

Synthesis of mixed Phosphorotrithiotes via coupling of Thiol with Bis(diisopropylamino)chlorophosphine and Sulphenyl Chloride

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Supplementary Data

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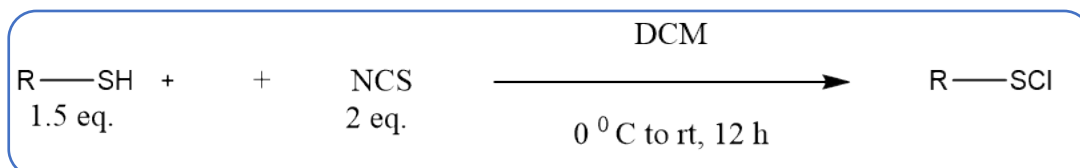
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1. General information:

All chemicals were obtained from Sigma-Aldrich, Toyoko Chemical Industry, S. D. Fine and the CHCl₃ Solvent used in the reaction is from Qualigen having 0.05% water content, the progress of the reactions was monitored by thin-layer chromatography (TLC) on pre-coated silica-gel plates using Merck Silica Gel 60 F254, Cat. No. 1.05554.0007 and visualized by short-wave ultraviolet light as well as by treatment with KMnO₄, Dragendroff reagent. Column chromatography was performed by hand using silica-gel (100–200 mesh, Silicycle). ¹H, ¹³C, ³¹P NMR spectra were recorded on Bruker-Advance DPX FT-NMR 500 and 400 MHz instruments. Chemical data for protons are reported in parts per million (ppm) downfield from tetramethylsilane and are referenced to the residual proton in the NMR solvent (CDCl₃: 7.26 ppm and CD₄O 4.78, 3.30 ppm). Carbon nuclear magnetic resonance spectra (¹³C NMR solvent CDCl₃: 77.0 ppm and CD₄O 49 ppm) were recorded at 125 MHz or 100 MHz: chemical data for carbons are reported in parts per million (ppm, δ scale) down field from tetramethylsilane and are referenced to the carbon resonance of the solvent. ESI-MS and HRMS spectra were recorded on Agilent 1100 LC-Q-TOF and HRMS-6540-UHD machines respectively.

2. General procedure for synthesis of Starting Materials.

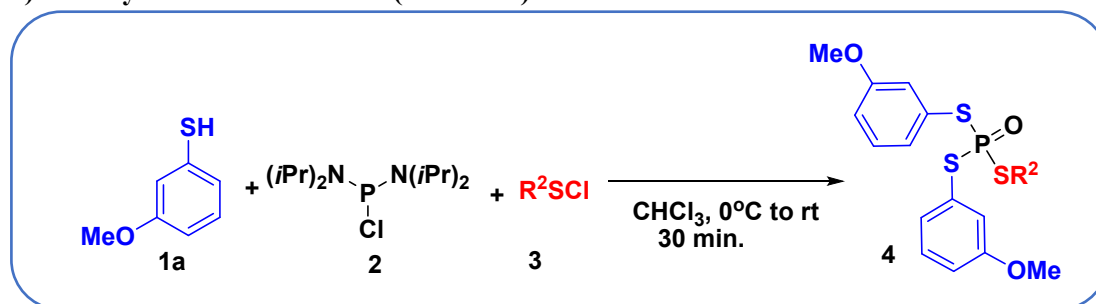
2.1 For the synthesis of Sulphenyl Chloride.



Sulfonyl Chlorides were prepared according to a reported literature. N-chlorosuccinimide (2mmol) was placed in oven dried 100 mL round bottom flask and dissolved in Dichloromethane (50 mL). Corresponding thiol (1.5 mmol) was added slowly at 0 °C and the reaction mixture was stirred at room temperature for 12 hours. After completion of the reaction as monitored by TLC, the reaction mixture was extracted with ethyl acetate (25 ml x 3) and washed with water (50 ml x 2). The organic layer was dried over anhydrous Na₂SO₄, concentrated via rotatory evaporation, and purified by column chromatography on silica gel using Hexane and Ethyl acetate as eluent.

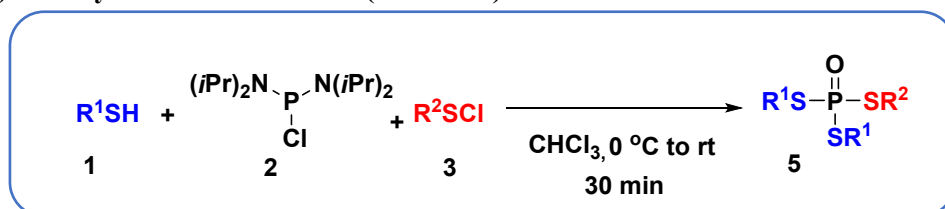
3. General procedure for synthesis of products Materials.

a) Synthesis of Product 4 (Scheme 1)



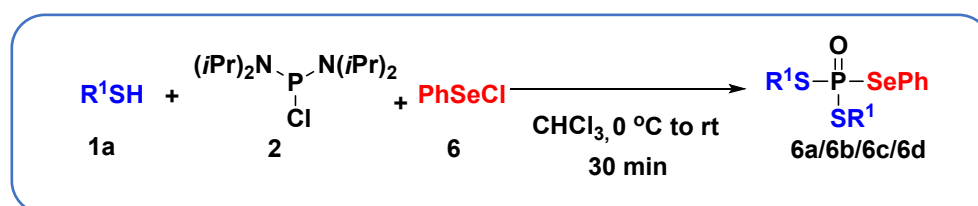
To a 25 ml of oven dried round bottom flask dissolve 2 mmol of reagent 2 in 6 ml CHCl₃. Add reagent 1 (3 mmol) dropwise, then reagent 3 (1 mmol) at 0°C and stir for 10 minutes, and then continue stirring at room temperature (rt) for 20 min. After completion of reaction as monitored by TLC, the reaction mixture was extracted with ethyl acetate (25 ml x 3) and washed with water (50 ml x 2). The organic layer was dried over anhydrous Na₂SO₄, concentrated via rotary evaporation. All compounds were purified by column chromatography with the eluent EtOAc/ hexane.

b) Synthesis of Product 5 (Scheme 2)



To a 25 ml oven dried round bottom flask dissolve 2 mmol of reagent 2 in 6 ml CHCl₃. Add reagent 1 (3 mmol) dropwise, then reagent 3 (1 mmol) at 0°C, stir for 10 minutes, and then continue stirring at room temperature (rt) for 20 min. After completion of reaction as monitored by TLC, the reaction mixture was extracted with ethyl acetate (25 ml x 3) and washed with water (50 ml x 2). The organic layer was dried over anhydrous Na₂SO₄, concentrated via rotary evaporation. All compounds were purified by column chromatography with the eluent EtOAc/ hexane.

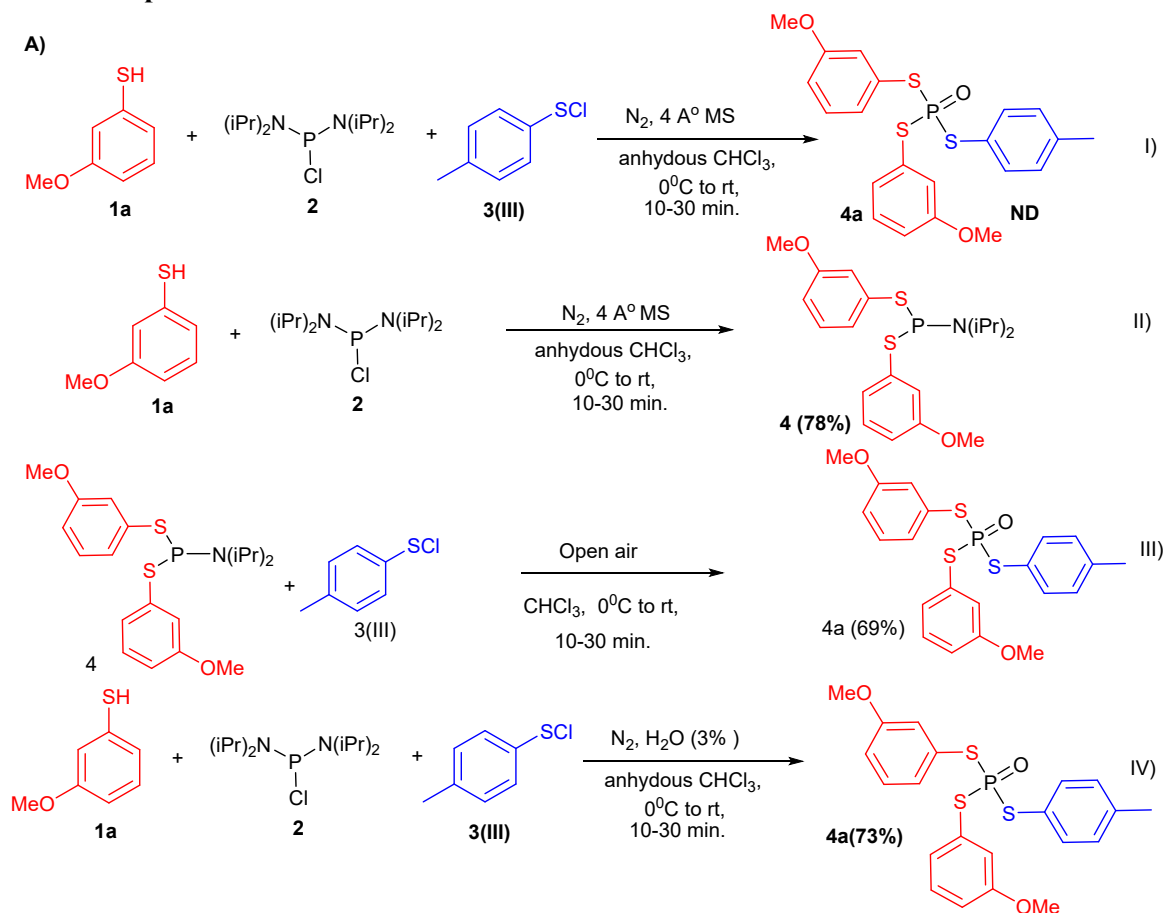
c) Synthesis of Product 6a/6b/6c/6d (Scheme 3)



To a 25 ml oven dried round bottom flask dissolve 2 mmol of reagent 2 in 6 ml CHCl₃. Add reagent 1 (3 mmol) dropwise, then reagent 6 (1 mmol) at 0°C, stir for 10 minutes, and then continue stirring at

room temperature (rt) for 20 min. After completion of reaction as monitored by TLC, the reaction mixture was extracted with ethyl acetate (25 ml x 3) and washed with water (50 ml x 2). The organic layer was dried over anhydrous Na₂SO₄, concentrated via rotary evaporation. All compounds were purified by column chromatography with the eluent EtOAc/ hexane

d) Control Experiments.



In experiment I) 3-methoxybenzenethiol **1a** (3 mmol) is added dropwise in oven dried round bottom flask having solution of reagent **2** (2 mmol) and anhydrous CHCl₃ under nitrogen atmosphere and had activated 4° molecular sieves. Followed by addition of reagent **3(III)** (1 mmol) at 0 °C and stirred for 30 minutes but no product is determined on TLC.

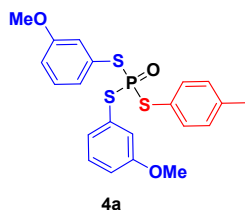
In experiment II) 3-methoxybenzenethiol **1a** (3 mmol) is added dropwise in oven dried round bottom flask having solution of reagent **2** (2 mmol) and anhydrous CHCl₃ under nitrogen atmosphere and had activated 4° molecular sieves and stirred the reaction for 10 minutes at 0 °C and for another 20 minutes at rt. After completion of reaction as monitored by TLC. The product **4** is isolated via column chromatography loaded with neutralized silica. Hexane and ethyl acetate were used as eluent.

In experiment III) Compound **4** (1.5 mmol) is added in oven dried round bottom flask containing CHCl₃ as solvent followed by addition of **3(III)** (1 mmol) and stirred the reaction for 10 minutes at 0 °C and for another 20 minutes at rt. After completion of reaction as monitored by TLC, the reaction mixture was extracted with ethyl acetate (25 ml x 3) and washed with water (50 ml x 2). The organic layer was dried over anhydrous Na₂SO₄, concentrated via rotary evaporation. Compound **4a** was purified by column chromatography with the eluent EtOAc/ hexane.

In experiment IV) 3-methoxybenzenethiol **1a** (3 mmol) is added dropwise in oven dried round bottom flask having solution of reagent **2** (2mmol) and anhydrous CHCl_3 under nitrogen atmosphere . Followed by addition of reagent **3(III)** (1 mmol) at 0 °C and 3% v/v H_2O (54 μl) was added to the reaction mixture and stirred for 30 minutes After completion of reaction as monitored by TLC. The product **4a** is isolated via column chromatography loaded with neutralized silica. Hexane and ethyl acetate were used as eluent.

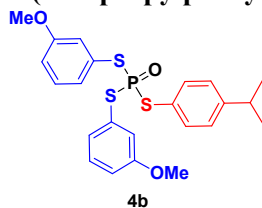
4. Characterization data of synthesized compounds

S,S-bis(3-methoxyphenyl) S-(p-tolyl) phosphorotrithioate (**4a**)



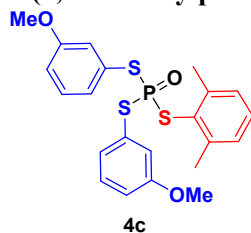
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 12:88); (327mg, 73%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.35 (dd, J = 8.2, 2.2 Hz, 2H), 7.19 (dd, J = 9.4, 6.6 Hz, 2H), 7.16 – 7.04 (m, 4H), 7.02 (dd, J = 4.1, 2.0 Hz, 2H), 6.94 – 6.81 (m, 2H), 3.71 (s, 6H), 2.29 (d, J = 2.5 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.8 (d, J = 2.7 Hz), 150.9 (d, J = 3.5 Hz), 135.8 (d, J = 4.6 Hz), 130.0 (d, J = 2.6 Hz), 127.7 (d, J = 5.6 Hz), 127.6 (d, J = 2.8 Hz), 123.1 (d, J = 7.0 Hz), 120.3 (d, J = 4.6 Hz), 116.1 (d, J = 3.2 Hz), 55.4 (s), 33.9 (s), 23.8 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 56.04 (s). HRMS (ESI) m/z : calcd for $\text{C}_{21}\text{H}_{21}\text{O}_3\text{PS}_3$ $[\text{M} + \text{H}]^+$, 449.0469; found, 449.0455.

S-(4-isopropylphenyl) S,S-bis(3-methoxyphenyl) phosphorotrithioate (**4b**)



The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 12:88); (333mg, 73%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.37 (dd, J = 8.4, 2.3 Hz, 2H), 7.15 (dd, J = 13.2, 8.2 Hz, 4H), 7.08 – 6.99 (m, 4H), 6.85 (m, 2H), 3.69 (s, 6H), 2.83 (dt, J = 13.8, 6.9 Hz, 1H), 1.16 (d, J = 6.9 Hz, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.82 (d, J = 2.7 Hz), 140.31 (s), 135.8 (d, J = 4.6 Hz), 130.2 (d, J = 2.9 Hz), 130.0 (d, J = 2.5 Hz), 127.7 (d, J = 5.3 Hz), 127.6 (s), 122.9 (d, J = 7.3 Hz), 120.3 (d, J = 4.6 Hz), 116.1 (d, J = 3.2 Hz), 55.4 (s), 30.3 (s), 21.3 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 55.86 (s). HRMS (ESI) m/z : calcd for $\text{C}_{23}\text{H}_{25}\text{O}_3\text{PS}_3$ $[\text{M} + \text{H}]^+$, 477.0731; found, 477.0726.

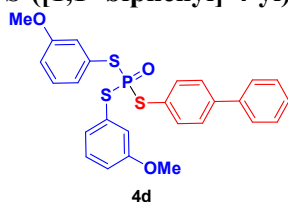
S-(2,6-dimethylphenyl) S,S-bis(3-methoxyphenyl) phosphorotrithioate (**4c**)



The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 12:88); (332mg, 73%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.18 (dd, J = 10.7, 5.3 Hz, 2H), 7.13 – 7.08 (m, 1H), 7.09 – 6.99 (m, 6H), 6.86 (m, 2H), 3.71 (s, 6H), 2.34 (d, J = 1.6 Hz, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.7 (s), 144.7 (d, J = 4.6 Hz), 130.0 (d, J = 3.8 Hz), 129.8 (d, J = 2.7 Hz), 128.6 (d,

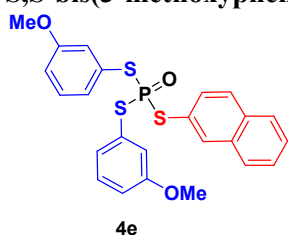
$J = 3.5$ Hz), 128.0 (d, $J = 6.9$ Hz), 127.9 (d, $J = 4.9$ Hz), 125.7 (s), 120.2 (d, $J = 4.5$ Hz), 116.1 (d, $J = 3.2$ Hz), 55.4 (s), 22.7 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 52.64 (s). HRMS (ESI) m/z : calcd for $\text{C}_{22}\text{H}_{23}\text{O}_3\text{PS}_3$ $[\text{M} + \text{H}]^+$, 463.0625; found, 463.0627.

S-([1,1'-biphenyl]-4-yl) S,S-bis(3-methoxyphenyl) phosphorotrithioate (4d)



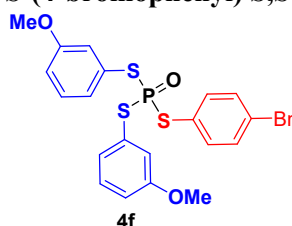
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 12:88); (362mg, 71%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.55 – 7.48 (m, 6H), 7.38 (dd, $J = 10.2$, 4.7 Hz, 2H), 7.32 – 7.27 (m, 1H), 7.22 – 7.17 (m, 2H), 7.12 – 7.02 (m, 4H), 6.87 (dt, $J = 8.3$, 1.9 Hz, 2H), 3.71 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.8 (d, $J = 2.7$ Hz), 142.7 (d, $J = 3.7$ Hz), 139.9 (s), 136.1 (d, $J = 4.7$ Hz), 130.0 (d, $J = 2.6$ Hz), 128.9 (s), 128.0 (d, $J = 2.7$ Hz), 127.9 (s), 127.8 (d, $J = 5.2$ Hz), 127.6 (d, $J = 6.9$ Hz), 127.1 (s), 125.3 (d, $J = 7.3$ Hz), 120.3 (d, $J = 4.6$ Hz), 116.2 (d, $J = 3.2$ Hz), 55.4 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 55.42 (s). HRMS (ESI) m/z : calcd for $\text{C}_{26}\text{H}_{23}\text{O}_3\text{PS}_3$ $[\text{M} + \text{H}]^+$, 511.0532; found, 511.0530.

S,S-bis(3-methoxyphenyl) S-(naphthalen-2-yl) phosphorotrithioate (4e)



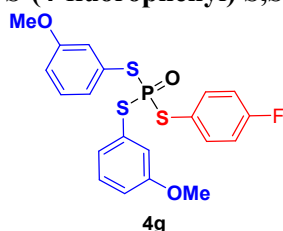
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 10:90); (333mg, 69%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 8.05 – 7.91 (m, 1H), 7.74 (d, $J = 8.0$ Hz, 3H), 7.57 – 7.42 (m, 3H), 7.19 (dd, $J = 9.3$, 6.7 Hz, 2H), 7.11 – 6.96 (m, 4H), 6.87 (m, 2H), 3.68 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.8 (d, $J = 2.7$ Hz), 136.0 (d, $J = 6.4$ Hz), 133.5 (d, $J = 6.2$ Hz), 131.8 (d, $J = 3.3$ Hz), 130.0 (d, $J = 2.5$ Hz), 129.0 (s), 128.8 (s), 128.1 (s), 127.8 (s), 127.8 (s), 127.7 (s), 127.6 (s), 127.5 (s), 127.4 (s), 126.7 (s), 123.9 (d, $J = 7.6$ Hz), 120.3 (d, $J = 4.6$ Hz), 116.2 (d, $J = 3.3$ Hz), 55.4 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 55.59 (s). HRMS (ESI) m/z : calcd for $\text{C}_{24}\text{H}_{21}\text{O}_3\text{PS}_3$ $[\text{M} + \text{H}]^+$, 485.0469; found, 485.0460.

S-(4-bromophenyl) S,S-bis(3-methoxyphenyl) phosphorotrithioate (4f)



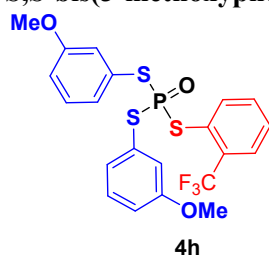
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 10:90); (362mg, 71%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.40 (d, $J = 8.2$ Hz, 2H), 7.33 – 7.28 (m, 2H), 7.24 – 7.16 (m, 2H), 7.06 (s, 2H), 7.00 (d, $J = 2.0$ Hz, 2H), 6.88 (dd, $J = 8.3$, 0.8 Hz, 2H), 3.72 (s, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.9 (d, $J = 2.4$ Hz), 137.1 (d, $J = 4.7$ Hz), 132.5 (d, $J = 2.7$ Hz), 130.1 (d, $J = 2.8$ Hz), 127.8 (d, $J = 5.2$ Hz), 127.2 (d, $J = 7.0$ Hz), 125.8 (d, $J = 7.0$ Hz), 124.7 (d, $J = 4.2$ Hz), 120.4 (d, $J = 4.6$ Hz), 116.2 (d, $J = 3.5$ Hz), 55.4 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 54.51 (s). HRMS (ESI) m/z : calcd for $\text{C}_{20}\text{H}_{18}\text{BrO}_3\text{PS}_3$ $[\text{M} + \text{H}]^+$, 512.9417; found, 512.9414.

S-(4-fluorophenyl) S,S-bis(3-methoxyphenyl) phosphorotrithioate (4g)



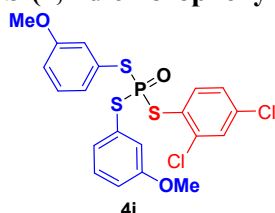
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 10:90); (307mg, 68%) as a colorless oil; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.42 (d, $J = 1.4$ Hz, 2H), 7.19 (dd, $J = 10.7, 5.3$ Hz, 2H), 7.05 (m, 4H), 7.03 – 6.93 (m, 2H), 6.87 (m, 2H), 3.70 (s, 6H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 163.89 (dd, $^1J_{\text{CF}} = 251.2, ^2J_{\text{CF}} = 3.6$ Hz), 159.89 (d, $J = 2.7$ Hz), 137.96 (d, $J = 4.5$ Hz), 137.88 (d, $J = 4.5$ Hz), 130.11 (d, $J = 2.5$ Hz), 127.80 (d, $J = 5.1$ Hz), 127.40 (d, $J = 7.1$ Hz), 120.41 (d, $J = 4.6$ Hz), 116.76 (d, $J = 2.8$ Hz), 116.54 (d, $J = 2.8$ Hz), 116.23 (d, $J = 3.3$ Hz), 55.46 (s). $^{31}\text{P NMR}$ (162 MHz, CDCl_3) δ 55.17 (d, $J = 6.0$ Hz). $^{19}\text{F NMR}$ (377 MHz, CDCl_3) δ -109.57 – -111.71 (m). HRMS (ESI) m/z : calcd for $\text{C}_{20}\text{H}_{18}\text{FO}_3\text{PS}_3$ [$\text{M} + \text{H}$] $^+$, 453.0218; found, 453.0216.

S,S-bis(3-methoxyphenyl) S-(2-(trifluoromethyl)phenyl) phosphorotrithioate (4h)



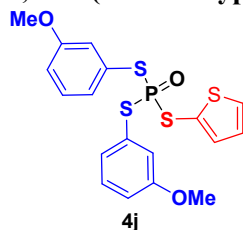
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 14:86); (336mg, 67%) as a colorless oil; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.75 – 7.60 (m, 1H), 7.47 – 7.38 (m, 1H), 7.19 (dd, $J = 10.2, 5.8$ Hz, 3H), 7.14 – 7.00 (m, 5H), 6.90 – 6.85 (m, 2H), 3.72 (d, $J = 1.2$ Hz, 6H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 159.85 (d, $J = 2.5$ Hz), 138.53 (d, $J = 4.6$ Hz), 138.39 (d, $J = 4.5$ Hz), 132.22 (s), 130.09 (s), 129.52 (d, $J = 2.4$ Hz), 128.82 (dd, $^1J_{\text{CF}} = 248.5, ^2J_{\text{CF}} = 4.8$ Hz), 127.99 (d, $J = 5.3$ Hz), 127.83 (d, $J = 5.2$ Hz), 127.19 (d, $J = 7.4$ Hz), 120.37 (d, $J = 4.6$ Hz), 116.46 (d, $J = 3.6$ Hz), 116.22 (d, $J = 3.2$ Hz), 55.46 (s). $^{31}\text{P NMR}$ (162 MHz, CDCl_3) δ 55.50 (d, $J = 6.1$ Hz). $^{19}\text{F NMR}$ (377 MHz, CDCl_3) δ -59.30 (d, $J = 6.6$ Hz). HRMS (ESI) m/z : calcd for $\text{C}_{21}\text{H}_{18}\text{F}_3\text{O}_3\text{PS}_3$ [$\text{M} + \text{H}$] $^+$, 503.0186; found, 503.0176.

S-(2,4-dichlorophenyl) S,S-bis(3-methoxyphenyl) phosphorotrithioate (4i)



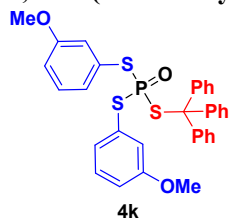
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 14:86); (355mg, 71%) as a colorless oil; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.50 (dd, $J = 8.5, 2.2$ Hz, 1H), 7.38 (dd, $J = 2.2, 0.7$ Hz, 1H), 7.19 (t, $J = 8.0$ Hz, 2H), 7.12 (m, 1H), 7.07 (m, 2H), 7.02 (dd, $J = 4.1, 2.2$ Hz, 2H), 6.87 (dd, $J = 8.3, 0.9$ Hz, 2H), 3.70 (s, 6H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 159.9 (d, $J = 2.5$ Hz), 139.6 (d, $J = 5.5$ Hz), 138.3 (d, $J = 4.0$ Hz), 136.7 (d, $J = 3.4$ Hz), 130.2 (d, $J = 2.3$ Hz), 130.1 (d, $J = 2.9$ Hz), 145.97 – 99.9 (m), 127.9 (d, $J = 5.2$ Hz), 127.8 (d, $J = 2.3$ Hz), 127.1 (d, $J = 7.1$ Hz), 125.1 (d, $J = 6.9$ Hz), 120.4 (d, $J = 4.6$ Hz), 116.4 (d, $J = 3.3$ Hz), 55.4 (s). $^{31}\text{P NMR}$ (162 MHz, CDCl_3) δ 53.81 (s). HRMS (ESI) m/z : calcd for $\text{C}_{20}\text{H}_{17}\text{Cl}_2\text{O}_3\text{PS}_3$ [$\text{M} + \text{H}$] $^+$, 502.9533; found, 502.9533.

S,S-bis(3-methoxyphenyl) S-(thiophen-2-yl) phosphorotrithioate (4j)



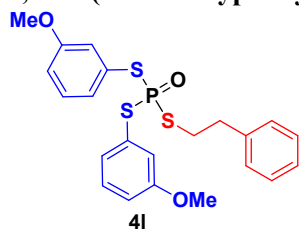
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 12:88); (294mg, 67%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.43 (dd, $J = 3.5, 1.3$ Hz, 1H), 7.19 (m, 3H), 7.11 – 6.96 (m, 5H), 6.93 – 6.79 (m, 2H), 3.70 (d, $J = 4.9$ Hz, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.9 (d, $J = 2.4$ Hz), 159.8 (d, $J = 2.6$ Hz), 138.2 (d, $J = 6.4$ Hz), 138.0 (d, $J = 6.3$ Hz), 132.8 (d, $J = 5.4$ Hz), 132.6 (d, $J = 5.3$ Hz), 130.1 (d, $J = 2.7$ Hz), 130.1 (d, $J = 2.5$ Hz), 128.2 (d, $J = 3.7$ Hz), 128.1 (d, $J = 3.8$ Hz), 127.9 (s), 127.8 (d, $J = 5.1$ Hz), 127.5 (d, $J = 7.0$ Hz), 127.1 (d, $J = 6.9$ Hz), 120.5 (d, $J = 19.3$ Hz), 116.3 (d, $J = 18.3$ Hz), 55.4 (d, $J = 3.4$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 59.70 (s). HRMS (ESI) m/z : calcd for $\text{C}_{18}\text{H}_{17}\text{O}_3\text{PS}_4$ $[\text{M} + \text{H}]^+$, 440.9876; found, 440.9876

S,S-bis(3-methoxyphenyl) S-trityl phosphorotrithioate (4k)



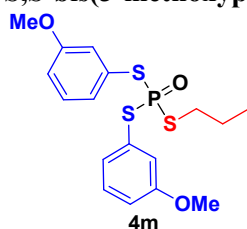
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 12:88); (294mg, 49%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.58 – 7.44 (m, 8H), 7.42 – 7.33 (m, 3H), 7.32 – 7.26 (m, 1H), 7.26 – 7.17 (m, 3H), 7.14 – 7.00 (m, 5H), 6.96 – 6.83 (m, 3H), 3.71 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.8 (s), 142.6 (s), 139.9 (s), 136.1 (d, $J = 4.7$ Hz), 130.0 (d, $J = 2.7$ Hz), 128.9 (s), 128.0 (d, $J = 2.7$ Hz), 127.9 (s), 127.8 (d, $J = 5.1$ Hz), 127.5 (d, $J = 6.9$ Hz), 127.1 (s), 125.3 (s), 120.3 (d, $J = 4.6$ Hz), 116.2 (d, $J = 3.5$ Hz), 55.4 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 55.52 (s). HRMS (ESI) m/z : calcd for $\text{C}_{33}\text{H}_{29}\text{O}_3\text{PS}_3$ $[\text{M} + \text{H}]^+$, 601.1013; found, 601.1011.

S,S-bis(3-methoxyphenyl) S-phenethyl phosphorotrithioate (4l)



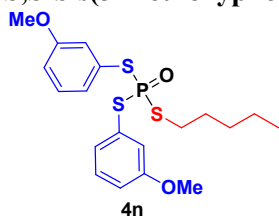
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 12:88); (362mg, 71%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.27 – 7.05 (m, 11H), 6.91 – 6.81 (m, 2H), 3.72 (s, 6H), 3.13 (m, 2H), 2.88 (t, $J = 7.6$ Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.8 (d, $J = 2.8$ Hz), 139.2 (s), 130.0 (d, $J = 2.5$ Hz), 128.6 (d, $J = 16.2$ Hz), 127.7 (t, $J = 6.8$ Hz), 126.7 (s), 120.4 (d, $J = 4.6$ Hz), 116. (d, $J = 3.5$ Hz), 55.4 (s), 36.9 (d, $J = 4.5$ Hz), 34.1 (d, $J = 3.8$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 59.15 (s). HRMS (ESI) m/z : calcd for $\text{C}_{22}\text{H}_{23}\text{O}_3\text{PS}_3$ $[\text{M} + \text{H}]^+$, 463.0546; found, 463.0538.

S,S-bis(3-methoxyphenyl) S-propyl phosphorotrithioate (4m)



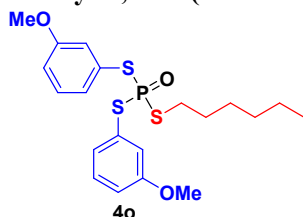
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 12:88); (276mg, 69%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.25 – 7.17 (m, 2H), 7.17 – 7.02 (m, 4H), 6.88 (m, 2H), 3.74 (s, 6H), 2.89 (dt, $J = 14.7, 7.3$ Hz, 2H), 1.72 – 1.57 (m, 2H), 0.90 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.8 (d, $J = 2.4$ Hz), 130.0 (d, $J = 2.6$ Hz), 127. (d, $J = 7.2$ Hz), 127.7 (d, $J = 5.2$ Hz), 120.3 (d, $J = 4.6$ Hz), 116.1 (d, $J = 3.3$ Hz), 55.4 (s), 34.9 (d, $J = 4.1$ Hz), 24.0 (d, $J = 4.9$ Hz), 13.2 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 59.42 (s). HRMS (ESI) m/z : calcd for $\text{C}_{17}\text{H}_{21}\text{O}_3\text{PS}_3$ [$\text{M} + \text{H}$] $^+$, 401.0469; found, 401.0463.

S,S-bis(3-methoxyphenyl) S-pentyl phosphorotrithioate (4n)



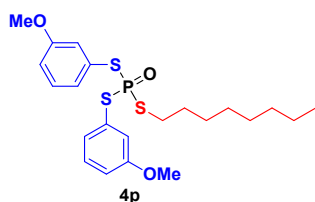
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 13:87); (295mg, 69%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.23 – 7.19 (m, 2H), 7.13 – 7.06 (m, 4H), 6.88 (m, 2H), 3.74 (s, 6H), 2.96 – 2.84 (m, 2H), 1.60 (s, 2H), 1.26 – 1.22 (m, 4H), 0.82 (d, $J = 7.0$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.8 (s), 130.0 (d, $J = 2.5$ Hz), 127.8 (dd, $J = 15.1, 6.2$ Hz), 120.3 (d, $J = 4.6$ Hz), 116.1 (d, $J = 3.2$ Hz), 55.4 (s), 33.0 (d, $J = 4.0$ Hz), 30.6 (s), 30.2 (d, $J = 5.1$ Hz), 22.1 (s), 13.9 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 59.38 (s). HRMS (ESI) m/z : calcd for $\text{C}_{19}\text{H}_{25}\text{O}_3\text{PS}_3$ [$\text{M} + \text{H}$] $^+$, 429.0764; found, 429.0766.

S-hexyl S,S-bis(3-methoxyphenyl) phosphorotrithioate (4o)



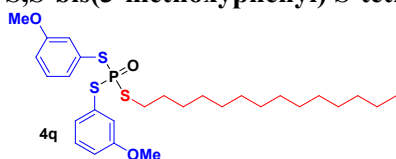
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 13:87); (309mg, 70%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.21 (t, $J = 7.7$ Hz, 2H), 7.12 – 7.06 (m, 4H), 6.88 (m, 2H), 3.74 (s, 6H), 2.96 – 2.86 (m, 2H), 1.58 (d, $J = 7.5$ Hz, 2H), 1.24 – 1.17 (m, 6H), 0.80 (t, $J = 6.9$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.8 (d, $J = 2.5$ Hz), 130.0 (d, $J = 2.6$ Hz), 127.9 (d, $J = 7.2$ Hz), 127.8 (dd, $J = 15.7, 6.2$ Hz), 127.7 (d, $J = 5.1$ Hz), 120.3 (d, $J = 4.7$ Hz), 55.4 (s), 33.1 (d, $J = 4.2$ Hz), 31.1 (s), 30.5 (d, $J = 4.9$ Hz), 28.2 (s), 22.4 (s), 14.0 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 59.35 (s). HRMS (ESI) m/z : calcd for $\text{C}_{20}\text{H}_{27}\text{O}_3\text{PS}_3$ [$\text{M} + \text{H}$] $^+$, 443.0911; found, 443.0910.

S,S-bis(3-methoxyphenyl) S-octyl phosphorotrithioate (4p)



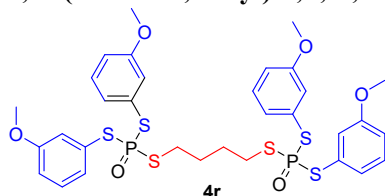
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 13:87); (470mg, 67%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.19 (d, $J = 8.0$ Hz, 2H), 7.13 – 7.05 (m, 4H), 6.92 – 6.81 (m, 2H), 3.73 (s, 6H), 2.98 – 2.82 (m, 2H), 1.59 (dd, $J = 14.8, 7.2$ Hz, 2H), 1.18 (s, 10H), 0.80 (t, $J = 6.9$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.8 (d, $J = 2.4$ Hz), 130.0 (d, $J = 2.5$ Hz), 127.9 (d, $J = 7.0$ Hz), 127.7 (d, $J = 4.9$ Hz), 120.3 (d, $J = 4.6$ Hz), 116.1 (d, $J = 3.2$ Hz), 55.4 (s), 33.1 (d, $J = 4.1$ Hz), 31.7 (s), 30.5 (d, $J = 5.1$ Hz), 29.1 (s), 28.9 (s), 28.5 (s), 22.6 (s), 14.1 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 59.39 (s). HRMS (ESI) m/z : calcd for $\text{C}_{22}\text{H}_{31}\text{O}_3\text{PS}_3$ $[\text{M} + \text{H}]^+$, 471.1233; found, 471.1229.

S,S-bis(3-methoxyphenyl) S-tetradecyl phosphorotrithioate (4q)



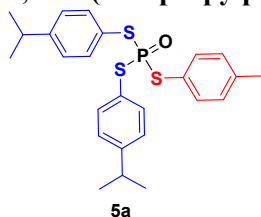
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 13:87); (376mg, 68%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.20 (t, $J = 8.0$ Hz, 2H), 7.14 – 7.03 (m, 4H), 6.87 (dd, $J = 8.3, 0.9$ Hz, 2H), 3.73 (s, 6H), 2.89 (dt, $J = 14.7, 7.4$ Hz, 2H), 1.58 (d, $J = 7.4$ Hz, 2H), 1.19 (d, $J = 13.9$ Hz, 22H), 0.80 (t, $J = 6.9$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.83 (d, $J = 2.7$ Hz), 130.05 (d, $J = 2.7$ Hz), 127.90 (d, $J = 7.2$ Hz), 127.76 (d, $J = 5.1$ Hz), 120.33 (d, $J = 4.8$ Hz), 116.10 (d, $J = 3.2$ Hz), 55.45 (s), 33.12 (d, $J = 3.9$ Hz), 31.96 (s), 30.56 (d, $J = 5.0$ Hz), 29.73 (s), 29.71 (s), 29.69 (s), 29.59 (s), 29.47 (s), 29.40 (s), 29.04 (s), 28.60 (s), 22.74 (s), 14.19 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 59.33 (s). HRMS (ESI) m/z : calcd for $\text{C}_{28}\text{H}_{43}\text{O}_3\text{PS}_3$ $[\text{M} + \text{H}]^+$, 554.2123; found, 554.2123.

S,S'-(butane-1,4-diyl) S,S,S',S'-tetrakis(3-methoxyphenyl) bis(phosphorotrithioate) (4r)



The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 18:82); (770mg, 56%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.19 (t, $J = 8.0$ Hz, 4H), 7.06 (d, $J = 7.7$ Hz, 4H), 7.02 (dd, $J = 4.1, 2.0$ Hz, 4H), 6.91 – 6.83 (m, 4H), 3.71 (s, 12H), 3.33 – 3.26 (m, 2H), 3.26 – 3.18 (m, 2H), 2.29 (dd, $J = 11.8, 6.0$ Hz, 2H), 1.94 (dd, $J = 13.6, 8.7$ Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.8 (d, $J = 2.5$ Hz), 130.0 (d, $J = 2.5$ Hz), 127.8 (d, $J = 5.2$ Hz), 127.6 (dd, $J = 24.0, 6.2$ Hz), 127.5 (d, $J = 7.2$ Hz), 120.4 (d, $J = 4.7$ Hz), 116.1 (d, $J = 3.2$ Hz), 59.7 (s), 55.4 (s), 35.2 (s), 26.2 (s), 25.1 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 55.67 (s). HRMS (ESI) m/z : calcd for $\text{C}_{32}\text{H}_{36}\text{O}_6\text{P}_2\text{S}_6$ $[\text{M} + \text{H}]^+$, 771.0336; found, 771.0339.

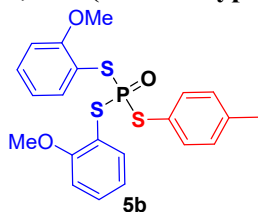
S,S-bis(4-isopropylphenyl) S-(p-tolyl) phosphorotrithioate (5a)



The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 18:82); (335mg, 71%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.36 (m, 6H), 7.13 (d, $J = 8.2$ Hz, 4H),

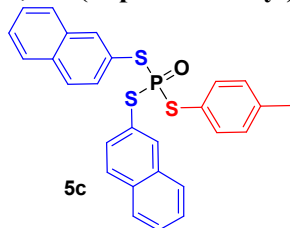
7.08 (d, $J = 8.0$ Hz, 2H), 2.83 (dt, $J = 13.8, 6.9$ Hz, 2H), 2.28 (d, $J = 2.1$ Hz, 3H), 1.17 (d, $J = 6.9$ Hz, 12H). ^{13}C NMR (101 MHz, CDCl_3) δ 150.7 (d, $J = 3.6$ Hz), 140.0 (d, $J = 3.7$ Hz), 135.8 (d, $J = 4.6$ Hz), 135.7 (d, $J = 4.7$ Hz), 130.1 (d, $J = 2.7$ Hz), 127.5 (d, $J = 2.5$ Hz), 123.5 (d, $J = 7.1$ Hz), 123.3 (d, $J = 6.9$ Hz), 33.9 (s), 29.7 (s), 23.8 (s), 21.3 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 56.64 (s). HRMS (ESI) m/z : calcd for $\text{C}_{25}\text{H}_{29}\text{OPS}_3$ $[\text{M} + \text{H}]^+$, 473.1132; found, 473.1129.

S,S-bis(2-methoxyphenyl) S-(p-tolyl) phosphorotrithioate (5b)



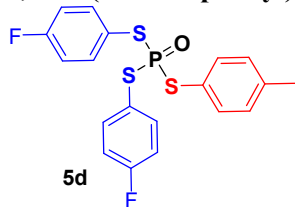
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 12:88); (327mg, 73%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.50 (dt, $J = 7.7, 1.9$ Hz, 2H), 7.36 (dd, $J = 8.2, 2.2$ Hz, 2H), 7.32 – 7.23 (m, 2H), 7.06 (d, $J = 8.2$ Hz, 2H), 6.84 (dd, $J = 12.5, 5.0$ Hz, 4H), 3.74 (s, 6H), 2.26 (d, $J = 2.4$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.9 (d, $J = 4.5$ Hz), 139.7 (d, $J = 3.5$ Hz), 137.6 (d, $J = 4.3$ Hz), 135.6 (d, $J = 4.7$ Hz), 131.4 (d, $J = 3.0$ Hz), 129.9 (d, $J = 2.6$ Hz), 123.8 (d, $J = 7.2$ Hz), 121.1 (d, $J = 2.4$ Hz), 115.4 (d, $J = 7.0$ Hz), 111.6 (d, $J = 2.2$ Hz), 55.9 (s), 21.3 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 55.78 (s). HRMS (ESI) m/z : calcd for $\text{C}_{21}\text{H}_{21}\text{O}_3\text{PS}_3$ $[\text{M} + \text{H}]^+$, 449.0469; found, 449.0455.

S,S-di(naphthalen-2-yl) S-(p-tolyl) phosphorotrithioate (5c)



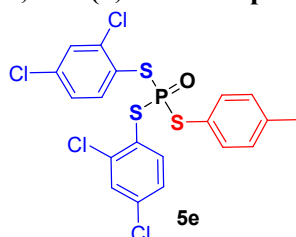
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 04:96); (309mg, 69%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.95 (d, $J = 2.2$ Hz, 2H), 7.87 – 7.60 (m, 6H), 7.57 – 7.33 (m, 8H), 7.08 (d, $J = 8.2$ Hz, 2H), 2.28 (d, $J = 2.5$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 140.3 (d, $J = 8.6$ Hz), 135.9 (d, $J = 6.4$ Hz), 135.8 (d, $J = 4.6$ Hz), 133.5 (d, $J = 2.9$ Hz), 133.4 (d, $J = 2.3$ Hz), 131.7 (d, $J = 3.5$ Hz), 130.2 (d, $J = 2.6$ Hz), 128.9 (d, $J = 1.9$ Hz), 128.0 (s), 127.7 (s), 127.3 (s), 126.7 (s), 124.0 (d, $J = 7.3$ Hz), 122.9 (d, $J = 10.1$ Hz), 21.3 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 55.92 (s).

