Electronic Supplementary Information

For

Additive-free, *N*-chlorosuccinimide-promoted electrophilic phosphorothiolation/cyclization of alkynes with P(O)SH compounds to access heterocyclic phosphorothiolated molecules

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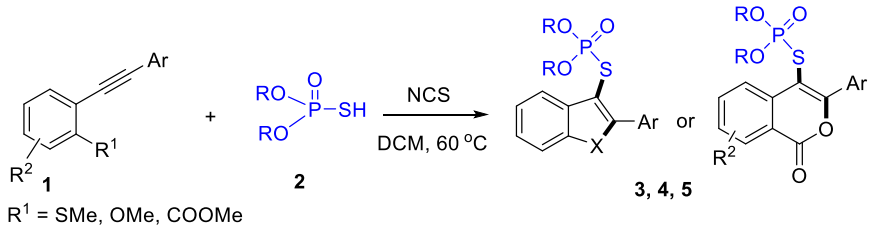
1. General

Substrates **1** was prepared according to the known references.[1,2] Other commercially available compounds were purchased from Aladdin, Aldrich, Energy chemical or Alfa Aesar and used without further purification, unless stated otherwise. ^{1}H NMR, ^{13}C NMR, ^{31}P NMR and ^{19}F NMR spectra were measured on Bruker AVIII 400M spectrometers with $CDCl_3$ as solvent and tetramethylsilane (TMS) as internal standard. Chemical shifts were reported in units (ppm) by assigning TMS resonance in the ^{1}H spectrum as 0.00 ppm and $CDCl_3$ resonance in the ^{13}C spectrum as 77.23 ppm. All coupling constants (*J* values) were reported in Hertz (Hz). Chemical shifts of common trace ^{1}H NMR impurities (ppm): H_2O: 1.56, $CHCl_3$: 7.26. HRMS analyses were obtained on a Finnigan-LCQDECA mass spectrometer and a Bruker Daltonics Bio-TOF-Q mass spectrometer by the ESI method, respectively.

2. Experimental procedure for *N*-chlorosuccinimide-promoted electrophilic phosphorothiolation/cyclization of alkynes with P(O)SH compounds



NCS (0.4 mmol, 2.0 equiv) was placed in a dry Schlenk-tube and compound **2** (0.4 mmol, 2.0 equiv) in DCM (2.5 mL) was then added and the mixture was stirred at 60 °C for 30 min under air. After that, compound **1** (0.2 mmol) was then added and the reaction mixture was stirred at 60 °C for 2 h. Upon completion, the solution was loaded directly onto a column and purified by flash column chromatography using a petroleum ether/AcOEt as the eluent to give the corresponding products **3**, **4** or **5**.

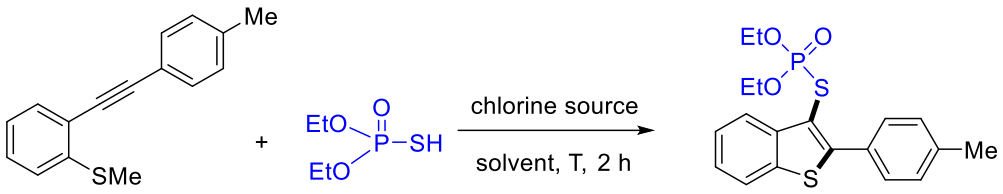
References:

1. T. Yao and R. C. Larock, *J. Org. Chem.*, 2003, **68**, 5936-5942.

2. J. Wang, Y.-J. Deng, X.-X. Yan, Y.-J. Liu, C.-P. Ge, Y. Yan, S. Chao and P.-X. Zhou, *Org. Chem. Front.*, 2020, **7**, 715-722.

3. Optimization of the reaction conditions

Table S1 Optimization of the chlorine sources[a]

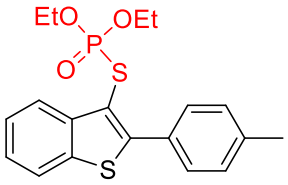


1a **2a** **3a**

Entry	Chlorine Source	T (°C)	Solvent	Yield (%)
1	NCS	60	DCM	96
2	CCl_4	60	DCM	0
3	*N*-chlorophthalimide	60	DCM	90
4	*N*-chlorosaccharin	60	DCM	0

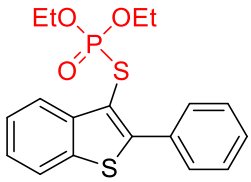
[a]Reaction conditions: Chlorine source (0.4 mmol), **2a** (0.4 mmol) were dissolved in solvent (2.5 mL) and stirred for 30 min; **1a** (0.2 mmol) were then added and the reaction mixture was stirred for 2 h under air atmosphere. Isolated yield.

O,O-diethyl *S*-(2-(*p*-tolyl)benzo[*b*]thiophen-3-yl) phosphorothioate (**3a**)



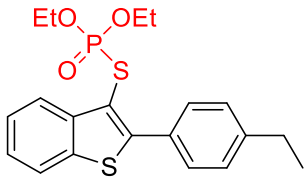
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 75.3 mg, 96%; Yellow Solid; m.p. 40.2-42.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.1 (d, *J* = 8.1 Hz, 1H), 7.8 (d, *J* = 7.9 Hz, 1H), 7.7 (d, *J* = 8.2 Hz, 2H), 7.5 – 7.4 (m, 1H), 7.4 – 7.3 (m, 1H), 7.3 – 7.2 (m, 2H), 4.0 – 3.9 (m, 2H), 3.8 – 3.6 (m, 2H), 2.4 (s, 3H), 1.0 (t, *J* = 6.6 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.3 (d, *J* = 8.1 Hz), 141.7 (d, *J* = 1.8 Hz), 139.2, 138.0 (d, *J* = 1.3 Hz), 130.6 (d, *J* = 2.6 Hz), 130.3 (d, *J* = 1.5 Hz), 129.4, 125.3, 125.0, 124.2, 122.2, 111.7 (d, *J* = 9.0 Hz), 64.2 (d, *J* = 6.6 Hz), 21.5, 16.0 (d, *J* = 7.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 21.22. HRMS: $[M+H]^+$ *m/z* calcd for $C_{19}H_{22}O_3PS_2^+$: 393.0742, found: 393.0749.

O,O-diethyl *S*-(2-phenylbenzo[*b*]thiophen-3-yl) phosphorothioate (**3b**)



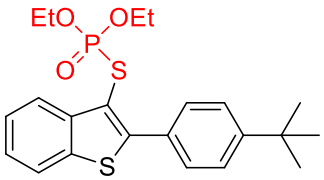
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 62.4 mg, 83%; Yellow Oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, *J* = 8.0 Hz, 1H), 7.87 – 7.74 (m, 3H), 7.53 – 7.36 (m, 5H), 3.97 – 3.82 (m, 2H), 3.77 – 3.61 (m, 2H), 1.03 (t, *J* = 7.2 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.1 (d, *J* = 8.1 Hz), 141.6 (d, *J* = 1.8 Hz), 138.1, 133.5 (d, *J* = 2.5 Hz), 130.4 (d, *J* = 1.5 Hz), 129.1, 128.6, 125.4, 125.1, 124.2, 122.2, 112.1 (d, *J* = 9.0 Hz), 64.2 (d, *J* = 6.6 Hz), 16.0 (d, *J* = 7.4 Hz).^{31}P NMR (162 MHz, $CDCl_3$) δ 21.07. HRMS: $[M+H]^+$ *m/z* calcd for $C_{18}H_{20}O_3PS_2^+$: 379.0586, found: 379.0590.

