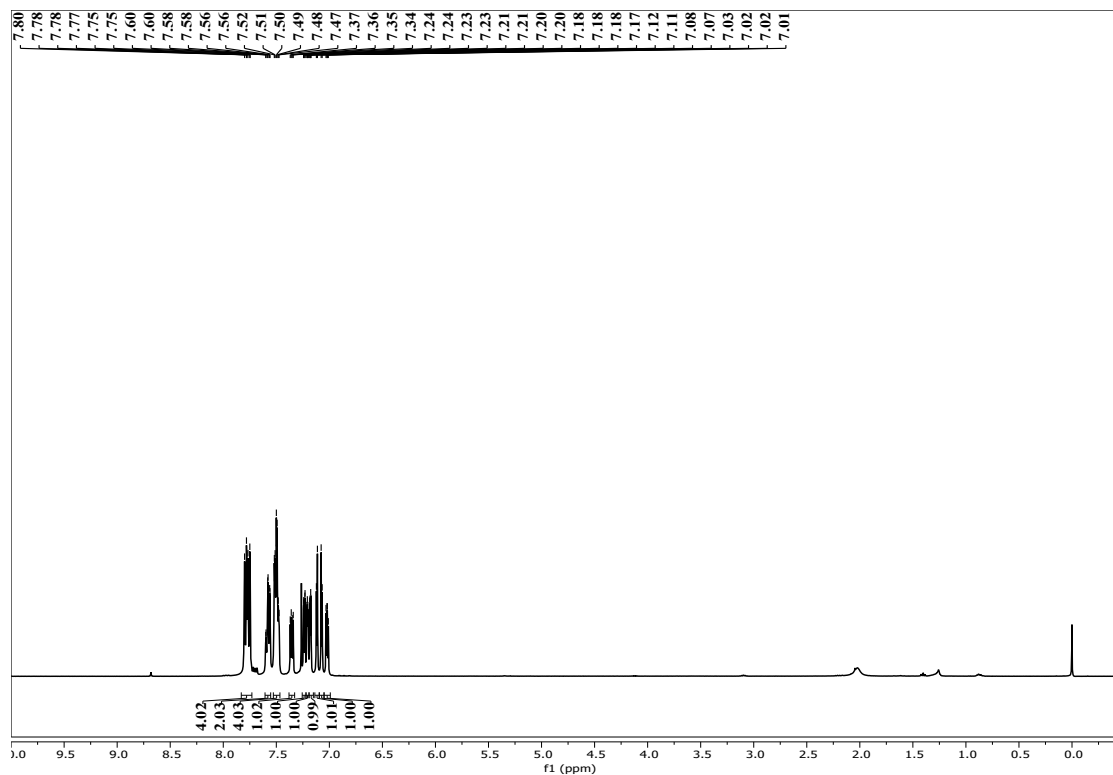


9b, ^{31}P NMR (162 MHz), CDCl_3

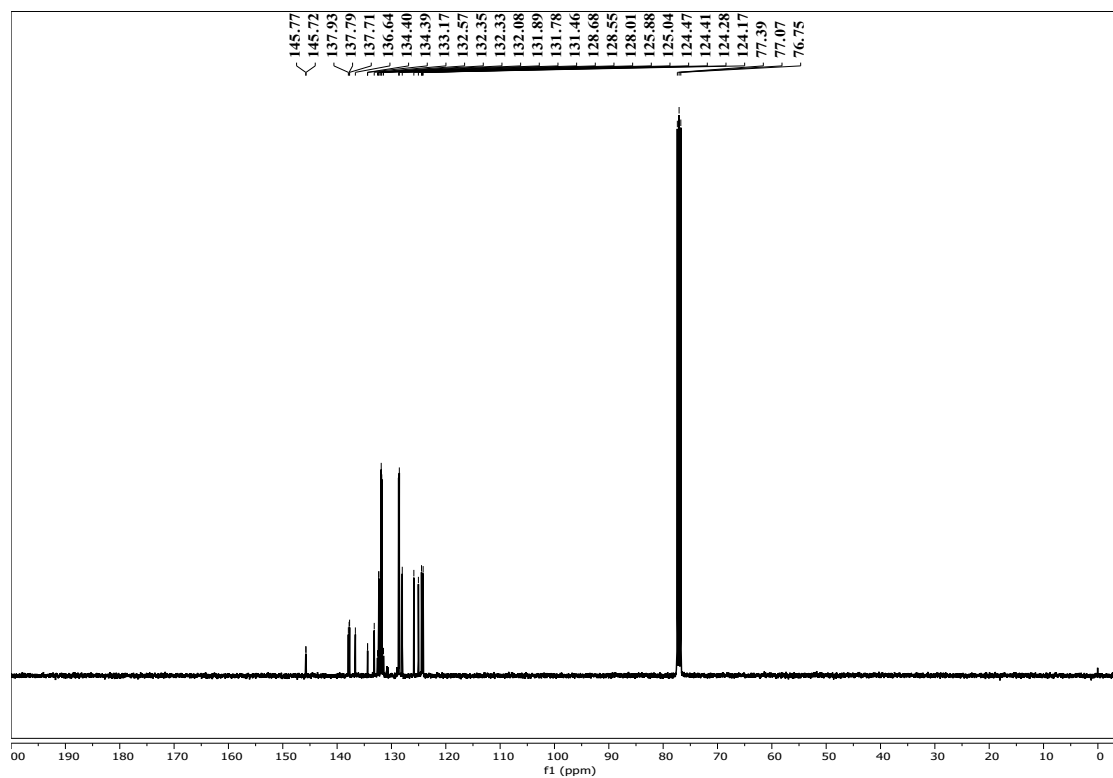




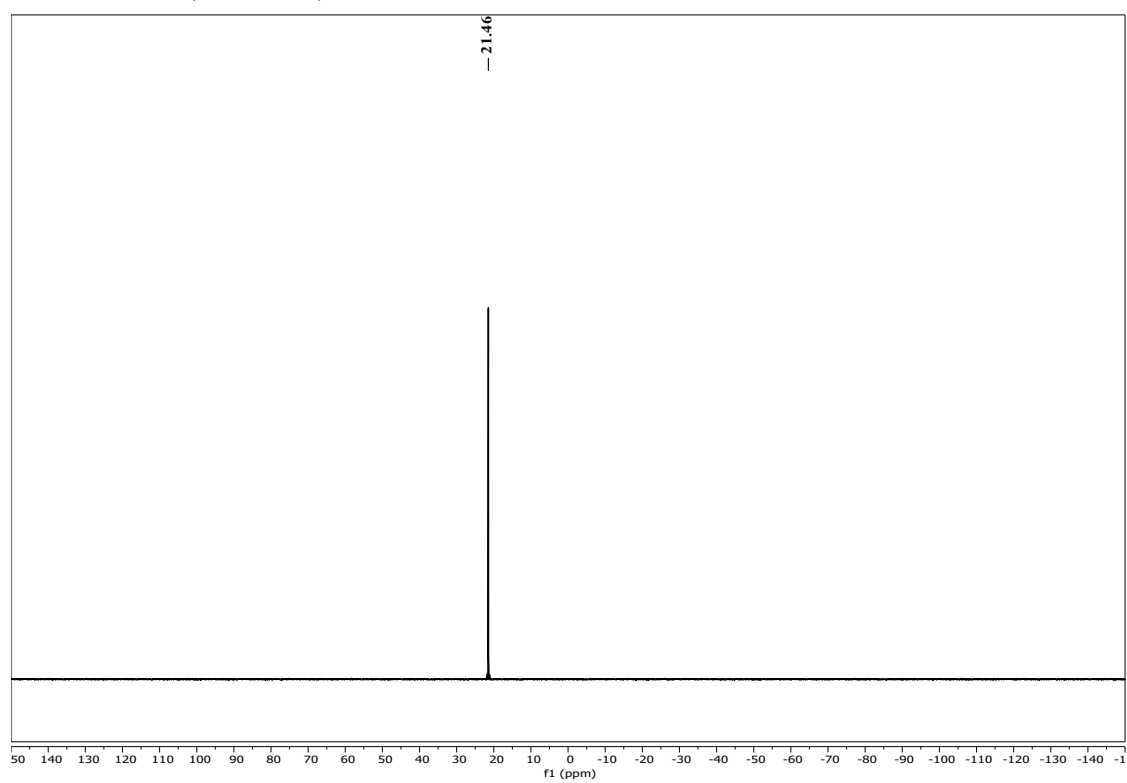
9c, ^1H NMR (400 MHz), CDCl_3

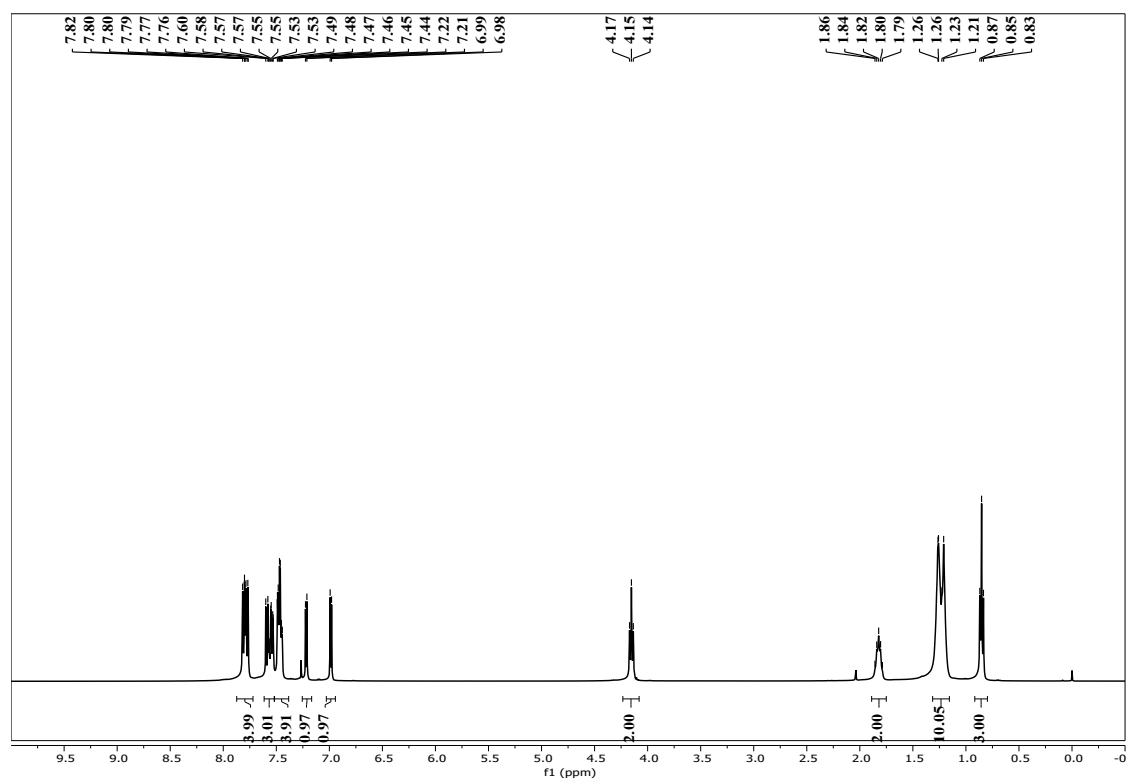
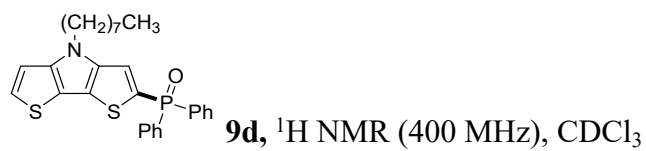


9c, ^{13}C NMR (101 MHz), CDCl_3

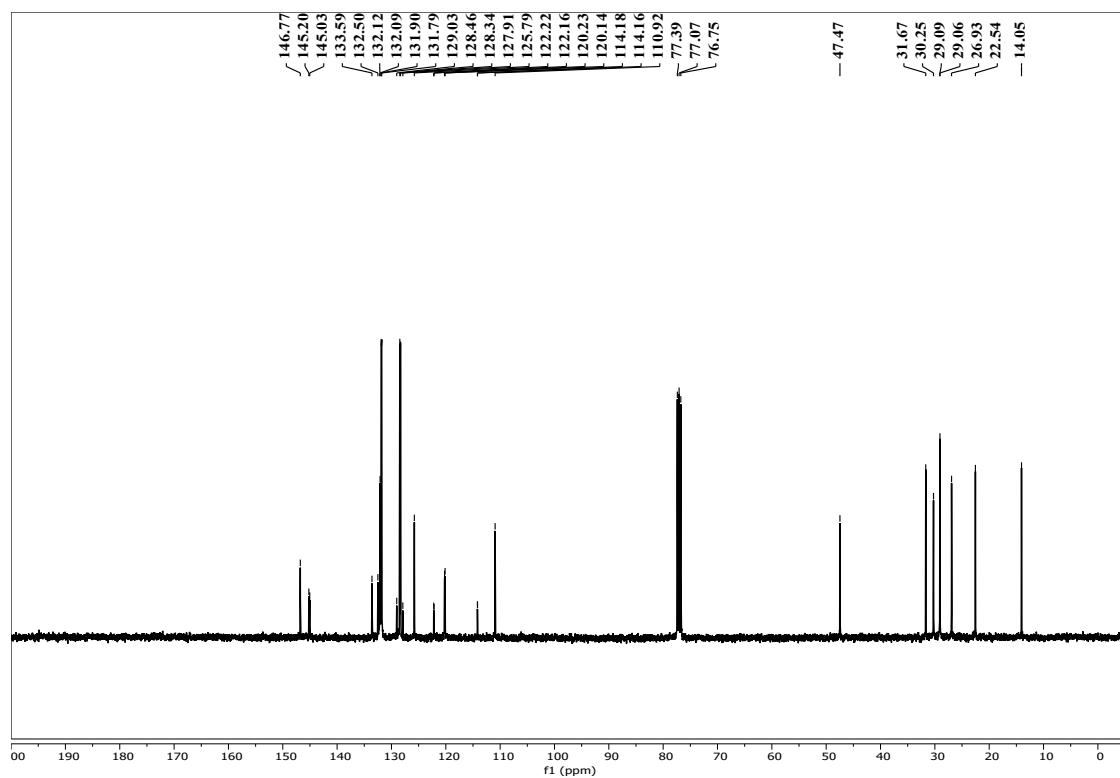


9c, ^{31}P NMR (162 MHz), CDCl_3

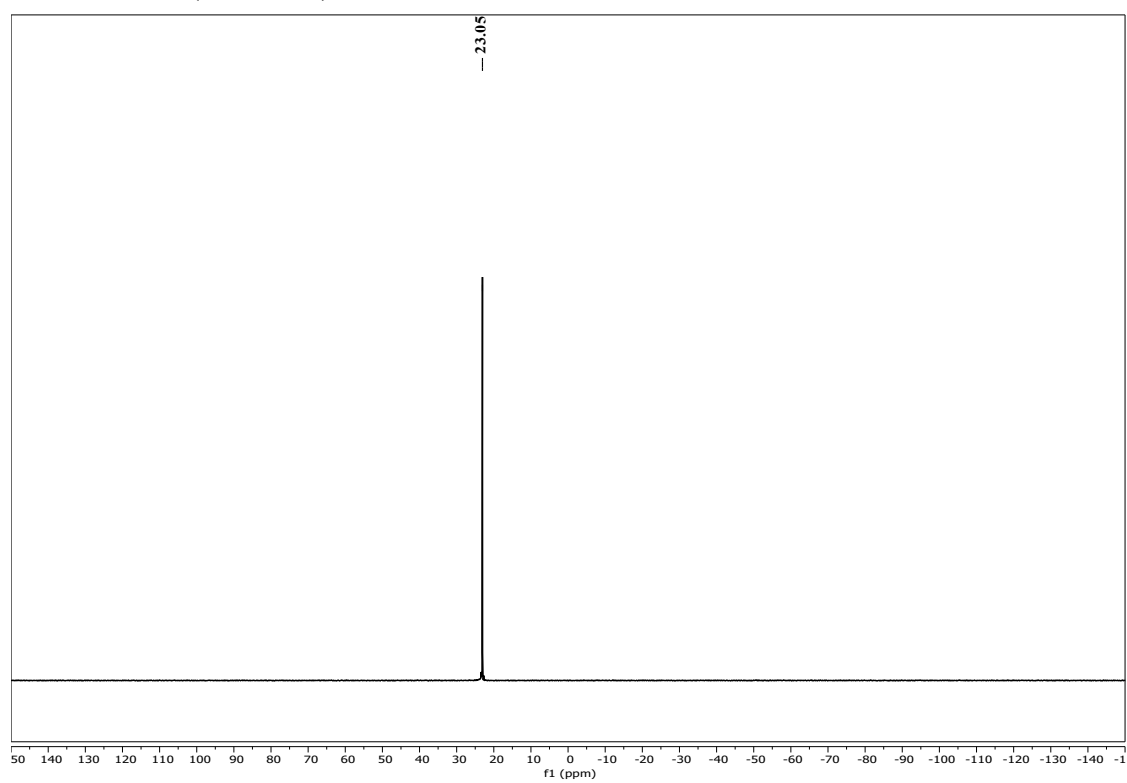


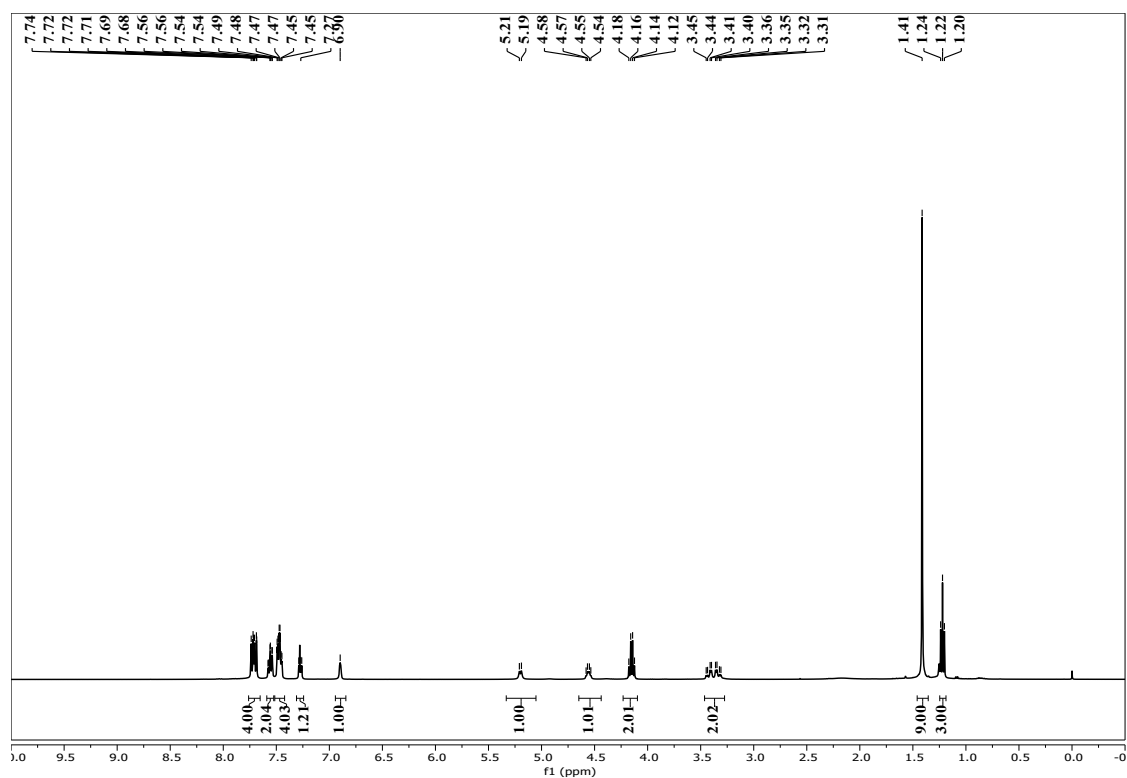
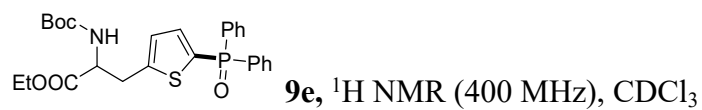


