

Electronic Supplementary Information

Catalyst- and solvent-free, atom and step economic synthesis of dithiophosphinates by one-pot domino introduction of sulfur atoms strategy

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Table of contents

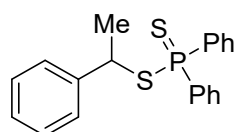
1. General experimental information.....	S2
2. Typical Procedure and Characterization of the Products.....	S3
3. Copies of the ¹ H and ¹³ C NMR Spectra of the Products.....	S16

1. General information

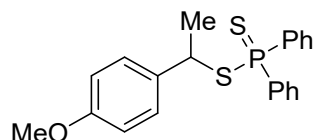
Unless otherwise noted, all chemicals were purchased and used without further purification and all reactions were carried out in sealed Schlenk tubes under N₂ and then monitored by TLC and/or GC-MS. ¹H-, ¹³C and ³¹P NMR spectra were measured on a JNM-ECZ600R/S3 (Jeol, Japan) (600, 151 and 243 MHz for ¹H-, ¹³C and ³¹P NMR, respectively) using CDCl₃ as the solvent. Chemical shifts for ¹H-, ¹³C and ³¹P NMR were referred to internal Me₄Si (0 ppm) as the standard. Mass spectra were measured on an Agilent GC-MS-7890A/5975C Plus spectrometer (EI). HRMS were recorded on a LC-TOF spectrometer (Xevo G2-XS QToF) using ESI techniques.

2. Typical Procedure and Characterization of the Products

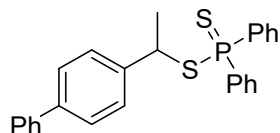
Typical procedure for Catalyst-Free Domino-Multicomponent Synthesis of Dithiophosphinates from Diphenylphosphine, S₈ and Alkenes or Alkynes: The mixture of Ph₂PH (174 μL, 1.0 mol), S₈ (64 mg, 0.25 mmol) and **1a** (115 μL, 1.0 mmol) sealed under N₂ in a 10 mL Schlenk tube was heated at 70 °C for 12 h. After cooling down to room temperature. The reaction mixture was analyzed by TLC/GC-MS and purified by flash column chromatography on silica gel using ethyl acetate and petroleum ether (0~ 1/10) as the eluent, giving **2a** in 83% isolated yield.



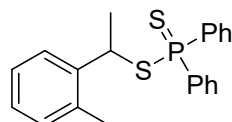
1-Phenylethyl diphenylphosphinodithioate (2a), colorless oil. ¹H NMR (600 MHz, CDCl₃) δ 7.98 – 7.89 (m, 2H), 7.80 – 7.70 (m, 2H), 7.52 – 7.47 (m, 1H), 7.47 – 7.42 (m, 2H), 7.40 – 7.35 (m, 1H), 7.31 – 7.22 (m, 4H), 7.18 – 7.13 (m, 2H), 7.13 – 7.09 (m, 1H), 4.71 (dq, *J* = 12.6, 7.2 Hz, 1H), 1.67 (d, *J* = 7.2 Hz, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 142.9 (d, *J* = 4.3 Hz), 135.2, 134.7, 134.1, 133.5, 131.9 (d, *J* = 2.6 Hz), 131.7, 131.6, 131.6 (d, *J* = 2.6 Hz), 131.5, 131.4, 128.7, 128.6, 128.5, 128.4, 128.3, 127.5, 127.4, 46.9, 24.1 (d, *J* = 4.8 Hz). ³¹P NMR (243 MHz, CDCl₃) δ 63.1. HRMS (ESI) for C₂₀H₂₀PS₂ (M+H)⁺: Calcd: 355.0744; found: 355.0739.



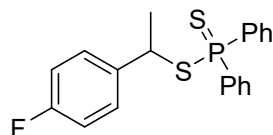
1-(4-Methoxyphenyl)ethyl diphenylphosphinodithioate (2b), colorless oil. ¹H NMR (600 MHz, CDCl₃) δ 7.92 (dd, *J* = 14.4, 7.2 Hz, 2H), 7.75 (dd, *J* = 14.4, 7.2 Hz, 2H), 7.52 – 7.47 (m, 1H), 7.46 – 7.41 (m, 2H), 7.41 – 7.36 (m, 1H), 7.32 – 7.27 (m, 2H), 7.17 (d, *J* = 8.4 Hz, 2H), 6.67 (d, *J* = 8.4 Hz, 2H), 4.70 (dq, *J* = 14.4, 7.2 Hz, 1H), 3.73 (s, 3H), 1.65 (d, *J* = 7.2 Hz, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 158.8, 135.3, 134.9 (d, *J* = 4.4 Hz), 134.7, 134.2, 133.6, 131.9 (d, *J* = 2.4 Hz), 131.7 (d, *J* = 11.4 Hz), 131.4 (d, *J* = 11.0 Hz), 128.6, 128.5, 128.3 (d, *J* = 13.2 Hz), 113.8, 55.3, 46.6, 24.1 (d, *J* = 4.4 Hz). ³¹P NMR (243 MHz, CDCl₃) δ 62.6. HRMS (ESI) for C₂₁H₂₂OPS₂ (M+H)⁺: Calcd: 385.0850; found: 385.0843.



1-([1,1'-Biphenyl]-4-yl)ethyl diphenylphosphinodithioate (2c), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.94 – 7.88 (m, 2H), 7.78 – 7.72 (m, 2H), 7.50 – 7.46 (m, 3H), 7.45 – 7.40 (m, 4H), 7.36 – 7.31 (m, 4H), 7.29 (d, $J = 8.4$ Hz, 2H), 7.26 – 7.21 (m, 2H), 4.83 – 4.71 (m, 1H), 1.71 (d, $J = 7.2$ Hz, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 141.9 (d, $J = 3.9$ Hz), 140.9, 140.3, 135.3, 134.7, 133.8, 133.3, 131.9, 131.8 (d, $J = 11.1$ Hz), 131.4, 131.4, 131.3, 128.8, 128.7 (d, $J = 13.1$ Hz), 128.3 (d, $J = 13.4$ Hz), 127.9, 127.4, 127.1 (d, $J = 5.7$ Hz), 46.6, 23.9 (d, $J = 5.4$ Hz). ^{31}P NMR (243 MHz, CDCl_3) δ 63.0. HRMS (ESI) for $\text{C}_{26}\text{H}_{24}\text{PS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 431.1057; found: 431.1051.

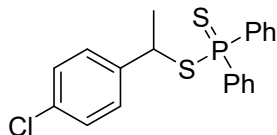


1-(o-Tolyl)ethyl diphenylphosphinodithioate (2d), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 8.07 – 8.01 (m, 2H), 7.75 (dd, $J = 14.4, 7.2$ Hz, 2H), 7.54 – 7.45 (m, 3H), 7.42 – 7.34 (m, 2H), 7.33 – 7.28 (m, 2H), 7.14 – 7.07 (m, 2H), 7.04 (d, $J = 7.2$ Hz, 1H), 4.93 (dq, $J = 13.8, 7.2$ Hz, 1H), 2.33 (s, 3H), 1.58 (d, $J = 7.2$ Hz, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 140.6 (d, $J = 6.1$ Hz), 135.5, 135.2, 134.9, 134.7, 134.3, 131.9 (d, $J = 2.6$ Hz), 131.8, 131.7, 131.4 (d, $J = 11.4$ Hz), 130.6, 128.6, 128.5, 128.5, 128.4, 127.4 (d, $J = 25.6$ Hz), 126.3, 43.6, 24.0, 19.7. ^{31}P NMR (243 MHz, CDCl_3) δ 62.6. HRMS (ESI) for $\text{C}_{21}\text{H}_{22}\text{PS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 369.0901; found: 369.0911.

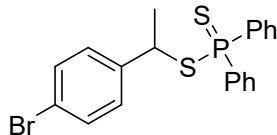


1-(4-Fluorophenyl)ethyl diphenylphosphinodithioate (2e), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.93 – 7.85 (m, 2H), 7.72 (dd, $J = 14.4, 7.2$ Hz, 2H), 7.52 – 7.47 (m, 1H), 7.47 – 7.42 (m, 2H), 7.42 – 7.37 (m, 1H), 7.31 – 7.26 (m, 2H), 7.23 – 7.17 (m, 2H), 6.78 (t, $J = 8.4$ Hz, 2H), 4.73 (dq, $J = 14.4, 7.2$ Hz, 1H), 1.66 (d, $J = 7.2$ Hz, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 161.9 (d, $J = 246.5$ Hz), 138.7, 135.1, 134.6, 133.7, 133.1, 132.0 (d, $J = 2.5$ Hz), 131.8 (d, $J = 11.3$ Hz), 131.6 (d, $J = 2.6$ Hz), 131.3 (d, $J =$

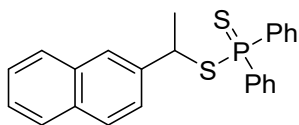
11.4 Hz), 129.1 (d, $J = 7.7$ Hz), 128.7 (d, $J = 13.1$ Hz), 128.3 (d, $J = 13.3$ Hz), 115.2 (d, $J = 21.3$ Hz), 46.1, 24.1 (d, $J = 5.6$ Hz). ^{31}P NMR (243 MHz, CDCl_3) δ 62.9. HRMS (ESI) for $\text{C}_{20}\text{H}_{19}\text{FPS}_2(\text{M}+\text{H})^+$: Calcd: 373.0650; found: 373.0642.



1-(4-Chlorophenyl)ethyl diphenylphosphinodithioate (2f), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.88 (dd, $J = 14.4, 7.2$ Hz, 2H), 7.71 (dd, $J = 14.4, 7.2$ Hz, 2H), 7.51 – 7.47 (m, 1H), 7.46 – 7.38 (m, 3H), 7.31 – 7.26 (m, 2H), 7.15 (d, $J = 8.4$ Hz, 2H), 7.04 (d, $J = 8.4$ Hz, 2H), 4.70 (dq, $J = 14.4, 7.2$ Hz, 1H), 1.66 (d, $J = 7.2$ Hz, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 141.4 (d, $J = 2.8$ Hz), 135.1, 134.5, 133.5, 133.0, 132.9, 132.0 (d, $J = 2.4$ Hz), 131.8 (d, $J = 11.2$ Hz), 131.5 (d, $J = 2.6$ Hz), 131.3 (d, $J = 11.1$ Hz), 128.9, 128.7 (d, $J = 13.7$ Hz), 128.4, 128.3 (d, $J = 13.2$ Hz), 46.0, 23.8 (d, $J = 5.7$ Hz). ^{31}P NMR (243 MHz, CDCl_3) δ 63.1. HRMS (ESI) for $\text{C}_{20}\text{H}_{19}\text{ClPS}_2(\text{M}+\text{H})^+$: Calcd: 389.0354; found: 389.0347.

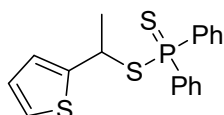


1-(4-Bromophenyl)ethyl diphenylphosphinodithioate (2g), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.91 – 7.84 (m, 2H), 7.73 – 7.66 (m, 2H), 7.51 – 7.47 (m, 1H), 7.46 – 7.39 (m, 3H), 7.31 – 7.26 (m, 2H), 7.19 (d, $J = 8.4$ Hz, 2H), 7.08 (d, $J = 8.4$ Hz, 2H), 4.69 (dq, $J = 14.4, 7.2$ Hz, 1H), 1.66 (d, $J = 7.2$ Hz, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 141.9 (d, $J = 3.4$ Hz), 135.1, 134.5, 133.4, 132.8, 132.0, 131.8 (d, $J = 11.5$ Hz), 131.5 (d, $J = 2.5$ Hz), 131.4, 131.3 (d, $J = 11.4$ Hz), 129.2, 128.7 (d, $J = 13.1$ Hz), 128.3 (d, $J = 13.2$ Hz), 121.2, 46.1, 23.7 (d, $J = 5.8$ Hz). ^{31}P NMR (243 MHz, CDCl_3) δ 63.1. HRMS (ESI) for $\text{C}_{20}\text{H}_{19}\text{BrPS}_2(\text{M}+\text{H})^+$: Calcd: 432.9849; found: 432.9838.

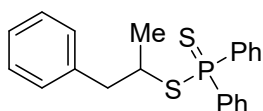


1-(Naphthalen-2-yl)ethyl diphenylphosphinodithioate (2h), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.91 (dd, $J = 14.4, 7.2$ Hz, 2H), 7.73 – 7.66 (m, 3H), 7.65 – 7.61

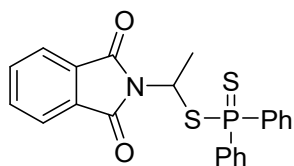
(m, 2H), 7.58 (s, 1H), 7.50 – 7.45 (m, 1H), 7.45 – 7.36 (m, 5H), 7.16 – 7.11 (m, 1H), 7.11 – 7.04 (m, 2H), 4.89 (dq, $J = 14.4, 7.2$ Hz, 1H), 1.77 (d, $J = 7.2$ Hz, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 139.9 (d, $J = 3.8$ Hz), 135.3, 134.7, 133.5, 133.0, 133.0, 132.7, 131.9, 131.7 (d, $J = 11.4$ Hz), 131.4 (d, $J = 11.3$ Hz), 131.2, 128.6 (d, $J = 13.2$ Hz), 128.4, 128.1, 128.0, 127.5, 126.3, 126.0 (d, $J = 19.0$ Hz), 125.4, 47.1, 23.8 (d, $J = 4.9$ Hz). ^{31}P NMR (243 MHz, CDCl_3) δ 63.2. HRMS (ESI) for $\text{C}_{24}\text{H}_{22}\text{PS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 405.0901; found: 405.0911.



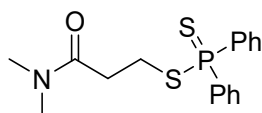
1-(Thiophen-2-yl)ethyl diphenylphosphinodithioate (2j), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.92 (dd, $J = 14.4, 7.2$ Hz, 2H), 7.82 (dd, $J = 14.4, 7.2$ Hz, 2H), 7.52 – 7.38 (m, 4H), 7.34 (td, $J = 7.8, 3.6$ Hz, 2H), 7.08 (d, $J = 5.4$ Hz, 1H), 6.86 (d, $J = 3.6$ Hz, 1H), 6.70 (dd, $J = 4.8, 3.6$ Hz, 1H), 5.03 (dq, $J = 14.4, 7.2$ Hz, 1H), 1.74 (d, $J = 7.2$ Hz, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 146.8 (d, $J = 4.6$ Hz), 135.1, 134.5, 134.1, 133.6, 132.0 (d, $J = 2.5$ Hz), 131.7, 131.6, 131.5 (d, $J = 11.4$ Hz), 128.7 (d, $J = 13.1$ Hz), 128.5 (d, $J = 13.2$ Hz), 126.5, 125.5, 124.9, 42.5, 25.3 (d, $J = 4.1$ Hz). ^{31}P NMR (243 MHz, CDCl_3) δ 63.0. HRMS (ESI) for $\text{C}_{18}\text{H}_{18}\text{PS}_3$ ($\text{M}+\text{H}$) $^+$: Calcd: 361.0308; found: 361.0303.



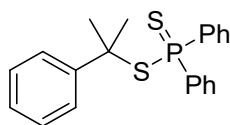
1-Phenylpropan-2-yl diphenylphosphinodithioate (2k), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.97 – 7.86 (m, 4H), 7.52 – 7.37 (m, 6H), 7.27 – 7.23 (m, 2H), 7.22 – 7.18 (m, 1H), 7.16 – 7.12 (m, 2H), 3.77 – 3.65 (m, 1H), 3.07 (dd, $J = 13.8, 5.4$ Hz, 1H), 2.70 (dd, $J = 13.8, 9.0$ Hz, 1H), 1.27 (d, $J = 6.6$ Hz, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 138.7, 135.5, 135.2, 134.9, 134.7, 131.8, 131.8, 131.8, 131.8, 131.6, 131.5 (d, $J = 4.3$ Hz), 131.4, 129.6, 128.6 (d, $J = 13.3$ Hz), 128.4, 126.6, 44.9 (d, $J = 4.2$ Hz), 44.8, 22.0 (d, $J = 4.7$ Hz). ^{31}P NMR (243 MHz, CDCl_3) δ 62.8. HRMS (ESI) for $\text{C}_{21}\text{H}_{22}\text{PS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 369.0901; found: 369.0911.



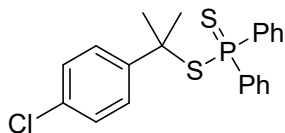
1-(1,3-Dioxoisindolin-2-yl)ethyl diphenylphosphinodithioate (2l), white solid. ^1H NMR (600 MHz, CDCl_3) δ 7.87 (ddd, $J = 15.6, 8.4, 1.2$ Hz, 4H), 7.69 – 7.62 (m, 4H), 7.49 (dd, $J = 7.8, 5.4$ Hz, 1H), 7.46 – 7.40 (m, 2H), 7.14 – 7.05 (m, 3H), 6.09 (dq, $J = 12.6, 7.2$ Hz, 1H), 1.88 (d, $J = 7.2$ Hz, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 166.2, 134.3, 134.0, 133.7, 132.8, 132.3, 132.2 (d, $J = 2.1$ Hz), 132.1 (d, $J = 11.5$ Hz), 131.5 (d, $J = 12.3$ Hz), 131.1 (d, $J = 10.9$ Hz), 128.8 (d, $J = 13.7$ Hz), 128.3 (d, $J = 13.2$ Hz), 123.3, 50.7, 22.4 (d, $J = 6.1$ Hz). ^{31}P NMR (243 MHz, CDCl_3) δ 61.9. HRMS (ESI) for $\text{C}_{22}\text{H}_{19}\text{NO}_2\text{PS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 424.0595; found: 424.0587.



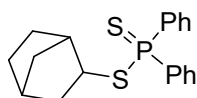
3-(Dimethylamino)-3-oxopropyl diphenylphosphinodithioate (2m), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 8.00 – 7.93 (m, 4H), 7.53 – 7.48 (m, 2H), 7.48 – 7.43 (m, 4H), 3.26 – 3.16 (m, 2H), 2.91 (s, 3H), 2.82 (s, 3H), 2.67 (t, $J = 6.6$ Hz, 2H). ^{13}C NMR (151 MHz, CDCl_3) δ 170.6, 134.8, 134.3, 132.0 (d, $J = 2.5$ Hz), 131.6 (d, $J = 11.4$ Hz), 128.7 (d, $J = 13.2$ Hz), 37.0, 35.5, 34.3, 27.1. ^{31}P NMR (243 MHz, CDCl_3) δ 64.7. HRMS (ESI) for $\text{C}_{17}\text{H}_{21}\text{NOPS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 350.0802; found: 350.0796.



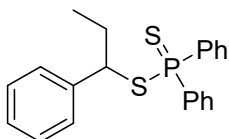
2-Phenylpropan-2-yl diphenylphosphinodithioate (2n), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.90 (dd, $J = 14.4, 7.8$ Hz, 4H), 7.43 (d, $J = 7.2$ Hz, 4H), 7.40 – 7.34 (m, 4H), 7.17 (t, $J = 7.8$ Hz, 2H), 7.11 (t, $J = 7.2$ Hz, 1H), 1.98 (s, 6H). ^{13}C NMR (151 MHz, CDCl_3) δ 145.3 (d, $J = 4.3$ Hz), 135.3, 134.7, 131.7 (d, $J = 11.2$ Hz), 131.5 (d, $J = 2.4$ Hz), 128.4 (d, $J = 13.4$ Hz), 128.1, 127.3, 126.5, 57.2 (d, $J = 3.9$ Hz), 31.1 (d, $J = 2.7$ Hz). ^{31}P NMR (243 MHz, CDCl_3) δ 56.1. HRMS (ESI) for $\text{C}_{21}\text{H}_{22}\text{PS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 369.0901; found: 369.0913.



2-(4-Chlorophenyl)propan-2-yl diphenylphosphinodithioate (2o), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.86 – 7.77 (m, 4H), 7.48 – 7.41 (m, 2H), 7.39 – 7.33 (m, 4H), 7.29 (d, $J = 8.4$ Hz, 2H), 6.99 (d, $J = 8.4$ Hz, 2H), 1.96 (s, 6H). ^{13}C NMR (151 MHz, CDCl_3) δ 143.1 (d, $J = 3.5$ Hz), 134.9, 134.3, 133.0, 131.7 (d, $J = 10.7$ Hz), 131.5 (d, $J = 2.5$ Hz), 128.4 (d, $J = 13.1$ Hz), 128.2, 127.9, 56.1 (d, $J = 3.1$ Hz), 31.3 (d, $J = 3.4$ Hz). ^{31}P NMR (243 MHz, CDCl_3) δ 56.3. HRMS (ESI) for $\text{C}_{21}\text{H}_{21}\text{ClPS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 403.0511; found: 403.0506.

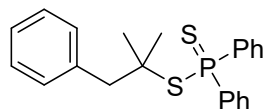


Bicyclo[2.2.1]heptan-2-yl diphenylphosphinodithioate (2p), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 8.02 – 7.96 (m, 2H), 7.95 – 7.88 (m, 2H), 7.52 – 7.40 (m, 6H), 3.40 – 3.28 (m, 1H), 2.25 (s, 1H), 2.18 (d, $J = 4.2$ Hz, 1H), 1.83 – 1.73 (m, 1H), 1.58 – 1.52 (m, 2H), 1.51 – 1.44 (m, 1H), 1.44 – 1.37 (m, 1H), 1.23 – 1.15 (m, 2H), 1.13 – 1.05 (m, 1H). ^{13}C NMR (151 MHz, CDCl_3) δ 135.3 (d, $J = 9.5$ Hz), 134.8 (d, $J = 8.7$ Hz), 131.8, 131.8, 131.8, 131.8, 131.6, 131.5, 131.5, 131.4, 128.6 (d, $J = 13.3$ Hz), 48.6, 44.3, 40.6 (d, $J = 6.6$ Hz), 36.7, 36.4, 28.7, 28.4. ^{31}P NMR (243 MHz, CDCl_3) δ 62.1. HRMS (ESI) for $\text{C}_{19}\text{H}_{22}\text{PS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 345.0901; found: 345.0892.

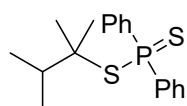


1-Phenylpropyl diphenylphosphinodithioate (2q), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.90 – 7.84 (m, 2H), 7.74 – 7.66 (m, 2H), 7.51 – 7.46 (m, 1H), 7.45 – 7.40 (m, 2H), 7.34 (ddd, $J = 7.2, 2.4, 1.2$ Hz, 1H), 7.23 (ddd, $J = 7.8, 3.6, 1.8$ Hz, 2H), 7.16 (dd, $J = 7.8, 1.2$ Hz, 2H), 7.13 – 7.05 (m, 3H), 4.50 – 4.37 (m, 1H), 2.13 – 1.94 (m, 2H), 0.85 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 141.4 (d, $J = 3.4$ Hz), 135.5, 135.0, 134.0, 133.4, 131.8, 131.8, 131.4 (d, $J = 3.2$ Hz), 131.2 (d, $J = 10.9$ Hz), 128.6 (d, $J = 13.5$ Hz), 128.3, 128.2 (d, $J = 13.8$ Hz), 128.0, 127.2, 53.5, 31.1 (d, $J = 5.2$ Hz),

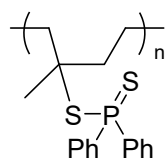
12.3. ^{31}P NMR (243 MHz, CDCl_3) δ 63.5. HRMS (ESI) for $\text{C}_{21}\text{H}_{22}\text{PS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 369.0901; found: 369.0913.



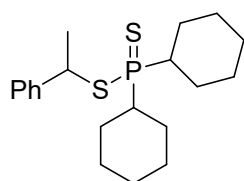
2-Methyl-1-phenylpropan-2-yl diphenylphosphinodithioate (2r), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 8.03 – 7.95 (m, 4H), 7.51 – 7.46 (m, 2H), 7.46 – 7.41 (m, 4H), 7.31 – 7.21 (m, 5H), 3.24 (s, 2H), 1.43 (s, 6H). ^{13}C NMR (151 MHz, CDCl_3) δ 137.2, 136.0, 135.4, 131.7, 131.7, 131.4, 128.5 (d, J = 13.1 Hz), 127.9, 126.6, 57.0, 49.7, 29.9 (d, J = 4.7 Hz). ^{31}P NMR (243 MHz, CDCl_3) δ 55.9. HRMS (ESI) for $\text{C}_{22}\text{H}_{24}\text{PS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 383.1057; found: 383.1051.



2,3-Dimethylbutan-2-yl diphenylphosphinodithioate (2s), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 8.06 – 7.96 (m, 4H), 7.51 – 7.41 (m, 6H), 2.10 – 1.96 (m, 1H), 1.47 (s, 6H), 0.98 (d, J = 6.6 Hz, 6H). ^{13}C NMR (151 MHz, CDCl_3) δ 136.2, 135.6, 131.7 (d, J = 11.3 Hz), 131.6 (d, J = 2.5 Hz), 128.4 (d, J = 13.1 Hz), 62.1 (d, J = 4.2 Hz), 39.7 (d, J = 4.3 Hz), 27.3 (d, J = 3.3 Hz), 18.0. ^{31}P NMR (243 MHz, CDCl_3) δ 55.7. HRMS (ESI) for $\text{C}_{18}\text{H}_{24}\text{PS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 335.1057; found: 335.1051.

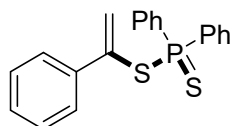


Polymer (2t), white solid. ^1H NMR (600 MHz, CDCl_3) δ 7.98 (s, 4H), 7.40 (d, J = 15.6 Hz, 6H), 1.79 – 1.19 (m, 9H). ^{13}C NMR (151 MHz, CDCl_3) δ 136.2, 135.6, 131.7, 128.6 (d, J = 12.7 Hz), 62.0, 42.1, 27.3 (d, J = 22.1 Hz), 19.7. ^{31}P NMR (243 MHz, CDCl_3) δ 55.0.

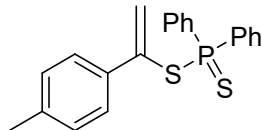


1-Phenylethyl dicyclohexylphosphinodithioate (2u), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.39 (d, J = 7.2 Hz, 2H), 7.31 (t, J = 7.8 Hz, 2H), 7.23 (t, J = 7.2 Hz,

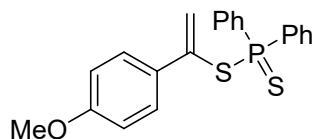
1H), 4.54 (dq, $J = 14.4, 7.2$ Hz, 1H), 2.08 – 1.77 (m, 8H), 1.72 (d, $J = 7.2$ Hz, 3H), 1.73-1.72 (m, 2H), 1.56 – 1.44 (m, 2H), 1.37 – 1.18 (m, 7H), 1.17 – 1.06 (m, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 144.5, 128.6, 127.6, 127.2, 45.7, 42.3 (d, $J = 47.2$ Hz), 41.2 (d, $J = 47.7$ Hz), 26.5, 26.5, 26.4, 26.4, 26.3, 25.9, 25.8, 25.8, 25.7, 25.5 (d, $J = 2.6$ Hz). ^{31}P NMR (243 MHz, CDCl_3) δ 92.3. HRMS (ESI) for $\text{C}_{20}\text{H}_{32}\text{PS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 367.1683; found: 367.1692.



1-Phenylvinyl diphenylphosphinodithioate (6a), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.94 – 7.87 (m, 4H), 7.47 – 7.43 (m, 2H), 7.40 – 7.33 (m, 6H), 7.18 – 7.10 (m, 3H), 5.98 (d, $J = 3.6$ Hz, 1H), 5.77 (d, $J = 3.6$ Hz, 1H). ^{13}C NMR (151 MHz, CDCl_3) δ 139.5, 136.6 (d, $J = 7.0$ Hz), 134.0, 133.4, 131.9, 131.9, 131.8, 128.5 (d, $J = 13.7$ Hz), 128.3, 128.0, 127.5, 127.3 (d, $J = 8.2$ Hz). ^{31}P NMR (243 MHz, CDCl_3) δ 64.9. HRMS (ESI) for $\text{C}_{20}\text{H}_{18}\text{PS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 353.0588; found: 353.0593.

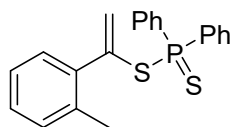


1-(p-Tolyl)vinyl diphenylphosphinodithioate (6b), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.94 – 7.86 (m, 4H), 7.48 – 7.43 (m, 2H), 7.41 – 7.35 (m, 4H), 7.27 – 7.23 (m, 2H), 6.98 – 6.89 (m, 2H), 5.93 (d, $J = 3.6$ Hz, 1H), 5.70 (d, $J = 3.6$ Hz, 1H), 2.26 (s, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 138.2, 136.7, 136.6 (d, $J = 6.7$ Hz), 134.1, 133.5, 131.9, 131.8, 128.7, 128.5 (d, $J = 13.2$ Hz), 127.5, 126.1 (d, $J = 8.1$ Hz), 21.2. ^{31}P NMR (243 MHz, CDCl_3) δ 64.7. HRMS (ESI) for $\text{C}_{21}\text{H}_{20}\text{PS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 367.0744; found: 367.0749.

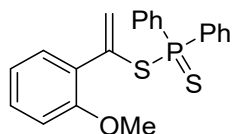


1-(4-Methoxyphenyl)vinyl diphenylphosphinodithioate (6c), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.96 – 7.86 (m, 4H), 7.50 – 7.43 (m, 2H), 7.42 – 7.37 (m, 4H), 7.32 – 7.28 (m, 2H), 6.70 – 6.63 (m, 2H), 5.88 (d, $J = 3.6$ Hz, 1H), 5.64 (d, $J = 3.6$ Hz,

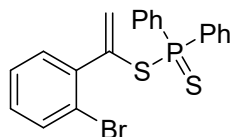
1H), 3.75 (s, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 159.7, 136.2 (d, $J = 6.5$ Hz), 134.1, 133.5, 132.1, 131.9, 131.9, 131.8, 128.9, 128.5 (d, $J = 13.4$ Hz), 125.3 (d, $J = 7.6$ Hz), 113.3, 55.4. ^{31}P NMR (243 MHz, CDCl_3) δ 64.6. HRMS (ESI) for $\text{C}_{21}\text{H}_{20}\text{OPS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 383.0693; found: 383.0678.



1-(o-Tolyl)vinyl diphenylphosphinodithioate (6d), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.90 – 7.75 (m, 4H), 7.44 – 7.39 (m, 2H), 7.36 – 7.30 (m, 4H), 7.03 – 6.94 (m, 3H), 6.88 – 6.82 (m, 1H), 6.01 (d, $J = 3.6$ Hz, 1H), 5.69 (d, $J = 3.6$ Hz, 1H), 2.28 (s, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 139.8, 136.3 (d, $J = 6.9$ Hz), 135.3, 133.7, 133.2, 131.8, 131.7, 129.9, 129.7, 129.6, 128.4 (d, $J = 13.6$ Hz), 128.0, 125.3, 20.2. ^{31}P NMR (243 MHz, CDCl_3) δ 63.9. HRMS (ESI) for $\text{C}_{21}\text{H}_{20}\text{PS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 367.0744; found: 367.0749.

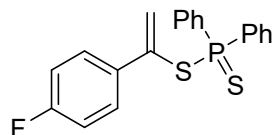


1-(2-Methoxyphenyl)vinyl diphenylphosphinodithioate (6e), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.89 – 7.80 (m, 4H), 7.44 – 7.38 (m, 2H), 7.35 – 7.30 (m, 4H), 7.13 – 7.08 (m, 1H), 6.91 (dd, $J = 7.2, 1.8$ Hz, 1H), 6.67 (d, $J = 8.4$ Hz, 1H), 6.63 (td, $J = 7.2, 1.2$ Hz, 1H), 5.98 (d, $J = 3.6$ Hz, 1H), 5.84 (d, $J = 3.6$ Hz, 1H), 3.77 (s, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 156.2, 134.0, 133.9 (d, $J = 7.1$ Hz), 133.5, 131.8 (d, $J = 10.8$ Hz), 131.7 (d, $J = 2.5$ Hz), 130.6 (d, $J = 7.7$ Hz), 130.5, 129.6, 129.5, 128.3 (d, $J = 13.2$ Hz), 120.1, 110.4, 55.6. ^{31}P NMR (243 MHz, CDCl_3) δ 64.8. HRMS (ESI) for $\text{C}_{21}\text{H}_{20}\text{OPS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 383.0693; found: 383.0675.

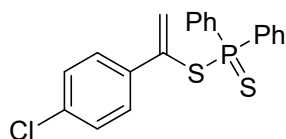


1-(2-Bromophenyl)vinyl diphenylphosphinodithioate (6f), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.90 – 7.78 (m, 4H), 7.44 – 7.36 (m, 3H), 7.35 – 7.29 (m, 4H), 6.97 – 6.90 (m, 2H), 6.87 (td, $J = 7.2, 1.2$ Hz, 1H), 6.06 (d, $J = 3.6$ Hz, 1H), 5.86 (d, J

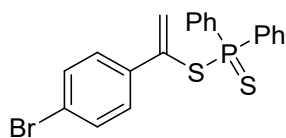
= 3.6 Hz, 1H). ^{13}C NMR (151 MHz, CDCl_3) δ 140.8, 136.0 (d, $J = 7.0$ Hz), 133.6, 133.0, 132.5 (d, $J = 7.4$ Hz), 132.3, 131.8, 131.8, 131.7, 131.4, 129.3, 128.4 (d, $J = 13.5$ Hz), 126.8, 122.2. ^{31}P NMR (243 MHz, CDCl_3) δ 64.9. HRMS (ESI) for $\text{C}_{20}\text{H}_{17}\text{BrPS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 430.9693; found: 430.9686.



1-(4-Fluorophenyl)vinyl diphenylphosphinodithioate (6g), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.92 – 7.85 (m, 4H), 7.48 – 7.43 (m, 2H), 7.41 – 7.35 (m, 4H), 7.33 – 7.28 (m, 2H), 6.83 – 6.74 (m, 2H), 5.92 (d, $J = 3.6$ Hz, 1H), 5.74 (d, $J = 3.6$ Hz, 1H). ^{13}C NMR (151 MHz, CDCl_3) δ 162.7 (d, $J = 248.3$ Hz), 135.7, 135.7, 135.7, 133.9, 133.3, 132.0 (d, $J = 2.6$ Hz), 131.9, 131.8, 129.5 (d, $J = 8.0$ Hz), 128.5 (d, $J = 13.5$ Hz), 127.5 (d, $J = 7.7$ Hz), 114.8 (d, $J = 21.7$ Hz). ^{31}P NMR (243 MHz, CDCl_3) δ 64.7. HRMS (ESI) for $\text{C}_{20}\text{H}_{17}\text{FPS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 371.0493; found: 371.0477.

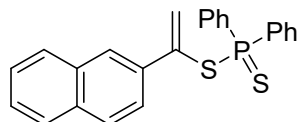


1-(4-Chlorophenyl)vinyl diphenylphosphinodithioate (6h), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.88 (dd, $J = 14.4, 7.2$ Hz, 4H), 7.50 – 7.45 (m, 2H), 7.39 (td, $J = 7.8, 3.6$ Hz, 4H), 7.25 (s, 2H), 7.06 (d, $J = 8.4$ Hz, 2H), 5.98 (d, $J = 3.6$ Hz, 1H), 5.77 (d, $J = 3.6$ Hz, 1H). ^{13}C NMR (151 MHz, CDCl_3) δ 138.0, 135.7 (d, $J = 6.7$ Hz), 134.1, 133.8, 133.3, 132.0 (d, $J = 2.5$ Hz), 131.8 (d, $J = 10.7$ Hz), 128.9, 128.5 (d, $J = 13.3$ Hz), 128.1, 128.0. ^{31}P NMR (243 MHz, CDCl_3) δ 64.8. HRMS (ESI) for $\text{C}_{20}\text{H}_{17}\text{ClPS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 387.0198; found: 387.0189.

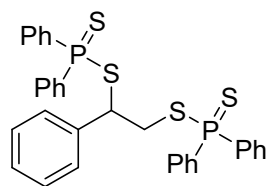


1-(4-Bromophenyl)vinyl diphenylphosphinodithioate (6i), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.91 – 7.84 (m, 4H), 7.50 – 7.46 (m, 2H), 7.39 (ddd, $J = 7.2, 3.6, 1.8$ Hz, 4H), 7.24 – 7.16 (m, 4H), 5.99 (d, $J = 3.6$ Hz, 1H), 5.78 (d, $J = 3.6$ Hz, 1H). ^{13}C NMR (151 MHz, CDCl_3) δ 138.5, 135.8 (d, $J = 6.3$ Hz), 133.8, 133.2, 132.0 (d, $J = 2.5$

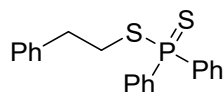
Hz), 131.8 (d, $J = 11.3$ Hz), 131.0, 129.3, 128.5 (d, $J = 13.2$ Hz), 128.1 (d, $J = 8.0$ Hz), 122.4. ^{31}P NMR (243 MHz, CDCl_3) δ 64.8. HRMS (ESI) for $\text{C}_{20}\text{H}_{17}\text{BrPS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 430.9693; found: 430.9687.