O,O-diethyl *S*-(2-(4-ethylphenyl)benzo[*b*]thiophen-3-yl) phosphorothioate (**3c**)



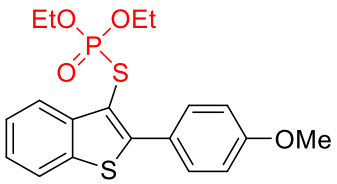
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 76.3 mg, 94%; Yellow Solid; m.p. 40.5-42.7 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (d, *J* = 8.1 Hz, 1H), 7.81 (d, *J* = 8.0 Hz, 1H), 7.73 (d, *J* = 7.8 Hz, 2H), 7.48 (t, *J* = 7.6 Hz, 1H), 7.38 (t, *J* = 7.5 Hz, 1H), 7.32 (d, *J* = 7.8 Hz, 2H), 3.99 – 3.83 (m, 2H), 3.77 – 3.56 (m, 2H), 2.72 (q, *J* = 7.6 Hz, 2H), 1.28 (t, *J* = 7.6 Hz, 3H), 1.03 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.3 (d, *J* = 7.9 Hz), 145.5, 141.7 (d, *J* = 1.8 Hz), 138.0, 130.8 (d, *J* = 2.6 Hz), 130.4 (d, *J* = 1.4 Hz), 128.2, 125.2, 125.0, 124.2, 122.2, 111.7 (d, *J* = 9.0 Hz), 64.1 (d, *J* = 6.6 Hz), 28.9, 16.0 (d, *J* = 7.4 Hz), 15.8. ^{31}P NMR (162 MHz, $CDCl_3$) δ 21.15. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{20}H_{23}NaO_3PS_2^+$: 429.0718, found: 429.0720.

S-(2-(4-(*tert*-butyl)phenyl)benzo[*b*]thiophen-3-yl) *O,O*-diethyl phosphorothioate (**3d**)



Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 74.6 mg, 86%; Yellow Solid; m.p. 84.1-86.3 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (d, *J* = 8.0 Hz, 1H), 7.81 (d, *J* = 8.0 Hz, 1H), 7.75 (d, *J* = 8.4 Hz, 2H), 7.55 – 7.44 (m, 3H), 7.42 – 7.33 (m, 1H), 3.97 – 3.81 (m, 2H), 3.72 – 3.53 (m, 2H), 1.37 (s, 9H), 1.00 (t, *J* = 6.6 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 152.3, 150.2 (d, *J* = 8.0 Hz), 141.7 (d, *J* = 1.8 Hz), 138.0 (d, *J* = 1.3 Hz), 130.6 (d, *J* = 2.5 Hz), 130.1 (d, *J* = 1.4 Hz), 125.6, 125.2, 125.0, 124.1, 122.2, 111.7 (d, *J* = 9.2 Hz), 64.1 (d, *J* = 6.4 Hz), 34.9, 31.4, 16.0 (d, *J* = 7.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 21.09. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{22}H_{27}NaO_3PS_2^+$: 457.1031, found: 457.1038.

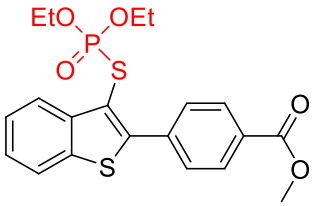
O,O-diethyl *S*-(2-(4-methoxyphenyl)benzo[*b*]thiophen-3-yl) phosphorothioate (**3e**)



Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 81.0 mg, 99%; Yellow Solid; m.p. 82.2-84.1 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 7.6 Hz, 1H), 7.79 (d, *J* = 8.0 Hz, 1H), 7.77 – 7.74 (m, 2H), 7.50 – 7.43

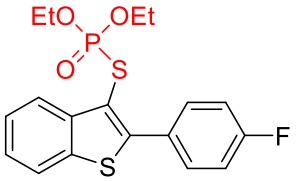
(m, 1H), 7.40 – 7.33 (m, 1H), 7.07 – 6.95 (m, 2H), 3.99 – 3.88 (m, 2H), 3.86 (s, 3H), 3.80 – 3.70 (m, 2H), 1.06 (td, J = 7.0, 0.9 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.4, 150.2 (d, J = 8.1 Hz), 141.8 (d, J = 1.9 Hz), 137.9, 131.7 (d, J = 1.4 Hz), 125.9 (d, J = 2.5 Hz), 125.2, 125.0, 124.2, 122.2, 114.1, 111.2 (d, J = 8.8 Hz), 64.2 (d, J = 6.6 Hz), 55.6, 16.1 (d, J = 7.3 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 21.30. HRMS: $[M+H]^+$ *m/z* calcd for $C_{20}H_{24}O_3PS_2^+$: 407.0899, found: 407.0896.

Methyl 4-(3-((diethoxyphosphoryl)thio)benzo[*b*]thiophen-2-yl)benzoate (**3f**)



Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 1:1); Yield: 45.3 mg, 52%; Yellow Oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 – 8.06 (m, 3H), 7.95 – 7.87 (m, 2H), 7.83 (d, J = 7.9 Hz, 1H), 7.53 – 7.46 (m, 1H), 7.44 – 7.38 (m, 1H), 4.01 – 3.87 (m, 5H), 3.81 – 3.68 (m, 2H), 1.05 (td, J = 7.1, 0.9 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.8, 148.6 (d, J = 8.1 Hz), 141.5 (d, J = 1.9 Hz), 138.3, 138.0 (d, J = 2.6 Hz), 130.5 – 130.3 (m), 129.8, 125.8, 125.3, 124.5, 122.3, 113.3 (d, J = 9.0 Hz), 64.4 (d, J = 6.7 Hz), 52.5, 16.1 (d, J = 7.3 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.82. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{20}H_{21}NaO_5PS_2^+$: 459.0460, found: 459.0470.

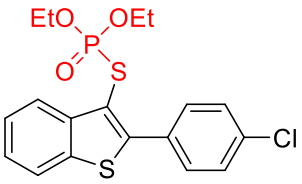
O,O-diethyl *S*-(2-(4-fluorophenyl)benzo[*b*]thiophen-3-yl) phosphorothioate (**3g**)



Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 75.6 mg, 96%; Yellow Solid; m.p. 38.5-40.3 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (d, J = 8.0 Hz, 1H), 7.86 – 7.74 (m, 3H), 7.52 – 7.46 (m, 1H), 7.44 – 7.37 (m, 1H), 7.23 – 7.12 (m, 2H), 4.01 – 3.89 (m, 2H), 3.86 – 3.71 (m, 2H), 1.09 (td, J = 7.1, 0.9 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.3 (d, J = 249.9 Hz), 149.0 (d, J = 8.1 Hz), 141.5 (d, J = 1.8 Hz), 138.0 (d, J = 1.3 Hz), 132.3 (dd, J = 8.2, 1.5 Hz), 129.6 (dd, J = 2.9 Hz), 125.5, 125.2, 124.3, 122.2, 115.7 (d, J = 21.7 Hz), 112.3 (d, J = 8.8 Hz), 64.3 (d, J = 6.7 Hz), 16.1 (d, J = 7.2 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 21.10. ^{19}F

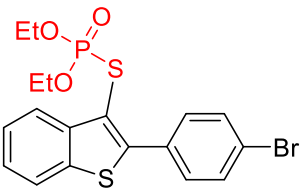
NMR (376 MHz, $CDCl_3$) δ -111.94. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{18}H_{18}NaO_3PS_2^+$: 419.0311, found: 419.0299.