S,S-bis(4-fluorophenyl) S-(p-tolyl) phosphorotrithioate (5d)



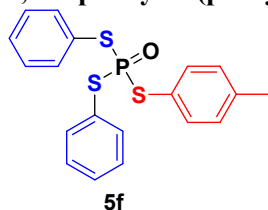
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 04:96); (296mg, 70%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.45 – 7.37 (m, 4H), 7.35 – 7.31 (m, 2H), 7.09 (d, $J = 8.3$ Hz, 2H), 7.01 – 6.94 (m, 4H), 2.28 (d, $J = 2.5$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 163.89 (dd, $J = 251.1, 3.7$ Hz), 140.46 (d, $J = 3.7$ Hz), 137.89 (d, $J = 4.5$ Hz), 137.81 (d, $J = 4.4$ Hz), 135.75 (d, $J = 4.6$ Hz), 130.33 (d, $J = 2.7$ Hz), 122.58 (d, $J = 7.0$ Hz), 121.79 (d, $J = 3.4$ Hz), 121.72 (d, $J = 3.5$ Hz), 116.77 (d, $J = 2.4$ Hz), 116.55 (d, $J = 2.4$ Hz), 21.37 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 55.16 (t, $J = 6.5$ Hz). ^{19}F NMR (377 MHz, CDCl_3) δ -99.04 – -126.58 (m). HRMS (ESI) m/z : calcd for $\text{C}_{19}\text{H}_{15}\text{F}_2\text{OPS}_3$ $[\text{M} + \text{H}]^+$, 425.0013; found, 425.0008.

S,S-bis(2,4-dichlorophenyl) S-(p-tolyl) phosphorotrithioate (5e)



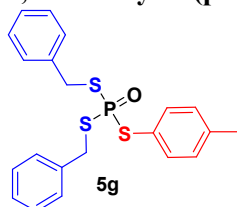
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 04:96); (355mg, 68%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.49 (dd, $J = 8.5, 2.2$ Hz, 2H), 7.36 (m, 4H), 7.14 – 7.05 (m, 4H), 2.27 (d, $J = 2.7$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 140.7 (d, $J = 3.9$ Hz), 139.8 (d, $J = 5.6$ Hz), 138.3 (d, $J = 4.2$ Hz), 136.9 (d, $J = 3.7$ Hz), 135.8 (d, $J = 4.7$ Hz), 130.3 (d, $J = 3.0$ Hz), 130.3 (d, $J = 2.6$ Hz), 127.8 (d, $J = 2.4$ Hz), 127.5 (s), 124.8 (d, $J = 6.9$ Hz), 122.1 (d, $J = 7.5$ Hz), 21.4 (d, $J = 1.0$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 52.69 (s). HRMS (ESI) m/z : calcd for $\text{C}_{19}\text{H}_{13}\text{Cl}_4\text{OPS}_3$ [$\text{M} + \text{H}$] $^+$, 524.8669; found, 524.8666.

S,S-diphenyl S-(p-tolyl) phosphorotrithioate (5f)



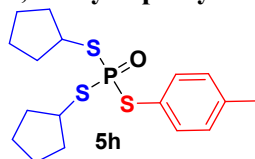
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 04:96); (275mg, 71%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.54 (dd, $J = 5.1, 2.9$ Hz, 4H), 7.38 (m, 8H), 7.16 (d, $J = 7.9$ Hz, 2H), 2.35 (d, $J = 2.5$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 140.26 (d, $J = 3.7$ Hz), 135.81 (s), 135.75 (d, $J = 4.6$ Hz), 130.27 (d, $J = 2.7$ Hz), 129.84 (d, $J = 3.2$ Hz), 129.77 (d, $J = 3.3$ Hz), 129.44 (s), 129.41 (d, $J = 2.7$ Hz), 126.89 (d, $J = 7.0$ Hz), 126.77 (d, $J = 7.1$ Hz), 123.02 (d, $J = 7.2$ Hz), 21.40 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 55.93 (s). HRMS (ESI) m/z : calcd for $\text{C}_{19}\text{H}_{17}\text{OPS}_3$ [$\text{M} + \text{H}$] $^+$, 389.0219; found, 389.0216.

S,S-dibenzyl S-(p-tolyl) phosphorotrithioate (5g)



The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 04:96); (291mg, 70%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.37 (dd, $J = 8.2, 2.3$ Hz, 2H), 7.32 – 7.15 (m, 10H), 7.10 (d, $J = 8.4$ Hz, 2H), 4.13 – 4.09 (m, 3H), 4.09 – 4.04 (m, 2H), 2.28 (d, $J = 2.4$ Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 140.3 (d, $J = 3.8$ Hz), 136.2 (d, $J = 6.3$ Hz), 135.78 (d, $J = 4.8$ Hz), 130.3 (d, $J = 3.0$ Hz), 129.3 (d, $J = 4.0$ Hz), 128.8 (d, $J = 4.5$ Hz), 127.8 (d, $J = 4.3$ Hz), 123.1 (d, $J = 7.2$ Hz), 36.9 (d, $J = 3.7$ Hz), 36.8 (d, $J = 3.7$ Hz), 21.4 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 61.79 (s). HRMS (ESI) m/z : calcd for $\text{C}_{21}\text{H}_{21}\text{OPS}_3$ [$\text{M} + \text{H}$] $^+$, 417.0570; found, 417.0574.

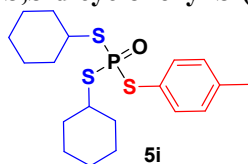
S,S-dicyclopentyl S-(p-tolyl) phosphorotrithioate (5h)



The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 04:96); (260mg, 70%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.46 – 7.37 (m, 2H), 7.11 (d, $J = 8.3$ Hz,

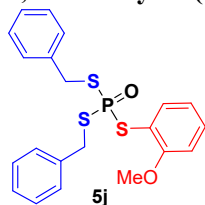
2H), 3.68 – 3.53 (m, 2H), 2.29 (d, $J = 2.4$ Hz, 3H), 2.18 – 1.98 (m, 4H), 1.71 – 1.45 (m, 12H), 1.21 (dd, $J = 11.7, 6.8$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 139.9 (d, $J = 3.7$ Hz), 135.5 (d, $J = 4.6$ Hz), 130.1 (d, $J = 2.6$ Hz), 124.0 (d, $J = 7.3$ Hz), 46.8 (d, $J = 3.9$ Hz), 35.2 (d, $J = 2.2$ Hz), 35.2 (d, $J = 2.1$ Hz), 24.3 (d, $J = 1.3$ Hz), 21.3 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 59.80 (s). HRMS (ESI) m/z : calcd for $\text{C}_{17}\text{H}_{25}\text{OPS}_3$ [$\text{M} + \text{H}$] $^+$, 373.0883; found, 373.0887.

S,S-dicyclohexyl S-(p-tolyl) phosphorotrithioate (5i)



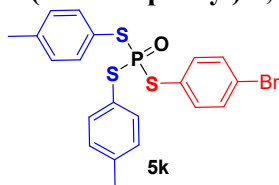
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 06:94); (276mg, 69%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.42 (m, 2H), 7.11 (d, $J = 8.4$ Hz, 2H), 3.39 (dd, $J = 10.2, 3.8$ Hz, 2H), 2.29 (d, $J = 2.4$ Hz, 3H), 2.21 – 1.81 (m, 4H), 1.87 – 1.60 (m, 4H), 1.48 (d, 5H), 1.33 (m, 4H), 1.26 – 1.10 (m, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 139.8 (d, $J = 3.5$ Hz), 135.5 (d, $J = 4.6$ Hz), 130.0 (d, $J = 2.6$ Hz), 124.3 (d, $J = 7.0$ Hz), 47.8 (d, $J = 3.7$ Hz), 35.3 (d, $J = 4.7$ Hz), 35.2 (d, $J = 4.7$ Hz), 25.9 (s), 25.3 (s), 21.3 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 59.63 (s). HRMS (ESI) m/z : calcd for $\text{C}_{19}\text{H}_{29}\text{OPS}_3$ [$\text{M} + \text{H}$] $^+$, 401.1196; found, 401.1189.

S,S-dibenzyl S-(2-methoxyphenyl) phosphorotrithioate (5j)



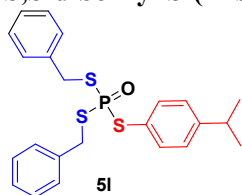
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 12:88); (306mg, 71%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.66 – 7.62 (m, 1H), 7.45 – 7.38 (m, 1H), 7.34 – 7.21 (m, 10H), 7.01 – 6.92 (m, 2H), 4.25 – 4.13 (m, 4H), 3.86 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 160.1 (d, $J = 15.5$ Hz), 138.0 (d, $J = 4.5$ Hz), 136.3 (d, $J = 10.3$ Hz), 131.9 (d, $J = 13.2$ Hz), 129.3 (s), 128.7 (s), 127.7 (s), 121.4 (s), 114.8 (d, $J = 16.3$ Hz), 111.7 (s), 55.9 (s), 36.8 (d, $J = 3.5$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 61.32 (s). HRMS (ESI) m/z : calcd for $\text{C}_{21}\text{H}_{21}\text{O}_2\text{PS}_3$ [$\text{M} + \text{H}$] $^+$, 433.0416; found, 433.0419.

S-(4-bromophenyl) S,S-di-p-tolyl phosphorotrithioate (5k)



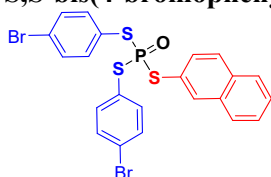
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 5:95); (330mg, 69%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.48 – 7.27 (m, 8H), 7.15 – 7.03 (m, 4H), 2.29 (d, $J = 2.5$ Hz, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 140.3 (d, $J = 10.3$ Hz), 137.0 (d, $J = 4.6$ Hz), 135.7 (d, $J = 4.6$ Hz), 132.4 (d, $J = 2.3$ Hz), 130.2 (d, $J = 2.8$ Hz), 126.1 (d, $J = 6.9$ Hz), 124.5 (d, $J = 3.8$ Hz), 122.8 (d, $J = 3.0$ Hz), 21.3 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 55.34 (s). HRMS (ESI) m/z : calcd for $\text{C}_{20}\text{H}_{18}\text{BrOPS}_3$ [$\text{M} + \text{H}$] $^+$, 480.9519; found, 480.9519.

S,S-dibenzyl S-(4-isopropylphenyl) phosphorotrithioate (5l)



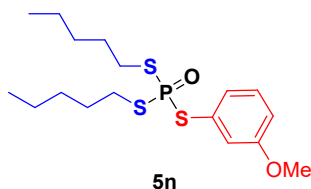
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 5:95); (319mg, 69%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.42 (dd, $J = 8.3, 2.3$ Hz, 2H), 7.35 – 6.90 (m, 12H), 4.09 (dd, $J = 12.2, 4.9$ Hz, 4H), 2.85 (dt, $J = 13.8, 6.9$ Hz, 1H), 1.18 (d, $J = 4.4$ Hz, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 151.0 (d, $J = 3.7$ Hz), 136.2 (s), 135.8 (d, $J = 4.6$ Hz), 129.32 (s), 128.7 (s), 127.8 (s), 127.7 (d, $J = 2.7$ Hz), 123.3 (d, $J = 12.3$ Hz), 36.8 (d, $J = 3.4$ Hz), 33.9 (s), 23.8 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 60.61 (s). HRMS (ESI) m/z : calcd for $\text{C}_{23}\text{H}_{25}\text{OPS}_3$ $[\text{M} + \text{H}]^+$, 445.0832; found, 445.0831.

S,S-bis(4-bromophenyl) S-(naphthalen-2-yl) phosphorotrithioate (5m)



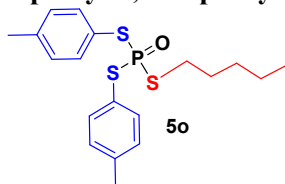
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 5:95); (393mg, 69%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.95 (s, 2H), 7.70 (m, 6H), 7.58 – 7.39 (m, 7H). ^{13}C NMR (101 MHz, CDCl_3) δ 136.6 (d, $J = 2.1$ Hz), 136.0 (d, $J = 6.4$ Hz), 133.5 (d, $J = 2.9$ Hz), 133.4 (d, $J = 2.2$ Hz), 131.7 (d, $J = 3.3$ Hz), 129.5 (d, $J = 10.3$ Hz), 129.0 (d, $J = 1.7$ Hz), 128.0 (d, $J = 1.0$ Hz), 127.7 (d, $J = 1.2$ Hz), 127.4 (d, $J = 1.1$ Hz), 126.7 (s), 123.8 (d, $J = 7.2$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 55.95 (s). HRMS (ESI) m/z : calcd for $\text{C}_{22}\text{H}_{15}\text{Br}_2\text{OPS}_3$ $[\text{M} + \text{H}]^+$, 580.8468; found, 580.8469.

S-(3-methoxyphenyl) S,S-dipentyl phosphorotrithioate (5n)



The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 5:95); (274mg, 70%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.46 – 7.28 (m, 1H), 7.31 – 7.15 (m, 1H), 7.18 – 7.08 (m, 1H), 6.95 – 6.83 (m, 1H), 3.75 (s, 3H), 3.04 – 2.79 (m, 4H), 1.75 – 1.54 (m, 4H), 1.37 – 1.21 (m, 8H), 0.82 (t, $J = 7.1$ Hz, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.8 (d, $J = 2.8$ Hz), 135.6 (d, $J = 4.6$ Hz), 130.1 (d, $J = 2.8$ Hz), 127.6 (d, $J = 4.8$ Hz), 120.3 (d, $J = 4.6$ Hz), 116.01 (d, $J = 3.3$ Hz), 55.4 (s), 32.9 (d, $J = 3.9$ Hz), 30.7 (s), 30.2 (d, $J = 5.3$ Hz), 22.1 (s), 13.9 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 61.41 (s).

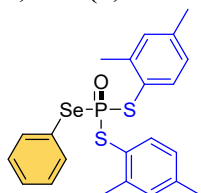
S-pentyl S,S-di-p-tolyl phosphorotrithioate (5o)



The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 5:95); (269mg, 68%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.40 (dd, $J = 8.2, 2.2$ Hz, 4H), 7.10 (d, $J = 8.4$ Hz, 4H), 2.98 – 2.70 (m, 2H), 2.28 (d, $J = 2.4$ Hz, 6H), 1.70 – 1.47 (m, 2H), 1.37 – 1.12 (m, 6H), 0.80 (t, $J = 6.9$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 140.1 (d, $J = 3.7$ Hz), 135.6 (d, $J = 4.7$

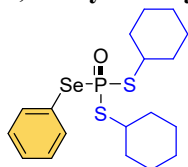
Hz), 130.2 (d, $J = 2.7$ Hz), 123.4 (d, $J = 7.1$ Hz), 32.9 (d, $J = 4.0$ Hz), 31.1 (s), 30.4 (d, $J = 5.1$ Hz), 28.2 (s), 22.4 (s), 21.3 (s), 14.0 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 60.05 (s).

S,S-bis(2,4-dimethylphenyl) Se-phenyl phosphoroselenodithioate (6a)



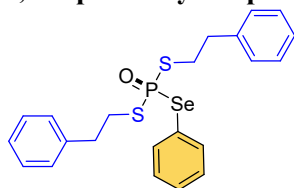
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 5:95); (338mg, 71%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.75 – 7.51 (m, 2H), 7.36 (dd, $J = 7.9$, 2.4 Hz, 2H), 7.34 – 7.27 (m, 1H), 7.23 (d, $J = 7.8$ Hz, 2H), 6.98 (s, 2H), 6.88 (t, $J = 7.3$ Hz, 2H), 2.22 (d, $J = 2.6$ Hz, 6H), 2.20 (d, $J = 1.3$ Hz, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 143.0 (d, $J = 4.5$ Hz), 140.4 (d, $J = 3.8$ Hz), 137.1 (d, $J = 4.4$ Hz), 136.5 (d, $J = 3.9$ Hz), 131.8 (d, $J = 2.9$ Hz), 129.3 (d, $J = 2.2$ Hz), 129.2 (d, $J = 2.8$ Hz), 127.5 (d, $J = 2.9$ Hz), 125.8 (d, $J = 7.5$ Hz), 123.2 (d, $J = 7.3$ Hz), 21.3 (d, $J = 19.2$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 44.10 (s), 42.67 (s), 41.24 (s). HRMS (ESI) m/z : calcd for $\text{C}_{22}\text{H}_{23}\text{OPS}_2\text{Se}$ [$\text{M} + \text{H}$] $^+$, 478.0146; found, 478.0142.

S,S-dicyclohexyl Se-phenyl phosphoroselenodithioate (6b)



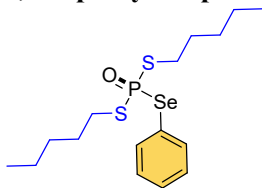
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 5:95); (303mg, 70%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.66 – 7.60 (m, 2H), 7.35 – 7.23 (m, 3H), 3.40 (dd, $J = 10.3$, 3.9 Hz, 2H), 2.07 – 1.96 (m, 4H), 1.68 – 1.60 (m, 4H), 1.51 – 1.41 (m, 5H), 1.33 (m, 4H), 1.20 (dd, $J = 11.7$, 4.5 Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 136.26 (d, $J = 4.1$ Hz), 129.41 (d, $J = 2.4$ Hz), 129.26 (d, $J = 2.9$ Hz), 126.76 (d, $J = 7.7$ Hz), 48.28 (d, $J = 3.8$ Hz), 35.31 (d, $J = 4.3$ Hz), 35.16 (d, $J = 4.7$ Hz), 25.93 (s), 25.30 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 48.27 (s), 46.90 (s), 45.52 (s). HRMS (ESI) m/z : calcd for $\text{C}_{18}\text{H}_{27}\text{OPS}_2\text{Se}$ [$\text{M} + \text{H}$] $^+$, 435.0484; found, 435.0482.

S,S-diphenethyl Se-phenyl phosphoroselenodithioate (6c)



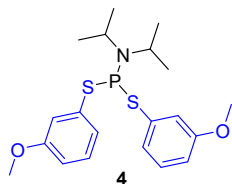
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 5:95); (329mg, 69%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.62 – 7.48 (m, 2H), 7.32 – 7.06 (m, 13H), 3.09 (dt, $J = 14.6$, 7.2 Hz, 4H), 2.90 (t, $J = 7.7$ Hz, 4H). ^{13}C NMR (101 MHz, CDCl_3) δ 139.2 (s), 136.4 (d, $J = 4.1$ Hz), 129.6 (s), 129.6 (s), 129.5 (s), 128.6 (d, $J = 14.6$ Hz), 126.7 (s), 125.9 (d, $J = 7.7$ Hz), 36.8 (d, $J = 4.5$ Hz), 34.4 (d, $J = 3.7$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 51.63 (s), 50.23 (s), 48.83 (s). HRMS (ESI) m/z : calcd for $\text{C}_{22}\text{H}_{23}\text{OPS}_2\text{Se}$ [$\text{M} + \text{H}$] $^+$, 479.0171; found, 479.0167.

S,S-dipentyl Se-phenyl phosphoroselenodithioate (6d)



^1H NMR (400 MHz, CDCl_3) δ 7.68 – 7.59 (m, 2H), 7.37 – 7.24 (m, 2H), 7.25 – 7.15 (m, 1H), 2.90 (m, 4H), 1.65 (dt, $J = 14.9, 7.4$ Hz, 4H), 1.36 – 1.21 (m, 8H), 0.82 (t, $J = 6.2$ Hz, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 136.3 (d, $J = 4.1$ Hz), 131.5 (s), 129.7 – 129.3 (m), 129.20(s), 127.7 (s), 126.1 (d, $J = 7.7$ Hz), 33.3 (d, $J = 3.9$ Hz), 30.7 (s), 30.1 (d, $J = 5.1$ Hz), 22.1 (s), 13.9(s). ^{31}P NMR (162 MHz, CDCl_3) δ 52.84 – 45.44 (m). HRMS (ESI) m/z : calcd for $\text{C}_{16}\text{H}_{27}\text{OPS}_2\text{Se}$ $[\text{M} + \text{H}]^+$, 410.0468; found, 410.0469.

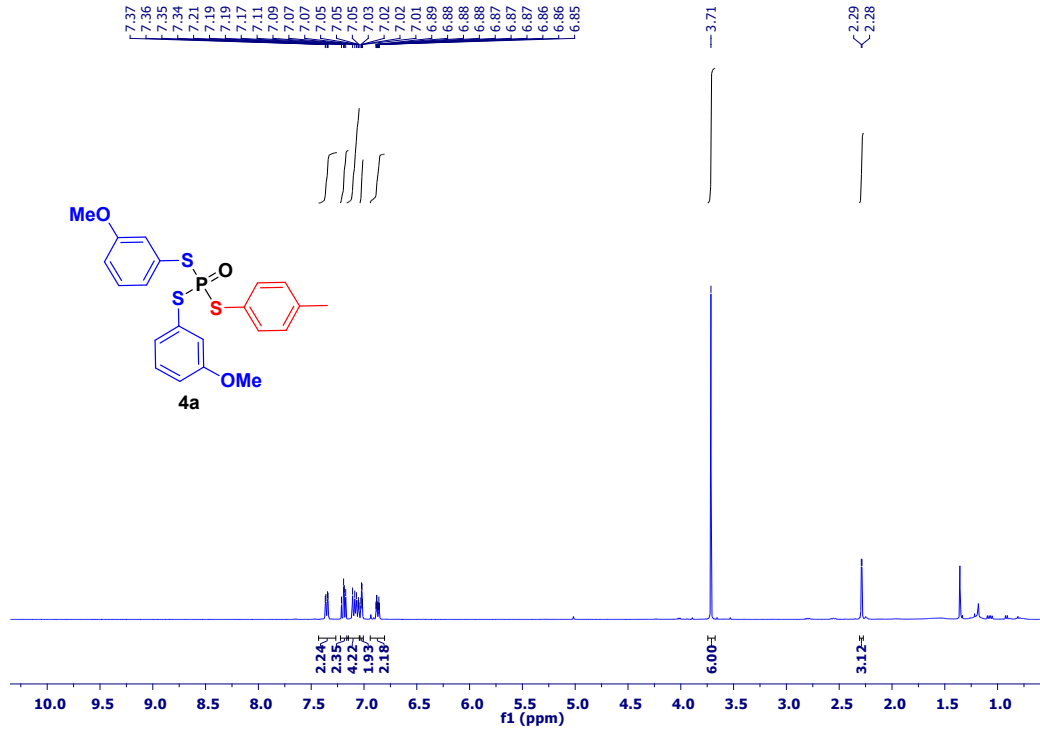
bis(3-methoxyphenyl) diisopropylphosphoramidodithioite



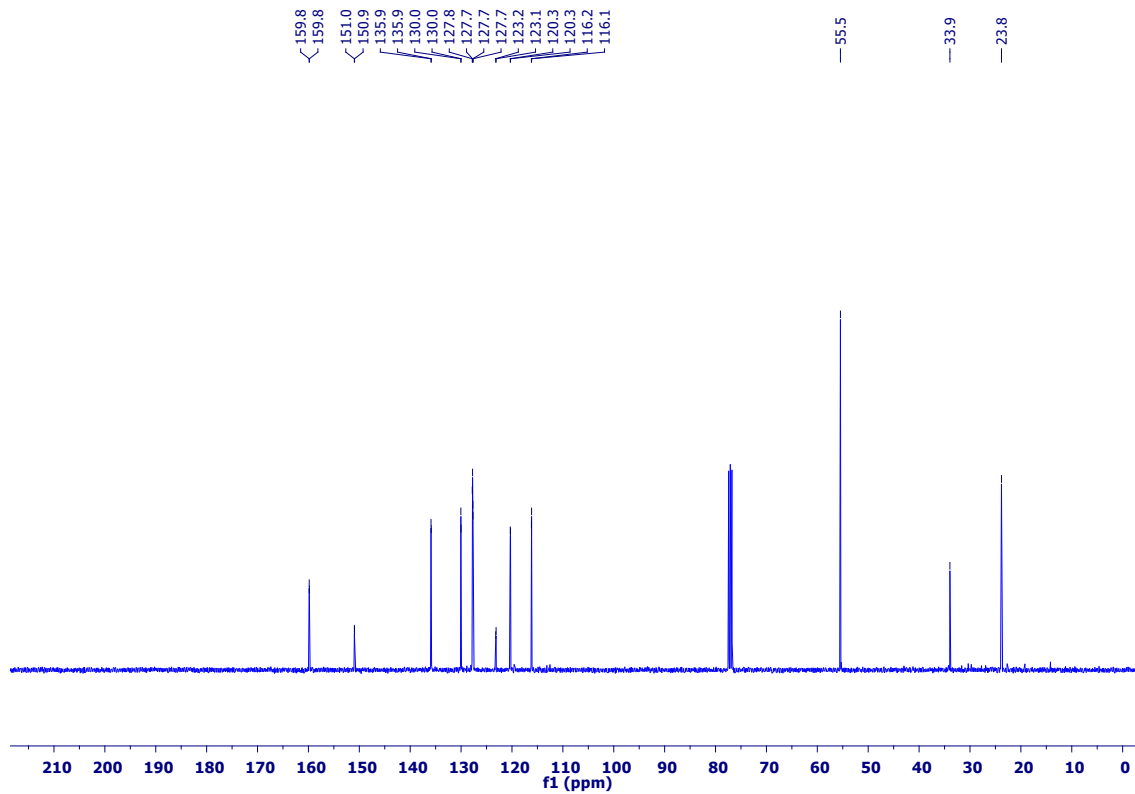
The title compound was purified by column chromatography with the eluent (EtOAc/hexane = 12:88); (333mg, 73%) as a colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 7.10 (d, $J = 8.0$ Hz, 2H), 7.07 – 6.98 (m, 4H), 6.71 (m, 2H), 3.72 (s, 6H), 3.68 – 3.54 (m, 2H), 0.91 (d, $J = 6.7$ Hz, 12H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.6 (s), 135.3 (d, $J = 12.7$ Hz), 129.5 (s), 126.2 (d, $J = 5.2$ Hz), 118.9 (d, $J = 4.9$ Hz), 113.5 (d, $J = 2.3$ Hz), 55.3 (s), 29.7 (s), 23.9 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 122.86 (s). HRMS (ESI) m/z : calcd for $\text{C}_{20}\text{H}_{28}\text{NO}_2\text{PS}_2$ $[\text{M} + \text{H}]^+$, 410.1332; found, 410.1336.

NMR Spectra

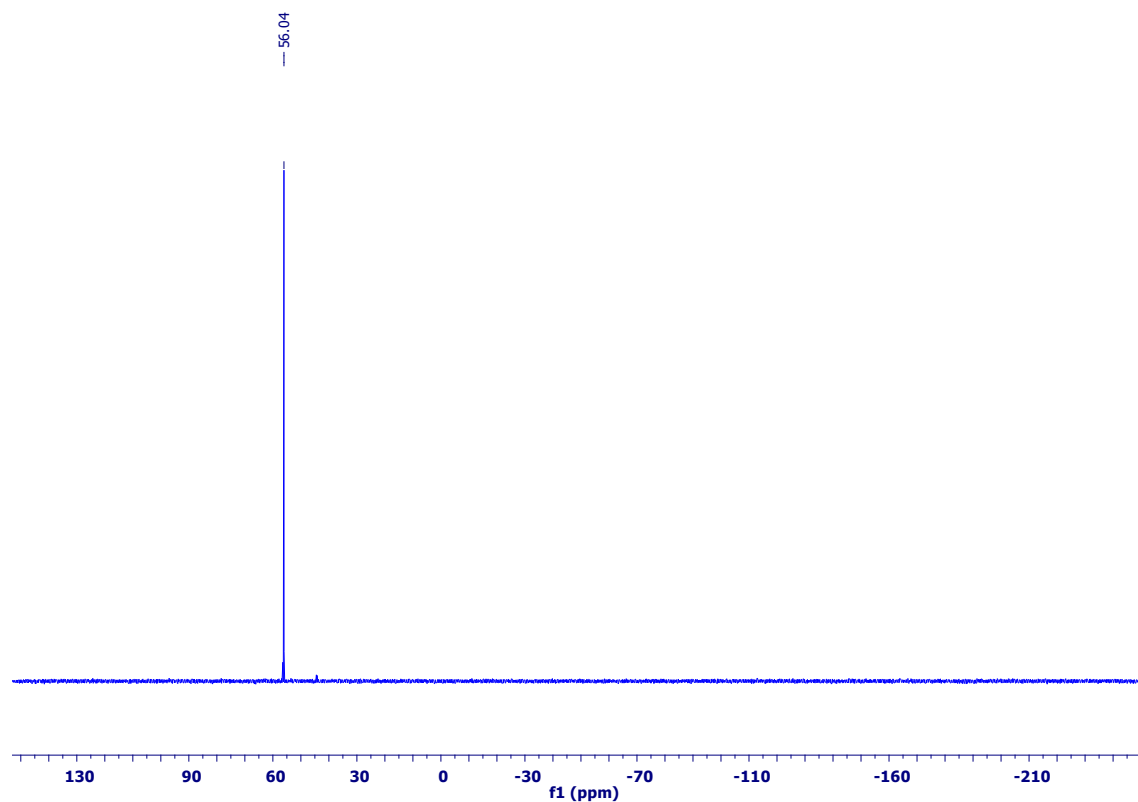
^1H NMR (400 MHz, CDCl_3)



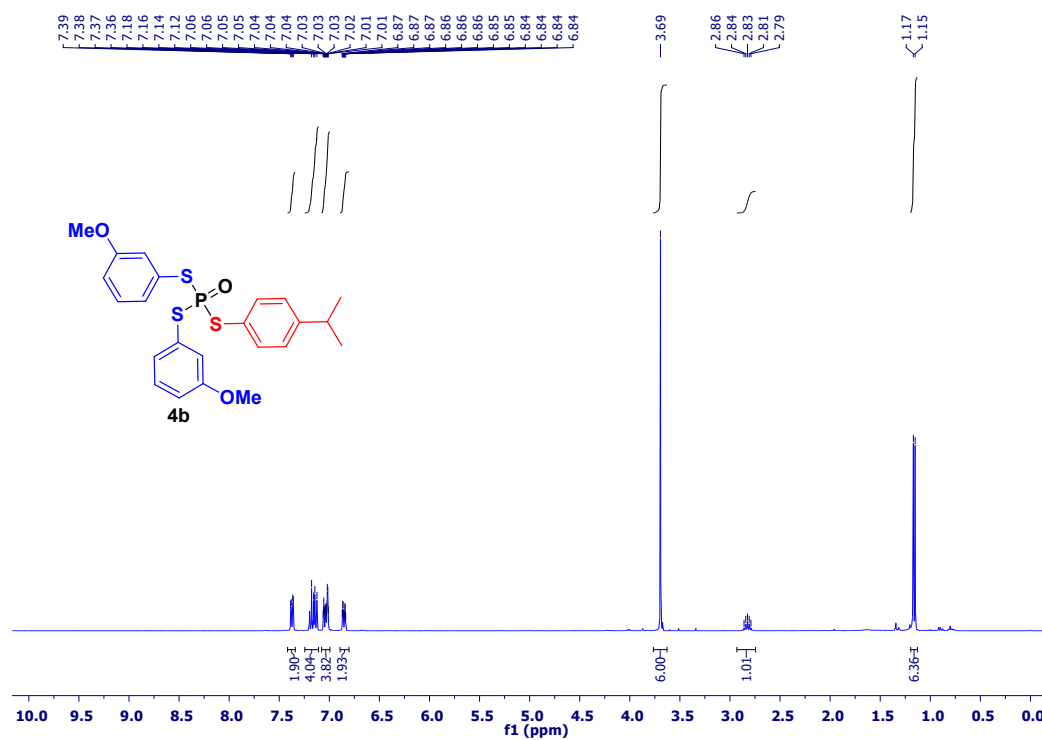
^{13}C NMR (101 MHz, CDCl_3)



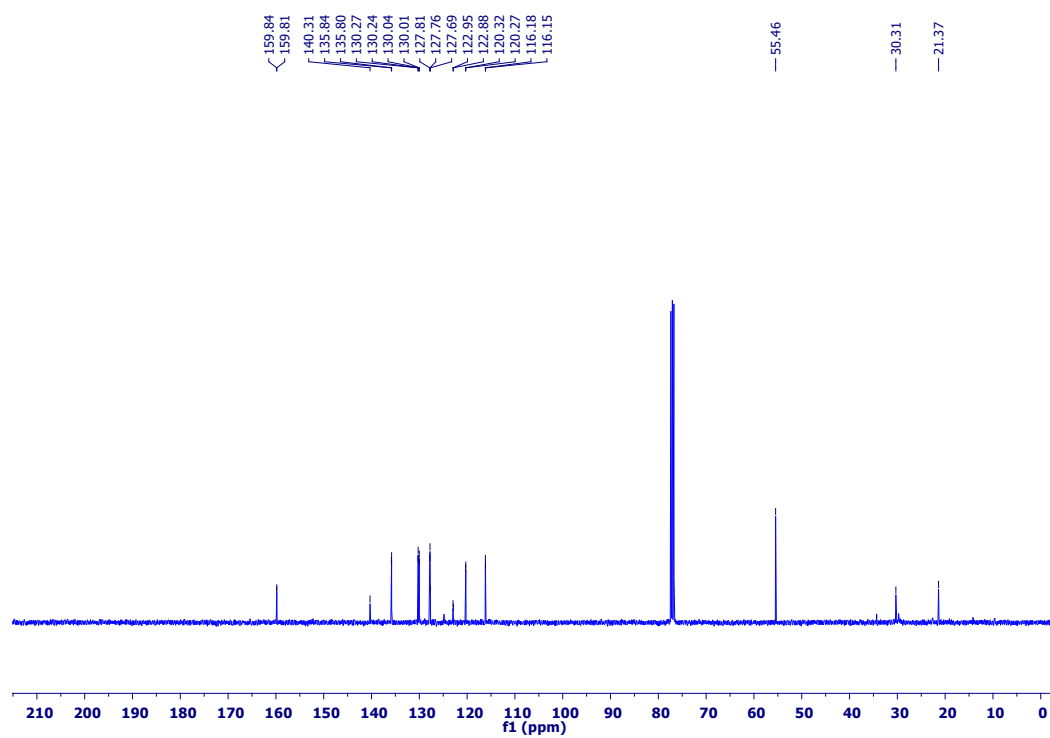
^{31}P NMR (162 MHz, CDCl_3)



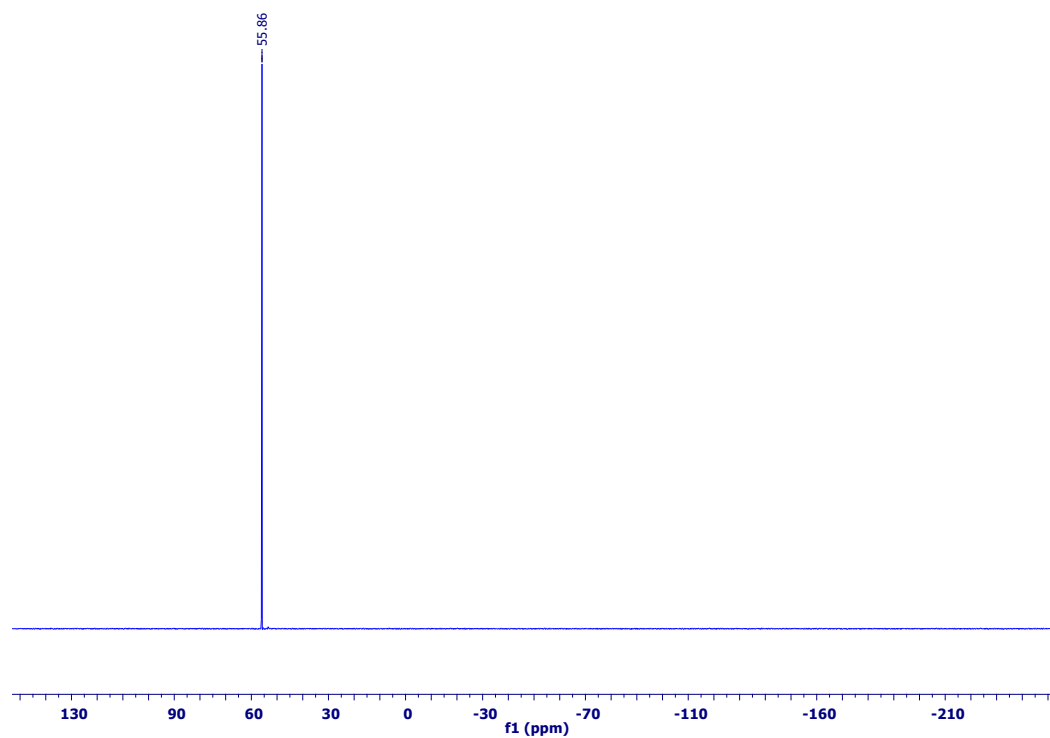
^1H NMR (400 MHz, CDCl_3)



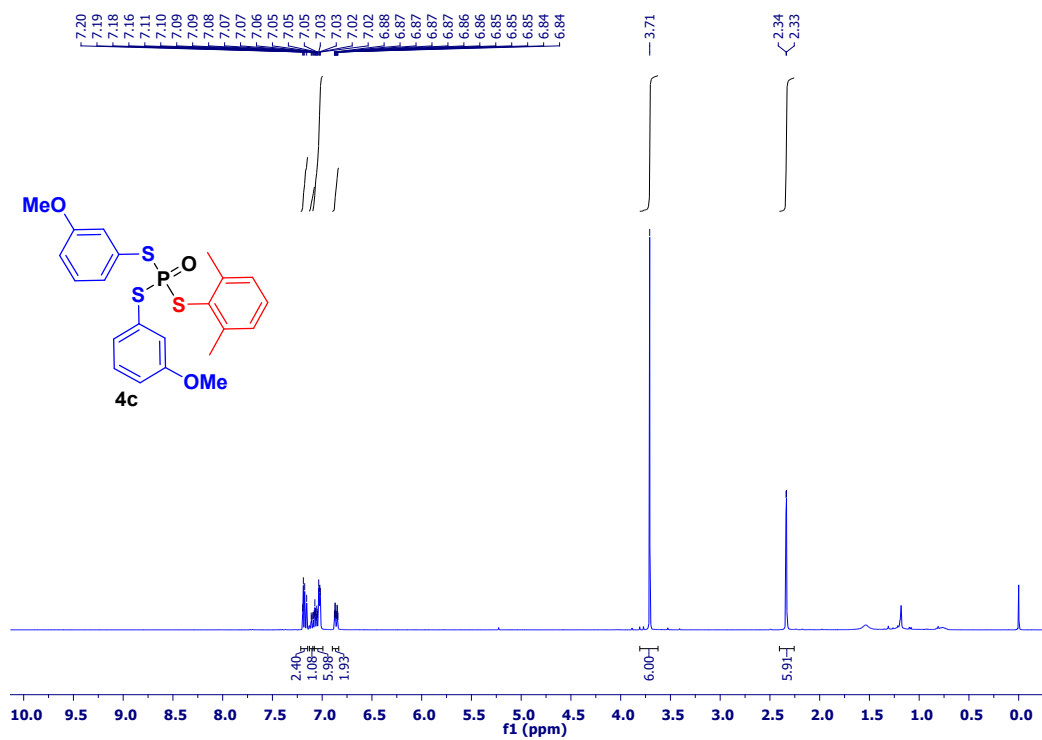
^{13}C NMR (101 MHz, CDCl_3)



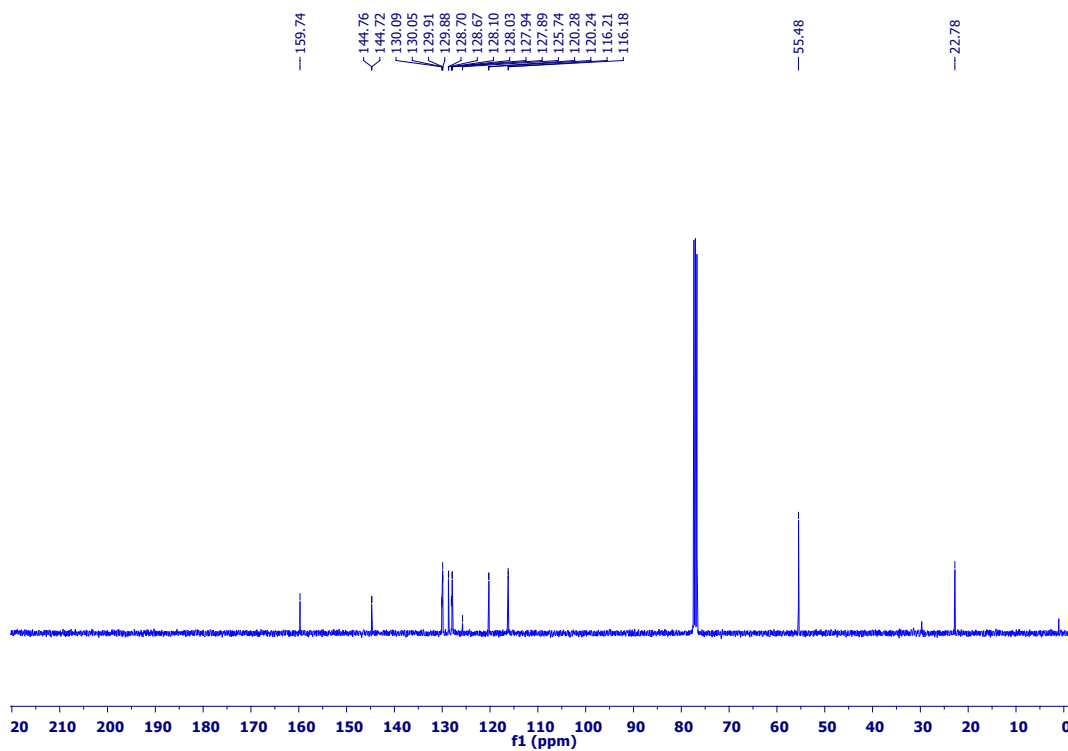
^{31}P NMR (162 MHz, CDCl_3)



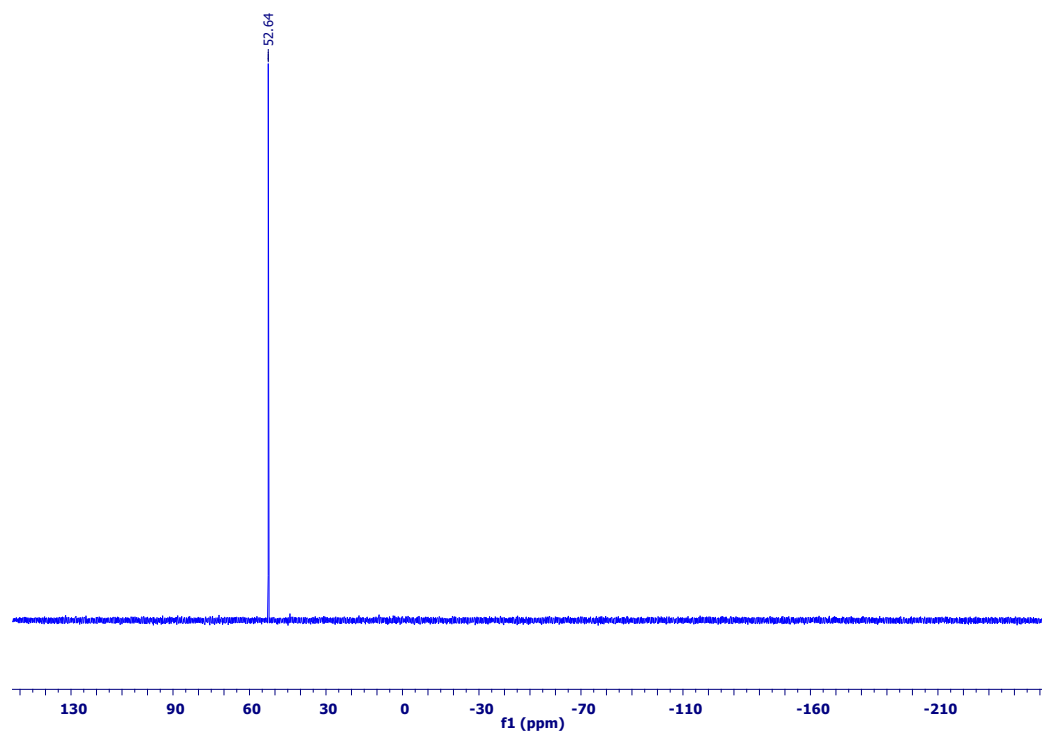
¹H NMR (400 MHz, CDCl₃)