9d, ^{13}C NMR (101 MHz), CDCl_3

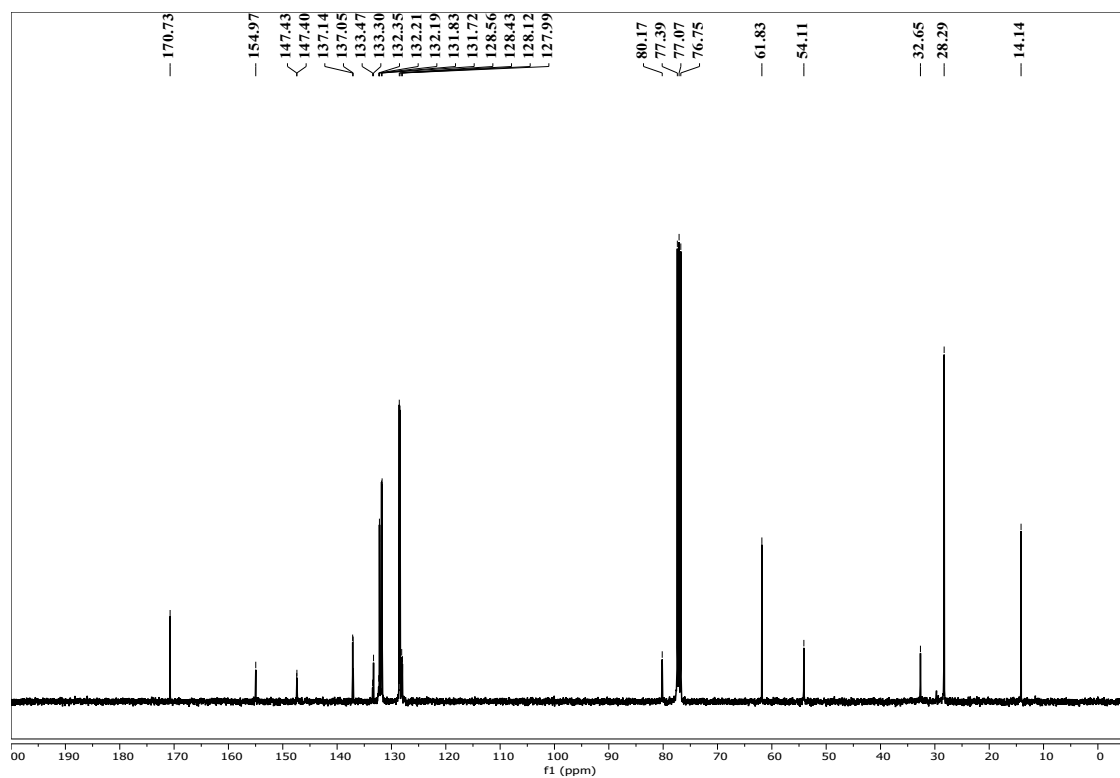


9d, ^{31}P NMR (162 MHz), CDCl_3

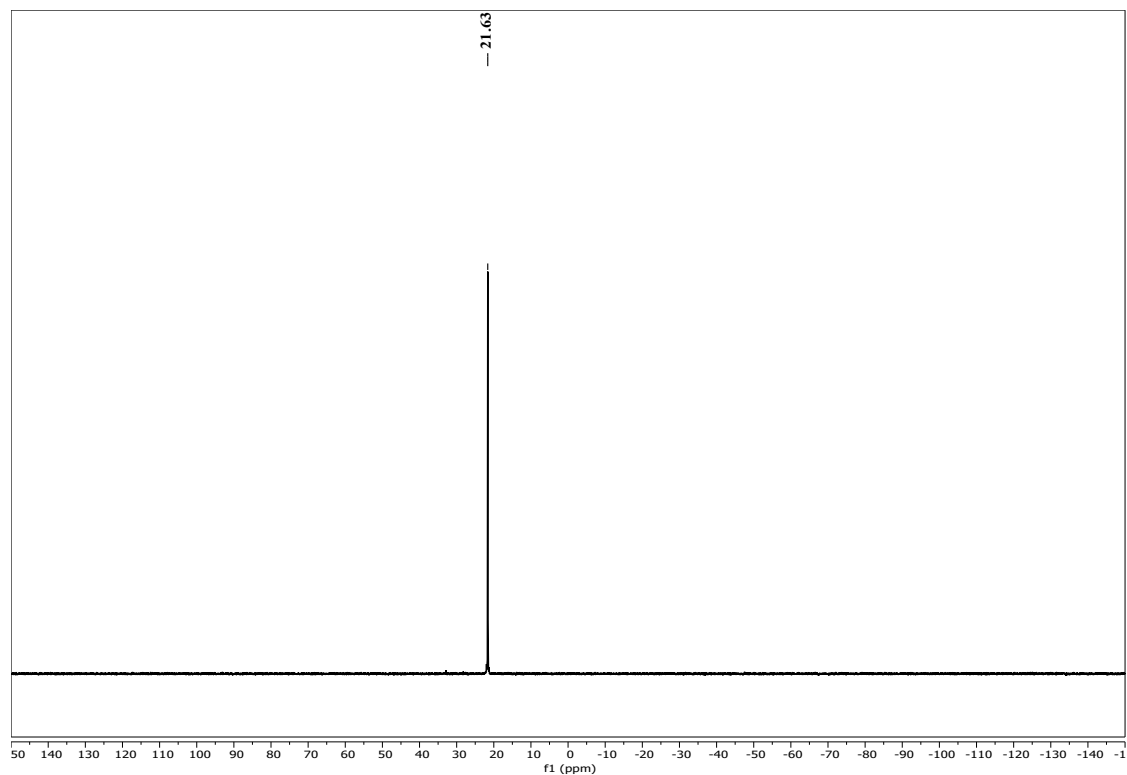


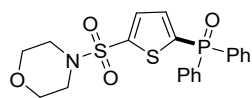


9e, ^{13}C NMR (101 MHz), CDCl_3

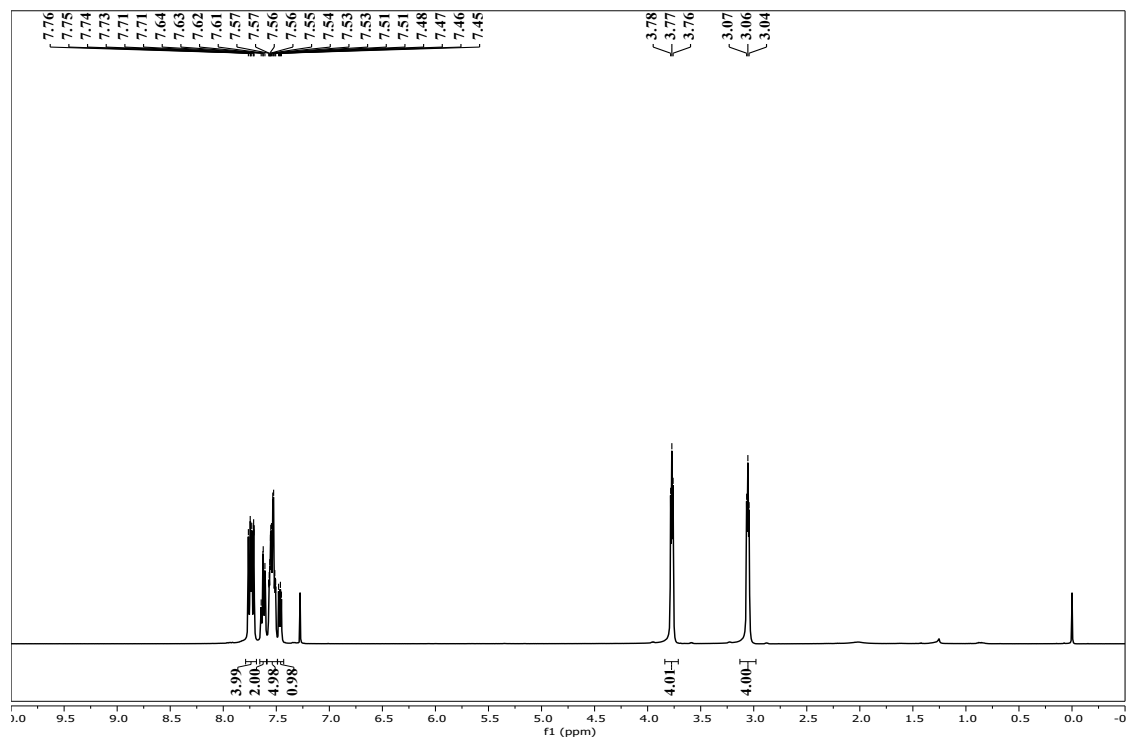


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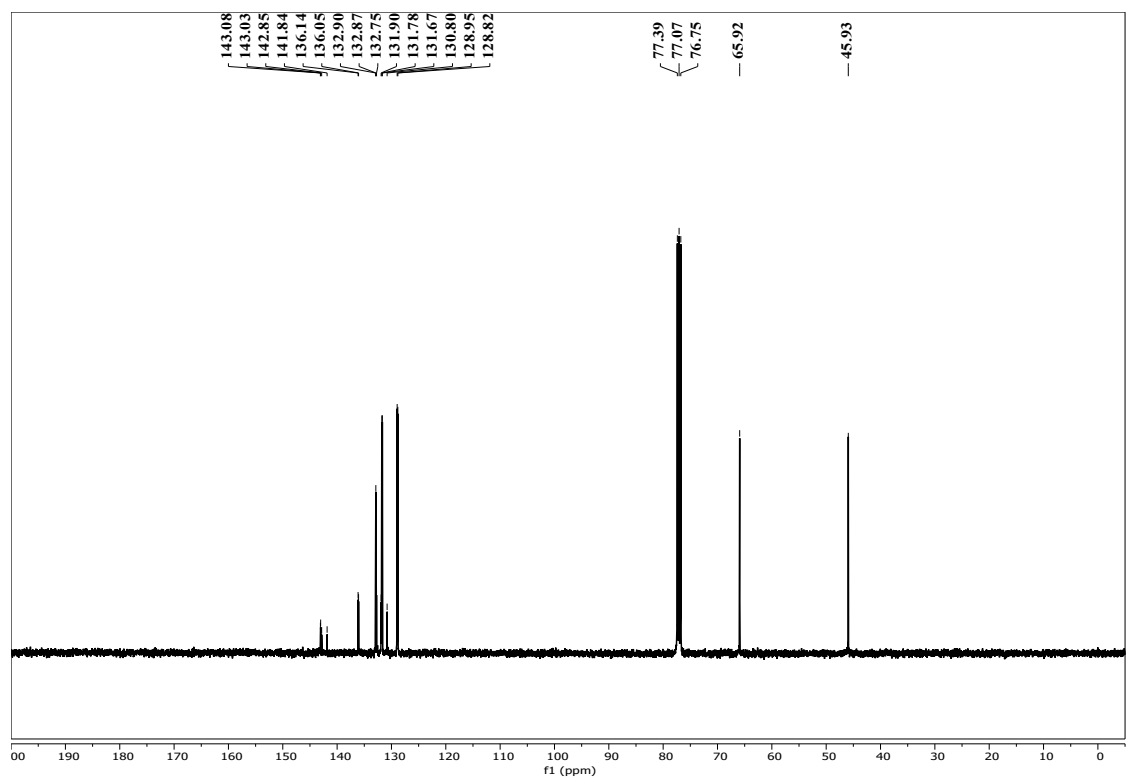




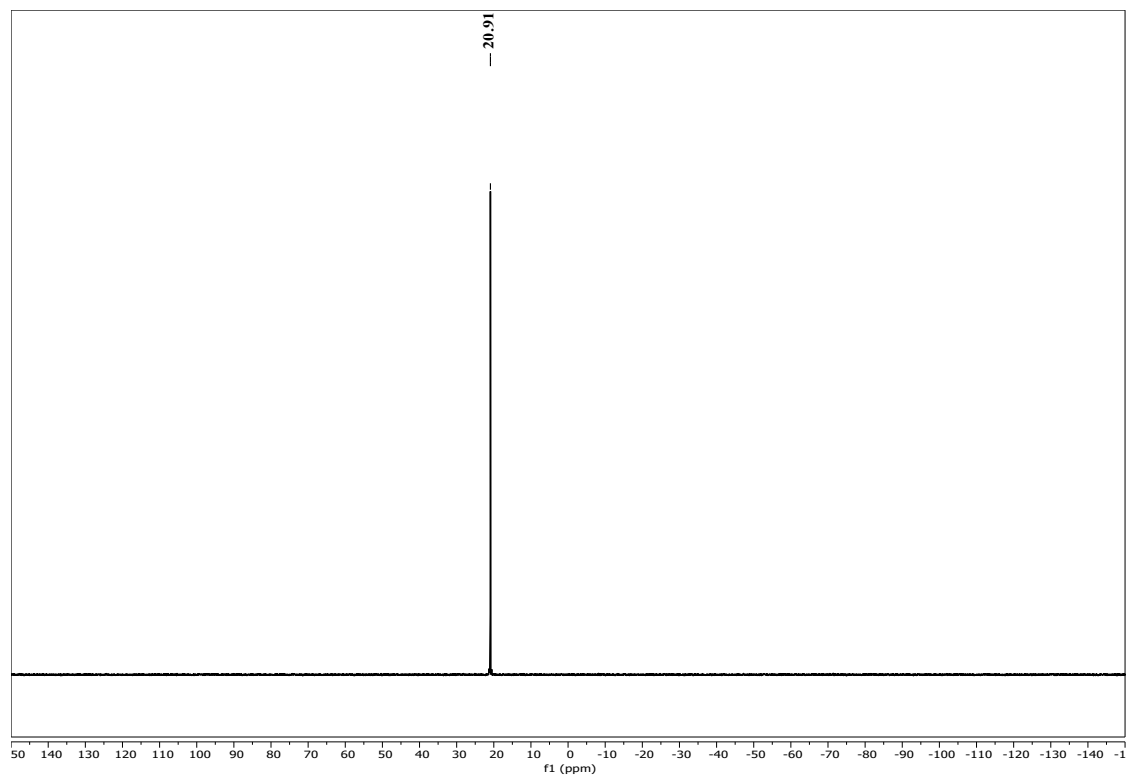
9f, ^1H NMR (400 MHz), CDCl_3

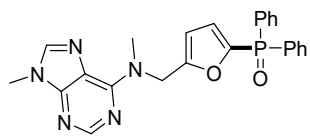


9f, ^{13}C NMR (101 MHz), CDCl_3

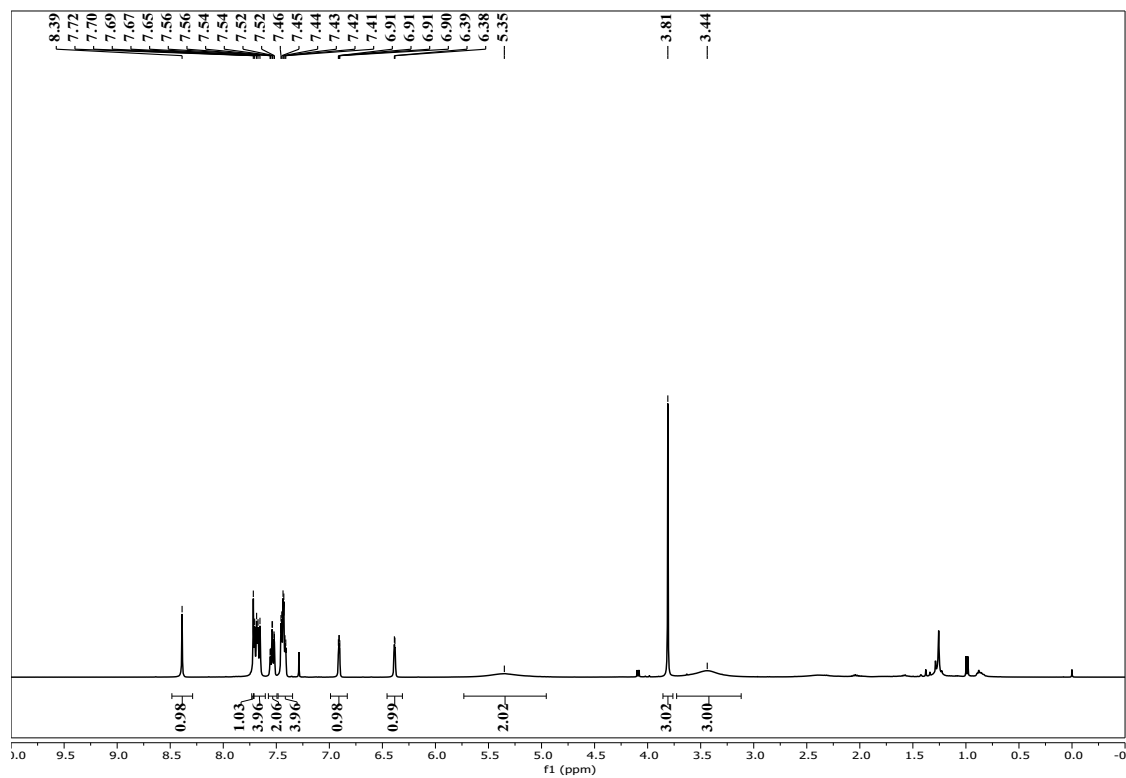


9f, ^{31}P NMR (162 MHz), CDCl_3

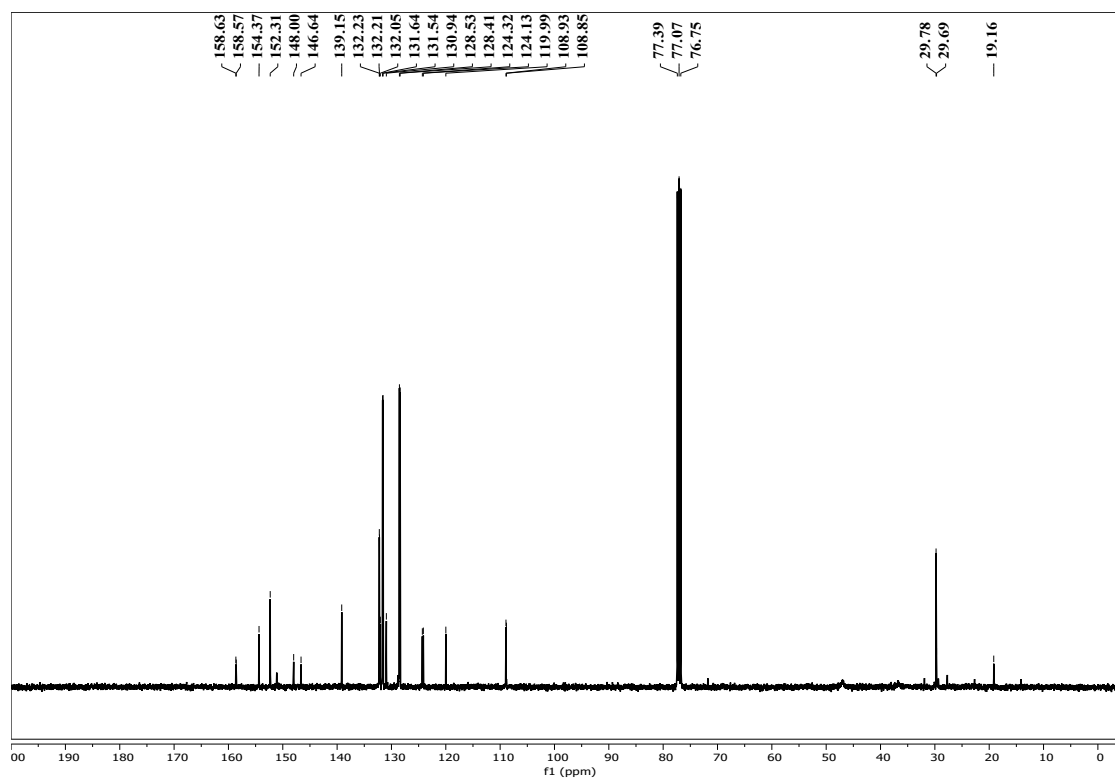




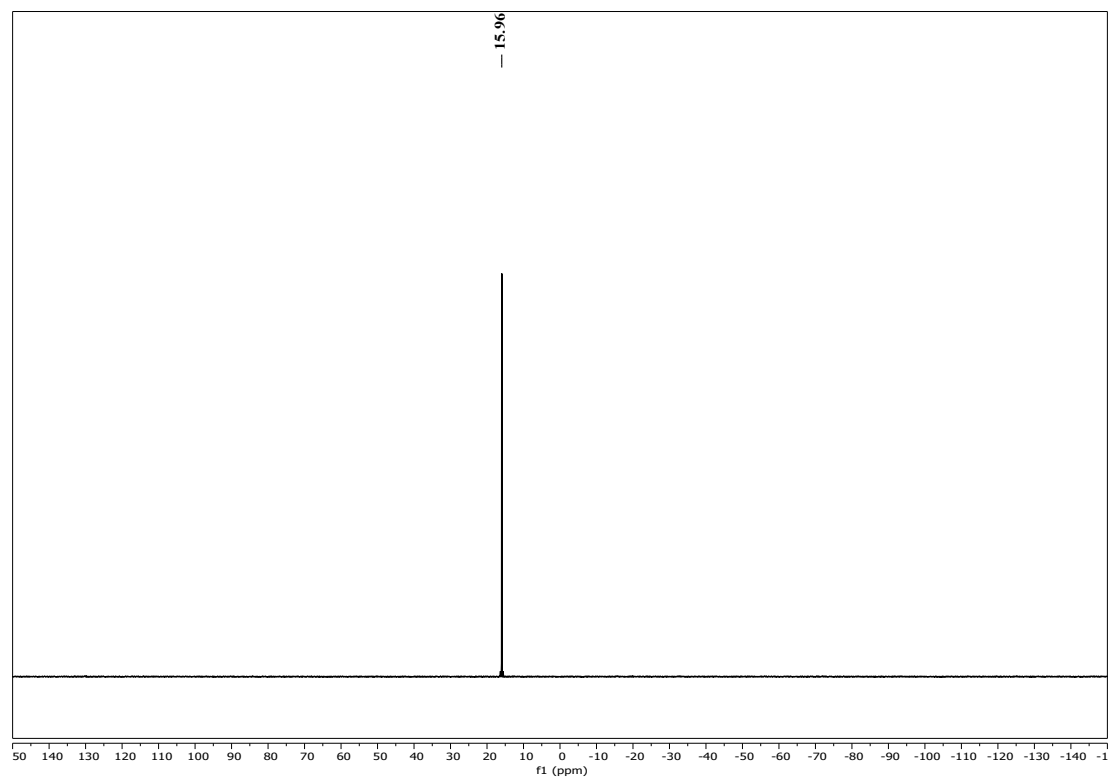
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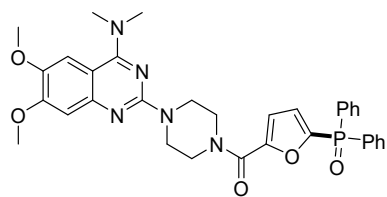