1-(Naphthalen-2-yl)vinyl diphenylphosphinodithioate (6j), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.93 – 7.87 (m, 4H), 7.76 – 7.70 (m, 2H), 7.64 – 7.59 (m, 2H), 7.47 (dd, $J = 8.4, 1.8$ Hz, 1H), 7.44 – 7.39 (m, 2H), 7.38 – 7.35 (m, 2H), 7.34 – 7.29 (m, 4H), 6.13 (d, $J = 3.6$ Hz, 1H), 5.89 (d, $J = 3.6$ Hz, 1H). ^{13}C NMR (151 MHz, CDCl_3) δ 136.7 (d, $J = 9.6$ Hz), 134.0, 133.4, 133.0, 132.8, 131.8 (d, $J = 11.7$ Hz), 128.4 (d, $J = 12.9$ Hz), 127.8 (d, $J = 7.6$ Hz), 127.7, 127.5, 127.3, 126.4, 126.2, 124.9. ^{31}P NMR (243 MHz, CDCl_3) δ 64.8. HRMS (ESI) for $\text{C}_{24}\text{H}_{20}\text{PS}_2$ ($\text{M}+\text{H}$) $^+$: Calcd: 403.0744; found: 403.0752.

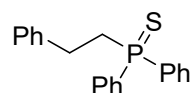


1-Phenylethane-1,2-diyl bis(diphenylphosphinodithioate) (6a'), colorless oil. ^1H NMR (600 MHz, CDCl_3) δ 7.93 – 7.87 (m, 2H), 7.86 – 7.79 (m, 2H), 7.72 – 7.65 (m, 2H), 7.51 – 7.41 (m, 7H), 7.40 – 7.36 (m, 2H), 7.34 (td, $J = 7.8, 3.6$ Hz, 2H), 7.29 – 7.24 (m, 1H), 7.15 (td, $J = 7.8, 3.0$ Hz, 2H), 6.96 – 6.90 (m, 3H), 6.87 (t, $J = 7.2$ Hz, 2H), 5.19 – 5.01 (m, 1H), 3.70 – 3.49 (m, 1H), 3.37 – 3.17 (m, 1H). ^{13}C NMR (151 MHz, CDCl_3) δ 138.7 (d, $J = 3.8$ Hz), 134.7, 134.2, 133.8, 133.5, 133.2, 132.9, 132.1, 132.0, 132.0, 132.0, 131.9, 131.5, 131.4, 131.3, 131.3, 131.2, 130.9 (d, $J = 2.1$ Hz), 128.7, 128.7, 128.6, 128.6, 128.5, 128.4, 128.1, 128.0, 127.7, 47.1, 40.3 (d, $J = 50.3$ Hz). ^{31}P NMR (243 MHz, CDCl_3) δ 62.2, 39.1. HRMS (ESI) for $\text{C}_{32}\text{H}_{29}\text{P}_2\text{S}_4$ ($\text{M}+\text{H}$) $^+$: Calcd: 603.0627; found: 603.0635.

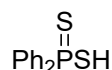


Phenethyl diphenylphosphinodithioate (3a), white solid. ^1H NMR (600 MHz,

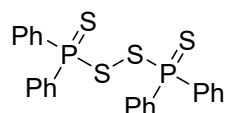
CDCl₃) δ 7.97 – 7.90 (m, 4H), 7.53 – 7.48 (m, 2H), 7.47 – 7.42 (m, 4H), 7.28 – 7.24 (m, 2H), 7.22 – 7.18 (m, 1H), 7.13 (d, J = 7.2 Hz, 2H), 3.27 – 3.09 (m, 2H), 2.98 – 2.75 (m, 2H). ¹³C NMR (151 MHz, CDCl₃) δ 139.7, 134.9, 134.3, 132.0 (d, J = 2.3 Hz), 131.5 (d, J = 11.4 Hz), 128.7, 128.6 (d, J = 5.7 Hz), 126.7, 36.6 (d, J = 4.7 Hz), 32.9. ³¹P NMR (243 MHz, CDCl₃) δ 64.8. HRMS (ESI) for C₂₀H₂₀PS₂ (M+H)⁺: Calcd: 355.0744; found: 355.0738.



Phenethyldiphenylphosphine sulfide (4a), white solid. ¹H NMR (600 MHz, CDCl₃) δ 7.90 – 7.82 (m, 4H), 7.53 – 7.42 (m, 6H), 7.28 – 7.22 (m, 2H), 7.20 – 7.13 (m, 3H), 3.00 – 2.87 (m, 2H), 2.82 – 2.70 (m, 2H). ¹³C NMR (151 MHz, CDCl₃) δ 141.0 (d, J = 17.1 Hz), 132.9, 132.4, 131.7 (d, J = 1.8 Hz), 131.2 (d, J = 10.1 Hz), 128.9, 128.8 (d, J = 8.3 Hz), 128.3, 126.5, 34.7 (d, J = 54.8 Hz), 28.5. ³¹P NMR (243 MHz, CDCl₃) δ 42.6. This compound is known in the literature: Moglie, Y.; González-Soria, M. J.; Martín-García, I.; Radivoy, G.; Alonso, F., *Green Chem.* **2016**, *18*, 4896.



Diphenylphosphinodithioic acid (7a), white solid. ¹H NMR (600 MHz, CDCl₃) δ 8.00 – 7.92 (m, 4H), 7.55 – 7.51 (m, 2H), 7.50 – 7.45 (m, 4H), 2.51 (d, J = 54.0 Hz, 1H). ¹³C NMR (151 MHz, CDCl₃) δ 135.9, 135.3, 132.1 (d, J = 2.2 Hz), 131.1 (d, J = 11.8 Hz), 128.8 (d, J = 13.6 Hz). ³¹P NMR (243 MHz, CDCl₃) δ 56.5. This compound is known in the literature: Wagner, J.; Ciesielski, M.; Fleckenstein, C. A.; Denecke, H.; Garlich, F.; Ball, A.; Doering, M. *Org. Process Res. Dev.* **2013**, *17*, 47.



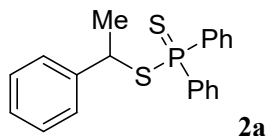
Bis(diphenylphosphinothioyl) Disulfide (8a), white solid. ¹H NMR (600 MHz, CDCl₃) δ 7.90 – 7.76 (m, 8H), 7.49 – 7.42 (m, 4H), 7.40 – 7.34 (m, 8H). ¹³C NMR (151 MHz, CDCl₃) δ 132.8 (d, J = 82.9 Hz), 132.4, 132.1 (d, J = 11.6 Hz), 128.6 (d, J = 14.0 Hz). ³¹P NMR (243 MHz, CDCl₃) δ 70.2. This compound is known in the literature:

Wagner, J.; Ciesielski, M.; Fleckenstein, C. A.; Denecke, H.; Garlich, F.; Ball, A.; Doering, M. *Org. Process Res. Dev.* **2013**, *17*, 47.

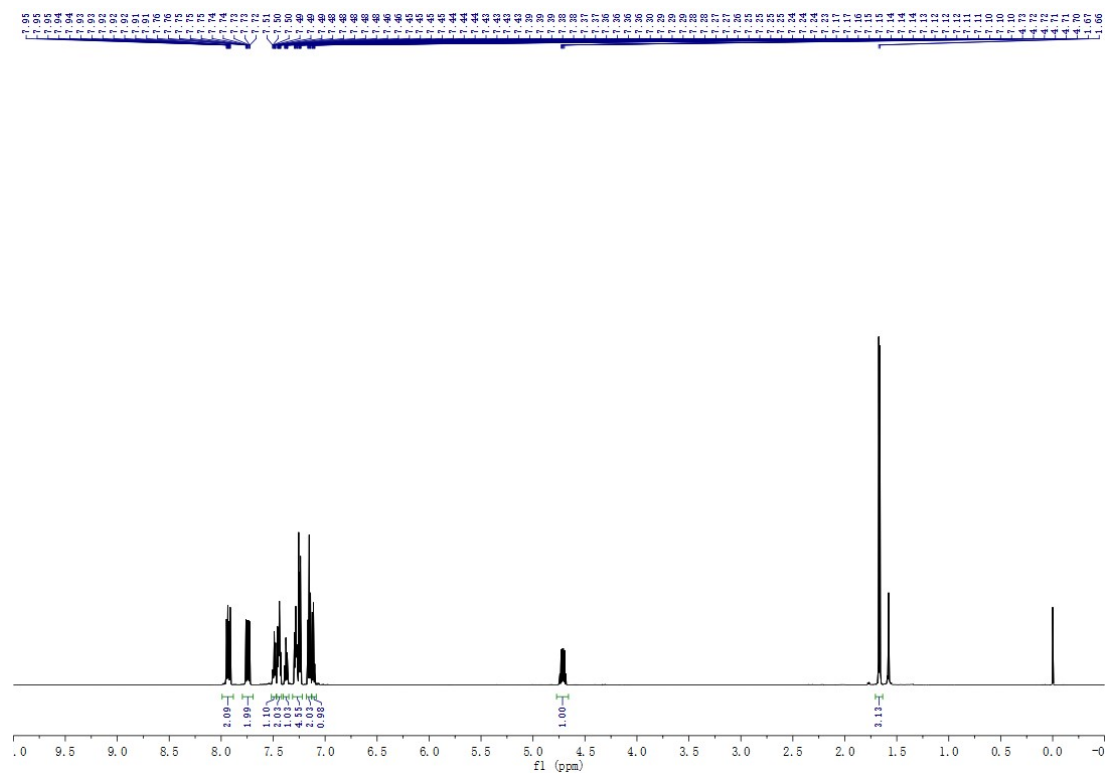


Diphenylphosphine sulfide (9a), white solid. ^[26] ¹H NMR (600 MHz, CDCl₃) δ 8.42 (s, 0.5 H), 7.81 – 7.74 (m, 4H), 7.64 (s, 0.5 H), 7.56 – 7.52 (m, 2H), 7.51 – 7.45 (m, 4H). ¹³C NMR (151 MHz, CDCl₃) δ 132.3 (d, J = 2.5 Hz), 131.37 (s), 131.23 (d, J = 11.6 Hz), 130.82 (s), 129.08 (d, J = 13.1 Hz). ³¹P NMR (243 MHz, CDCl₃) δ 23.5. This compound is known in the literature: Hirota, E.; Hirashima, S.; Morita, R.; Takase, J.; Matsushima, Y.; Nakashima, K.; Akutsu, H.; Miura, T. *Org. Lett.* **2024**, *26*, 1797.

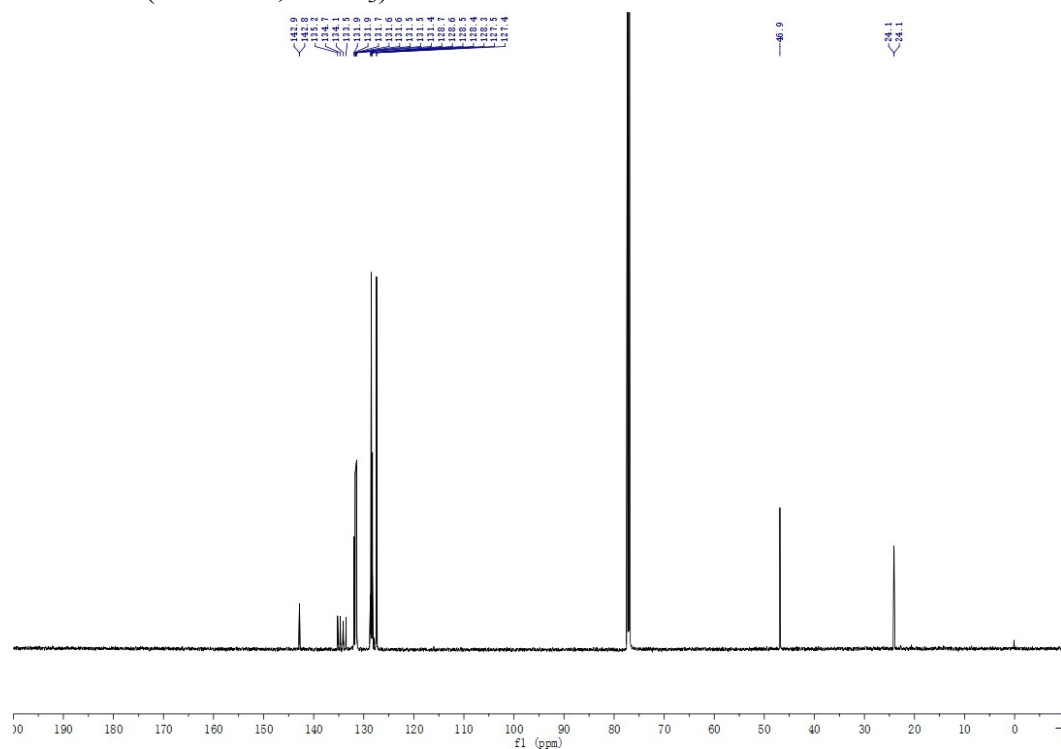
3. Copies of the ¹H and ¹³C NMR Spectra of the Products



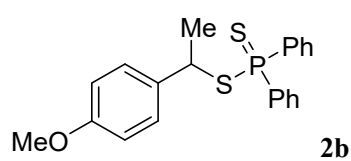
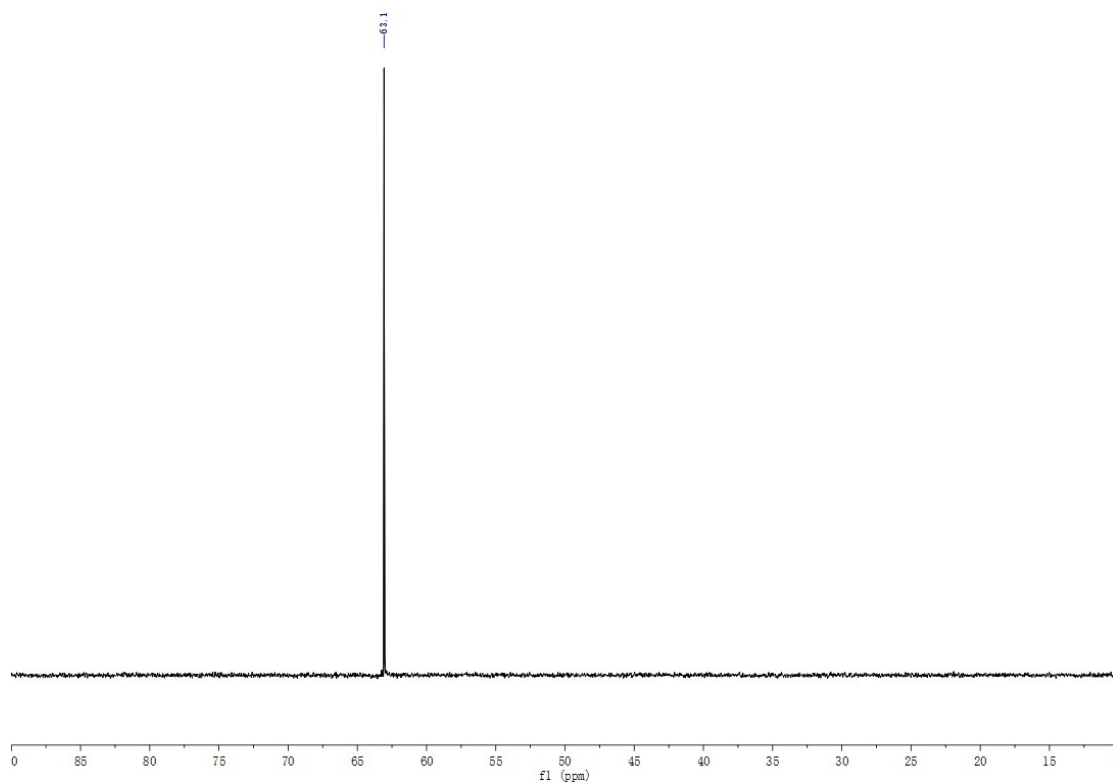
¹H NMR (600 MHz, CDCl₃)



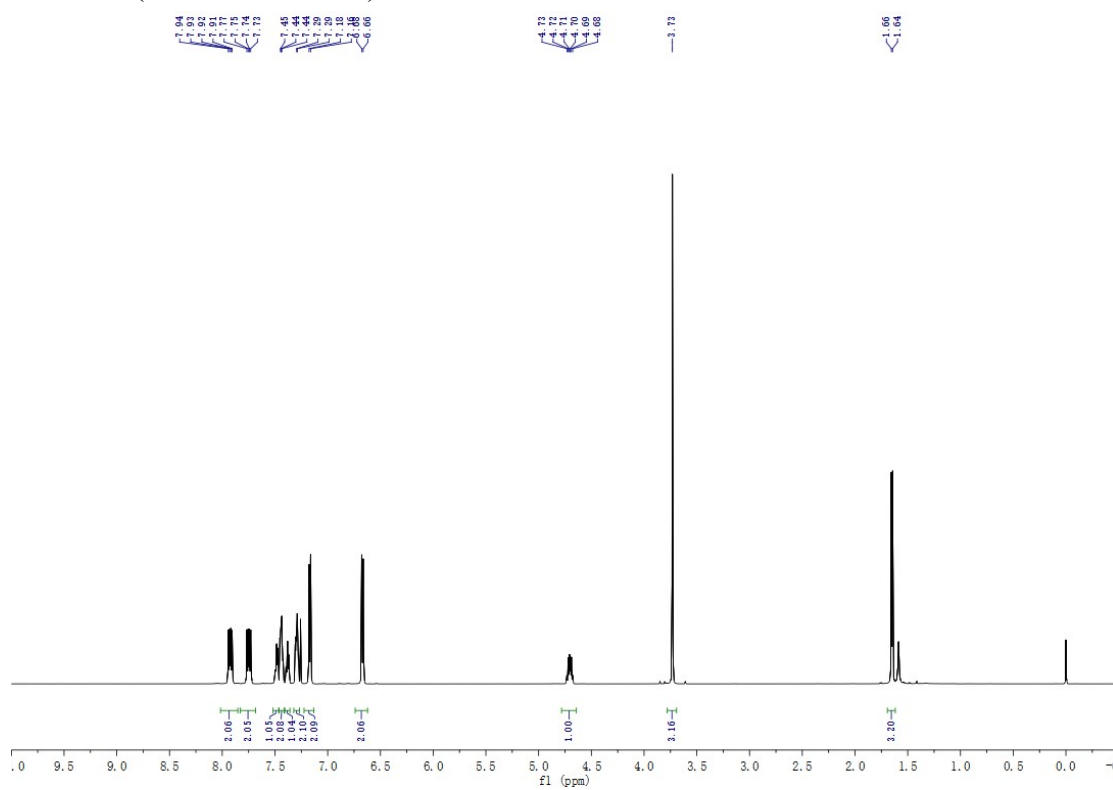
¹³C NMR (151 MHz, CDCl₃)



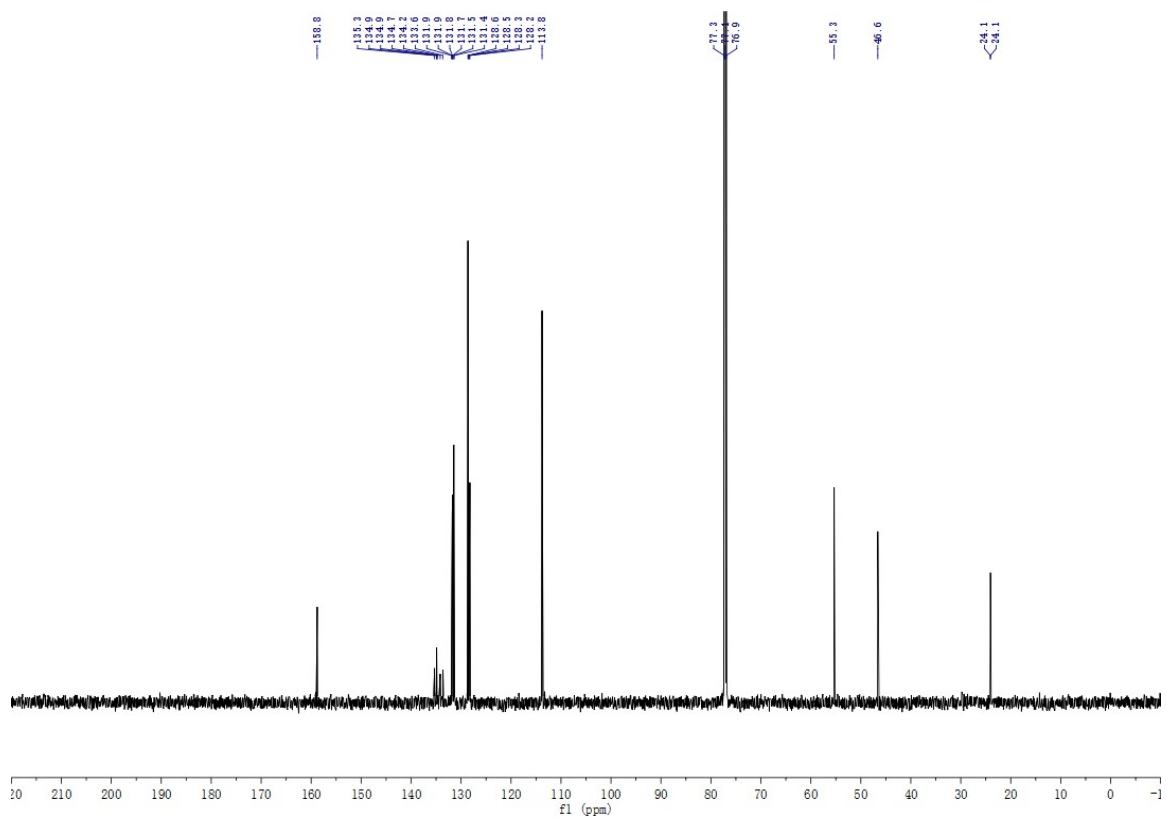
³¹P NMR (243 MHz, CDCl₃)



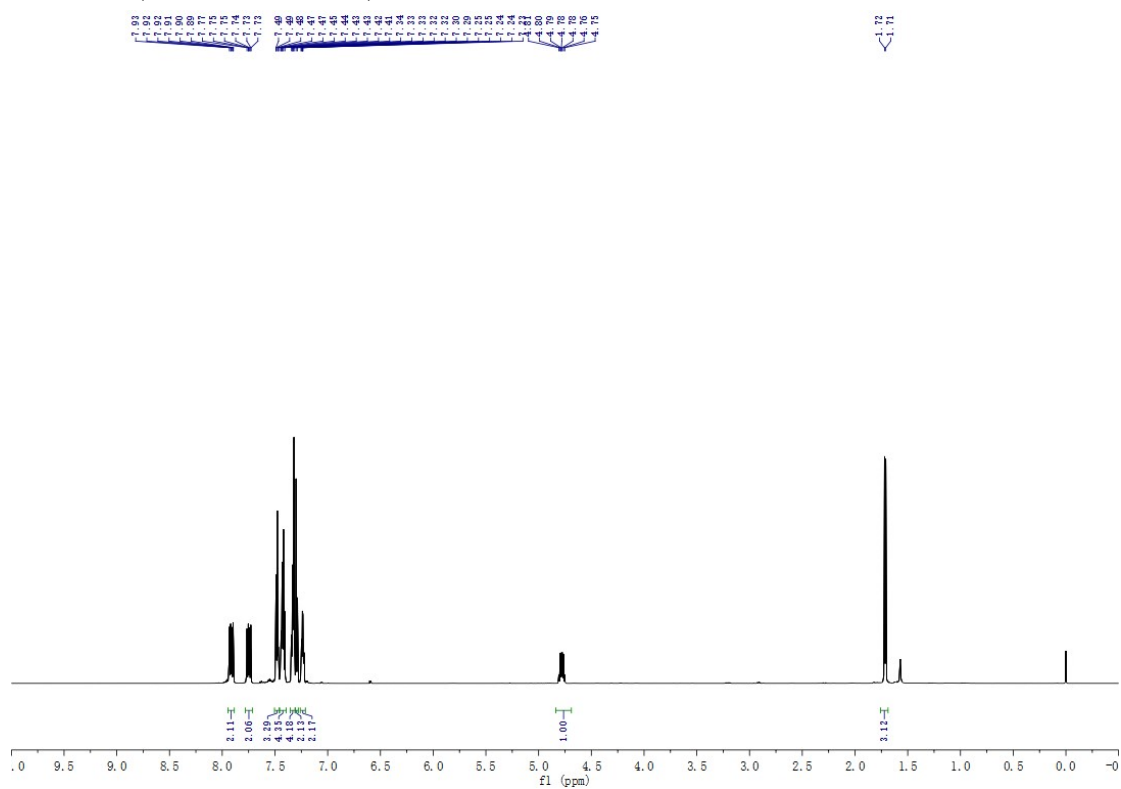
¹H NMR (600 MHz, CDCl₃)



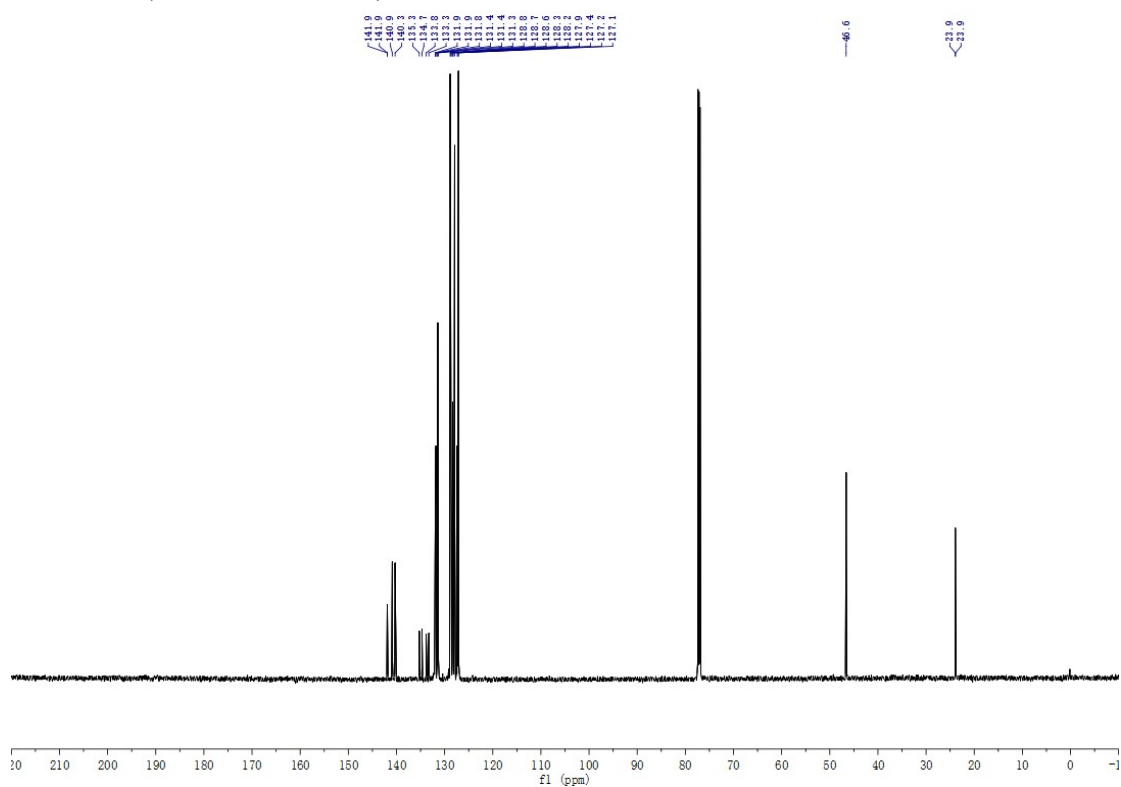
¹³C NMR (151 MHz, CDCl₃)



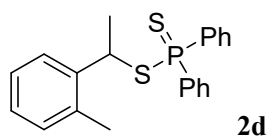
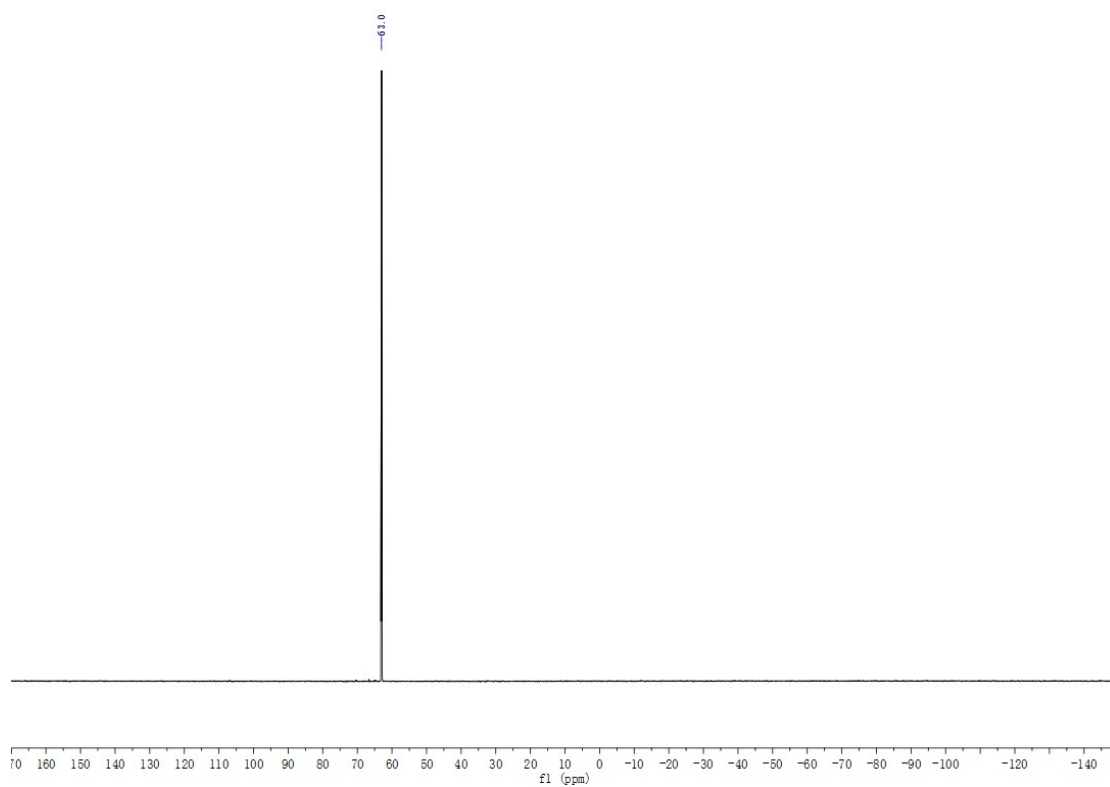
^1H NMR (600 MHz, CDCl_3)



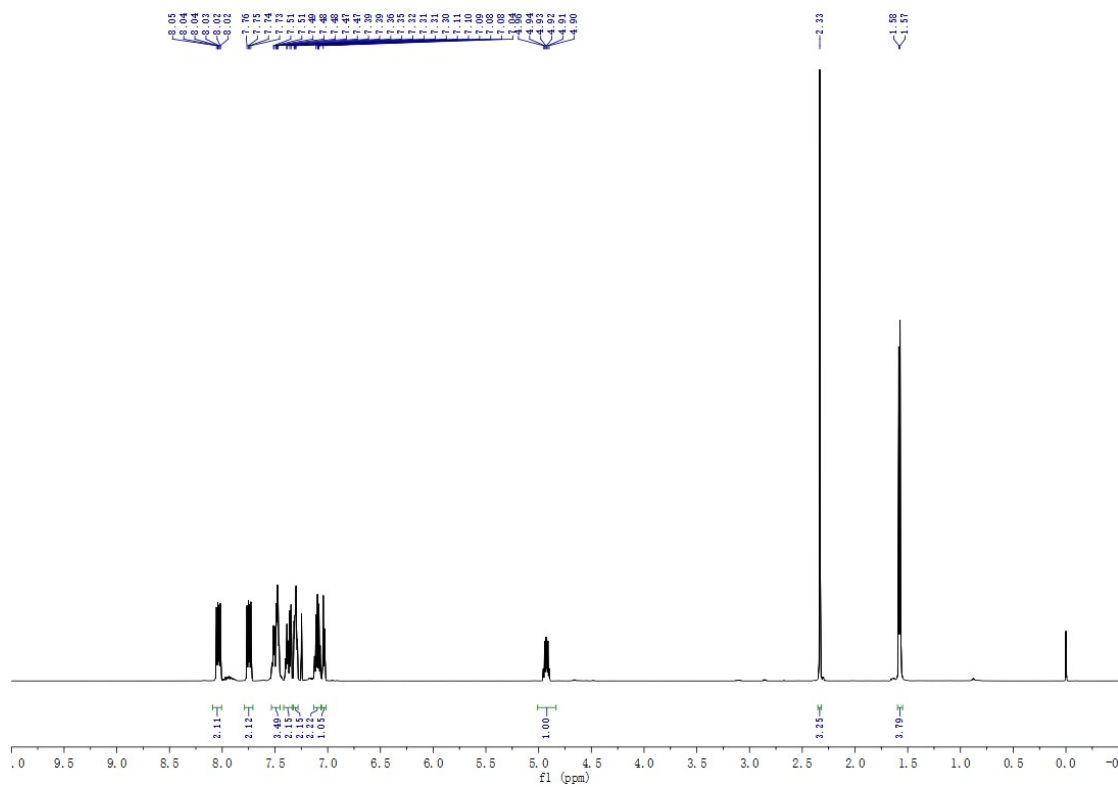
^{13}C NMR (151 MHz, CDCl_3)



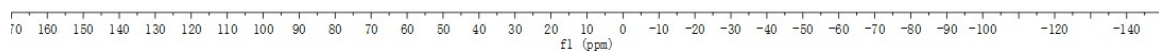
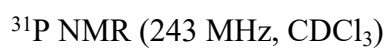
^{31}P NMR (243 MHz, CDCl_3)

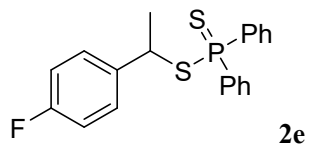


¹H NMR (600 MHz, CDCl₃)

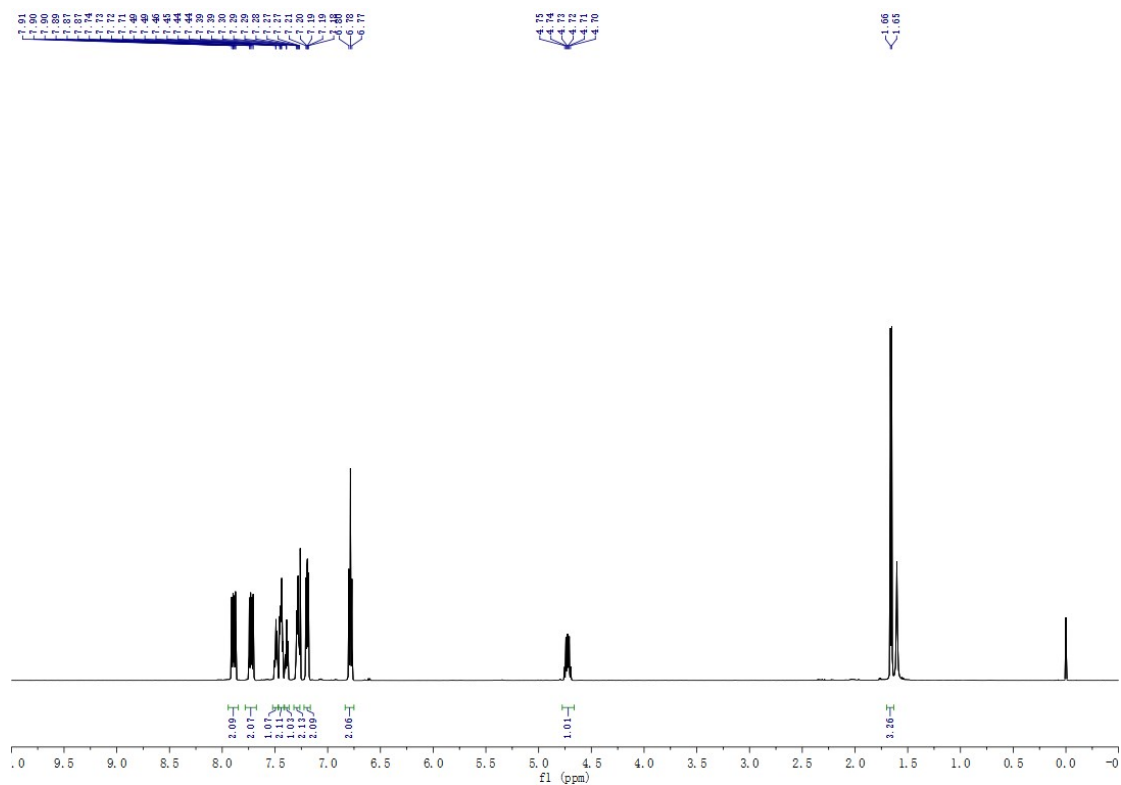


¹³C NMR (151 MHz, CDCl₃)