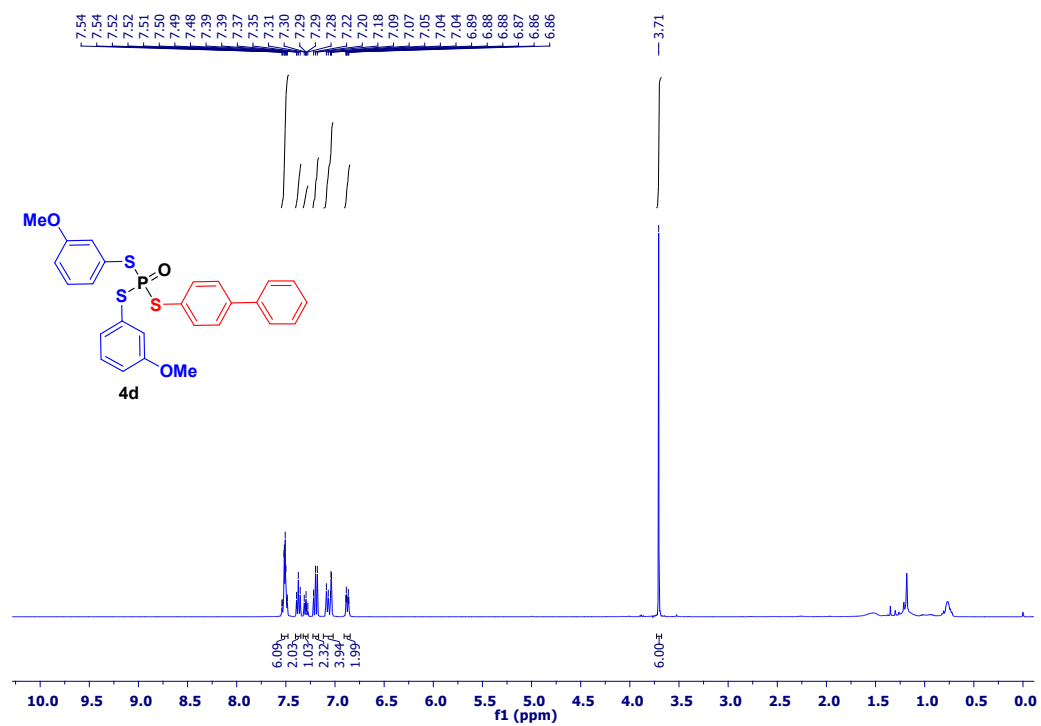
¹³C NMR (101 MHz, CDCl₃)



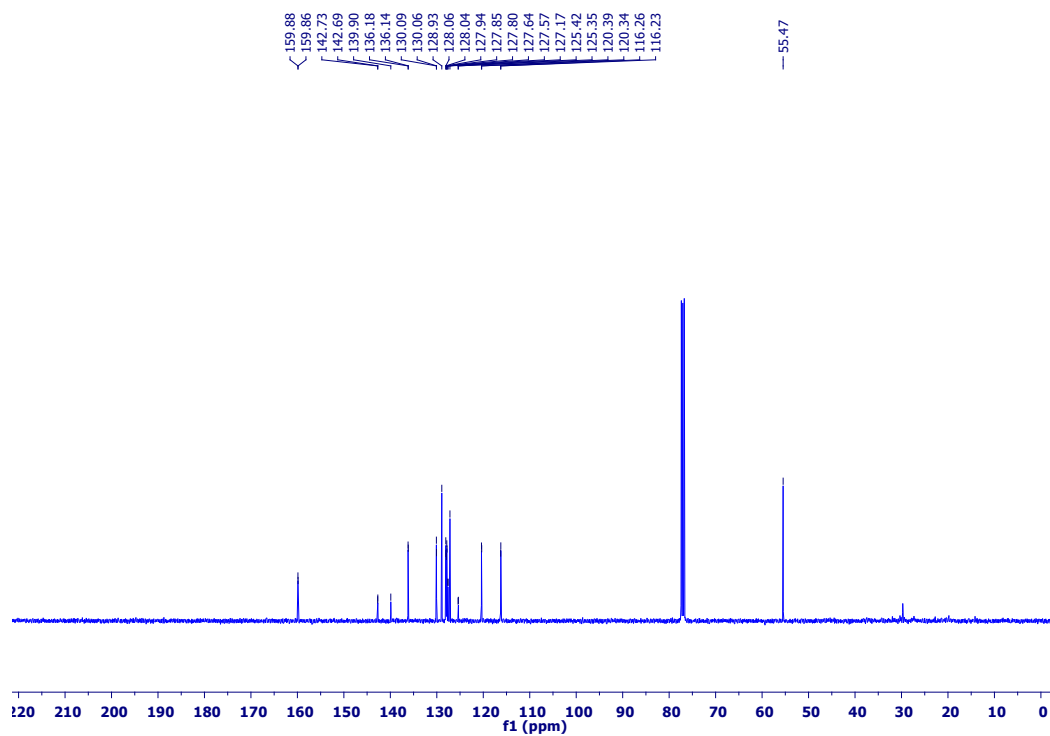
^{31}P NMR (162 MHz, CDCl_3)



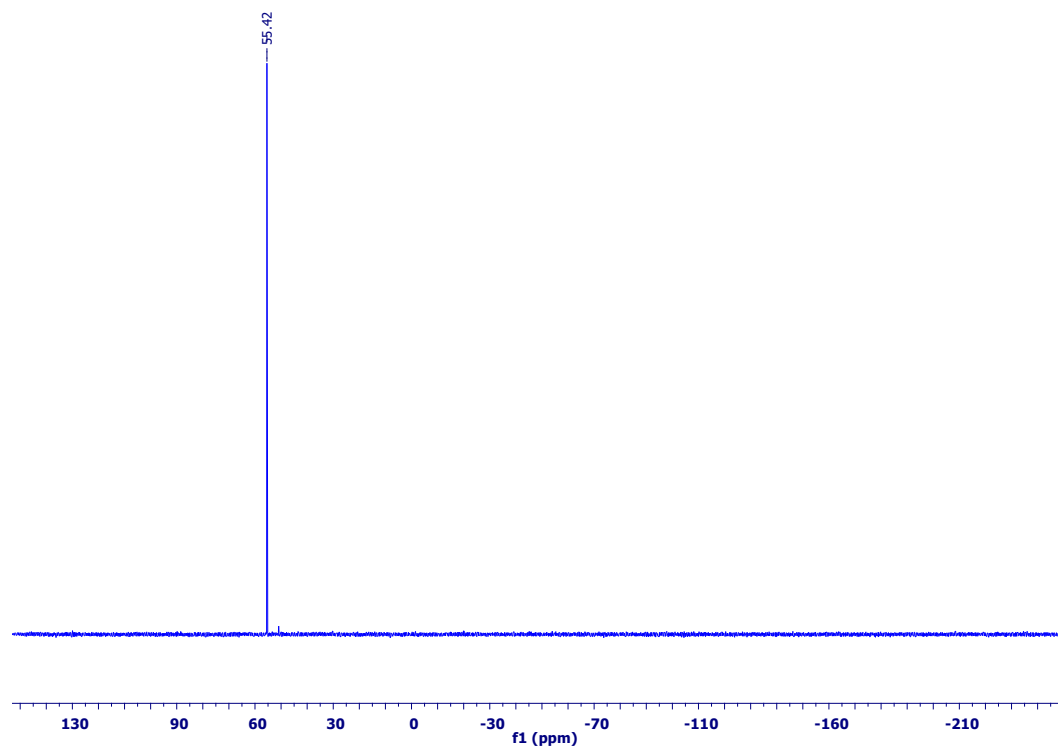
^1H NMR (400 MHz, CDCl_3)



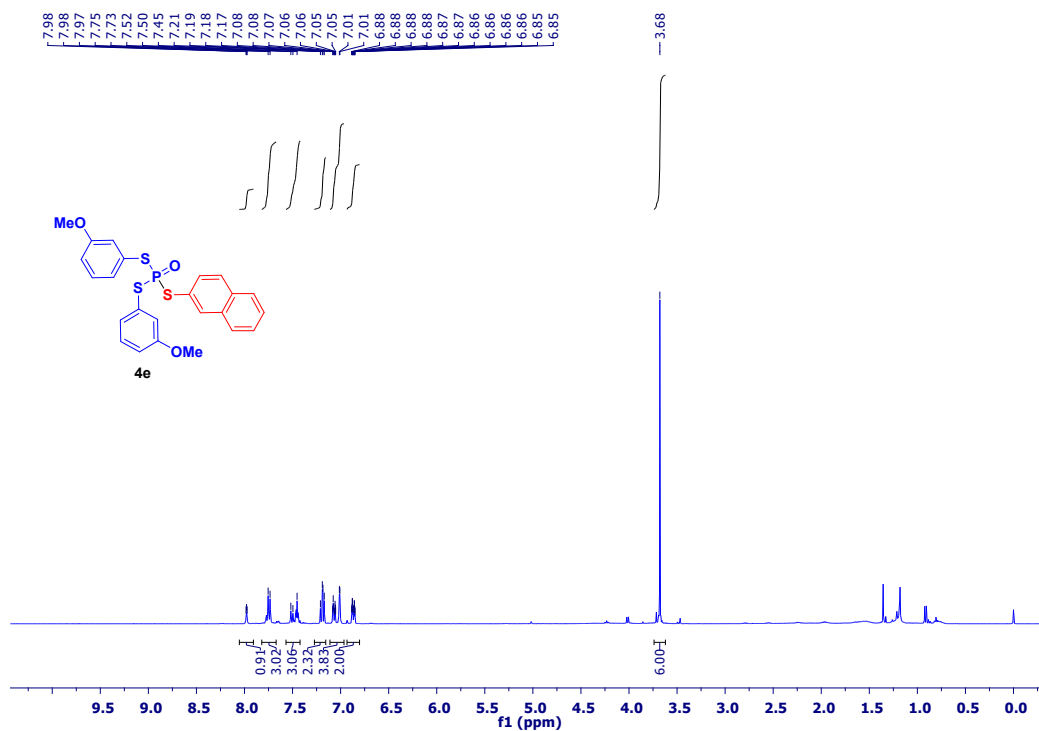
^{13}C NMR (101 MHz, CDCl_3)



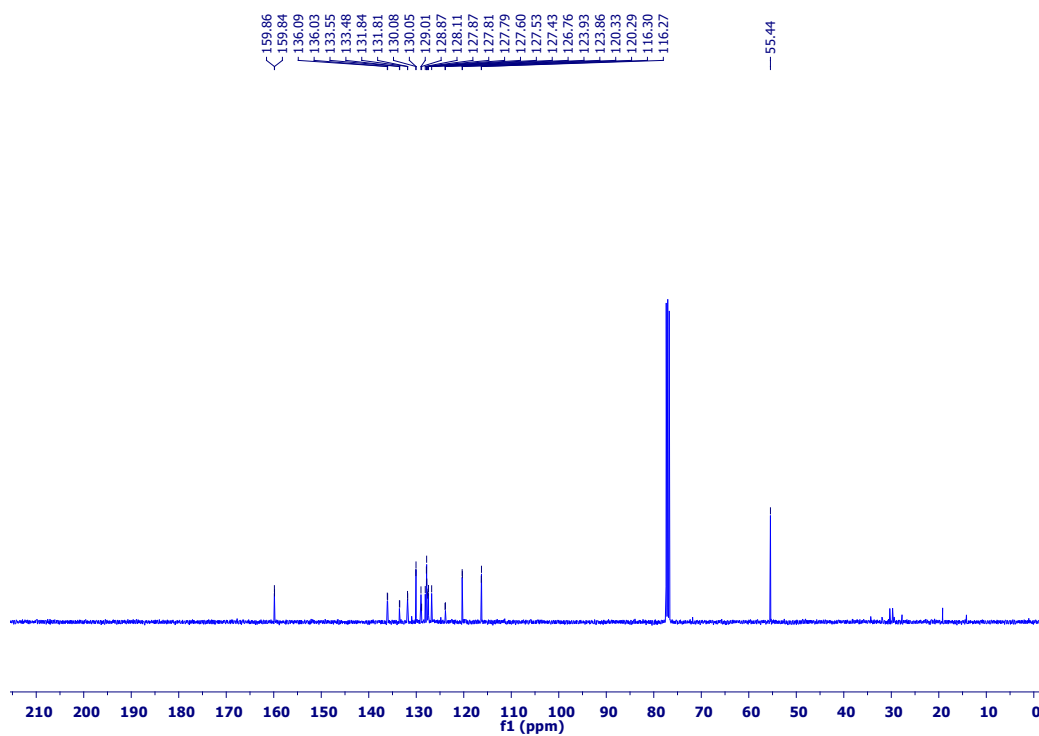
^{31}P NMR (162 MHz, CDCl_3)



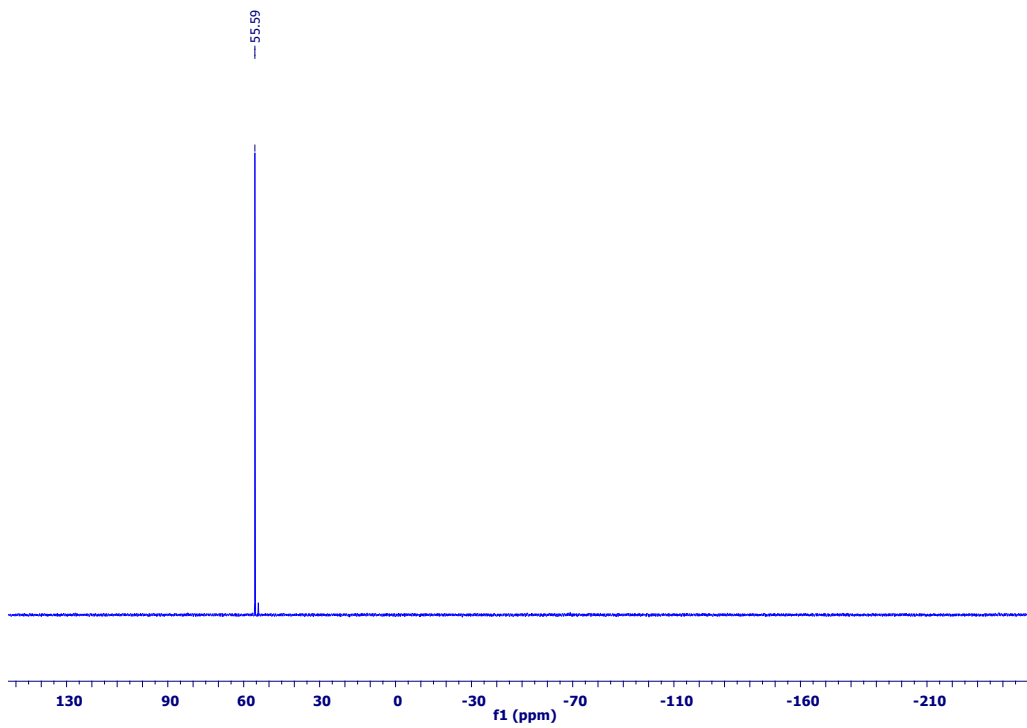
¹H NMR (400 MHz, CDCl₃)



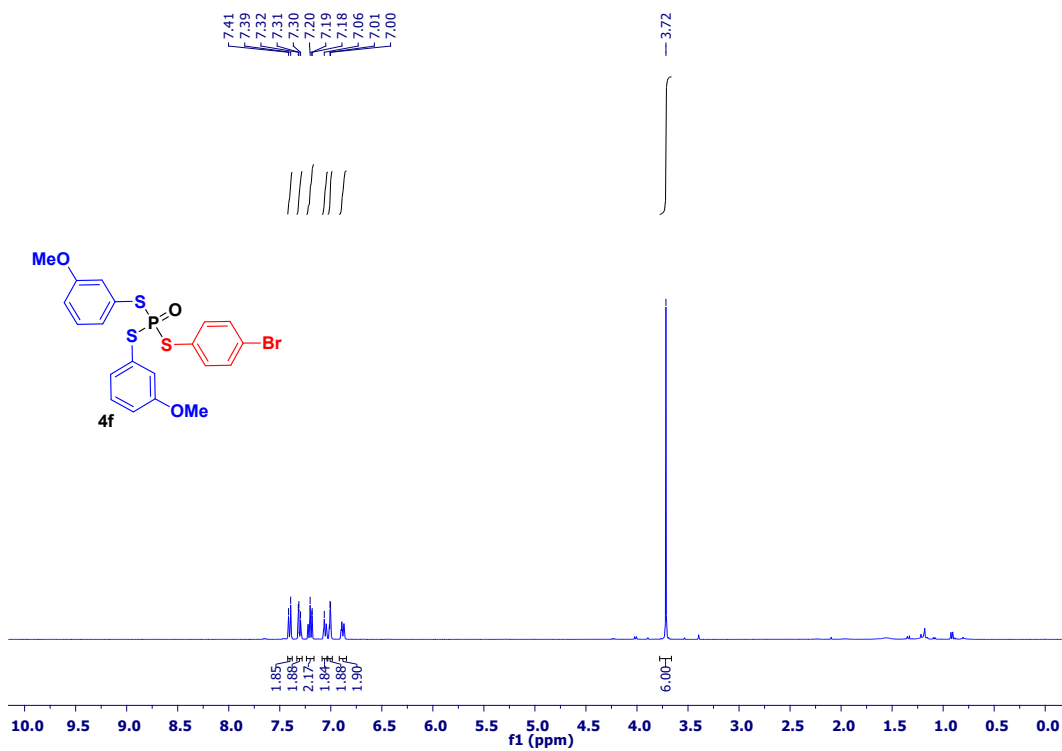
¹³C NMR (101 MHz, CDCl₃)



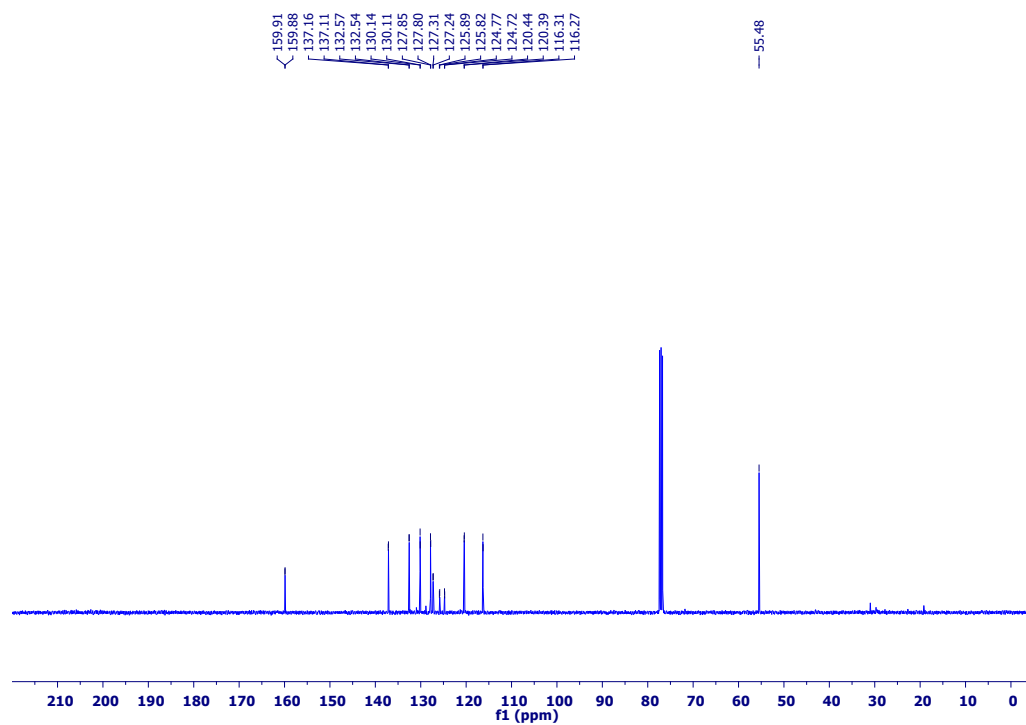
^{31}P NMR (162 MHz, CDCl_3)



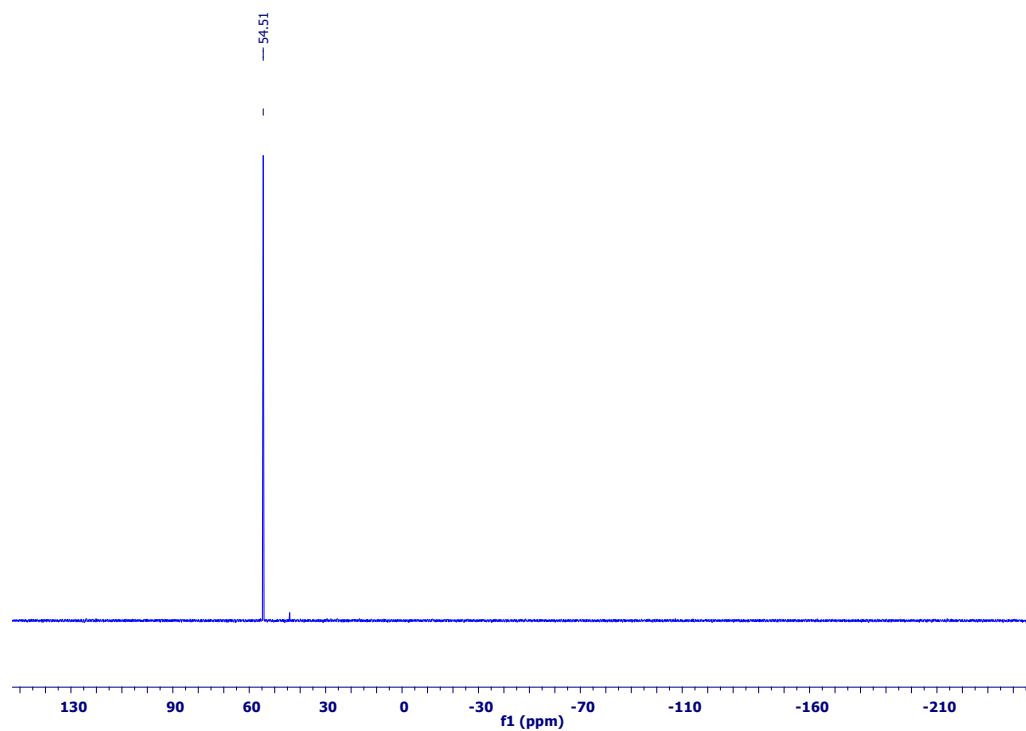
^1H NMR (400 MHz, CDCl_3)



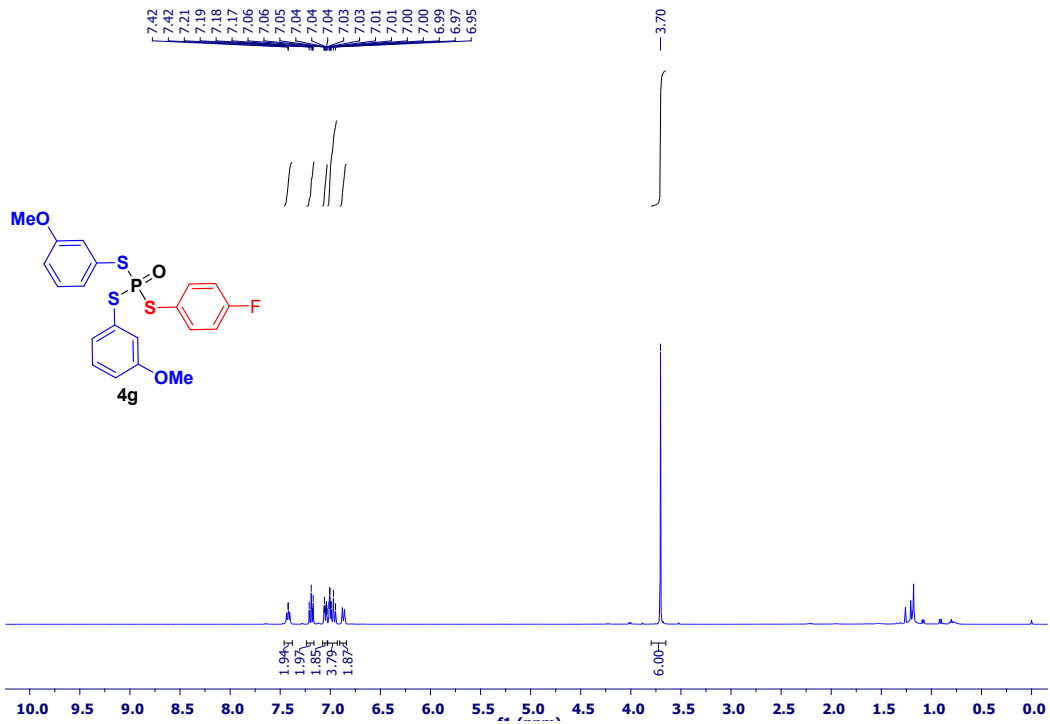
^{13}C NMR (101 MHz, CDCl_3)



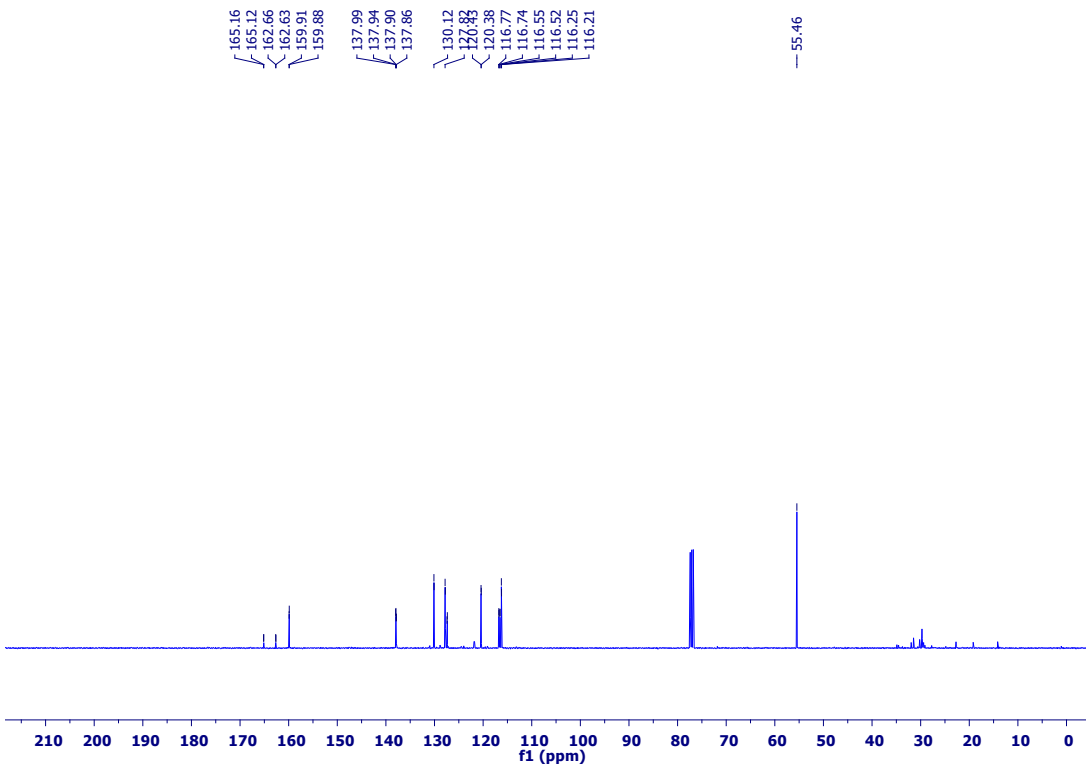
^{31}P NMR (162 MHz, CDCl_3)



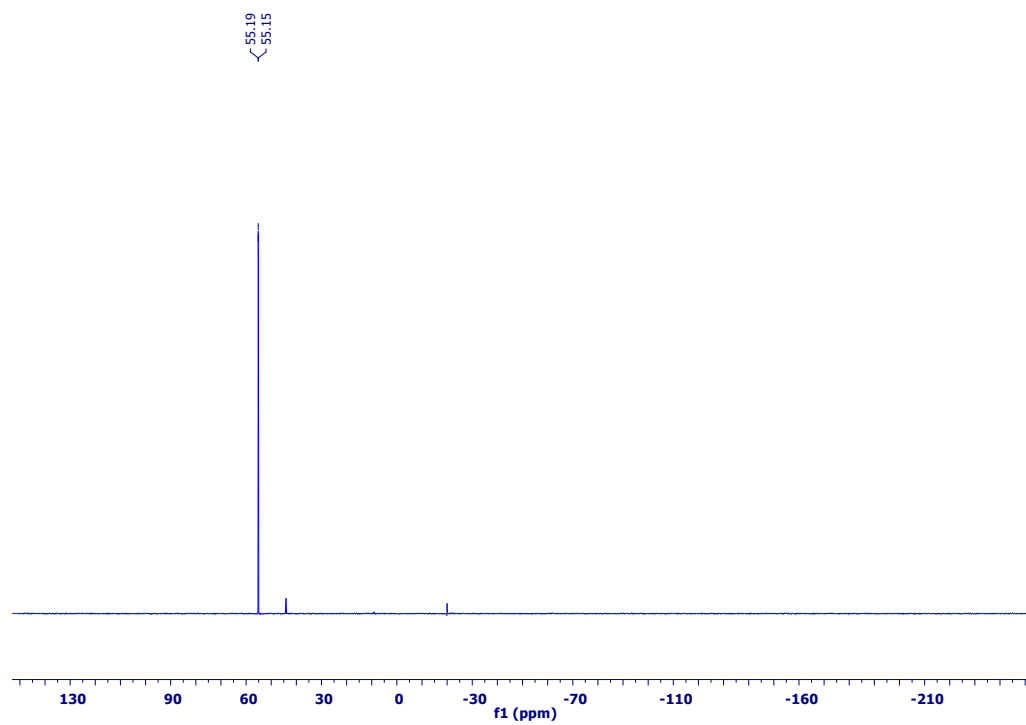
^1H NMR (400 MHz, CDCl_3)



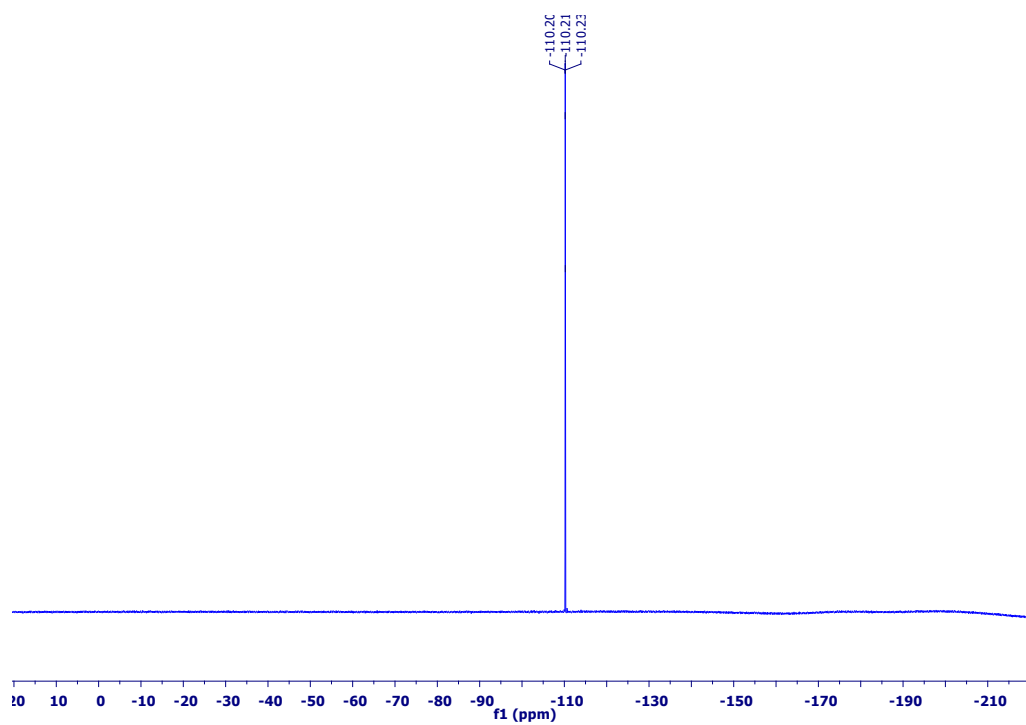
^{13}C NMR (101 MHz, CDCl_3)



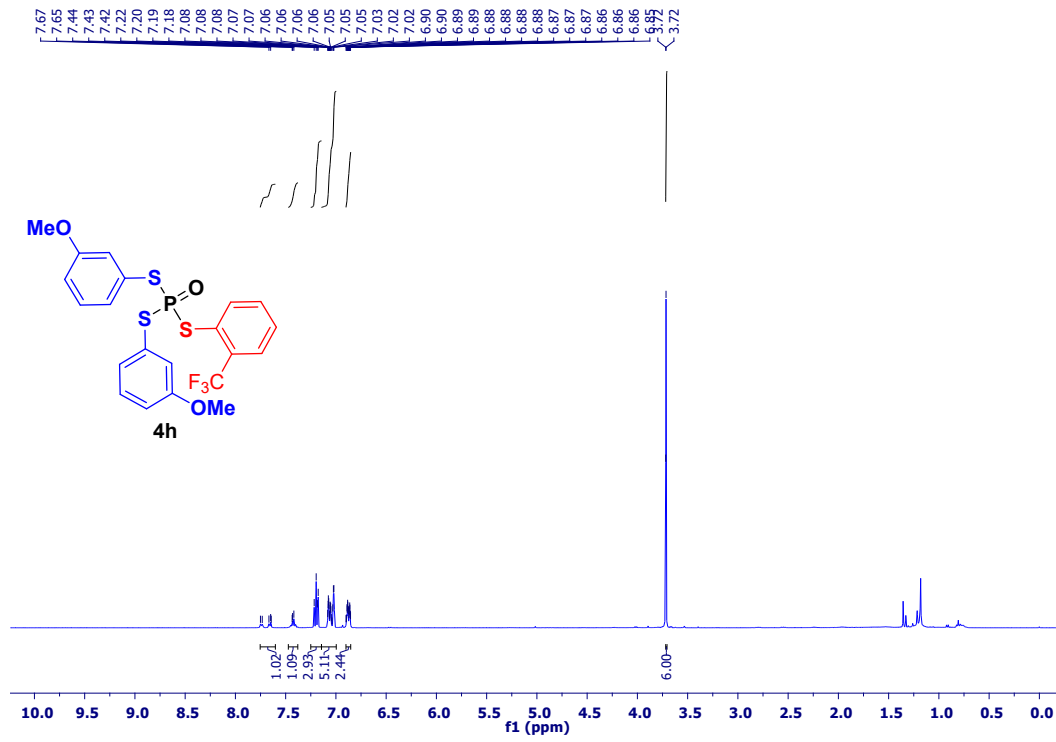
^{31}P NMR (162 MHz, CDCl_3)



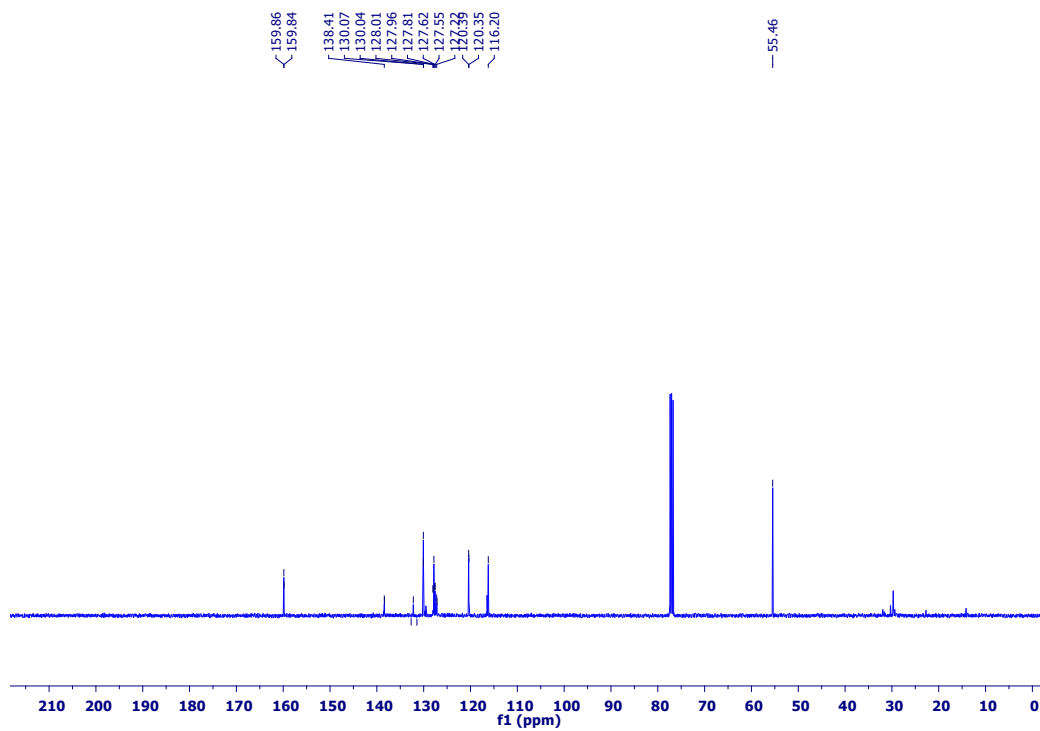
^{19}F NMR (377 MHz, CDCl_3)



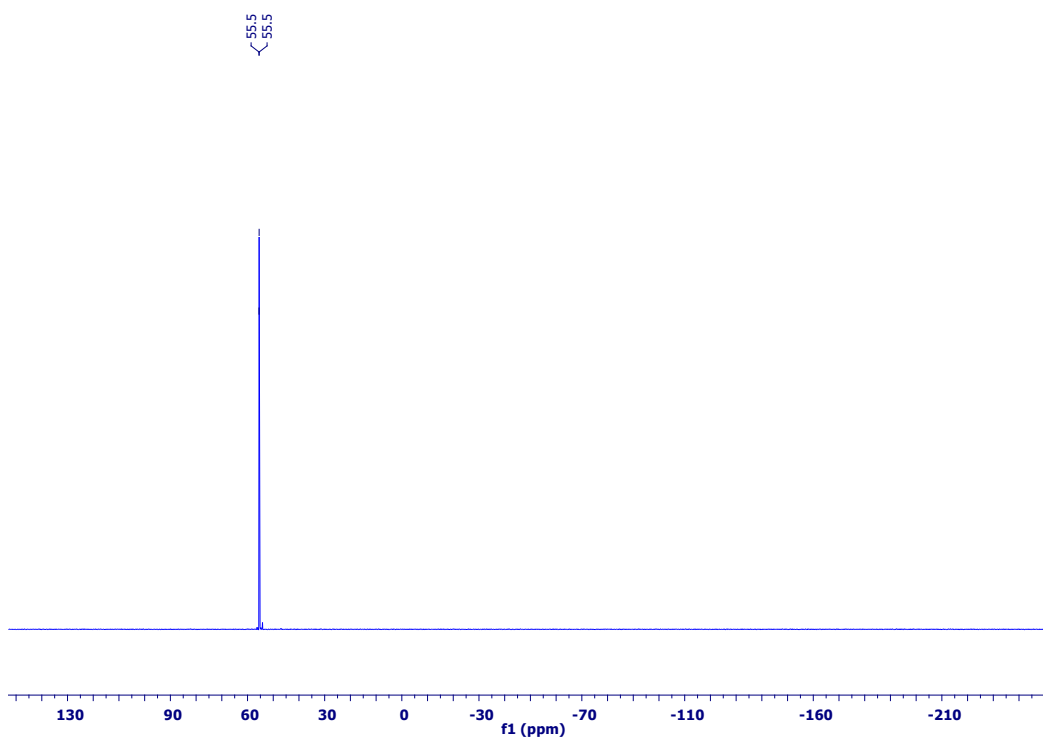
¹H NMR (400 MHz, CDCl₃)



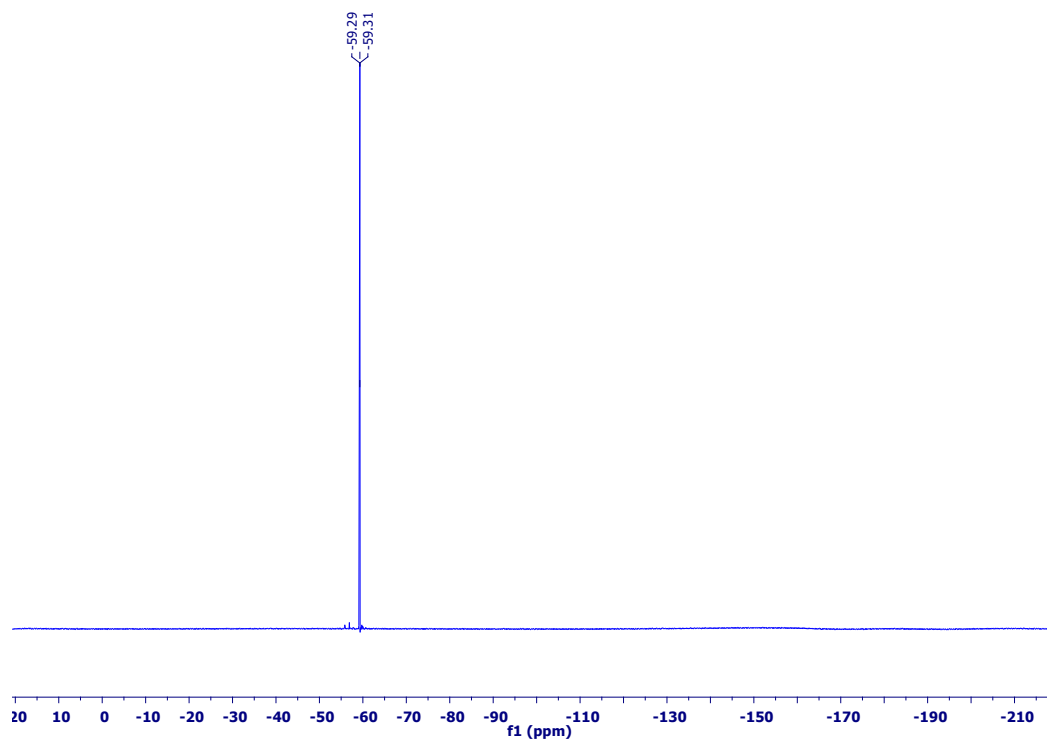
¹³C NMR (101 MHz, CDCl₃)



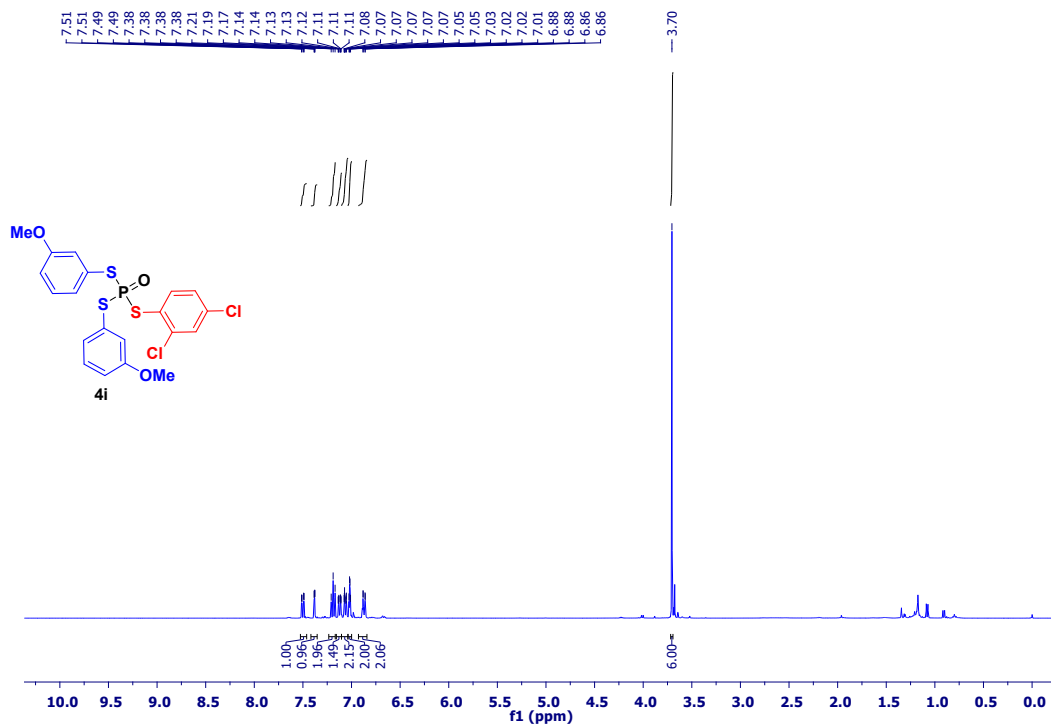
^{31}P NMR (162 MHz, CDCl_3)