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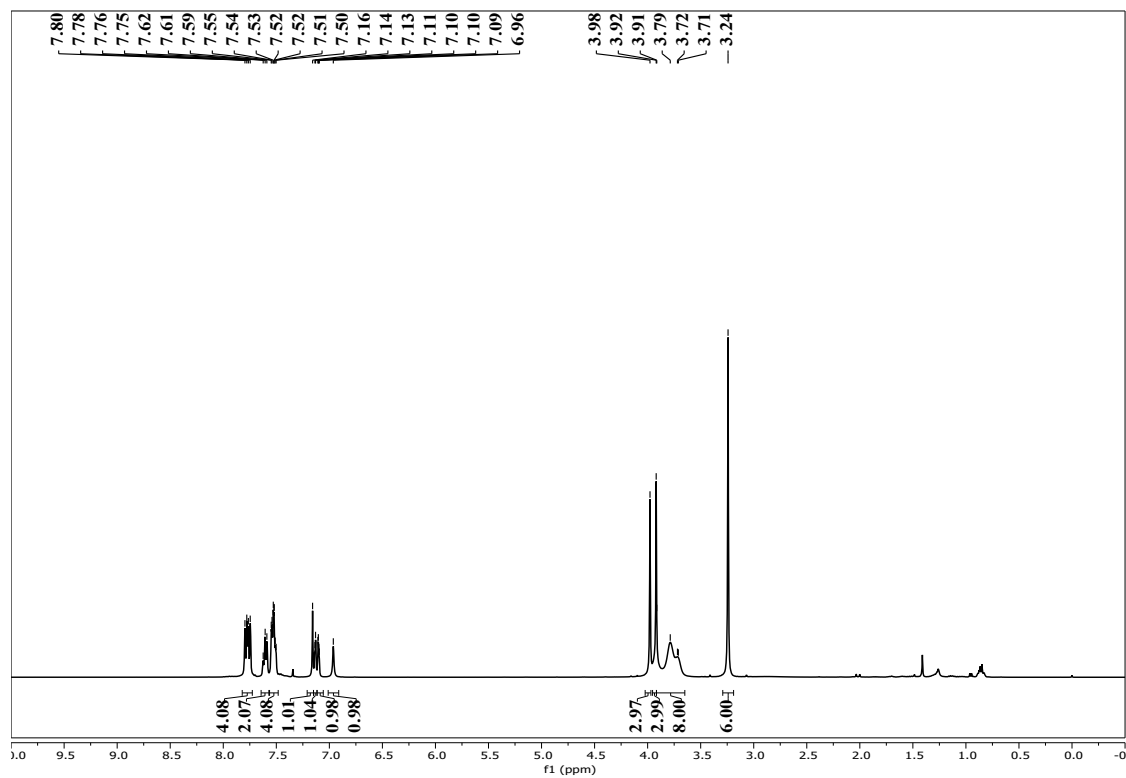


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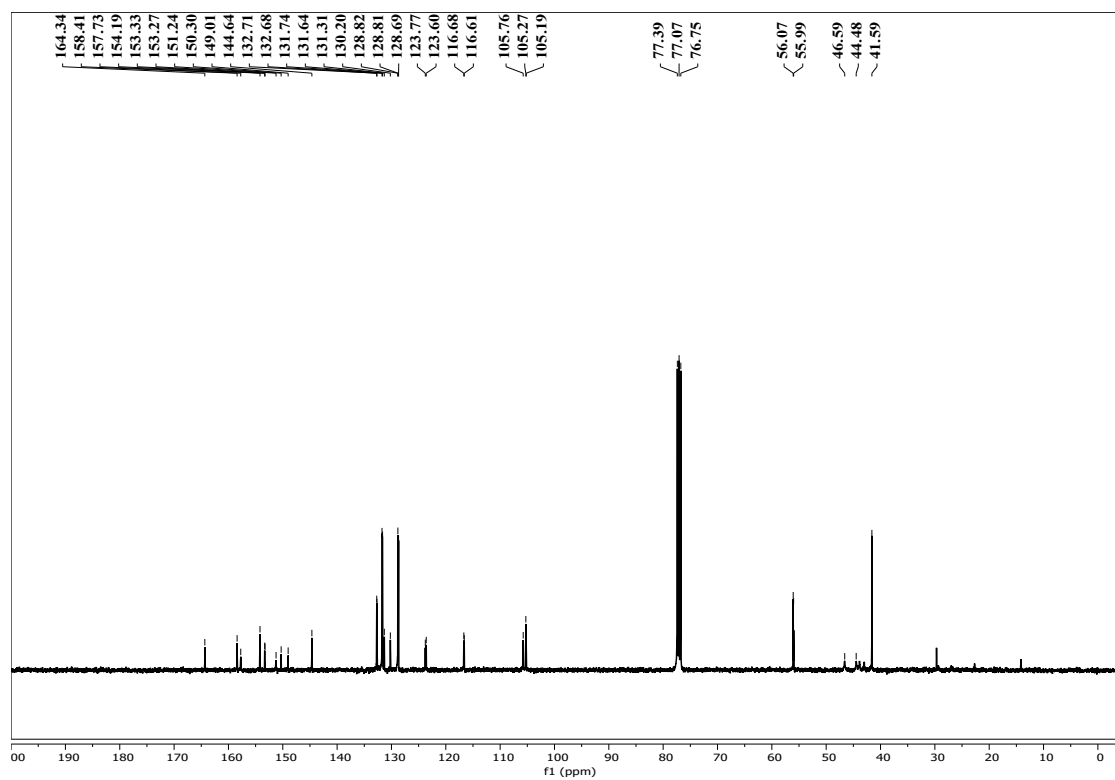




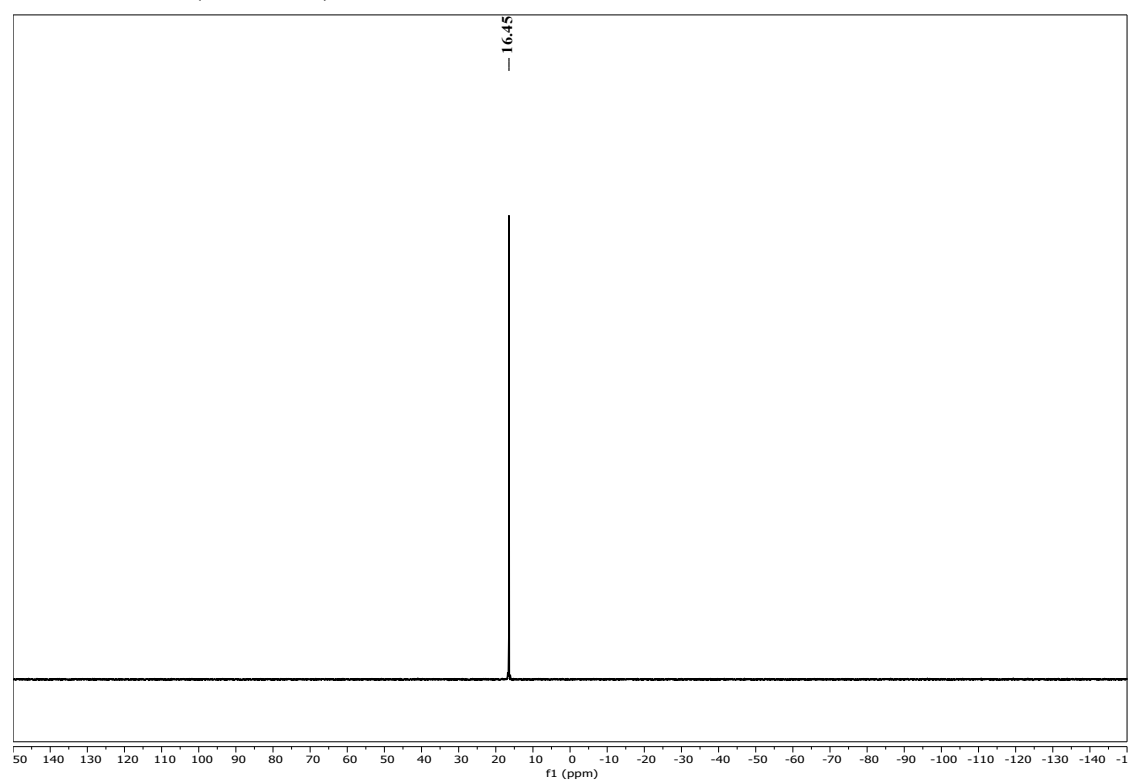
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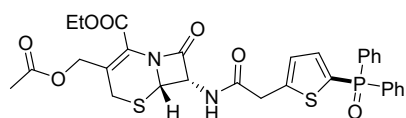


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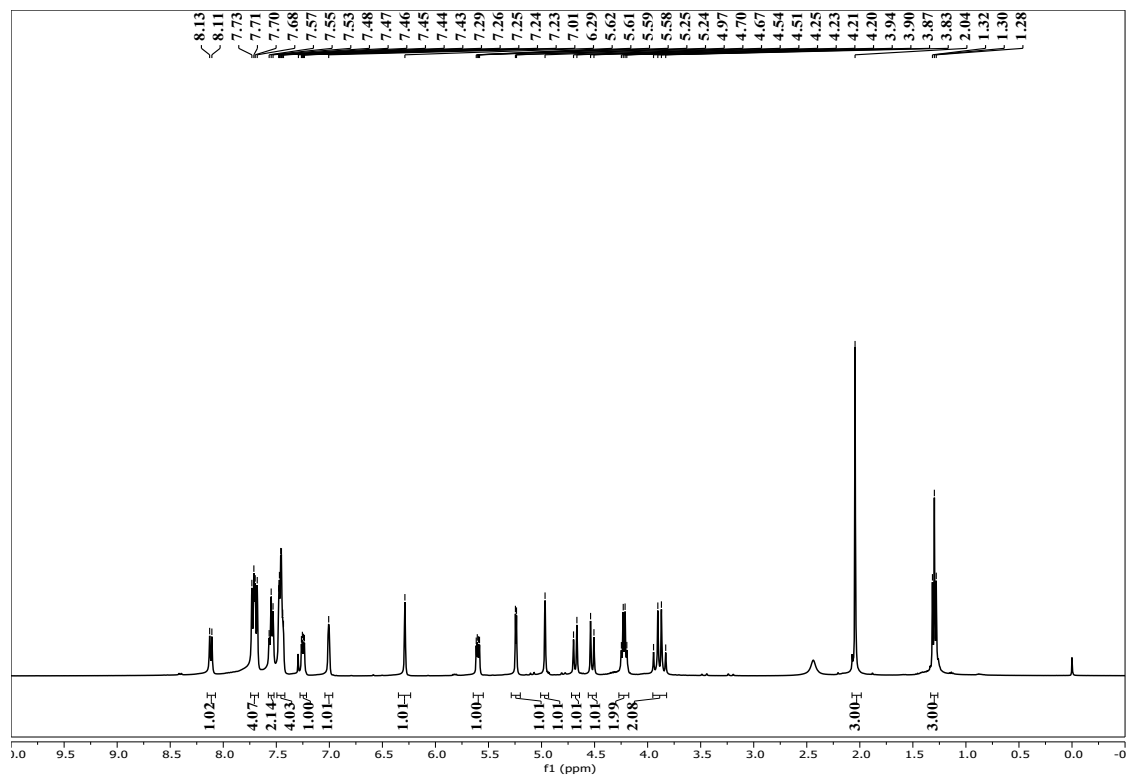


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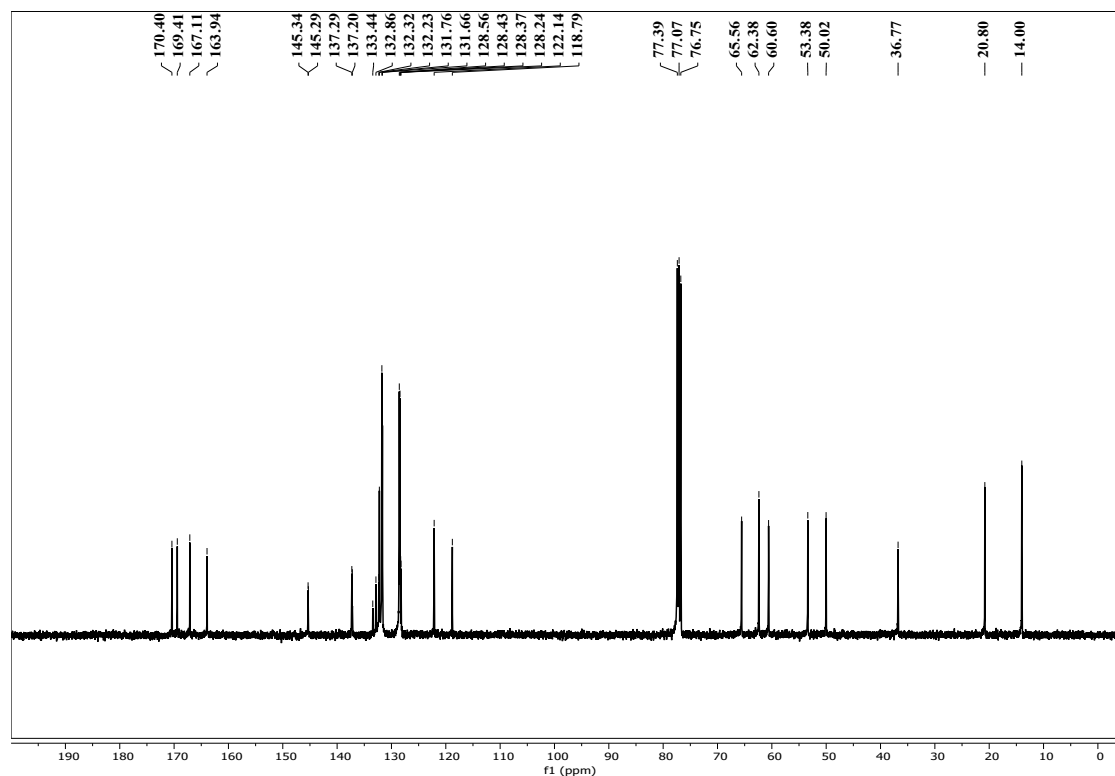




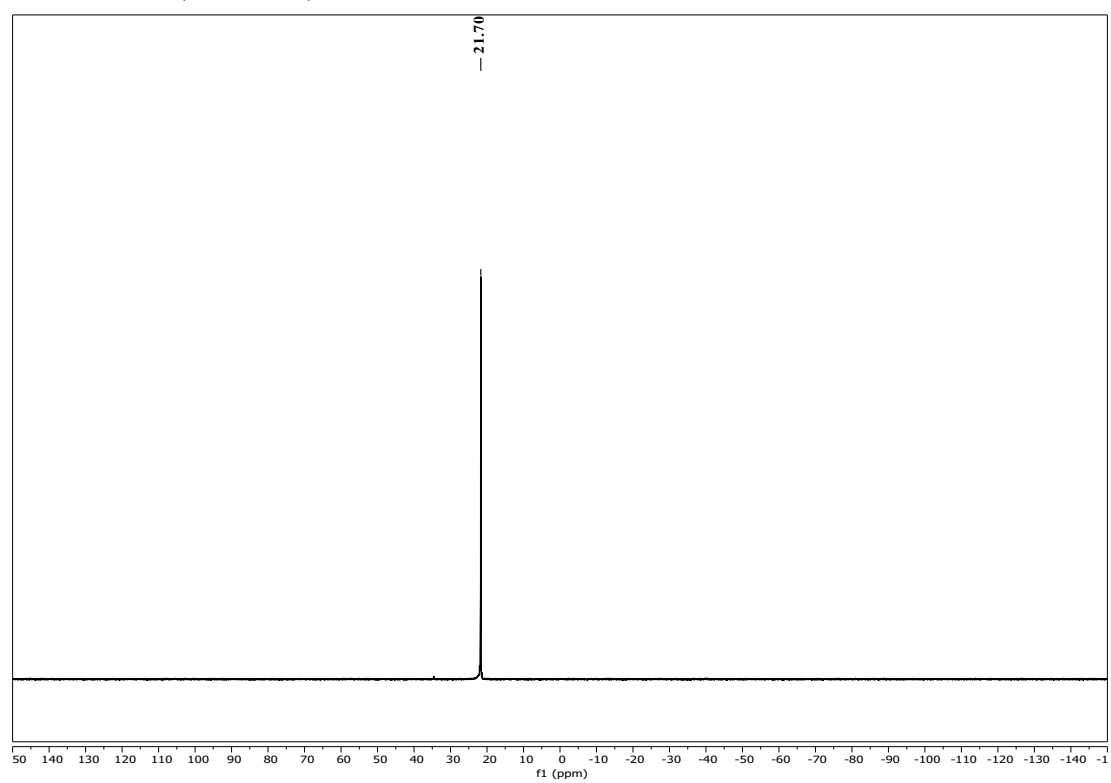
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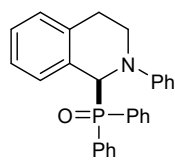


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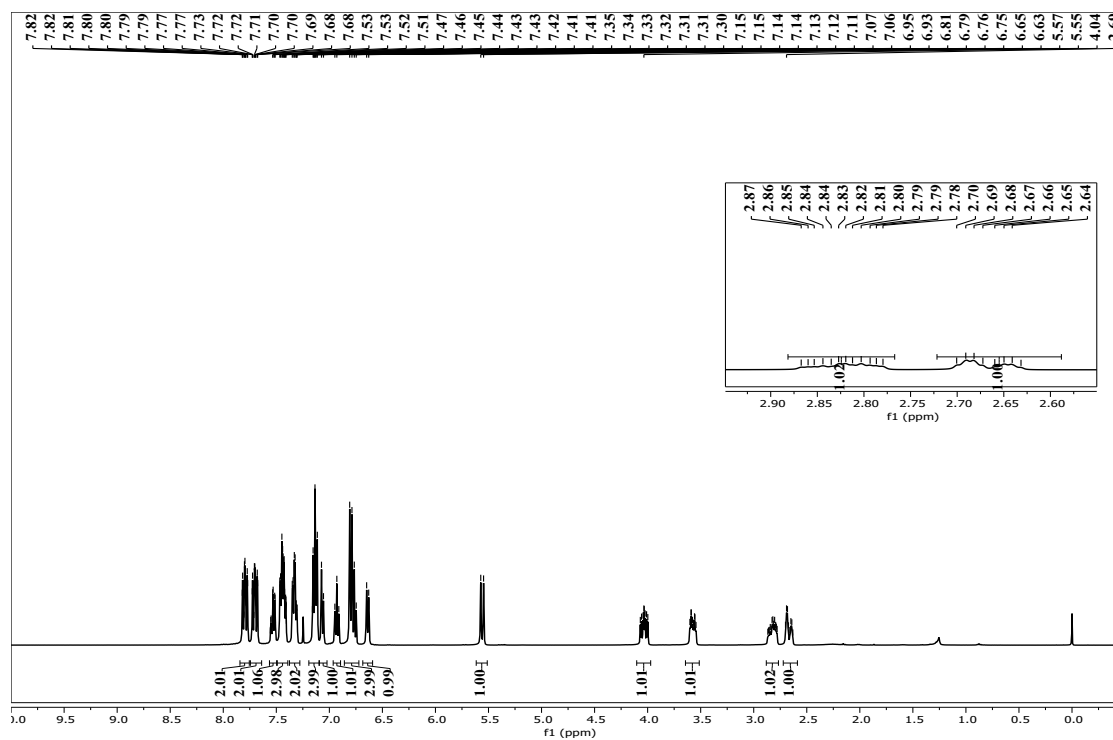