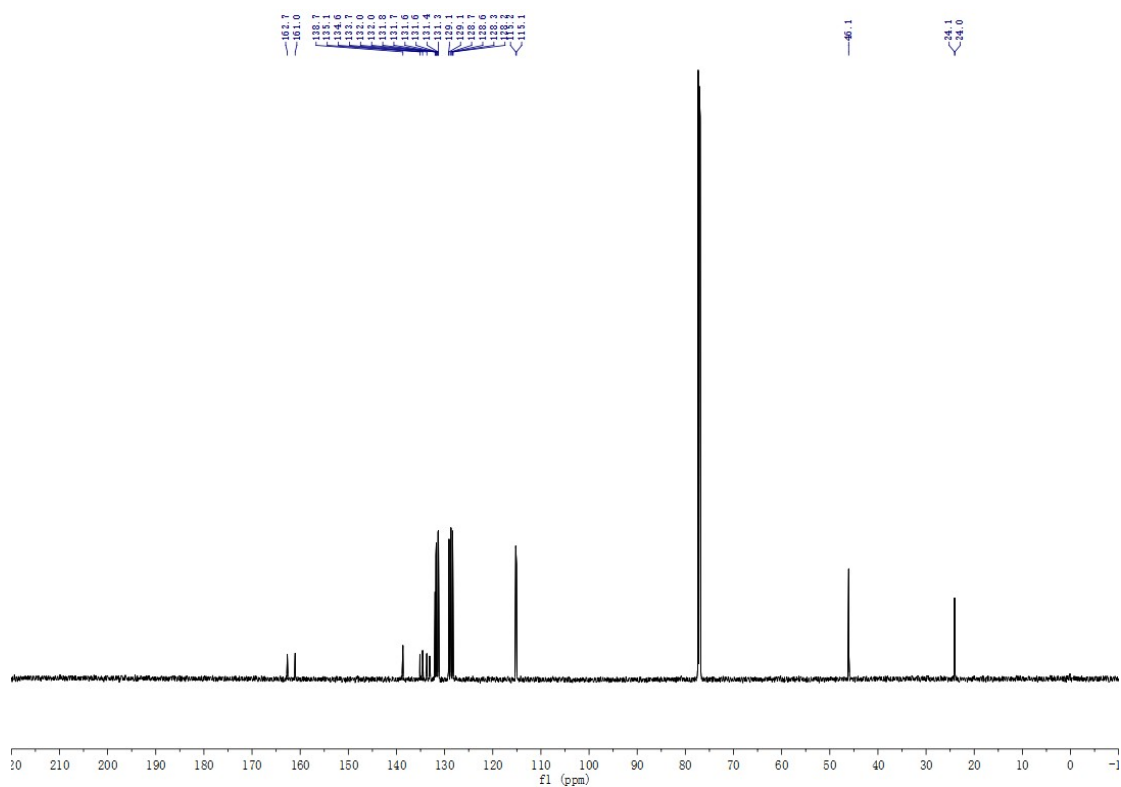




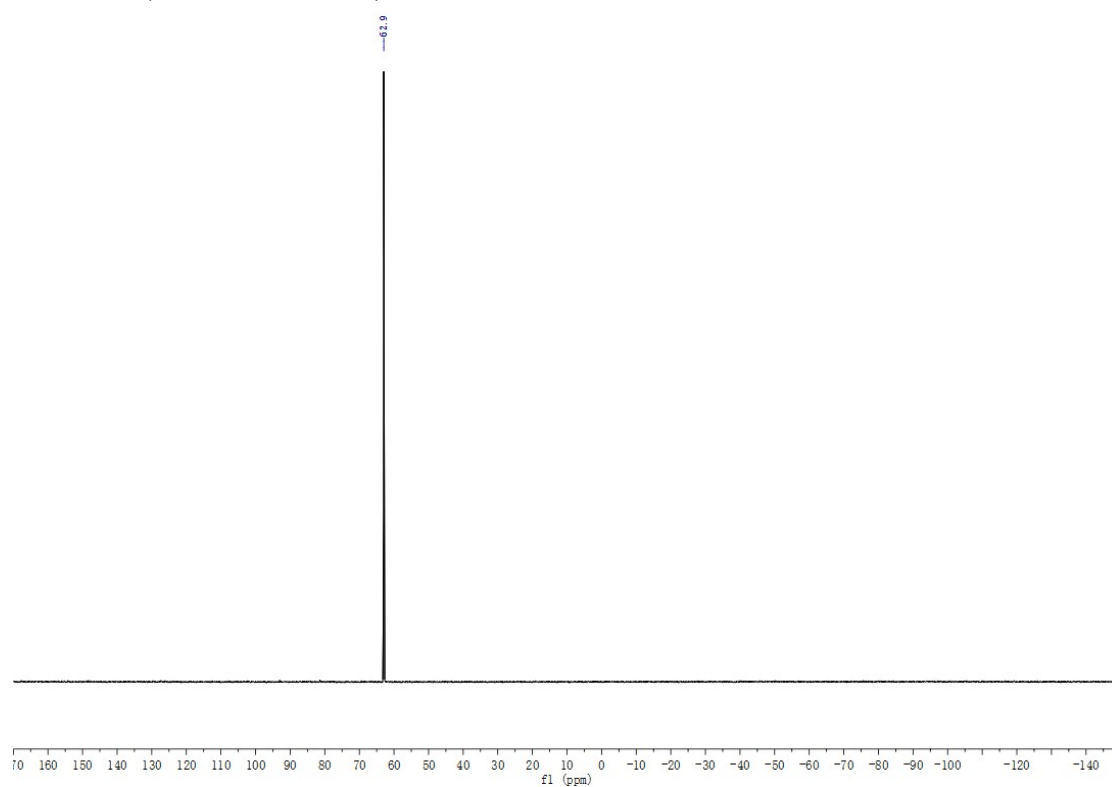
^1H NMR (600 MHz, CDCl_3)



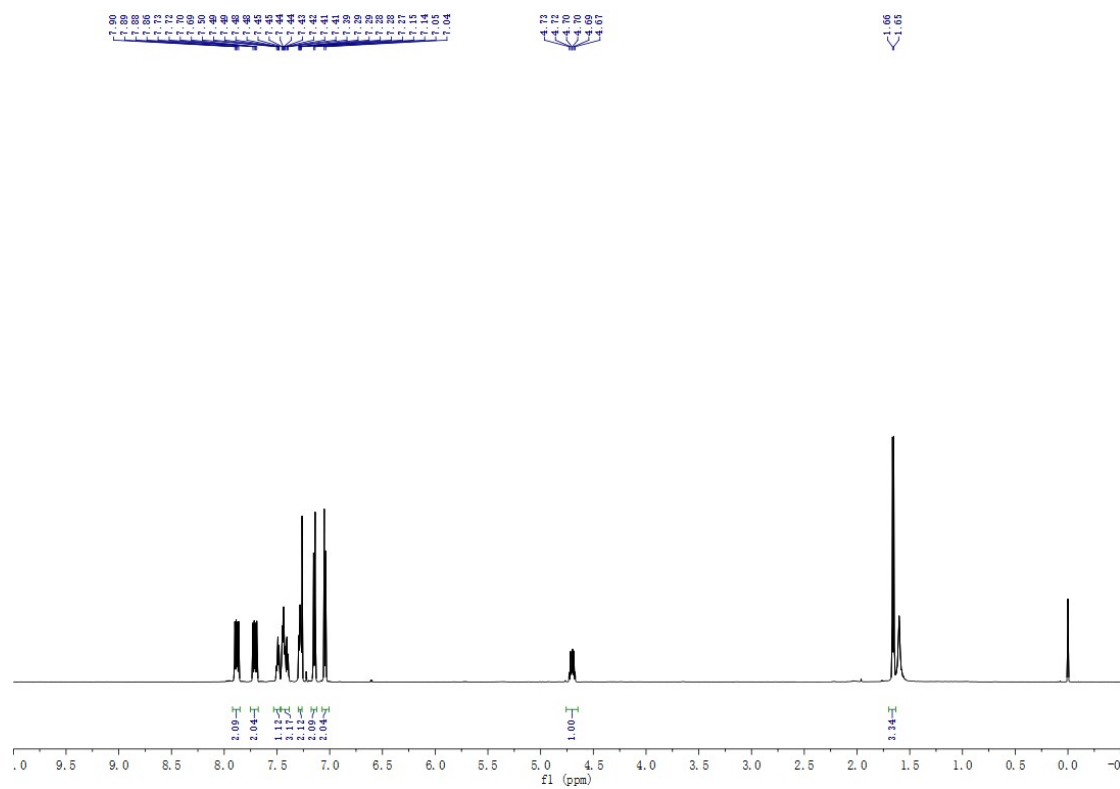
^{13}C NMR (151 MHz, CDCl_3)



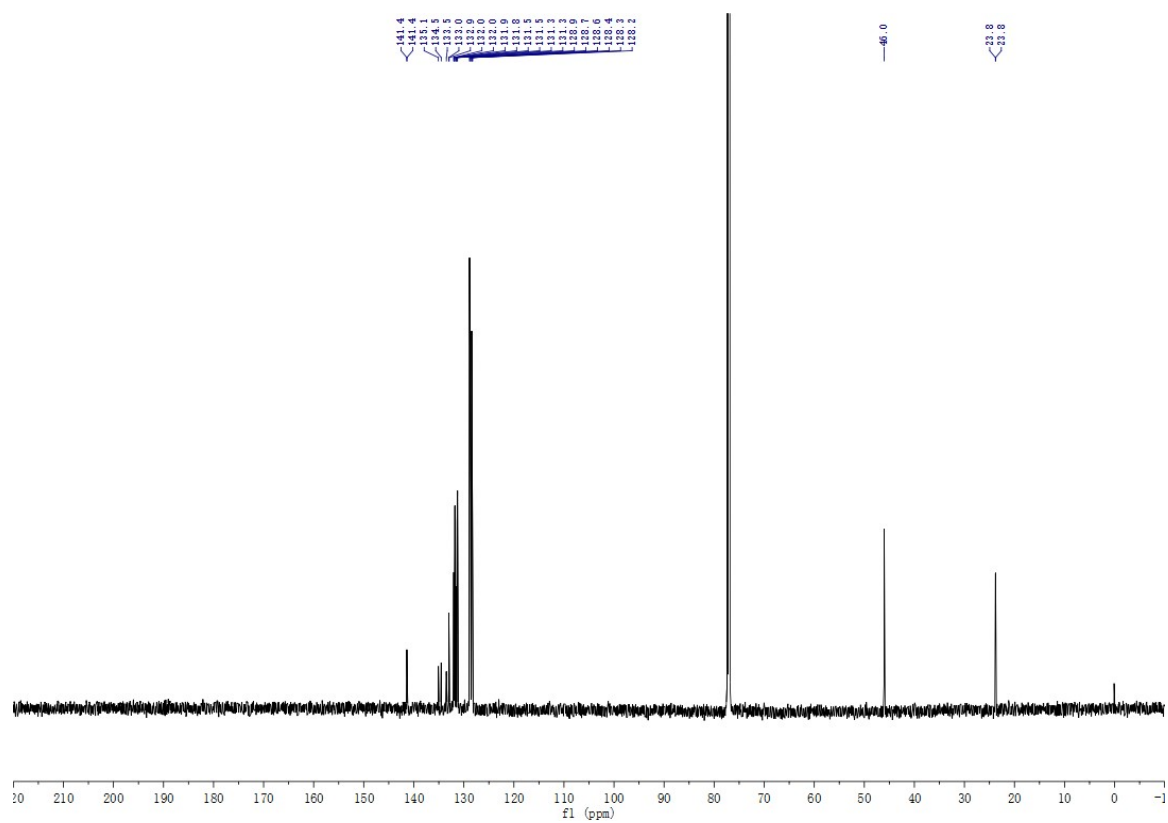
^{31}P NMR (243 MHz, CDCl_3)



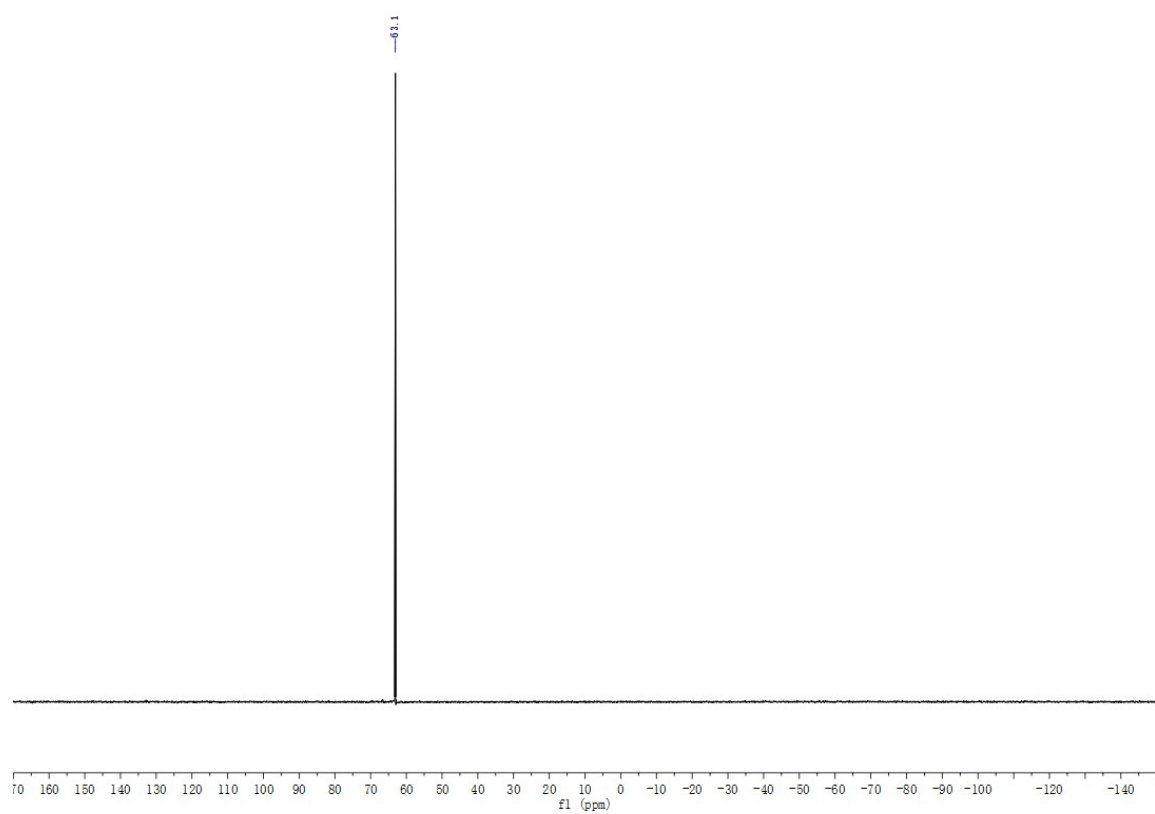
^1H NMR (600 MHz, CDCl_3)

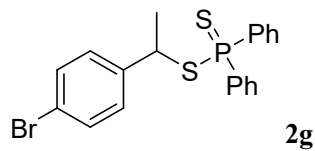


^{13}C NMR (151 MHz, CDCl_3)

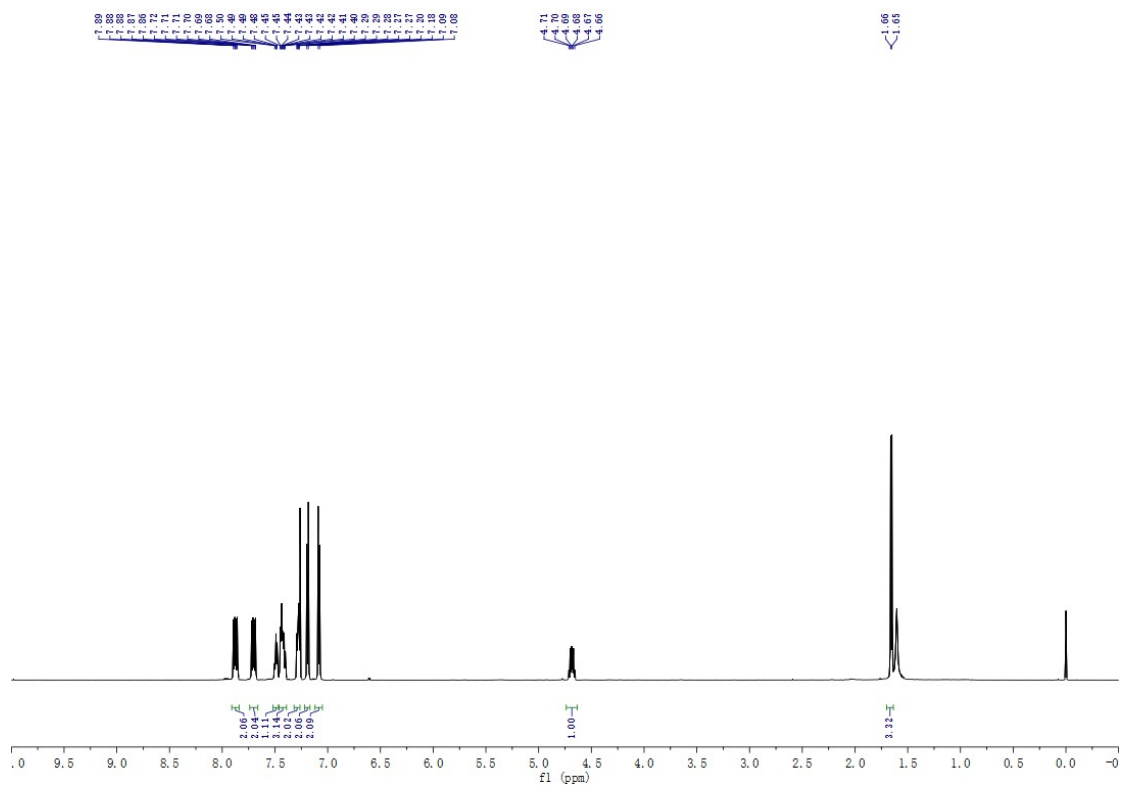


^{31}P NMR (243 MHz, CDCl_3)

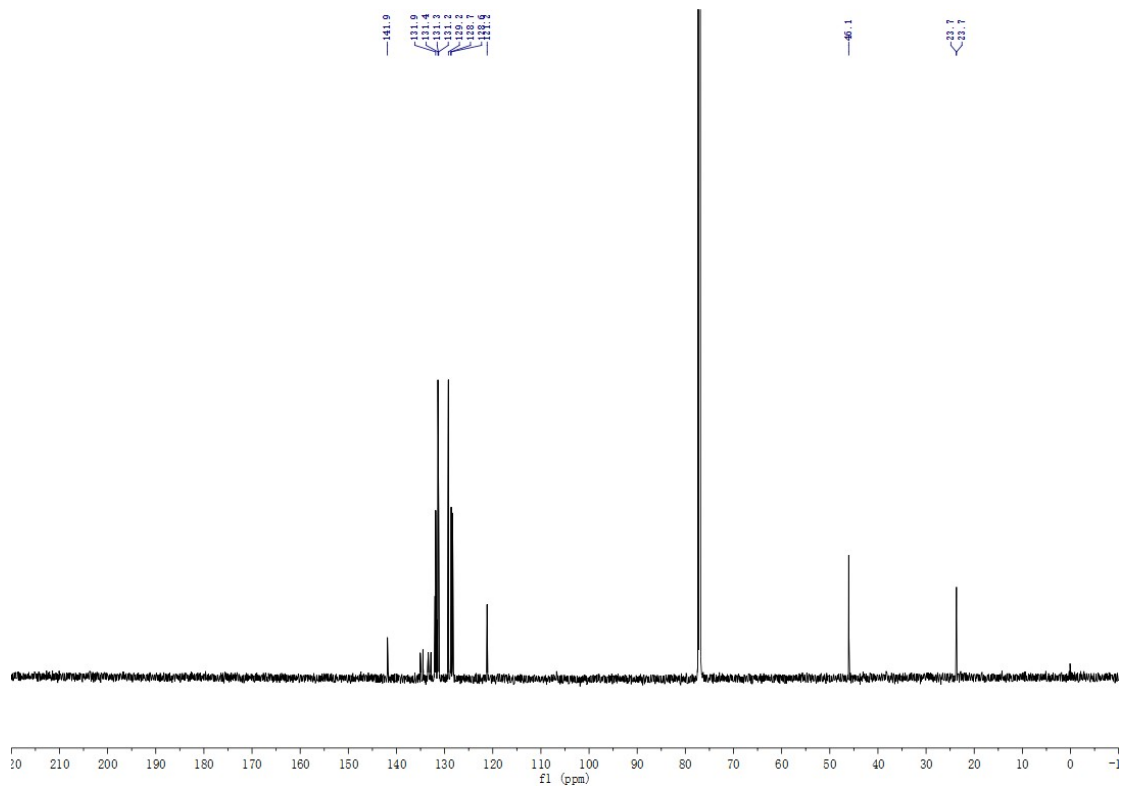




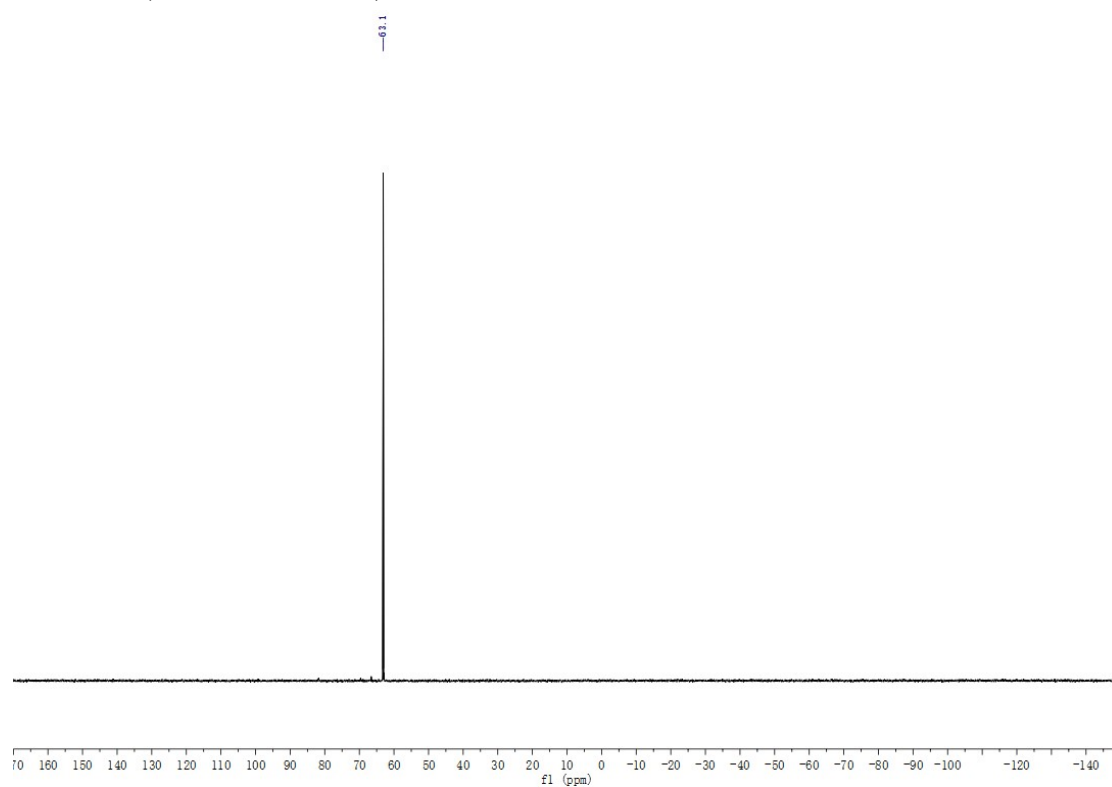
^1H NMR (600 MHz, CDCl_3)



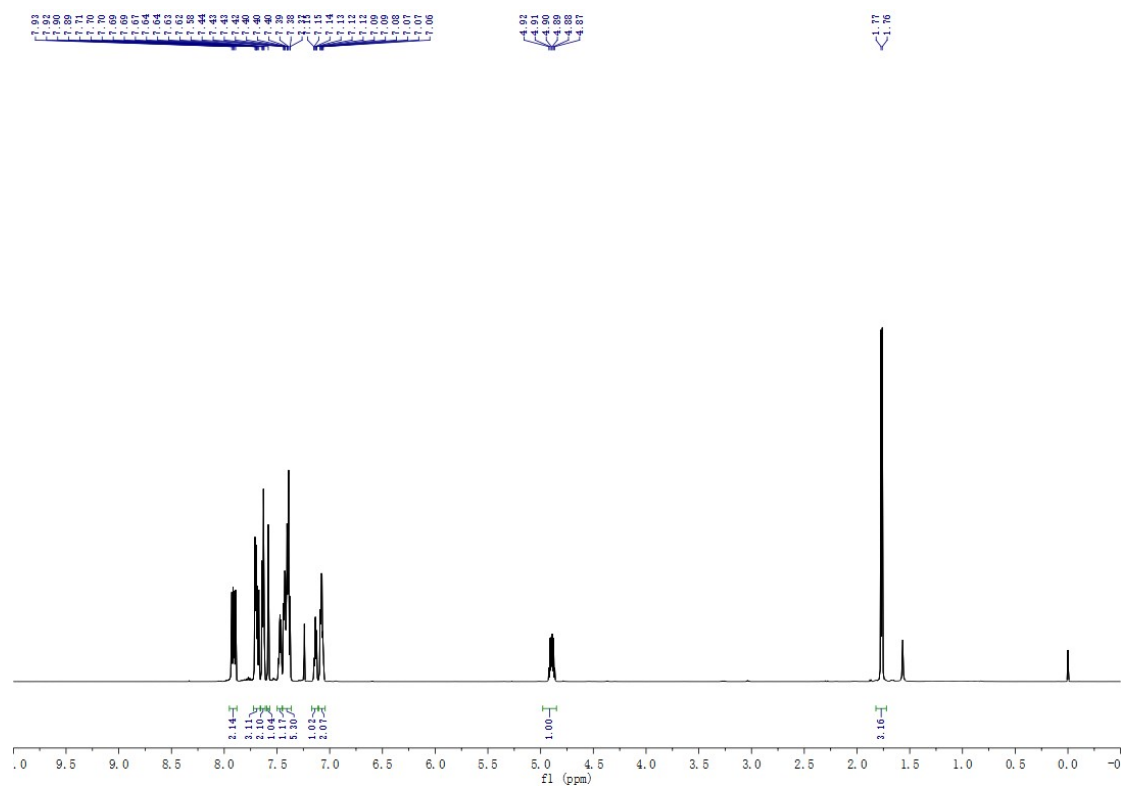
^{13}C NMR (151 MHz, CDCl_3)



^{31}P NMR (243 MHz, CDCl_3)

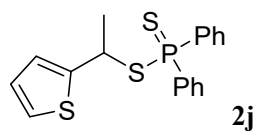


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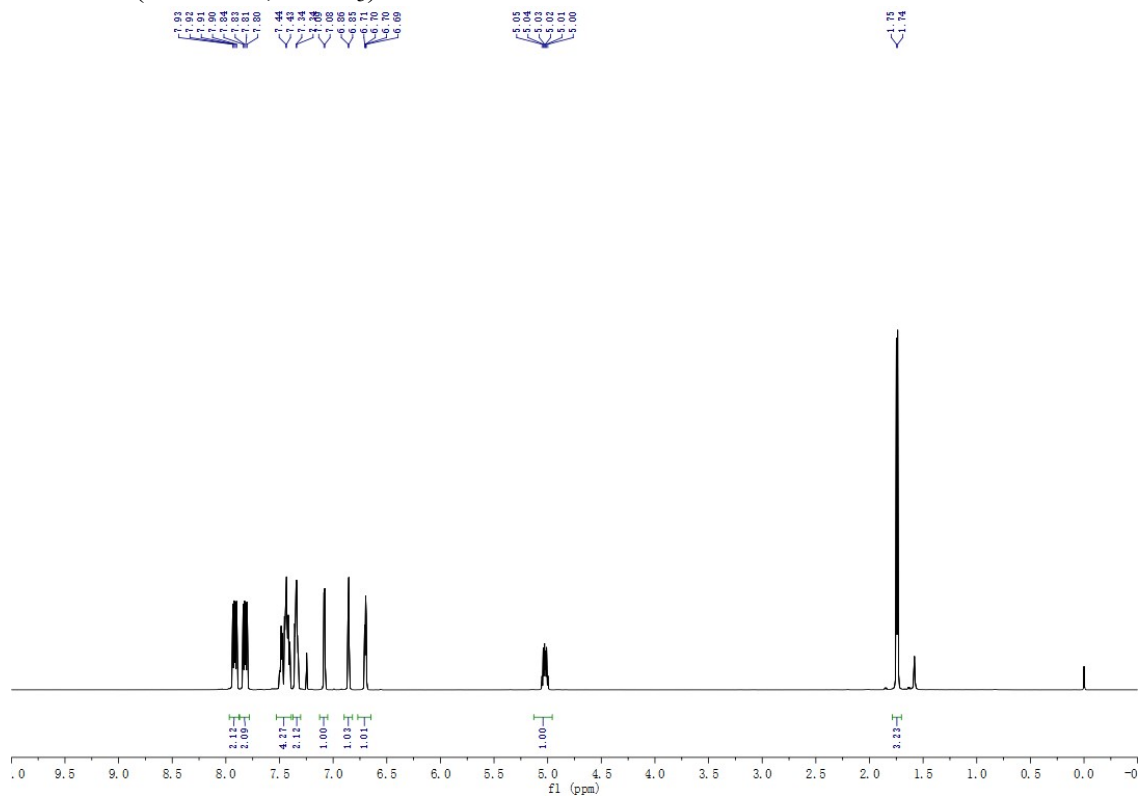


[illegible]

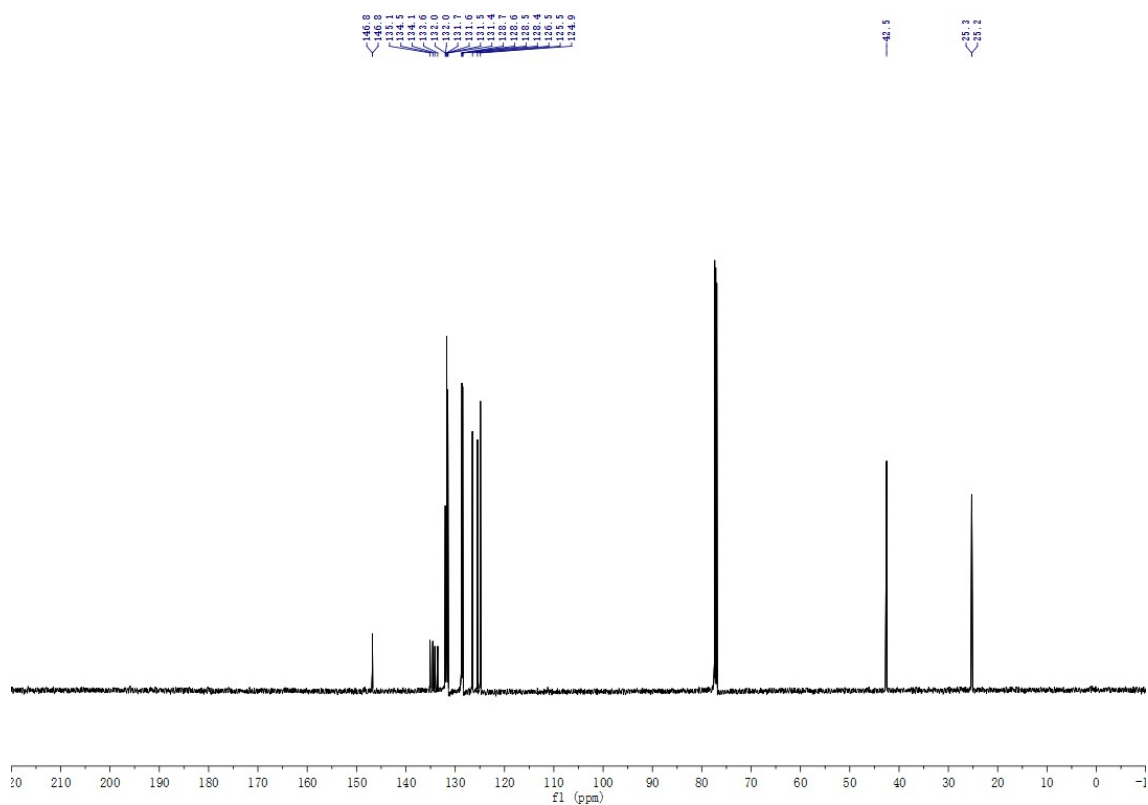
13C NMR spectrum of 1,2-dichloroethane in CDCl₃. The x-axis is labeled 'f1 (ppm)' and ranges from 170 to -140. A single sharp peak is visible at approximately 63 ppm, labeled '63.2'.