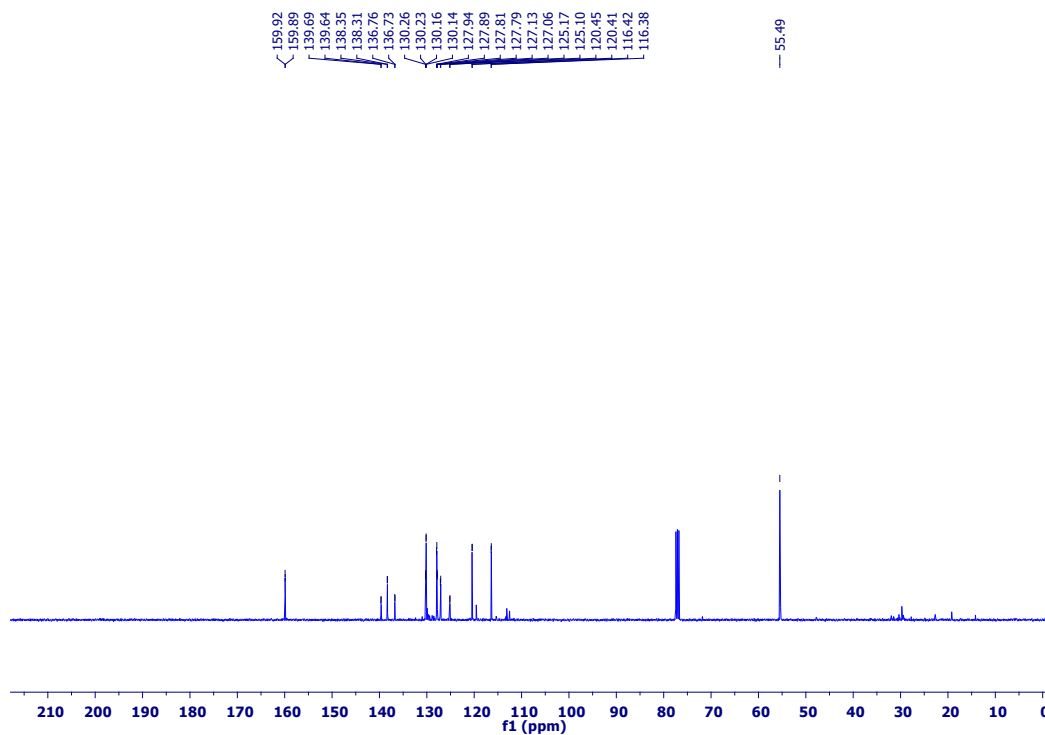
^{19}F NMR (377 MHz, CDCl_3)



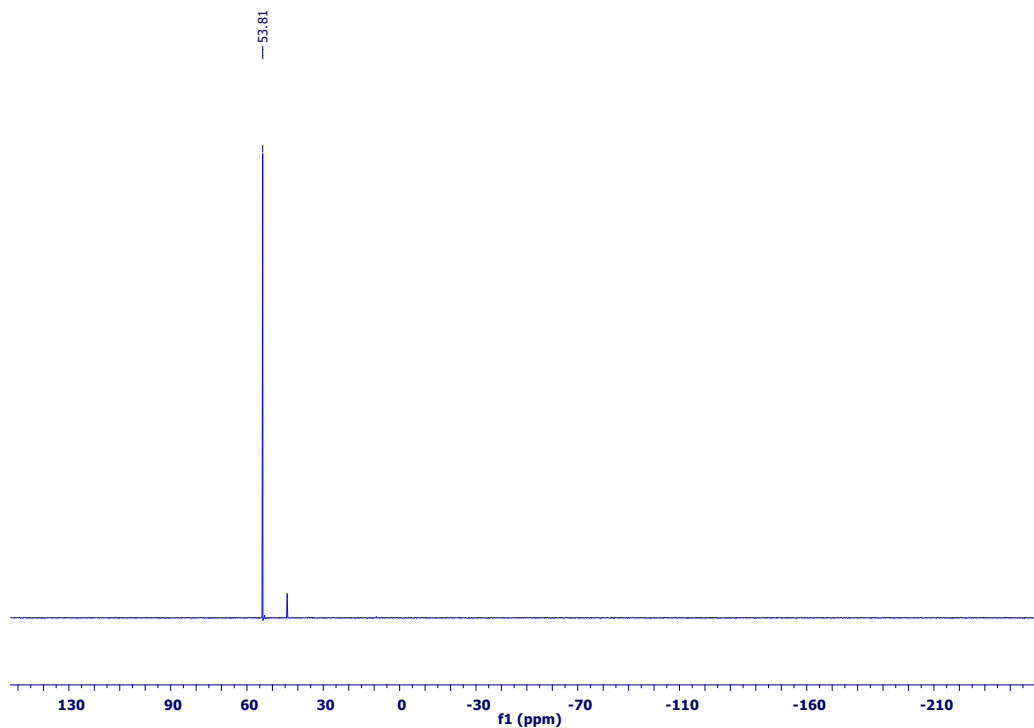
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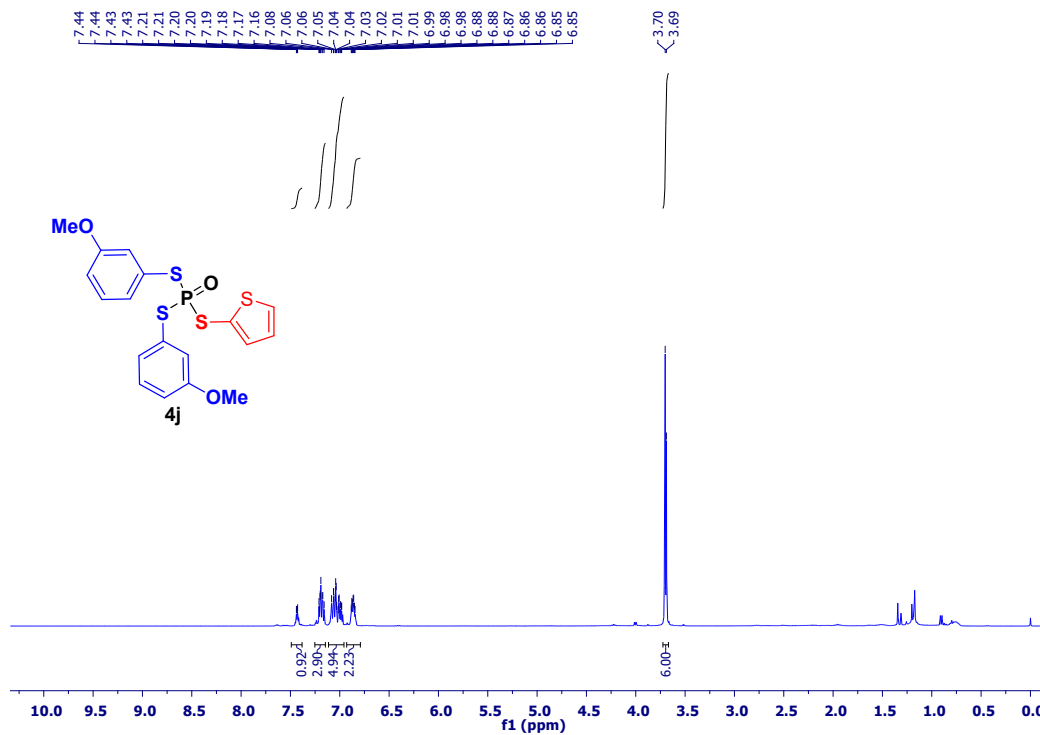
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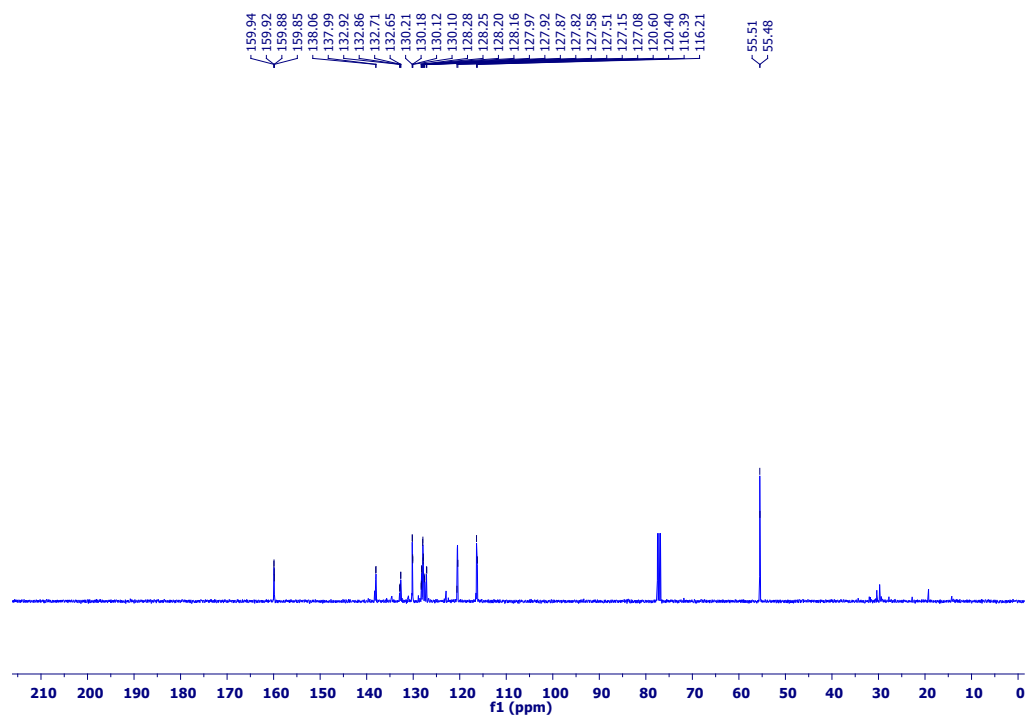
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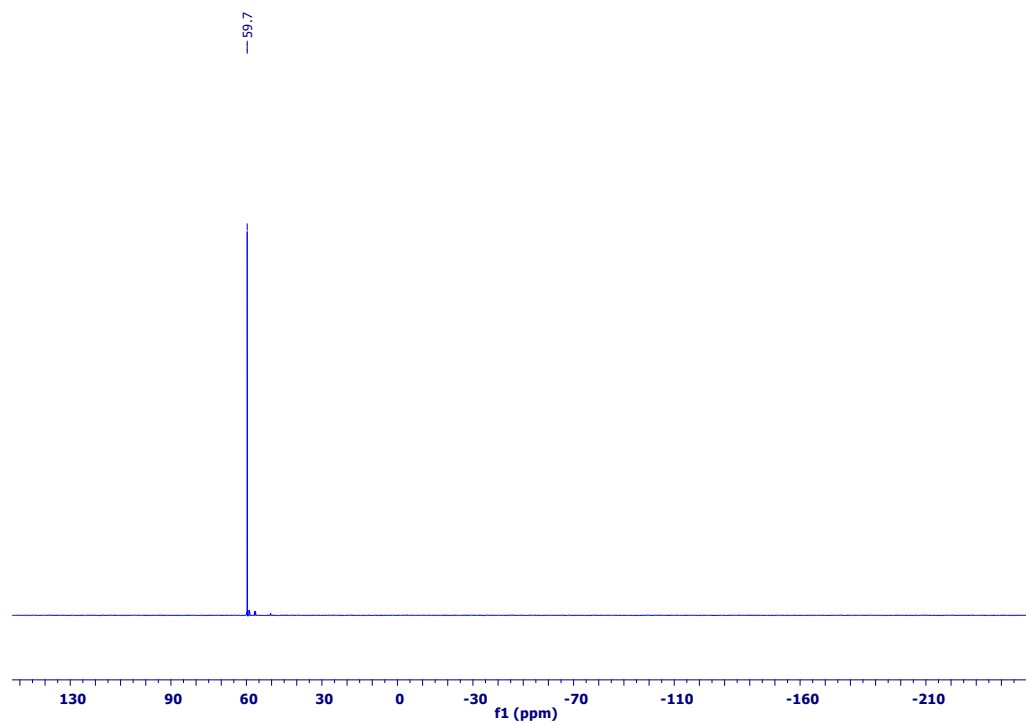
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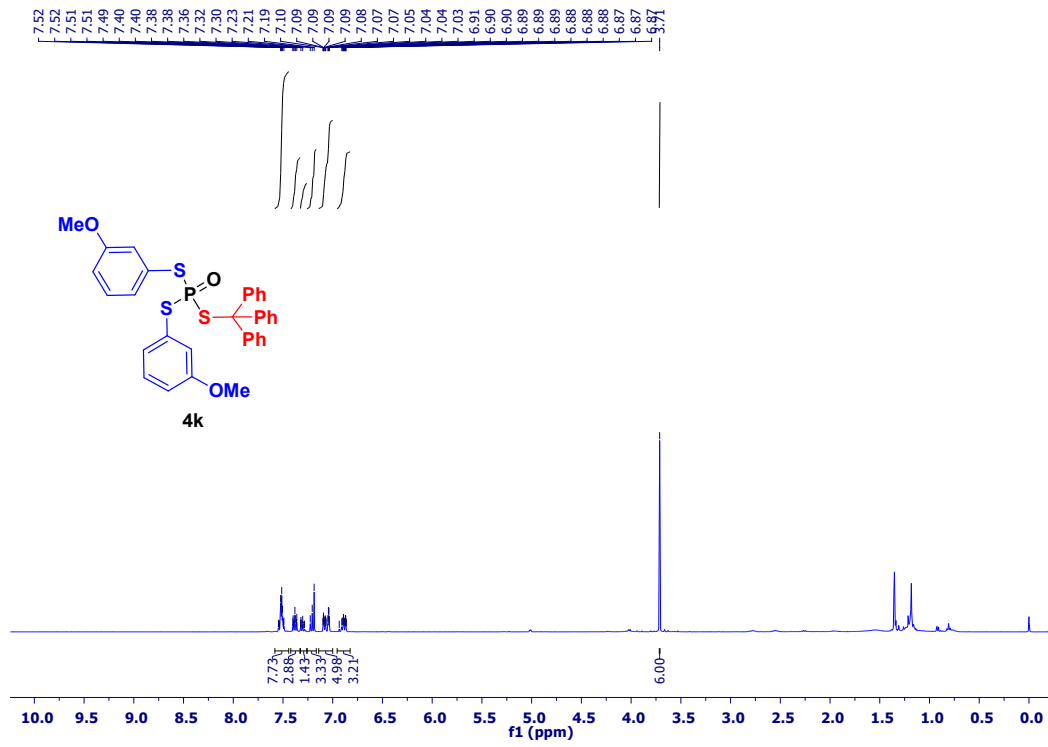
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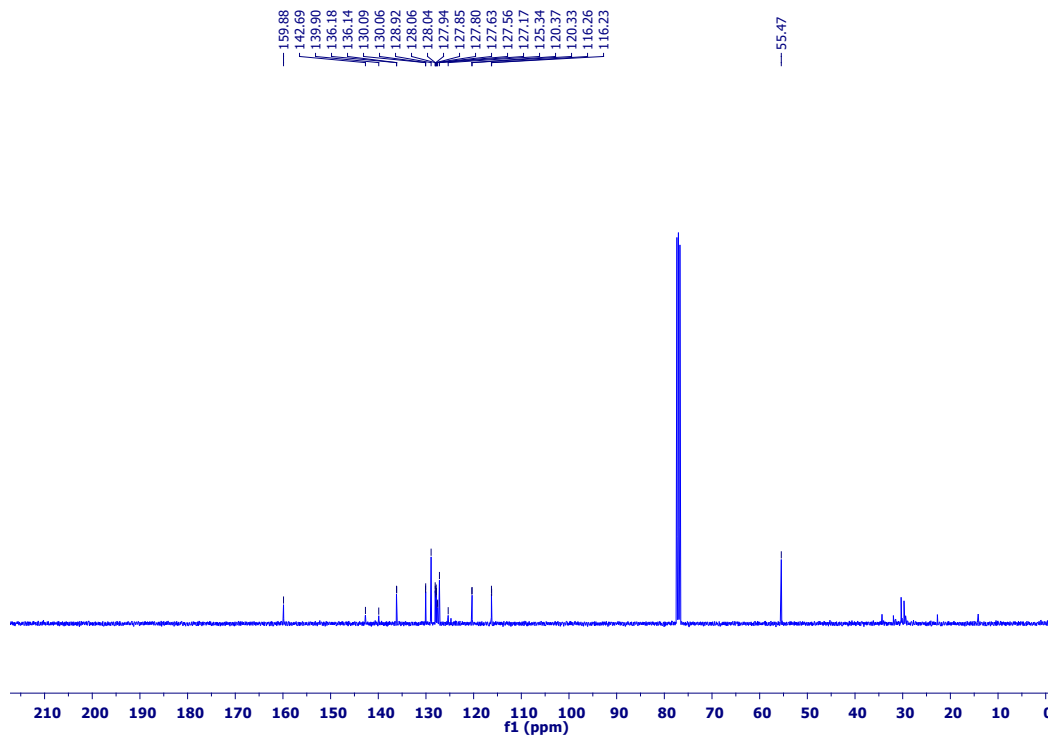
^{31}P NMR (162 MHz, CDCl_3)



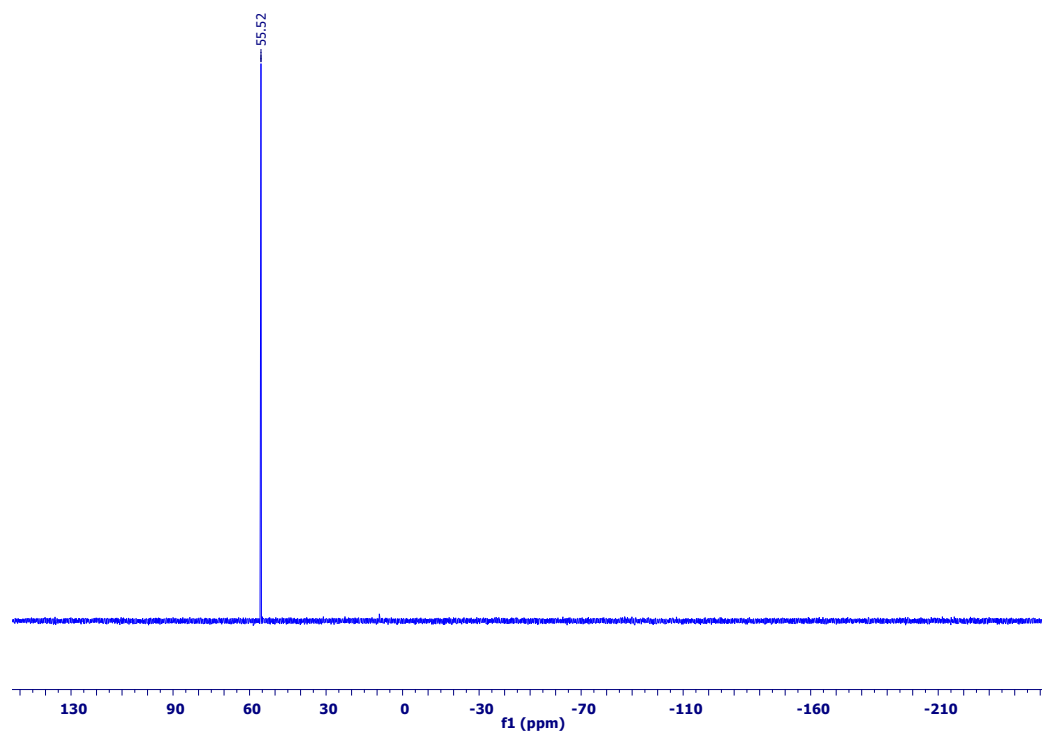
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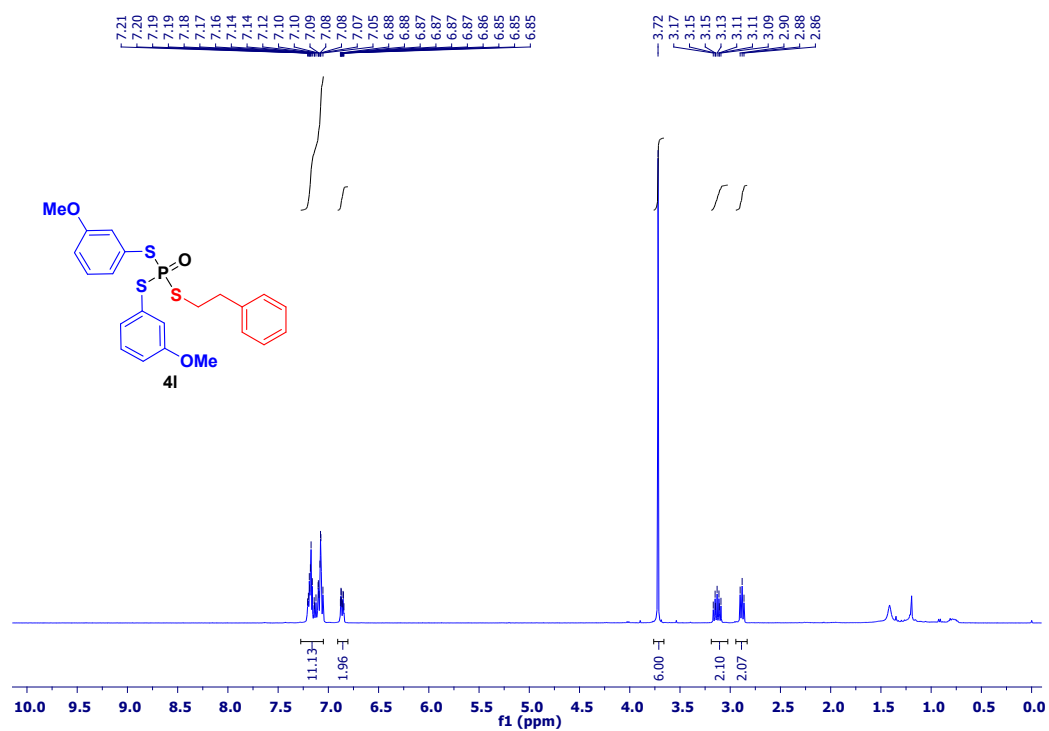
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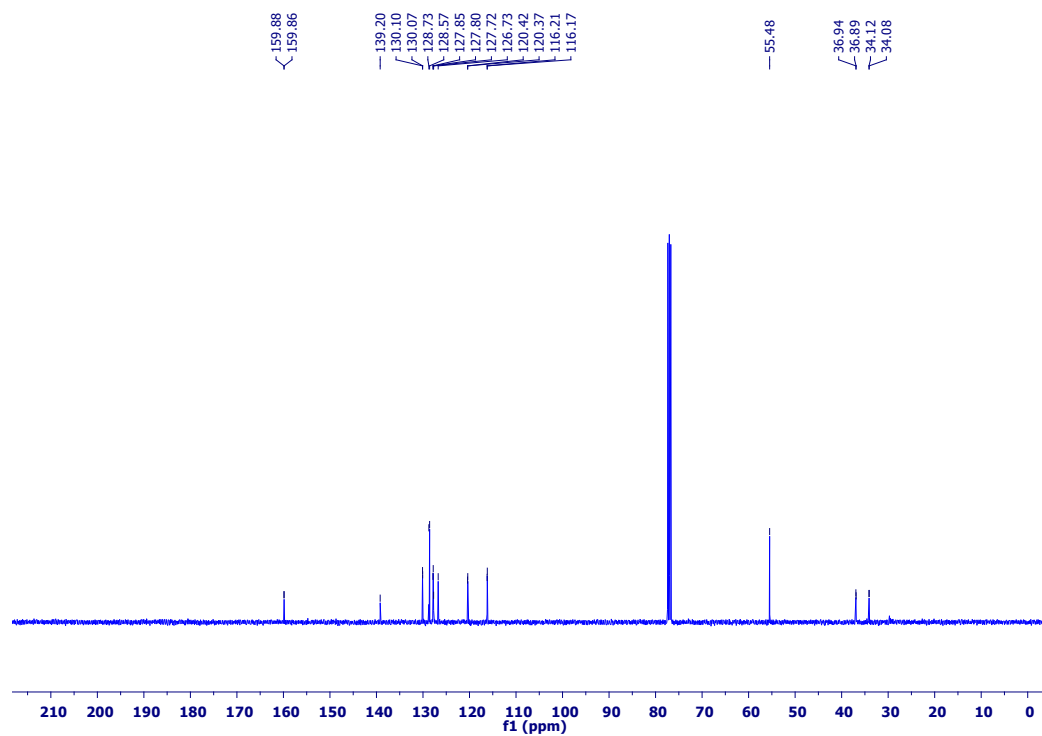
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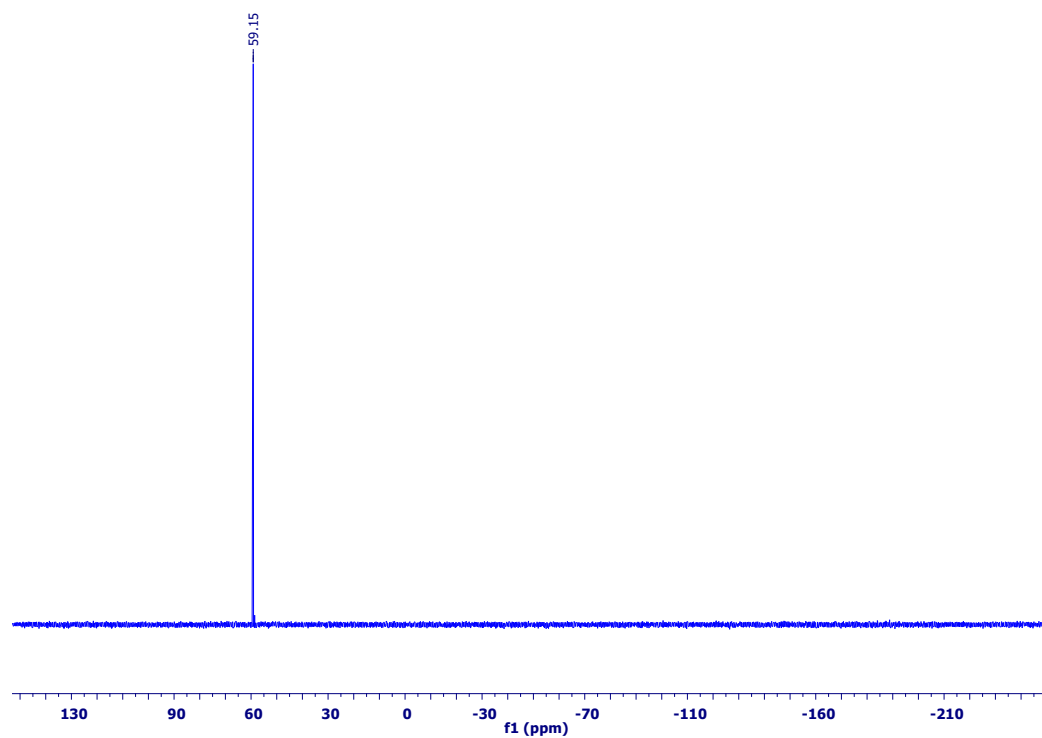
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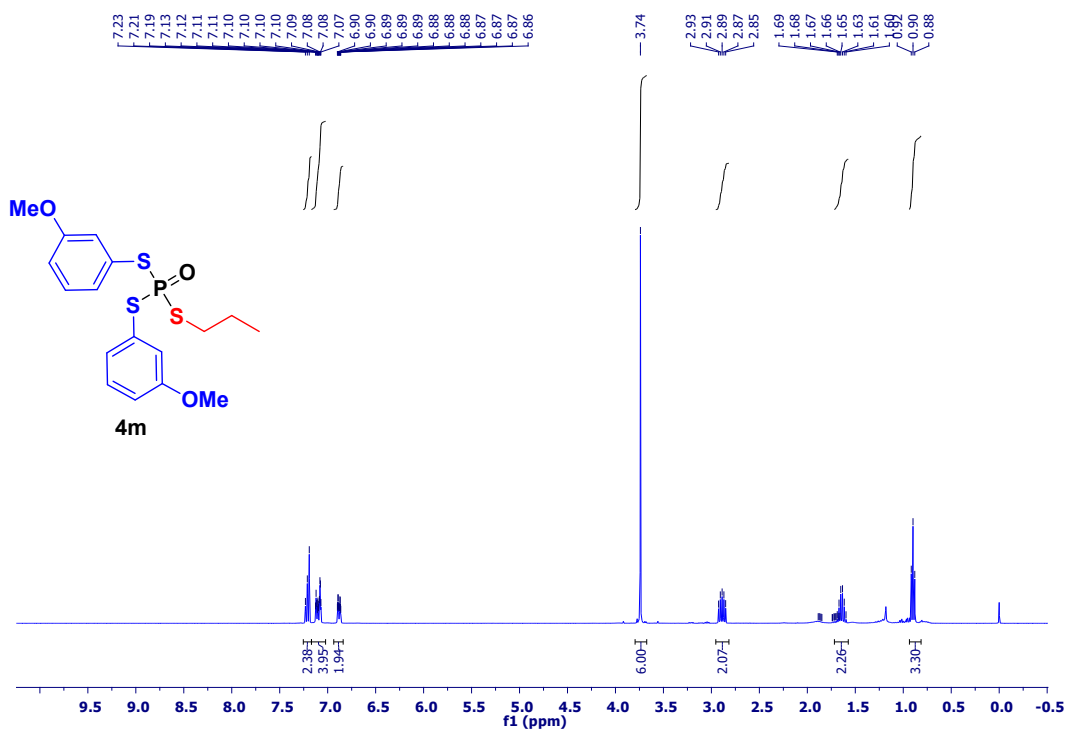
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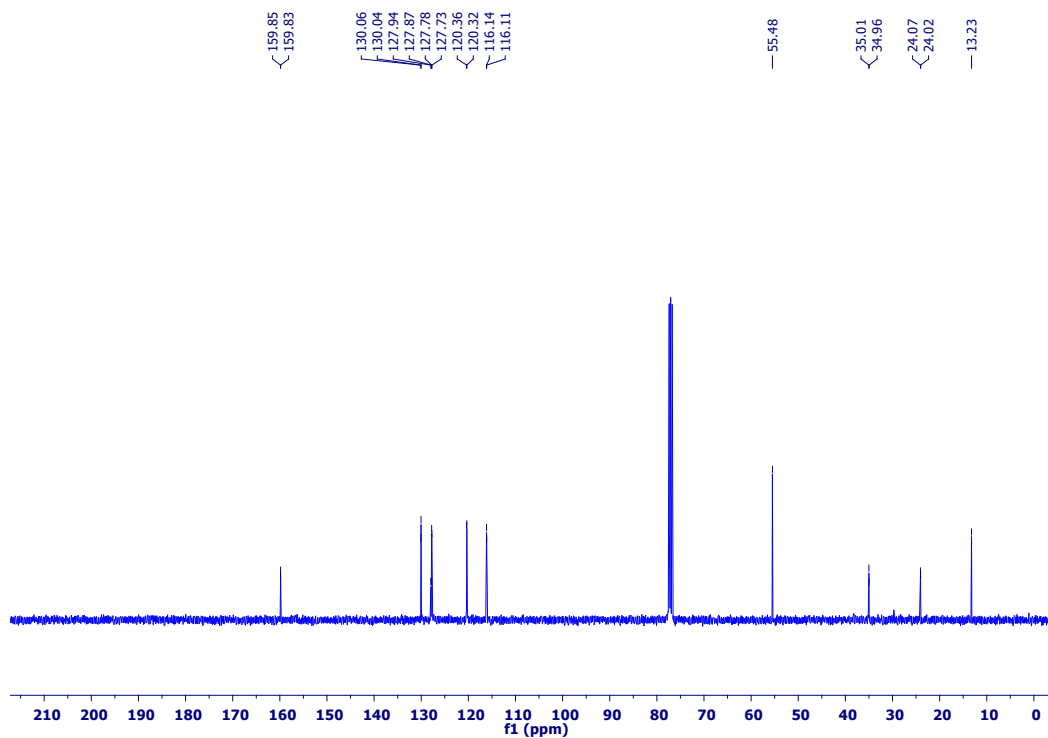
^{31}P NMR (162 MHz, CDCl_3)



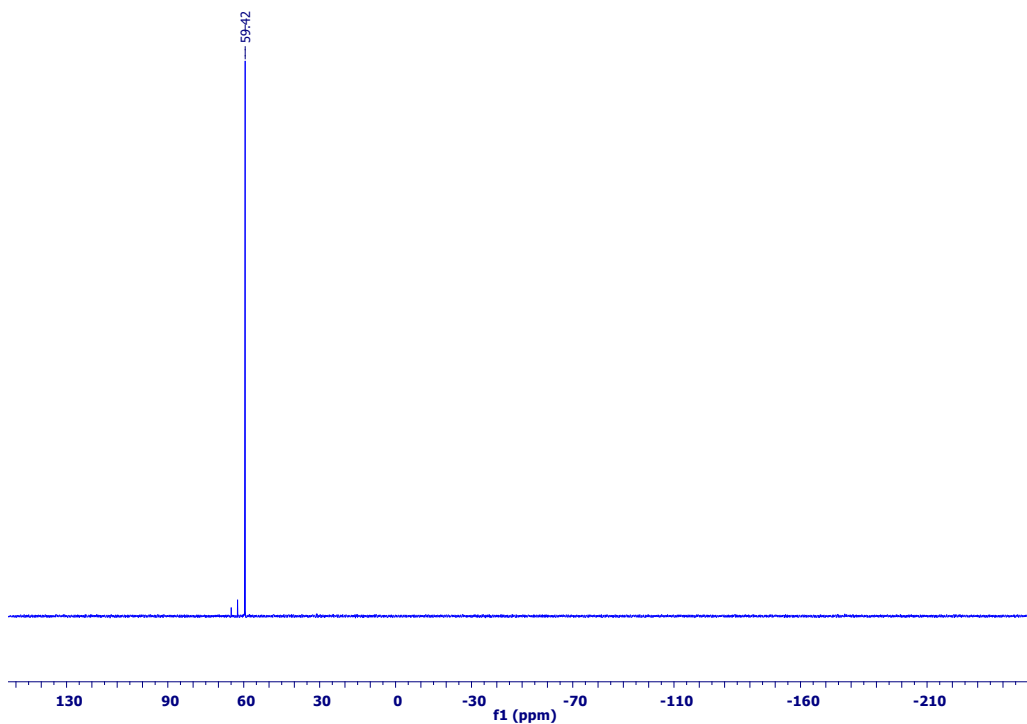
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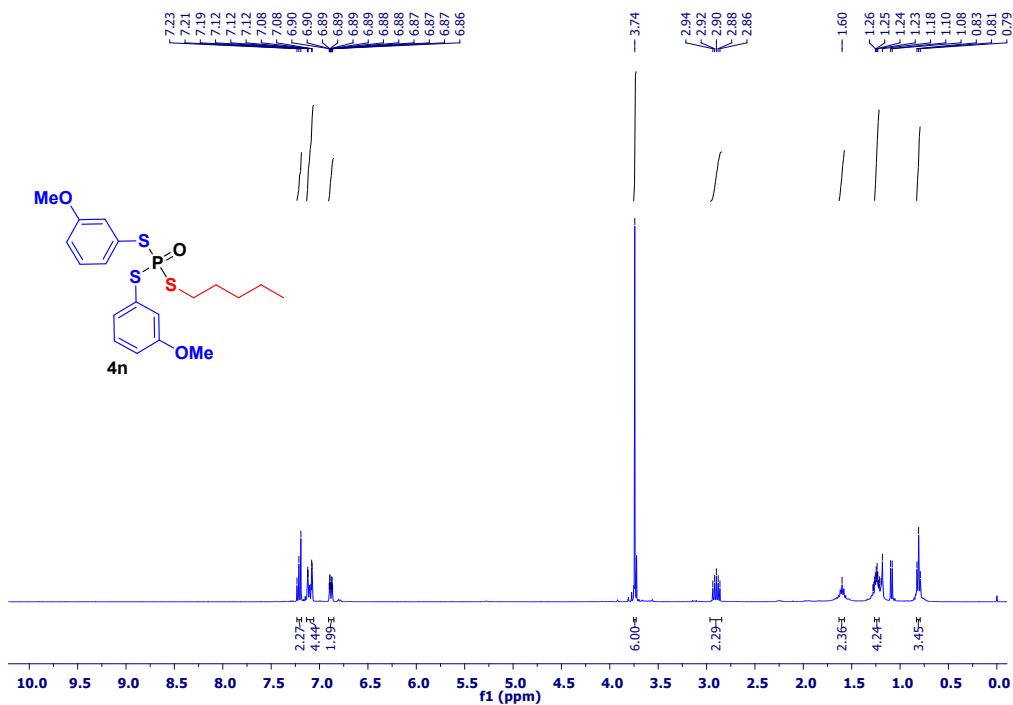
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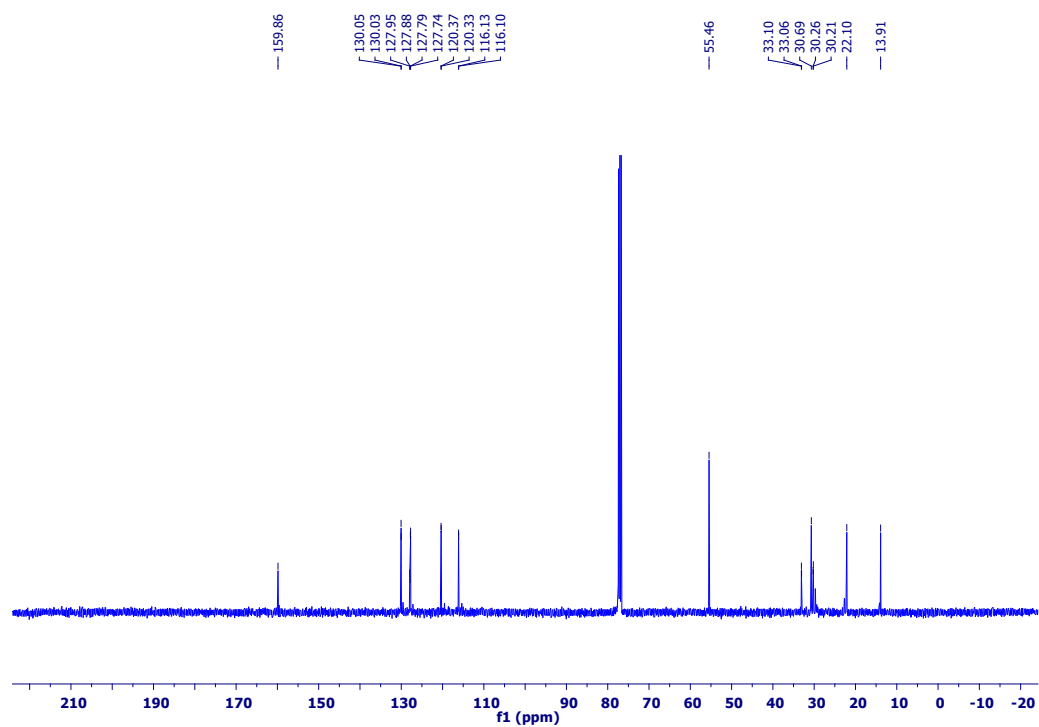
^{31}P NMR (162 MHz, CDCl_3)



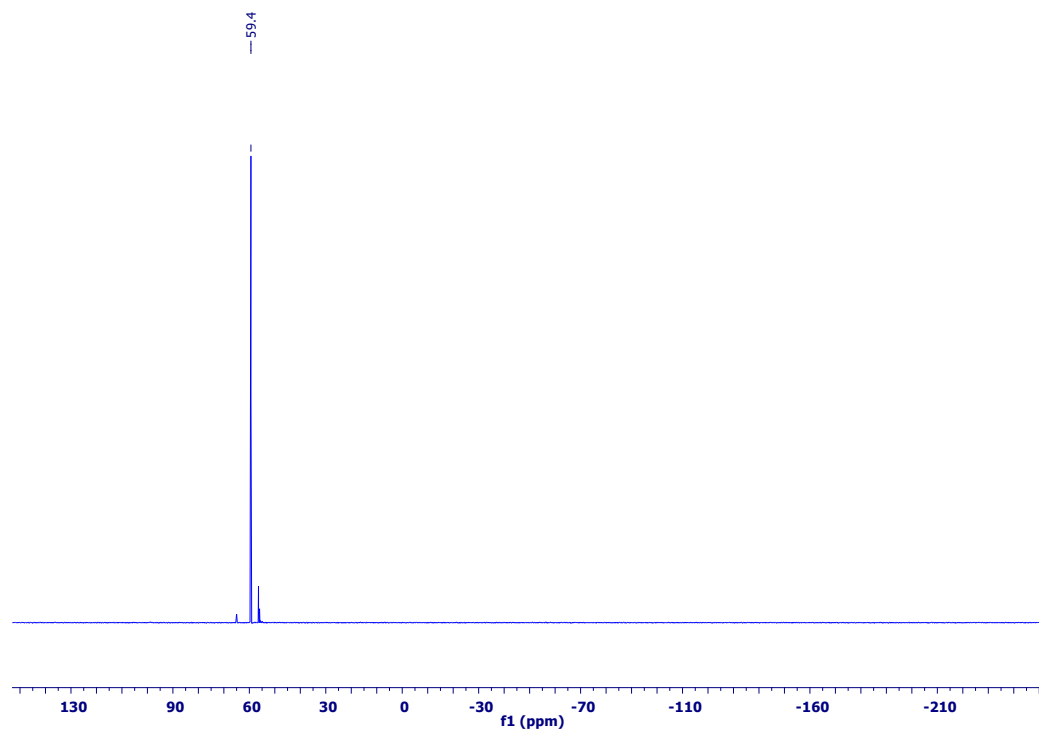
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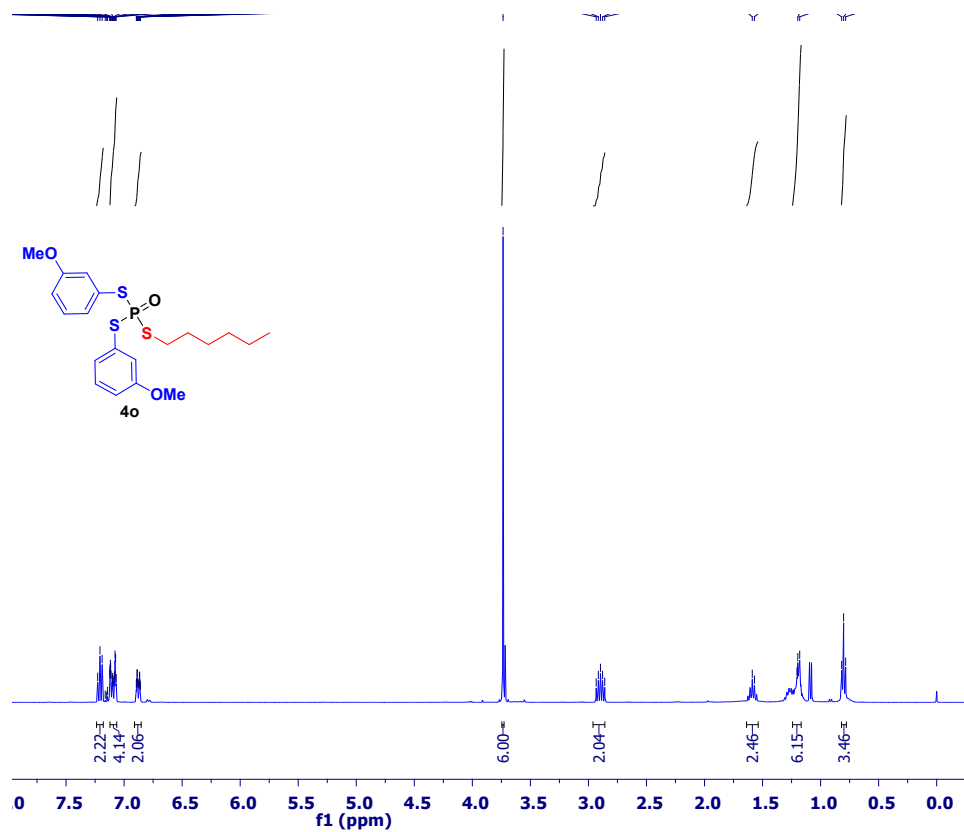
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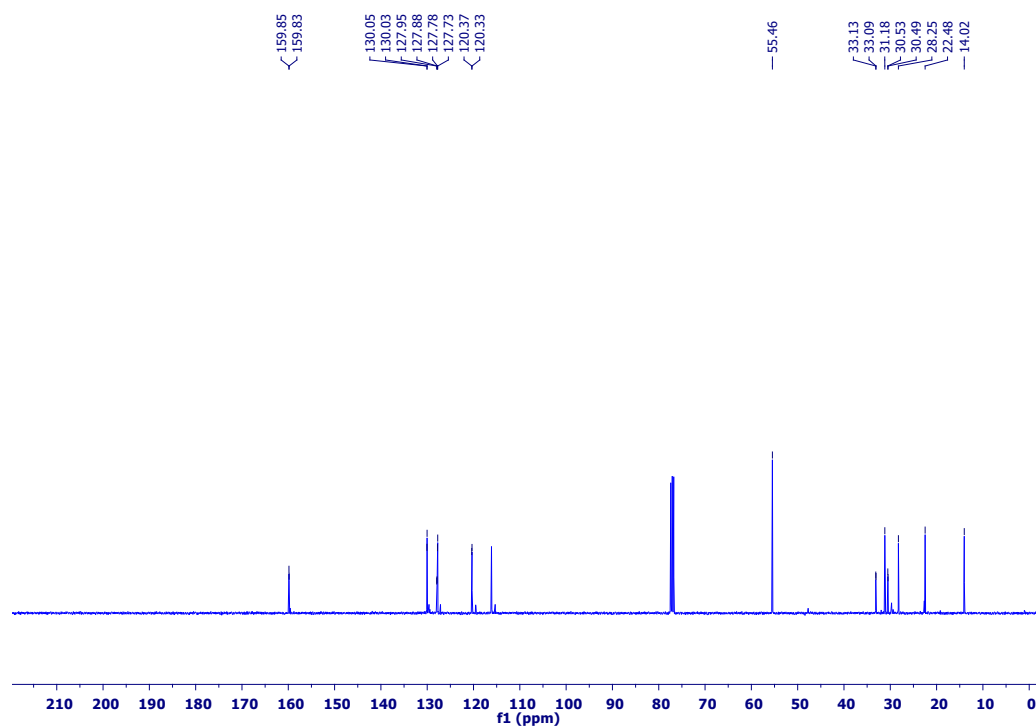
^{31}P NMR (162 MHz, CDCl_3)