S-(2-(4-chlorophenyl)benzo[*b*]thiophen-3-yl) *O,O*-diethyl phosphorothioate (**3h**)



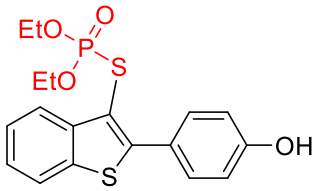
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 67.6 mg, 82%; Yellow Solid; m.p. 73.6-75.8 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 8.10 (d, *J* = 8.0 Hz, 1H), 7.82 (d, *J* = 8.0 Hz, 1H), 7.76 (d, *J* = 8.5 Hz, 2H), 7.54 – 7.37 (m, 4H), 4.05 – 3.89 (m, 2H), 3.86 – 3.73 (m, 2H), 1.09 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.7 (d, *J* = 8.2 Hz), 141.5 (d, *J* = 1.9 Hz), 138.0 (d, *J* = 1.3 Hz), 135.3, 132.0 (d, *J* = 2.5 Hz), 131.7 (d, *J* = 1.5 Hz), 128.9, 125.6, 125.2, 124.3, 122.3, 112.6 (d, *J* = 8.8 Hz), 64.4 (d, *J* = 6.7 Hz), 16.1 (d, *J* = 7.3 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 21.02. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{18}H_{18}ClNaO_3PS_2^+$: 435.0016, found: 435.0014.

S-(2-(4-bromophenyl)benzo[*b*]thiophen-3-yl) *O,O*-diethyl phosphorothioate (**3i**)



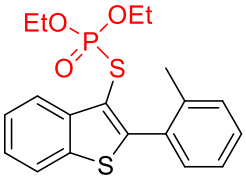
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 65.7 mg, 72%; Yellow Solid; m.p. 39.1-41.3 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 8.10 (d, *J* = 8.0 Hz, 1H), 7.82 (d, *J* = 7.9 Hz, 1H), 7.74 – 7.66 (m, 2H), 7.64 – 7.58 (m, 2H), 7.53 – 7.46 (m, 1H), 7.44 – 7.38 (m, 1H), 4.05 – 3.89 (m, 2H), 3.85 – 3.71 (m, 2H), 1.09 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.7 (d, *J* = 8.0 Hz), 141.5 (d, *J* = 1.7 Hz), 138.1, 132.5 (d, *J* = 2.5 Hz), 131.9 (d, *J* = 1.4 Hz), 131.8, 125.6, 125.2, 124.4, 123.5, 122.3, 112.7 (d, *J* = 8.8 Hz), 64.4 (d, *J* = 6.8 Hz), 16.1 (d, *J* = 7.2 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 21.00. HRMS: $[M+H]^+$ *m/z* calcd for $C_{18}H_{19}BrO_3PS_2^+$: 456.9691, found: 456.9694.

O,O-diethyl *S*-(2-(4-hydroxyphenyl)benzo[*b*]thiophen-3-yl) phosphorothioate (**3j**)



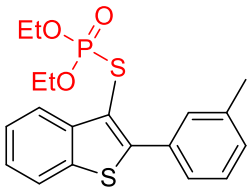
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 1:1); Yield: 54.4 mg, 69%; Yellow Solid; m.p. 165.5-167.3 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 8.2 Hz, 2H), 7.81 (d, *J* = 7.9 Hz, 1H), 7.68 – 7.63 (m, 2H), 7.52 – 7.43 (m, 1H), 7.41 – 7.35 (m, 1H), 6.90 (d, *J* = 8.6 Hz, 2H), 4.04 – 3.88 (m, 2H), 3.86 – 3.69 (m, 2H), 1.08 (t, *J* = 7.5 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.1, 151.3 (d, *J* = 8.1 Hz), 141.7 (d, *J* = 1.7 Hz), 137.8, 131.7 (d, *J* = 1.4 Hz), 125.2, 125.0, 124.6 (d, *J* = 2.5 Hz), 123.8, 122.3, 115.8, 109.8 (d, *J* = 9.1 Hz), 64.7 (d, *J* = 6.8 Hz), 16.1 (d, *J* = 7.3 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 22.04. HRMS: $[M+H]^+$ *m/z* calcd for $C_{18}H_{20}O_4PS_2^+$: 395.0535, found: 395.0531.

O,O-diethyl *S*-(2-(*o*-tolyl)benzo[*b*]thiophen-3-yl) phosphorothioate (**3k**)



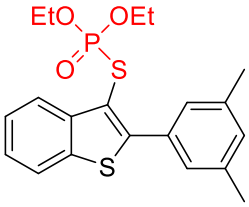
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 28.2 mg, 36%; Yellow Oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (dd, *J* = 7.7, 1.5 Hz, 1H), 7.77 (dd, *J* = 7.4, 1.4 Hz, 1H), 7.55 (dd, *J* = 7.7, 1.5 Hz, 1H), 7.51 – 7.40 (m, 2H), 7.36 (td, *J* = 7.7, 1.5 Hz, 1H), 7.22 (d, *J* = 8.0 Hz, 1H), 7.15 (t, *J* = 7.5, 1.1 Hz, 1H), 4.48 – 3.96 (m, 4H), 2.54 (s, 3H), 1.20 (t, *J* = 7.5 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 142.5, 139.8 (d, *J* = 2.1 Hz), 139.1, 132.8, 130.2 (d, *J* = 8.3 Hz), 129.9, 126.5, 125.4, 124.5 (d, *J* = 5.6 Hz), 122.3, 121.1 (d, *J* = 9.7 Hz), 120.4, 97.9 (d, *J* = 2.2 Hz), 88.2 (d, *J* = 4.5 Hz), 64.7 (d, *J* = 6.5 Hz), 16.2 (d, *J* = 7.5 Hz), 15.4. ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.66. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{19}H_{21}NaO_3PS_2^+$: 415.0562, found: 415.0569.

O,O-diethyl *S*-(2-(*m*-tolyl)benzo[*b*]thiophen-3-yl) phosphorothioate (**3l**)



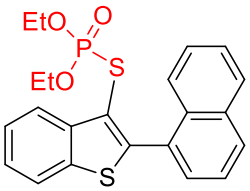
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 53.3 mg, 68%; Yellow Oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 – 7.98 (m, 1H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.59 – 7.49 (m, 2H), 7.42 – 7.36 (m, 1H), 7.34 – 7.23 (m, 2H), 7.19 – 7.12 (m, 1H), 3.93 – 3.74 (m, 2H), 3.68 – 3.54 (m, 2H), 2.35 (s, 3H), 0.95 (t, *J* = 7.5 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.3 (d, *J* = 8.0 Hz), 141.6 (d, *J* = 1.8 Hz), 138.3, 138.1 (d, *J* = 1.3 Hz), 133.4 (d, *J* = 2.6 Hz), 131.0 (d, *J* = 1.5 Hz), 129.8, 128.6, 127.5 (d, *J* = 1.4 Hz), 125.3, 125.0, 124.2, 122.2, 111.9 (d, *J* = 9.1 Hz), 64.1 (d, *J* = 6.5 Hz), 21.6, 16.0 (d, *J* = 7.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 21.15. HRMS: $[M+H]^+$ *m/z* calcd for $C_{19}H_{22}O_3PS_2^+$: 393.0742, found: 393.0739.

S-(2-(3,5-dimethylphenyl)benzo[*b*]thiophen-3-yl) *O,O*-diethyl phosphorothioate (**3m**)



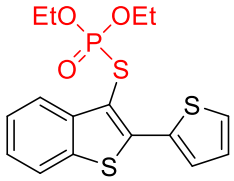
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 81 mg, 99%; Yellow Oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (d, *J* = 8.0 Hz, 1H), 7.81 (d, *J* = 8.0 Hz, 1H), 7.55 – 7.44 (m, 1H), 7.42 – 7.33 (m, 3H), 7.07 (s, 1H), 4.03 – 3.83 (m, 2H), 3.79 – 3.61 (m, 2H), 2.40 (s, 6H), 1.04 (t, *J* = 7.0 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.6 (d, *J* = 8.0 Hz), 141.7, 138.3, 138.1, 133.4 (d, *J* = 2.3 Hz), 130.8, 128.2, 125.3, 125.0, 124.2, 122.2, 111.8 (d, *J* = 8.9 Hz), 64.1 (d, *J* = 6.5 Hz), 21.5, 16.0 (d, *J* = 7.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 21.30. HRMS: $[M+H]^+$ *m/z* calcd for $C_{20}H_{24}O_3PS_2^+$: 407.0899, found: 407.0896.