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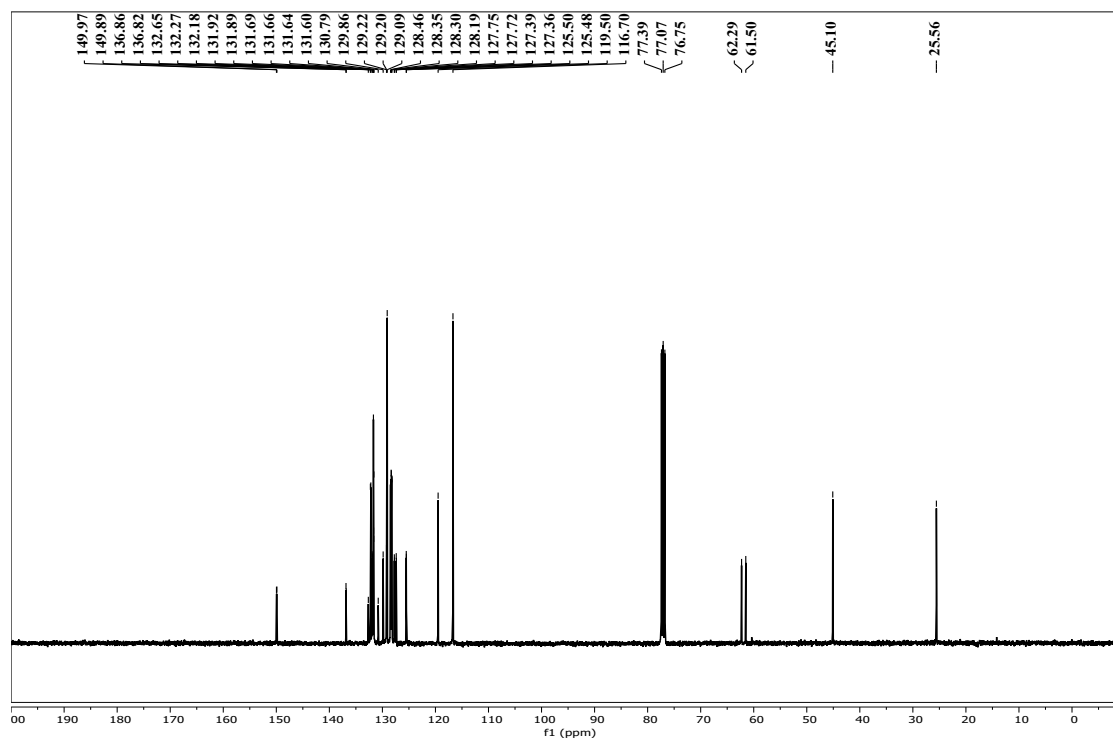




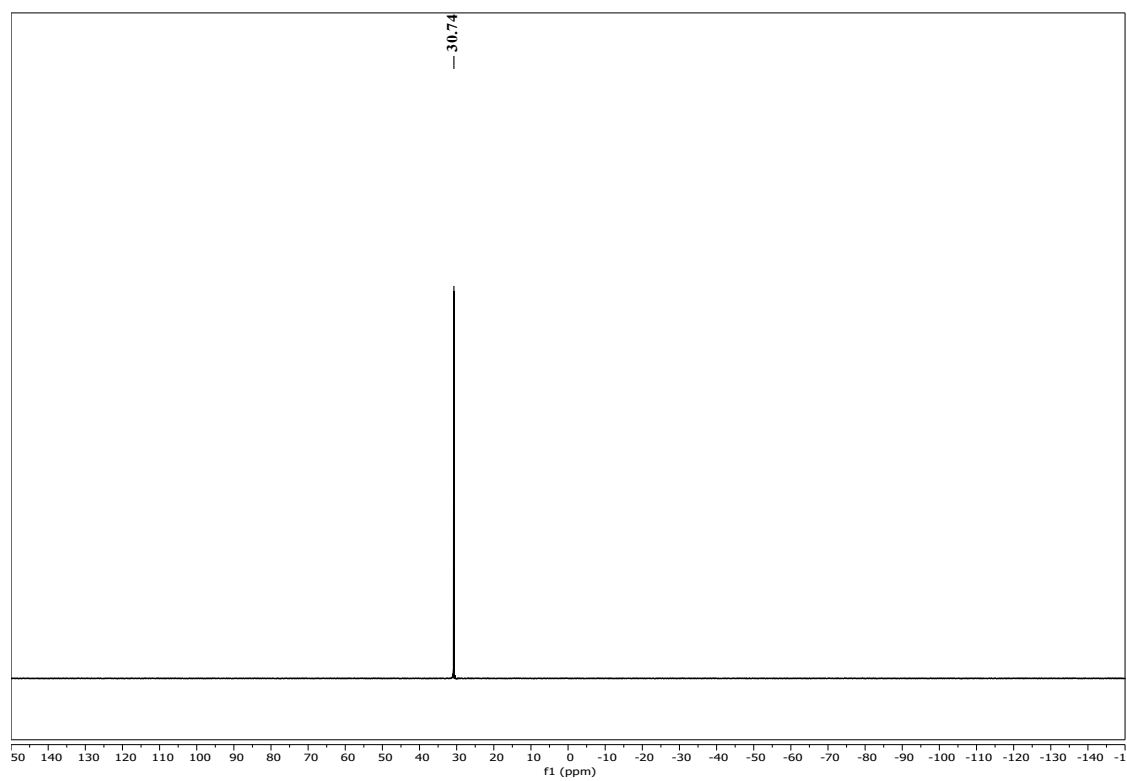
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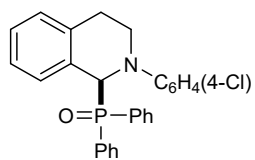


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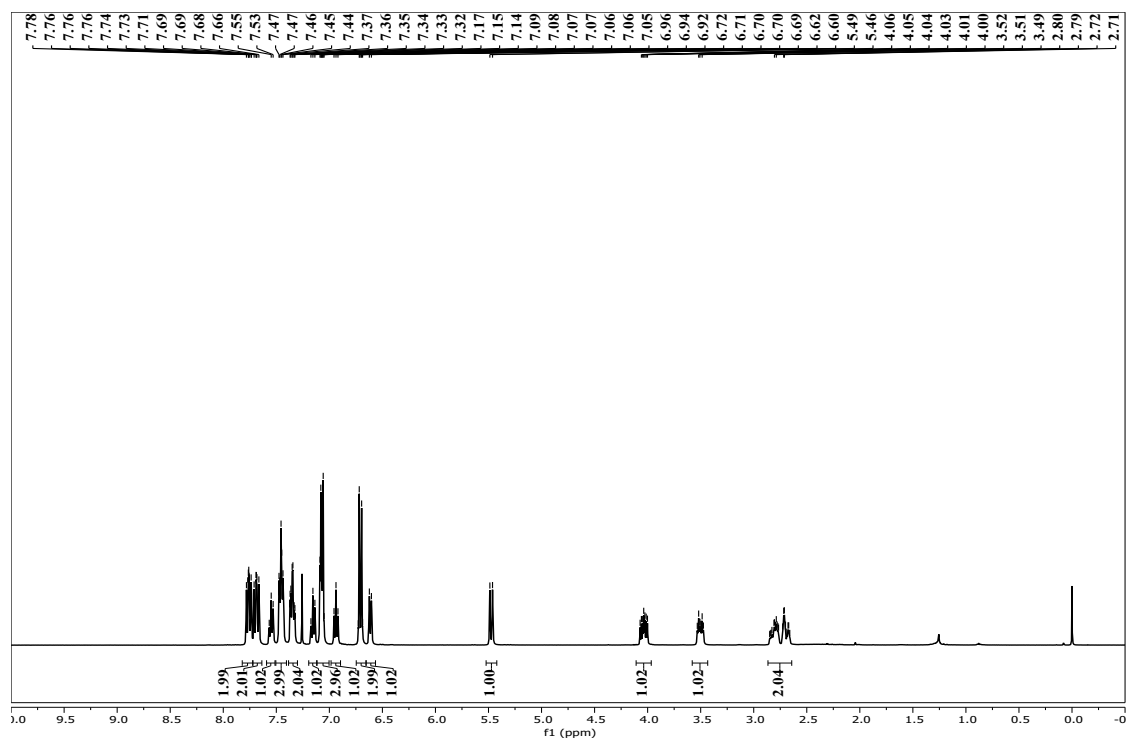


11a, ^{31}P NMR (162 MHz), CDCl_3

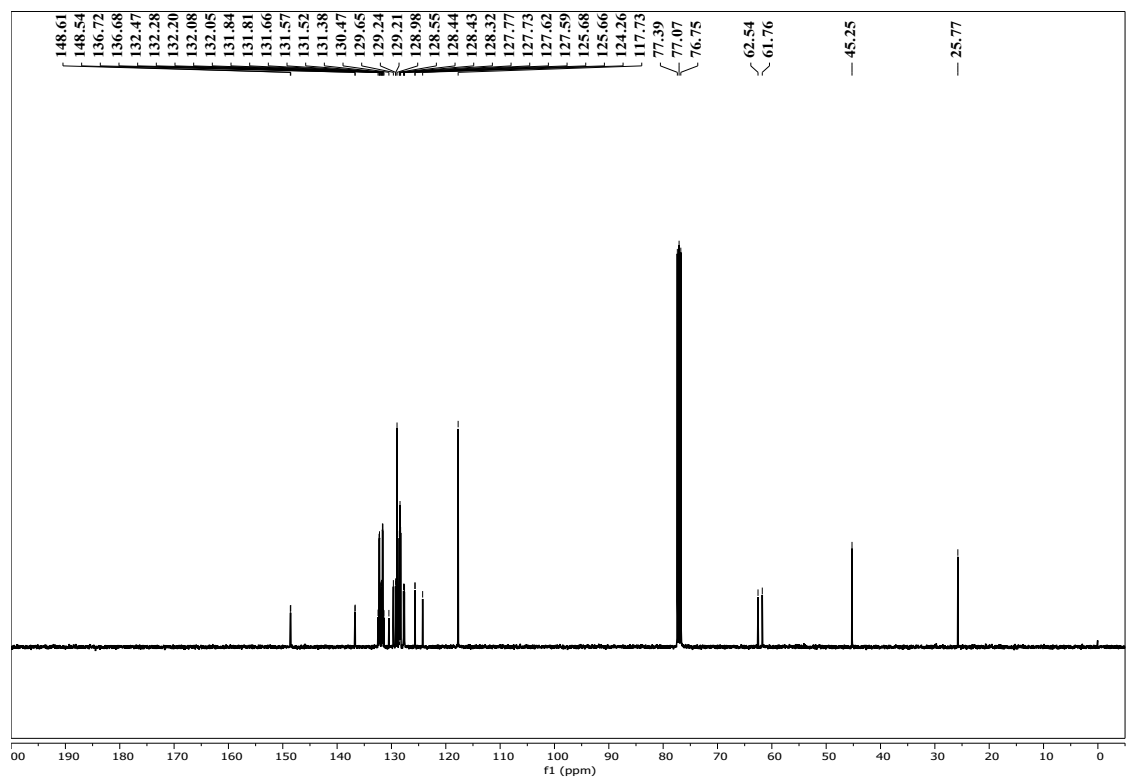




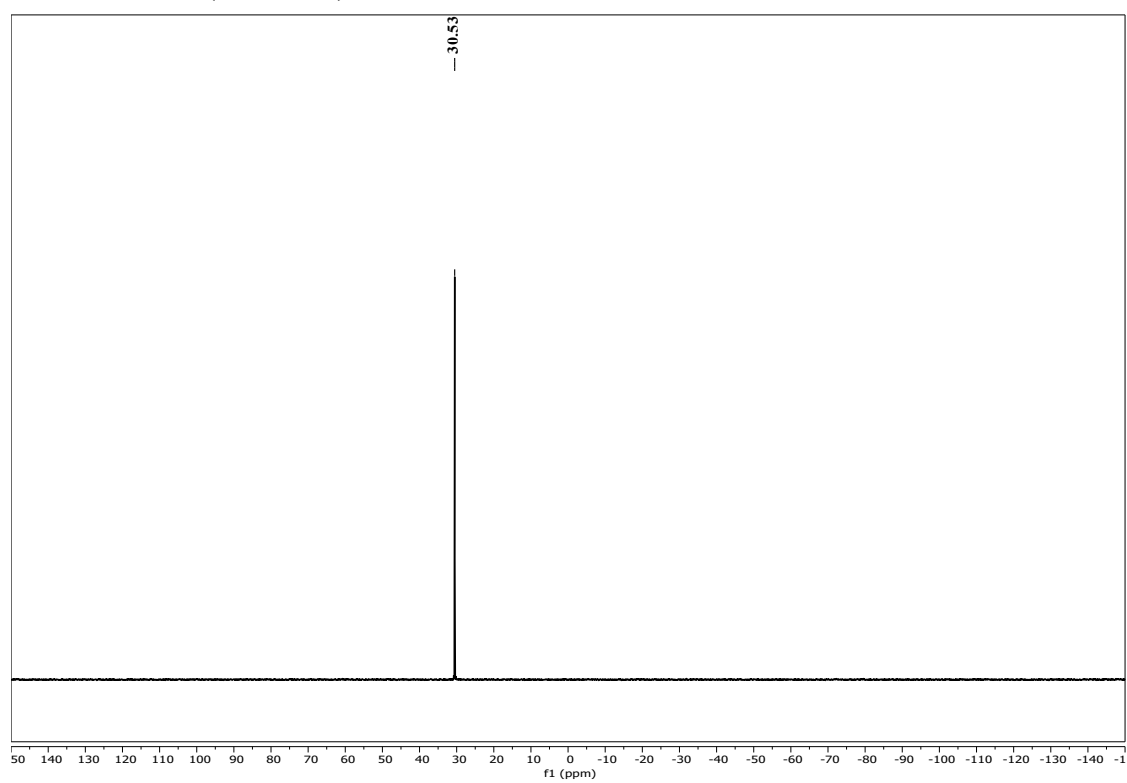
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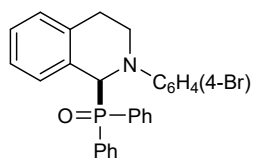


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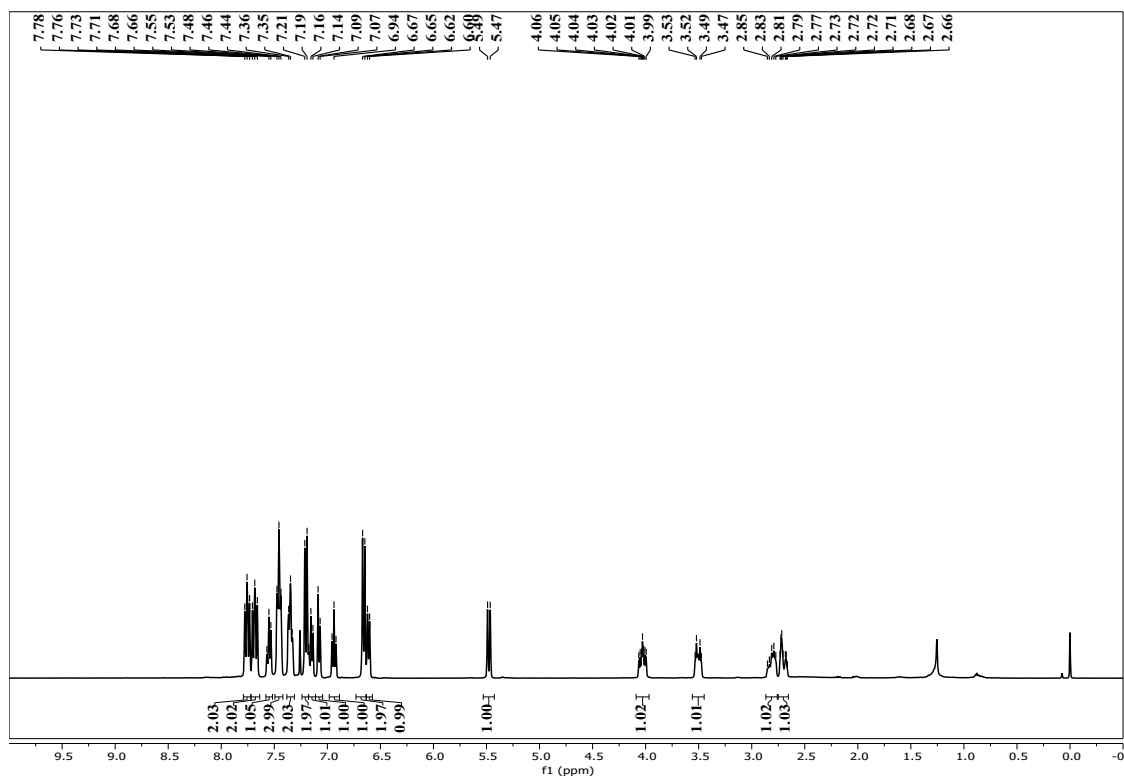


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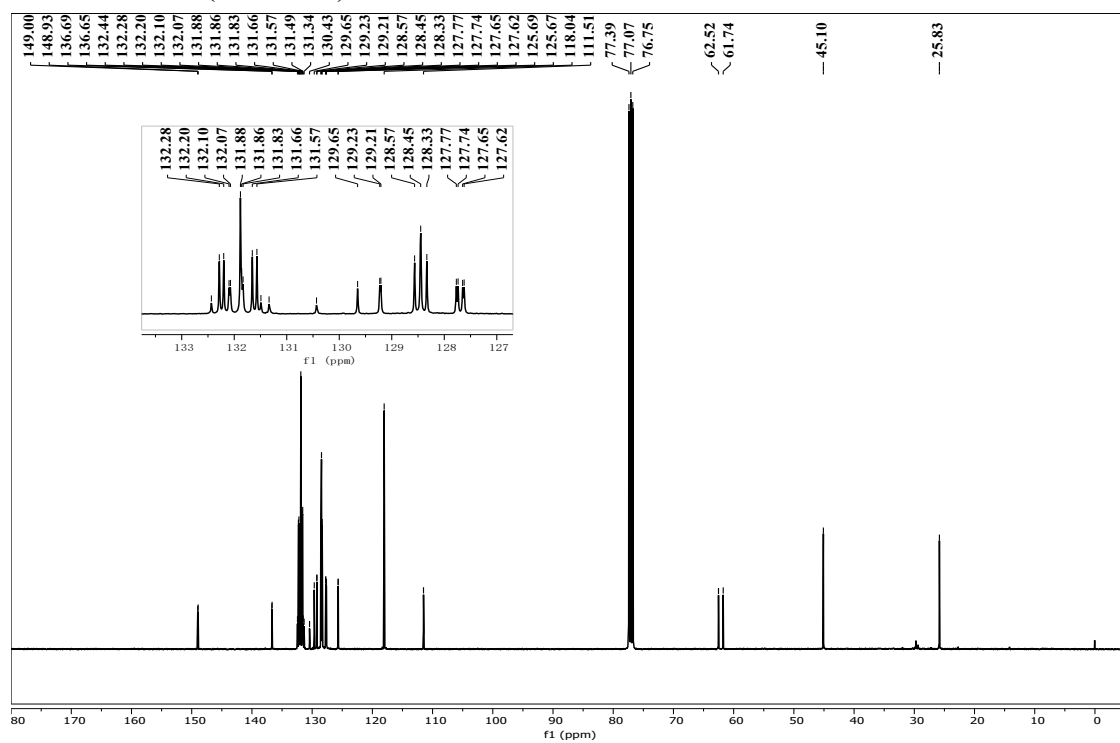




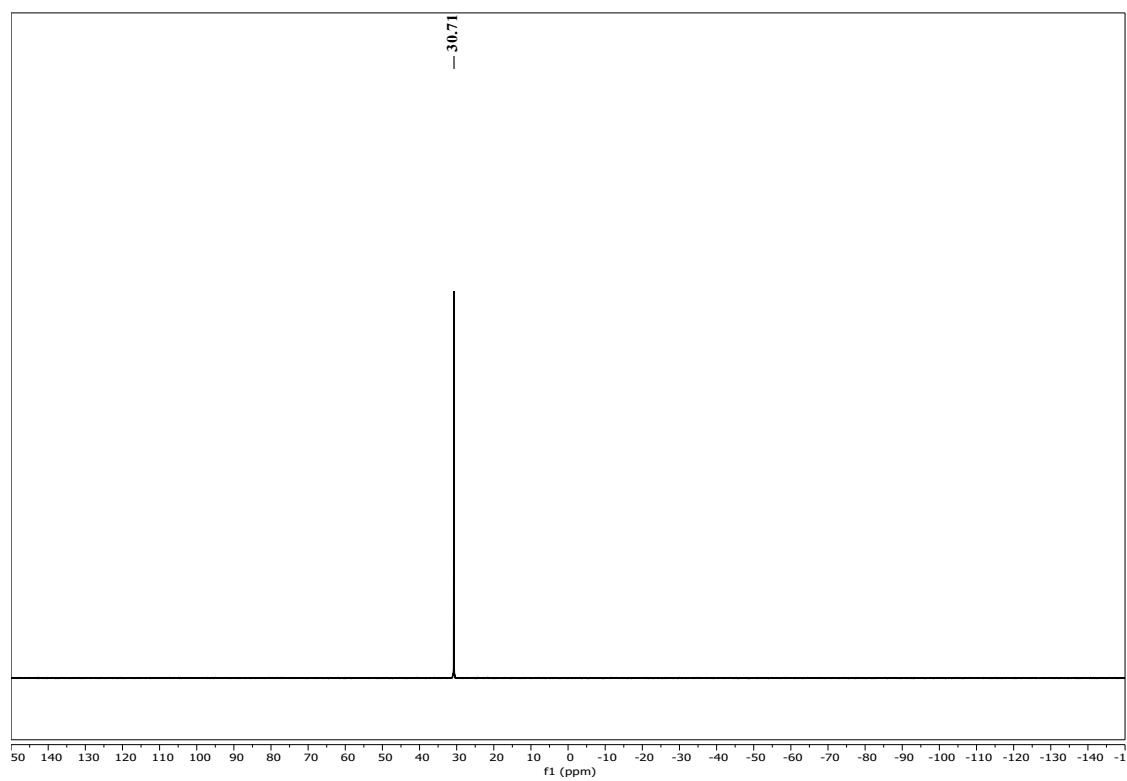
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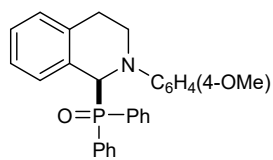