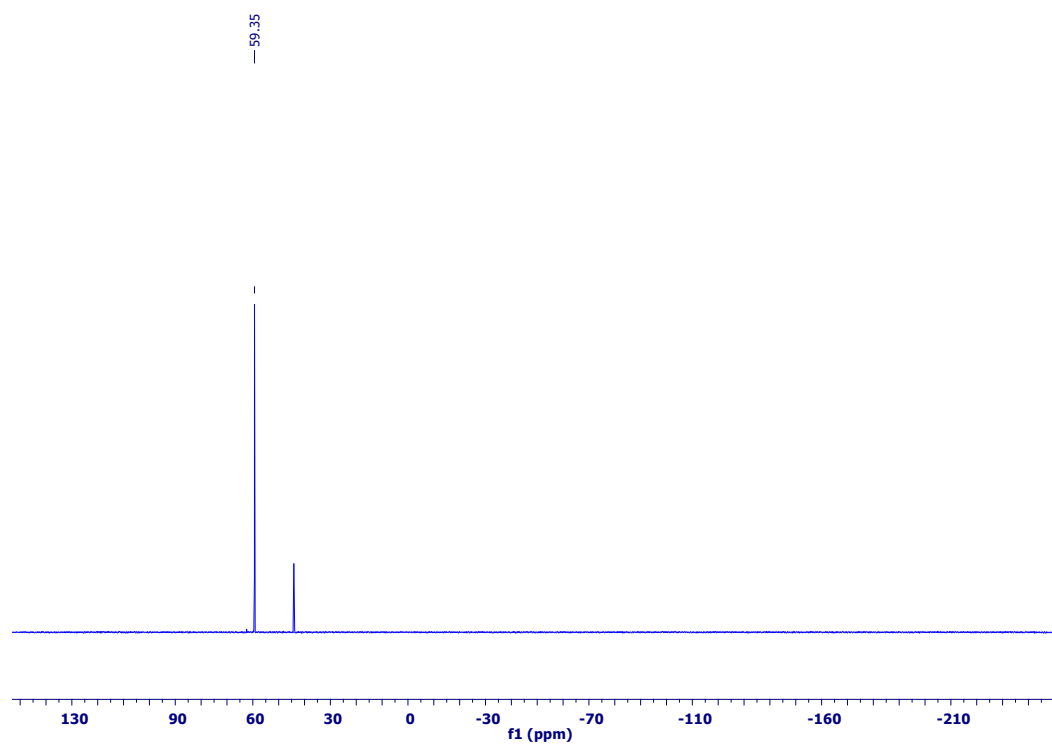
^1H NMR (400 MHz, CDCl_3)



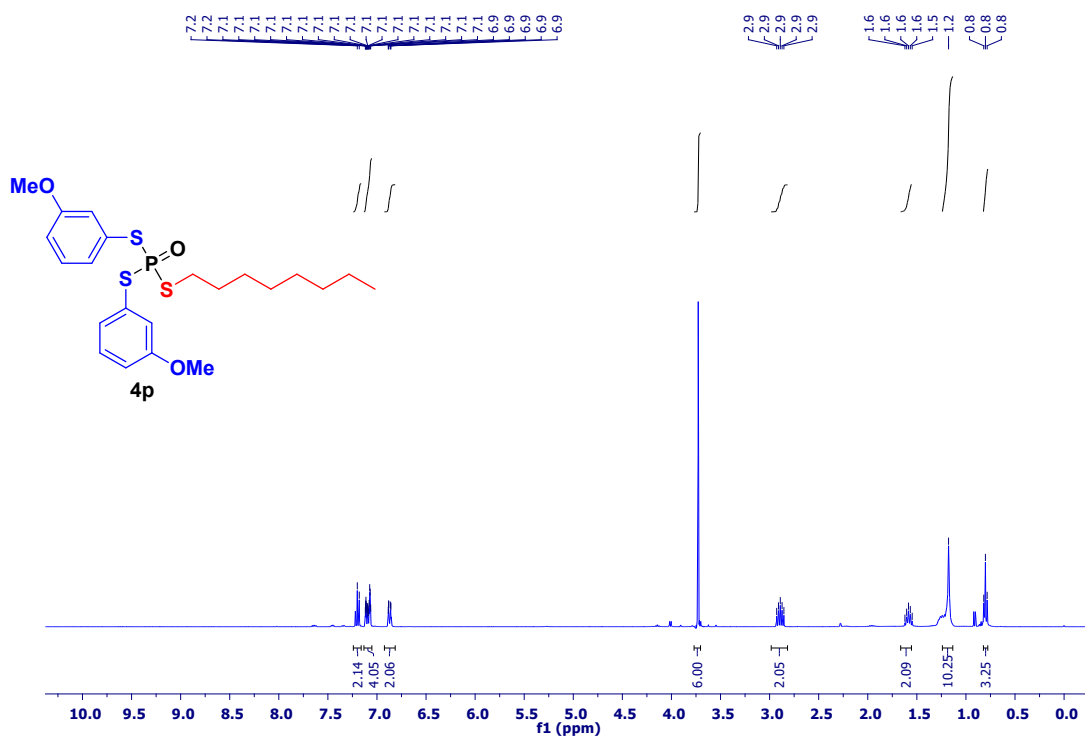
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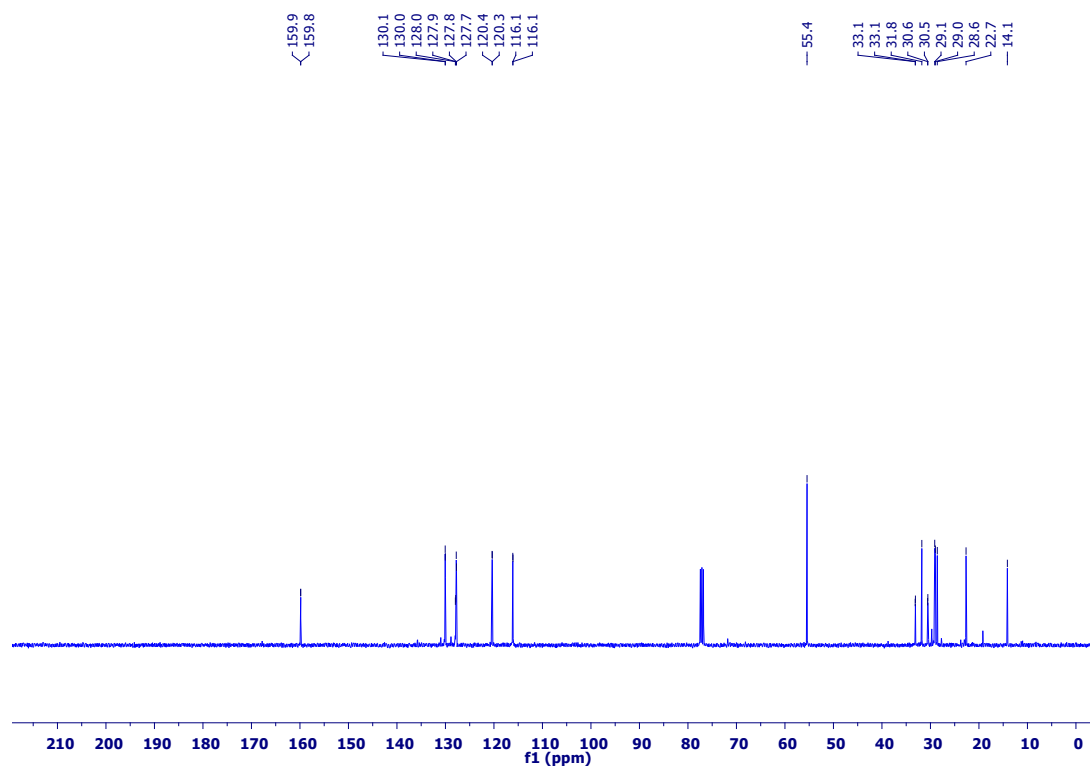
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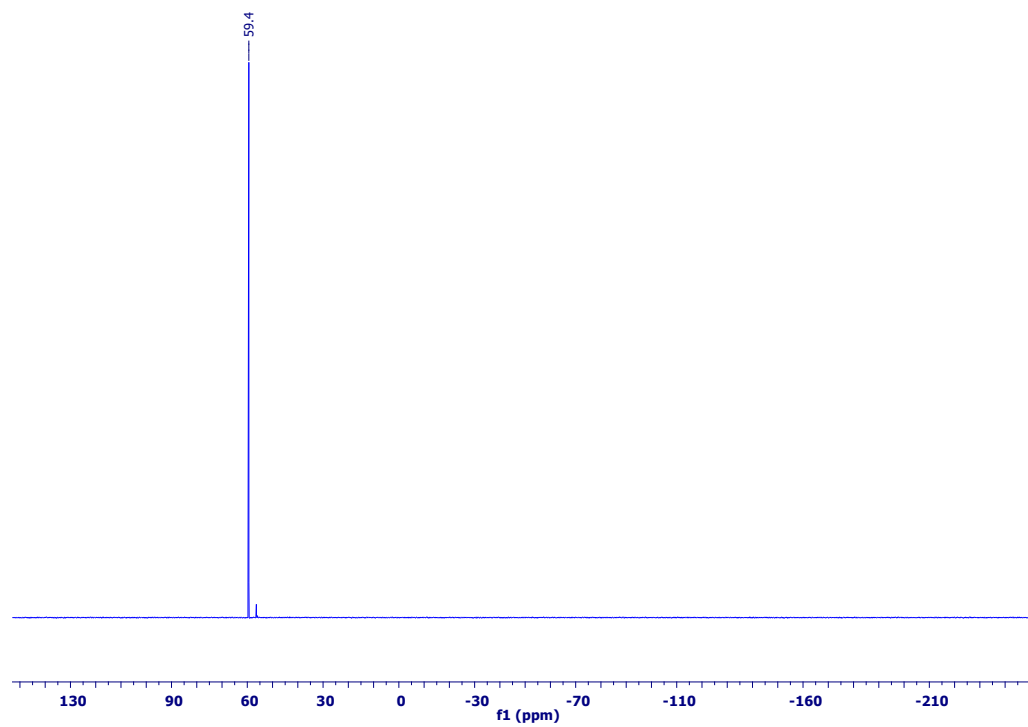
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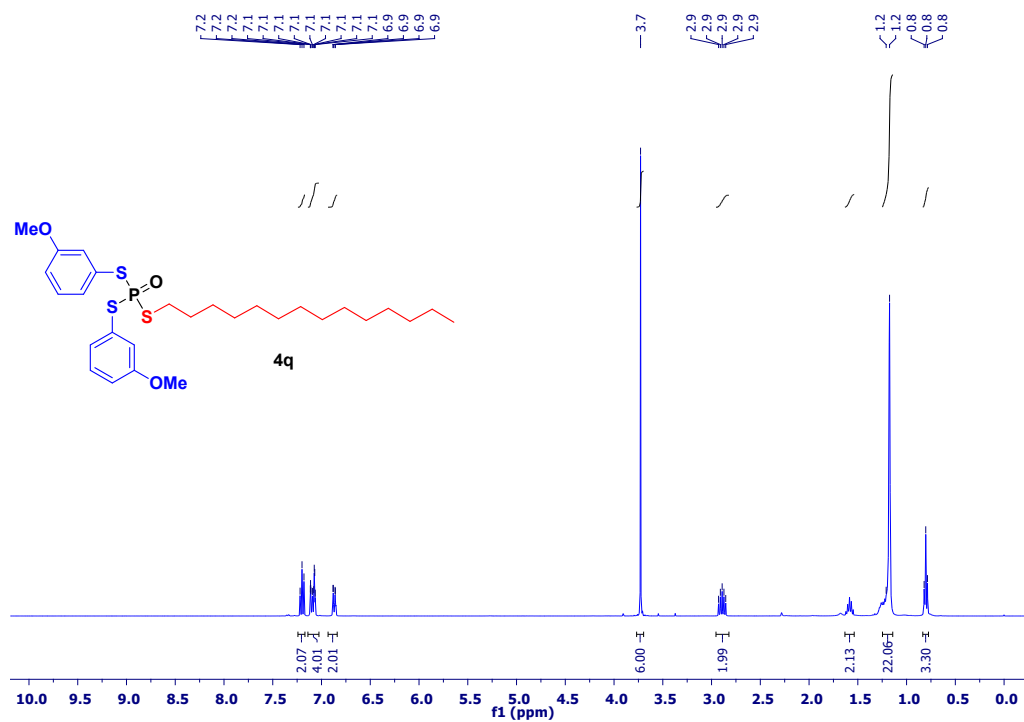
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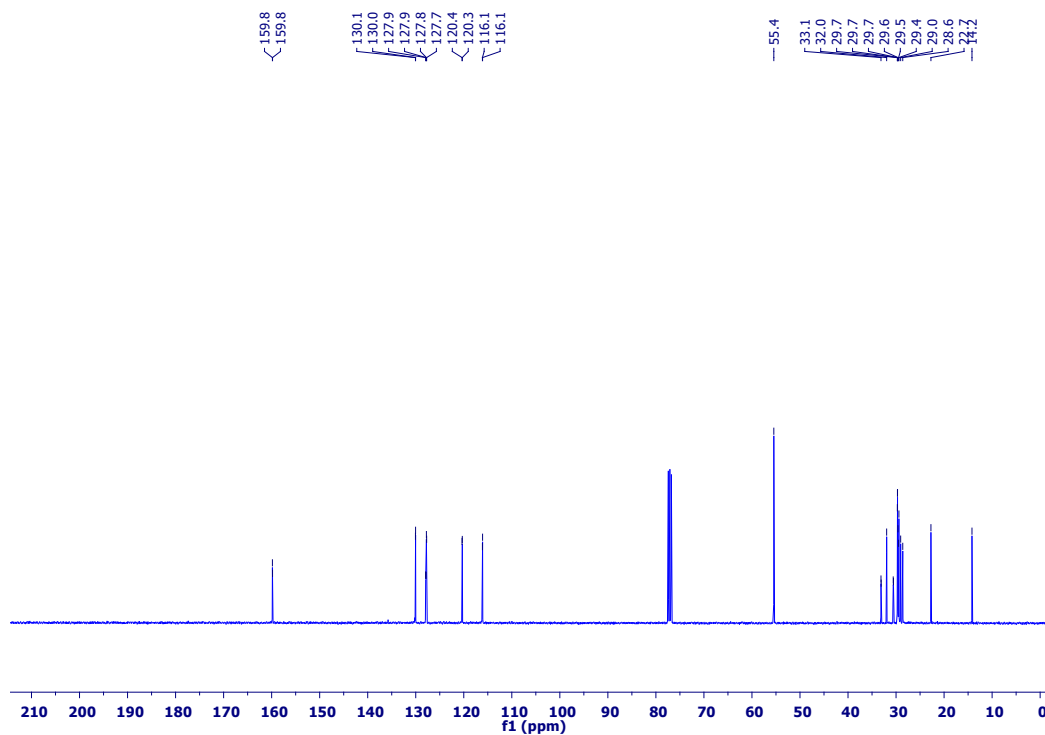
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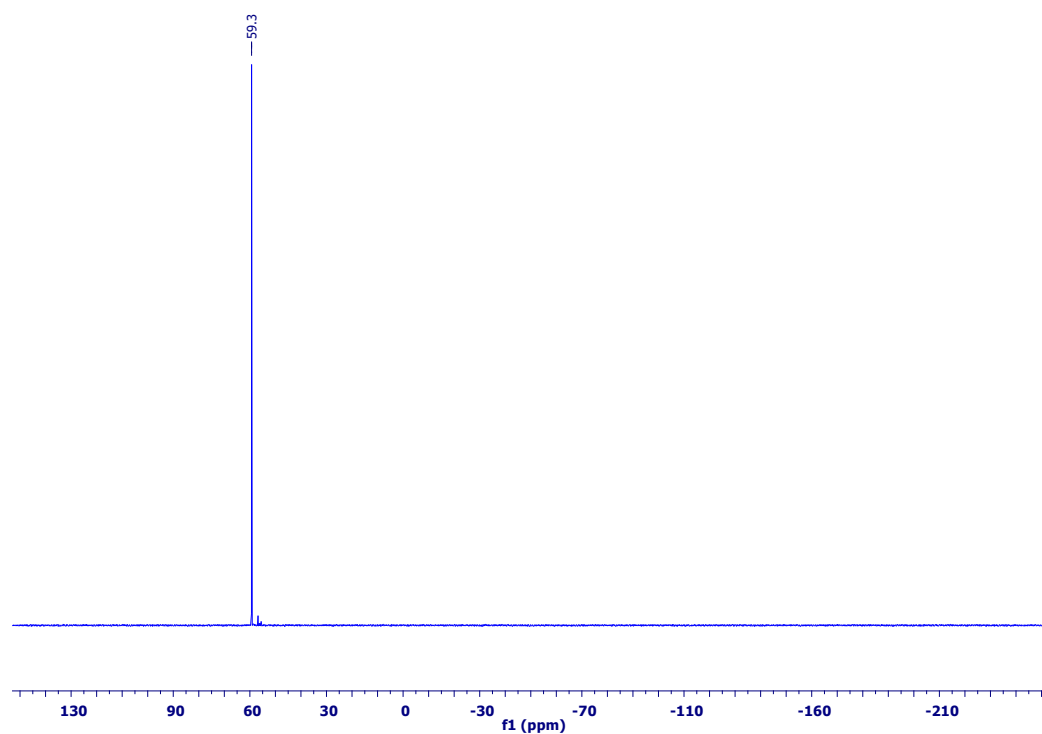
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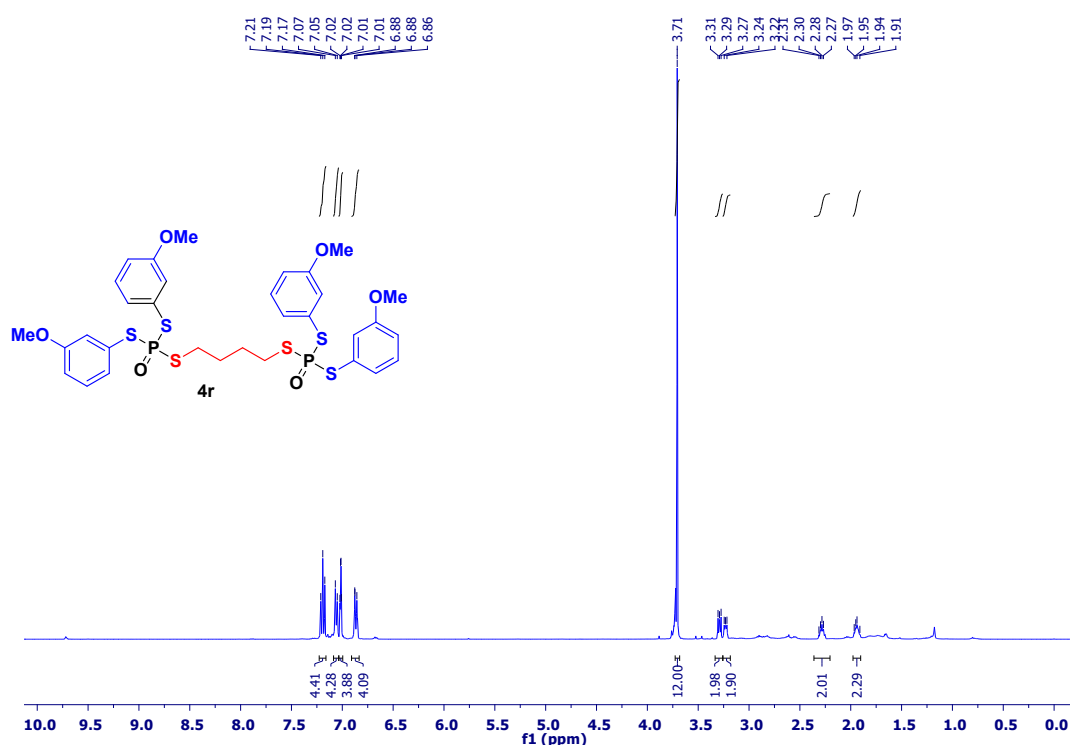
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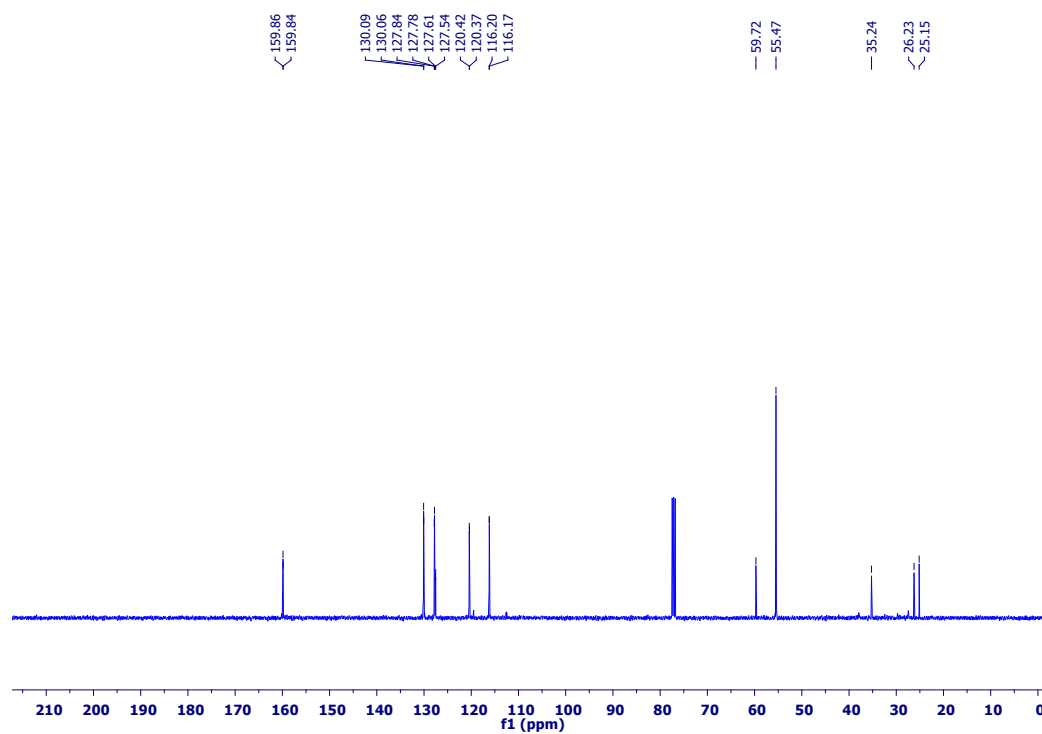
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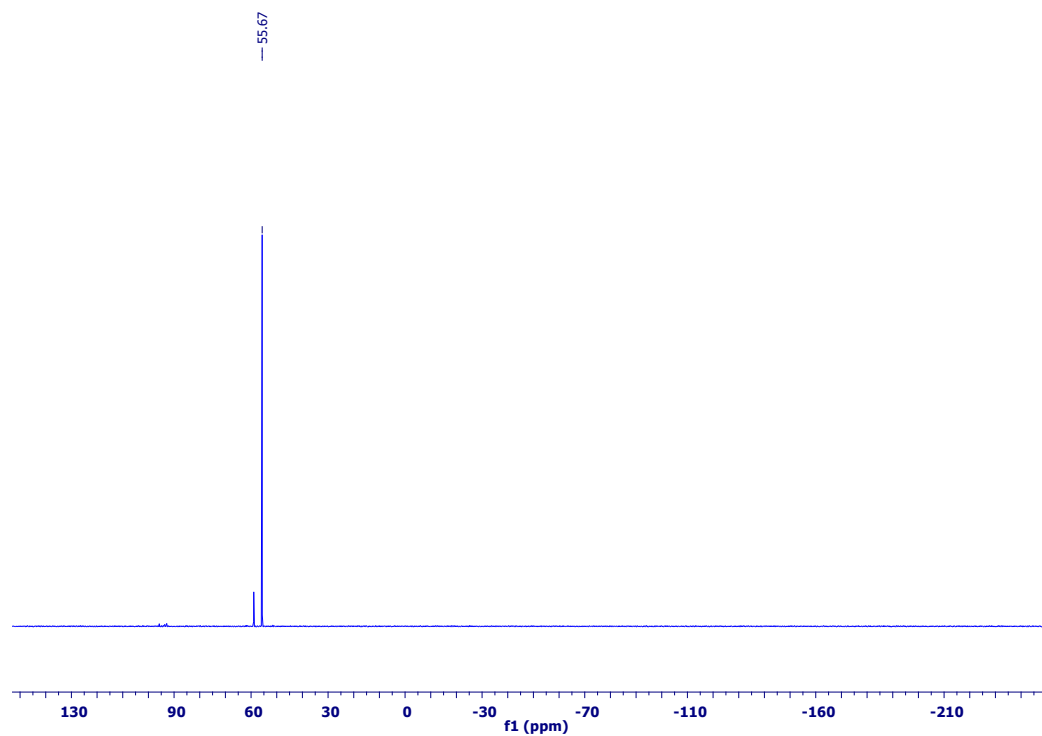
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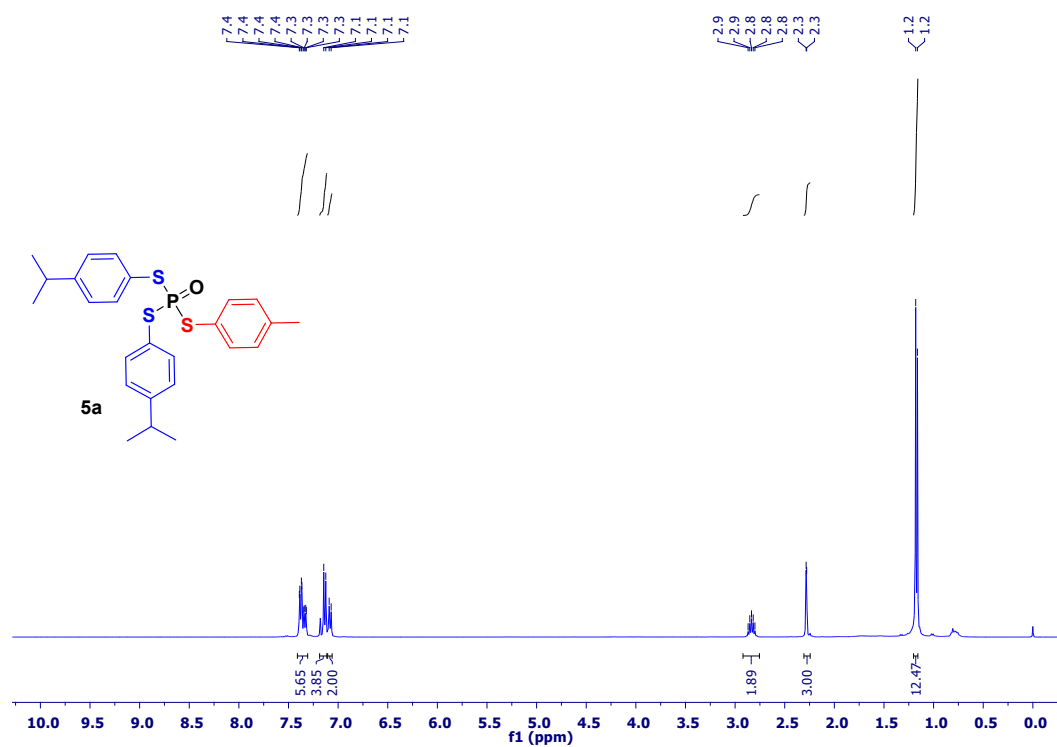
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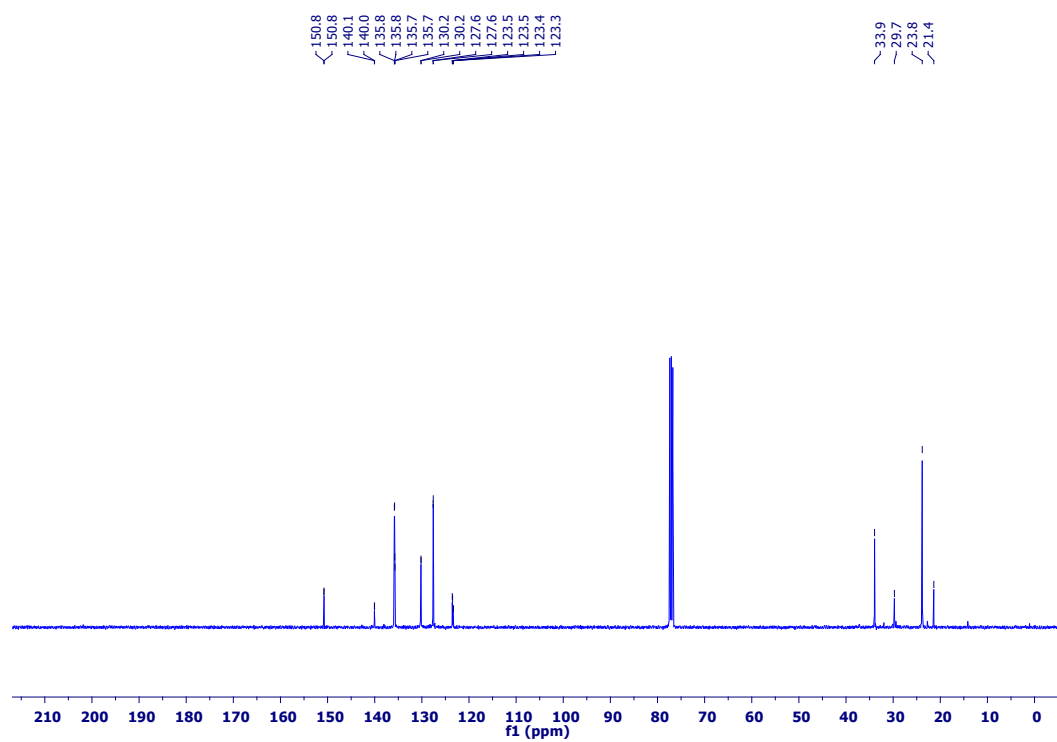
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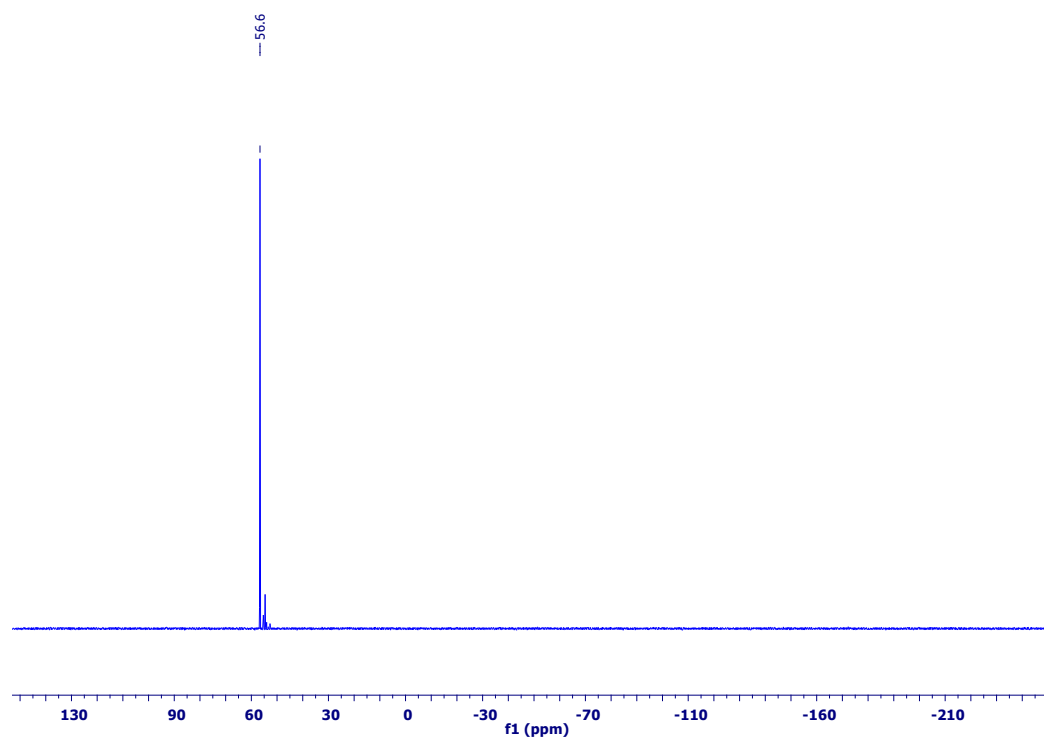
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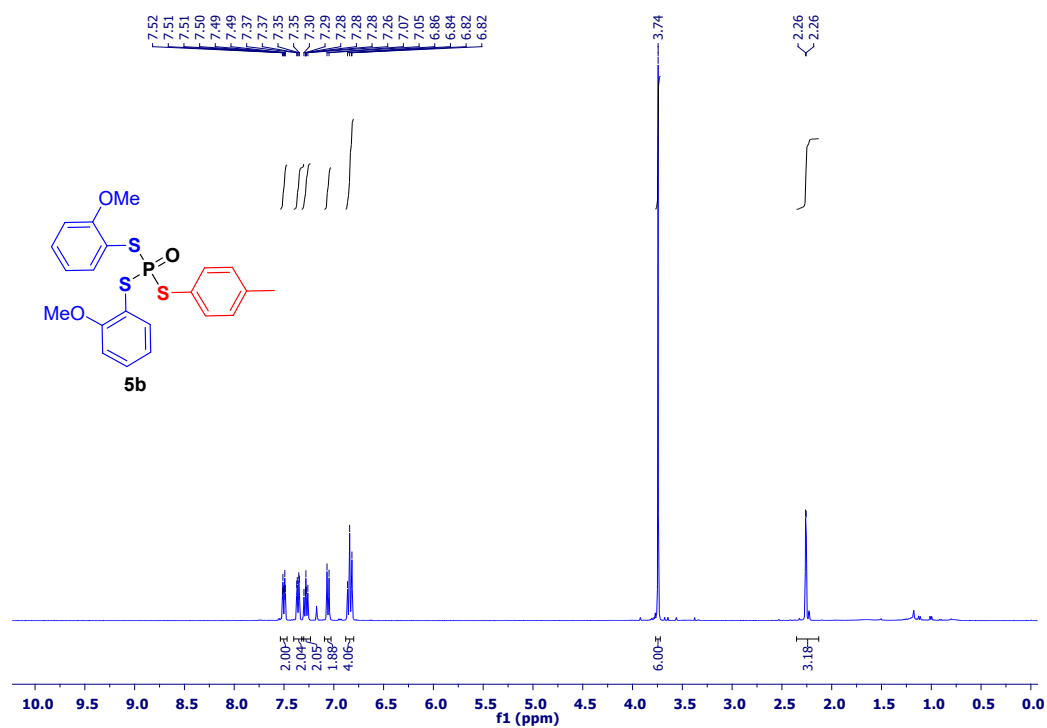
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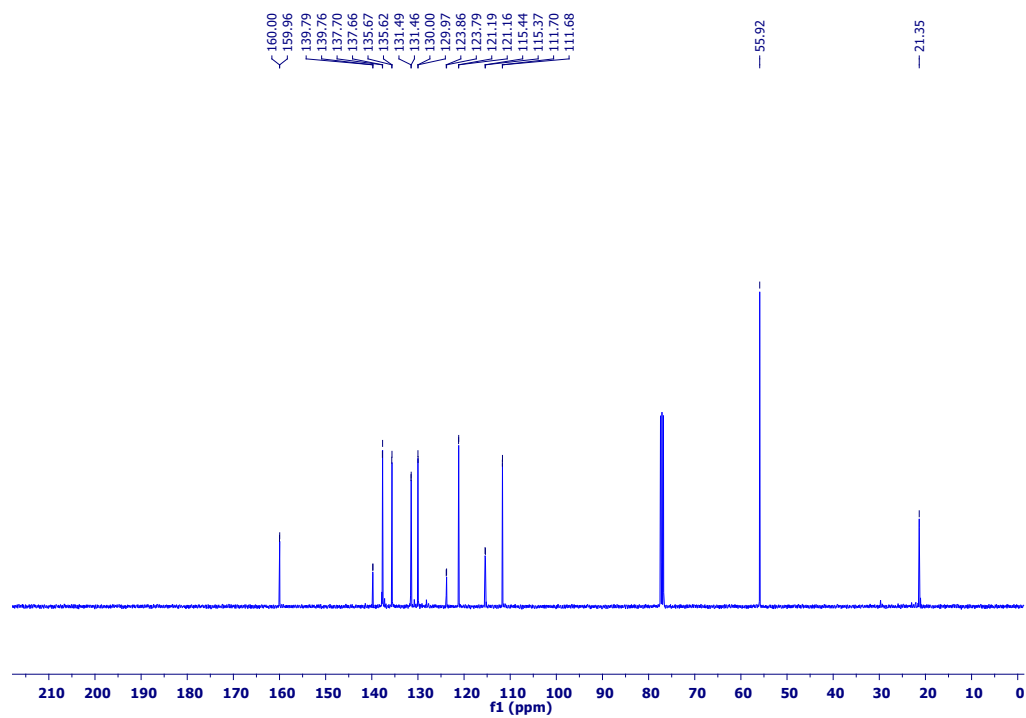
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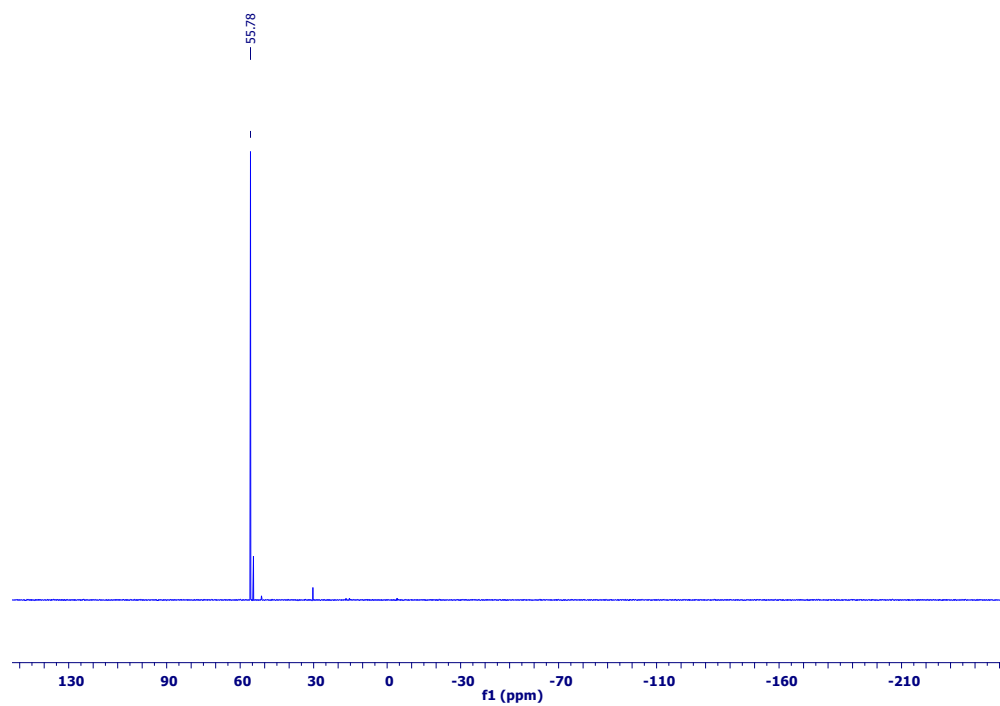
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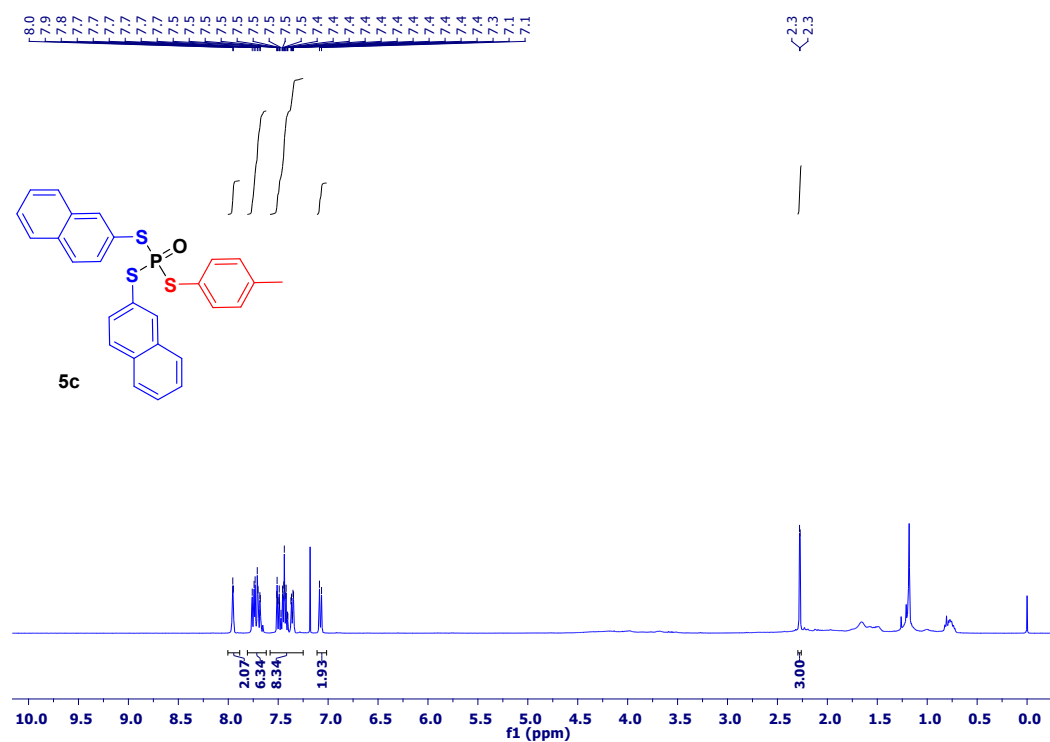
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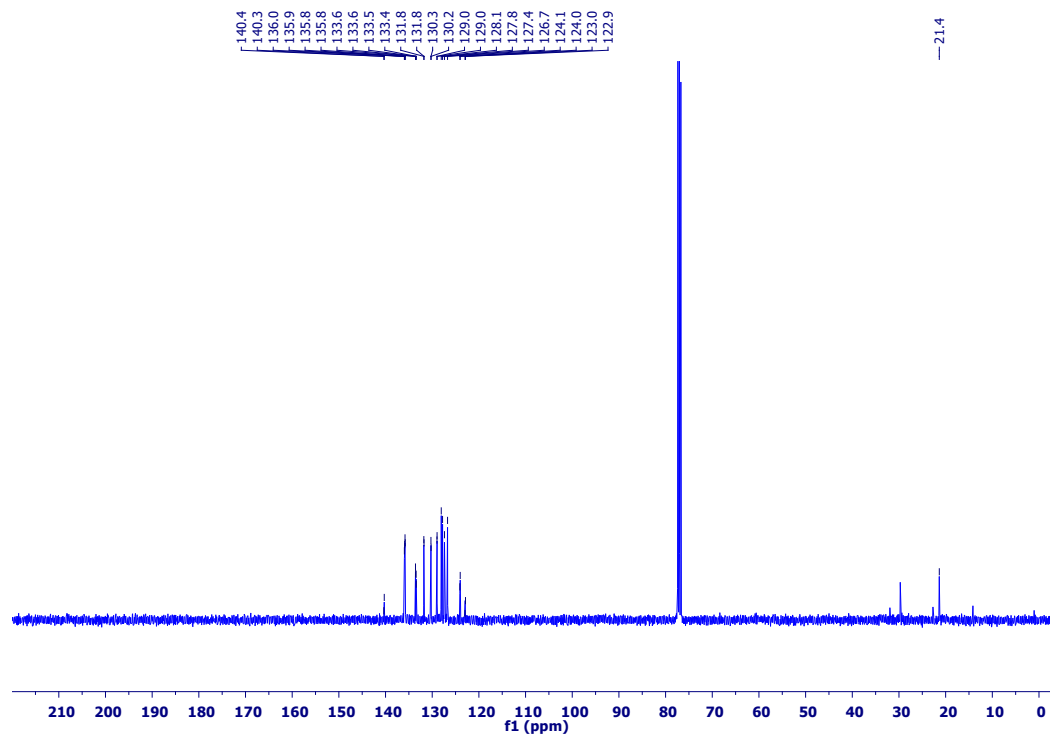
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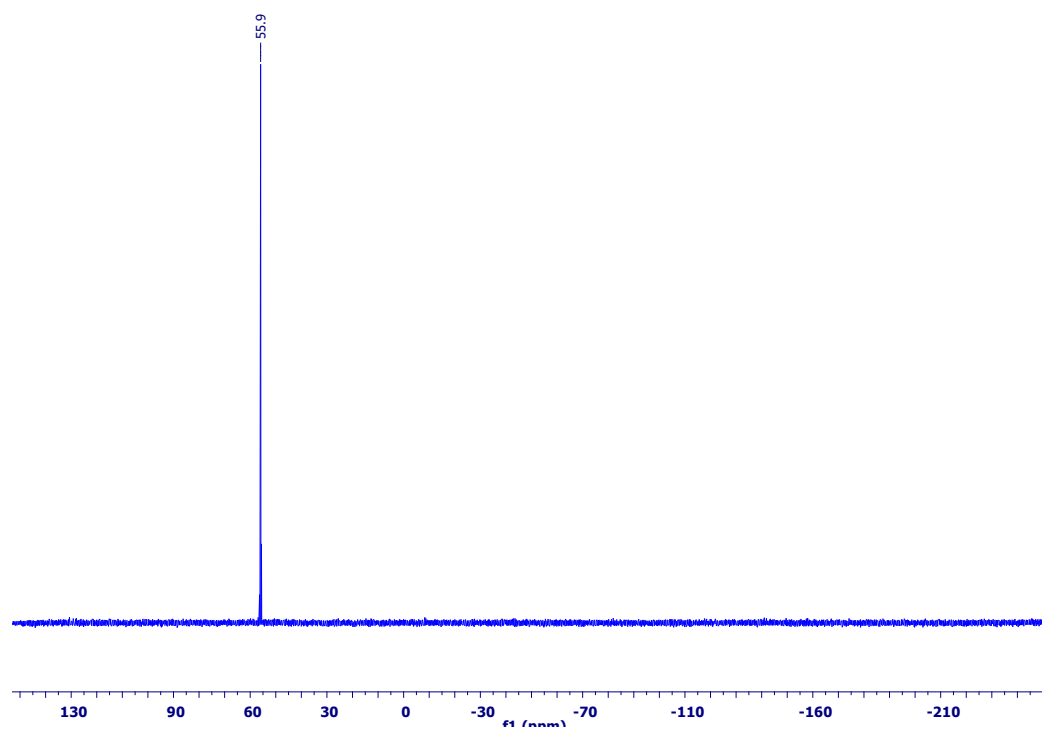
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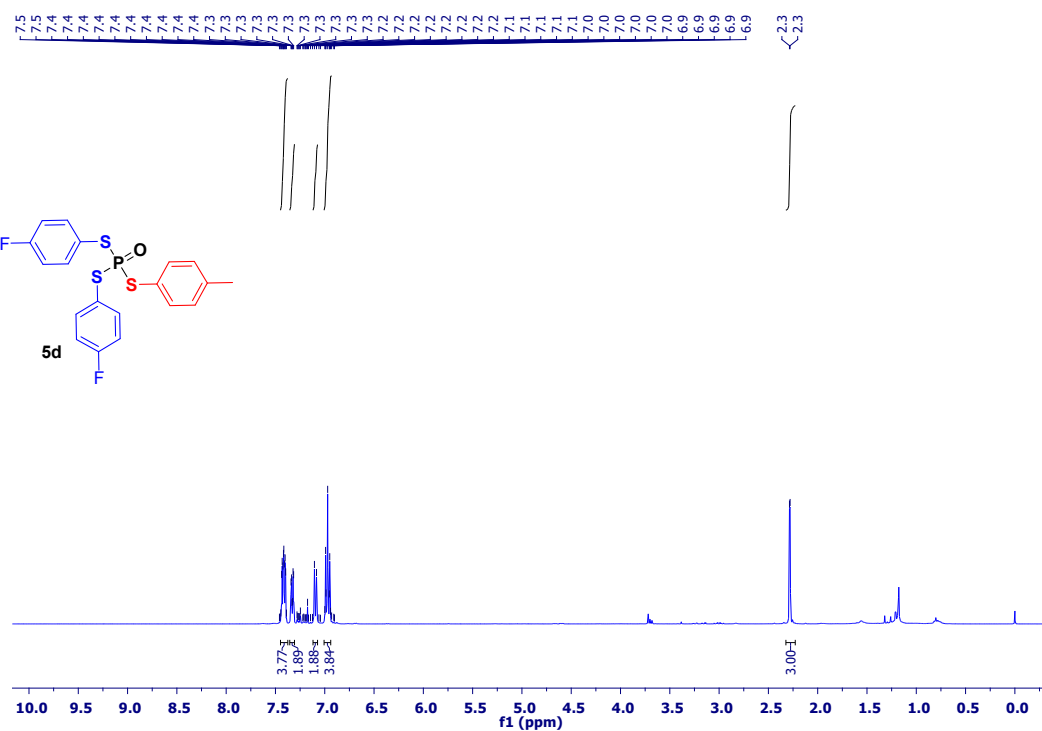
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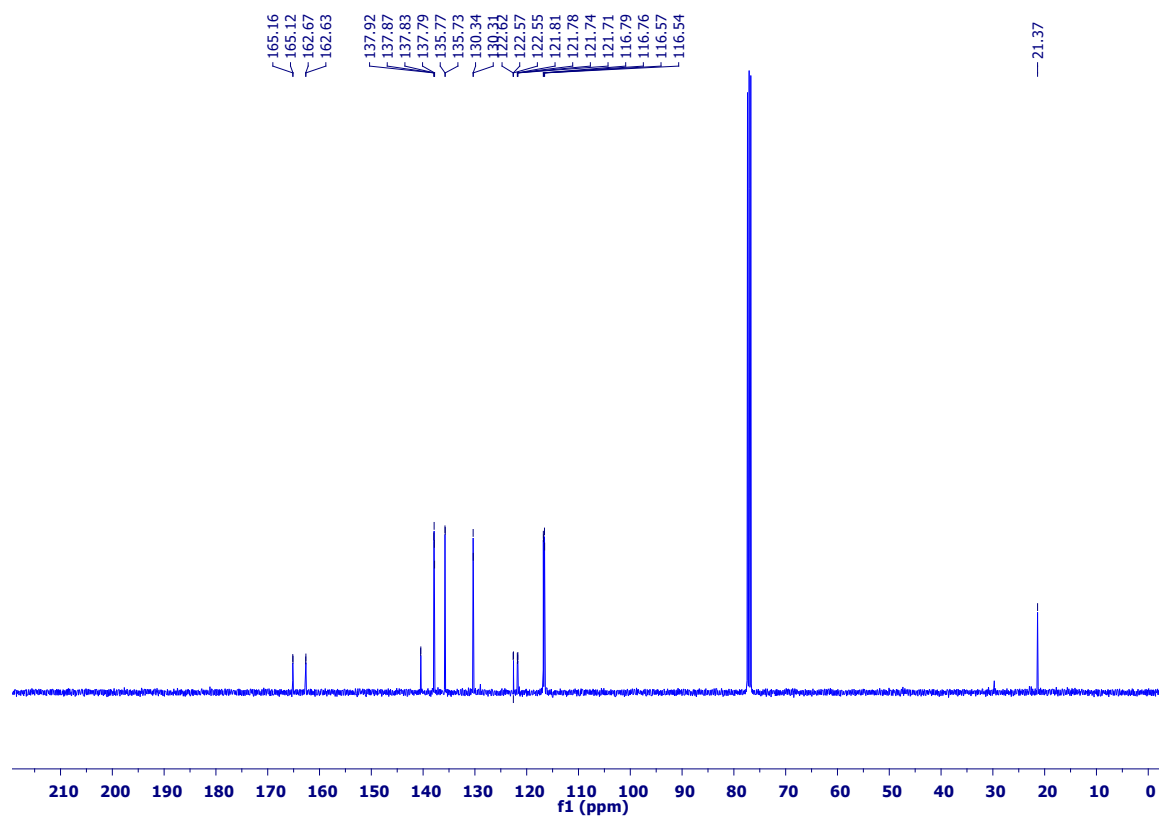
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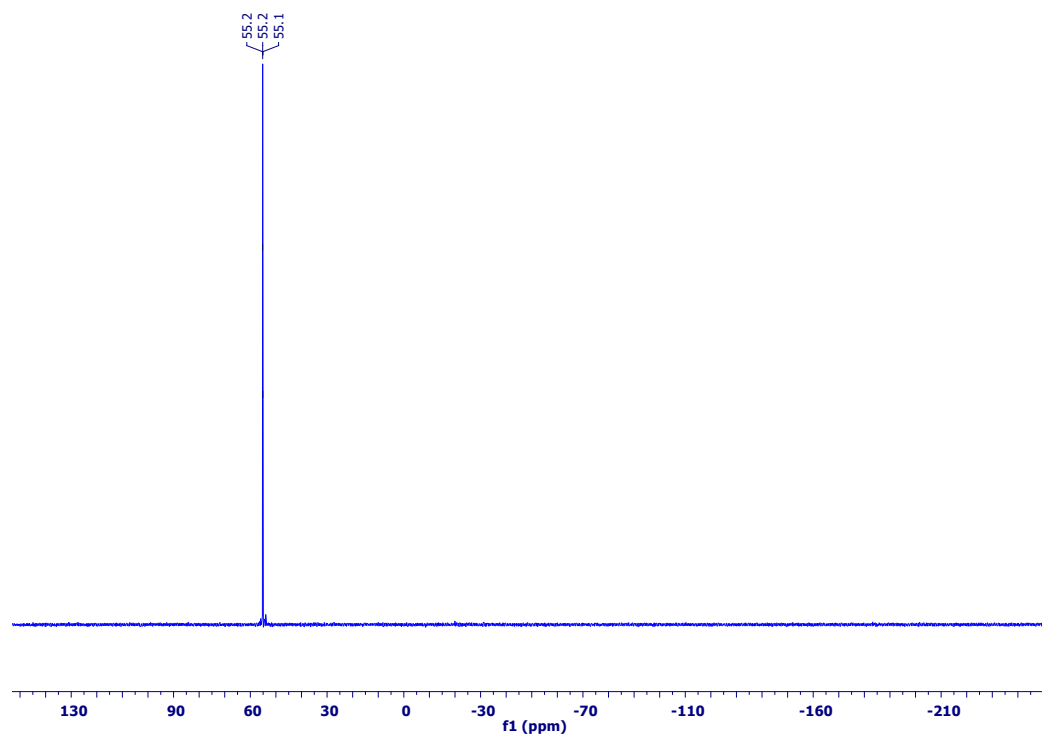
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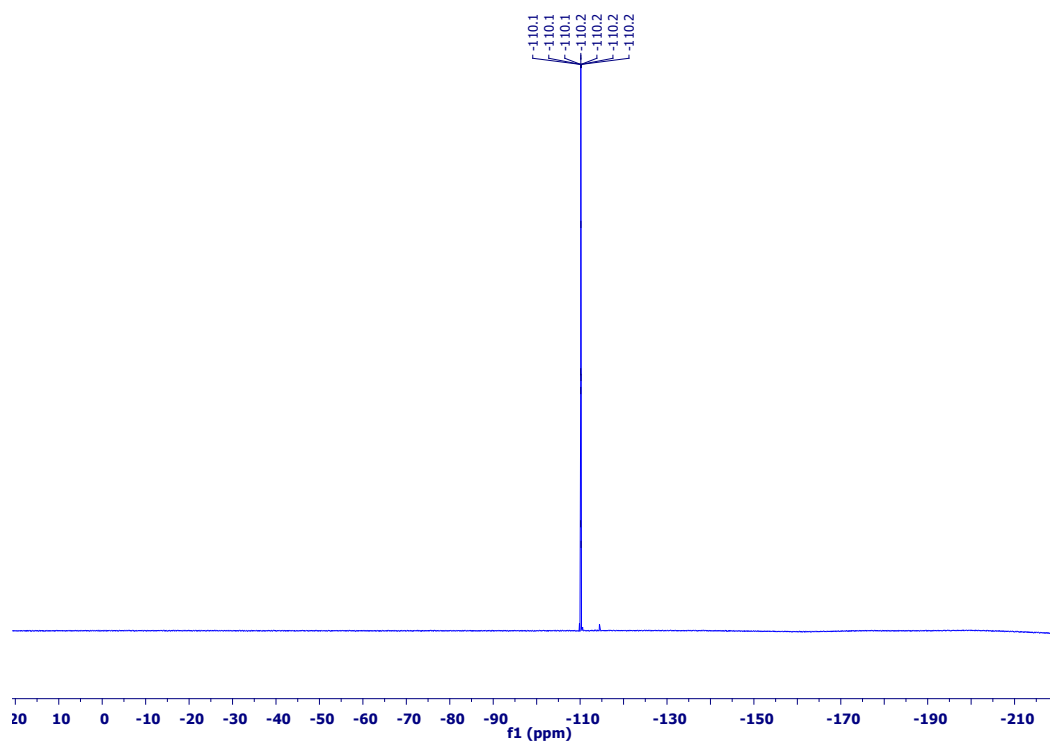
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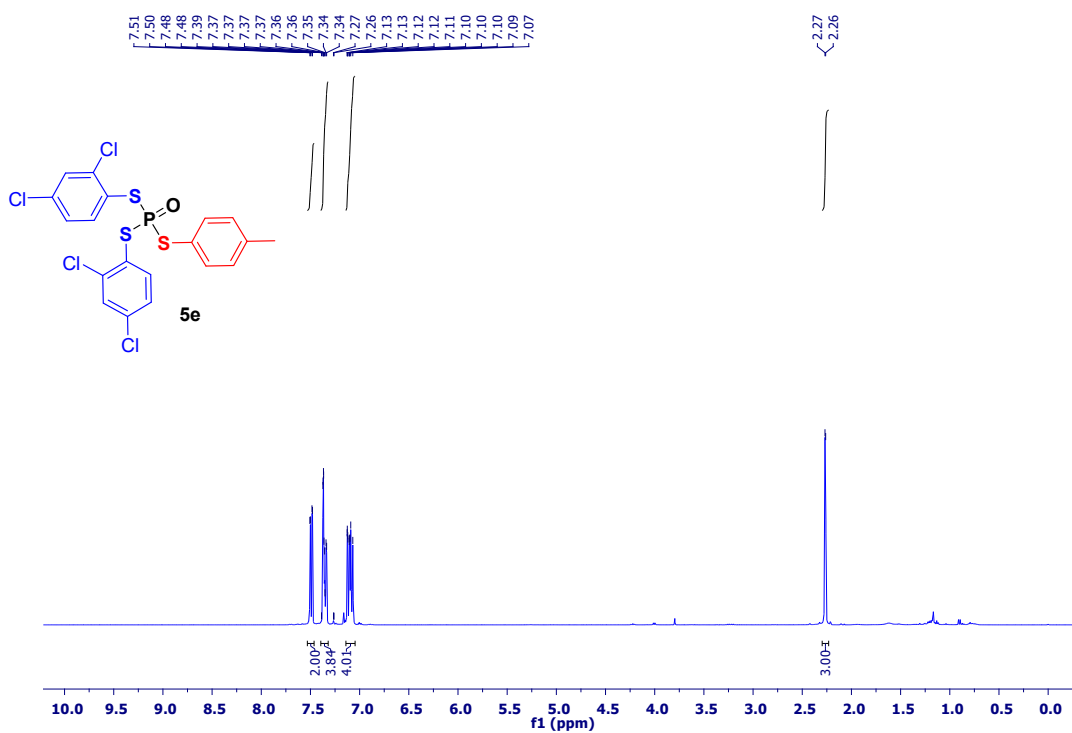
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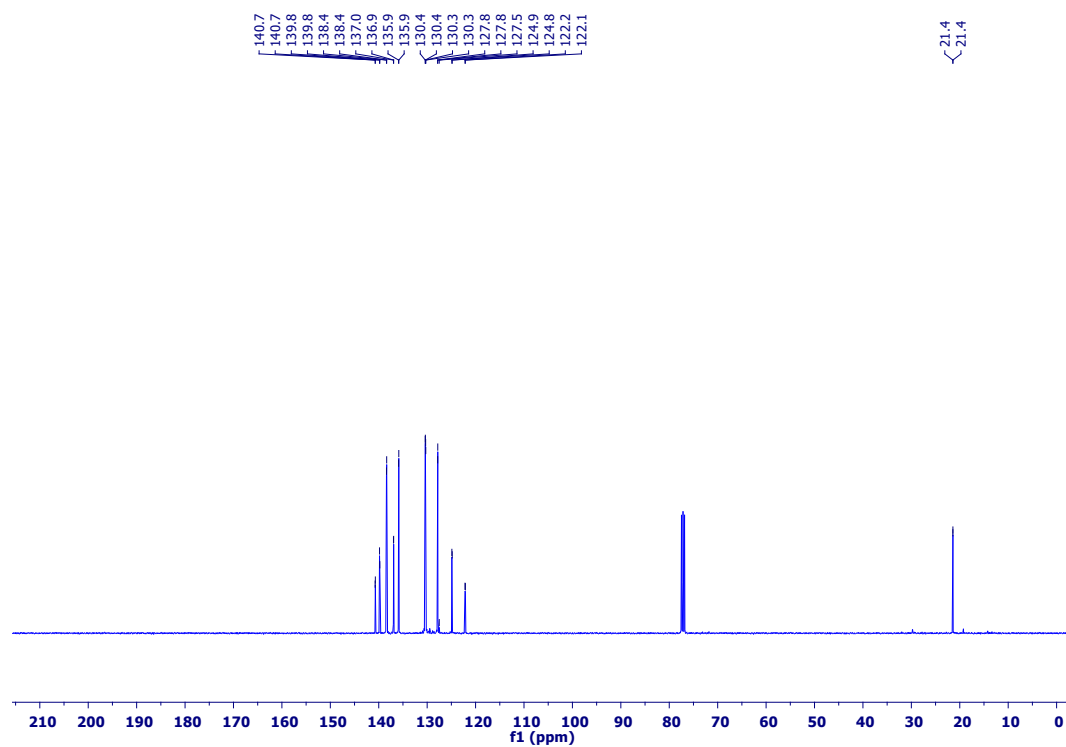
^{19}F NMR (377 MHz, CDCl_3)