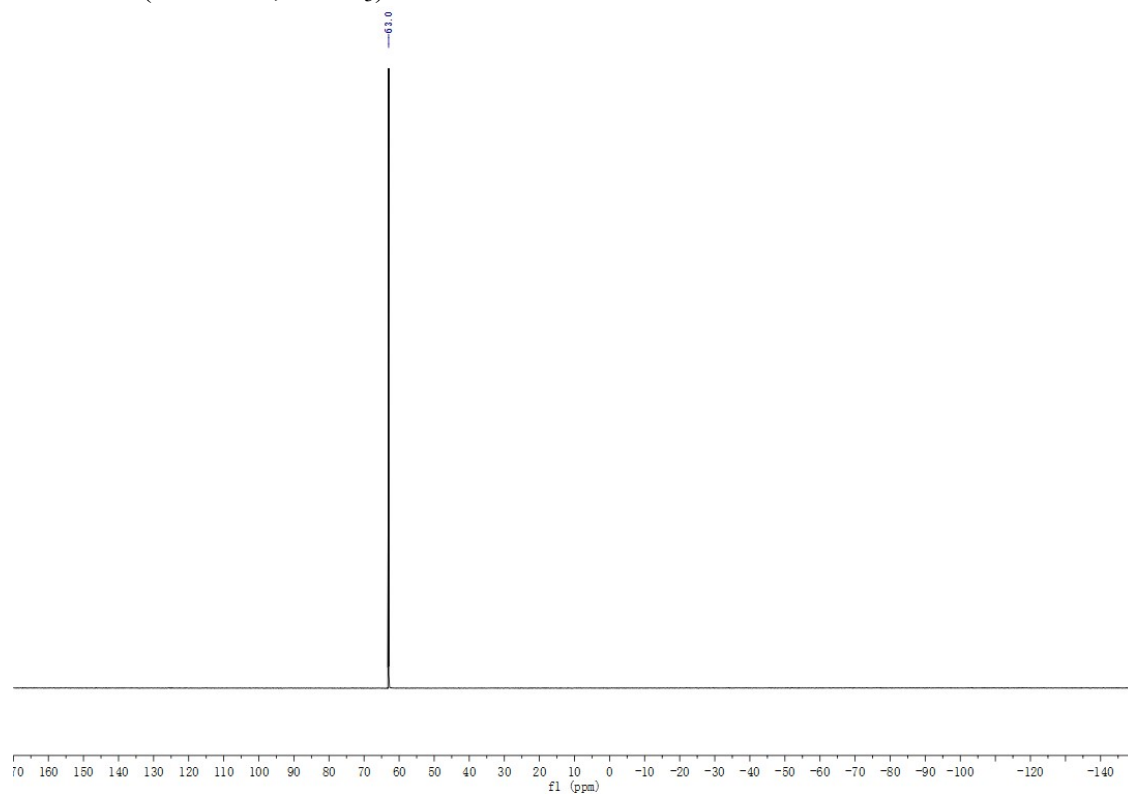
^1H NMR (600 MHz, CDCl_3)



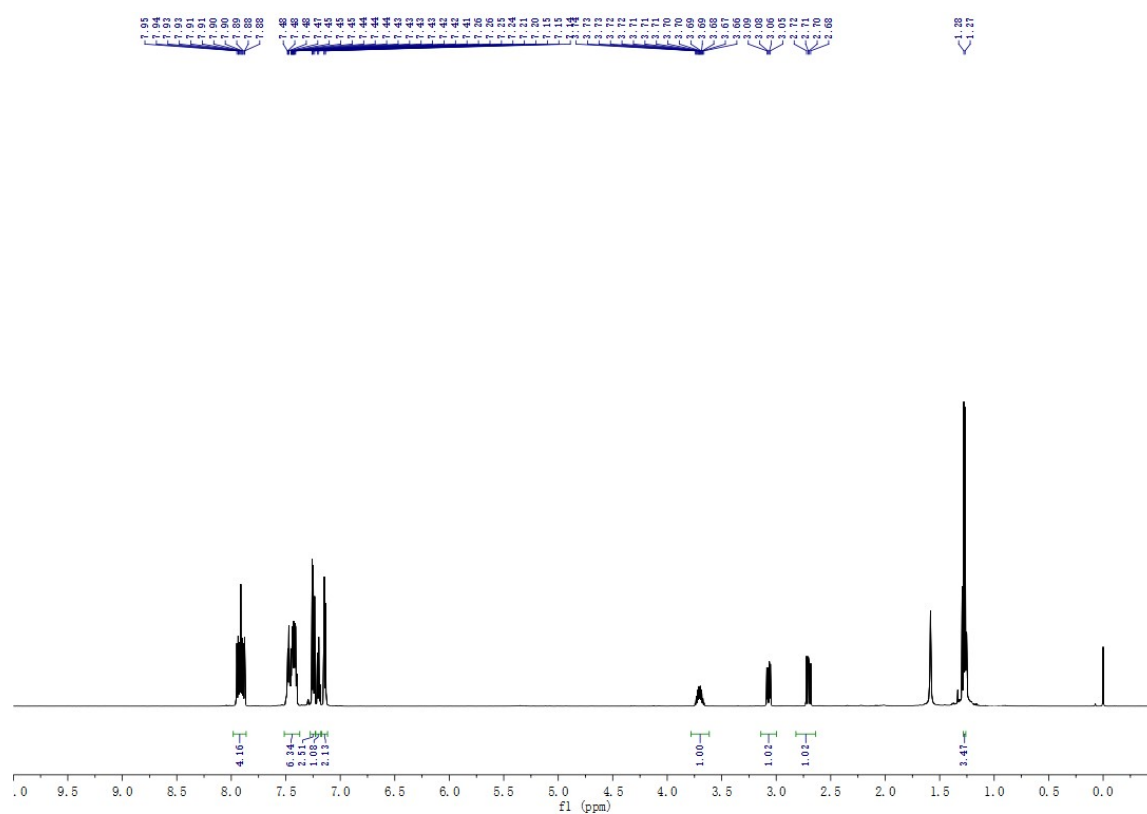
^{13}C NMR (151 MHz, CDCl_3)



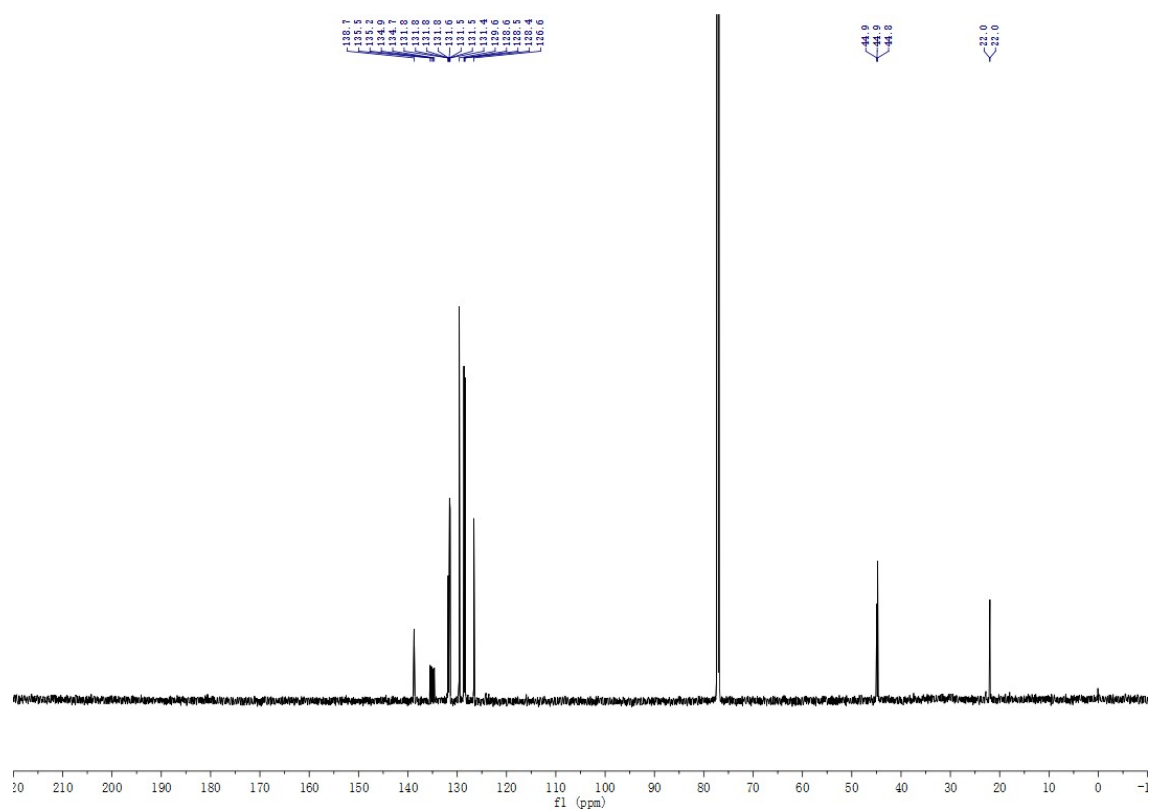
^{31}P NMR (243 MHz, CDCl_3)



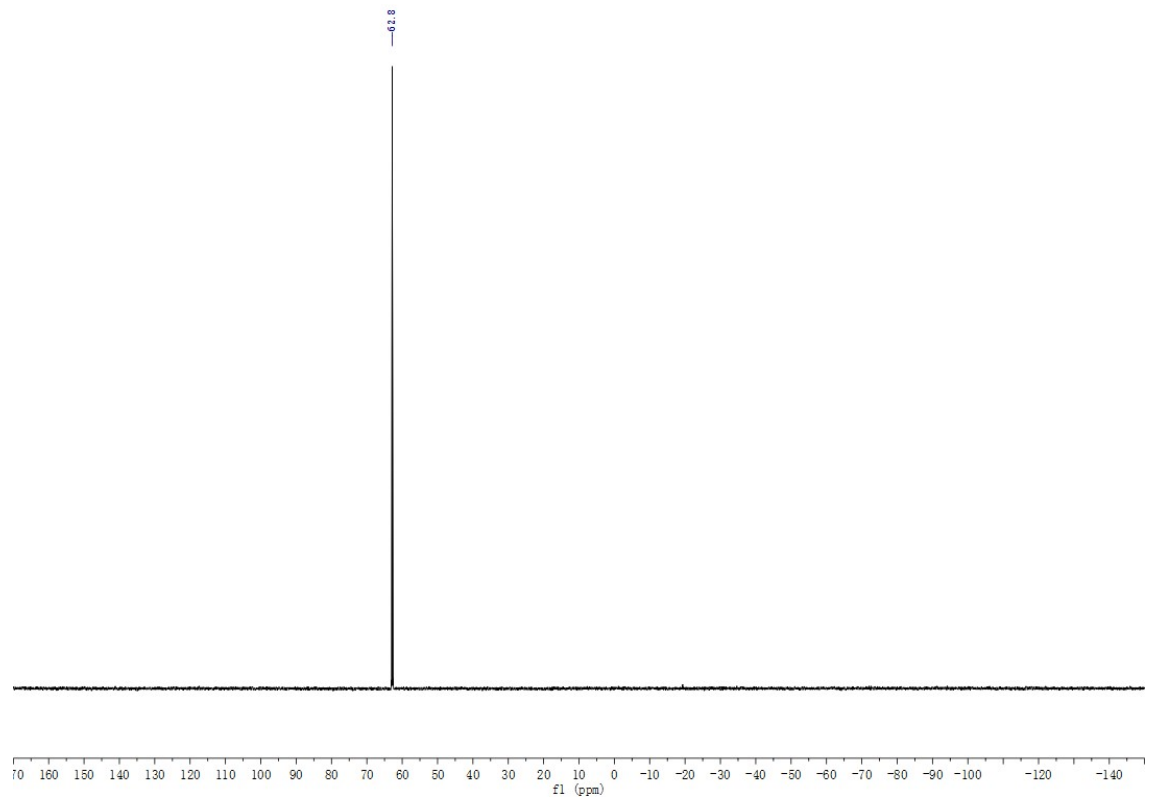
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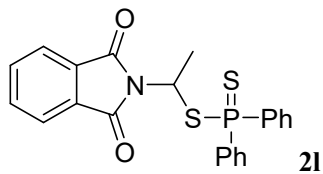


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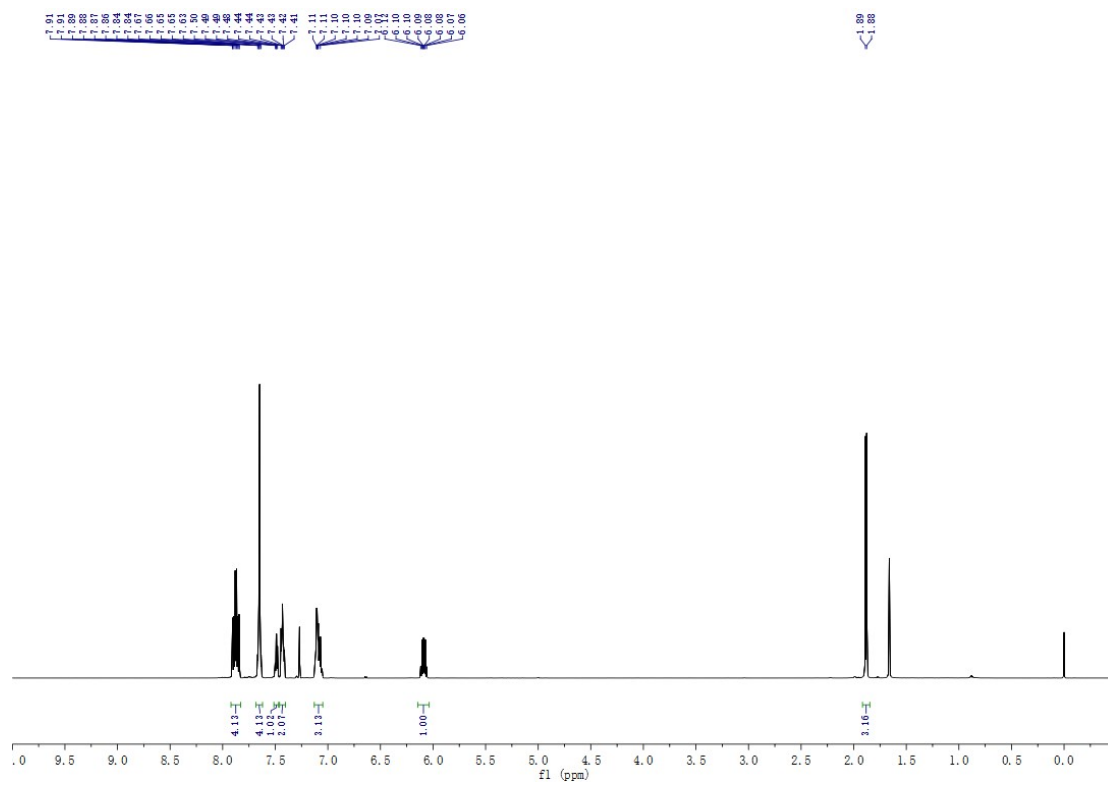


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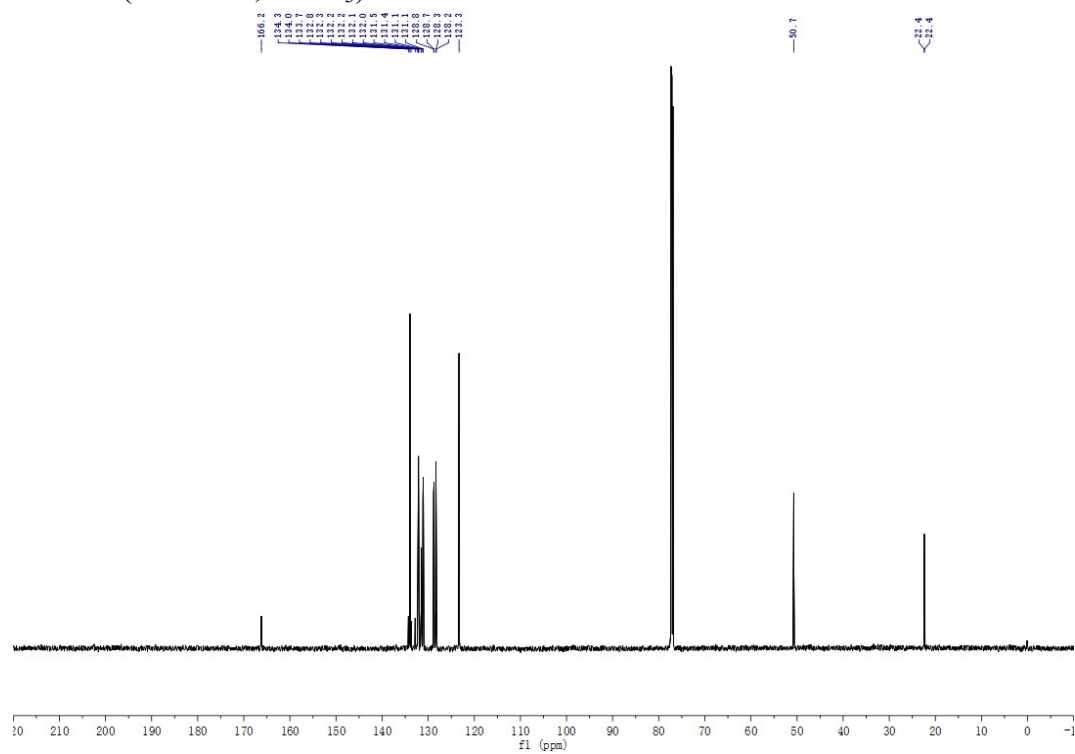




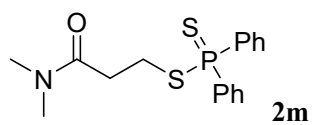
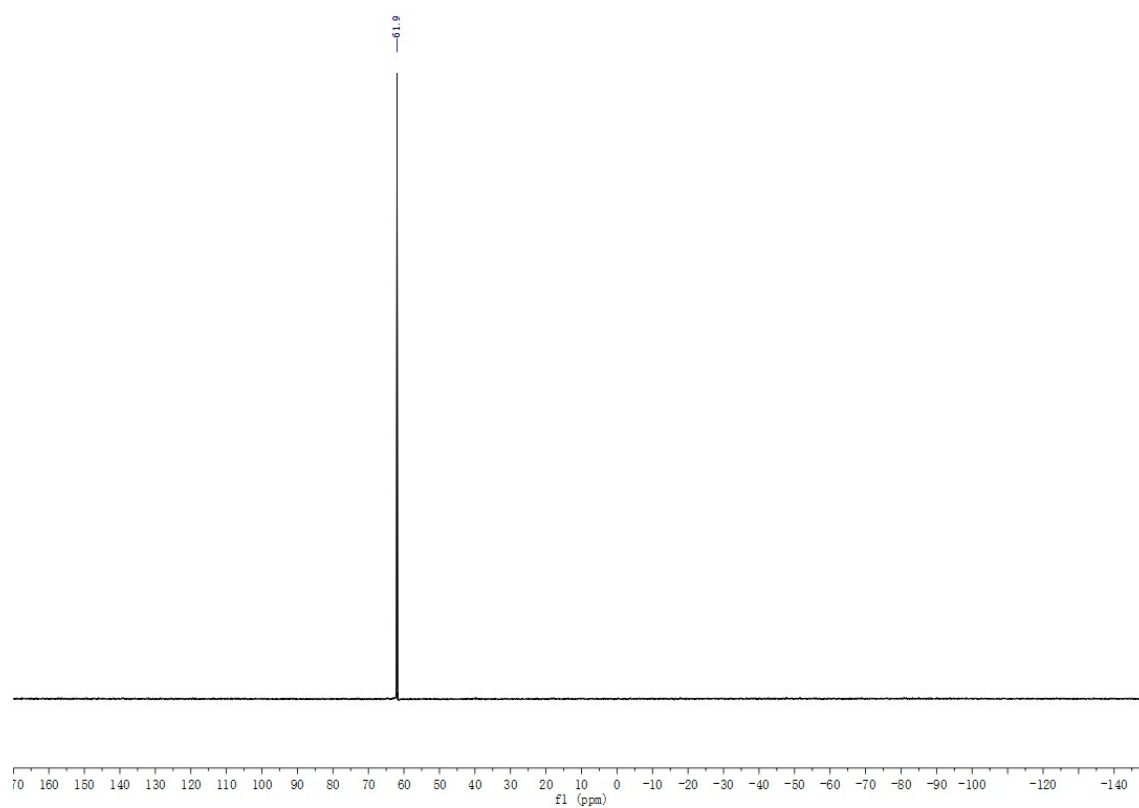
^1H NMR (600 MHz, CDCl_3)



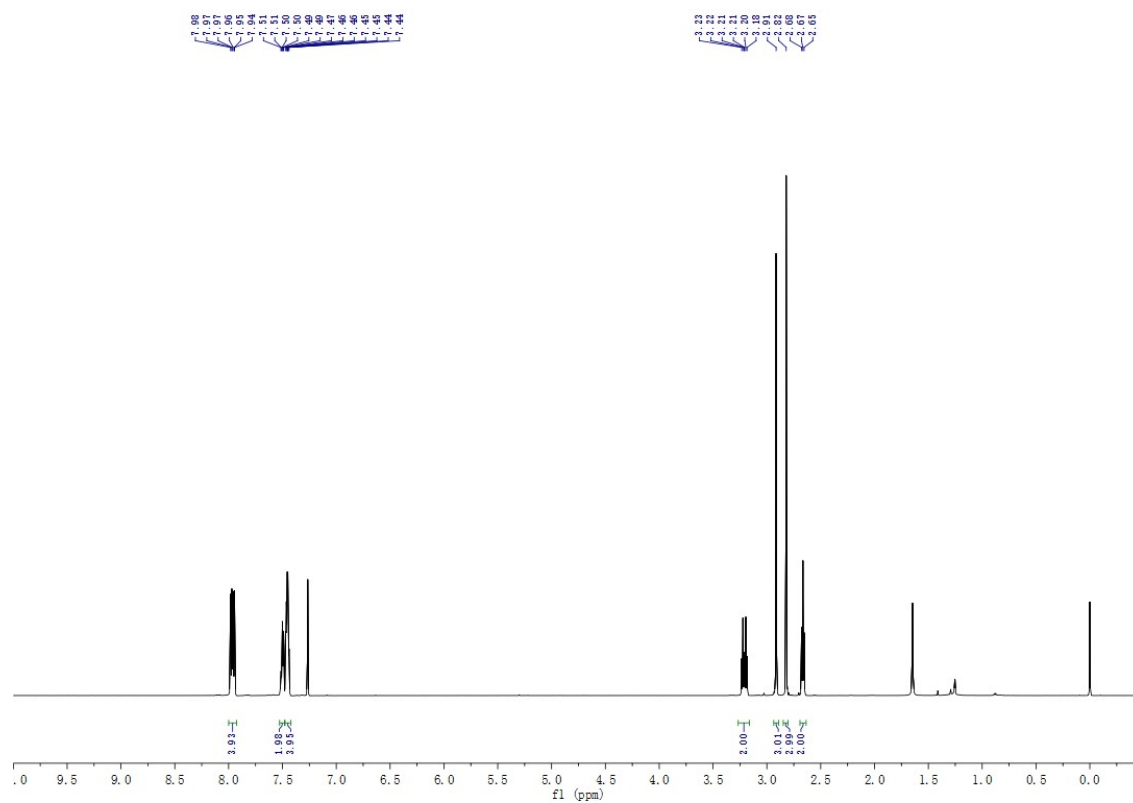
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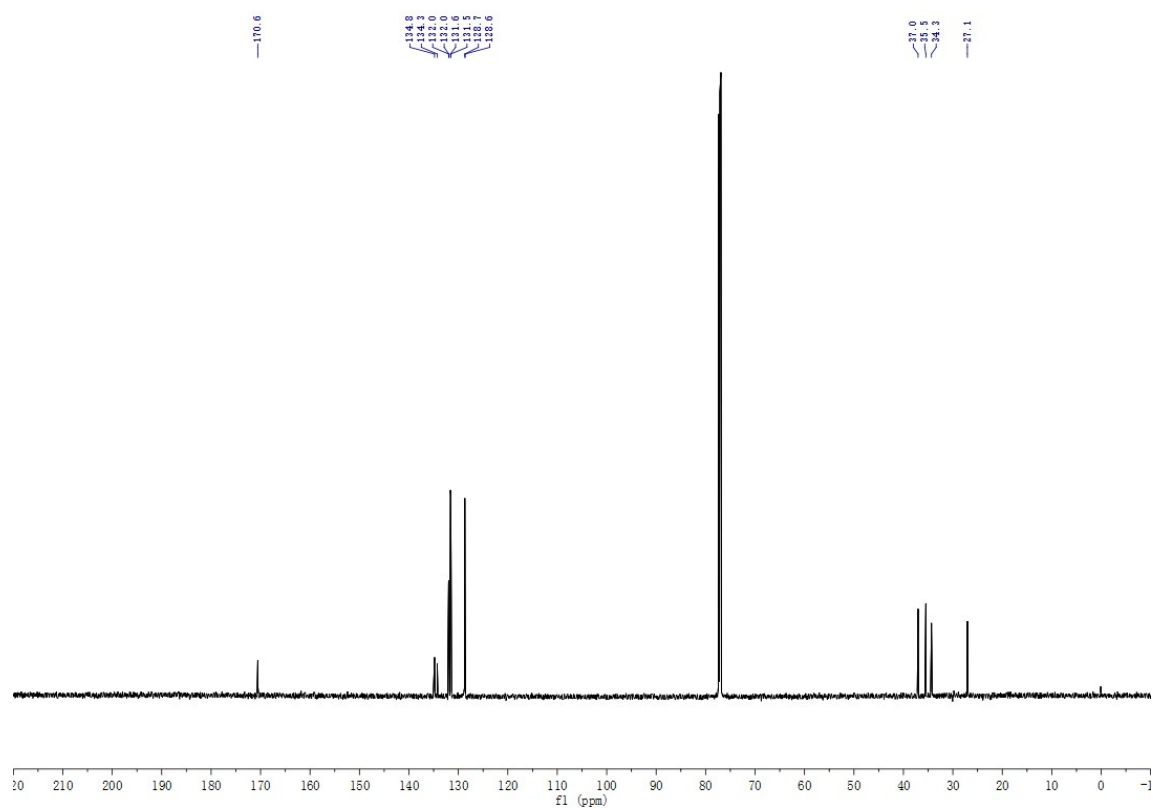
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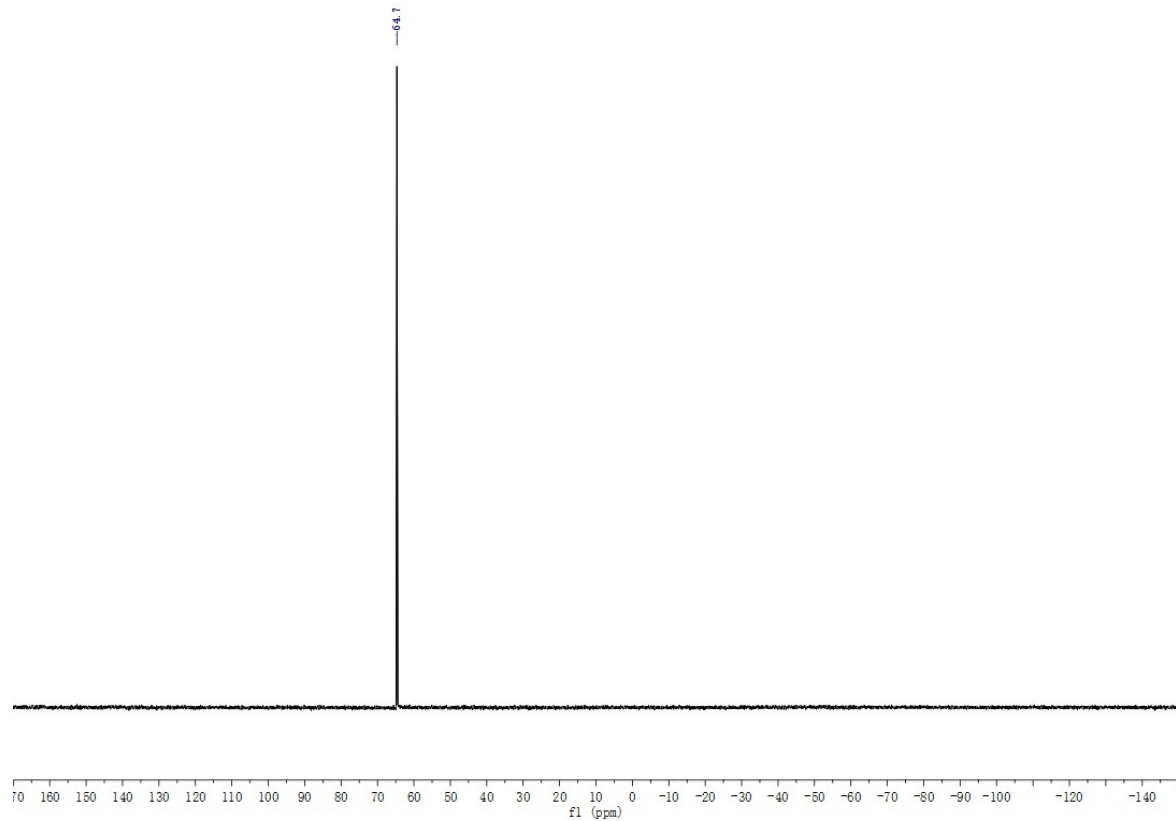
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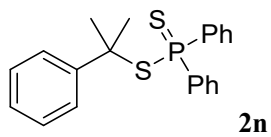


^{13}C NMR (151 MHz, CDCl_3)

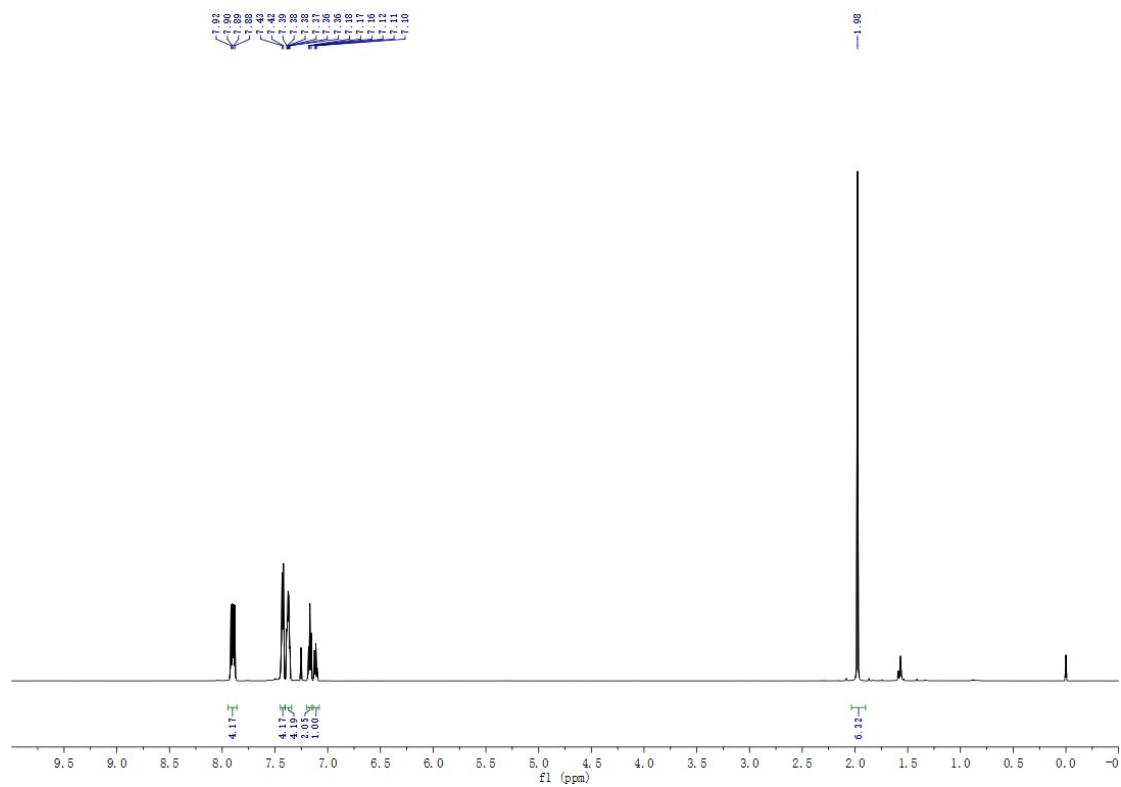


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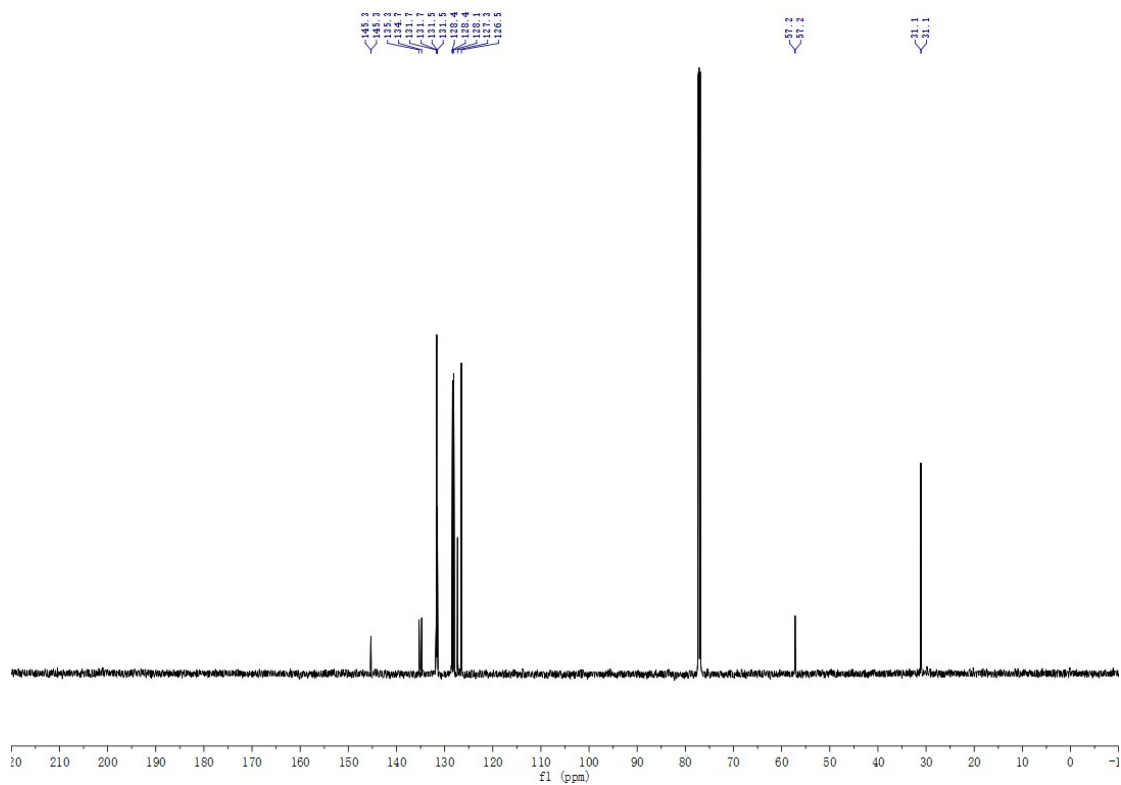