O,O-diethyl *S*-(2-(naphthalen-1-yl)benzo[*b*]thiophen-3-yl) phosphorothioate (**3n**)



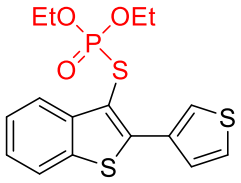
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 62.5 mg, 73%; Yellow Solid; m.p. 71.3-73.1 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, *J* = 8.0 Hz, 1H), 7.99 – 7.91 (m, 2H), 7.88 (d, *J* = 8.0 Hz, 1H), 7.82 (d, *J* = 8.3 Hz, 1H), 7.74 – 7.68 (m, 1H), 7.62 – 7.42 (m, 5H), 3.87 – 3.65 (m, 2H), 3.63 – 3.46 (m, 2H), 0.96 (t, *J* = 7.1 Hz, 6H ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.3 (d, *J* = 8.1 Hz), 140.9 (d, *J* = 1.8 Hz), 139.2, 133.6, 132.4 (d, *J* = 1.6 Hz), 130.7 (d, *J* = 2.2 Hz), 130.0 (d, *J* = 2.0 Hz), 129.8, 128.4, 126.9, 126.4, 126.2, 125.5, 125.2, 125.1, 124.2, 122.3, 115.7 (d, *J* = 8.7 Hz), 64.0 (d, *J* = 6.6 Hz), 15.9 (d, *J* = 7.3 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.86. HRMS: $[M+H]^+$ *m/z* calcd for $C_{22}H_{22}O_3PS_2^+$: 429.0742, found: 429.0743.

O,O-diethyl *S*-(2-(thiophen-2-yl)benzo[*b*]thiophen-3-yl) phosphorothioate (**3o**)



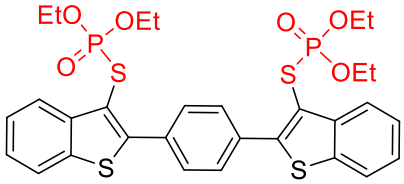
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 58.4 mg, 76%; Yellow Solid; m.p. 59.0-61.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.1 Hz, 1H), 7.76 (d, *J* = 7.9 Hz, 1H), 7.66 (d, *J* = 3.7 Hz, 1H), 7.51 – 7.42 (m, 2H), 7.41 – 7.32 (m, 1H), 7.19 – 7.09 (m, 1H), 4.11 – 3.99 (m, 2H), 3.98 – 3.88 (m, 2H), 1.10 (td, *J* = 7.1, 0.9 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.2 (d, *J* = 8.3 Hz), 141.8 (d, *J* = 1.9 Hz), 137.3, 135.0 (d, *J* = 3.4 Hz), 129.1 (d, *J* = 1.8 Hz), 128.3, 127.4, 125.7, 125.2, 124.3, 122.1, 111.6 (d, *J* = 9.4 Hz), 64.6 (d, *J* = 6.9 Hz), 16.1 (d, *J* = 7.1 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.57. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{16}H_{17}NaO_3PS_3^+$: 406.9970, found: 406.9967.

O,O-diethyl *S*-(2-(thiophen-3-yl)benzo[*b*]thiophen-3-yl) phosphorothioate (**3p**)



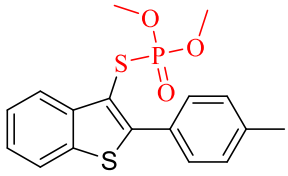
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 59.9 mg, 78%; Yellow Oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 – 7.95 (m, 2H), 7.78 (d, *J* = 8.0 Hz, 1H), 7.65 (d, *J* = 5.1 Hz, 1H), 7.54 – 7.33 (m, 3H), 4.12 – 3.94 (m, 2H), 3.92 – 3.68 (m, 2H), 1.08 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.8 (d, *J* = 8.2 Hz), 141.7 (d, *J* = 1.9 Hz), 137.4, 133.8 (d, *J* = 2.8 Hz), 128.9, 126.0 (d, *J* = 2.0 Hz), 126.0, 125.4, 125.1, 124.1, 122.1, 111.4 (d, *J* = 9.0 Hz), 64.4 (d, *J* = 6.9 Hz), 16.1 (d, *J* = 7.2 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 21.09. HRMS: $[M+H]^+$ *m/z* calcd for $C_{16}H_{18}O_3PS_3^+$: 385.0150, found: 385.0154.

S,S'-(1,4-phenylenebis(benzo[*b*]thiophene-2,3-diyl)) *O,O,O',O'*-tetraethyl bis(phosphorothioate) (**3q**)



Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 46.1 mg, 68%; Yellow Solid; m.p. 104.2-106.8 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (d, *J* = 8.0 Hz, 2H), 7.96 (s, 4H), 7.85 (d, *J* = 7.9 Hz, 2H), 7.62 – 7.48 (m, 2H), 7.46 – 7.39 (m, 2H), 4.06 – 3.94 (m, 4H), 3.90 – 3.78 (m, 4H), 1.11 (t, *J* = 7.5 Hz, 12H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.2 (d, *J* = 8.2 Hz), 141.7 (d, *J* = 1.8 Hz), 138.2, 134.2 (d, *J* = 2.6 Hz), 130.5 (d, *J* = 1.4 Hz), 125.7, 125.3, 124.4, 122.3, 112.8 (d, *J* = 8.9 Hz), 64.5 (d, *J* = 6.8 Hz), 16.2 (d, *J* = 7.1 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 21.07. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{30}H_{32}NaO_6P_2S_4^+$: 701.0449, found: 701.0432.

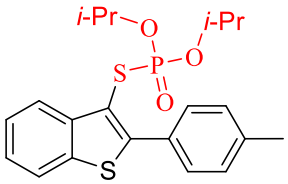
O,O-dimethyl *S*-(2-(*p*-tolyl)benzo[*b*]thiophen-3-yl) phosphorothioate (**3r**)



Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 40.8 mg, 56%; Yellow Solid; m.p. 71.1-73.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 – 8.01 (m, 1H), 7.89 – 7.79 (m, 1H), 7.72 – 7.60 (m, 2H), 7.56 – 7.45 (m, 1H),

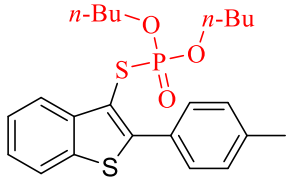
7.44 – 7.36 (m, 1H), 7.30 (d, J = 7.9 Hz, 2H), 3.43 (s, 3H), 3.39 (s, 3H), 2.42 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.7 (d, J = 8.0 Hz), 141.6 (d, J = 1.8 Hz), 139.3, 138.1, 130.6 (d, J = 2.5 Hz), 130.3 (d, J = 1.6 Hz), 129.5, 125.3, 125.2, 123.9, 122.3, 111.2 (d, J = 8.9 Hz), 54.2 (d, J = 6.2 Hz), 21.6. ^{31}P NMR (162 MHz, $CDCl_3$) δ 24.48. HRMS: $[M+H]^+$ *m/z* calcd for $C_{17}H_{18}O_3PS_2^+$: 365.0429, found: 365.0424.