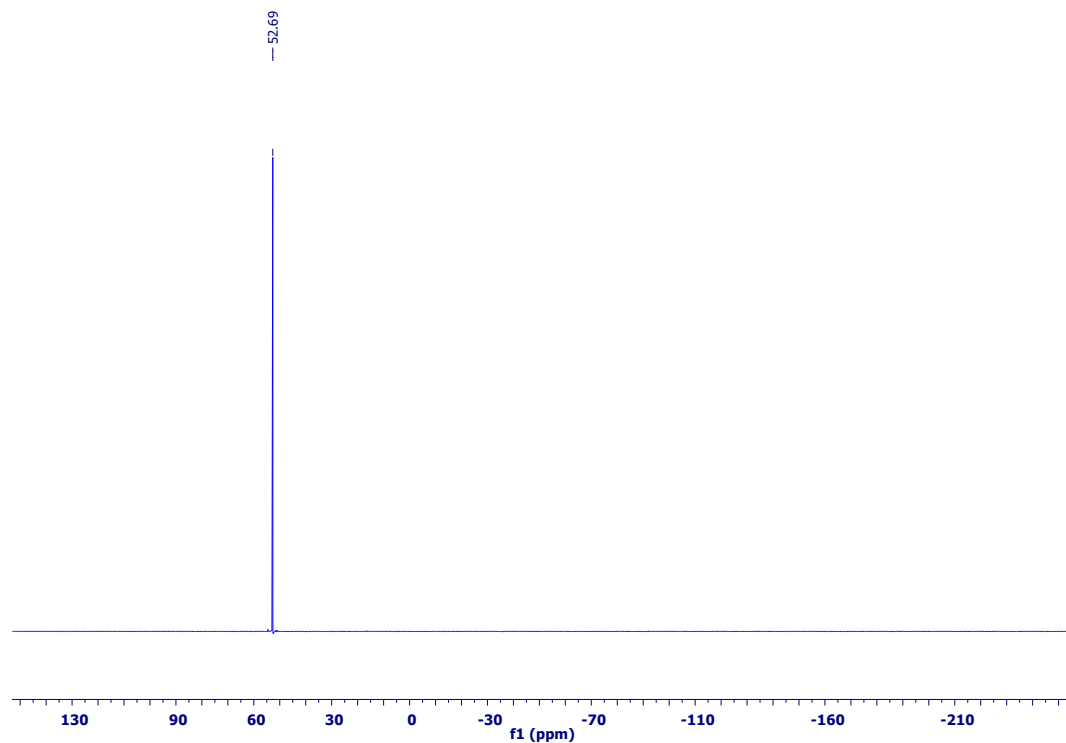
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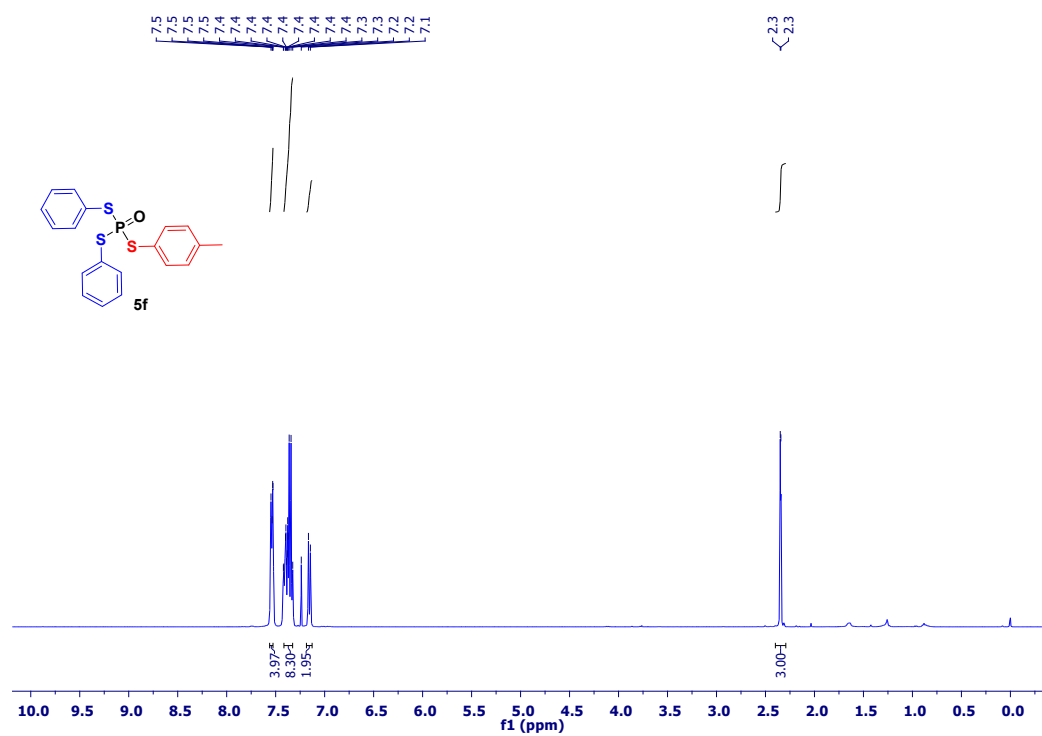
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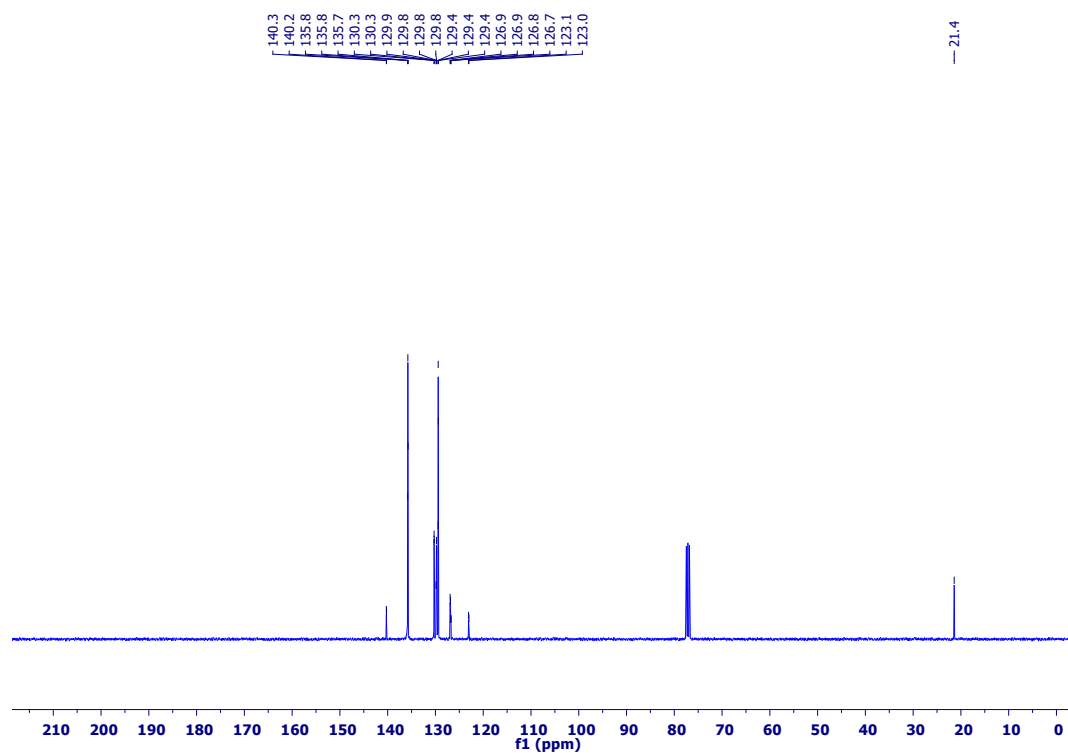
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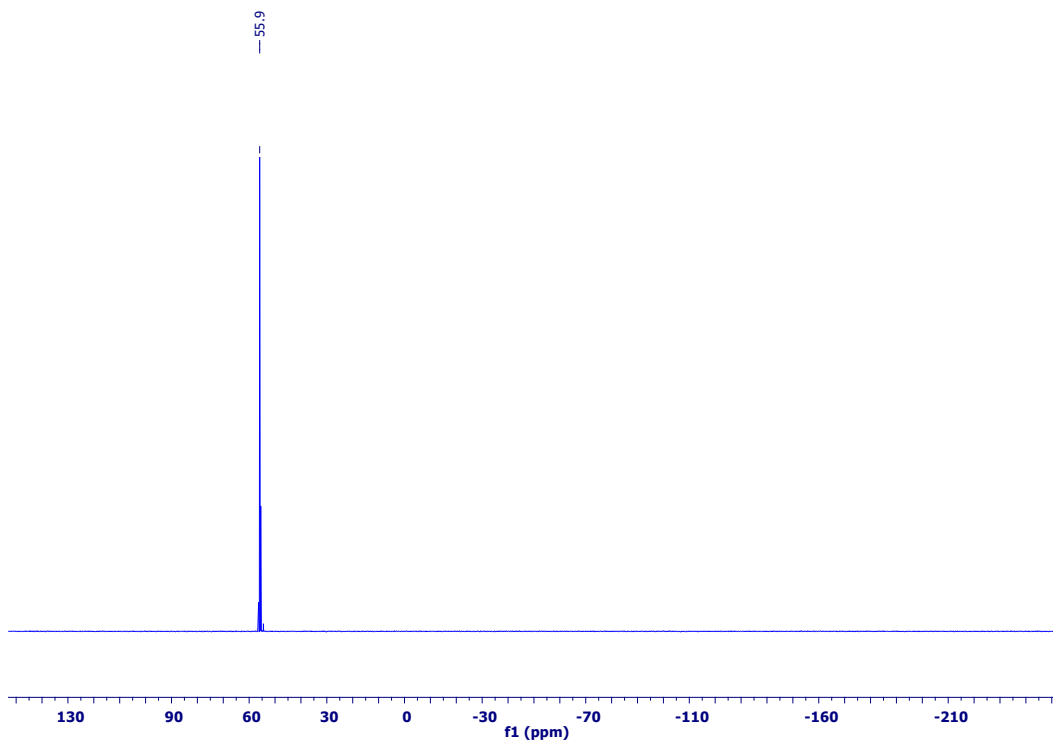
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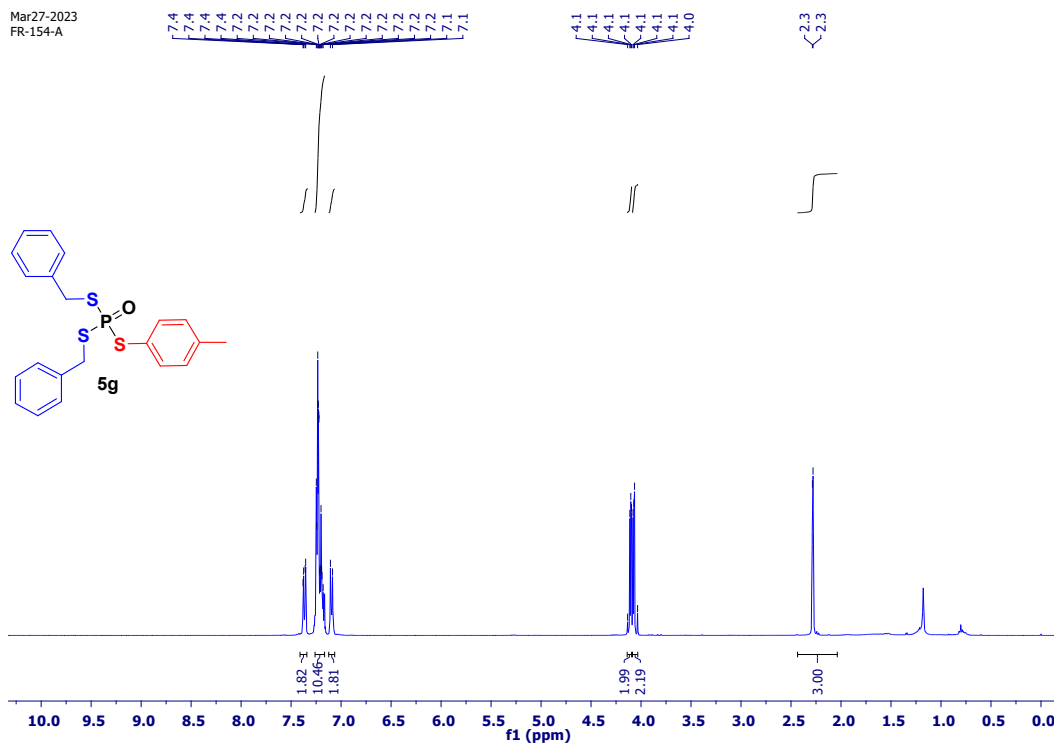
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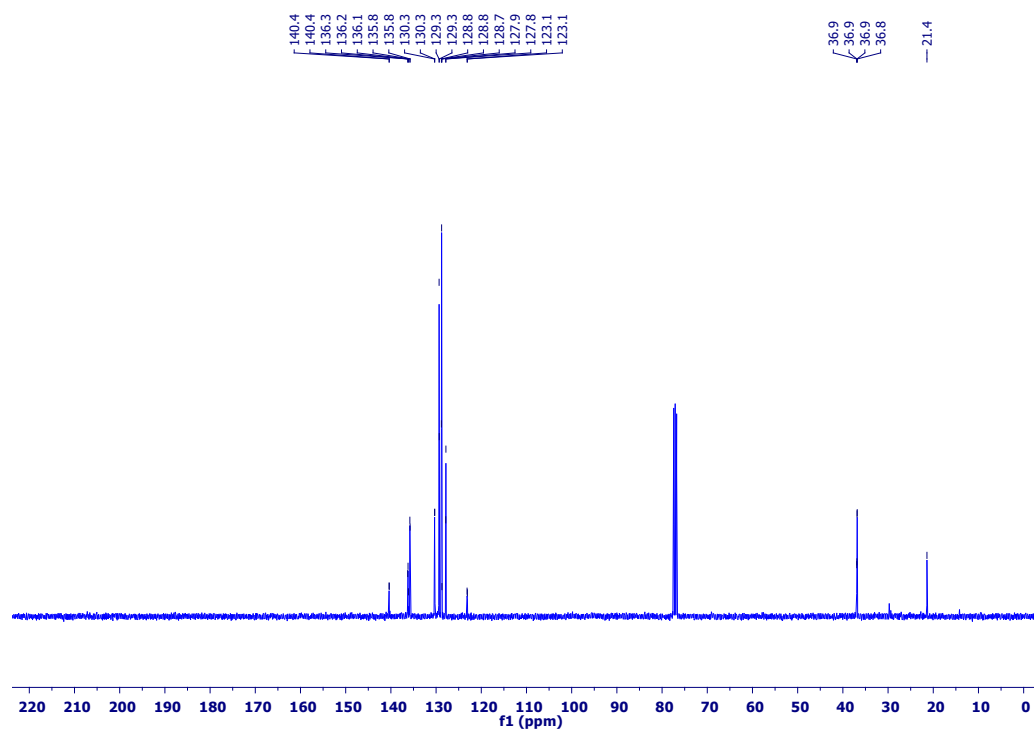
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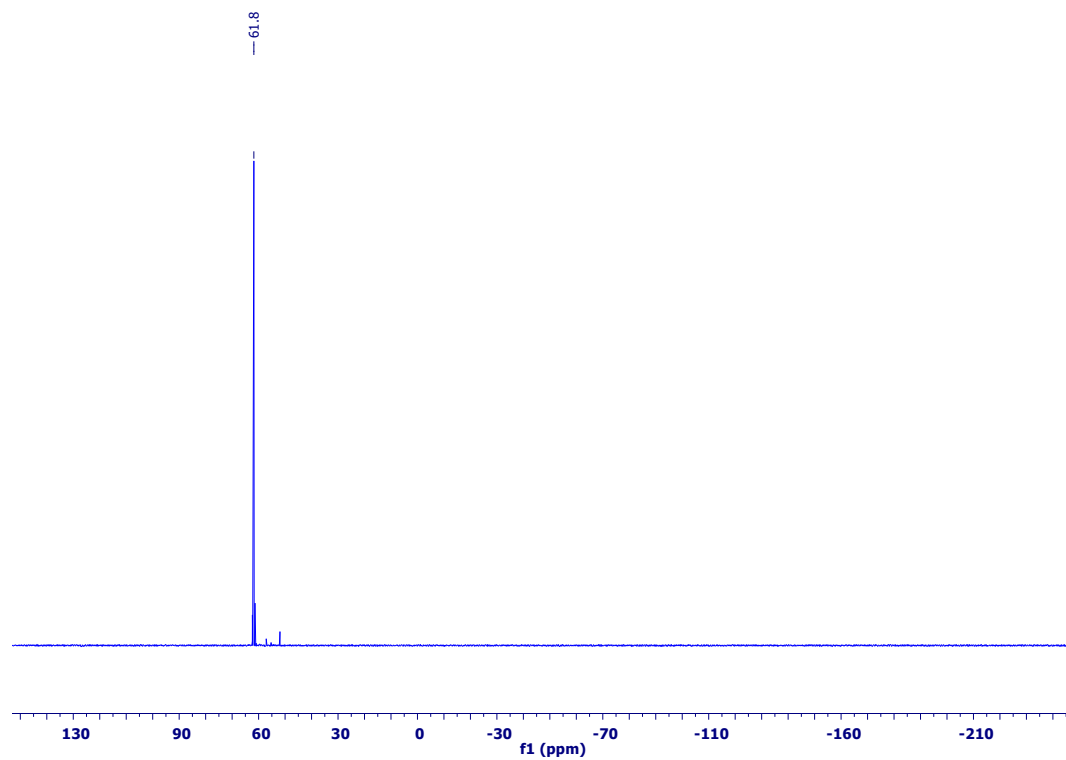
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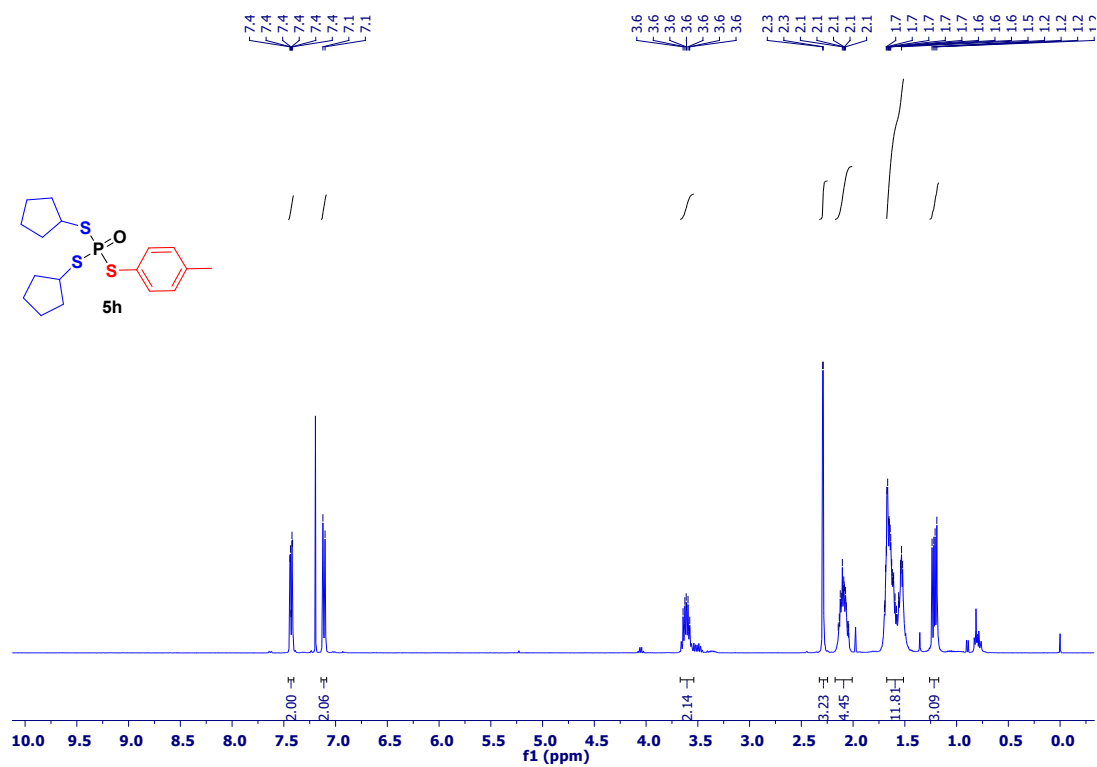
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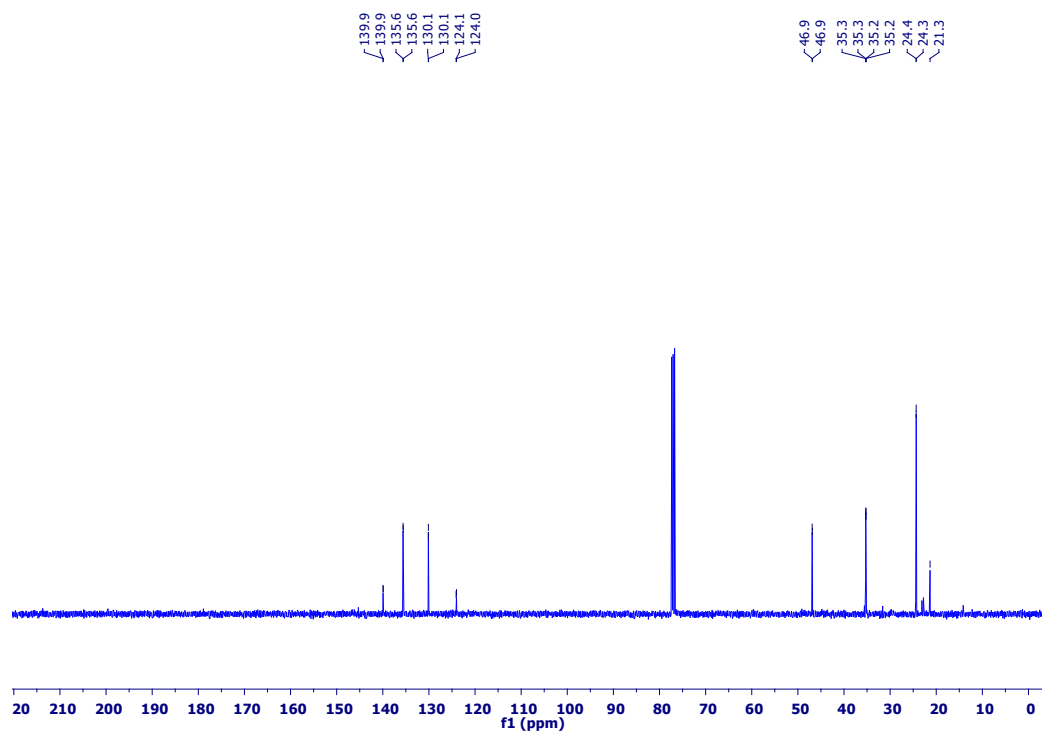
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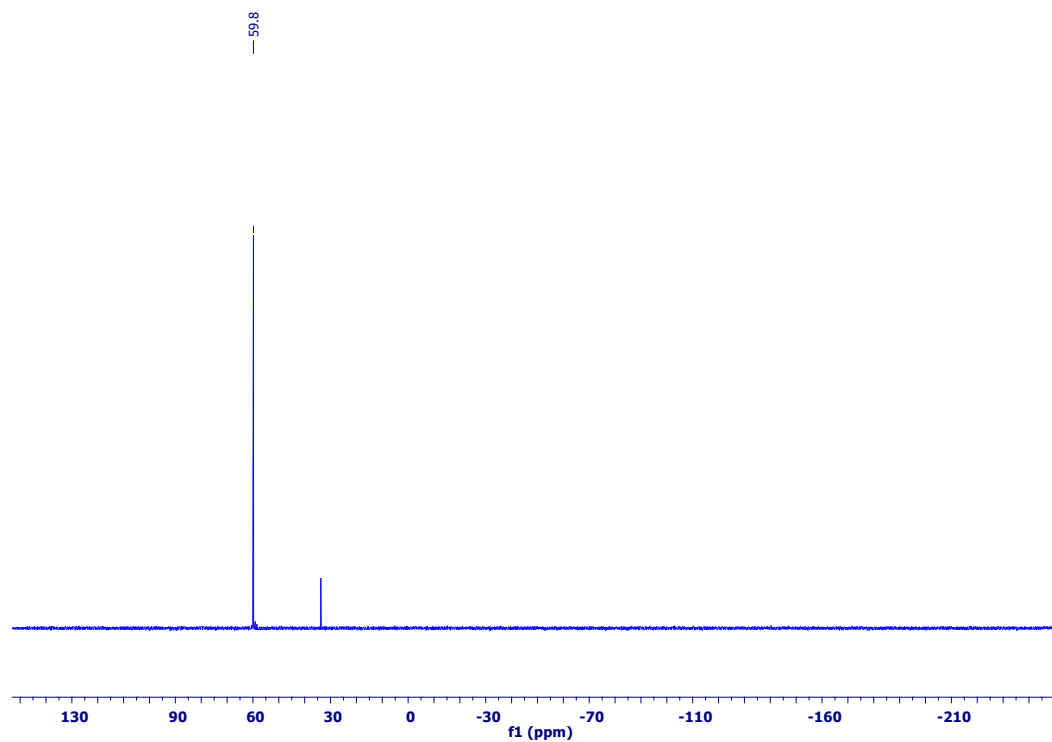
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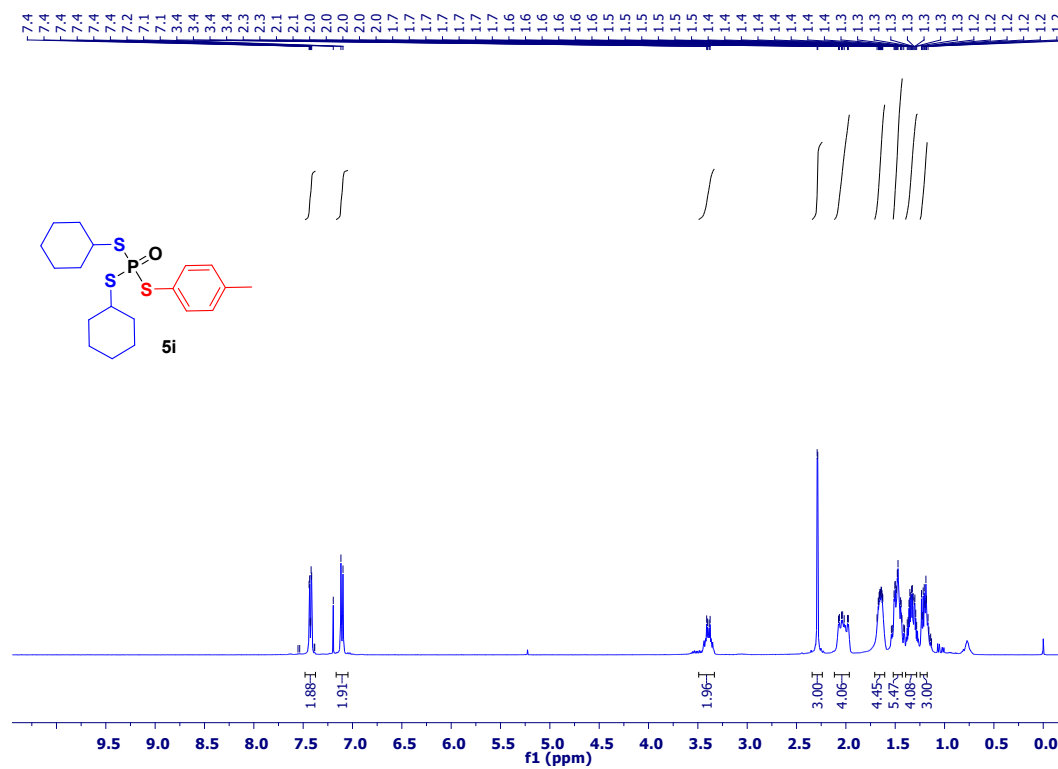
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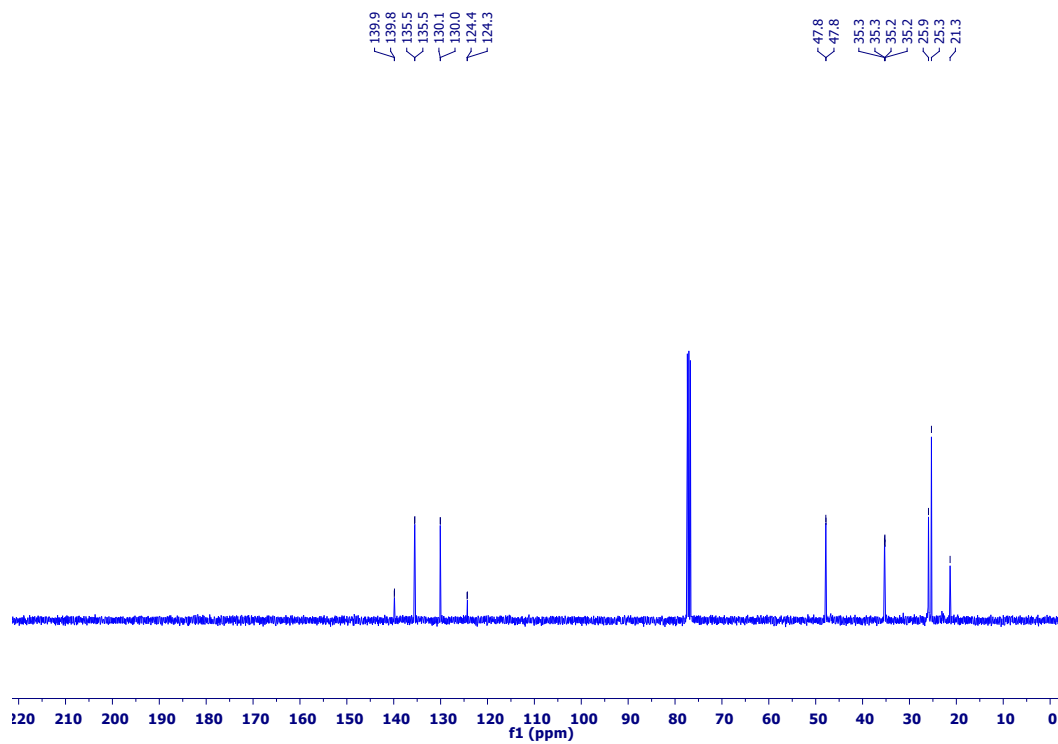
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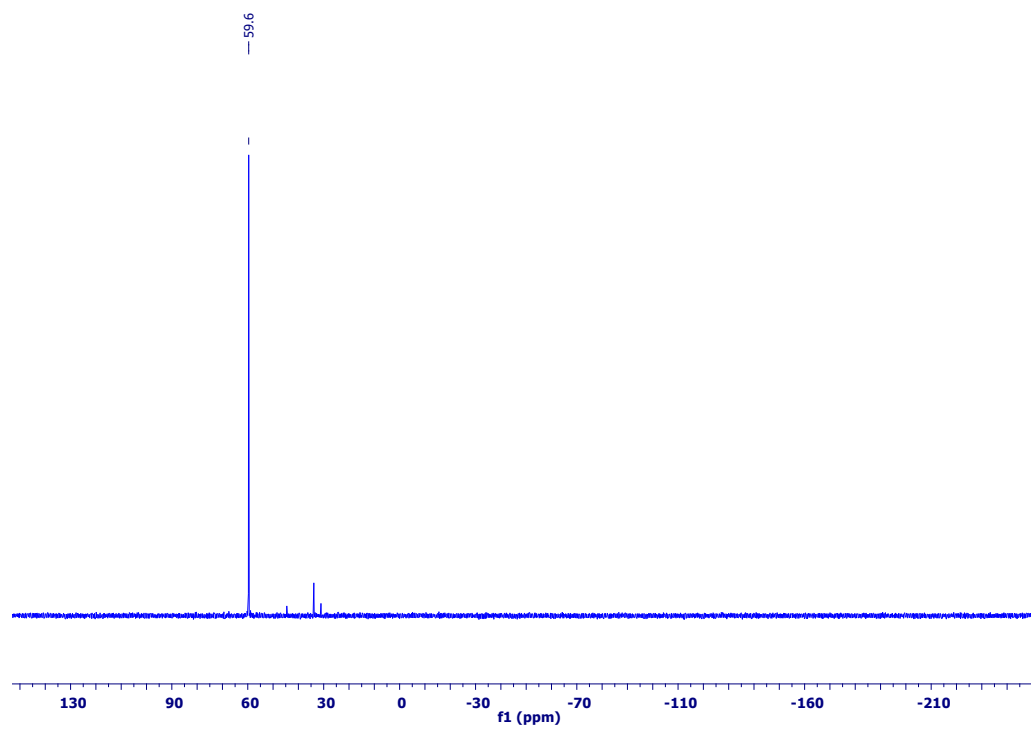
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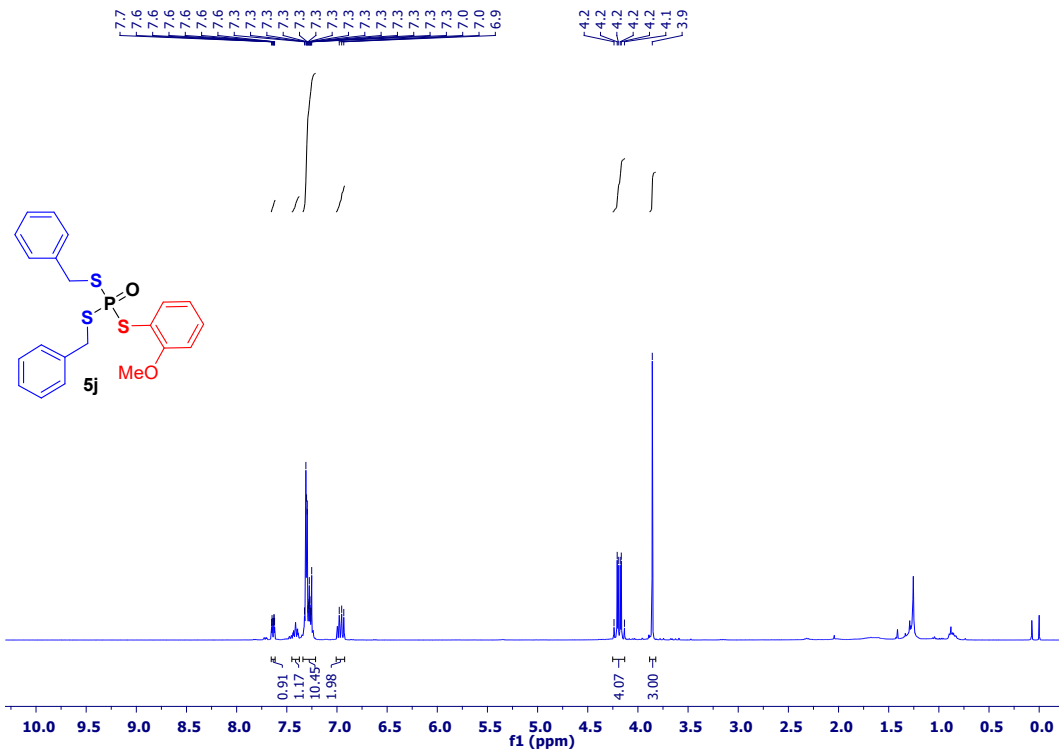
^{13}C NMR (101 MHz, CDCl_3)