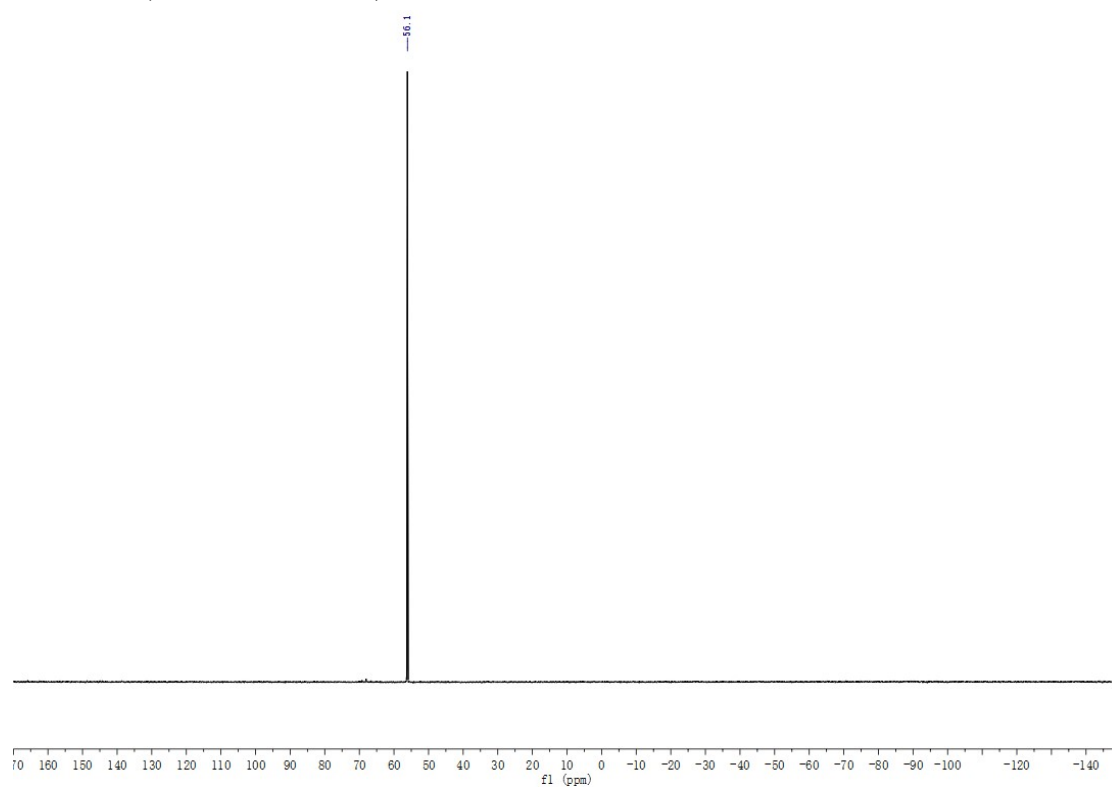
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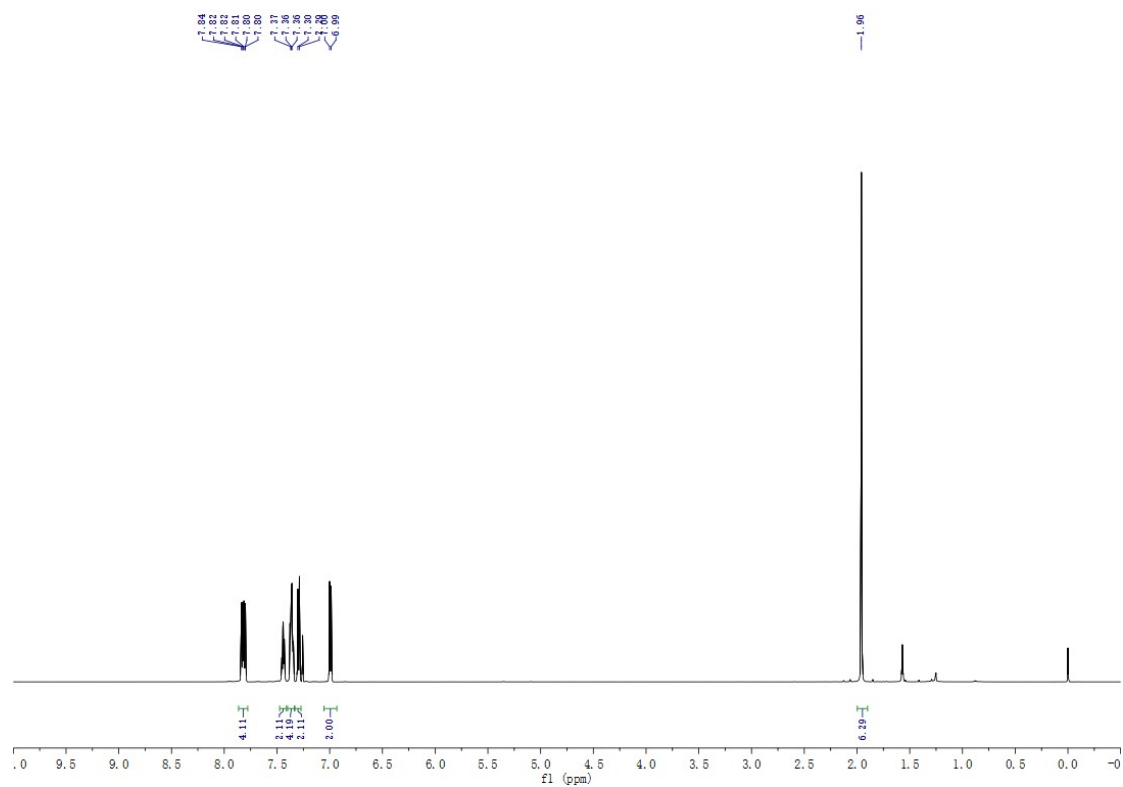
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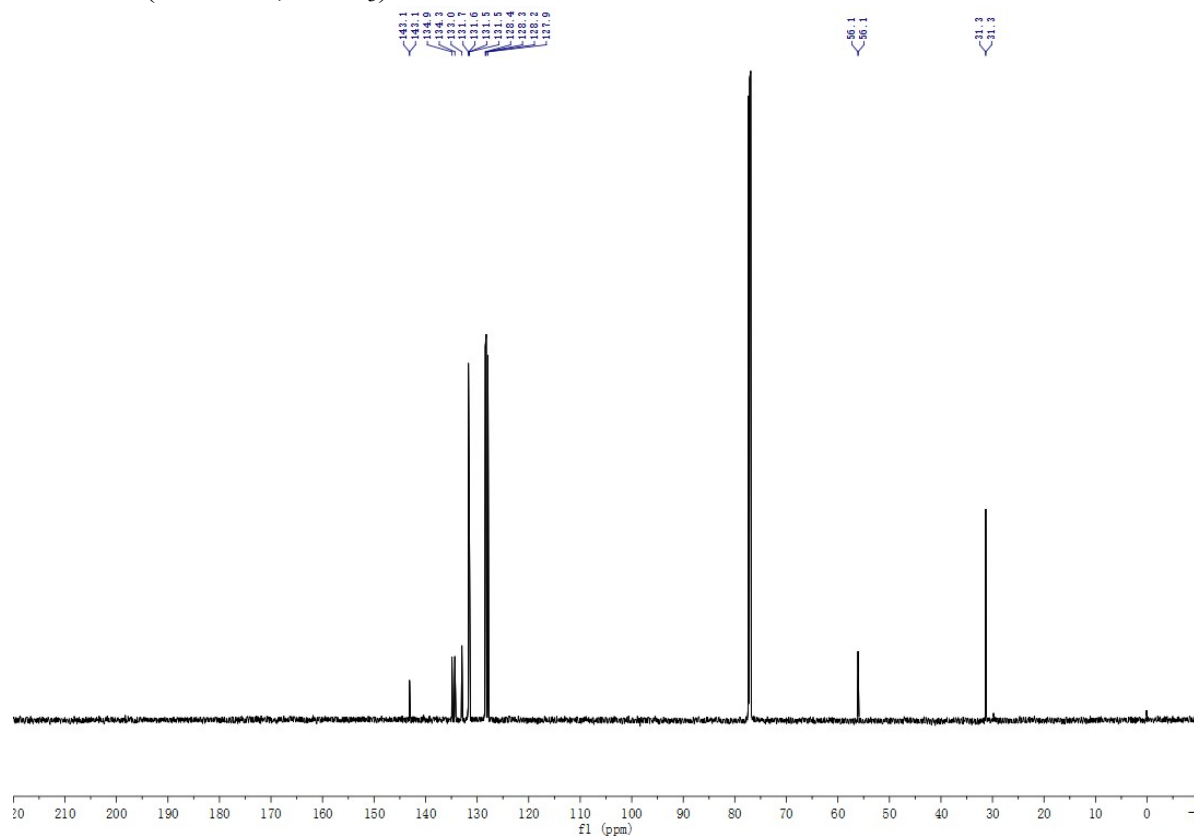
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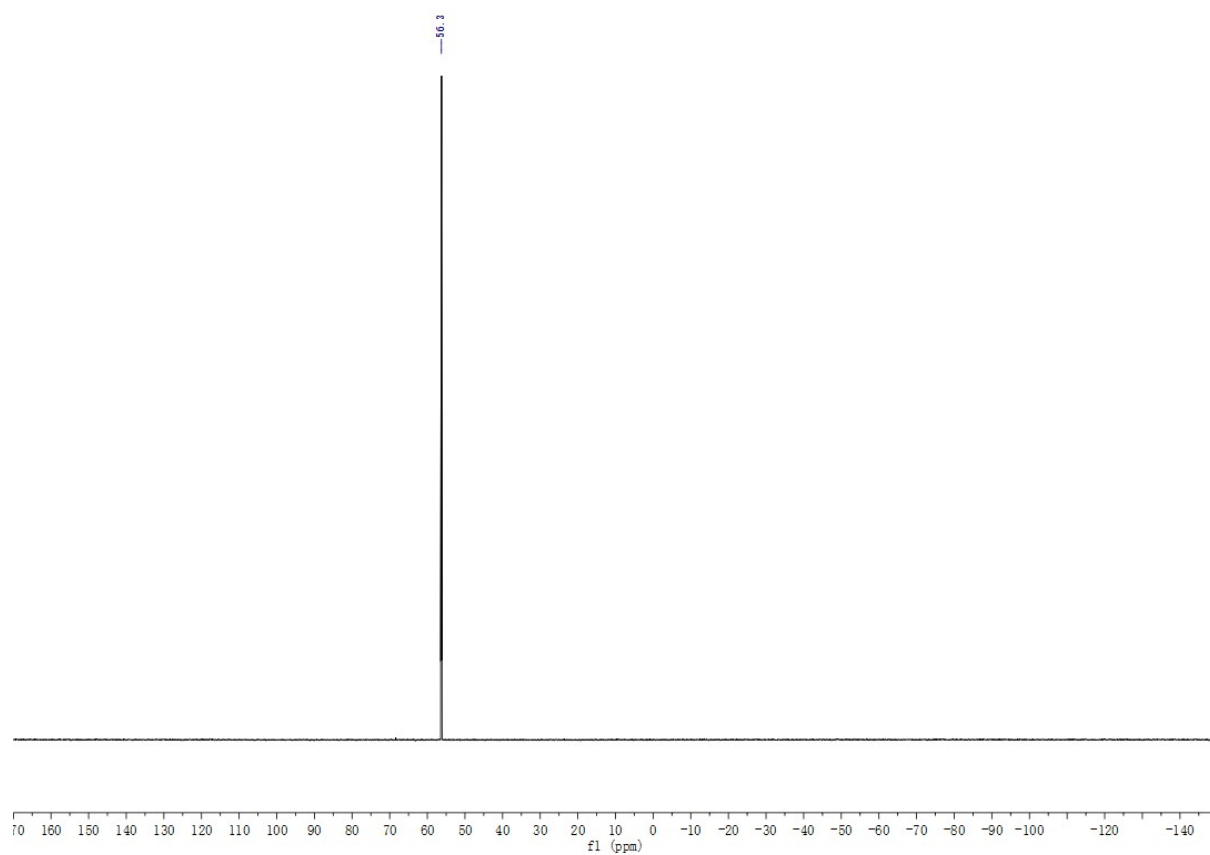
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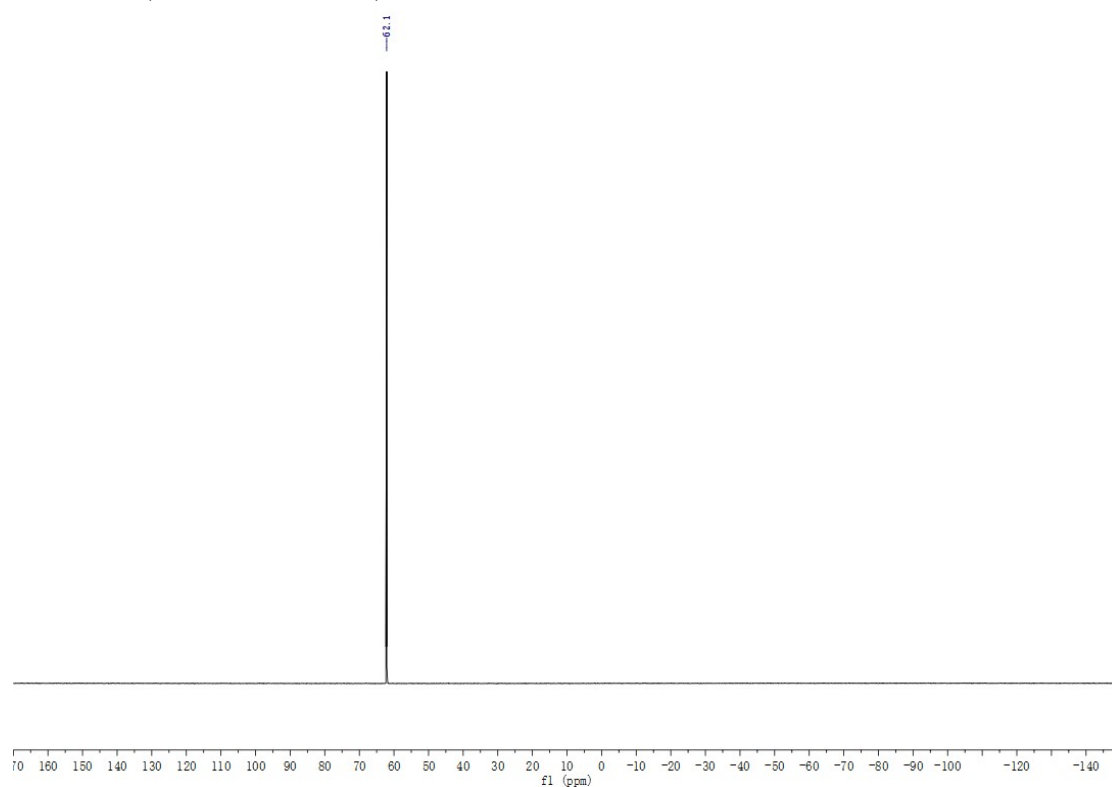


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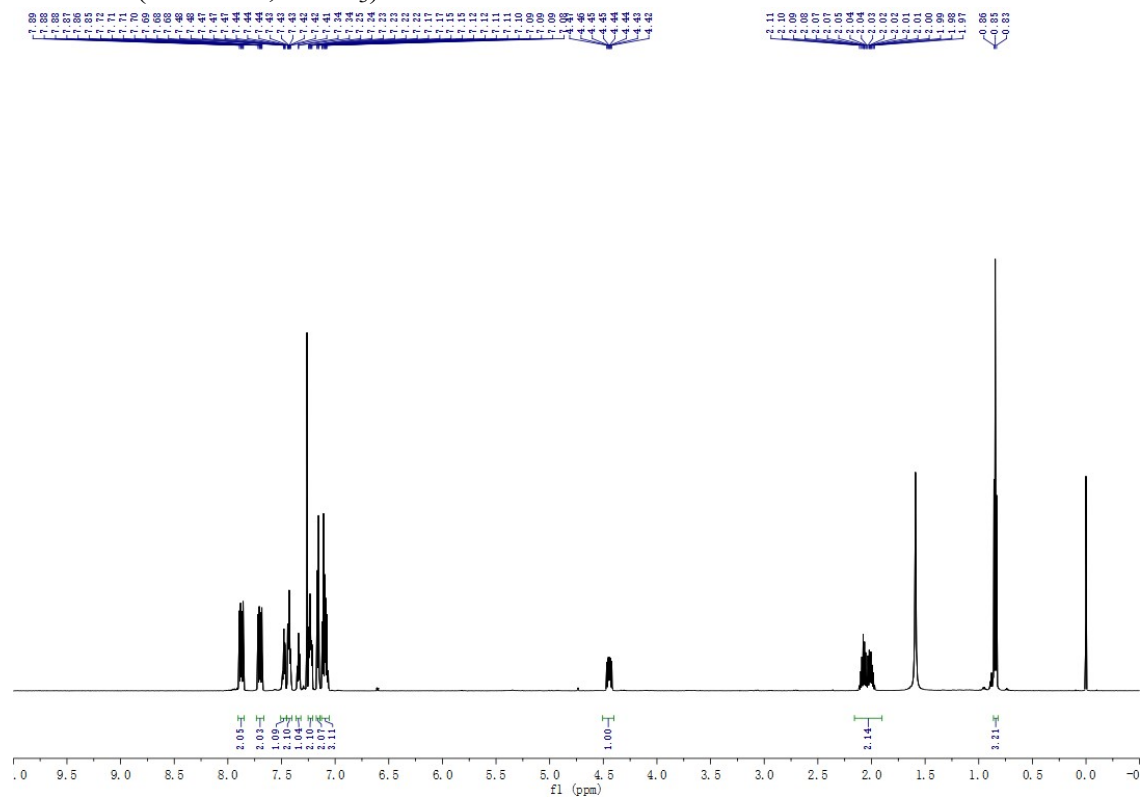




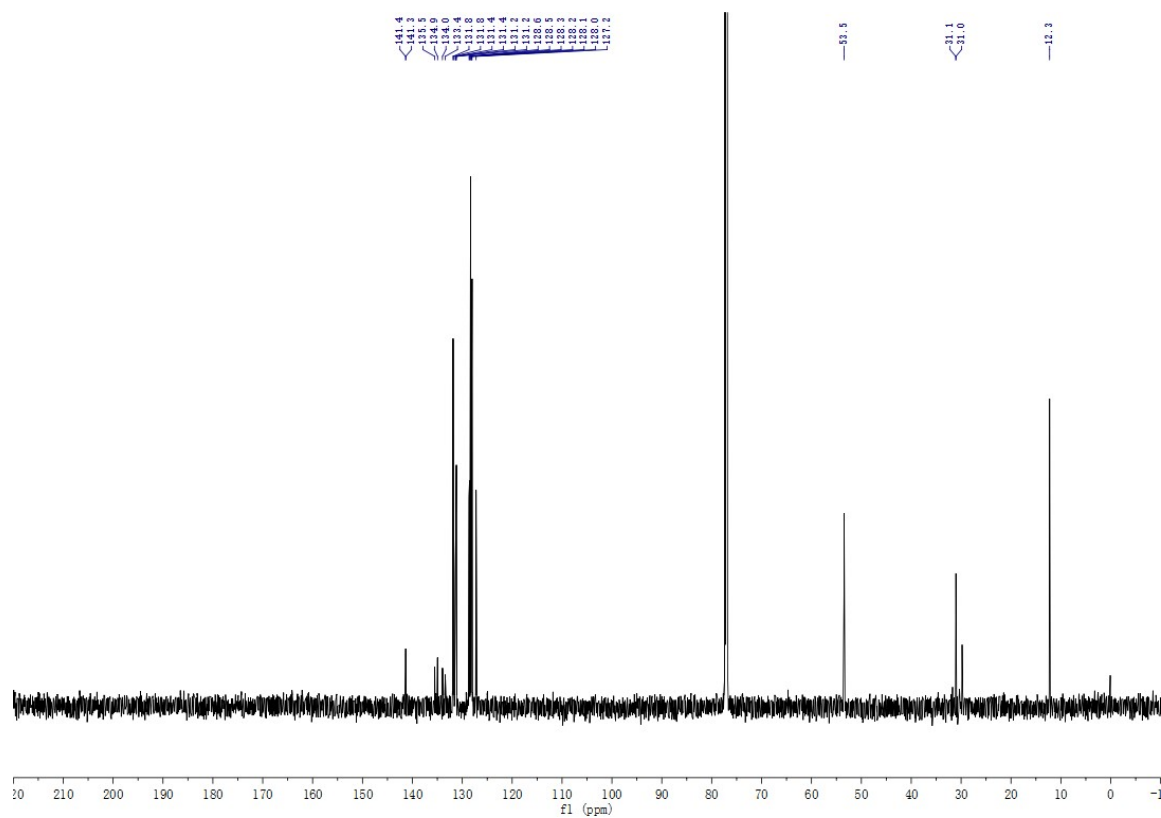
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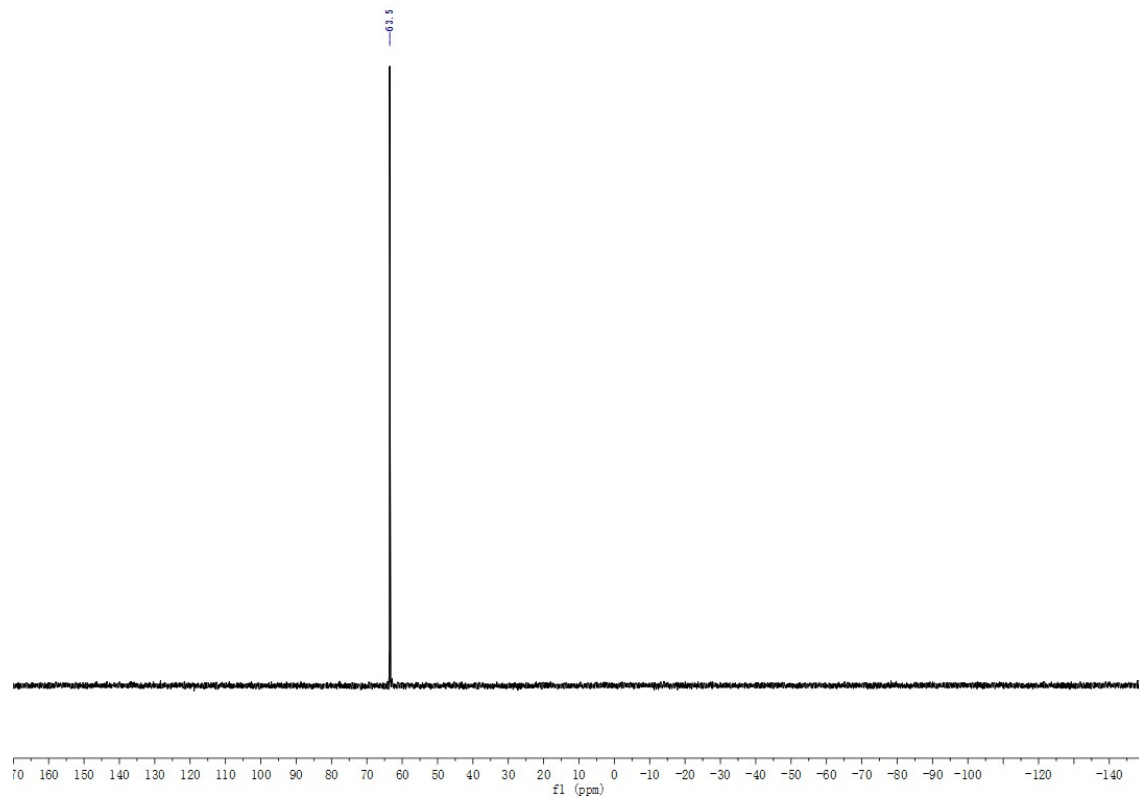
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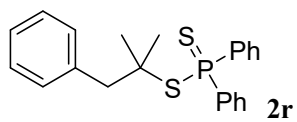


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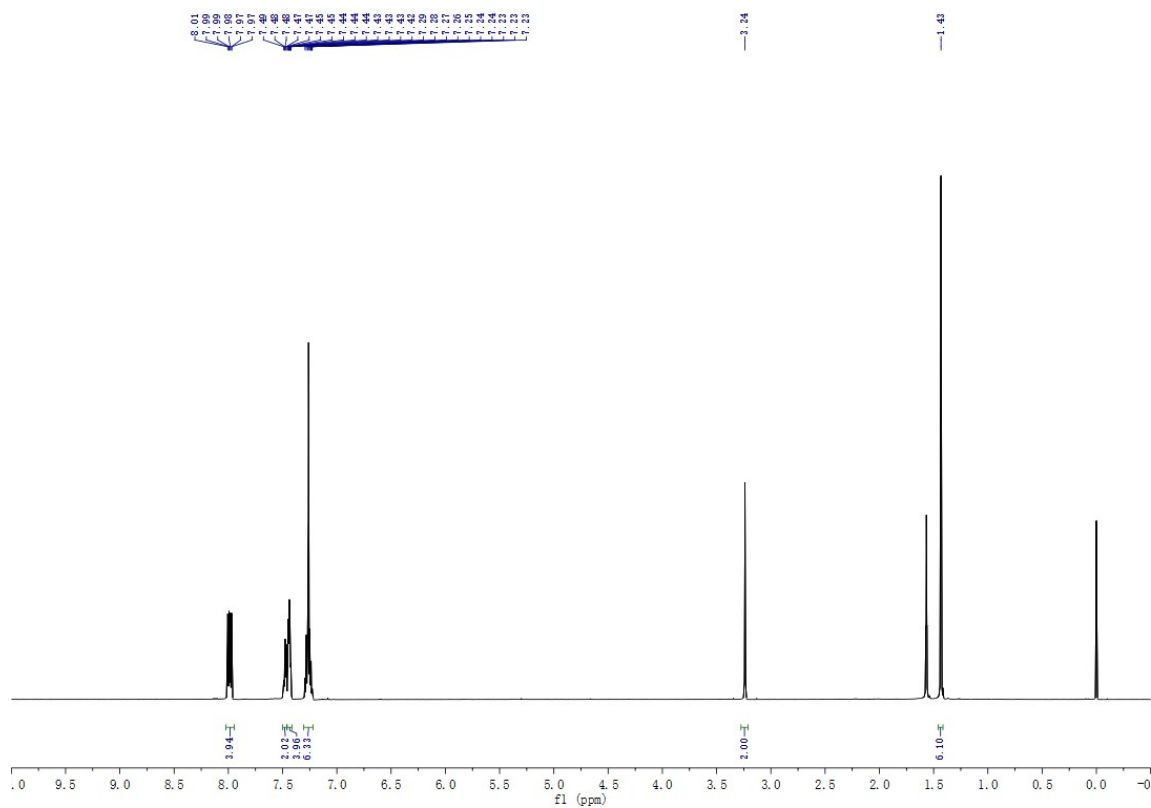


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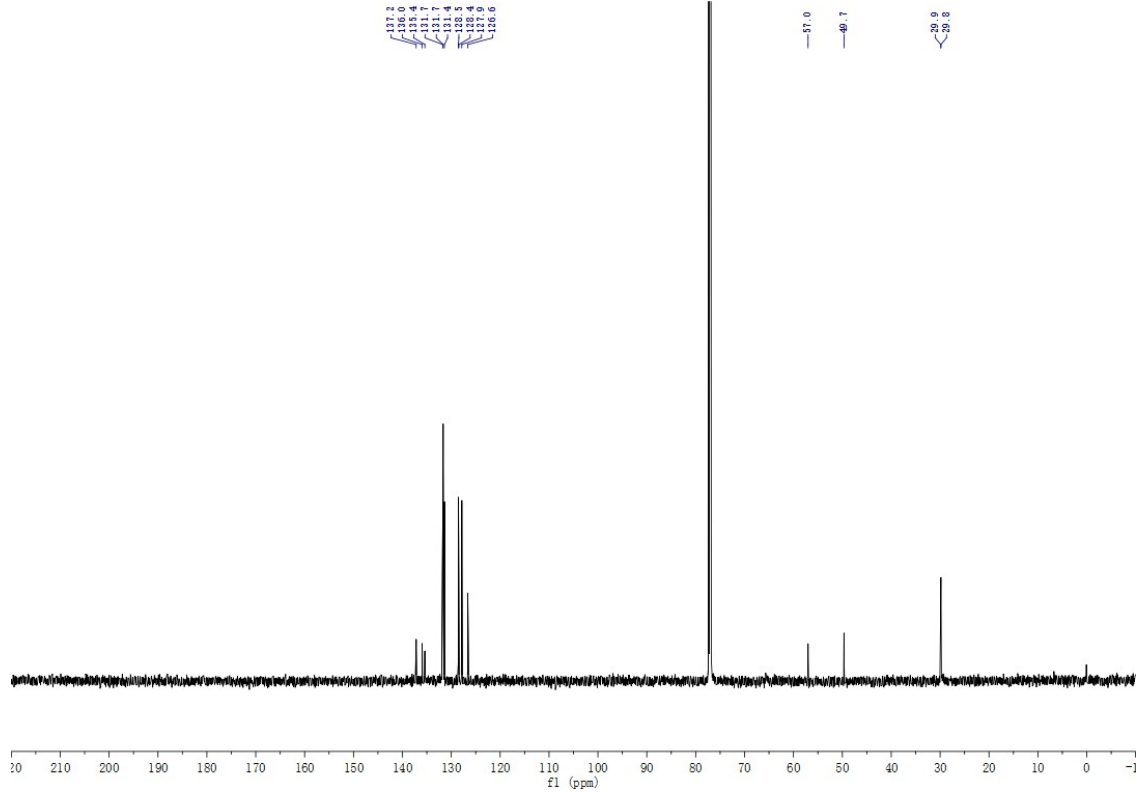




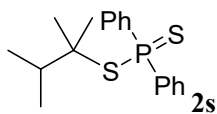
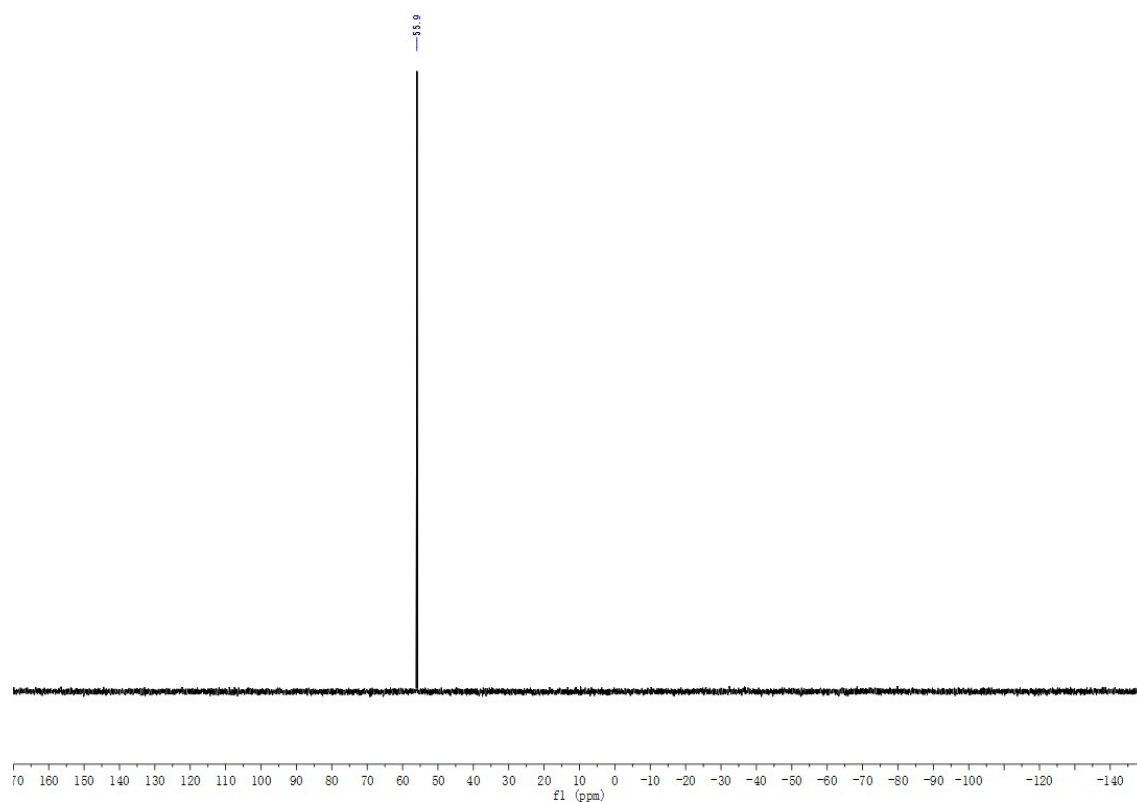
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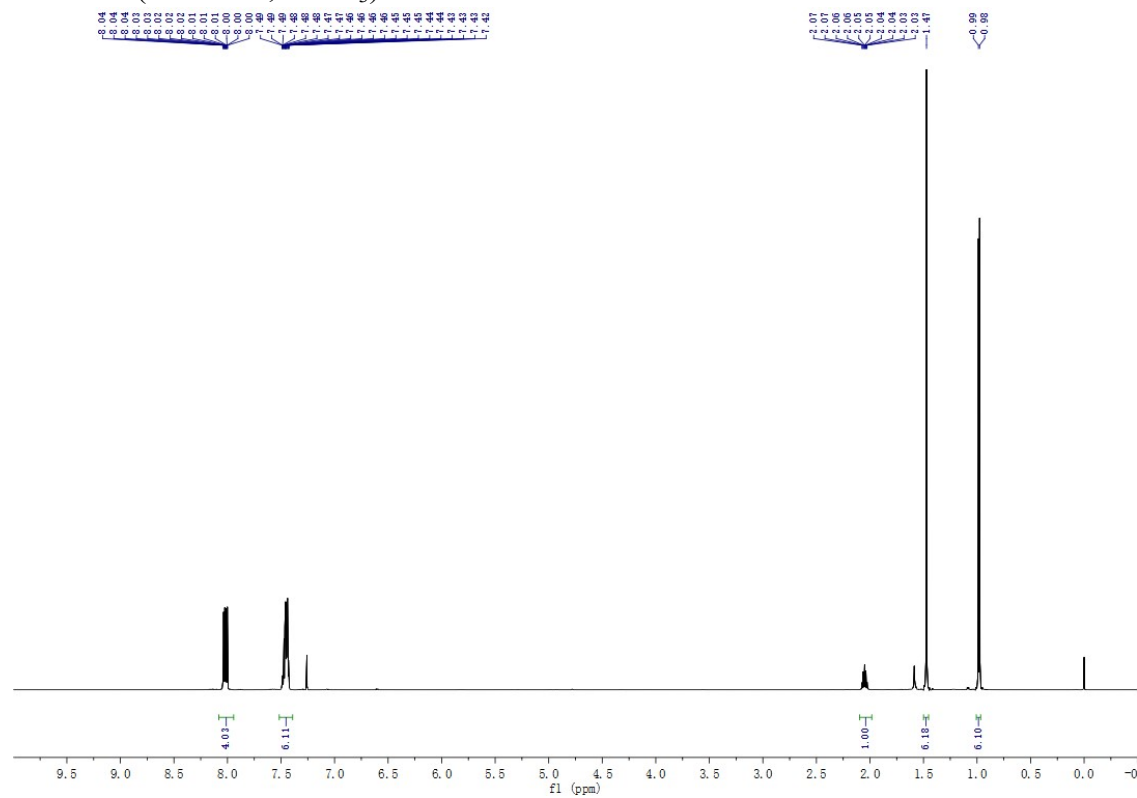
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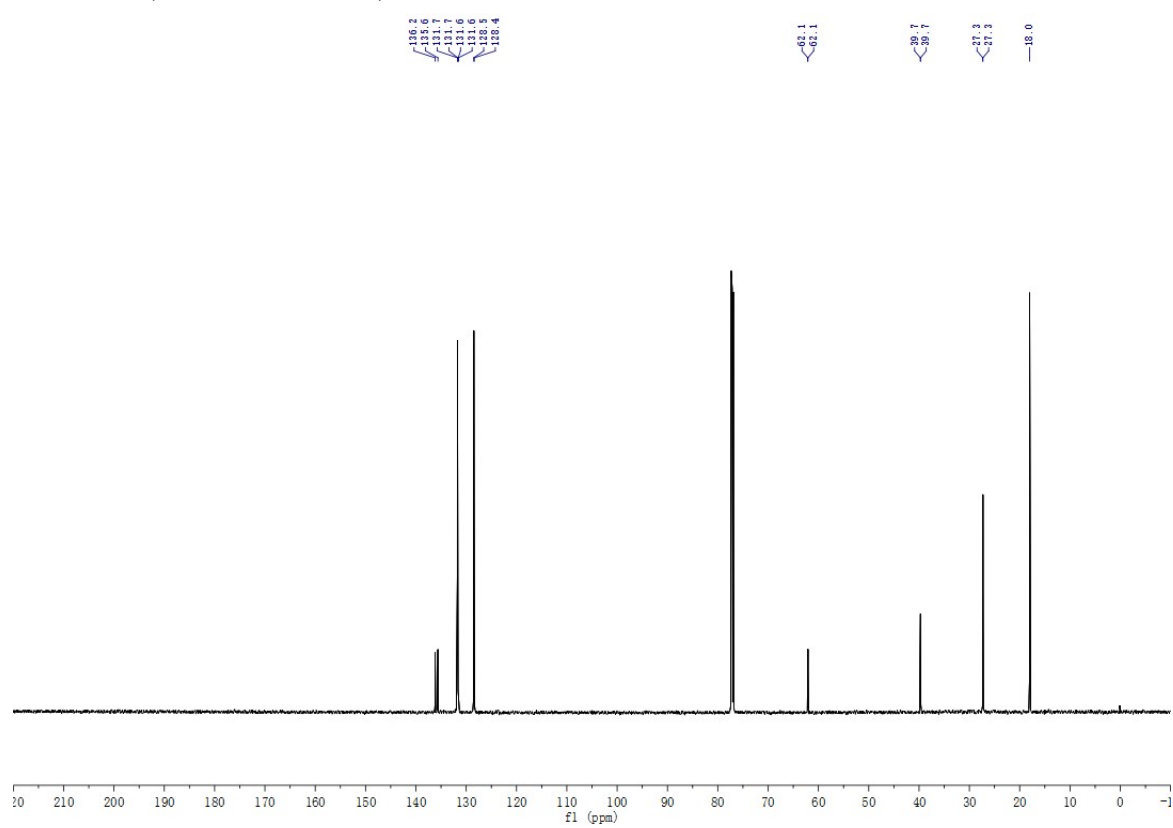
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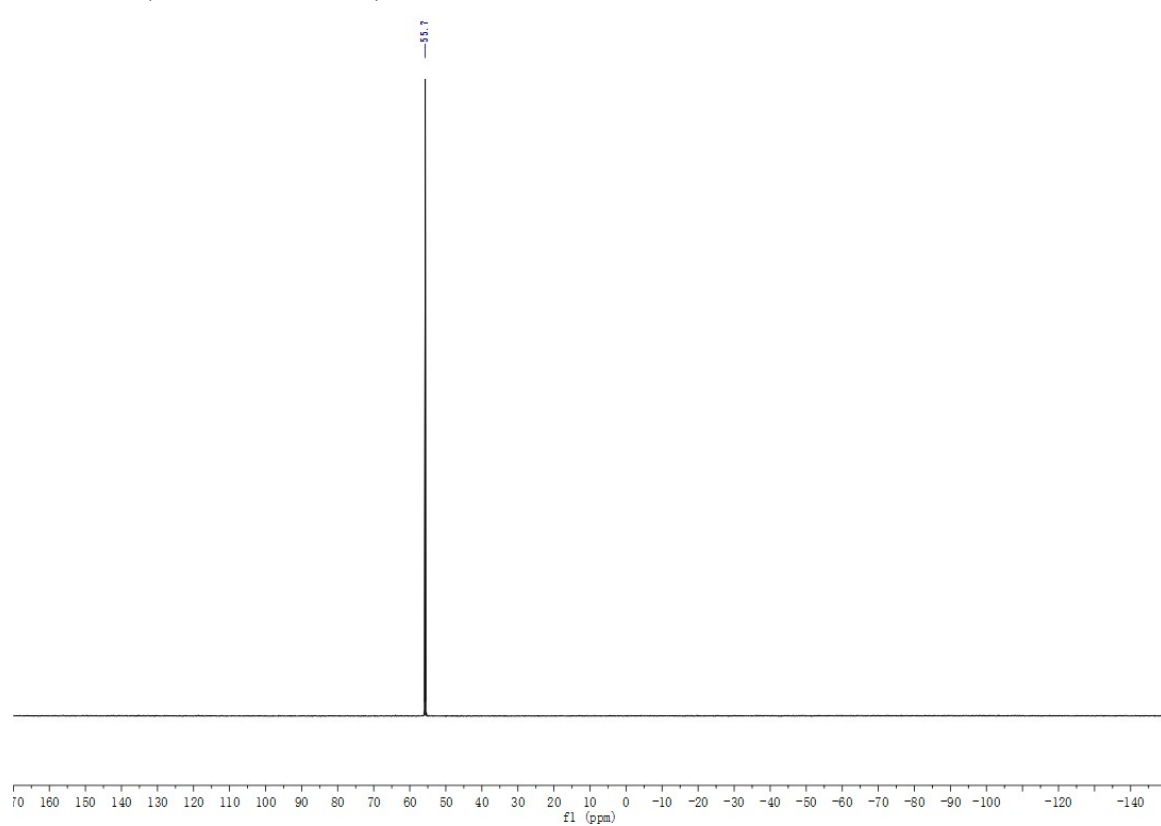
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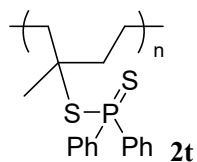


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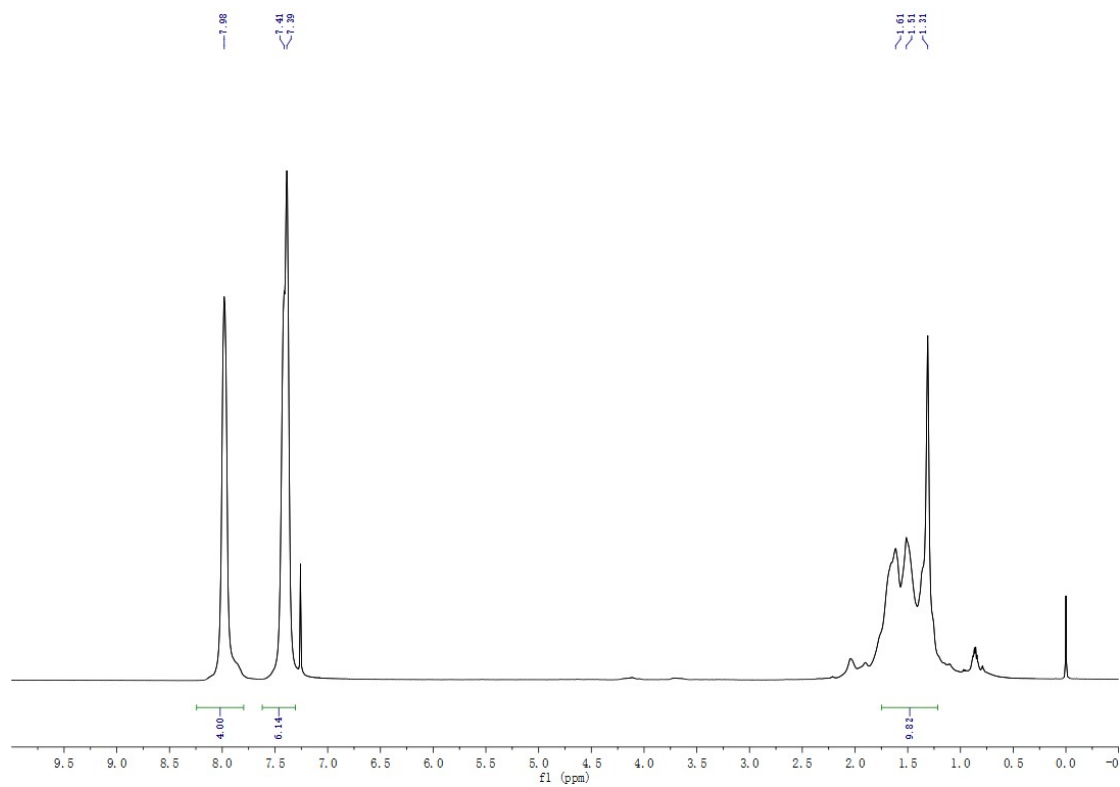