O,O-diisopropyl *S*-(2-(*p*-tolyl)benzo[*b*]thiophen-3-yl) phosphorothioate (**3s**)



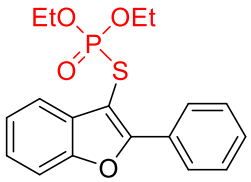
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 83.1 mg, 99%; Yellow Solid; m.p. 58.0-60.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 – 8.10 (m, 1H), 7.86 – 7.78 (m, 1H), 7.76 – 7.72 (m, 2H), 7.52 – 7.42 (m, 1H), 7.41 – 7.34 (m, 1H), 7.28 (d, J = 8.0 Hz, 2H), 4.65 – 4.47 (m, 2H), 2.42 (s, 3H), 1.18 (d, J = 6.2 Hz, 6H), 1.08 (d, J = 6.2 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.2 (d, J = 8.5 Hz), 141.8 (d, J = 1.8 Hz), 139.1, 138.0, 130.7 (d, J = 2.6 Hz), 130.4 (d, J = 1.5 Hz), 129.3, 125.2, 124.9, 124.8, 122.0, 112.3 (d, J = 8.7 Hz), 73.7 (d, J = 7.6 Hz), 23.9 (d, J = 4.1 Hz), 23.6 (d, J = 5.8 Hz), 21.5. ^{31}P NMR (162 MHz, $CDCl_3$) δ 19.73. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{21}H_{25}NaO_3PS_2^+$: 443.0875, found: 443.0878.

O,O-dibutyl *S*-(2-(*p*-tolyl)benzo[*b*]thiophen-3-yl) phosphorothioate (**3t**)



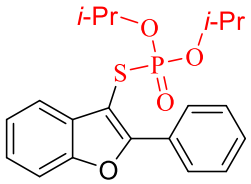
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 78.0 mg, 87%; Yellow Oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 – 8.03 (m, 1H), 7.88 – 7.76 (m, 1H), 7.70 (d, J = 8.1 Hz, 2H), 7.49 – 7.43 (m, 1H), 7.42 – 7.35 (m, 1H), 7.29 (d, J = 7.9 Hz, 2H), 3.91 – 3.74 (m, 2H), 3.69 – 3.55 (m, 2H), 2.42 (s, 3H), 1.41 – 1.29 (m, 4H), 1.20 – 1.06 (m, 4H), 0.80 (t, J = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.2 (d, J = 8.1 Hz), 141.8 (d, J = 1.7 Hz), 139.2, 138.0, 130.7 (d, J = 2.5 Hz), 130.3 (d, J = 1.7 Hz), 129.4, 125.2, 125.0, 124.3, 122.2, 111.7 (d, J = 9.0 Hz), 67.9 (d, J = 7.0 Hz), 32.2 (d, J = 7.1 Hz), 21.5, 18.6, 13.7. ^{31}P NMR (162 MHz, $CDCl_3$) δ 21.39. HRMS: $[M+H]^+$ *m/z* calcd for $C_{23}H_{30}O_3PS_2^+$: 449.1368, found: 449.1371.

O,O-diethyl *S*-(2-phenylbenzofuran-3-yl) phosphorothioate (**3u**)



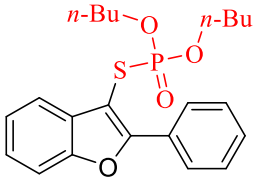
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 49.2 mg, 68%; Yellow Solid; m.p. 63.1-65.2 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 (d, *J* = 7.7 Hz, 2H), 7.82 – 7.71 (m, 1H), 7.55 – 7.41 (m, 4H), 7.38 – 7.29 (m, 2H), 4.13 – 4.02 (m, 2H), 3.99 – 3.88 (m, 2H), 1.08 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.2 (d, *J* = 8.5 Hz), 153.8, 131.1, 129.8, 129.7 (d, *J* = 3.3 Hz), 128.7, 128.0, 125.5, 123.6, 120.7, 111.5, 99.0 (d, *J* = 9.2 Hz), 64.5 (d, *J* = 6.6 Hz), 16.0 (d, *J* = 7.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 21.51. HRMS: $[M+H]^+$ *m/z* calcd for $C_{18}H_{20}O_4PS^+$: 363.0814, found: 363.0807.

O,O-diisopropyl *S*-(2-phenylbenzofuran-3-yl) phosphorothioate (**3v**)



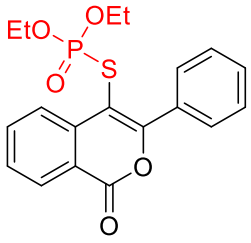
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 46.2 mg, 59%; Yellow Oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 – 8.25 (m, 2H), 7.85 – 7.80 (m, 1H), 7.53 – 7.46 (m, 3H), 7.45 – 7.40 (m, 1H), 7.36 – 7.30 (m, 2H), 4.73 – 4.64 (m, 2H), 1.21 (d, *J* = 6.2 Hz, 6H), 1.12 (d, *J* = 6.2 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.9 (d, *J* = 8.9 Hz), 153.8, 131.3 (d, *J* = 1.5 Hz), 129.8 (d, *J* = 3.1 Hz), 129.6, 128.6, 128.0 (d, *J* = 1.4 Hz), 125.4, 123.5, 121.4, 111.3, 99.5 (d, *J* = 8.9 Hz), 73.9 (d, *J* = 7.7 Hz), 24.0 (d, *J* = 4.0 Hz), 23.7 (d, *J* = 5.8 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 19.86. HRMS: $[M+H]^+$ *m/z* calcd for $C_{20}H_{24}O_4PS^+$: 391.1127, found: 391.1123.

O,O-dibutyl *S*-(2-phenylbenzofuran-3-yl) phosphorothioate (**3w**)



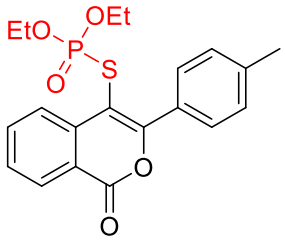
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 51.8 mg, 62%; Yellow Oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 – 8.18 (m, 2H), 7.78 – 7.71 (m, 1H), 7.55 – 7.46 (m, 3H), 7.46 – 7.40 (m, 1H), 7.37 – 7.30 (m, 2H), 4.04 – 3.93 (m, 2H), 3.91 – 3.82 (m, 2H), 1.42 – 1.35 (m, 4H), 1.22 – 1.12 (m, 4H), 0.79 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.1 (d, *J* = 8.5 Hz), 153.8, 131.2 (d, *J* = 1.5 Hz), 129.8, 129.7 (d, *J* = 3.1 Hz), 128.7, 128.0 (d, *J* = 1.4 Hz), 125.5, 123.6, 120.8, 111.5, 99.1 (d, *J* = 9.1 Hz), 68.2 (d, *J* = 7.1 Hz), 32.2 (d, *J* = 7.2 Hz), 18.7, 13.7. ^{31}P NMR (162 MHz, $CDCl_3$) δ 21.78. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{22}H_{27}NaO_4PS^+$: 441.1260, found: 441.1261.

O,O-diethyl *S*-(1-oxo-3-phenyl-1*H*-isochromen-4-yl) phosphorothioate (**4a**)



Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 65.5 mg, 84%; Yellow Solid; m.p. 70.3-72.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 (d, *J* = 7.9 Hz, 1H), 8.25 (d, *J* = 8.1 Hz, 1H), 7.91 – 7.77 (m, 3H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.49 – 7.44 (m, 3H), 3.93 – 3.74 (m, 2H), 3.70 – 3.52 (m, 2H), 1.05 (t, *J* = 7.0 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.3, 160.3 (d, *J* = 8.4 Hz), 138.5, 135.1, 133.0 (d, *J* = 2.8 Hz), 130.5 (d, *J* = 1.6 Hz), 130.4, 129.8, 129.0, 128.1, 126.5, 120.7, 102.9 (d, *J* = 9.0 Hz), 64.3 (d, *J* = 6.7 Hz), 16.0 (d, *J* = 7.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.72. HRMS: $[M+H]^+$ *m/z* calcd for $C_{19}H_{20}O_5PS^+$: 391.0764, found: 391.0762.