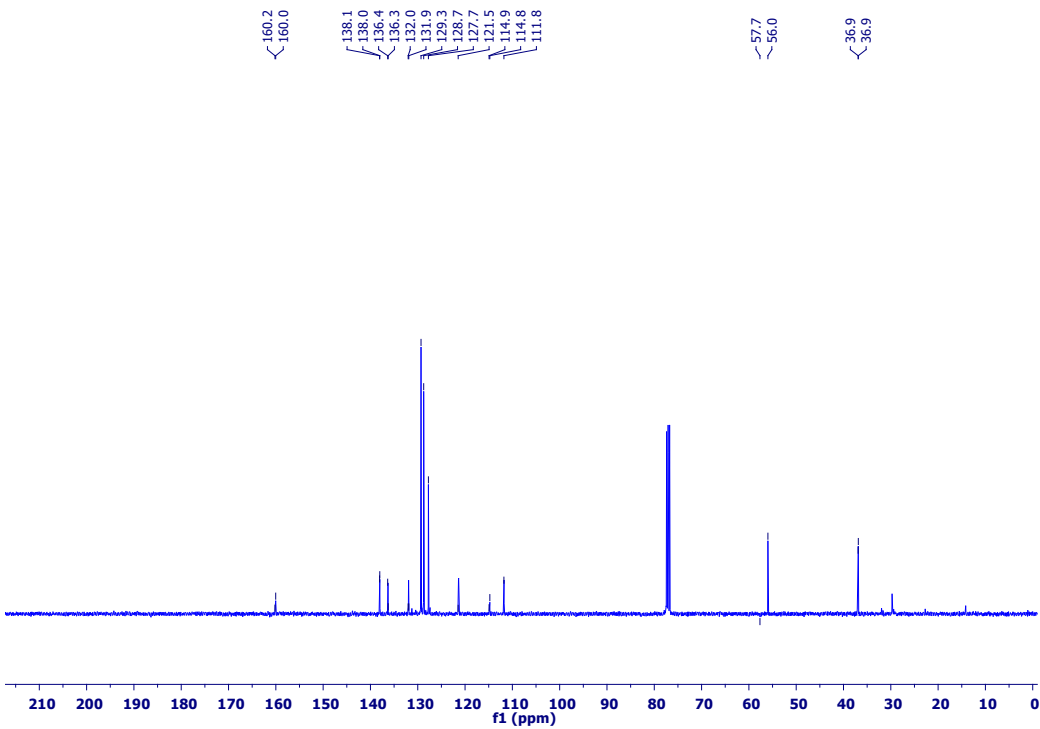
^{31}P NMR (162 MHz, CDCl_3)



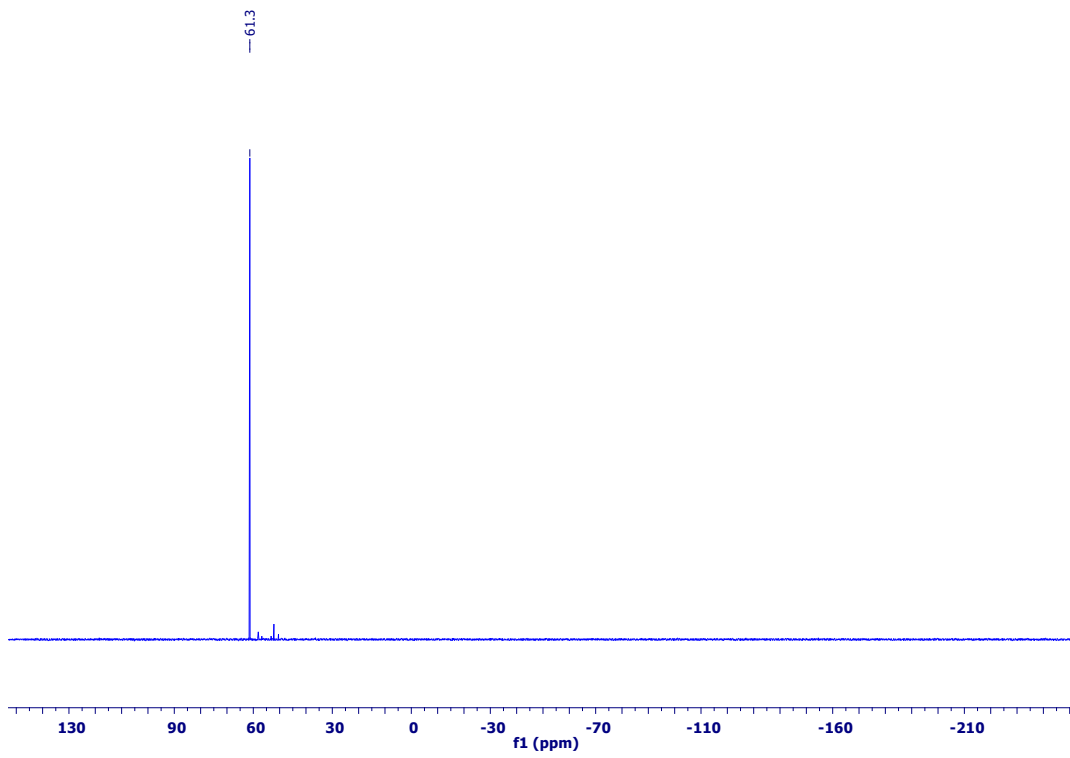
$^1\text{H NMR}$ (400 MHz, CDCl_3)



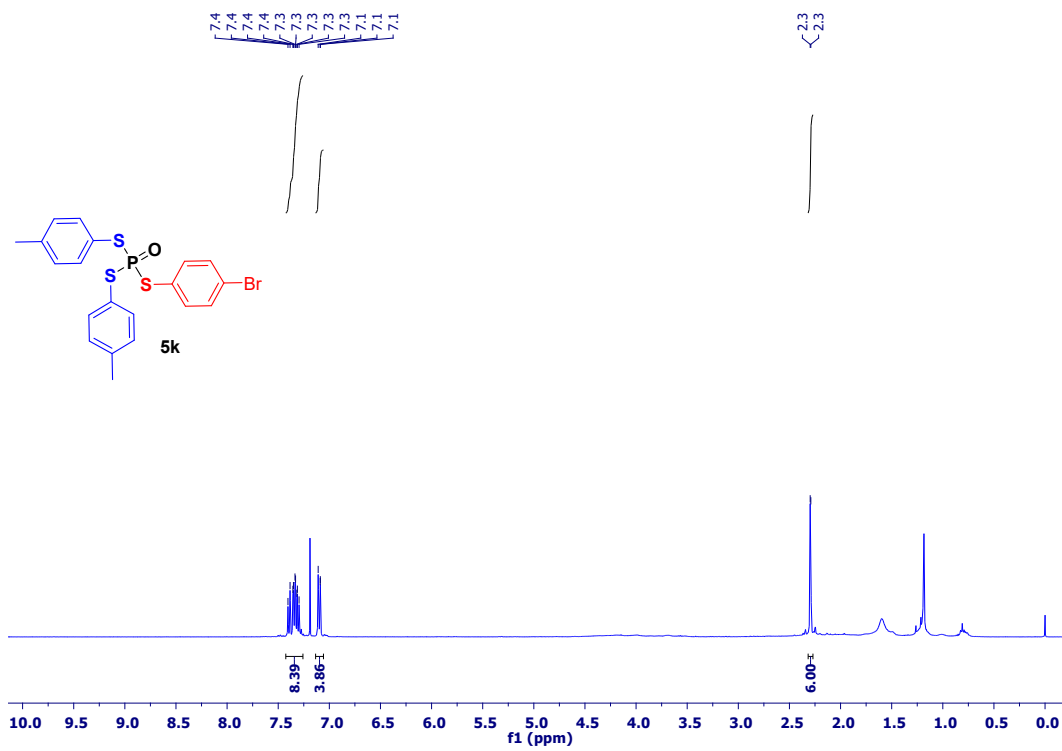
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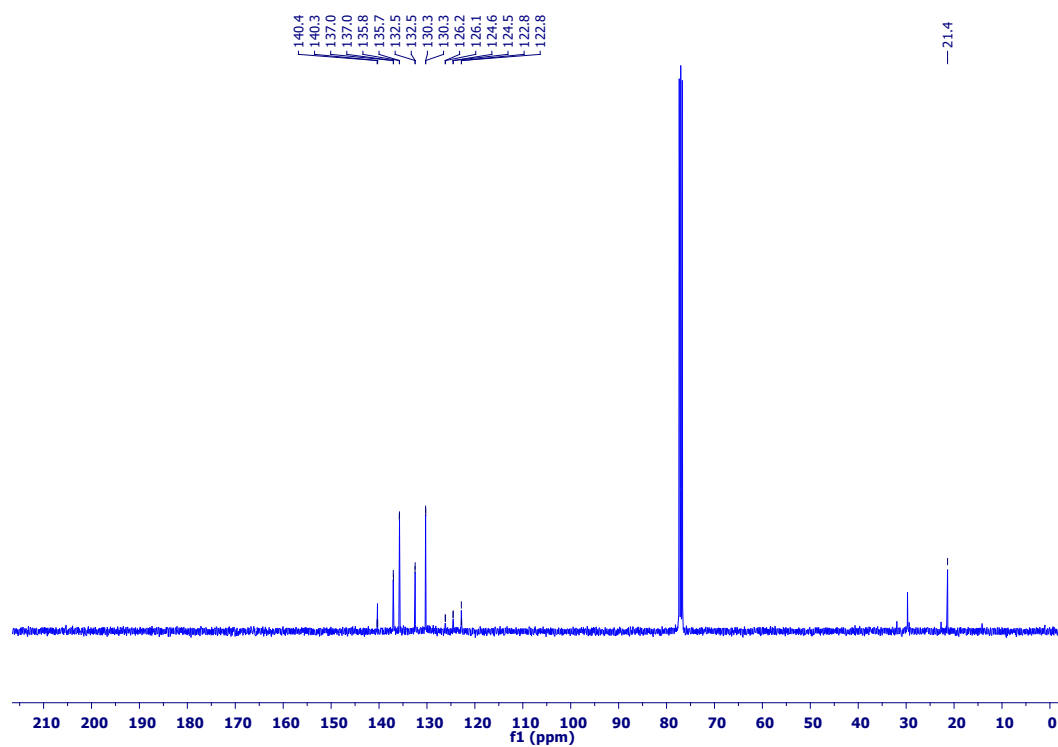
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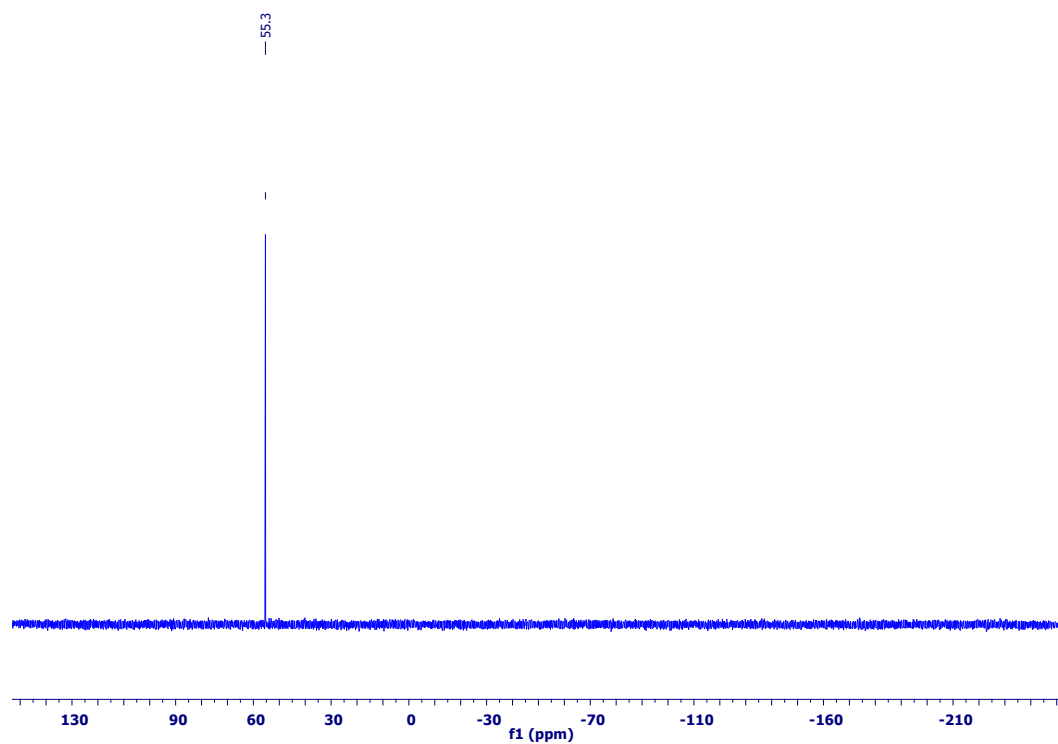
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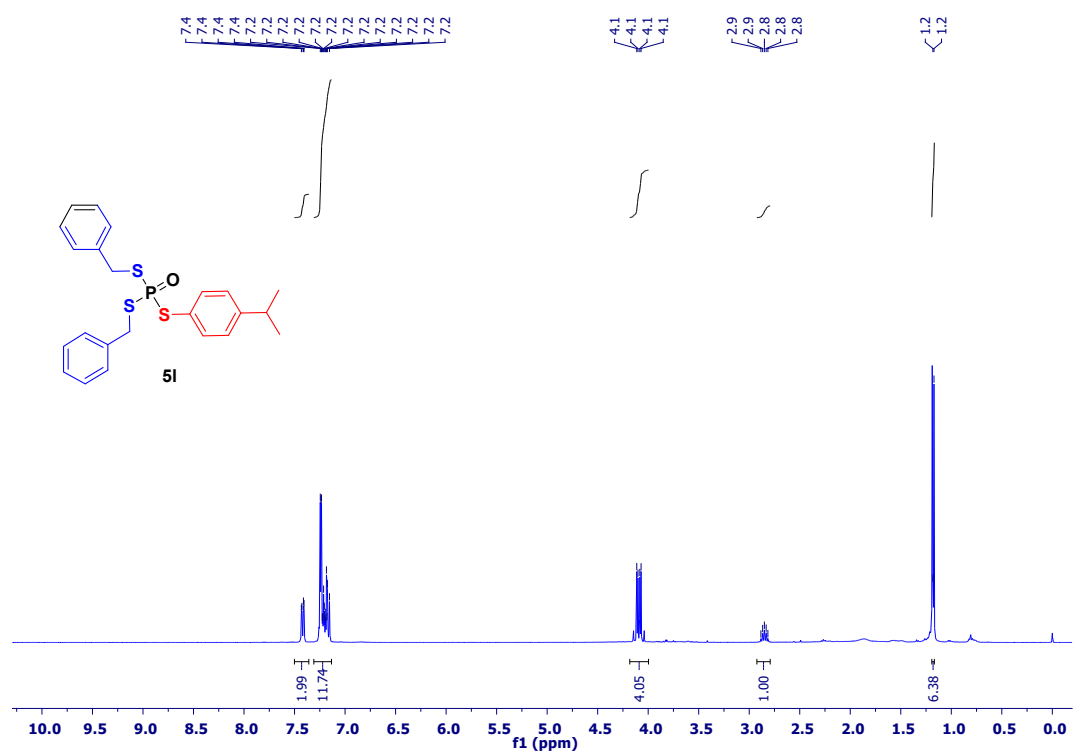
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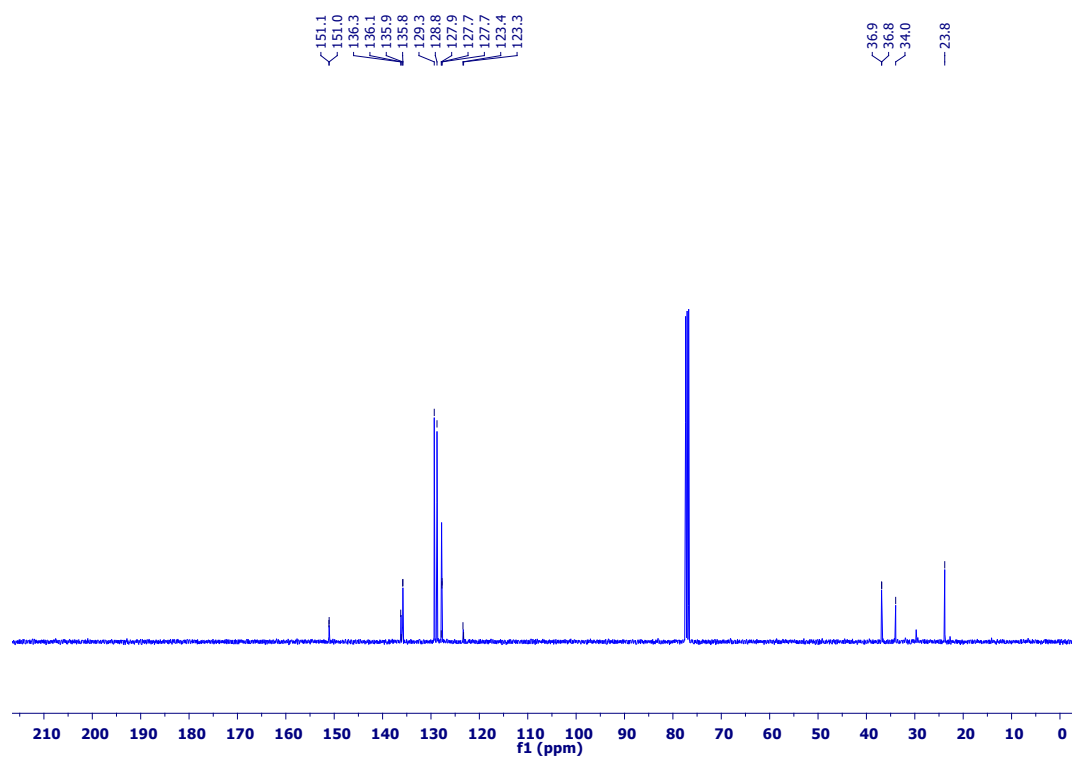
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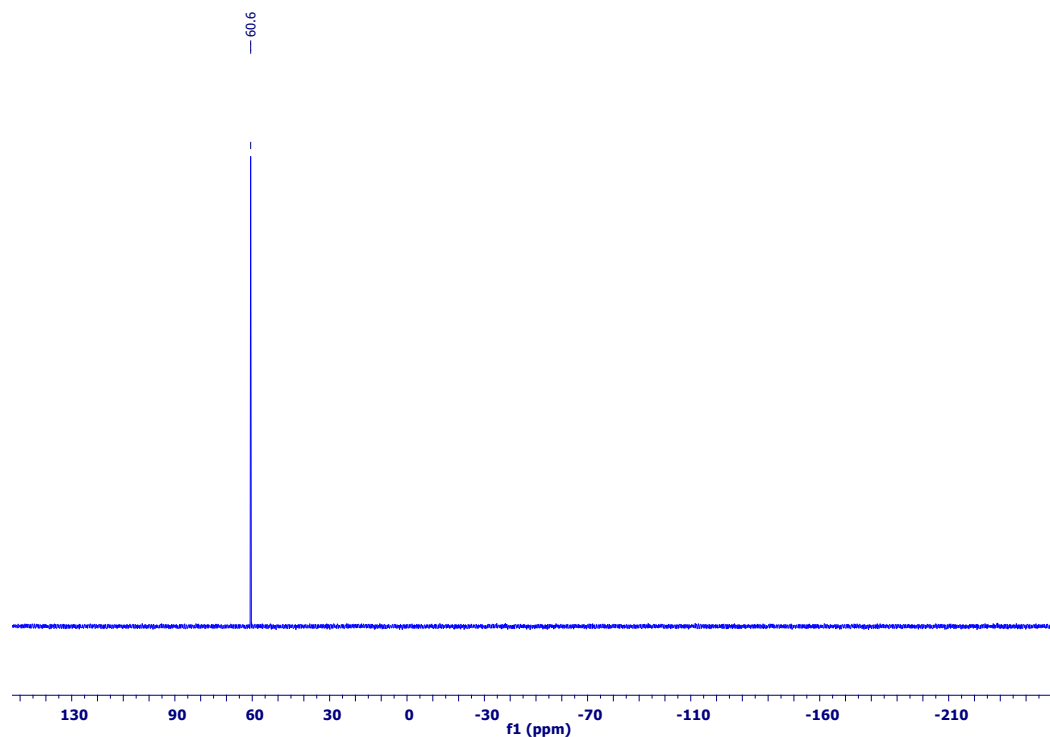
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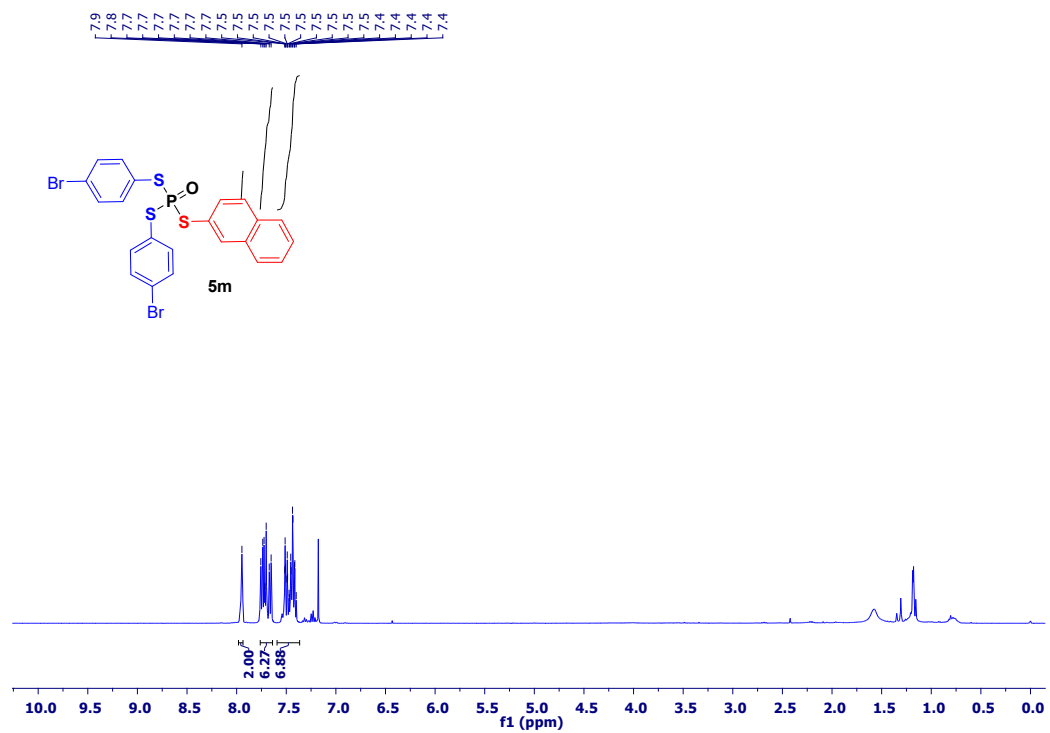
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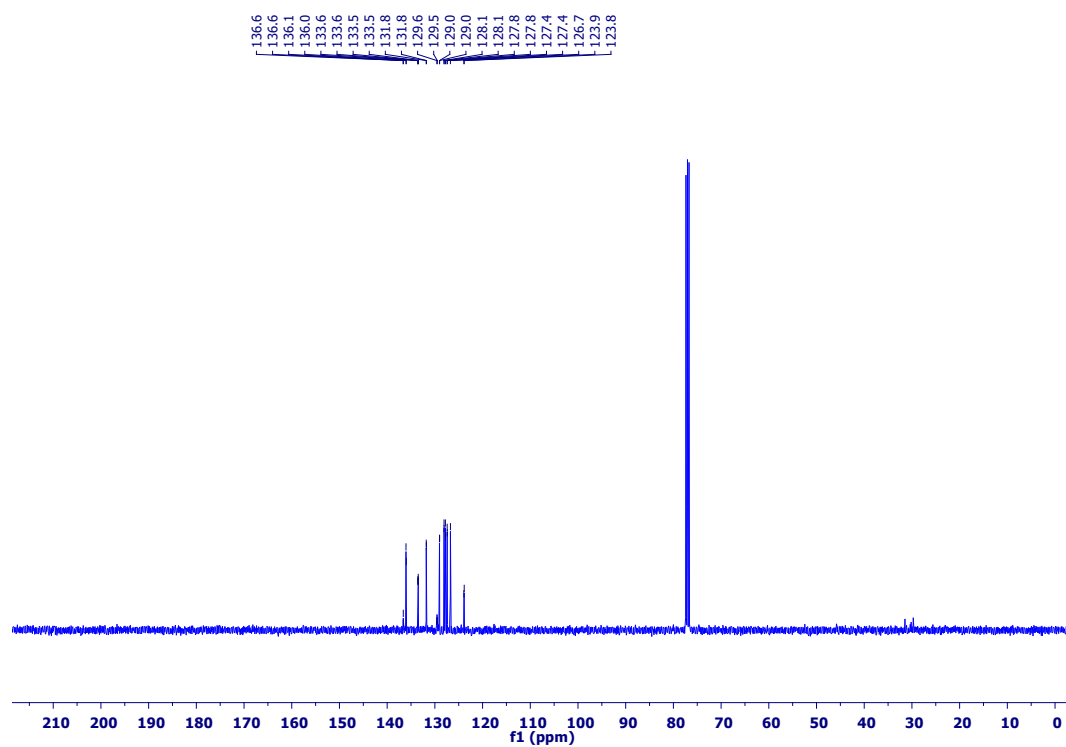
³¹P NMR (162 MHz, CDCl₃)