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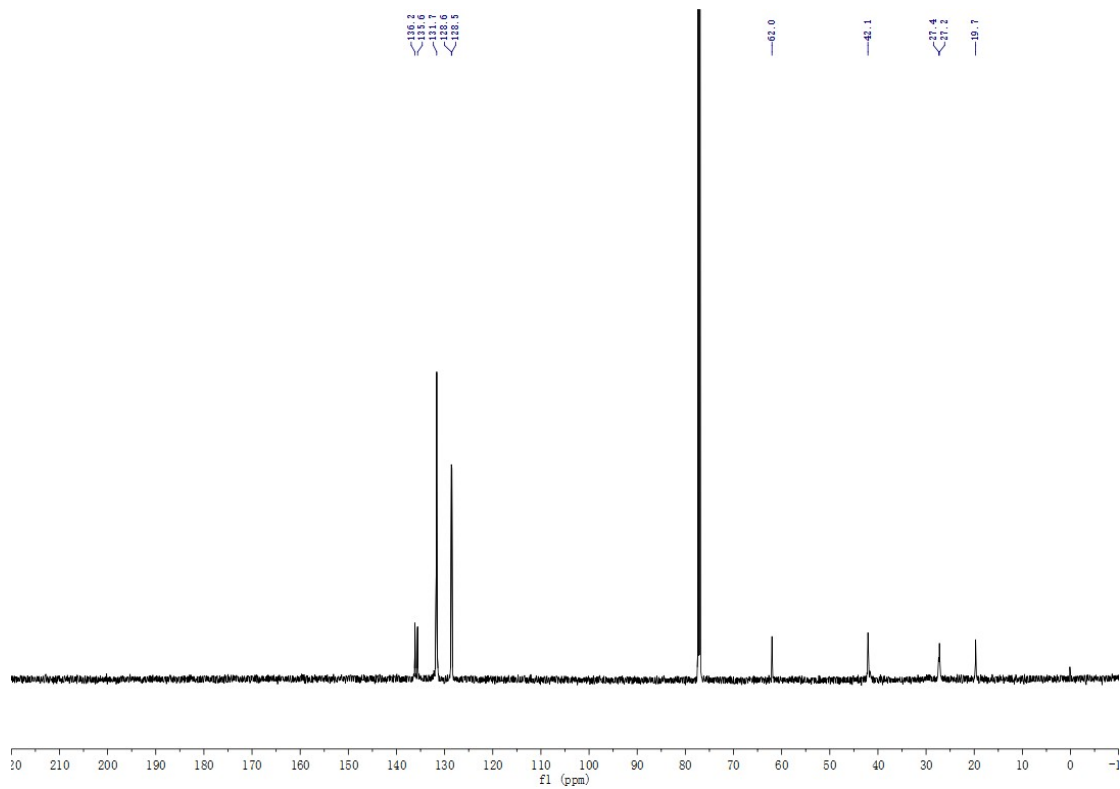




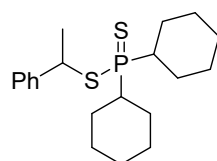
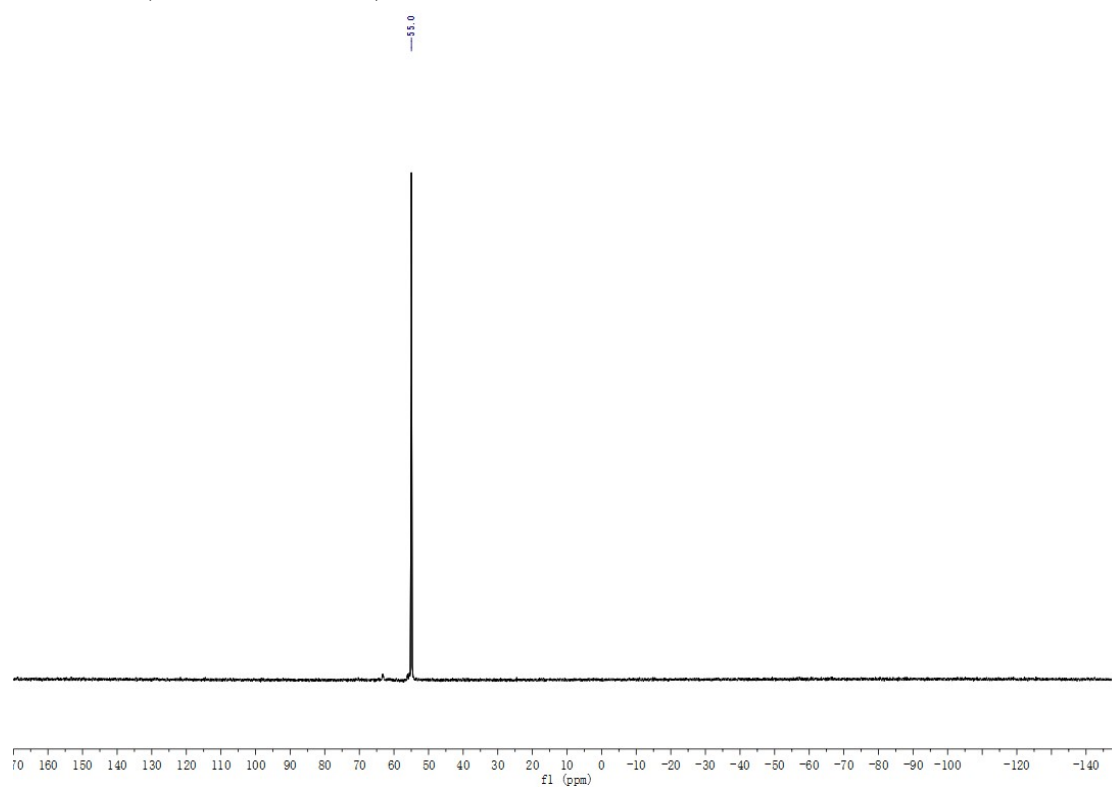
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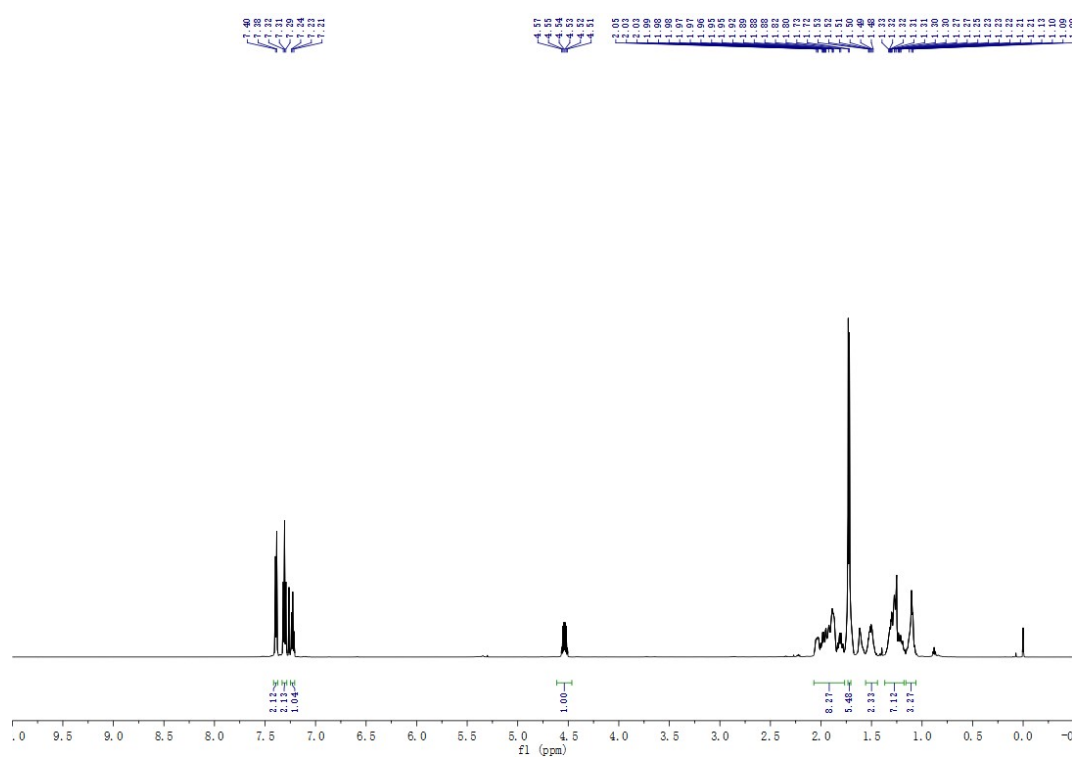


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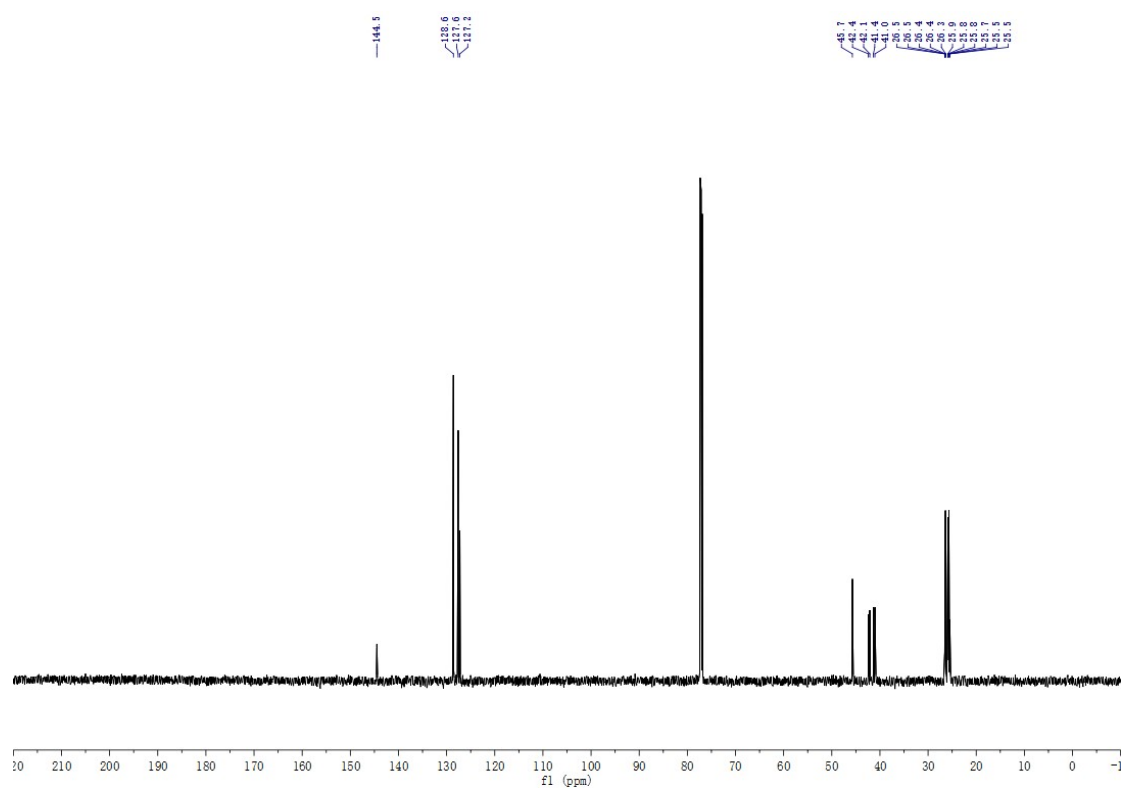


2u

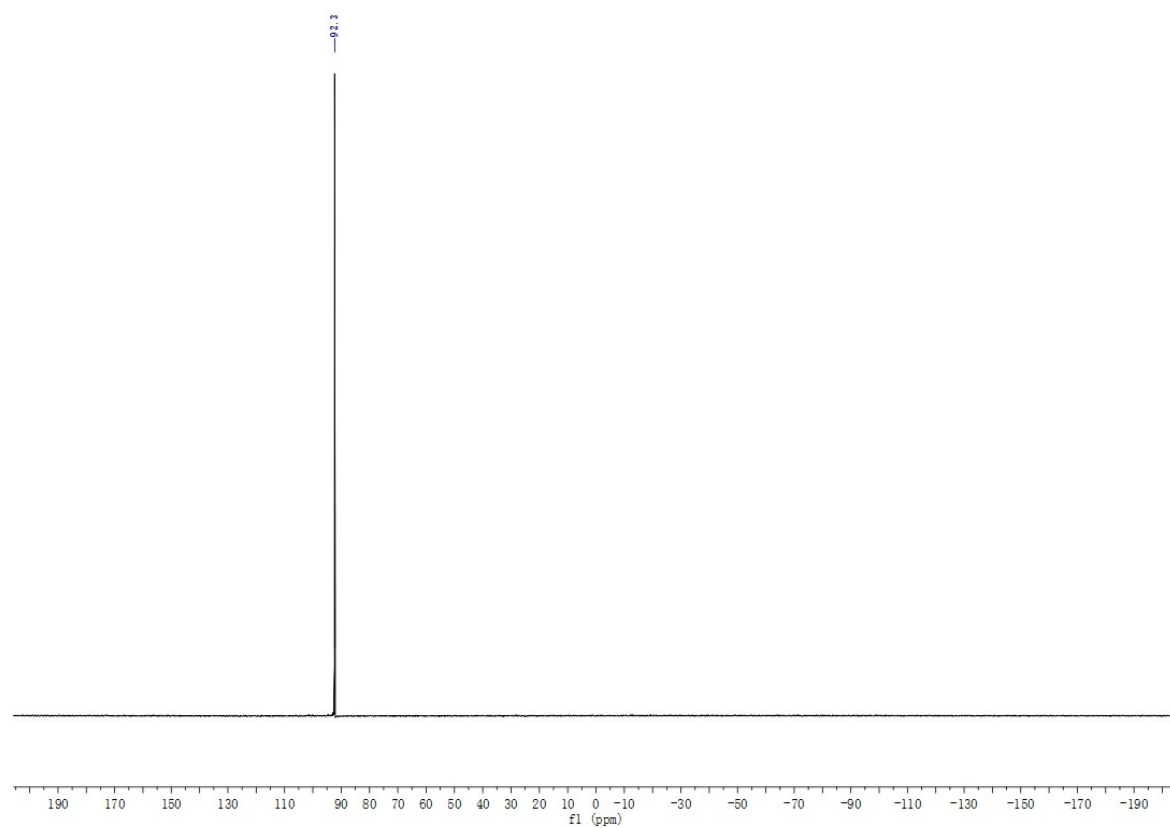
^1H NMR (600 MHz, CDCl_3)



^{13}C NMR (151 MHz, CDCl_3)



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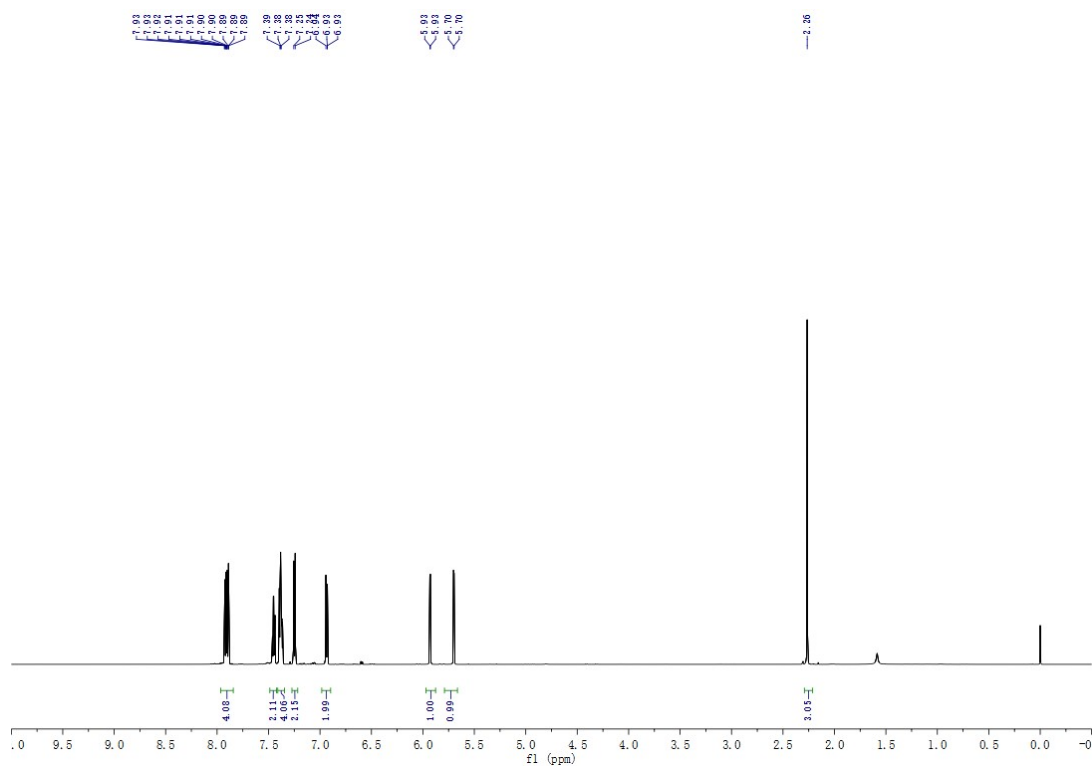




N	Nc
0	7.92
10	7.92
20	7.91
30	7.91
40	7.91
50	7.90
60	7.90
70	7.89
80	7.89
90	7.88
100	7.88



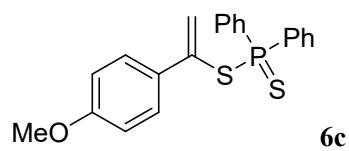
The figure displays a ^1H NMR spectrum. The horizontal axis represents the chemical shift in ppm, with major tick marks every 10 units from 170 down to -140. A single, very sharp and intense peak is present at a chemical shift of 64.9 ppm, as indicated by the label above the peak. The baseline is flat and stable across the entire range from 170 to -140 ppm.



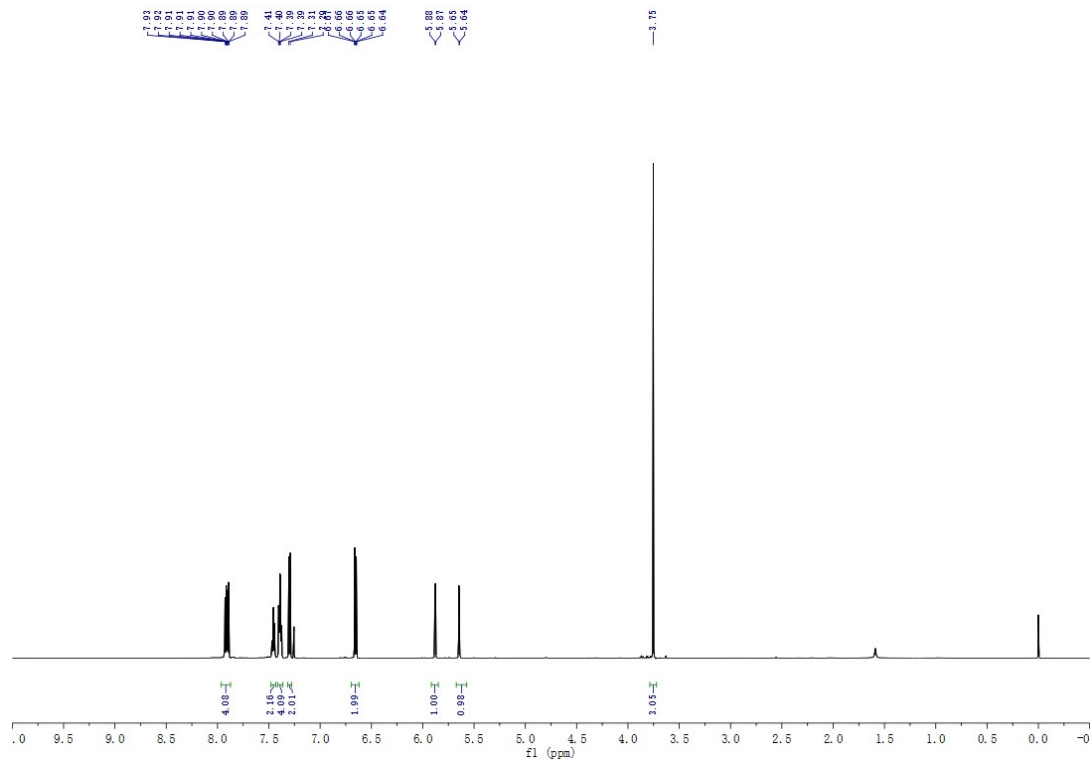
138.2
136.7
136.5
136.5
134.1
133.9
131.9
131.8
130.8
130.8
130.4
129.4
127.1
126.0
21.2

f1 (ppm)

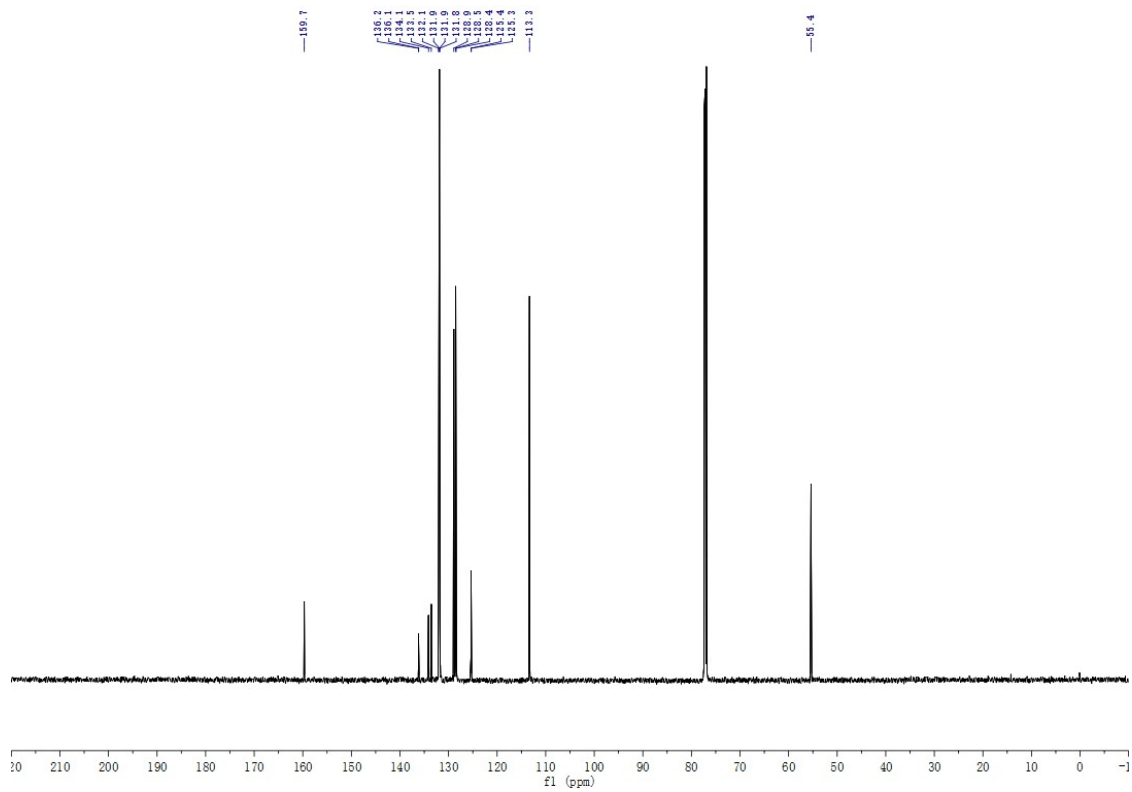
The image shows a ^1H NMR spectrum. The horizontal axis represents the chemical shift in ppm, ranging from 0 to 17. A single, sharp, vertical peak is present at approximately 3.7 ppm. Above this peak, the number '1.00' is printed, indicating the integration value. The baseline is flat and stable across the entire range.



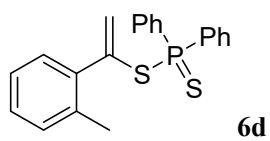
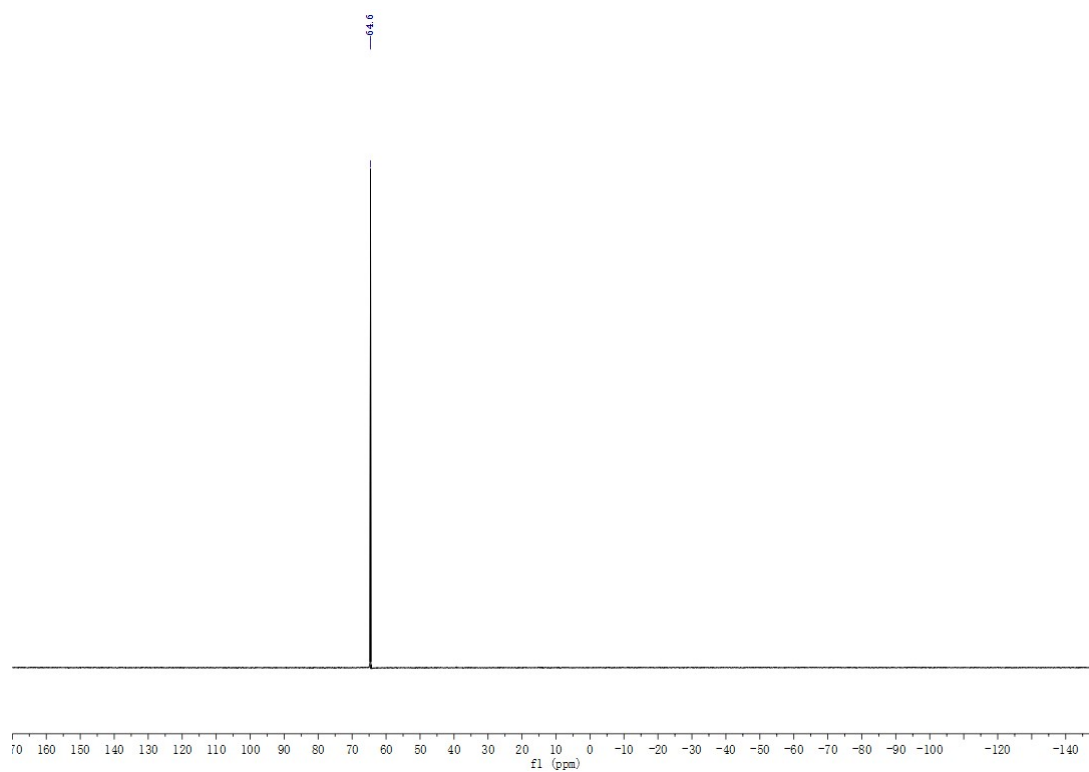
^1H NMR (600 MHz, CDCl_3)



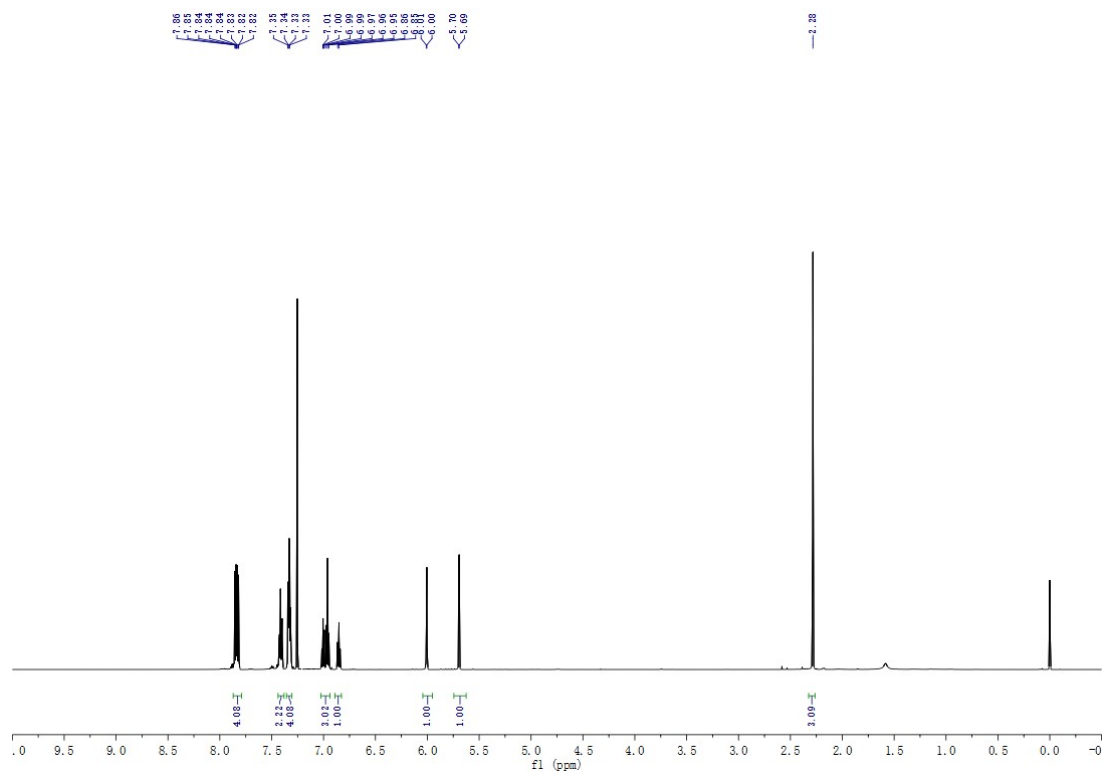
^{13}C NMR (151 MHz, CDCl_3)



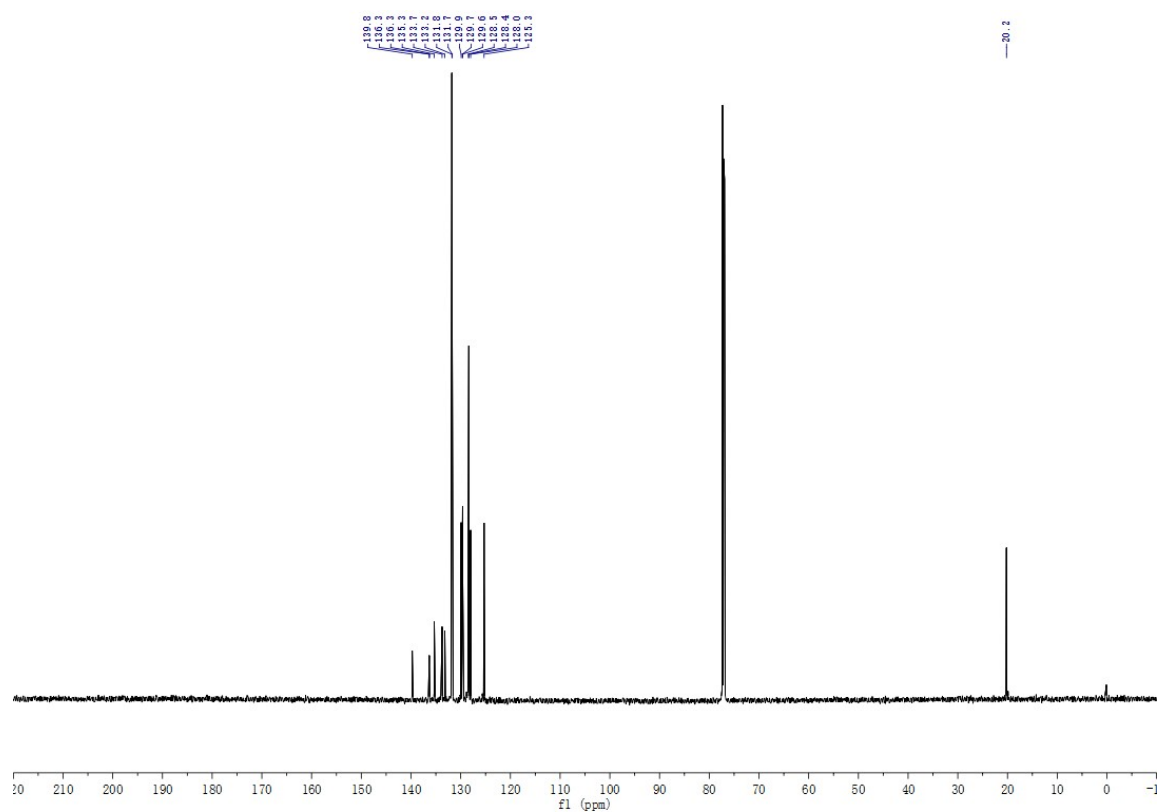
^{31}P NMR (243 MHz, CDCl_3)



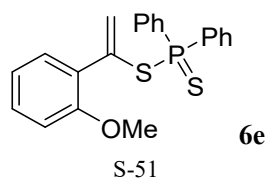
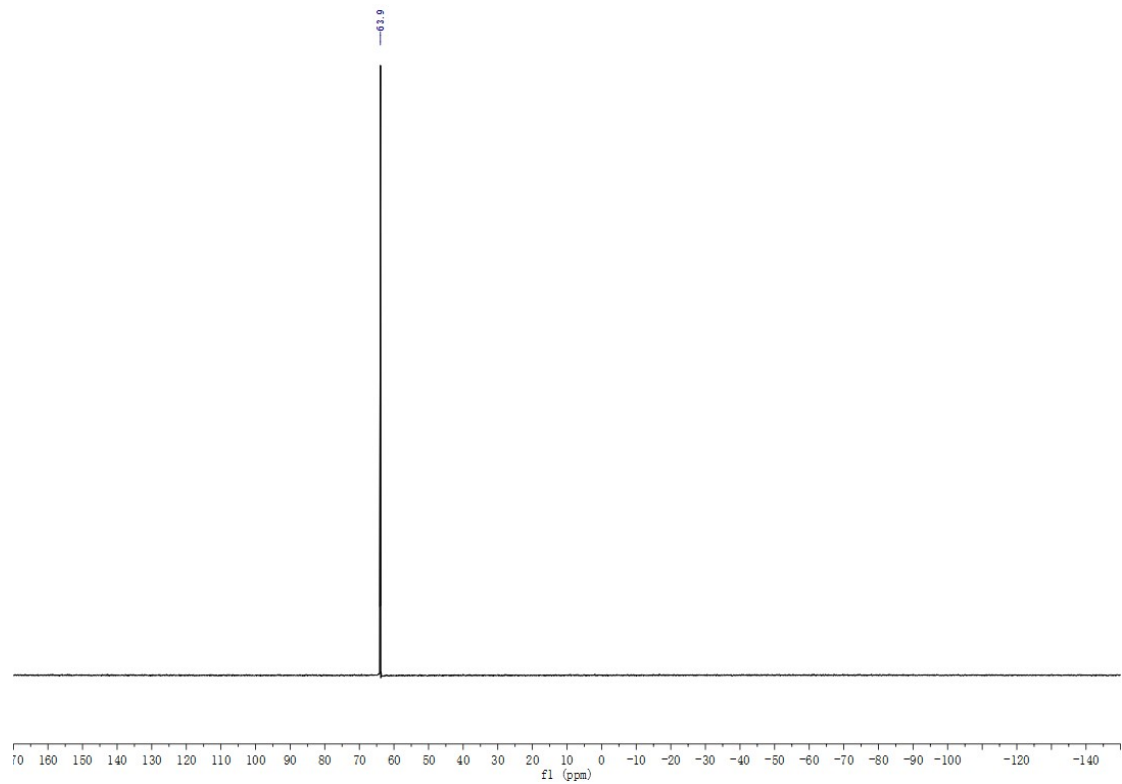
^1H NMR (600 MHz, CDCl_3)