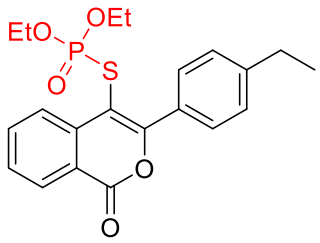
O,O-diethyl *S*-(1-oxo-3-(*p*-tolyl)-1*H*-isochromen-4-yl) phosphorothioate (**4b**)



Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 75.1 mg, 93%; Yellow Solid; m.p. 79.2-81.9 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.28 – 8.24 (m, 1H), 8.20 – 8.15 (m, 1H), 7.83 – 7.74 (m, 1H), 7.71 – 7.64 (m, 2H),

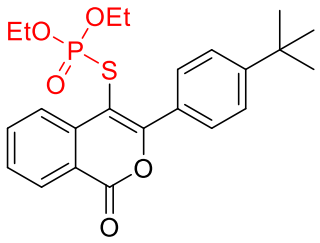
7.56 – 7.45 (m, 1H), 7.25 – 7.15 (m, 2H), 3.91 – 3.72 (m, 2H), 3.66 – 3.48 (m, 2H), 2.35 (s, 3H), 0.99 (t, J = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.4, 160.5 (d, J = 8.4 Hz), 140.8, 138.6 (d, J = 1.3 Hz), 135.1, 130.4 (d, J = 1.4 Hz), 130.1 (d, J = 2.7 Hz), 129.8, 128.8, 128.8, 126.5, 120.7, 102.4 (d, J = 8.9 Hz), 64.3 (d, J = 6.7 Hz), 21.7, 16.0 (d, J = 7.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.90. HRMS: $[M+H]^+$ *m/z* calcd for $C_{20}H_{22}O_5PS^+$: 405.0920, found: 405.0925.

O,O-diethyl *S*-(3-(4-ethylphenyl)-1-oxo-1*H*-isochromen-4-yl) phosphorothioate (**4c**)



Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 78.6 mg, 94%; Yellow Solid; m.p. 75.5-77.3 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.37 – 8.29 (m, 1H), 8.27 – 8.21 (m, 1H), 7.92 – 7.81 (m, 1H), 7.77 (d, J = 8.2 Hz, 2H), 7.64 – 7.49 (m, 1H), 7.30 (d, J = 8.3 Hz, 2H), 3.90 – 3.77 (m, 2H), 3.68 – 3.53 (m, 2H), 2.70 (q, 2H), 1.25 (t, J = 7.6 Hz, 3H), 1.04 (t, J = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.4, 160.5 (d, J = 8.3 Hz), 147.1, 138.6 (d, J = 1.4 Hz), 135.1, 130.6 (d, J = 1.5 Hz), 130.4 (d, J = 2.8 Hz), 129.8, 128.8, 127.6, 126.5, 120.7, 102.4 (d, J = 9.1 Hz), 64.2 (d, J = 6.7 Hz), 29.0, 16.0 (d, J = 7.4 Hz), 15.8. ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.79. HRMS: $[M+H]^+$ *m/z* calcd for $C_{21}H_{24}O_5PS^+$: 419.1077, found: 419.1073.

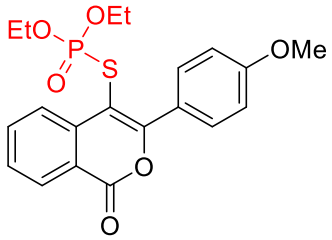
S-(3-(4-(*tert*-butyl)phenyl)-1-oxo-1*H*-isochromen-4-yl) *O,O*-diethyl phosphorothioate (**4d**)



Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 83 mg, 93%; Yellow Solid; m.p. 130.1-132.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (dd, J = 7.9, 1.4 Hz, 1H), 8.23 (d, J = 8.1 Hz, 1H), 7.87 – 7.73 (m, 3H), 7.61 – 7.53 (m, 1H), 7.51 – 7.42 (m, 2H), 3.92 – 3.67 (m, 2H), 3.64 – 3.42 (m, 2H), 1.33 (s, 9H), 1.01 (t, J = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.3, 160.3 (d, J

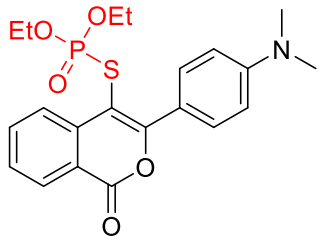
= 8.2 Hz), 153.9, 138.6 (d, J = 1.3 Hz), 135.0, 130.3 (d, J = 1.5 Hz), 130.1 (d, J = 2.8 Hz), 129.7, 128.8, 126.4, 125.0, 120.7, 102.4 (d, J = 9.1 Hz), 64.1 (d, J = 6.6 Hz), 35.1, 31.3, 16.0 (d, J = 7.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.73. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{23}H_{27}NaO_5PS$: 469.1209, found: 469.1201.

O,O-diethyl *S*-(3-(4-methoxyphenyl)-1-oxo-1*H*-isochromen-4-yl) phosphorothioate (**4e**)



Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 78.1 mg, 93%; Yellow Oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.36 – 8.27 (m, 1H), 8.27 – 8.18 (m, 1H), 7.88 – 7.74 (m, 3H), 7.65 – 7.50 (m, 1H), 7.02 – 6.93 (m, 2H), 3.96 – 3.81 (m, 5H), 3.74 – 3.59 (m, 2H), 1.06 (t, J = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.5, 161.3, 160.2 (d, J = 8.3 Hz), 138.8 (d, J = 1.4 Hz), 135.0, 132.2 (d, J = 1.5 Hz), 129.7, 128.7, 126.5, 125.3 (d, J = 2.8 Hz), 120.6, 113.5, 101.8 (d, J = 8.9 Hz), 64.3 (d, J = 6.8 Hz), 55.6, 16.1 (d, J = 7.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.93. HRMS: $[M+H]^+$ *m/z* calcd for $C_{20}H_{22}O_6PS^+$: 421.0869, found: 421.0873.

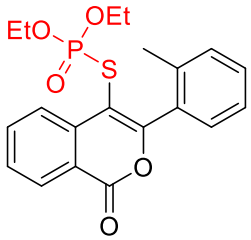
S-(3-(4-(dimethylamino)phenyl)-1-oxo-1*H*-isochromen-4-yl) *O,O*-diethyl phosphorothioate (**4f**)



Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 50.2 mg, 58%; Yellow Solid; m.p. 90.1-92.4 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 – 8.26 (m, 1H), 8.24 – 8.18 (m, 1H), 7.85 – 7.76 (m, 3H), 7.57 – 7.44 (m, 1H), 6.76 – 6.67 (m, 2H), 3.93 – 3.82 (m, 2H), 3.71 – 3.57 (m, 2H), 3.03 (s, 6H), 1.03 (t, J = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.8, 160.9 (d, J = 8.0 Hz), 151.6, 139.4, 134.9, 131.8 (d, J = 1.5 Hz), 129.6, 128.1, 126.3, 120.3, 119.8 (d, J = 2.6 Hz), 110.9, 99.8 (d, J = 9.0 Hz), 64.2 (d, J = 6.6 Hz), 40.3, 16.0 (d, J = 7.6 Hz). ^{31}P NMR (162 MHz,

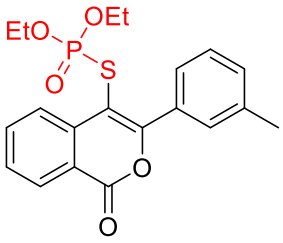
$CDCl_3$) δ 21.21. HRMS: $[M+H]^+$ *m/z* calcd for $C_{21}H_{25}NO_5PS^+$: 434.1186, found: 434.1183.