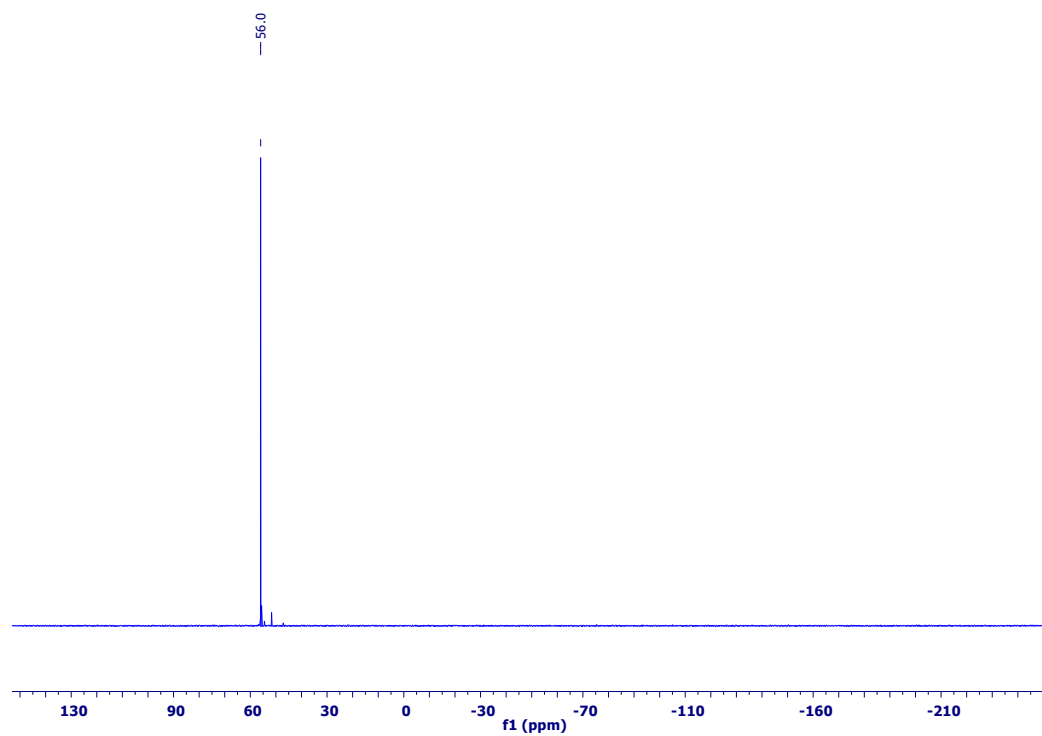
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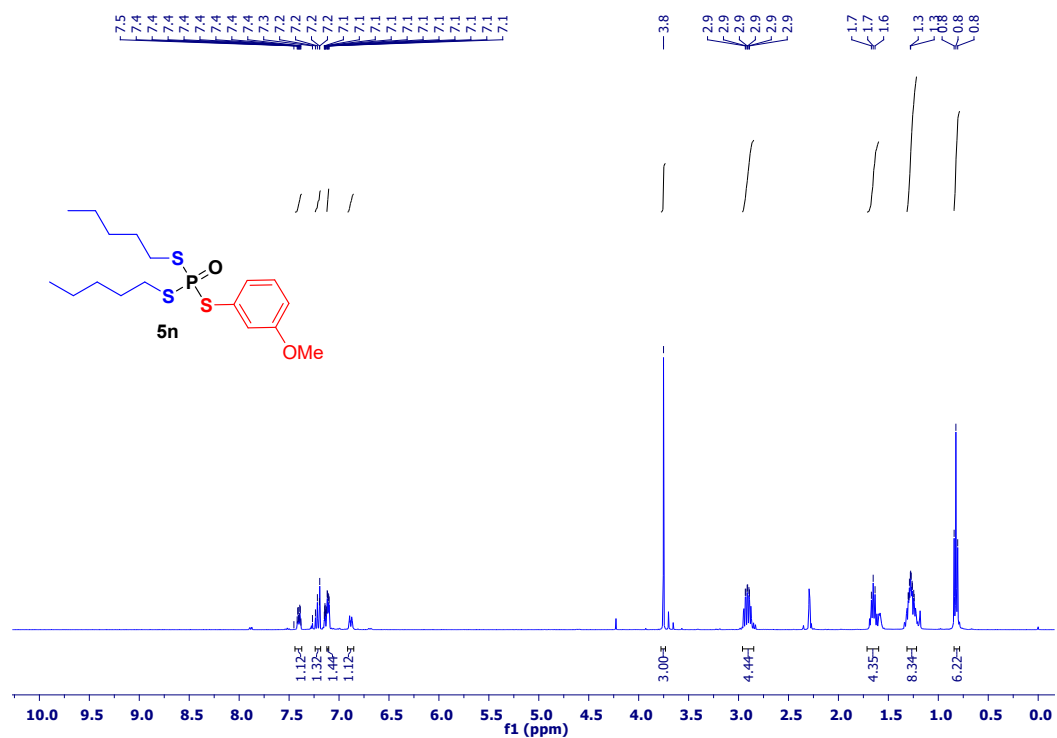
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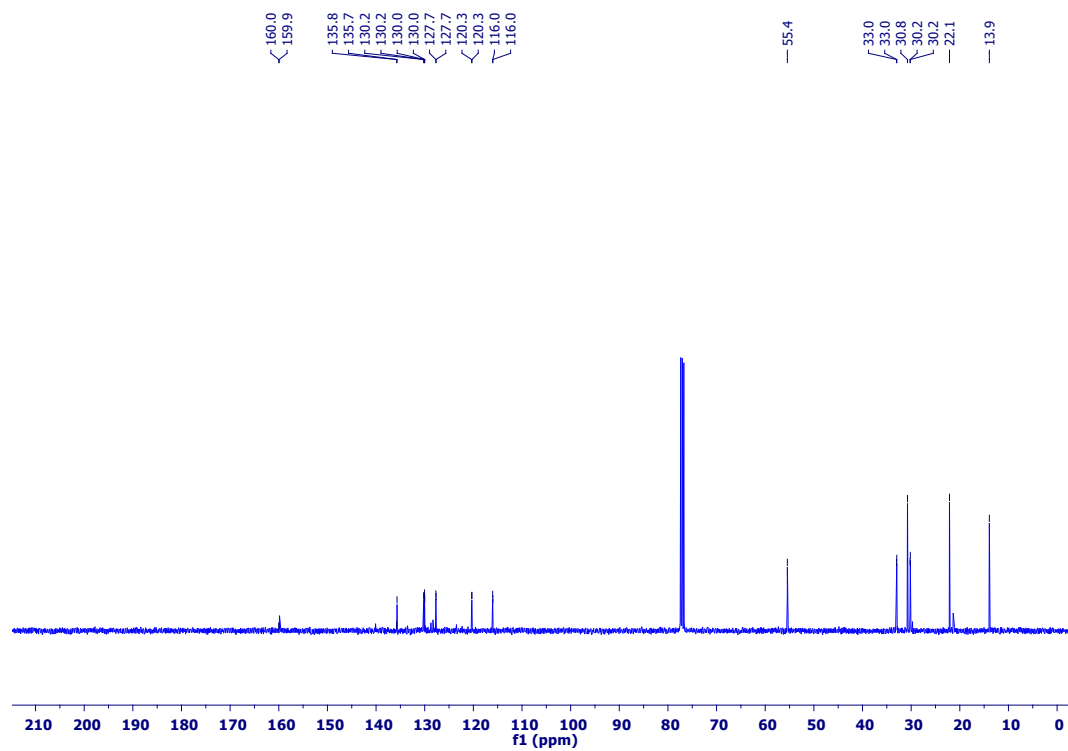
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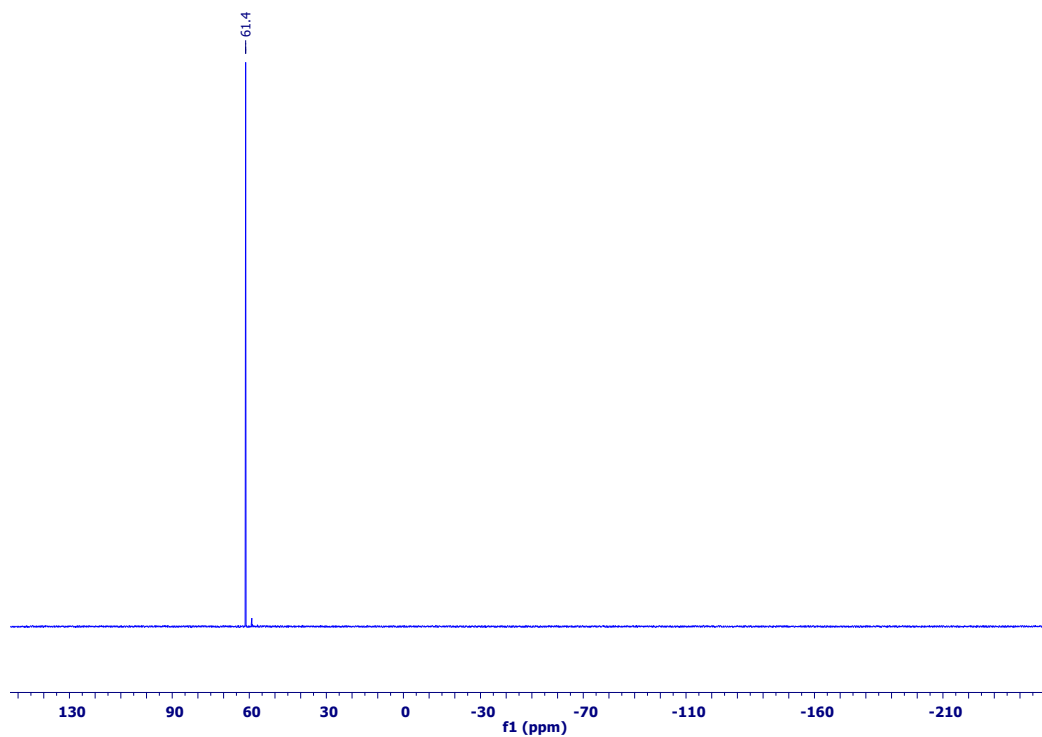
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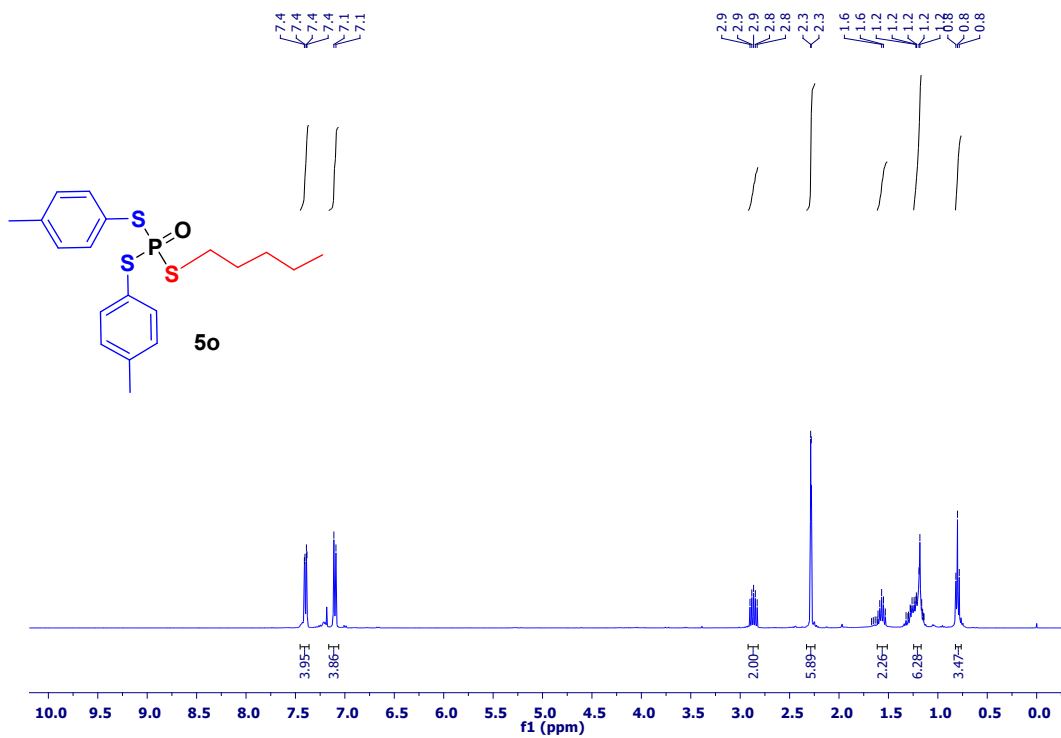
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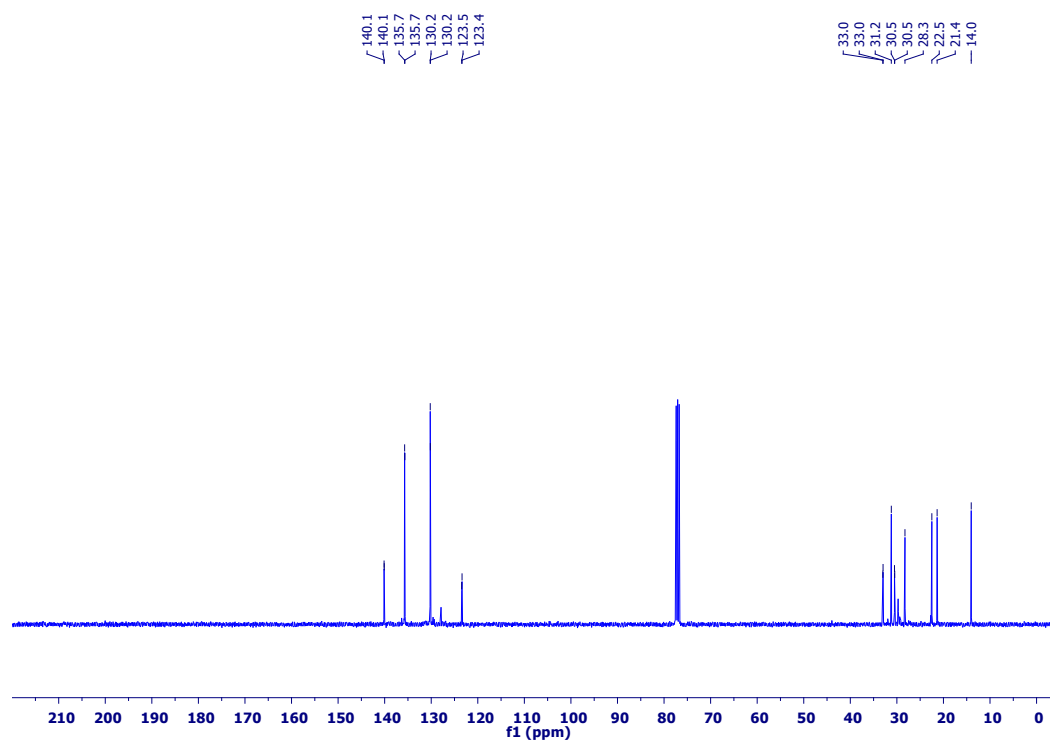
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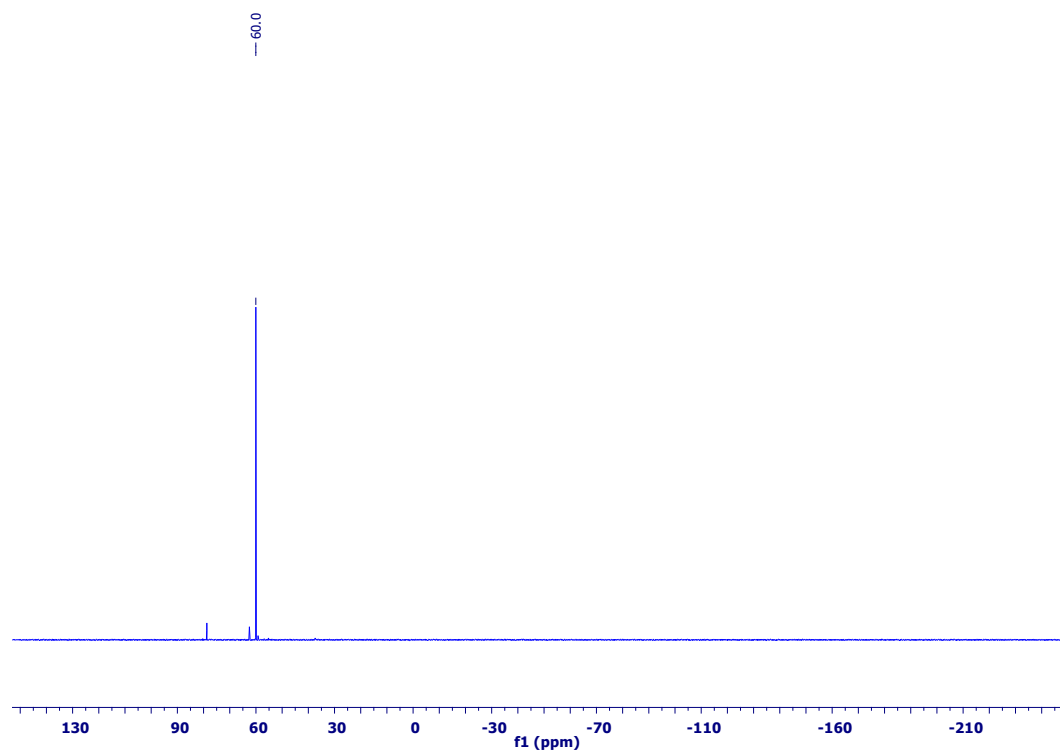
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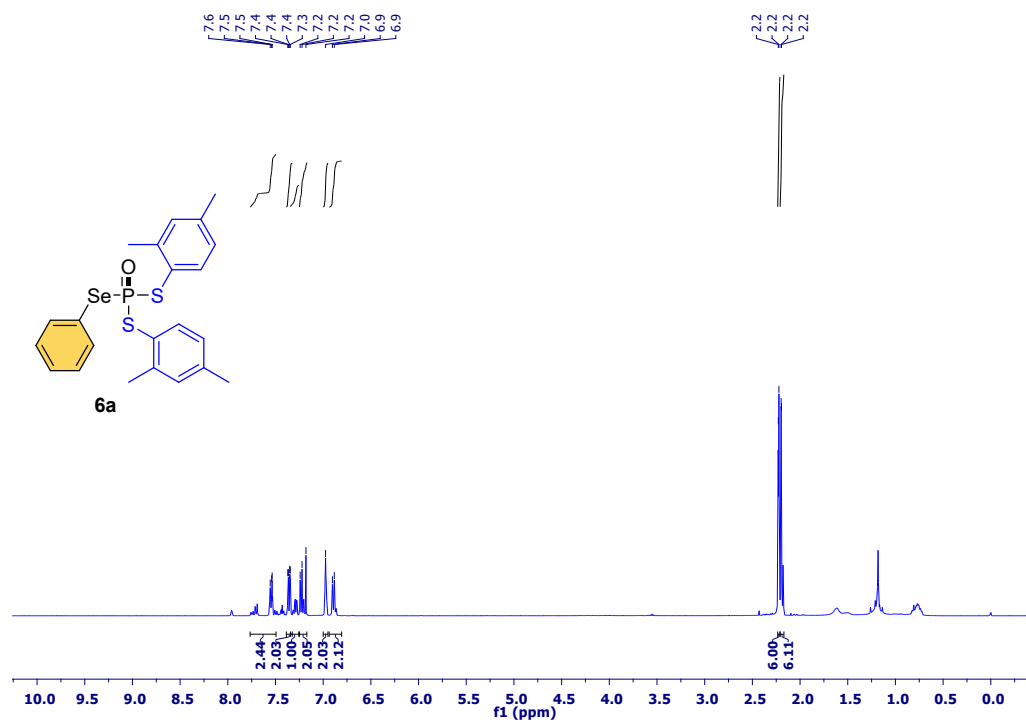
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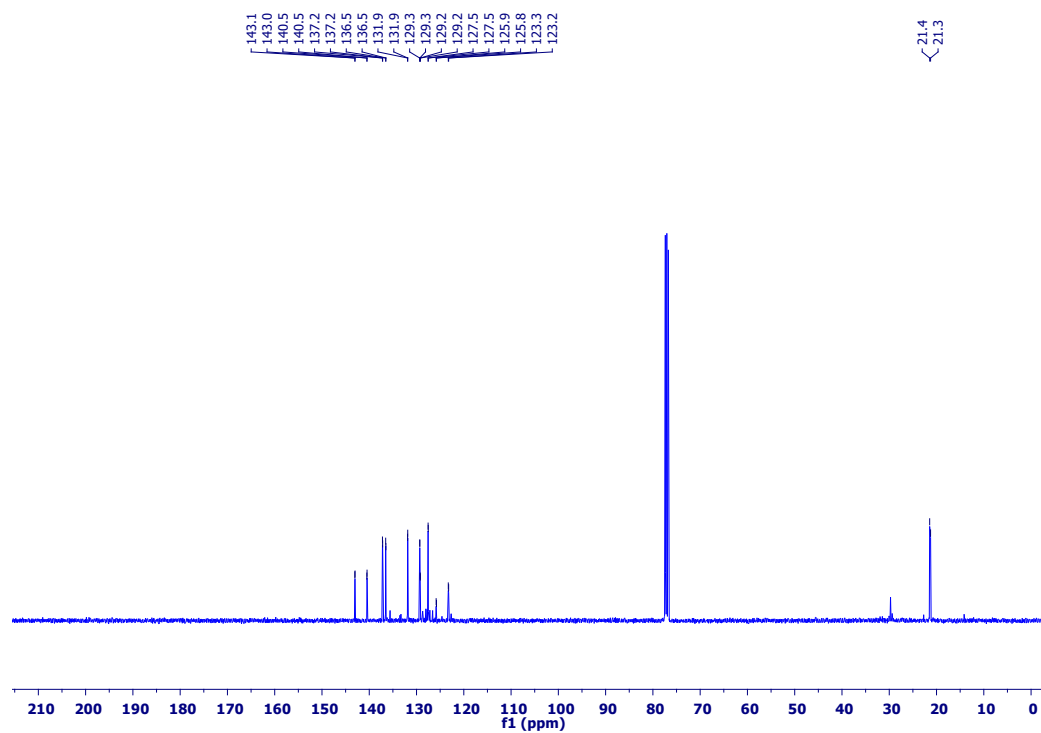
^{31}P NMR (162 MHz, CDCl_3)



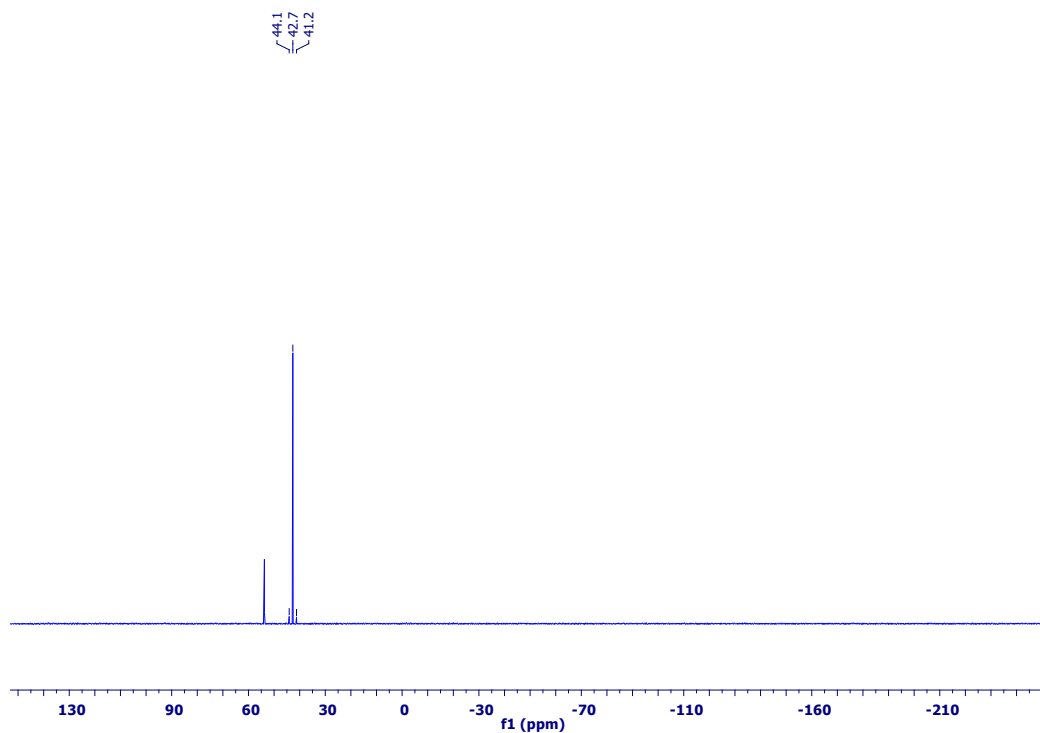
^1H NMR (400 MHz, CDCl_3)



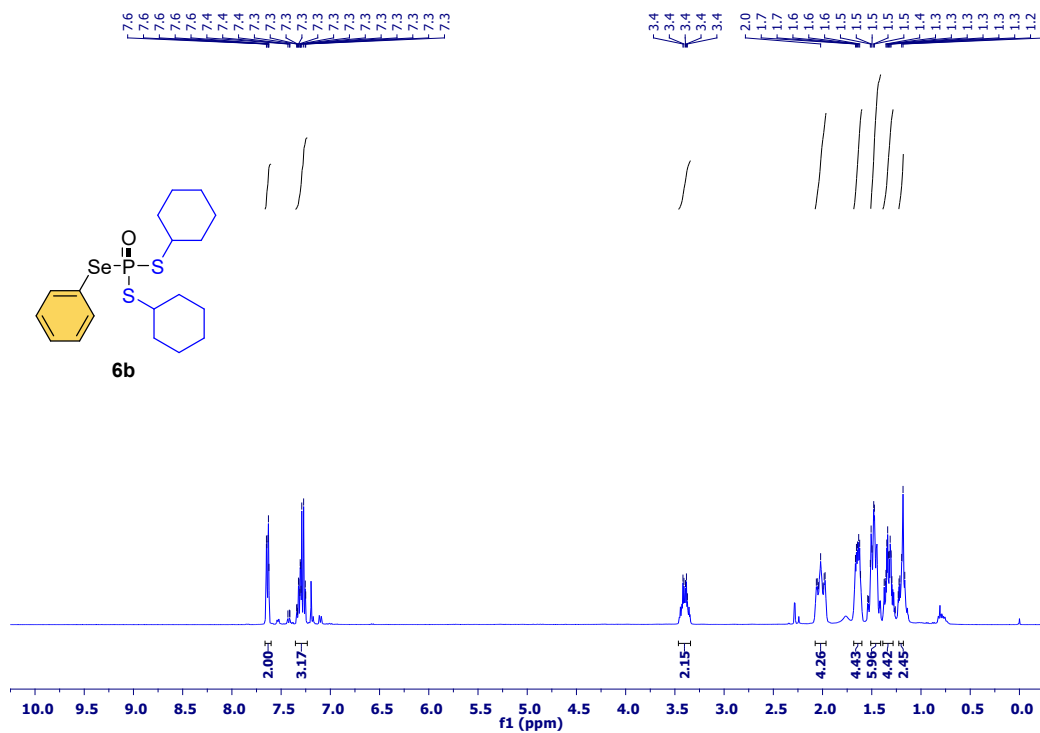
^{13}C NMR (101 MHz, CDCl_3)



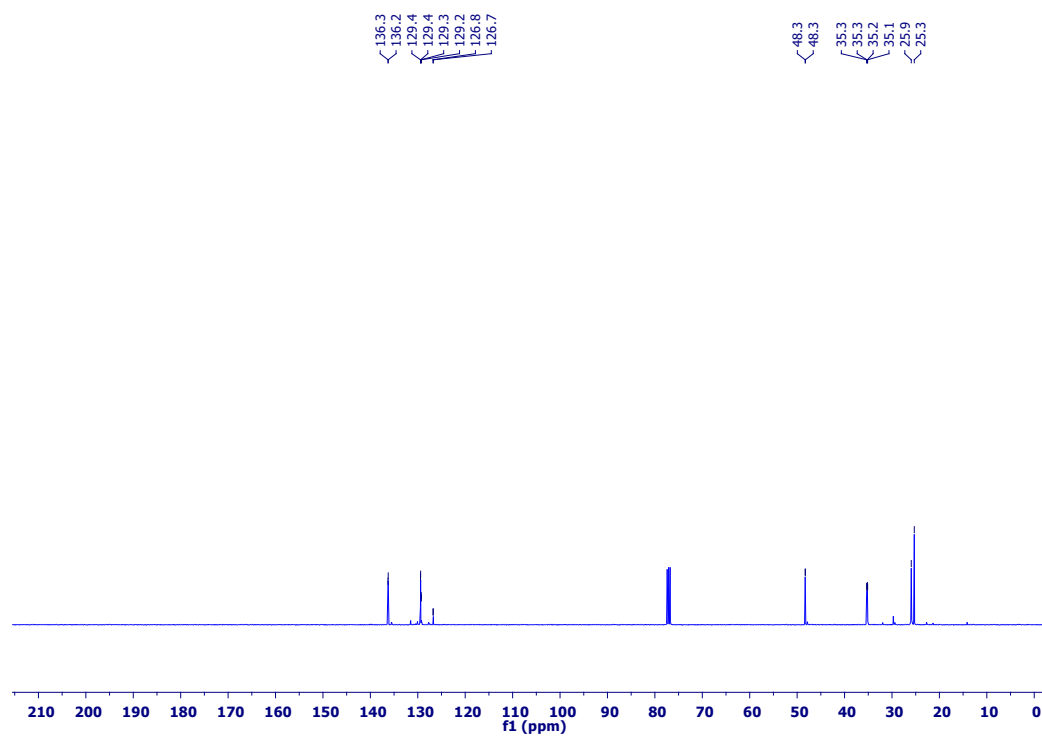
^{31}P NMR (162 MHz, CDCl_3)



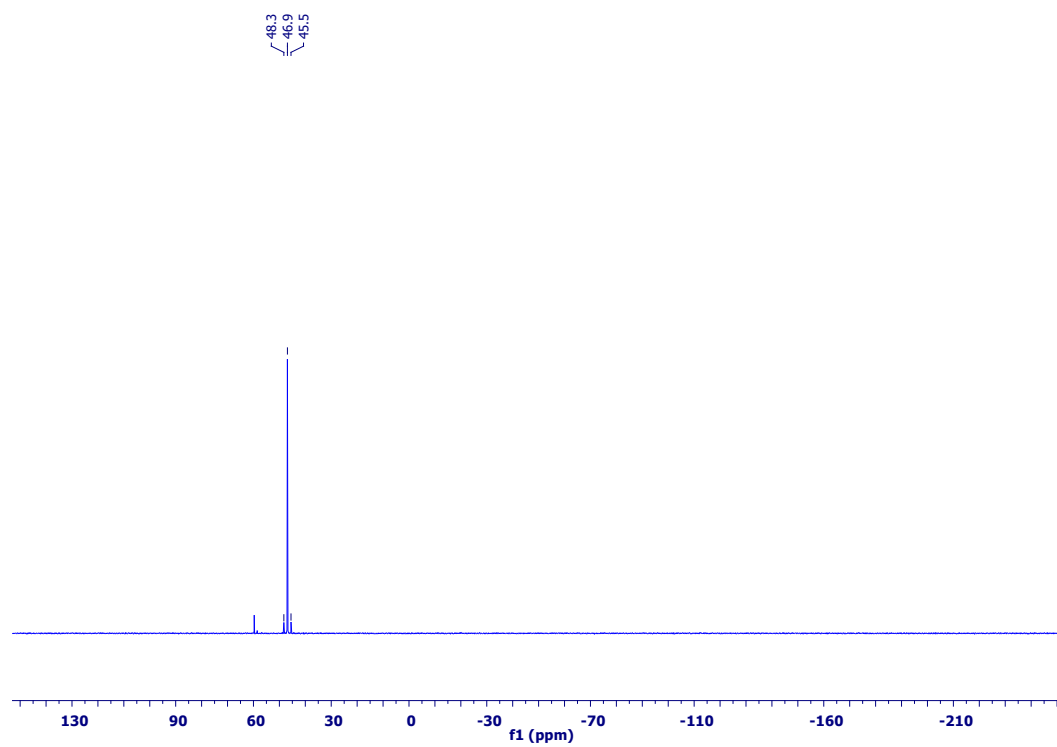
^1H NMR (400 MHz, CDCl_3)



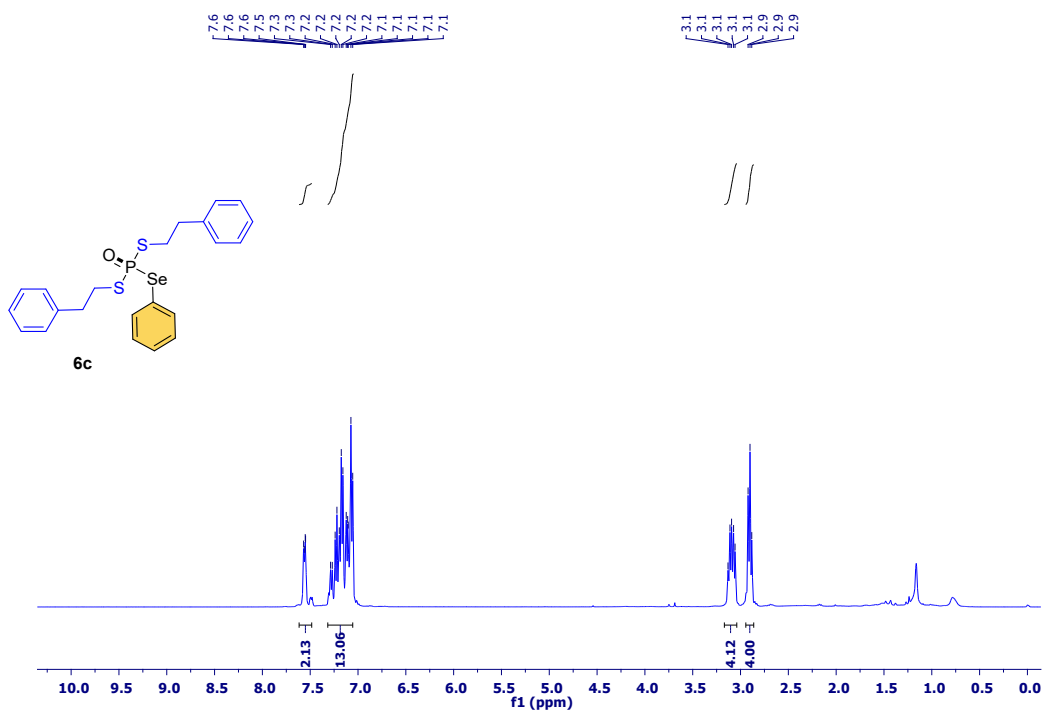
^{13}C NMR (101 MHz, CDCl_3)



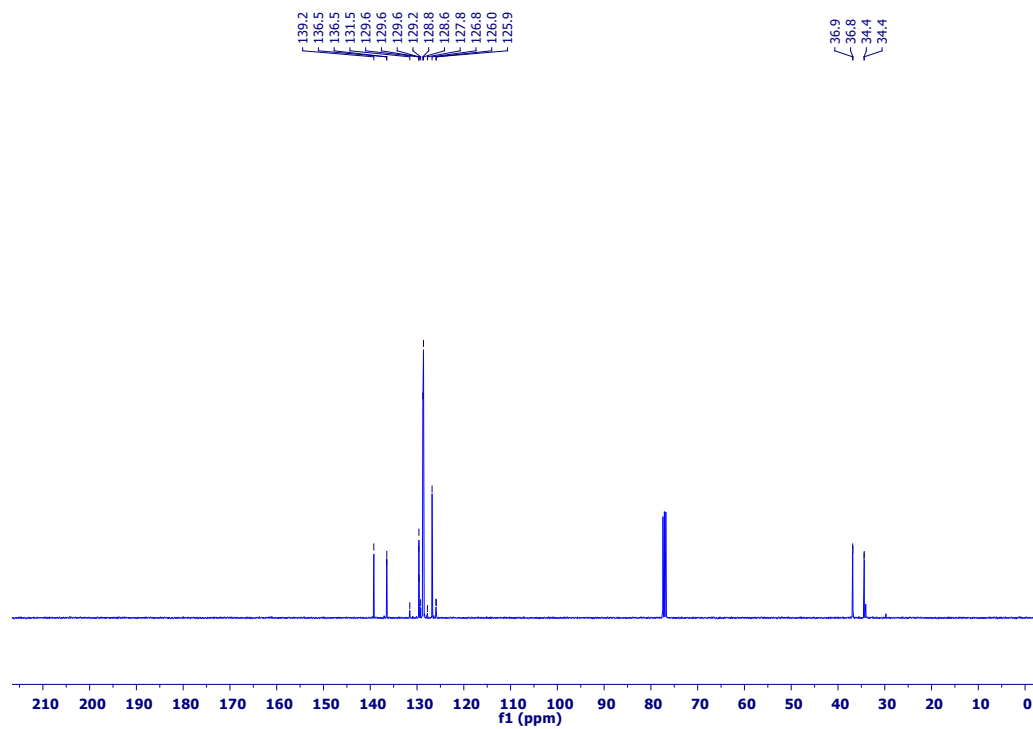
^{31}P NMR (162 MHz, CDCl_3)



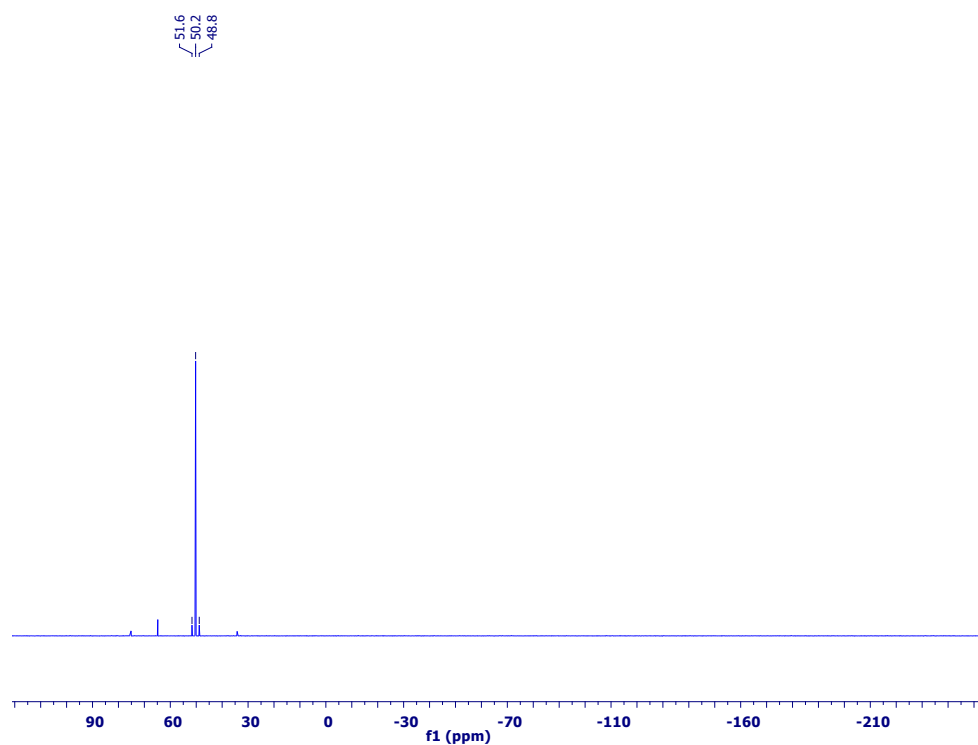
¹H NMR (400 MHz, CDCl₃)



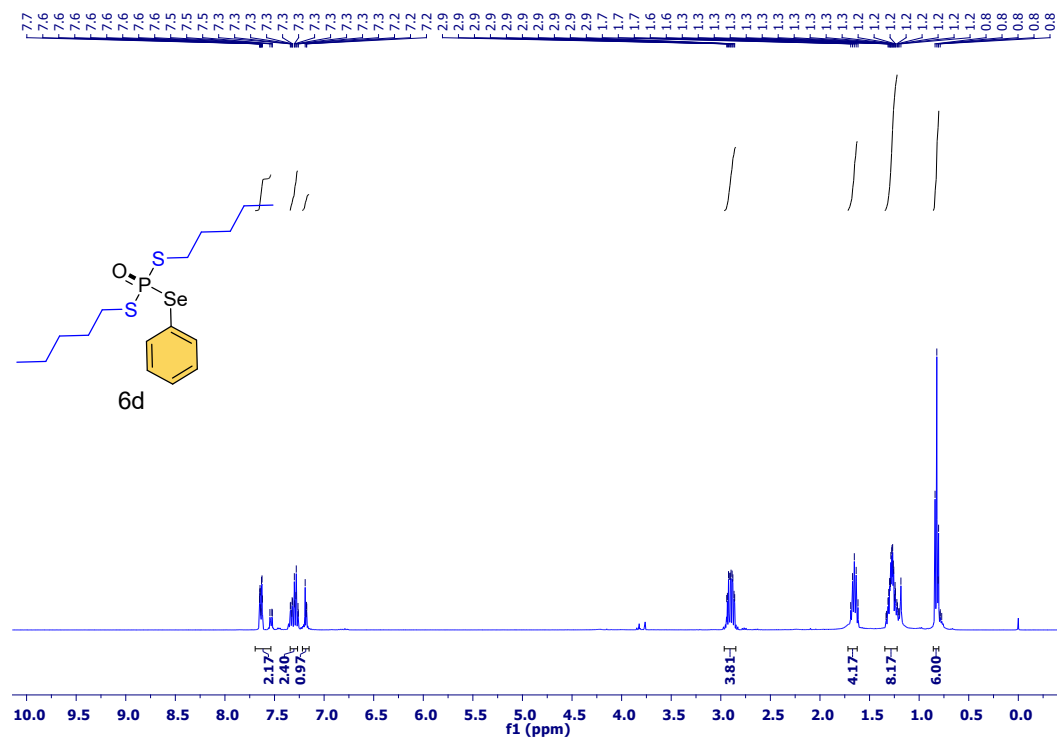
¹³C NMR (101 MHz, CDCl₃)



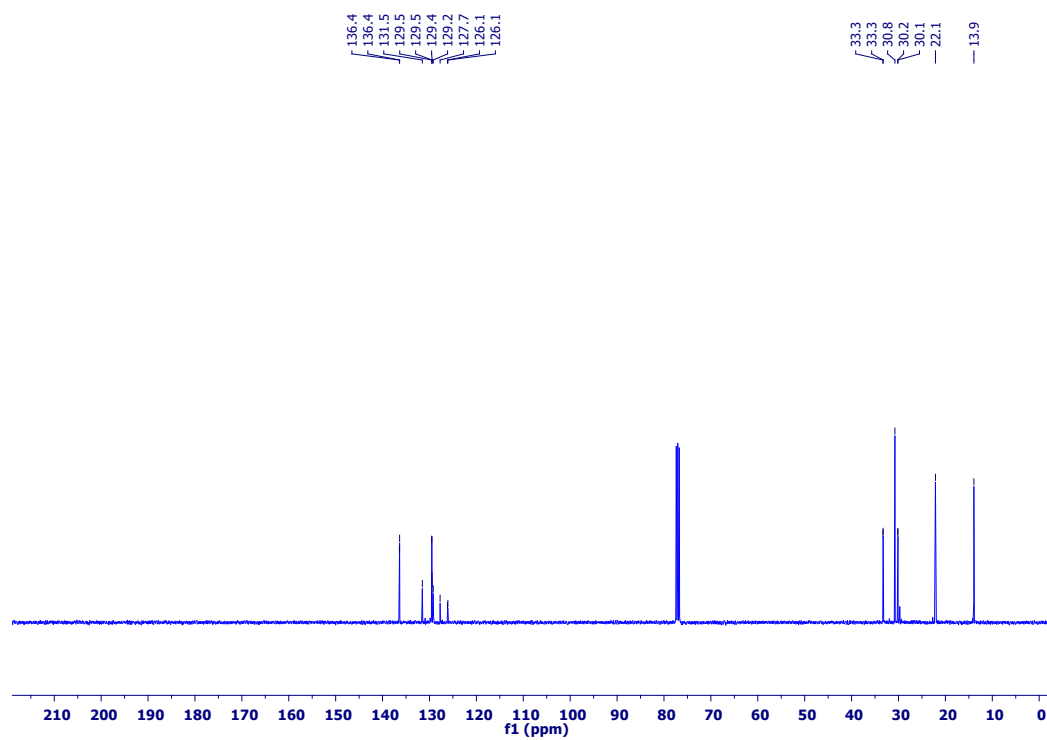
^{31}P NMR (162 MHz, CDCl_3)



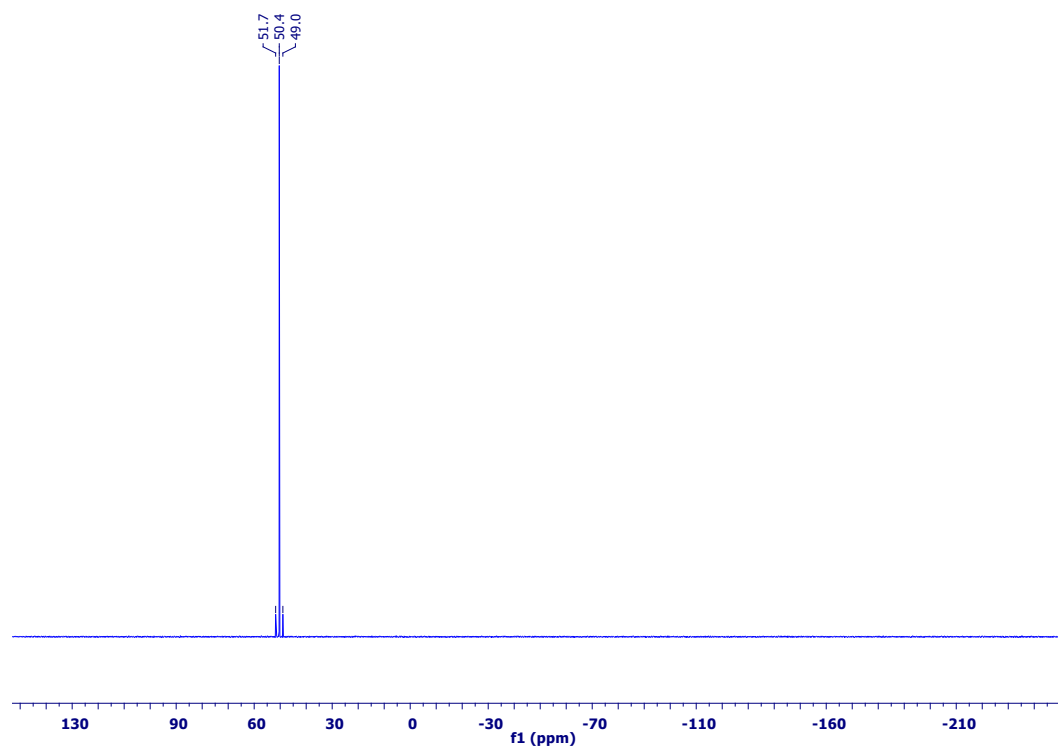
^1H NMR (400 MHz, CDCl_3)



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

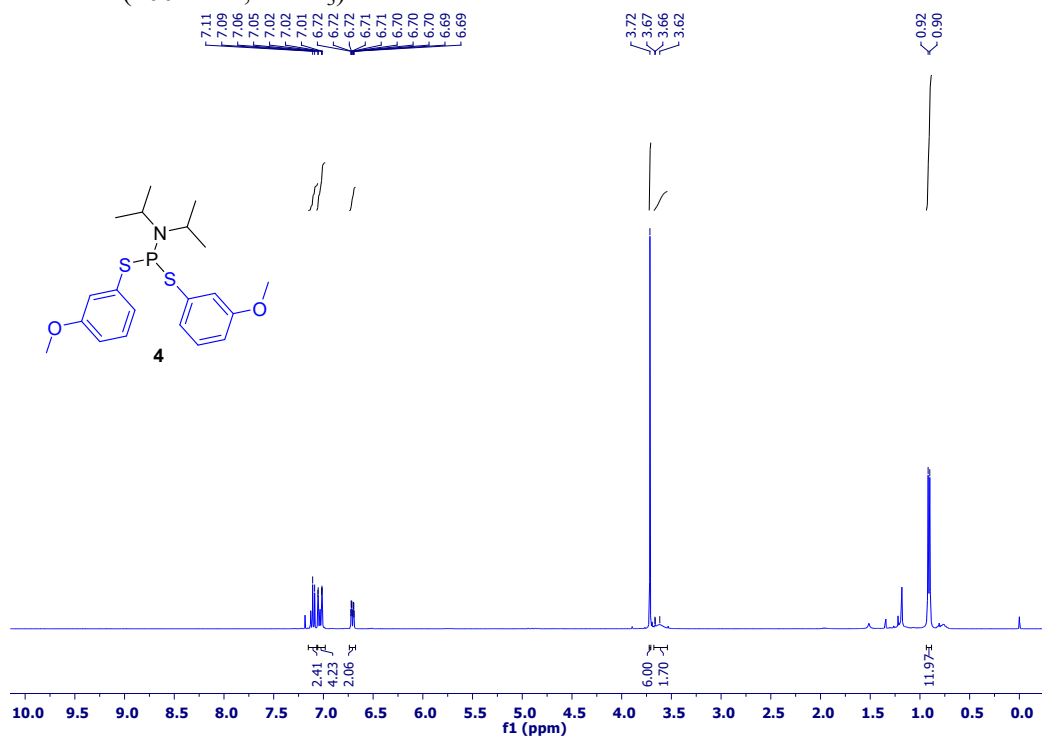


^{31}P NMR (162 MHz, CDCl_3)

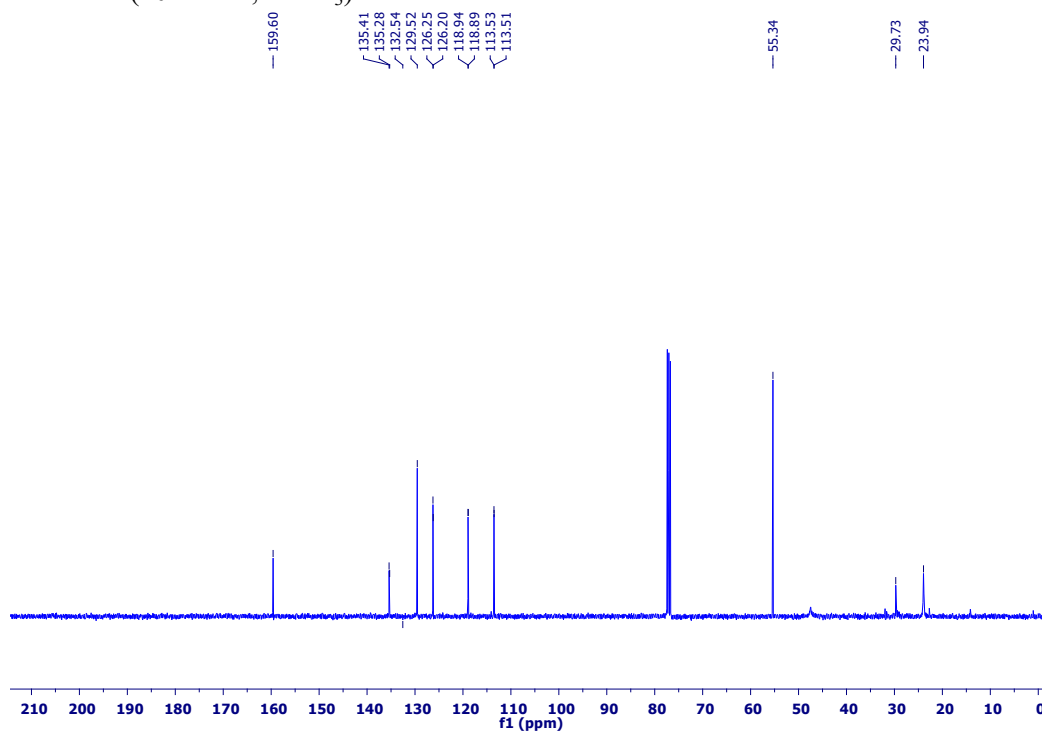


bis(3-methoxyphenyl) diisopropylphosphoramidodithioite

$^1\text{H NMR}$ (400 MHz, CDCl_3)



$^{13}\text{C NMR}$ (101 MHz, CDCl_3)



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