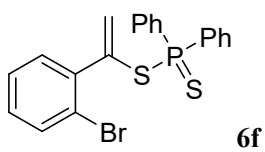
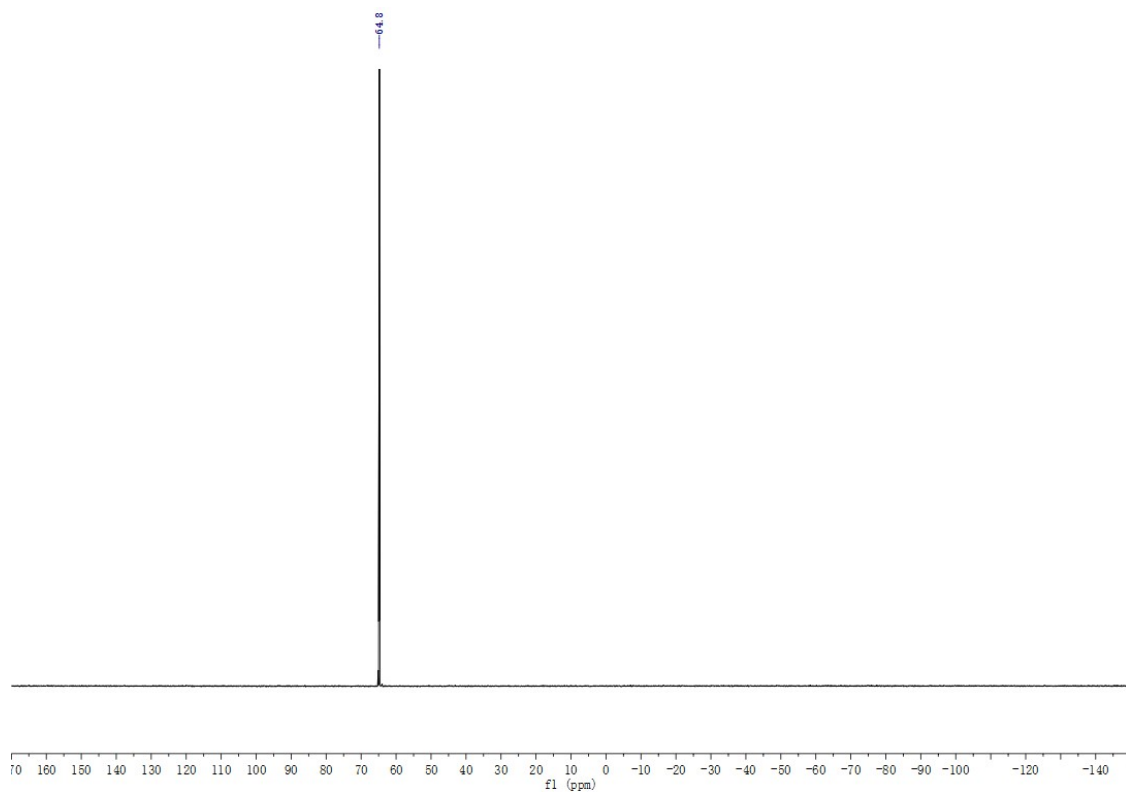


^{13}C NMR (151 MHz, CDCl_3)

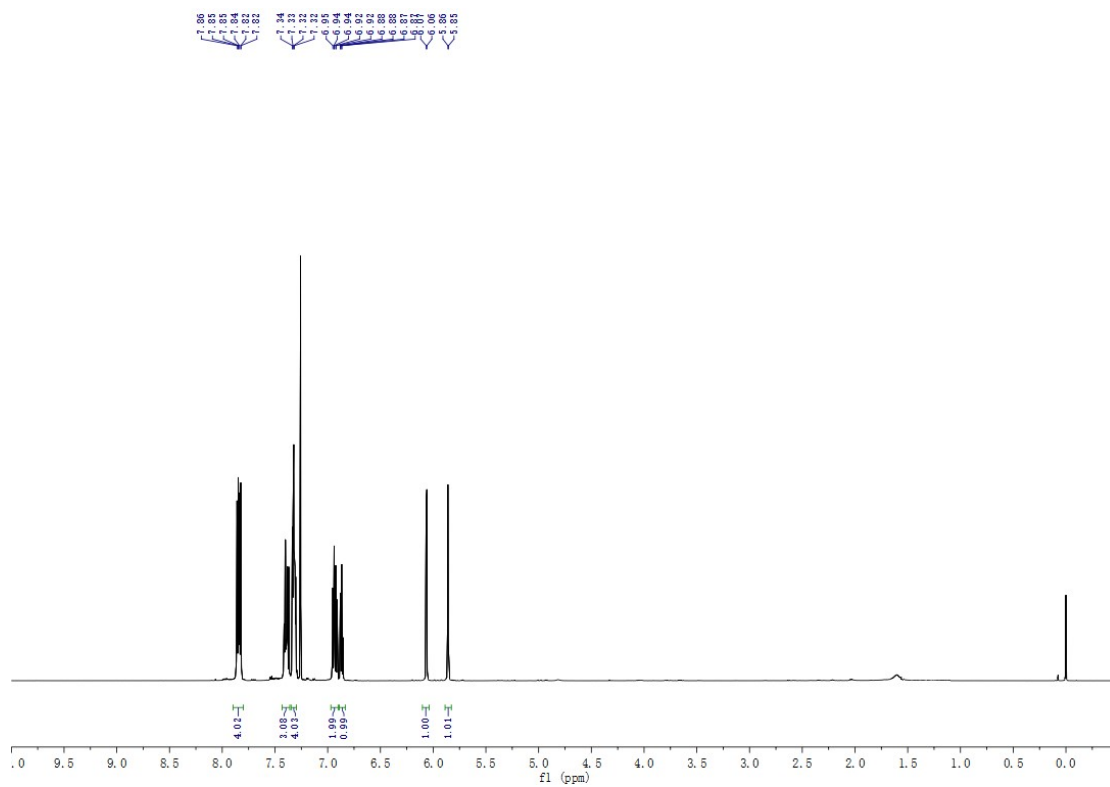


^{31}P NMR (243 MHz, CDCl_3)

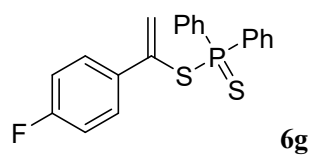
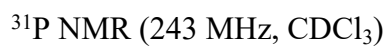




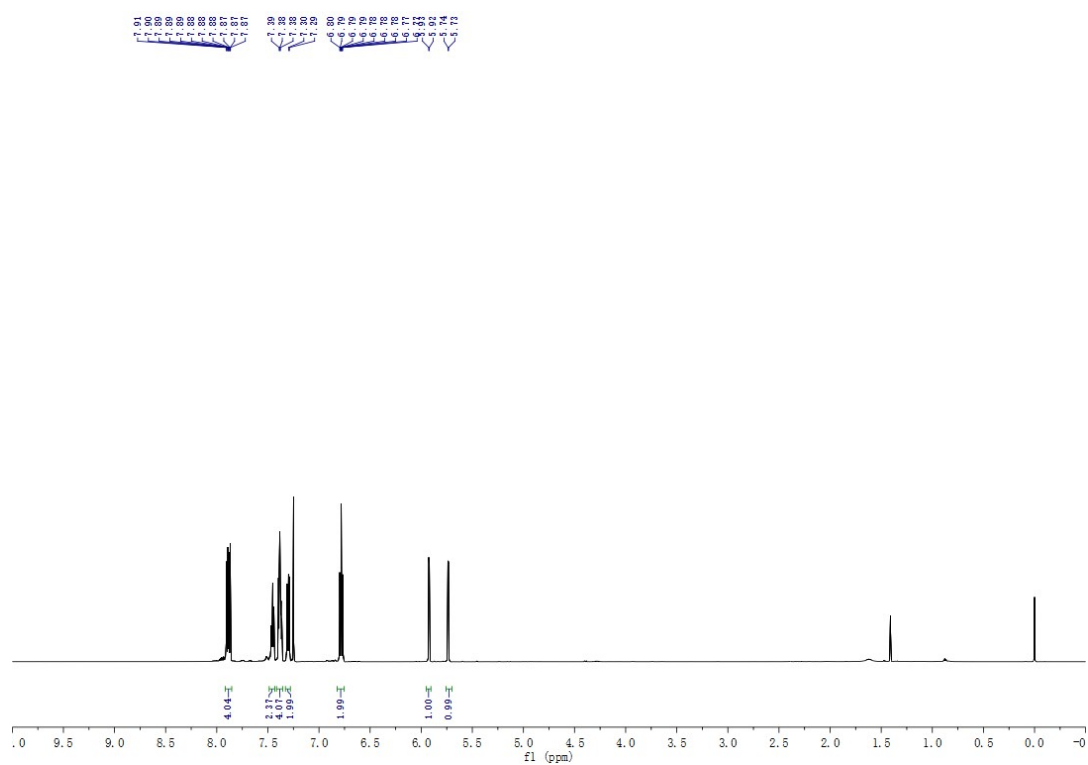
¹H NMR (600 MHz, CDCl₃)



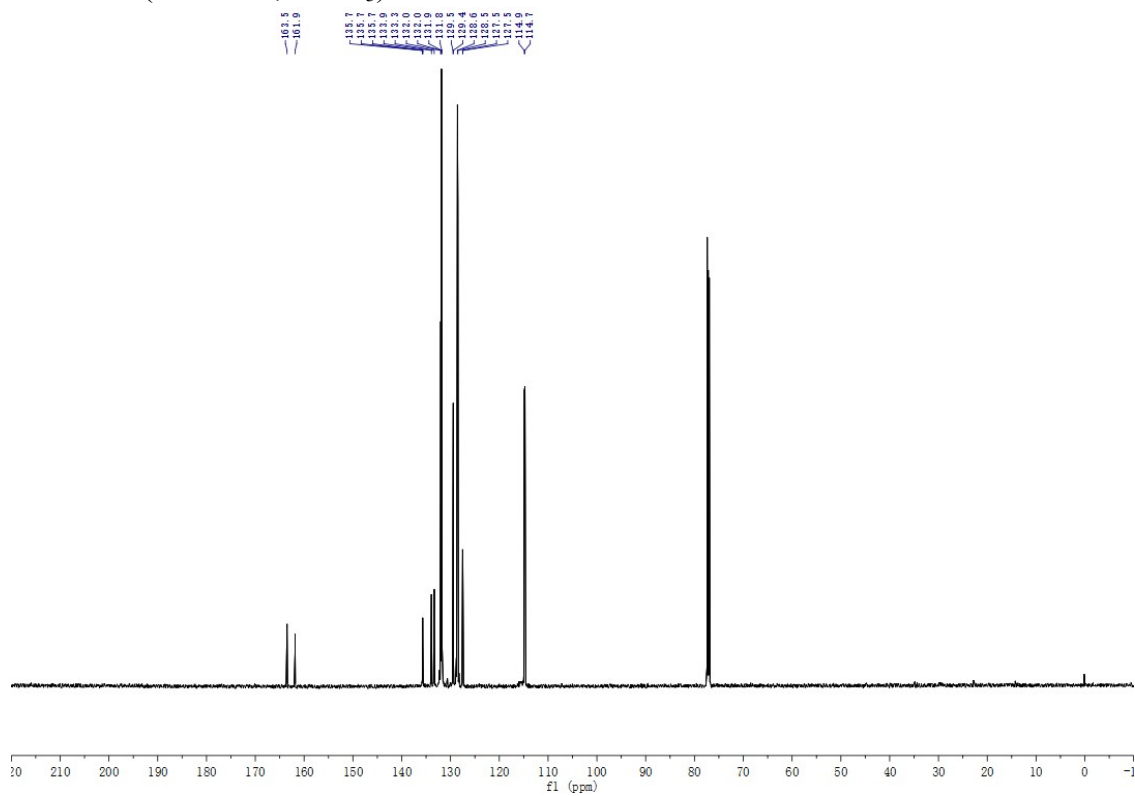
¹³C NMR (151 MHz, CDCl₃)



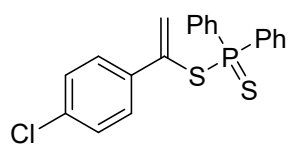
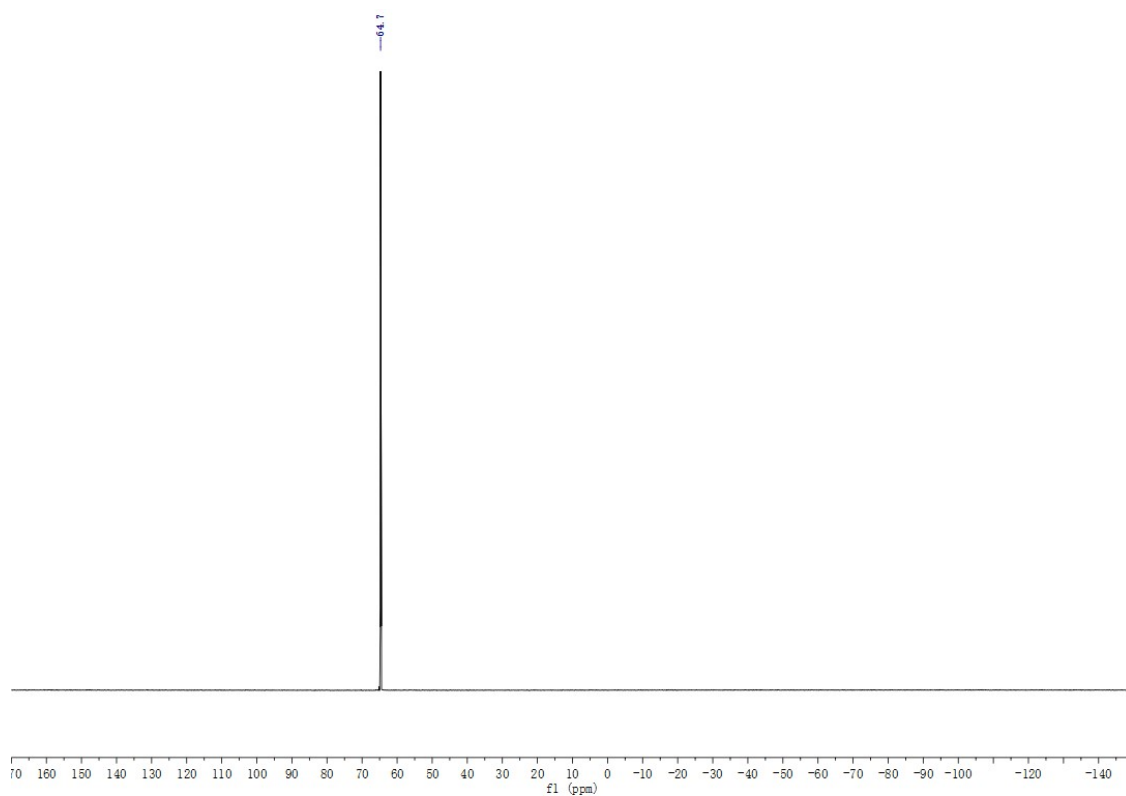
^1H NMR (600 MHz, CDCl_3)



^{13}C NMR (151 MHz, CDCl_3)

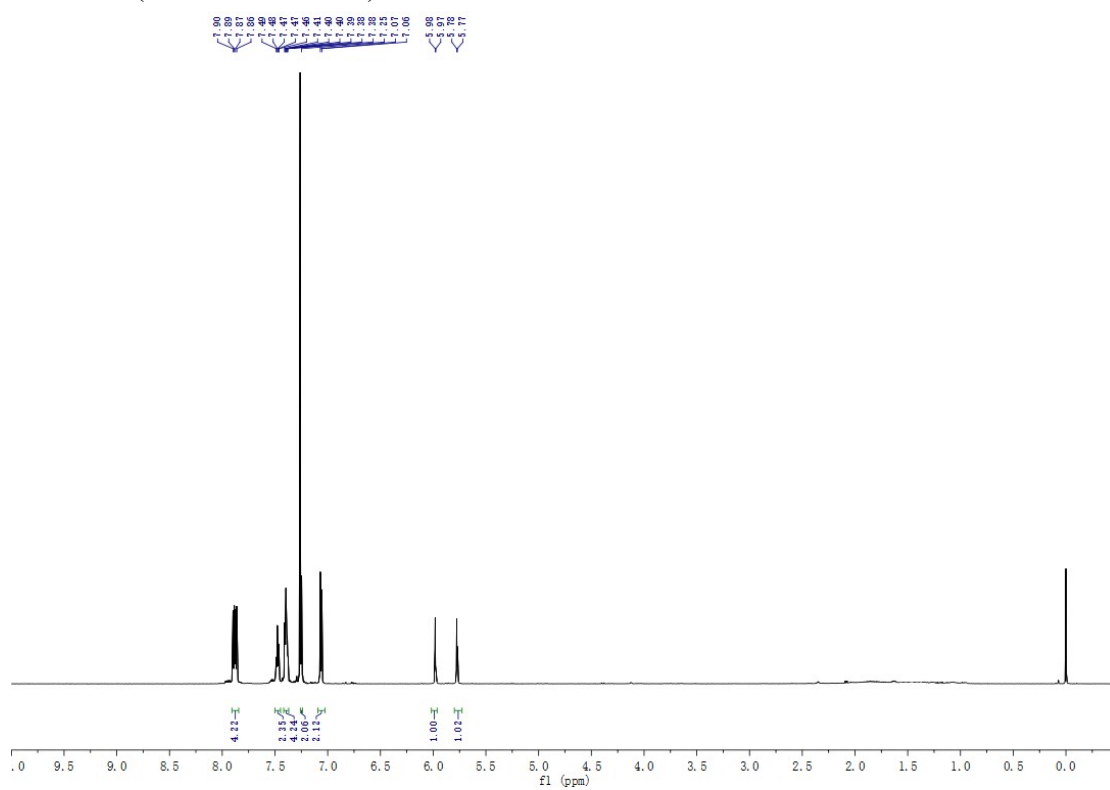


^{31}P NMR (243 MHz, CDCl_3)

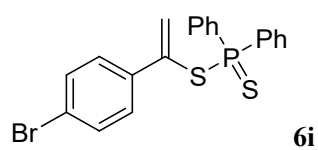
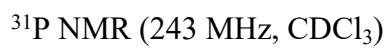


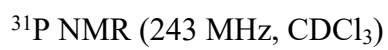
6h

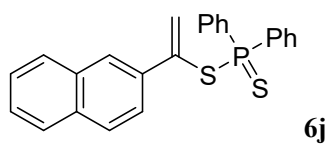
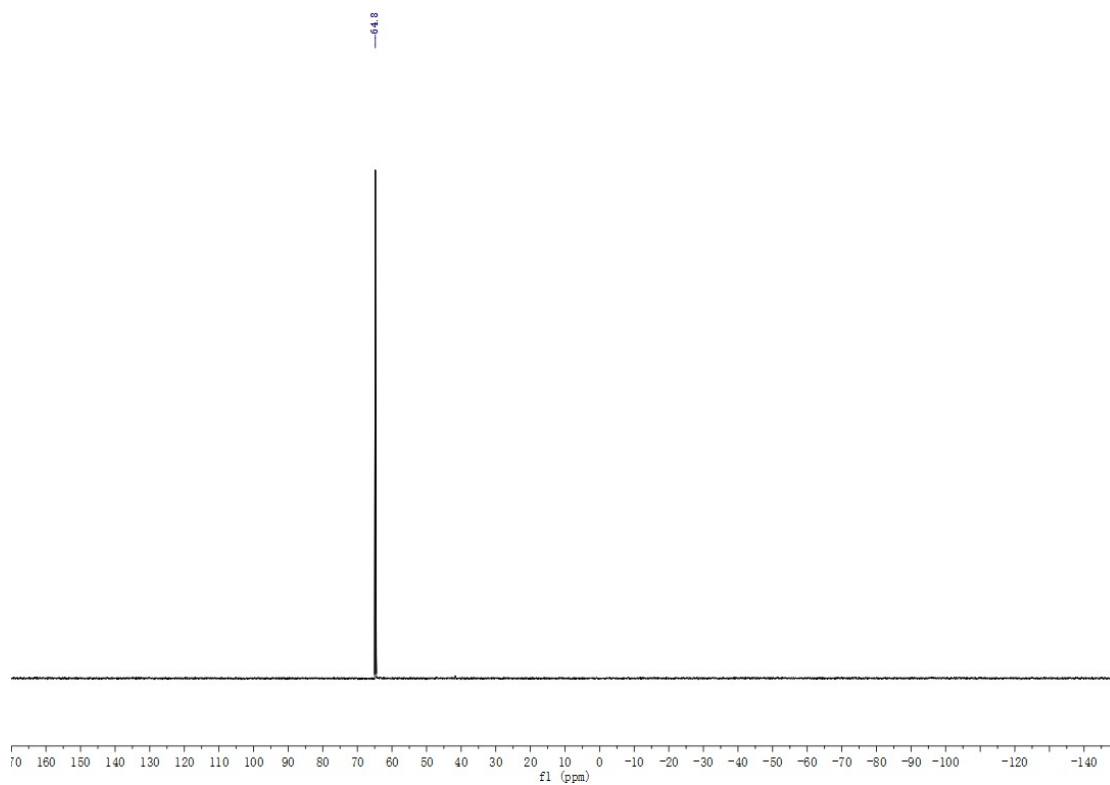
¹H NMR (600 MHz, CDCl₃)



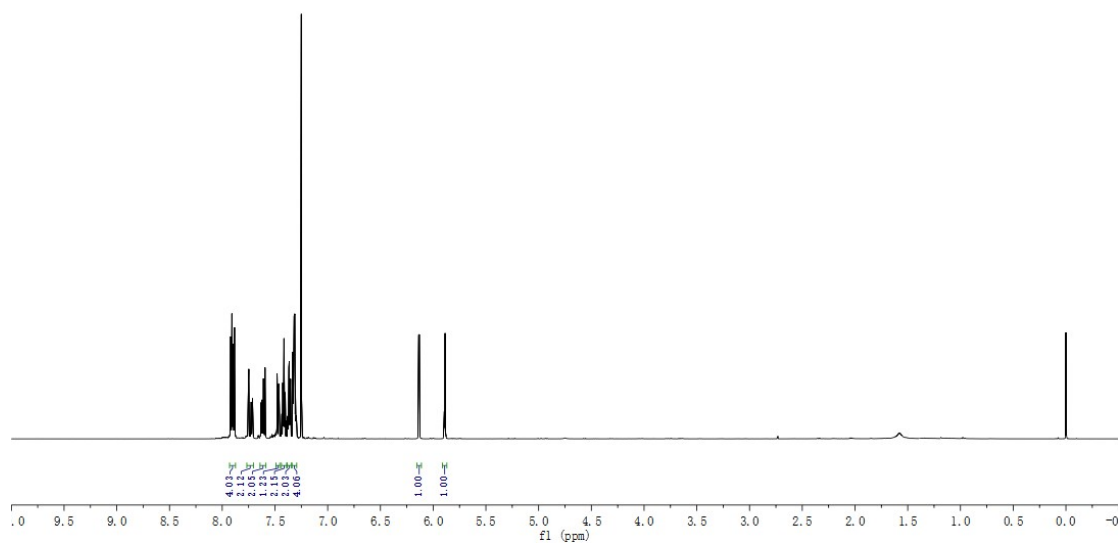
¹³C NMR (151 MHz, CDCl₃)



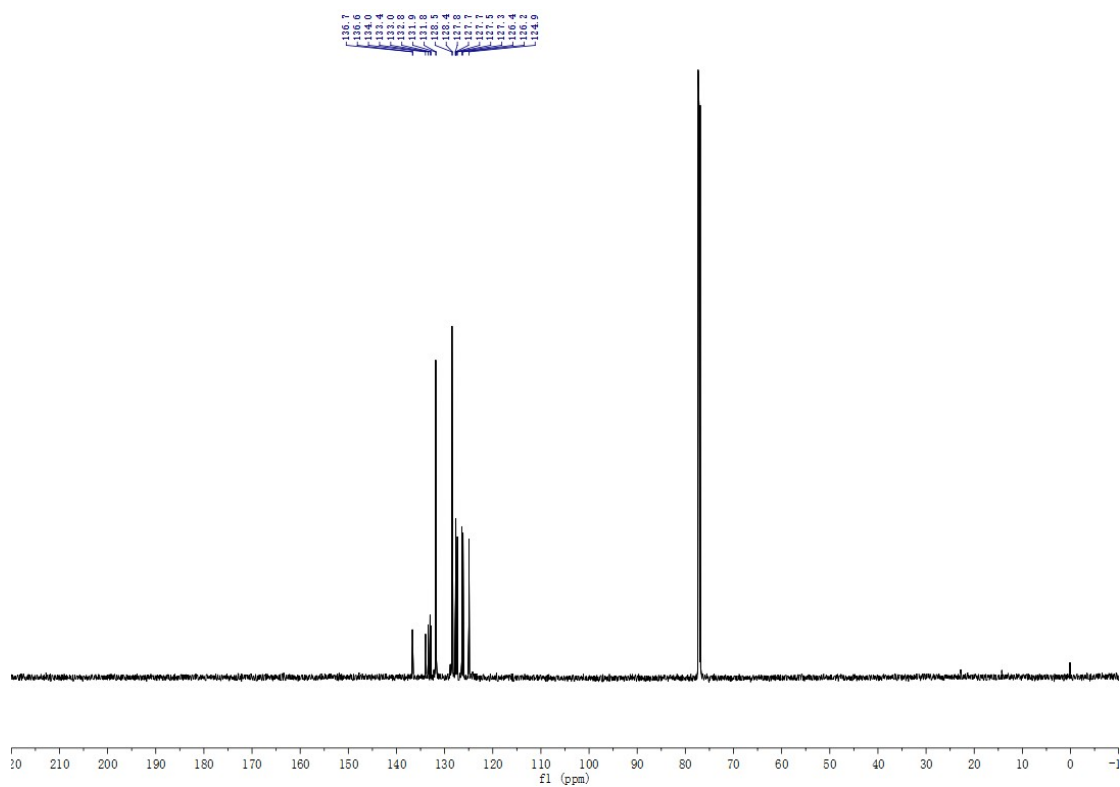
[illegible]



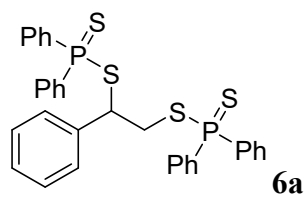
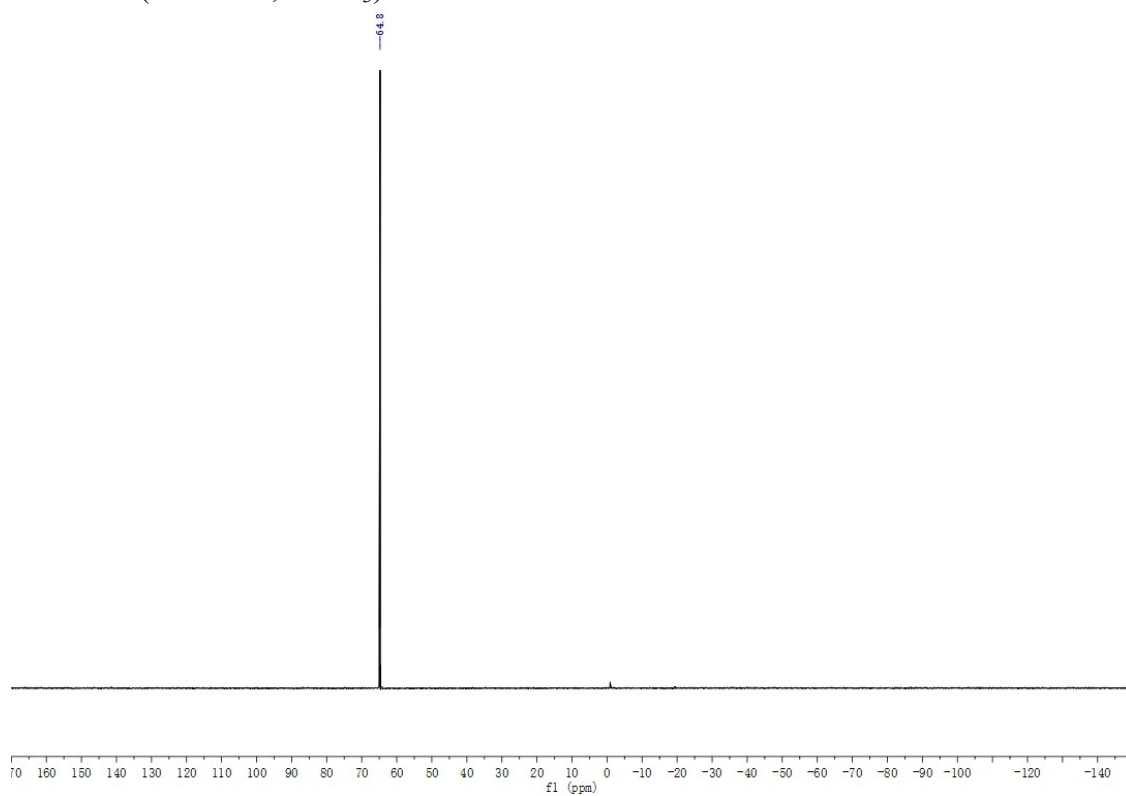
¹H NMR (600 MHz, CDCl₃)



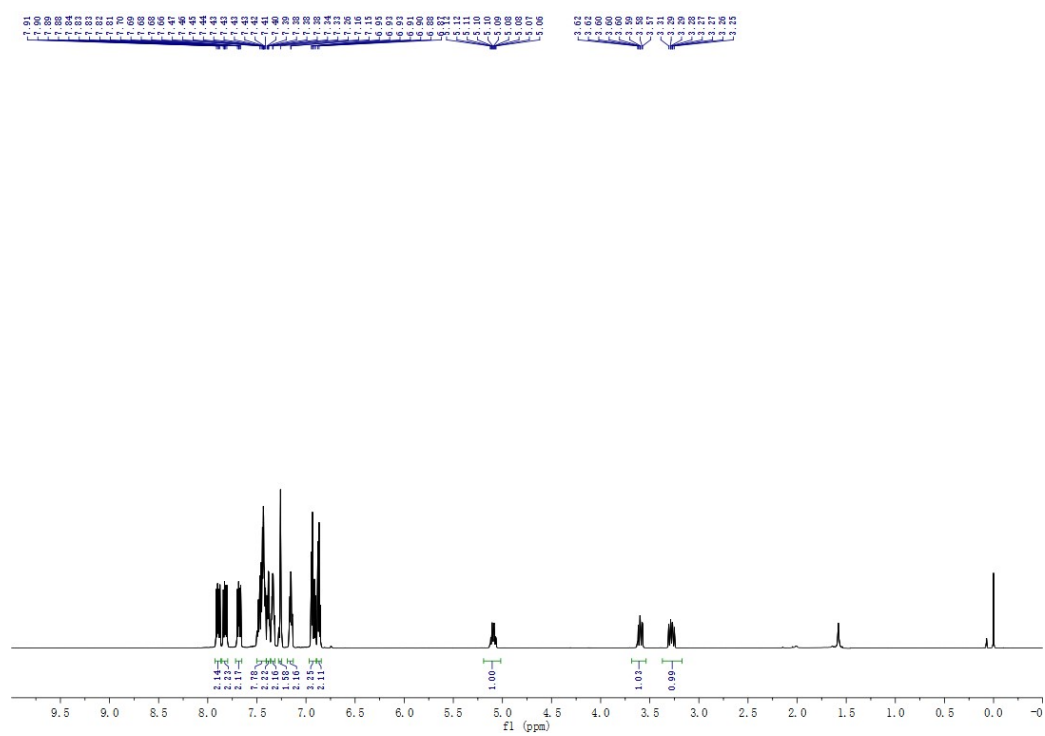
¹³C NMR (151 MHz, CDCl₃)



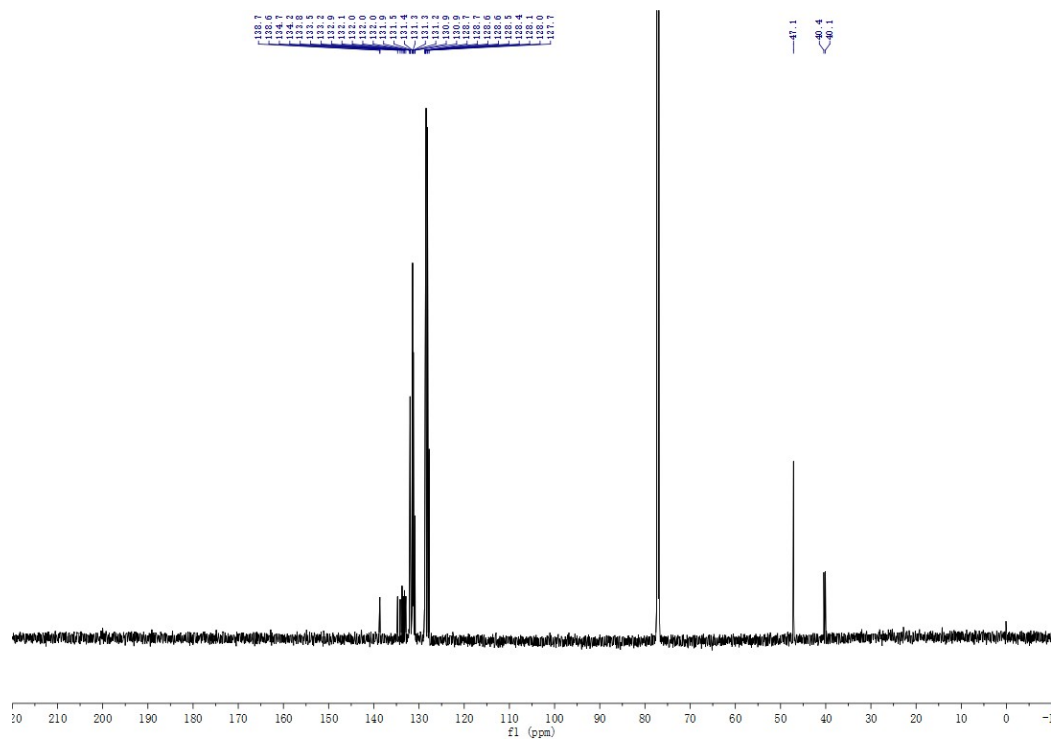
^{31}P NMR (243 MHz, CDCl_3)



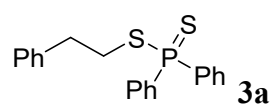
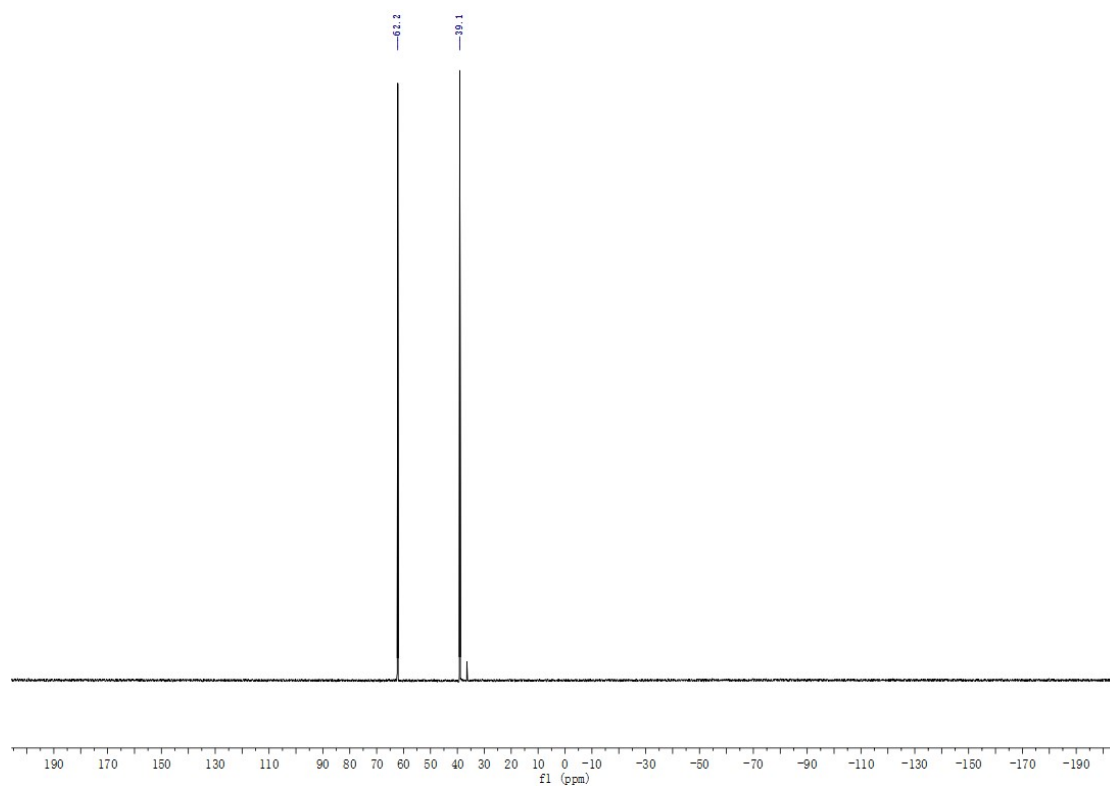
^1H NMR (600 MHz, CDCl_3)