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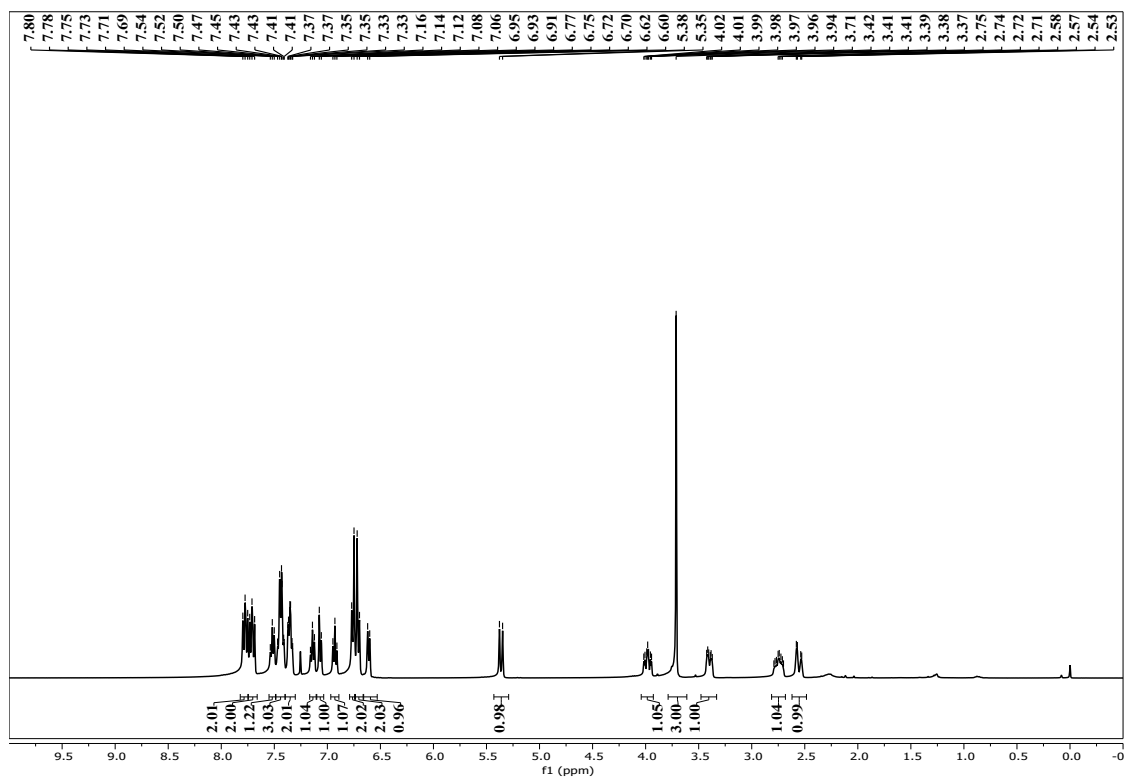


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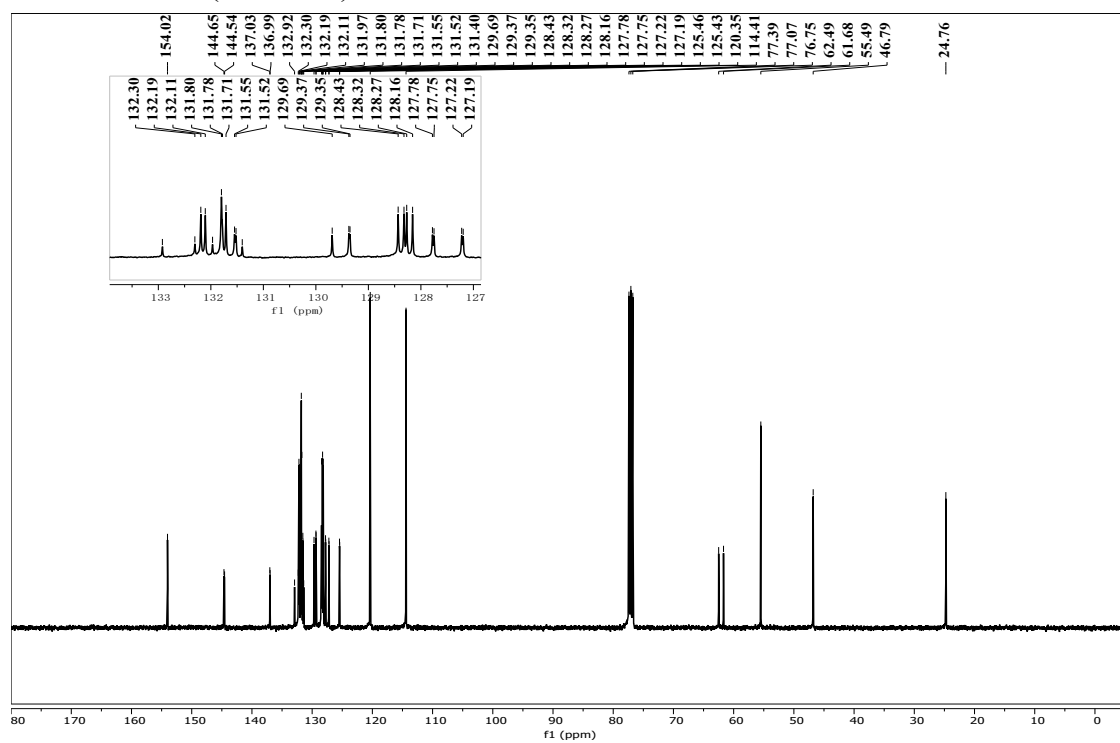




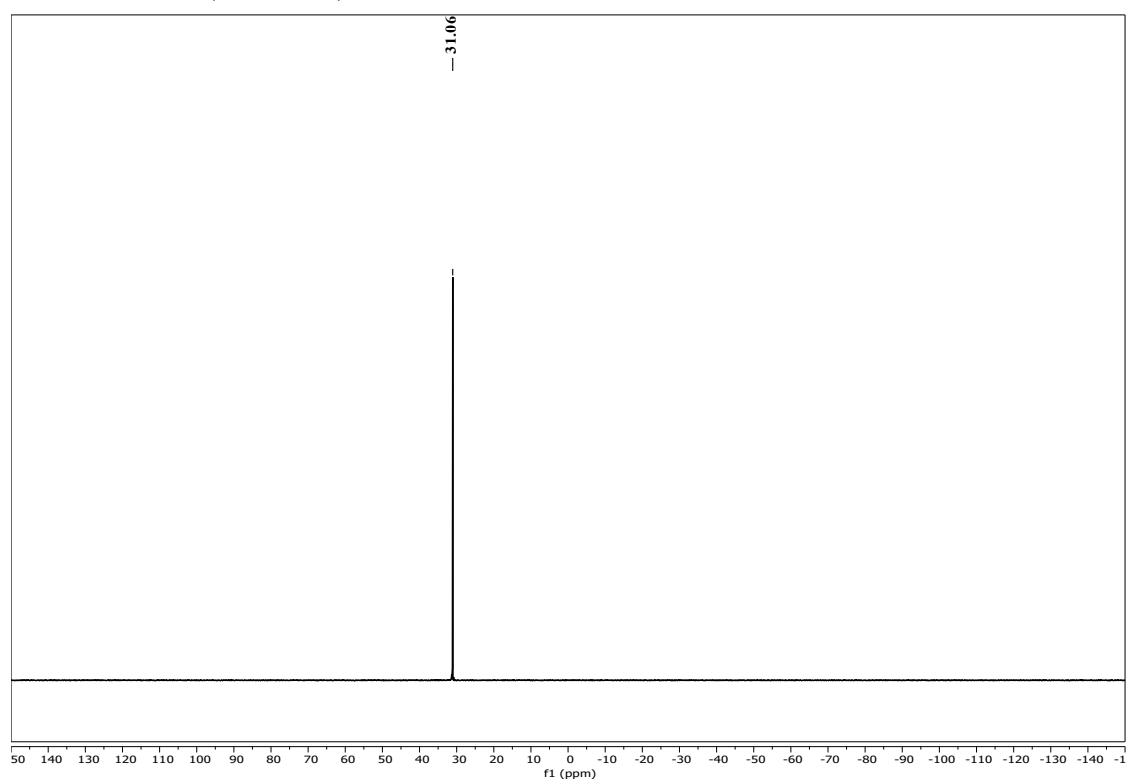
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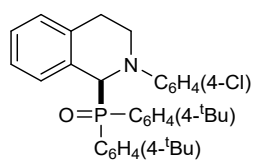


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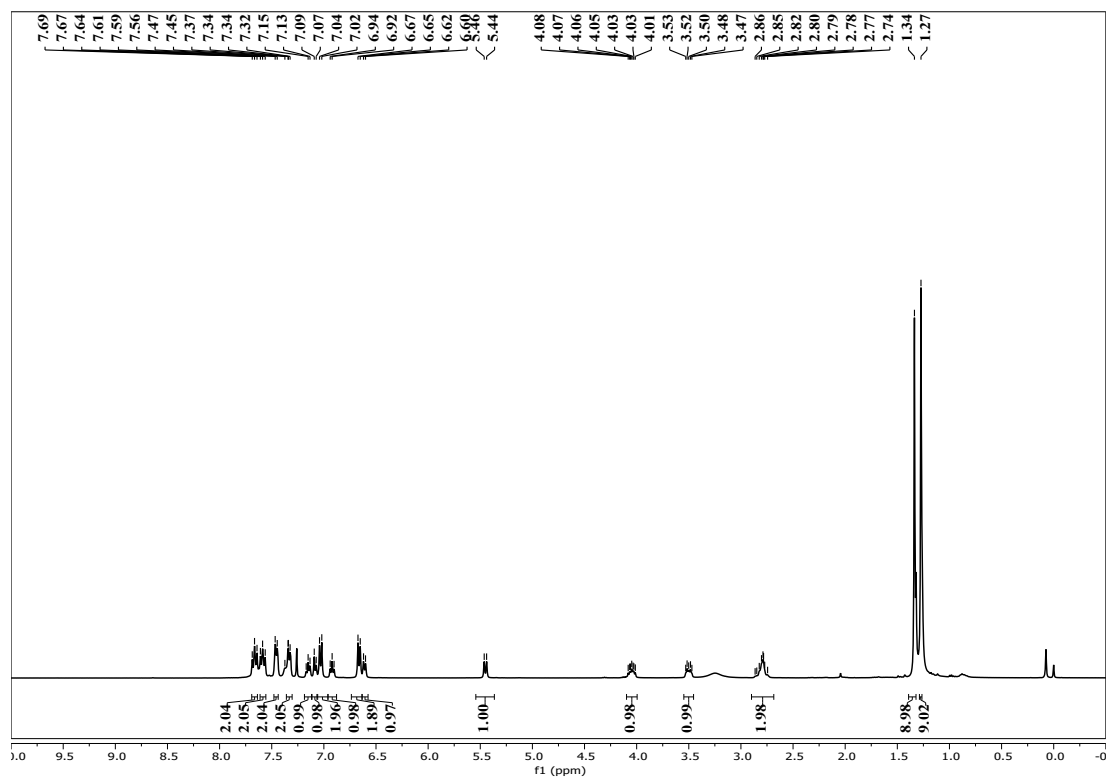


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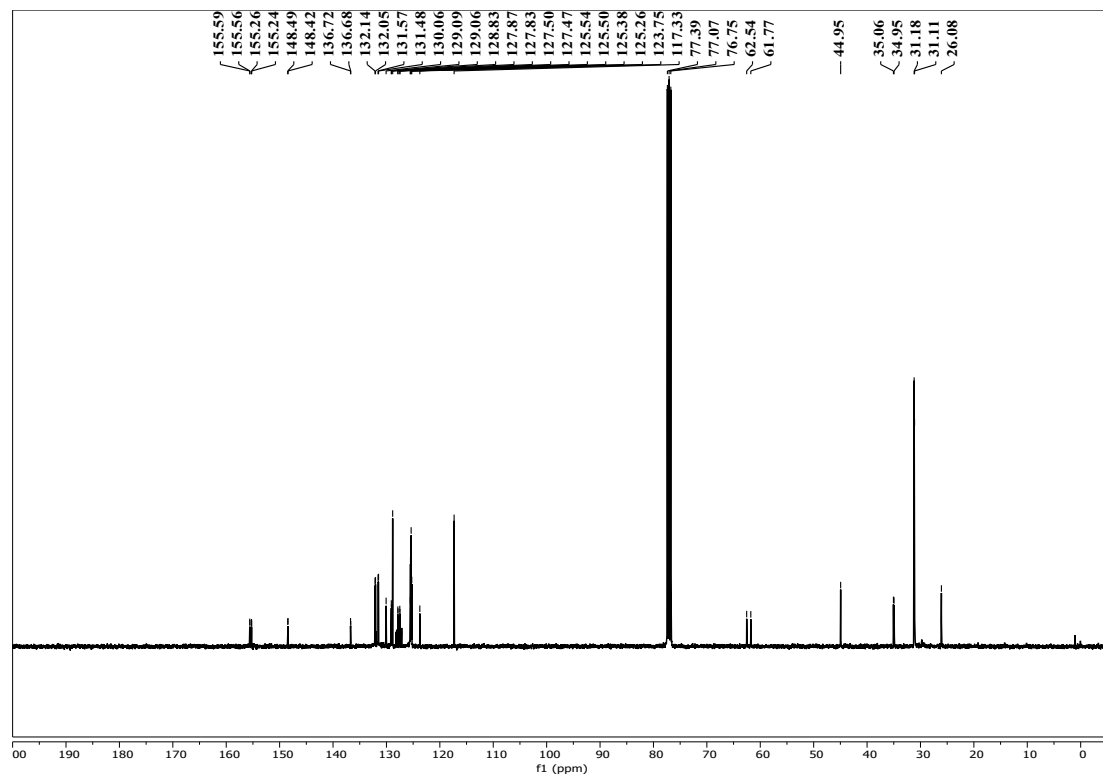




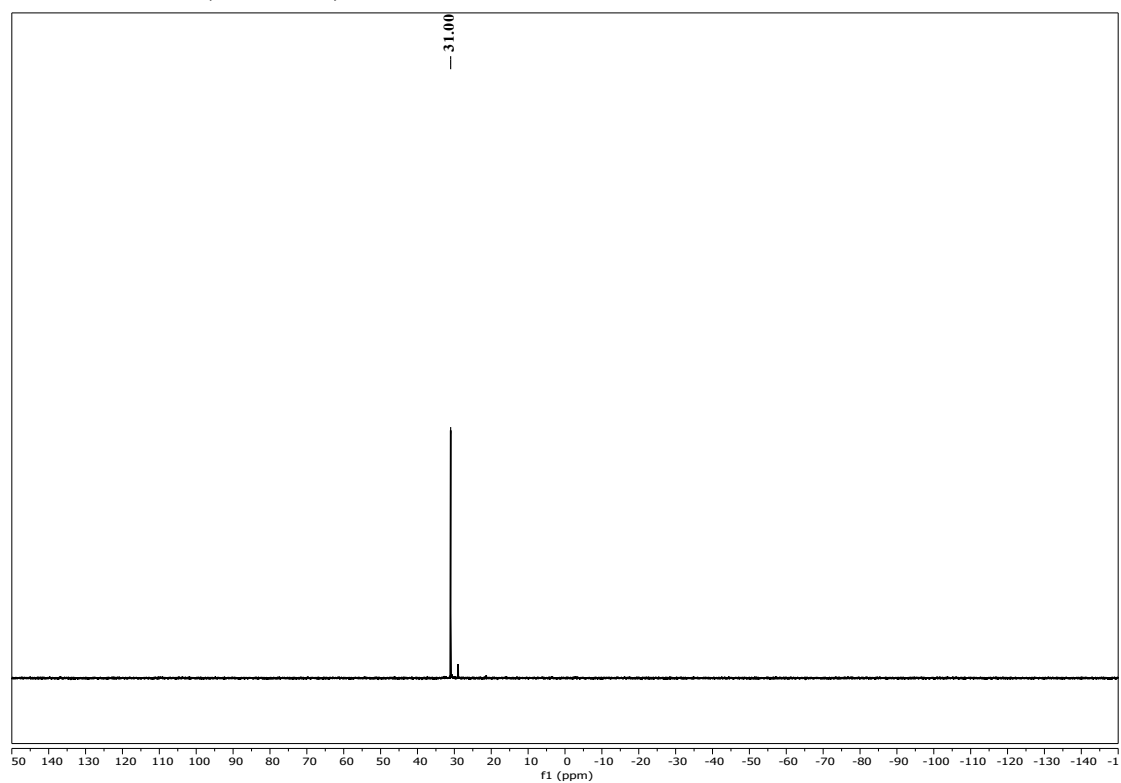
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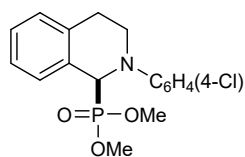


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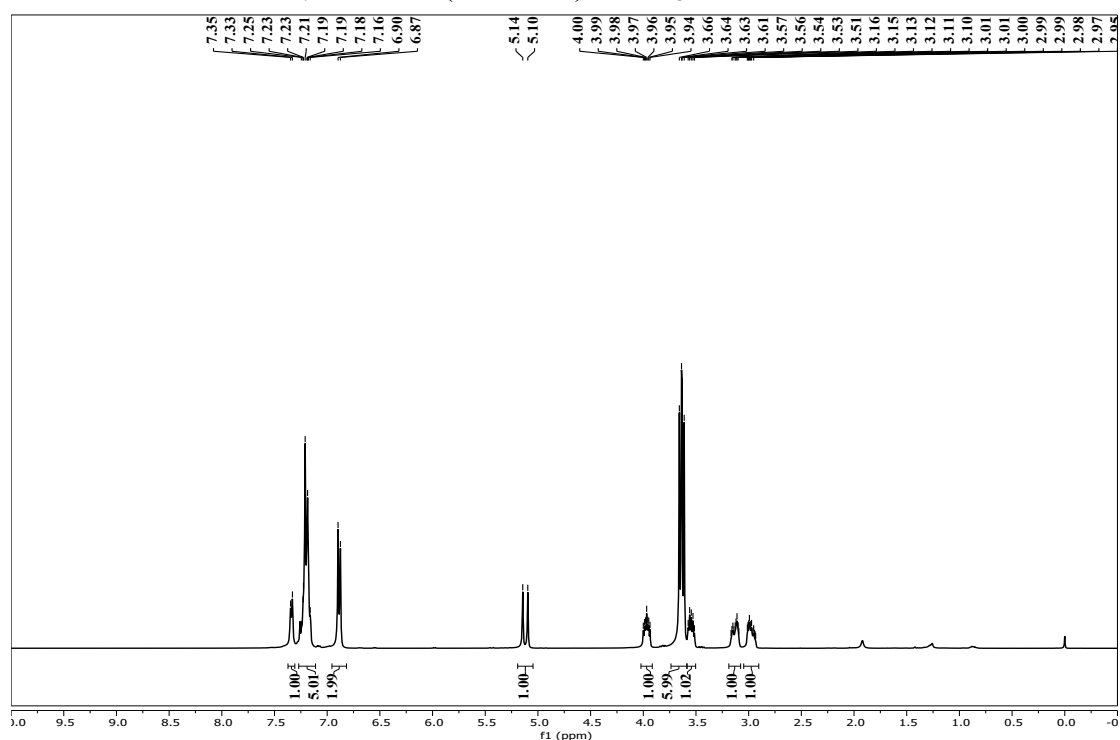