O,O-diethyl *S*-(1-oxo-3-(*o*-tolyl)-1*H*-isochromen-4-yl) phosphorothioate (**4g**)



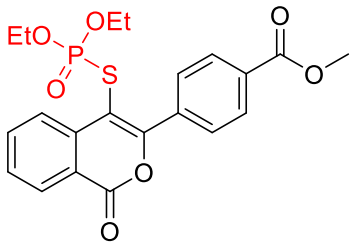
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 57.4 mg, 72%; Yellow Solid; m.p. 79.4-81.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 – 8.31 (m, 1H), 8.25 – 8.16 (m, 1H), 7.98 – 7.74 (m, 1H), 7.72 – 7.56 (m, 1H), 7.54 – 7.48 (m, 1H), 7.39 – 7.34 (m, 1H), 7.33 – 7.23 (m, 2H), 3.91 – 3.76 (m, 2H), 3.76 – 3.64 (m, 2H), 2.38 (s, 3H), 1.13 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.5, 161.2 (d, *J* = 8.5 Hz), 138.2, 137.9, 135.2, 132.9 (d, *J* = 2.5 Hz), 131.0, 130.5, 130.2, 130.0, 129.1, 126.3, 125.5, 121.0, 104.7 (d, *J* = 8.8 Hz), 64.3 (d, *J* = 6.9 Hz), 20.1, 16.1 (d, *J* = 7.2 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.90. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{20}H_{21}NaO_5PS^+$: 427.0740, found: 427.0743.

O,O-diethyl *S*-(1-oxo-3-(*m*-tolyl)-1*H*-isochromen-4-yl) phosphorothioate (**4h**)



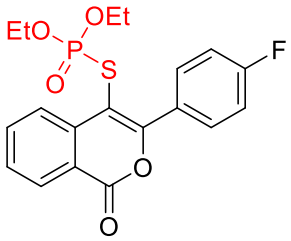
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 72.7 mg, 90%; Yellow Oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 – 8.23 (m, 1H), 8.21 – 8.14 (m, 1H), 7.85 – 7.71 (m, 1H), 7.66 – 7.55 (m, 2H), 7.54 – 7.46 (m, 1H), 7.29 (t, *J* = 7.9 Hz, 1H), 7.21 (d, *J* = 7.4 Hz, 1H), 3.87 – 3.69 (m, 2H), 3.64 – 3.44 (m, 2H), 2.35 (s, 3H), 0.99 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.3, 160.5 (d, *J* = 8.4 Hz), 138.5 (d, *J* = 1.4 Hz), 137.9, 135.1, 132.9 (d, *J* = 2.7 Hz), 131.1, 130.9 (d, *J* = 1.6 Hz), 129.8, 128.9, 128.0, 127.7 (d, *J* = 1.6 Hz), 126.5, 120.7, 102.7 (d, *J* = 8.9 Hz), 64.2 (d, *J* = 6.7 Hz), 21.5, 16.0 (d, *J* = 7.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.78. HRMS: $[M+H]^+$ *m/z* calcd for $C_{20}H_{22}O_5PS^+$: 405.0920, found: 405.0918.

Methyl 4-(4-((diethoxyphosphoryl)thio)-1-oxo-1*H*-isochromen-3-yl)benzoate (**4i**)



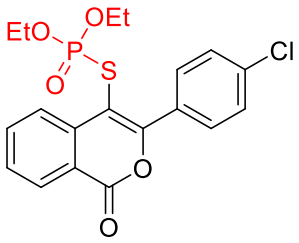
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 1:1); Yield: 52.9 mg, 59%; Yellow Solid; m.p. 116.2-118.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 – 8.33 (m, 1H), 8.28 – 8.23 (m, 1H), 8.17 – 8.11 (m, 2H), 7.97 – 7.91 (m, 2H), 7.91 – 7.84 (m, 1H), 7.66 – 7.58 (m, 1H), 3.96 (s, 3H), 3.94 – 3.83 (m, 2H), 3.78 – 3.64 (m, 2H), 1.08 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 161.0, 159.2 (d, *J* = 8.6 Hz), 138.2 (d, *J* = 1.4 Hz), 137.3 (d, *J* = 2.7 Hz), 135.3, 131.6, 130.6 (d, *J* = 1.6 Hz), 130.0, 129.4, 129.3, 126.7, 120.9, 104.1 (d, *J* = 8.9 Hz), 64.5 (d, *J* = 7.0 Hz), 52.6, 16.1 (d, *J* = 7.2 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.57. HRMS: $[M+H]^+$ *m/z* calcd for $C_{21}H_{22}O_7PS^+$: 449.0818, found: 449.0822.

O,O-diethyl *S*-(3-(4-fluorophenyl)-1-oxo-1*H*-isochromen-4-yl) phosphorothioate (**4j**)



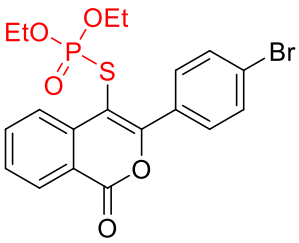
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 77.5 mg, 95%; Yellow Solid; m.p. 84.5-86.7 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 (d, *J* = 7.9 Hz, 1H), 8.21 (d, *J* = 8.1 Hz, 1H), 7.91 – 7.77 (m, 3H), 7.57 (t, *J* = 7.6 Hz, 1H), 7.15 (t, *J* = 8.5 Hz, 2H), 3.97 – 3.80 (m, 2H), 3.78 – 3.63 (m, 2H), 1.08 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.7 (d, *J* = 251.9 Hz), 161.0, 159.2 (d, *J* = 8.5 Hz), 138.2 (d, *J* = 1.4 Hz), 135.1, 132.6 (dd, *J* = 8.7, 1.6 Hz), 129.8, 129.2 (t, *J* = 3.1 Hz), 129.1, 126.5, 120.7, 115.2 (d, *J* = 21.9 Hz), 103.0 (d, *J* = 8.9 Hz), 64.3 (d, *J* = 6.9 Hz), 16.0 (d, *J* = 7.2 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.74. ^{19}F NMR (376 MHz, $CDCl_3$) δ -109.06. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{19}H_{18}FNaO_5PS^+$: 431.0489, found: 431.0486.

S-(3-(4-chlorophenyl)-1-oxo-1*H*-isochromen-4-yl) *O,O*-diethyl phosphorothioate (**4k**)



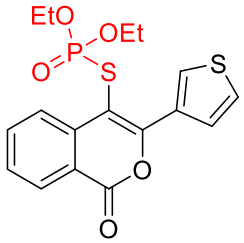
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 70.4 mg, 83%; Yellow Solid; m.p. 91.3-93.6 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 – 8.29 (m, 1H), 8.24 (d, *J* = 8.1 Hz, 1H), 7.92 – 7.74 (m, 3H), 7.60 (t, *J* = 7.6 Hz, 1H), 7.45 (d, *J* = 8.6 Hz, 2H), 3.98 – 3.84 (m, 2H), 3.80 – 3.67 (m, 2H), 1.10 (t, *J* = 7.0 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.1, 159.2 (d, *J* = 8.4 Hz), 138.2, 136.6, 135.2, 131.9 (d, *J* = 1.6 Hz), 131.5 (d, *J* = 2.7 Hz), 129.9, 129.2, 128.4, 126.6, 120.8, 103.4 (d, *J* = 8.9 Hz), 64.5 (d, *J* = 6.9 Hz), 16.1 (d, *J* = 7.3 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.71. HRMS: $[M+H]^+$ *m/z* calcd for $C_{19}H_{19}ClO_5PS^+$: 425.0374, found: 425.0376.

S-(3-(4-bromophenyl)-1-oxo-1*H*-isochromen-4-yl) *O,O*-diethyl phosphorothioate (**4l**)



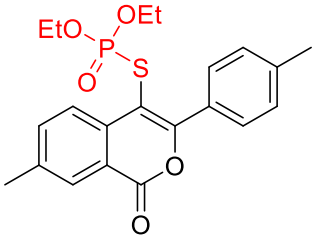
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 72.1 mg, 77%; Yellow Solid; m.p. 80.0-82.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 – 8.29 (m, 1H), 8.22 (d, *J* = 8.1 Hz, 1H), 7.96 – 7.80 (m, 1H), 7.78 – 7.68 (m, 2H), 7.64 – 7.54 (m, 3H), 3.98 – 3.81 (m, 2H), 3.79 – 3.65 (m, 2H), 1.09 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.0, 159.1 (d, *J* = 8.5 Hz), 138.2 (d, *J* = 1.5 Hz), 135.2, 132.0 (d, *J* = 1.7 Hz), 131.9 (d, *J* = 2.8 Hz), 131.3, 129.8, 129.2, 126.5, 124.9, 120.7, 103.4 (d, *J* = 9.1 Hz), 64.4 (d, *J* = 7.0 Hz), 16.0 (d, *J* = 7.2 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.64. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{19}H_{18}BrNaO_5PS^+$: 490.9688, found: 490.9677.