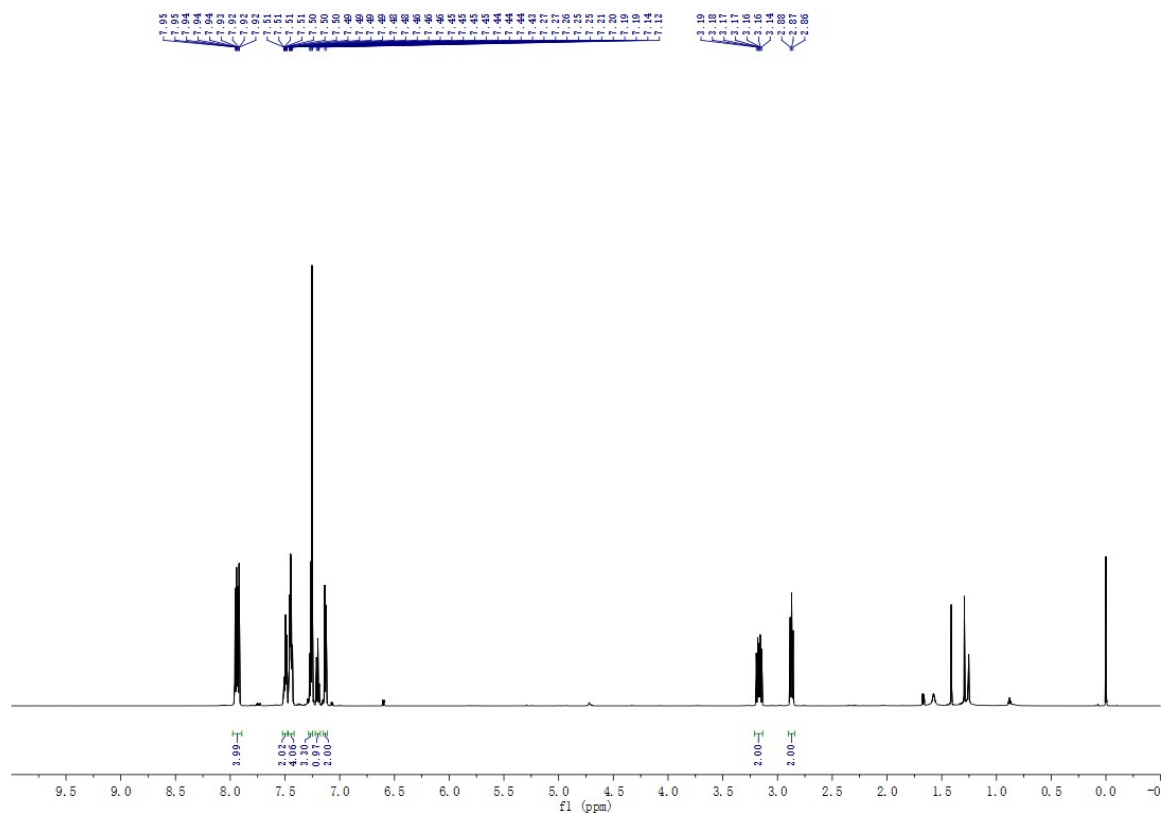
^{13}C NMR (151 MHz, CDCl_3)



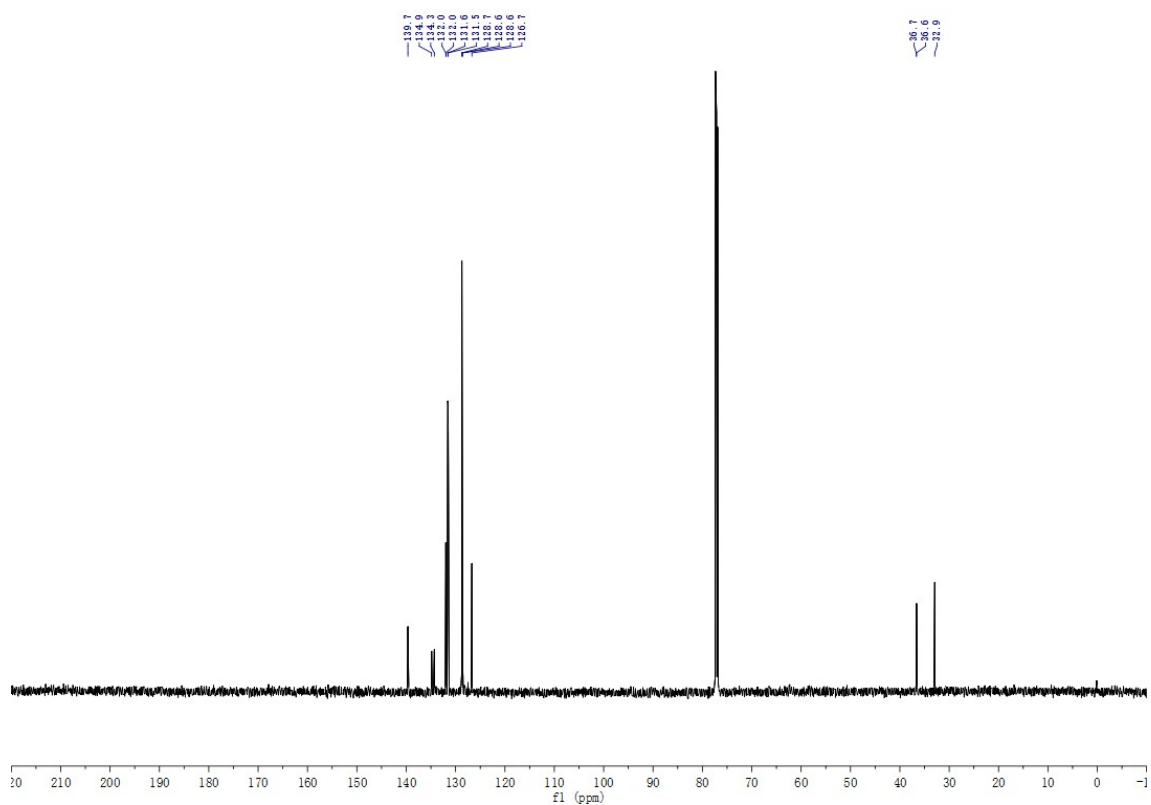
^{31}P NMR (243 MHz, CDCl_3) δ



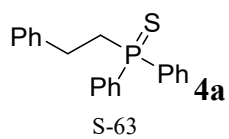
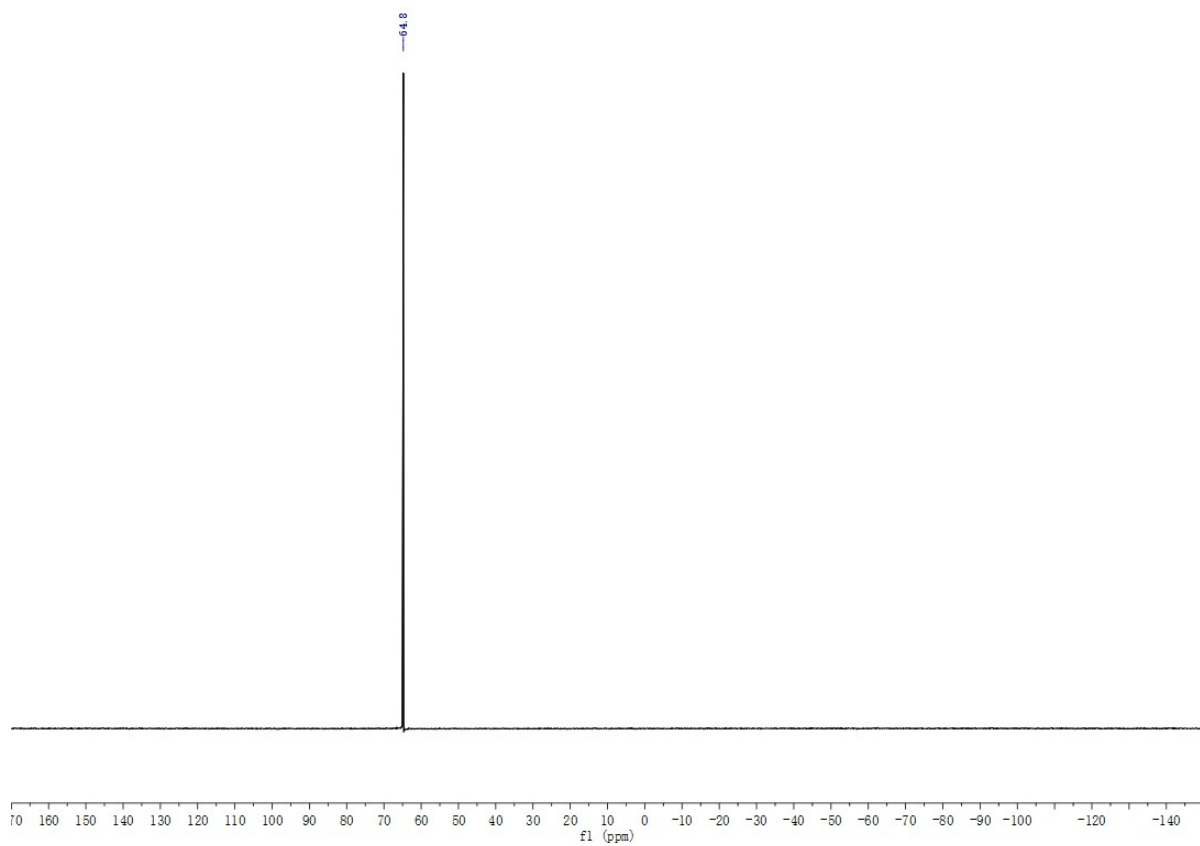
¹H NMR (600 MHz, CDCl₃)



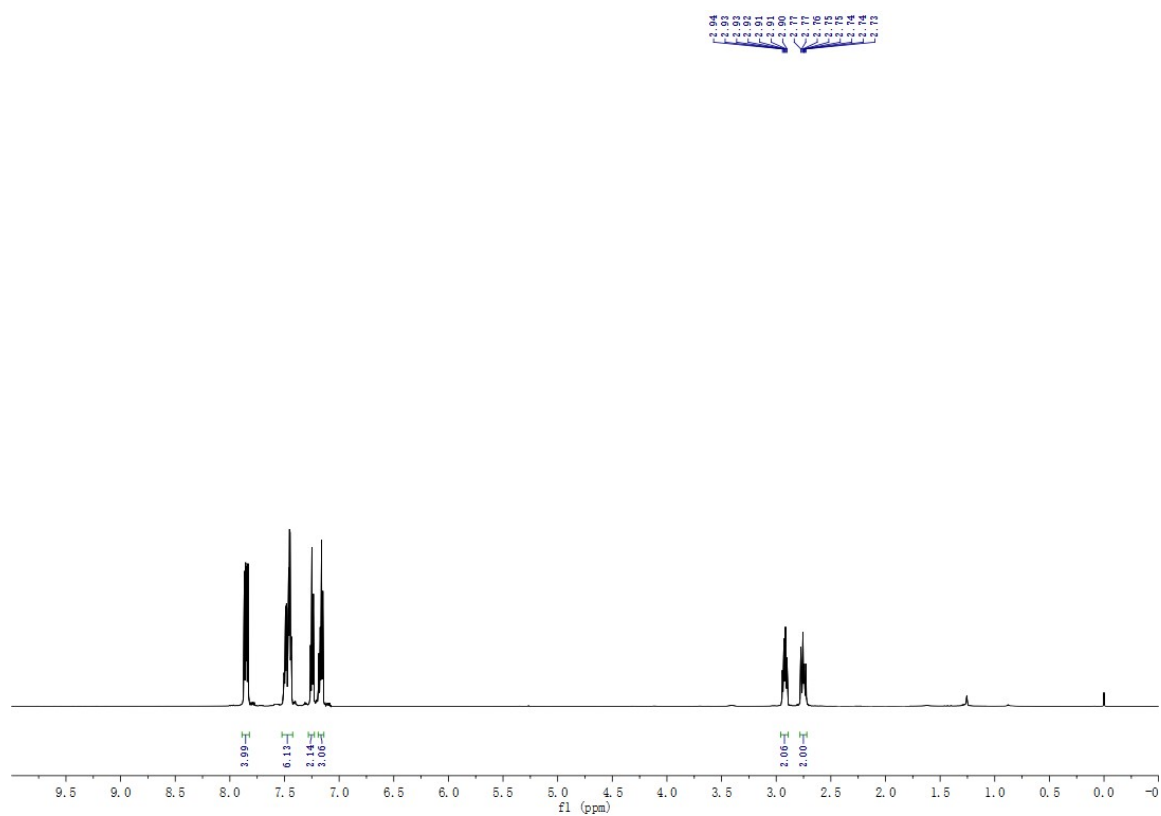
¹³C NMR (151 MHz, CDCl₃)



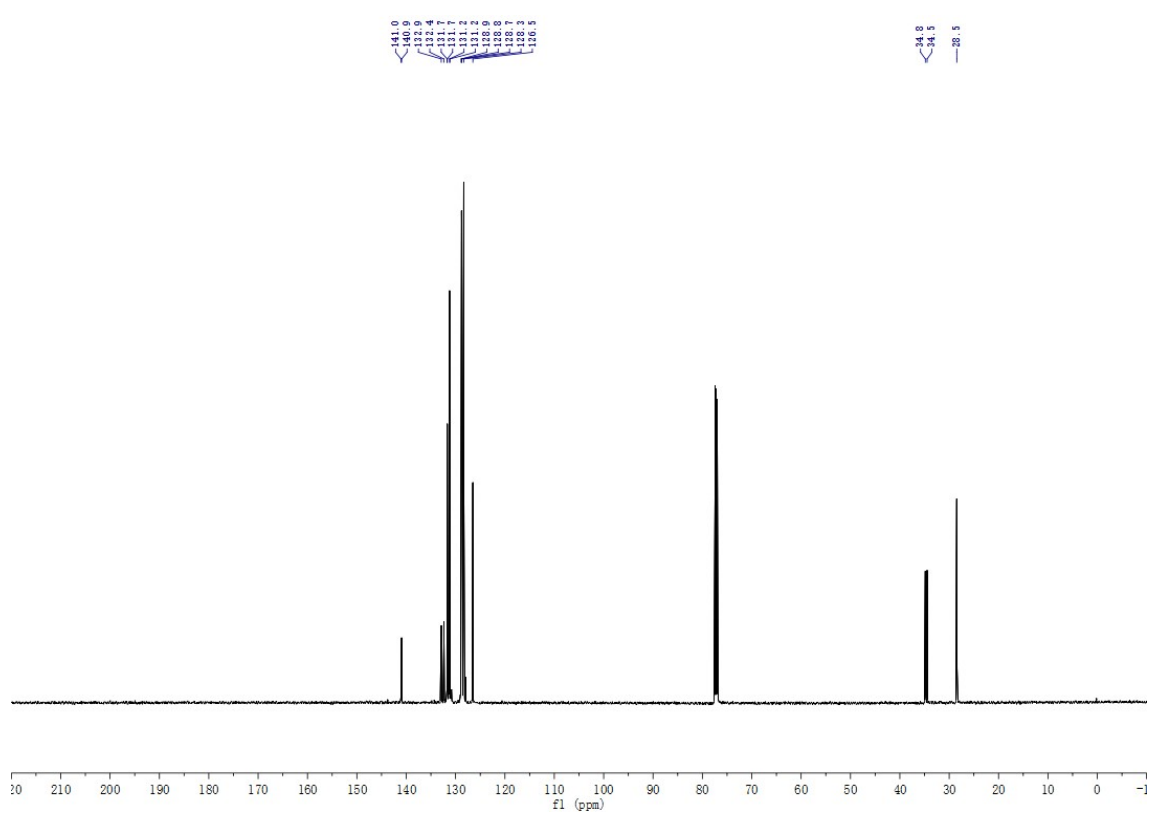
³¹P NMR (243 MHz, CDCl₃)



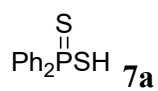
^1H NMR (600 MHz, CDCl_3)



^{13}C NMR (151 MHz, CDCl_3)

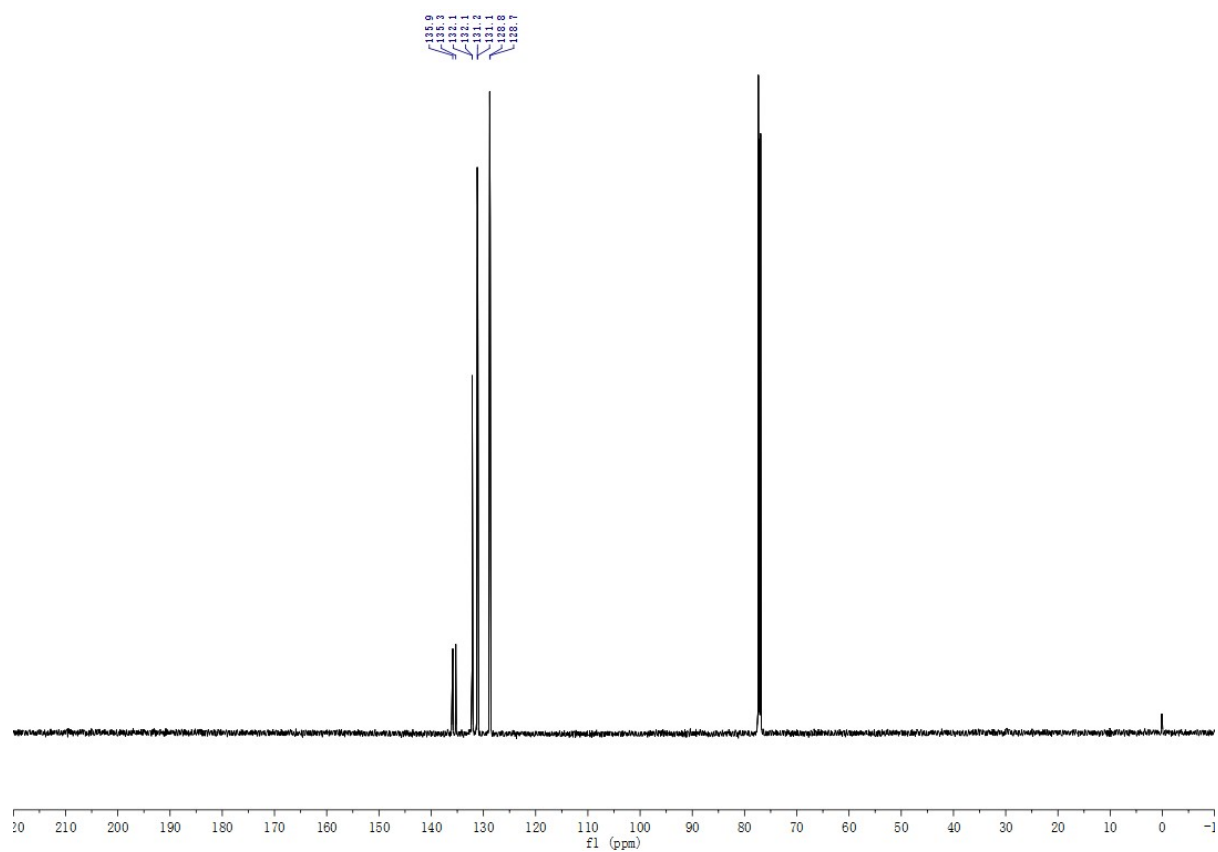


^{31}P NMR (243 MHz, CDCl_3)

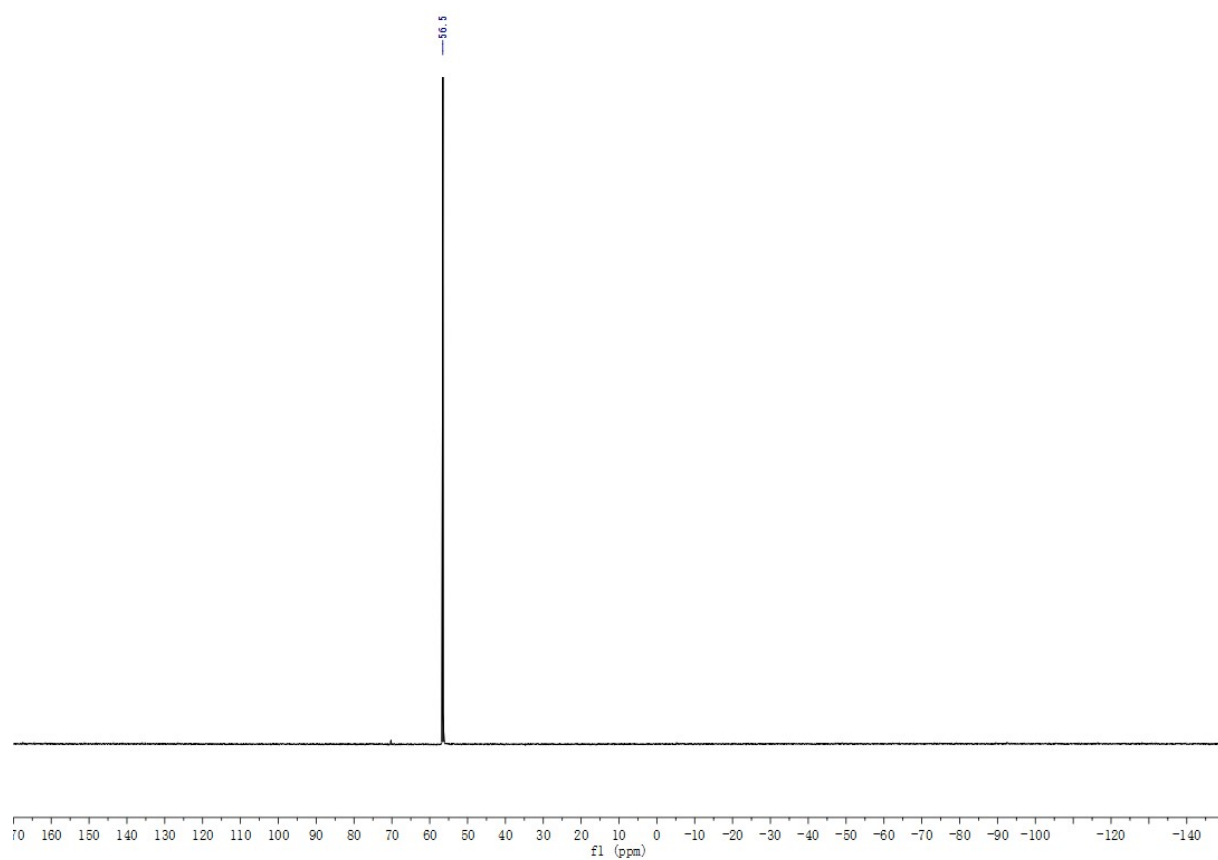


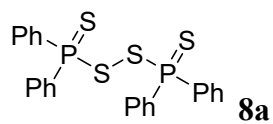
7.98
7.97
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7.46



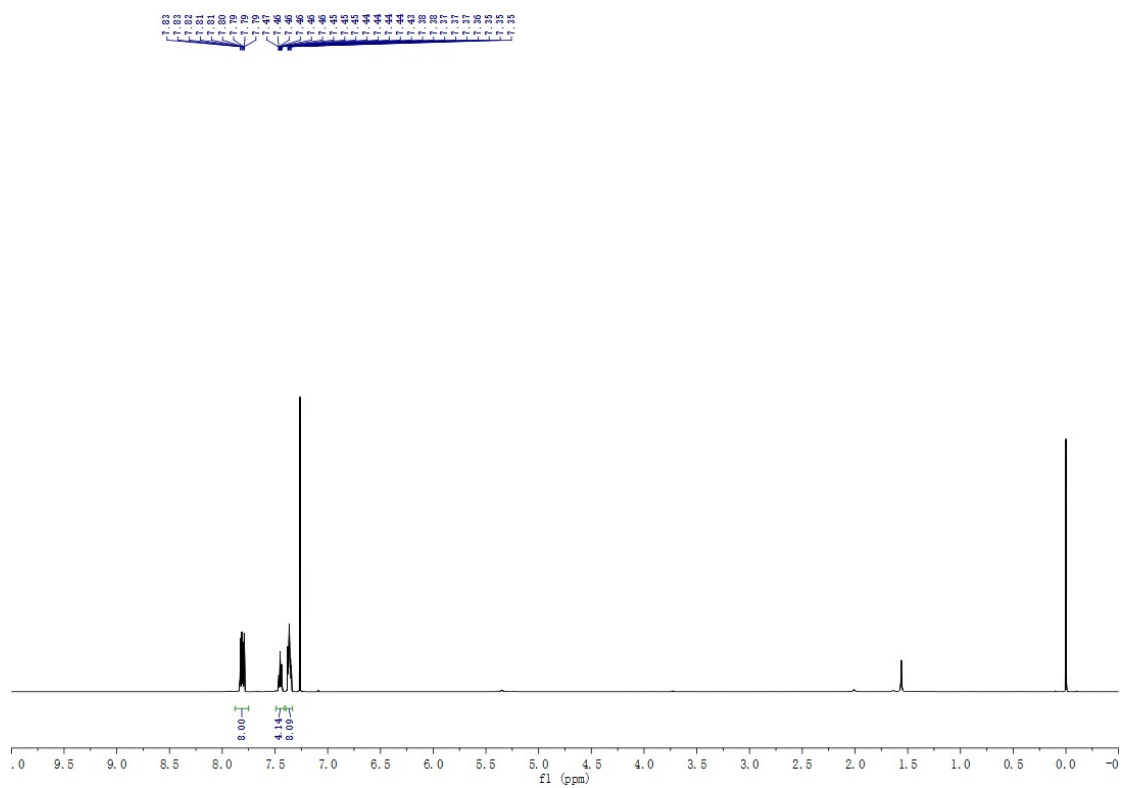


^{31}P NMR (243 MHz, CDCl_3)

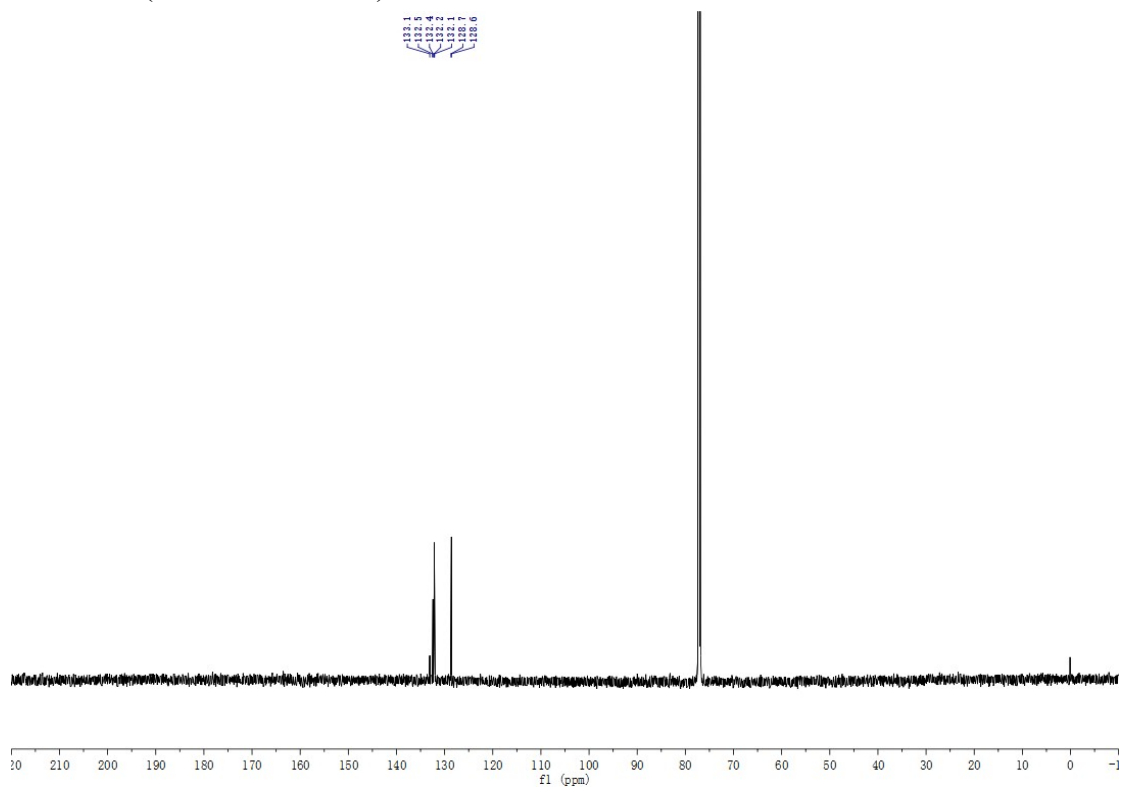




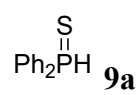
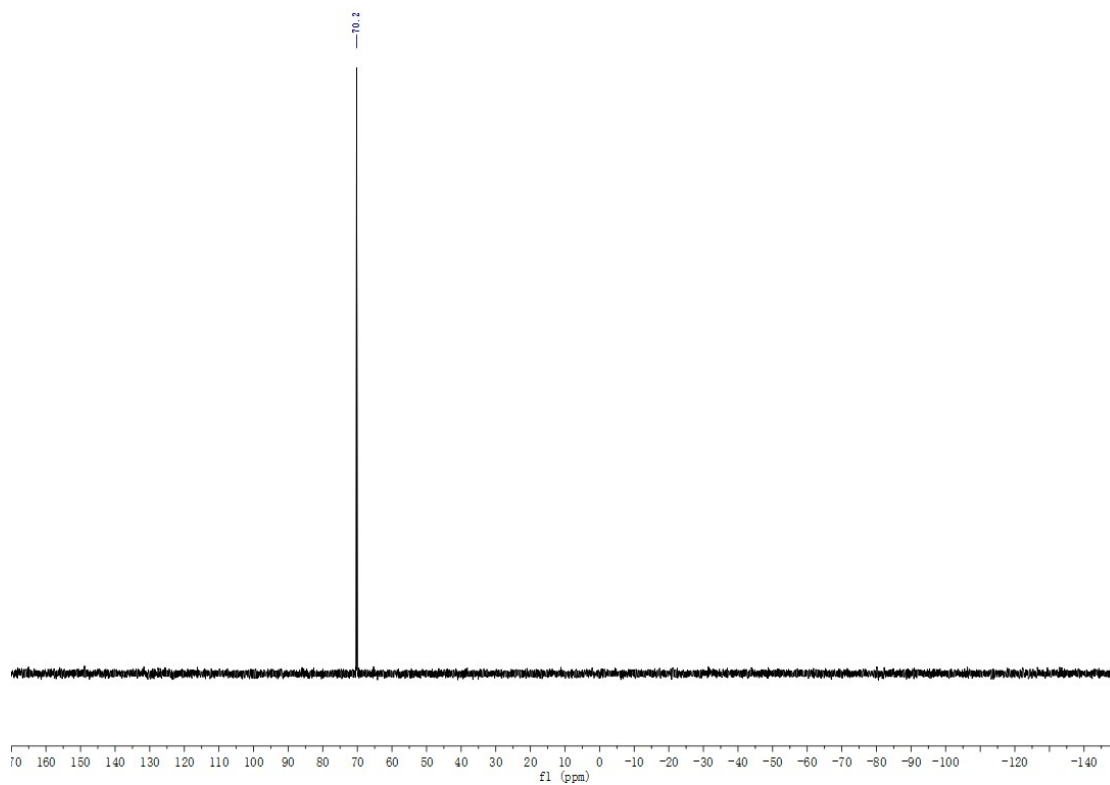
^1H NMR (600 MHz, CDCl_3)



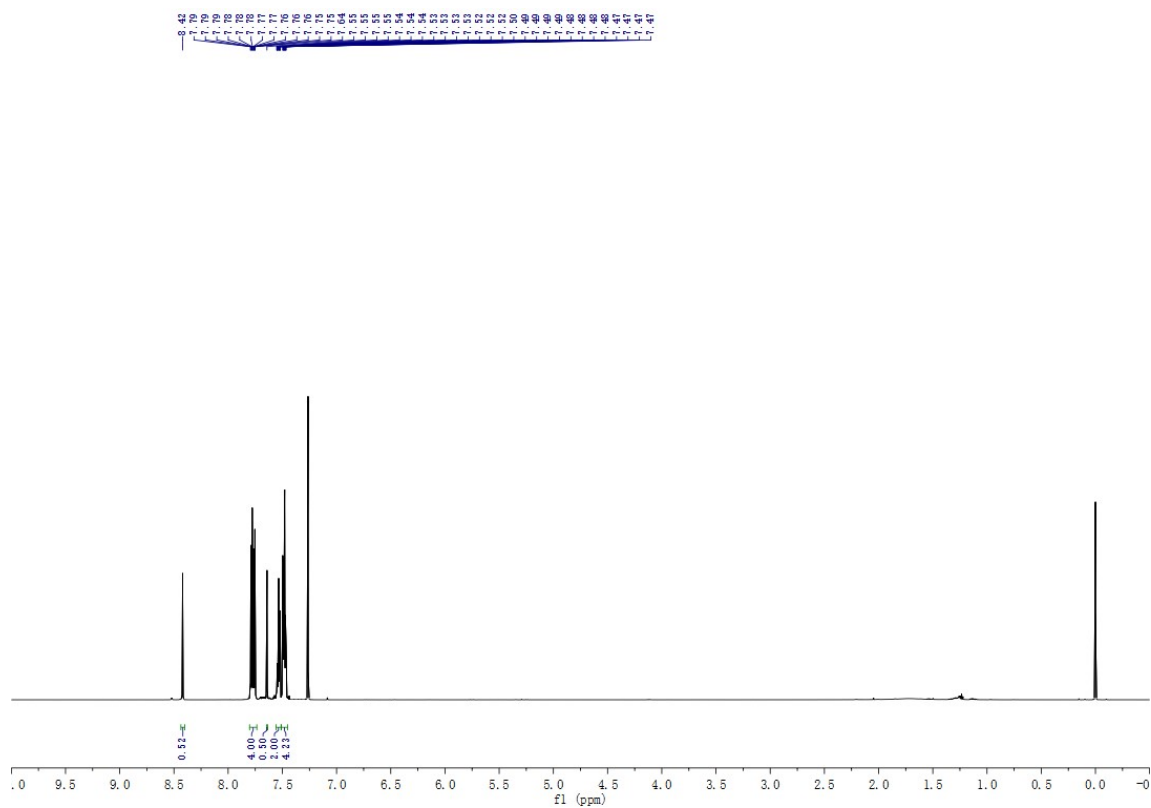
^{13}C NMR (151 MHz, CDCl_3)



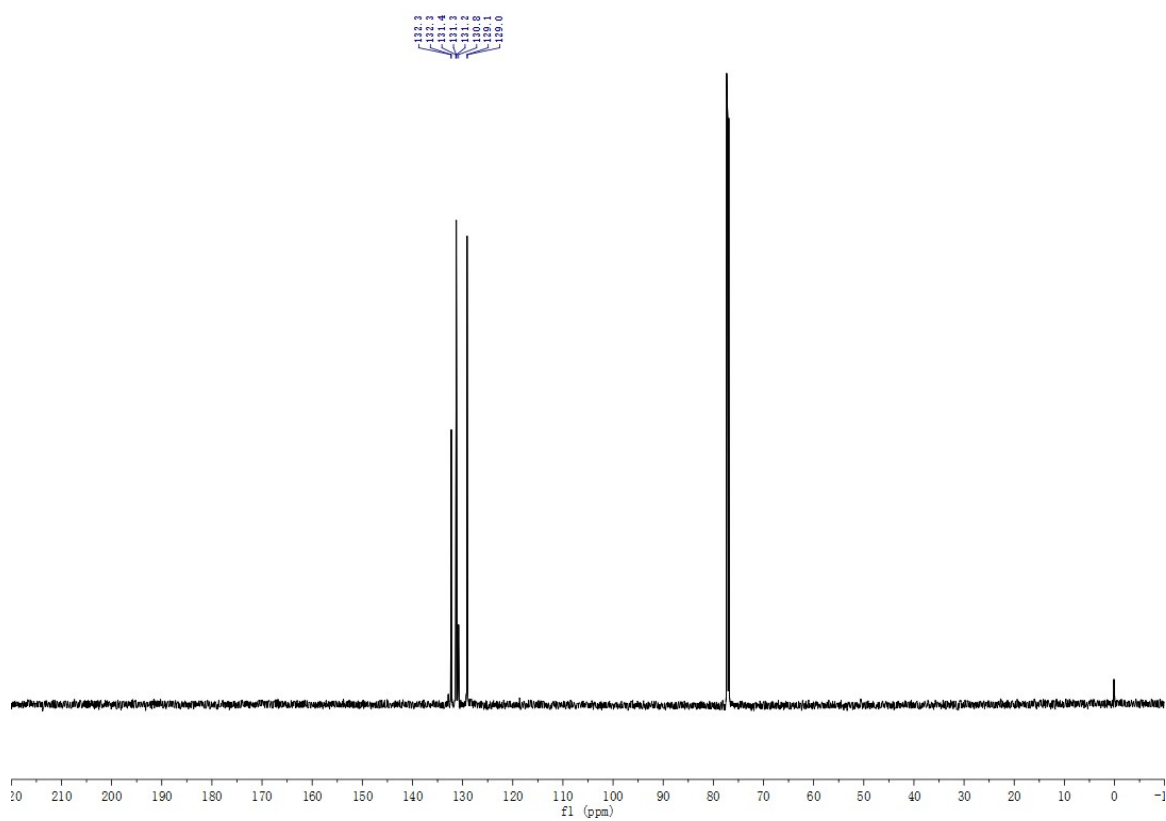
^{31}P NMR (243 MHz, CDCl_3) δ 70.2.



^1H NMR (600 MHz, CDCl_3)



^{13}C NMR (151 MHz, CDCl_3)



^{31}P NMR (243 MHz, CDCl_3)