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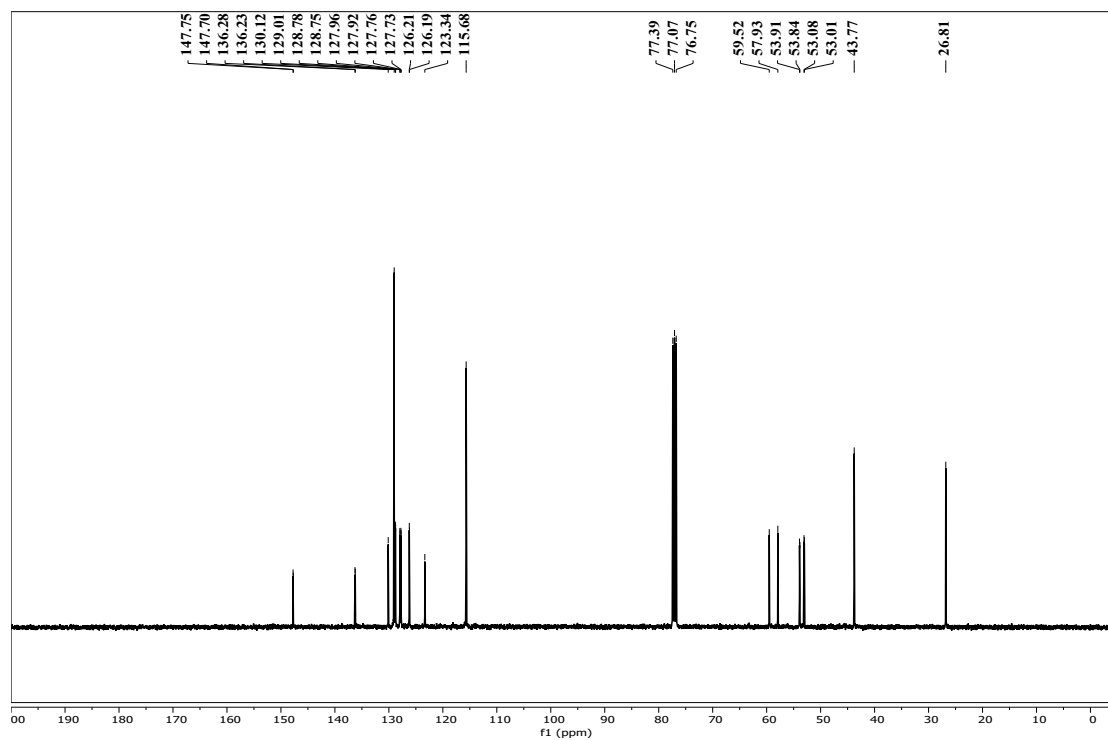




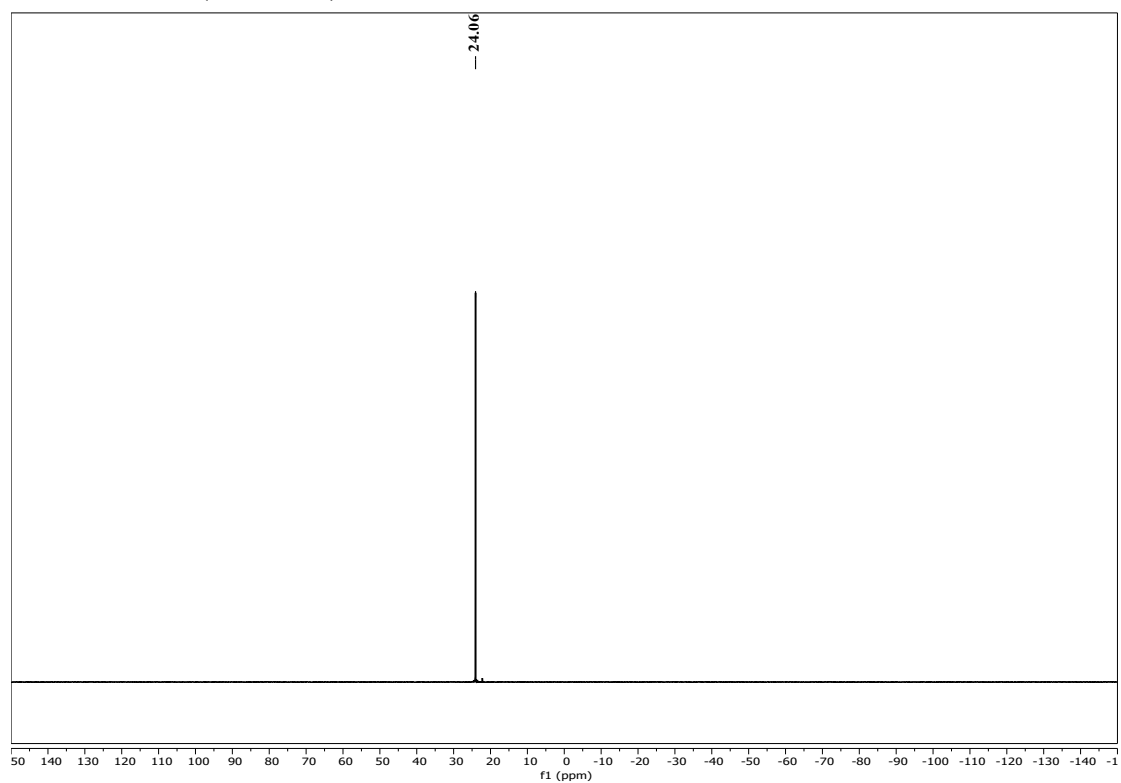
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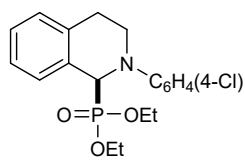


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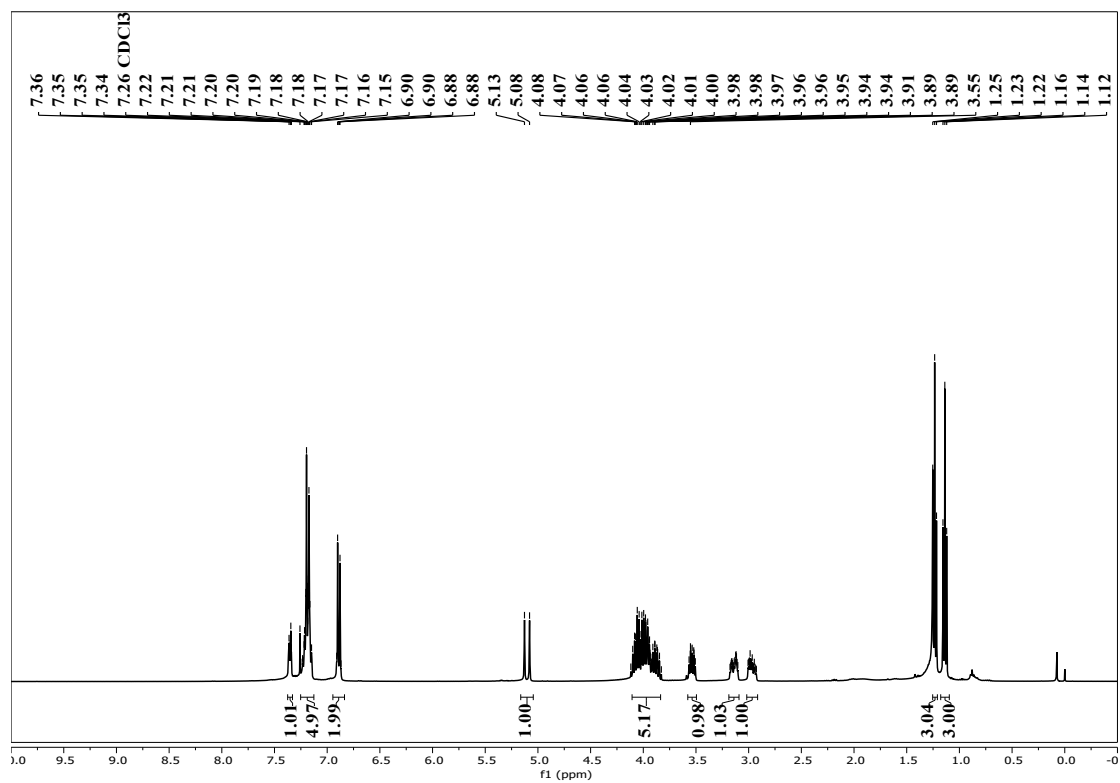


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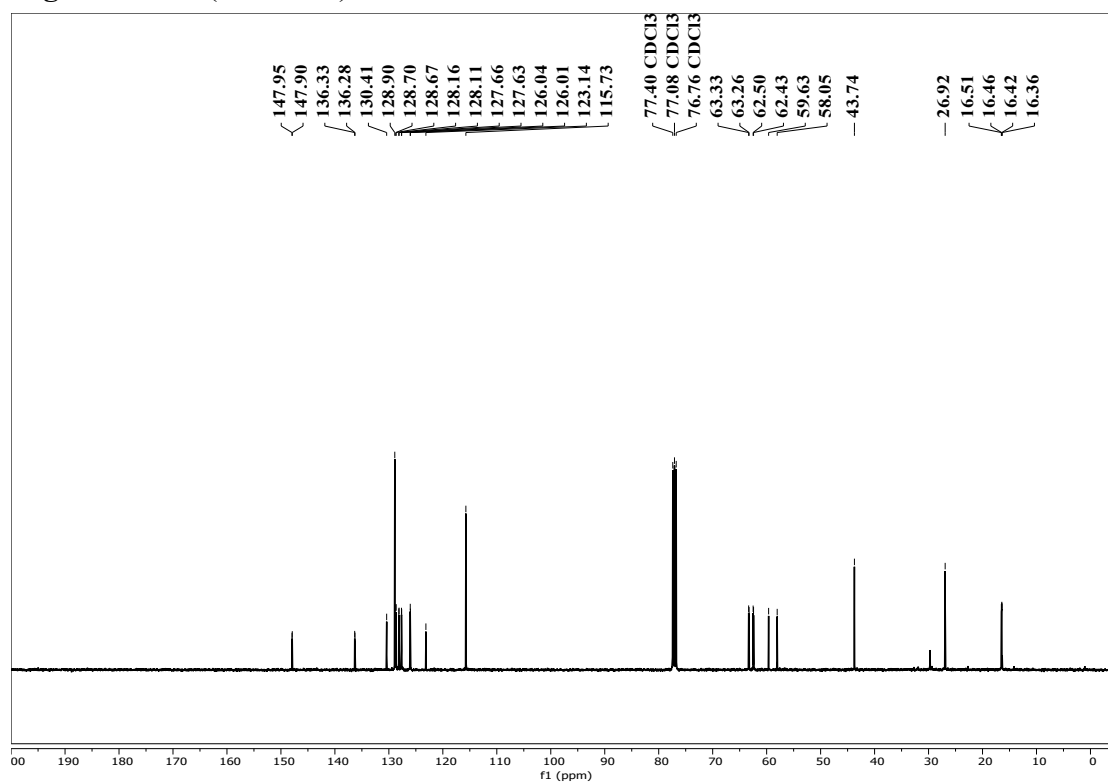




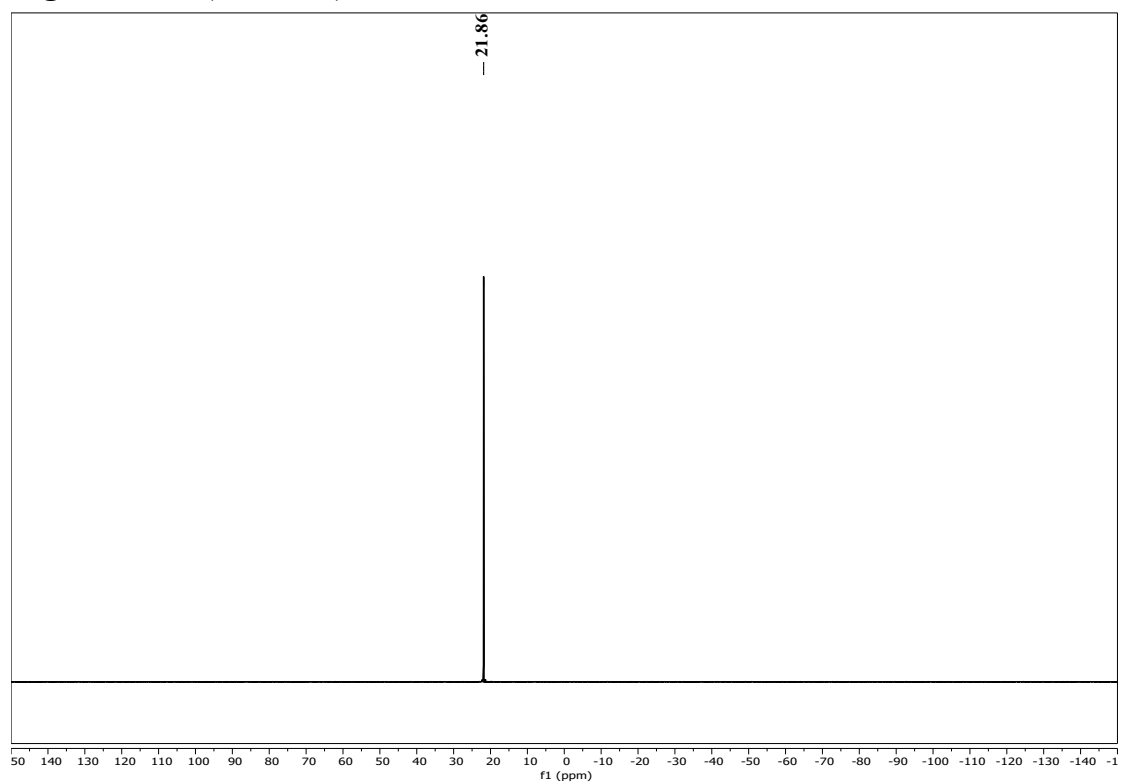
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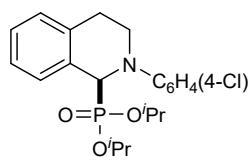


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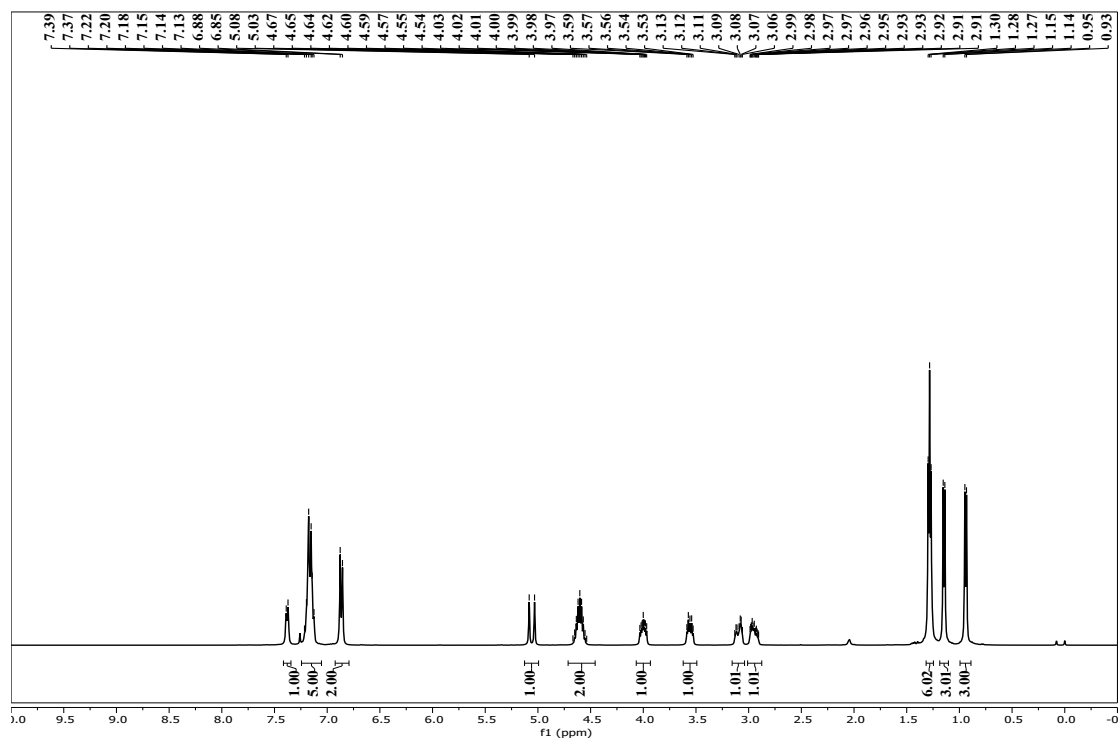


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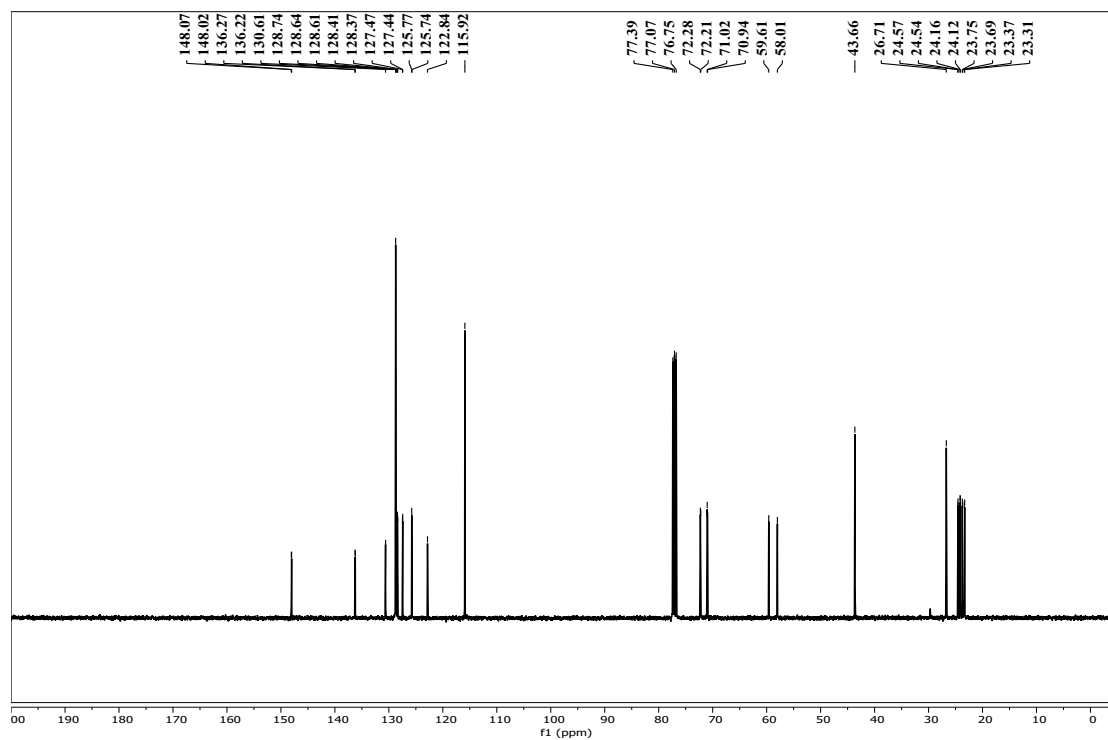




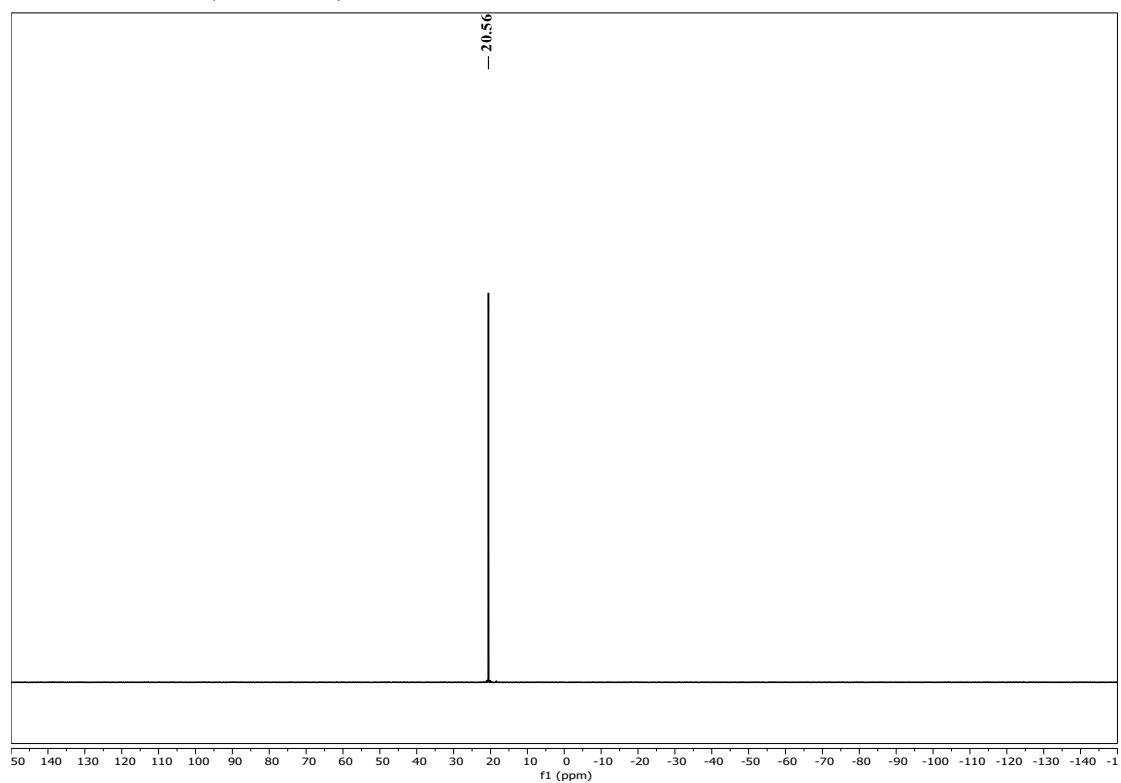
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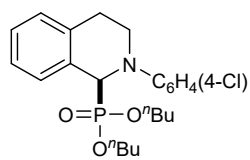


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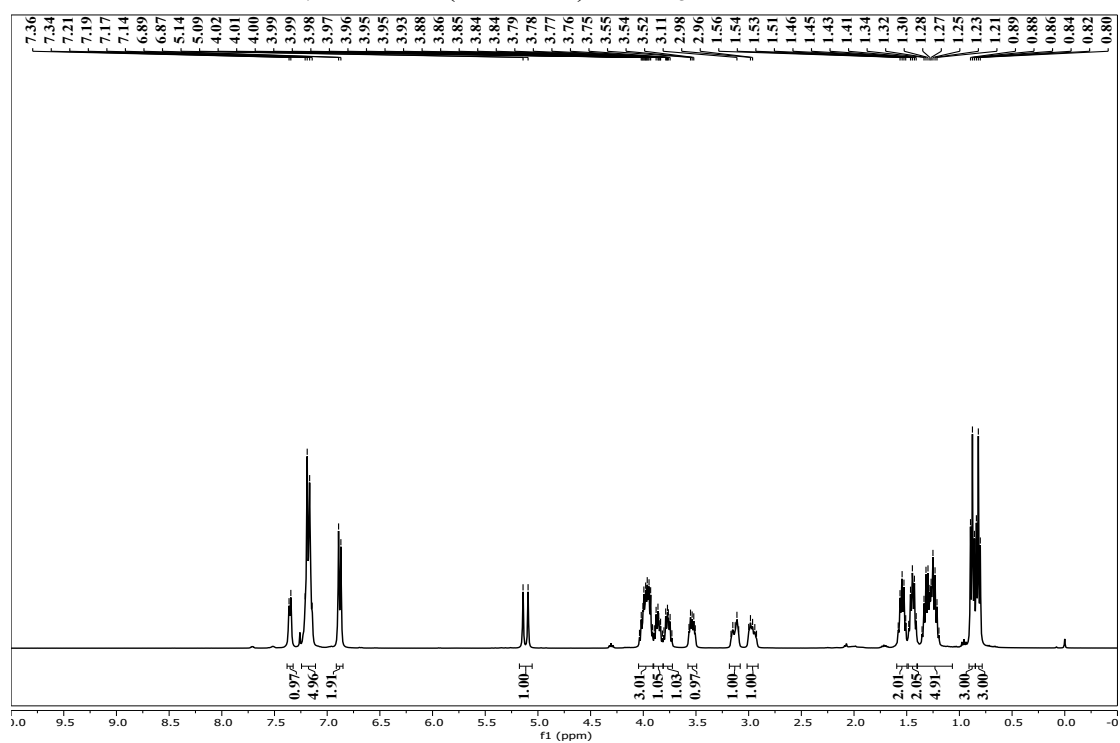


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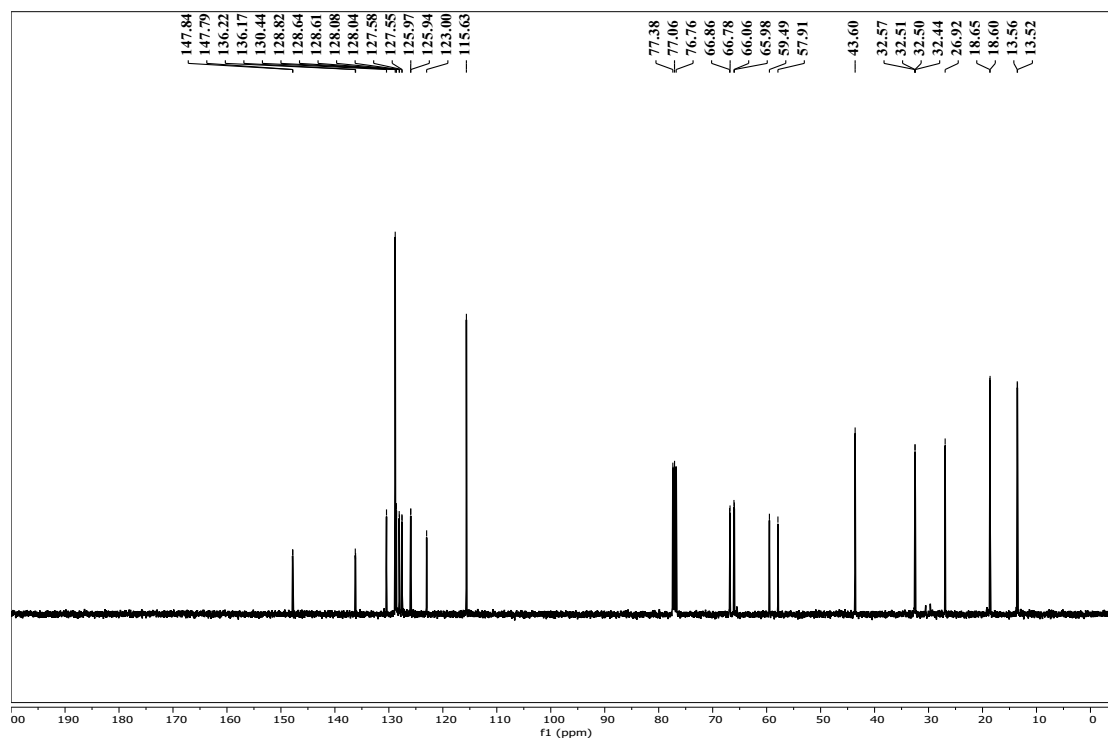




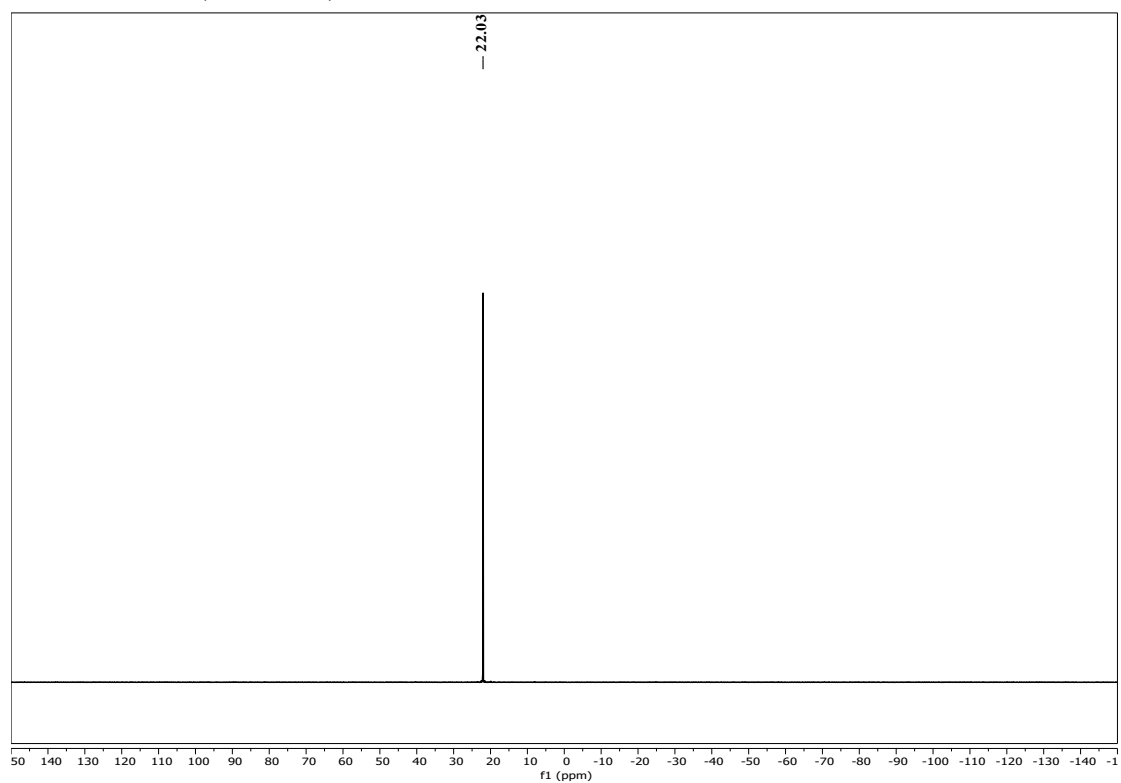
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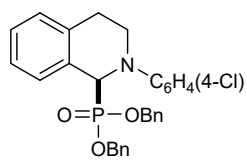


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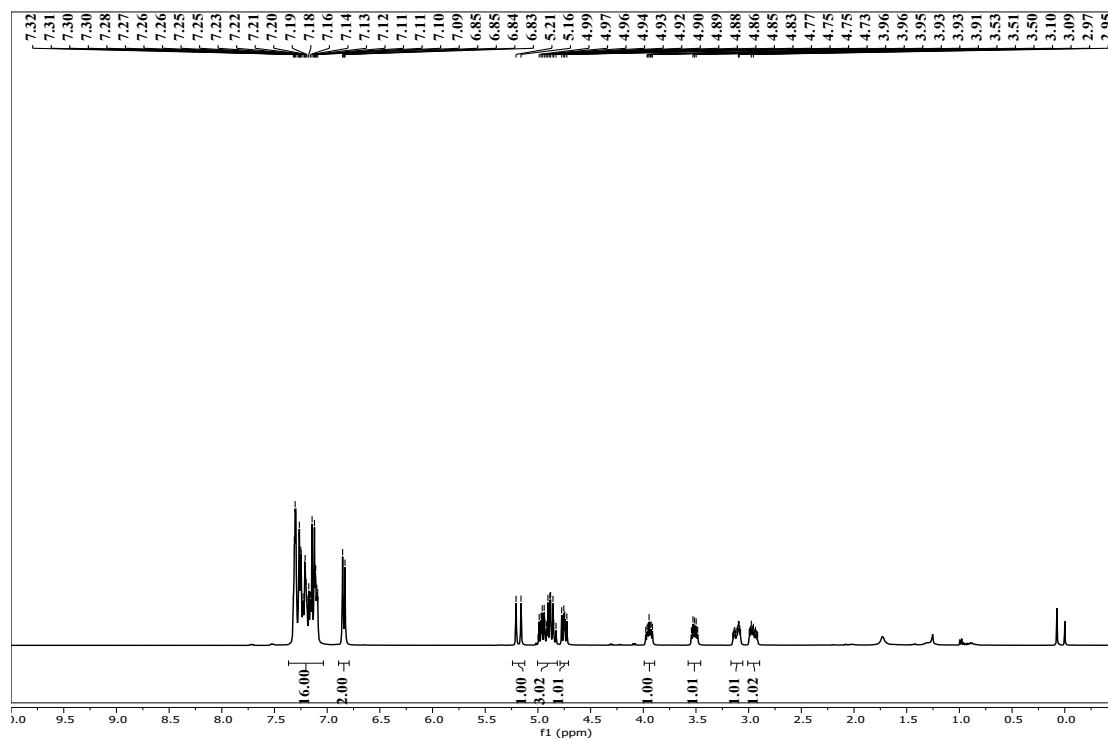


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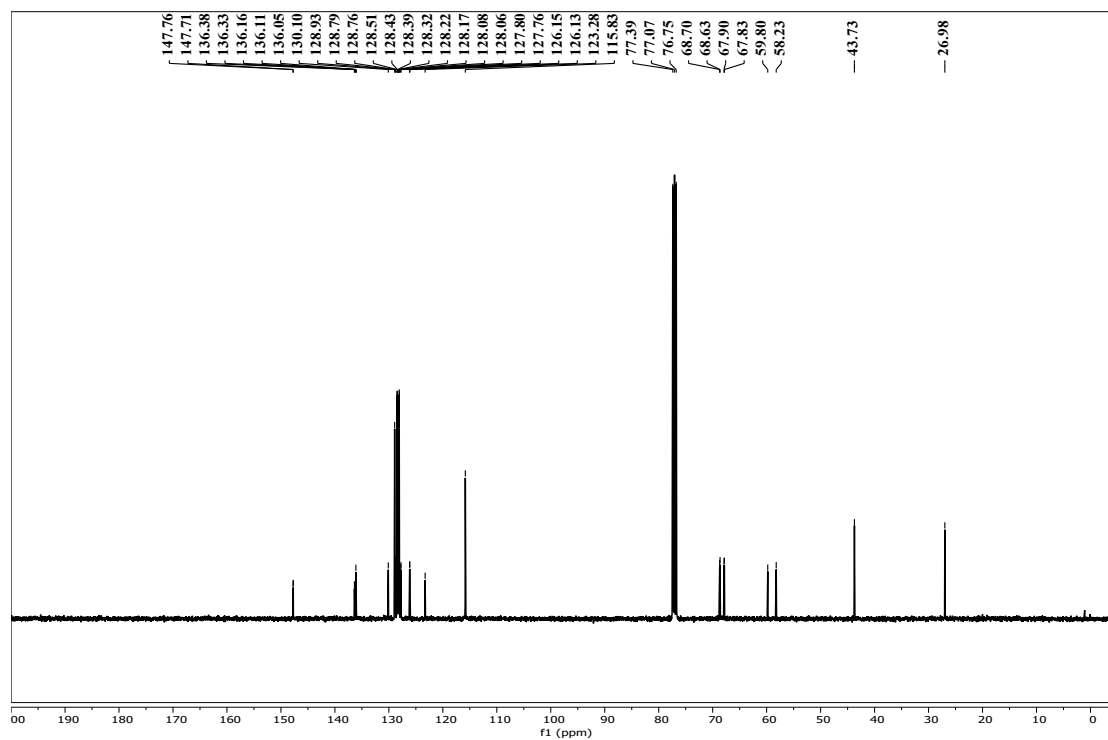




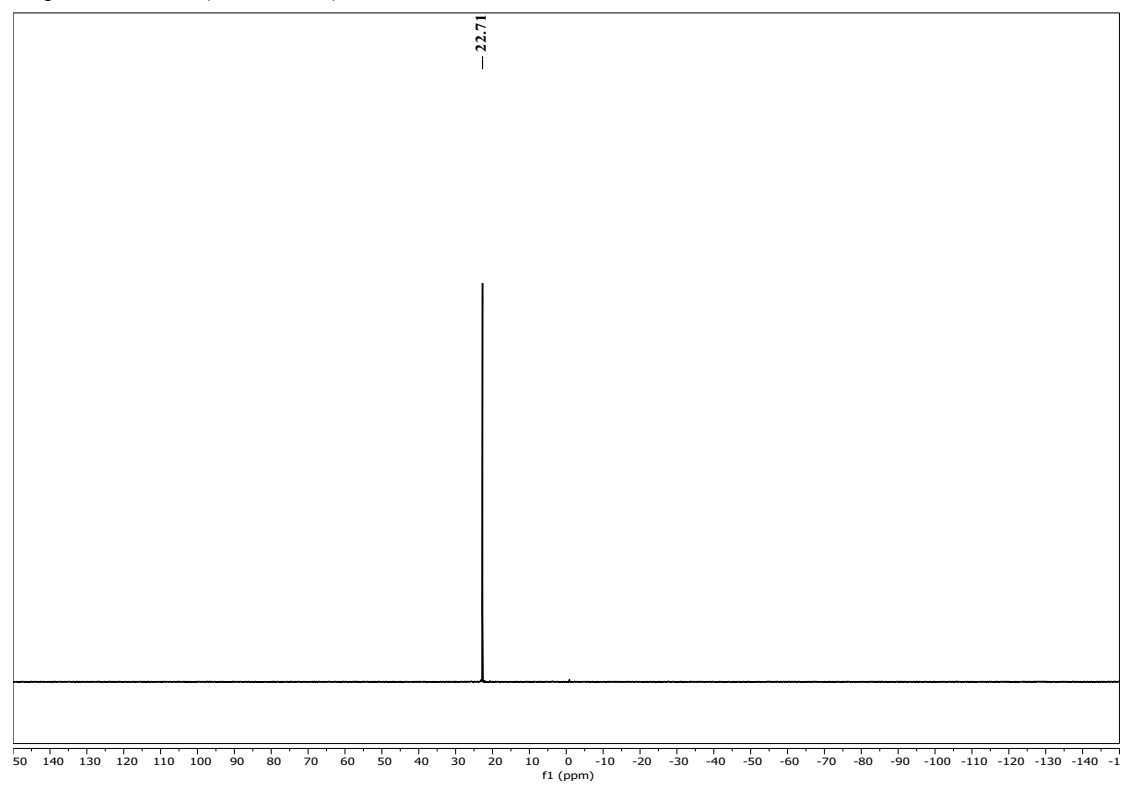
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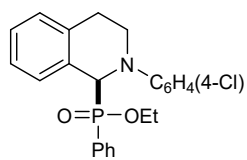


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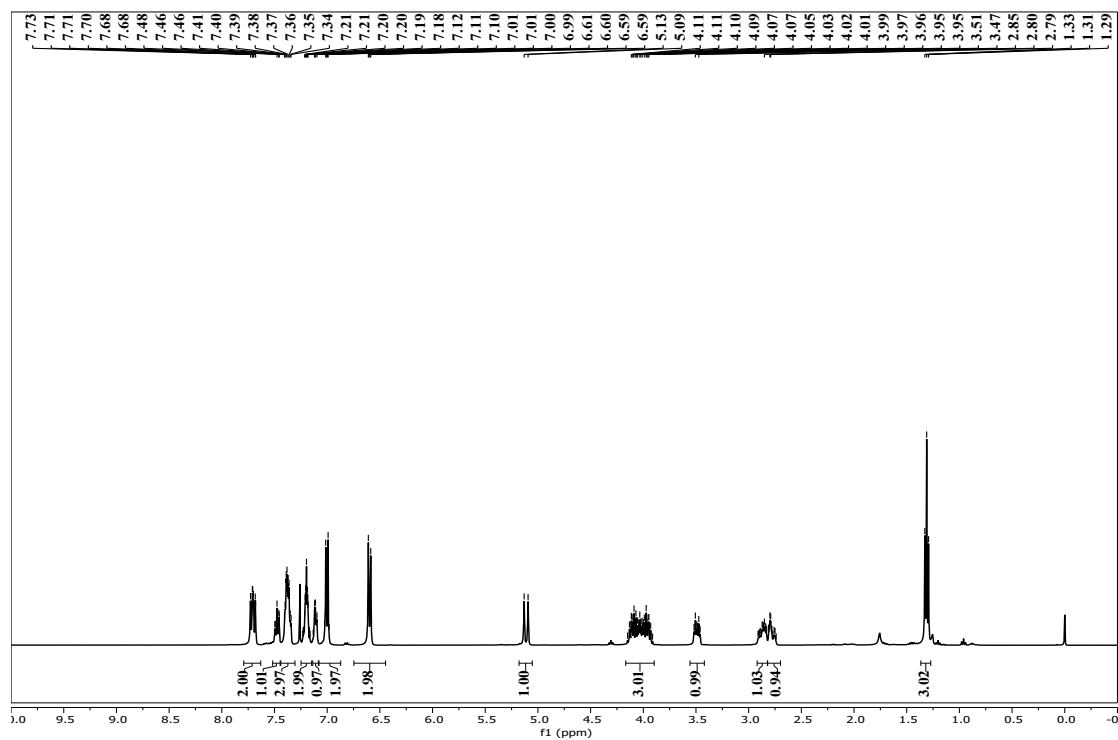


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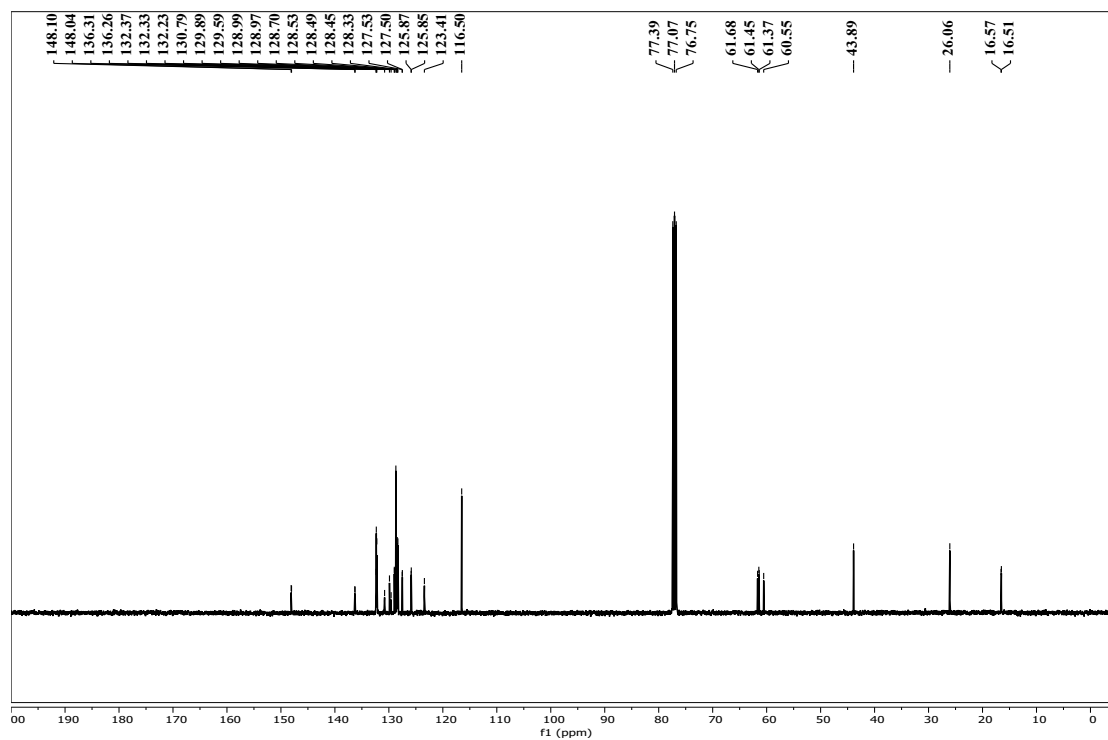




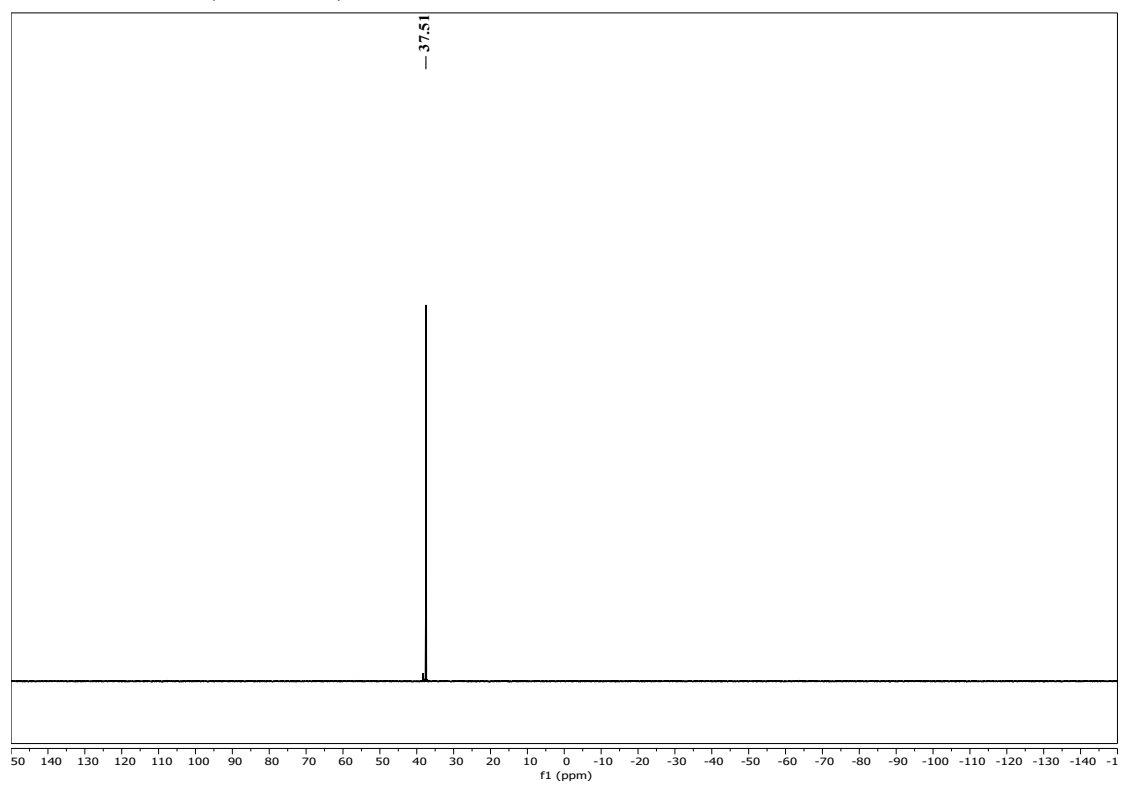
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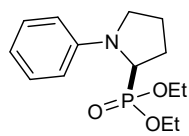


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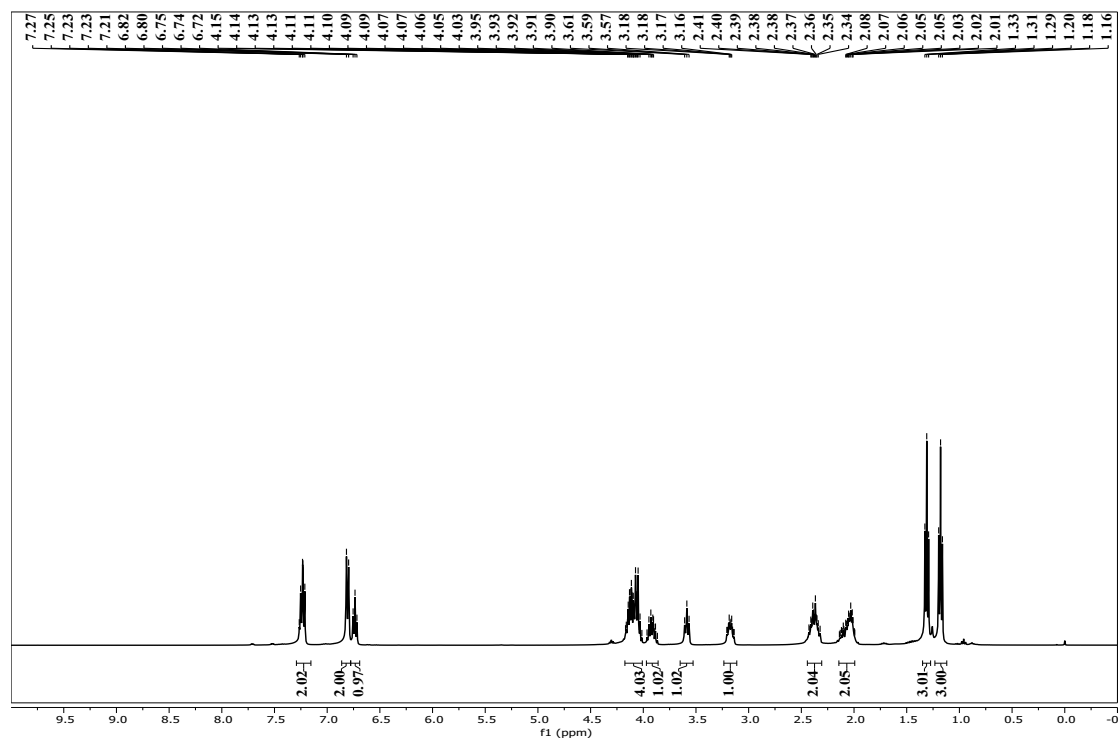


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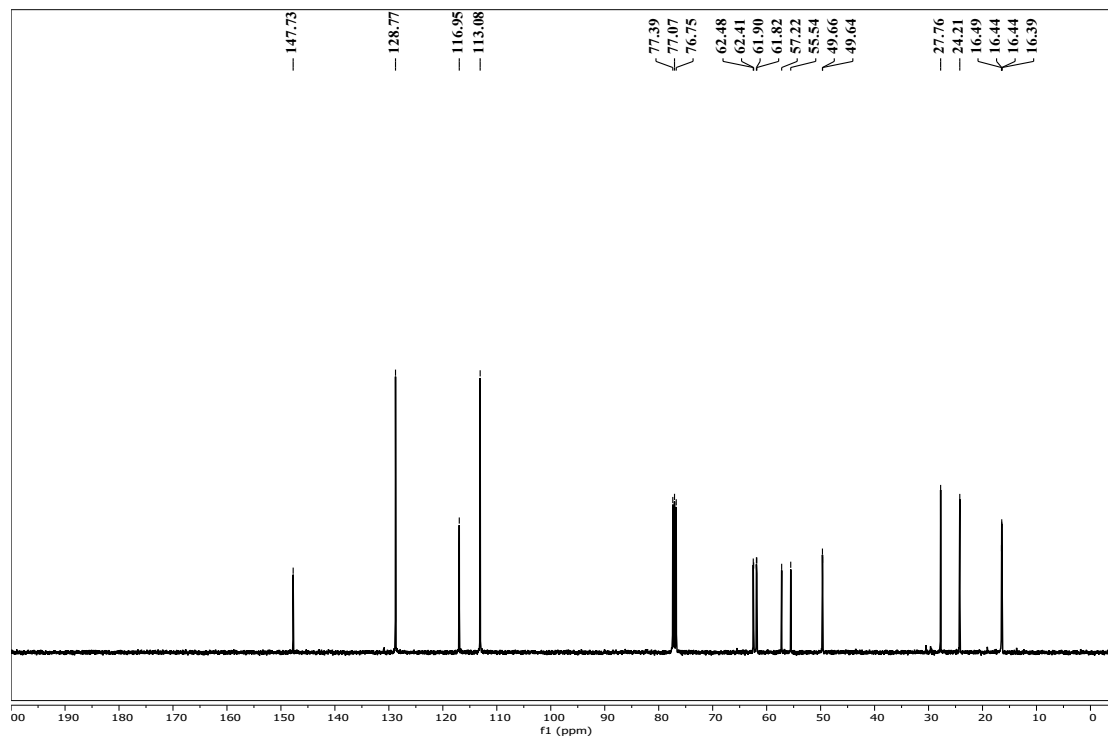




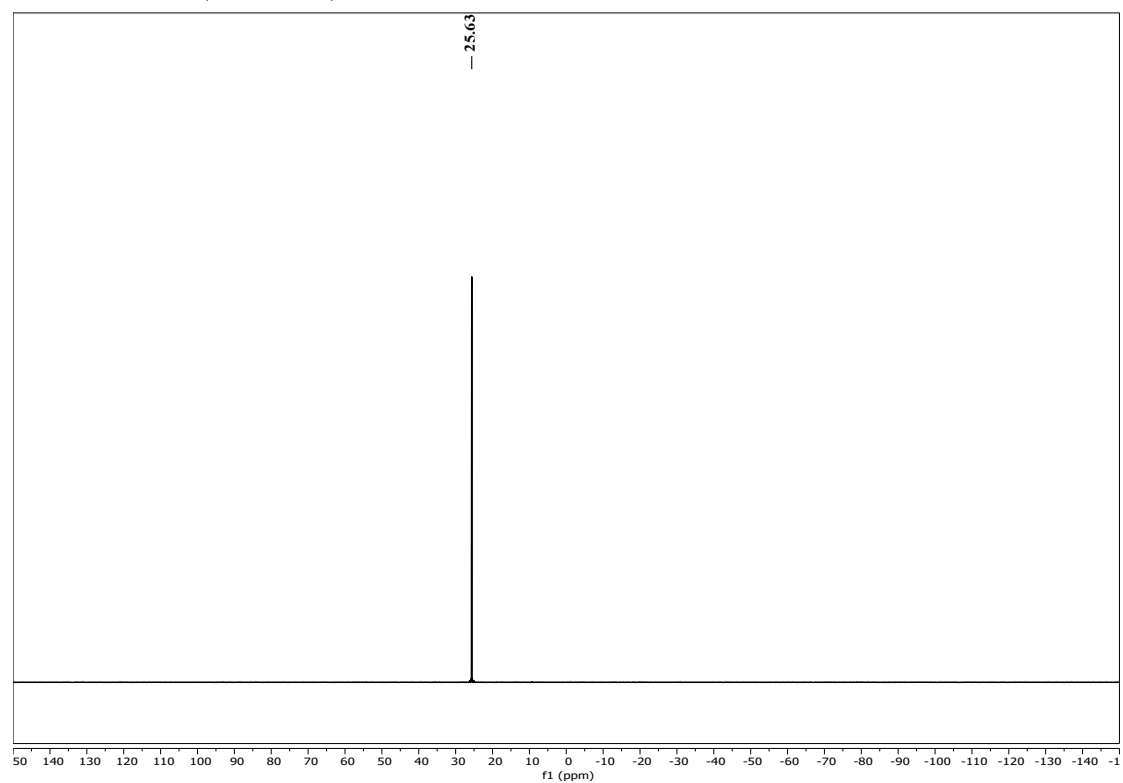
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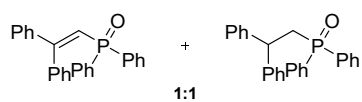


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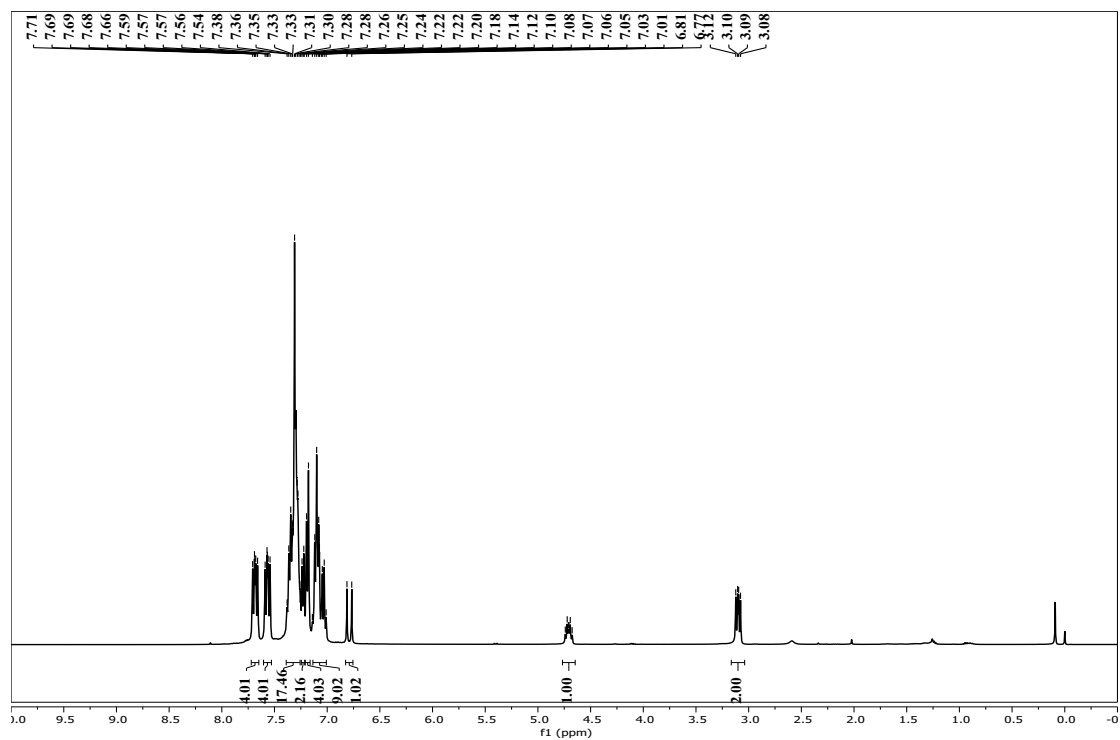


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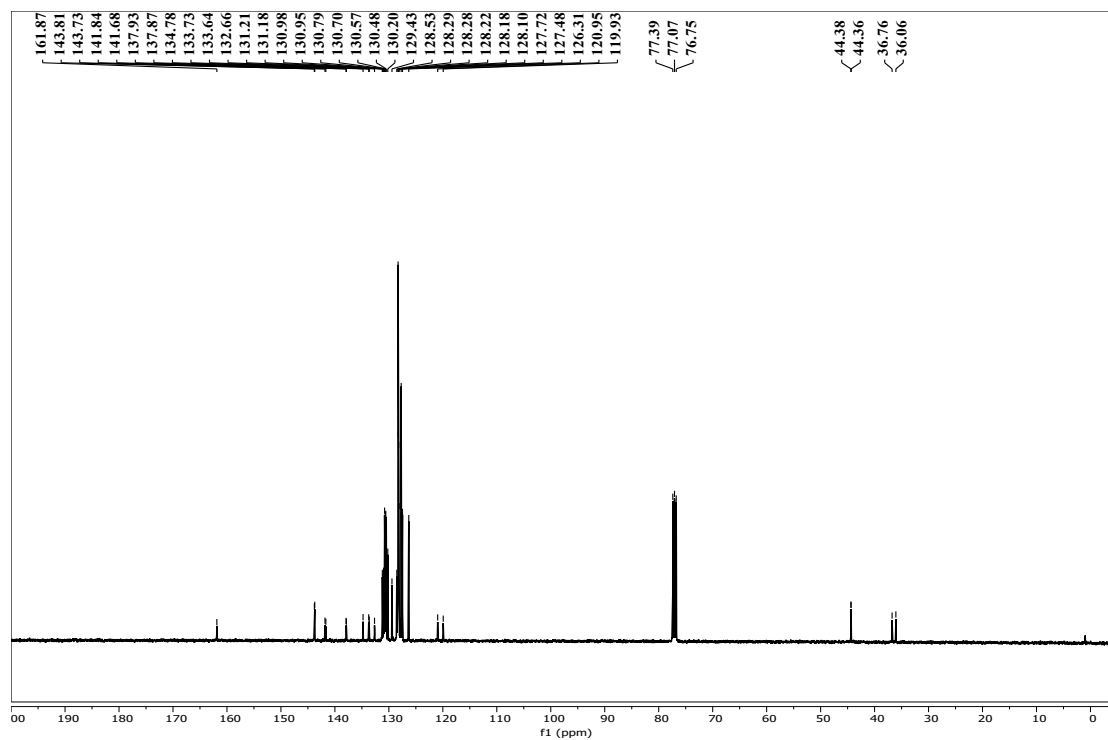




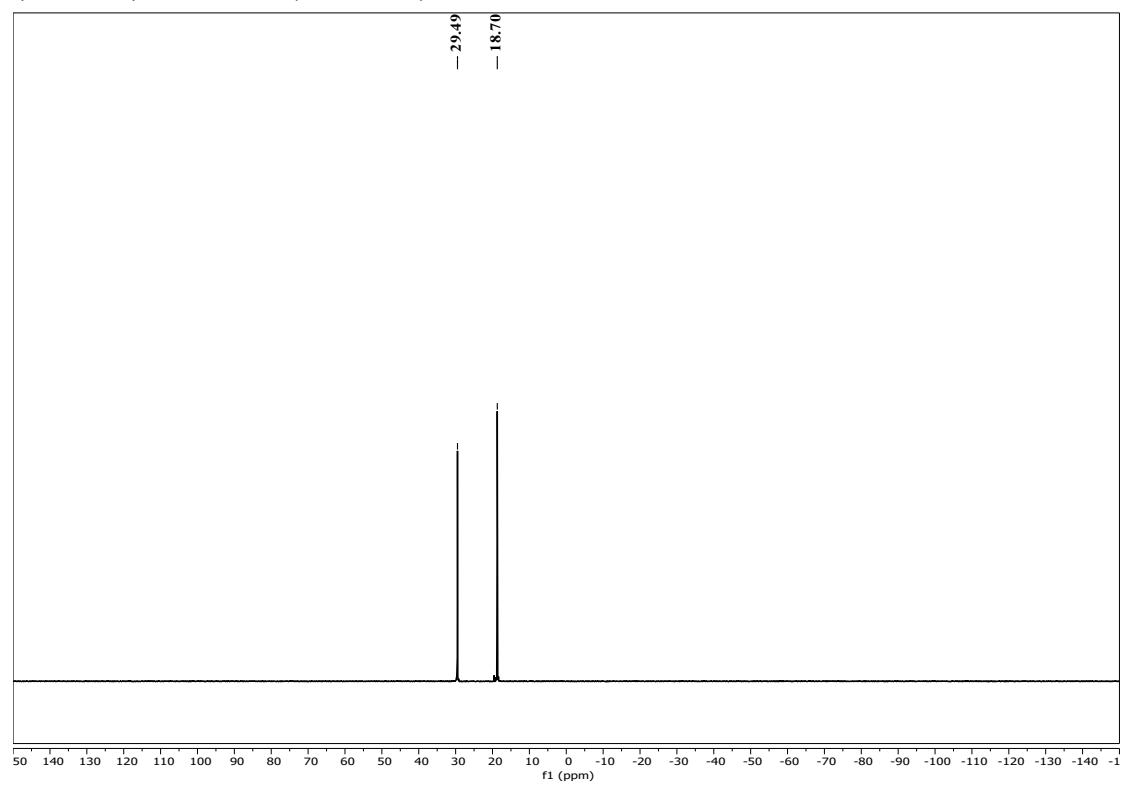
(13 + 13'), ¹H NMR (400 MHz), CDCl₃

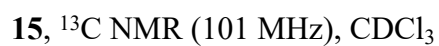
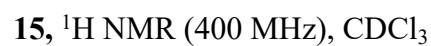


(13 + 13'), ¹³C NMR (101 MHz), CDCl₃



(13 + 13'), ^{31}P NMR (162 MHz), CDCl_3





15, ^{31}P NMR (162 MHz), CDCl_3