O,O-diethyl *S*-(1-oxo-3-(thiophen-3-yl)-1*H*-isochromen-4-yl) phosphorothioate (**4m**)



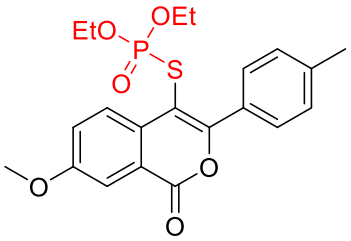
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 75.2 mg, 95%; Yellow Solid; m.p. 70.2-72.6 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 – 8.26 (m, 1H), 8.26 – 8.19 (m, 2H), 7.91 – 7.77 (m, 2H), 7.62 – 7.48 (m, 1H), 7.44 – 7.35 (m, 1H), 4.05 – 3.90 (m, 2H), 3.85 – 3.70 (m, 2H), 1.07 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.0, 155.3 (d, *J* = 8.4 Hz), 138.6 (d, *J* = 1.7 Hz), 135.0, 133.6 (d, *J* = 3.1 Hz), 130.3 (d, *J* = 1.8 Hz), 129.7, 129.1, 128.8, 126.6, 125.3, 120.5, 101.5 (d, *J* = 9.2 Hz), 64.5 (d, *J* = 6.9 Hz), 16.1 (d, *J* = 7.3 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.68. HRMS: $[M+H]^+$ *m/z* calcd for $C_{17}H_{18}O_5PS_2^+$: 397.0328, found: 397.0331.

O,O-diethyl *S*-(7-methyl-1-oxo-3-(*p*-tolyl)-1*H*-isochromen-4-yl) phosphorothioate (**4n**)



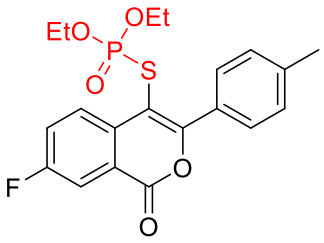
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 81.9 mg, 98%; Yellow Solid; m.p. 140.5-142.4 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 – 8.01 (m, 2H), 7.73 (d, *J* = 8.2 Hz, 2H), 7.67 – 7.61 (m, 1H), 7.26 (d, *J* = 8.1 Hz, 2H), 3.92 – 3.77 (m, 2H), 3.71 – 3.55 (m, 2H), 2.48 (s, 3H), 2.40 (s, 3H), 1.05 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.6, 159.5 (d, *J* = 8.5 Hz), 140.6, 139.2, 136.3, 136.2 (d, *J* = 1.4 Hz), 130.4 (d, *J* = 1.7 Hz), 130.2 (d, *J* = 2.8 Hz), 129.5, 128.7, 126.5, 120.5, 102.3 (d, *J* = 9.1 Hz), 64.2 (d, *J* = 6.7 Hz), 21.6, 21.4, 16.0 (d, *J* = 7.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.93. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{21}H_{23}NaO_5PS^+$: 441.0896, found: 441.0894.

O,O-diethyl *S*-(7-methoxy-1-oxo-3-(*p*-tolyl)-1*H*-isochromen-4-yl) phosphorothioate (**4o**)



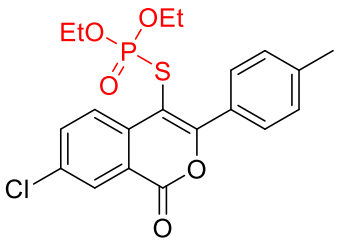
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 66 mg, 76%; Yellow Solid; m.p. 88.2-90.8 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, *J* = 8.9 Hz, 1H), 7.78 – 7.64 (m, 3H), 7.41 (dd, *J* = 8.9, 2.8 Hz,1H), 7.27 (s, 1H), 7.25 (s, 1H), 3.93 (s, 3H), 3.90 – 3.80 (m, 2H), 3.71 – 3.60 (m, 2H), 2.41 (s, 3H), 1.07 (t, *J* = 7.0 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.6, 160.1, 158.4 (d, *J* = 8.6 Hz), 140.5, 132.3, 130.4 (d, *J* = 1.6 Hz), 130.2 (d, *J* = 2.8 Hz), 128.8, 128.4, 124.5, 121.9, 110.2, 102.3 (d, *J* = 9.1 Hz), 64.3 (d, *J* = 6.8 Hz), 56.1, 21.7, 16.1 (d, *J* = 7.3 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.98. HRMS: $[M+H]^+$ *m/z* calcd for $C_{21}H_{24}O_6PS^+$: 435.1026, found: 435.1011.

O,O-diethyl *S*-(7-fluoro-1-oxo-3-(*p*-tolyl)-1*H*-isochromen-4-yl) phosphorothioate (**4p**)



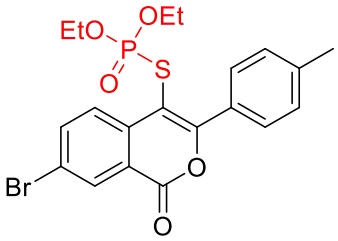
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 67.5 mg, 80%; Yellow Solid; m.p. 90.5-92.6 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 – 8.21 (m, 1H), 8.03 – 7.91 (m, 1H), 7.79 – 7.67 (m, 2H), 7.62 – 7.49 (m, 1H), 7.27 (d, *J* = 8.3 Hz, 2H), 3.94 – 3.78 (m, 2H), 3.74 – 3.61 (m, 2H), 2.41 (s, 3H), 1.08 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.3 (d, *J* = 251.4 Hz), 160.5 (d, *J* = 3.4 Hz), 159.9 (dd, *J* = 8.5, 2.6 Hz), 140.9, 135.1 (d, *J* = 1.7 Hz), 130.4 (d, *J* = 1.6 Hz), 129.8 (d, *J* = 2.8 Hz), 129.4 (d, *J* = 7.9 Hz), 128.9, 123.3 (d, *J* = 22.8 Hz), 122.4 (d, *J* = 8.2 Hz), 115.2 (d, *J* = 23.5 Hz), 101.9 (d, *J* = 9.1 Hz), 64.4 (d, *J* = 6.9 Hz), 21.7, 16.1 (d, *J* = 7.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.79. ^{19}F NMR (376 MHz, $CDCl_3$) δ -110.32. HRMS: $[M+H]^+$ *m/z* calcd for $C_{20}H_{21}FO_5PS^+$: 423.0826, found: 423.0824.

S-(7-chloro-1-oxo-3-(*p*-tolyl)-1*H*-isochromen-4-yl) *O,O*-diethyl phosphorothioate (**4q**)



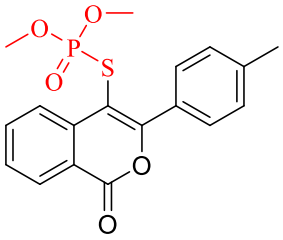
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 77.1 mg, 88%; Yellow Solid; m.p. 119.5-121.6 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, *J* = 2.3 Hz, 1H), 8.12 (d, *J* = 8.7 Hz, 1H), 7.78 – 7.64 (m, 3H), 7.26 – 7.11 (m, 2H), 3.92 – 3.71 (m, 2H), 3.68 – 3.53 (m, 2H), 2.35 (s, 3H), 1.02 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.7 (d, *J* = 8.4 Hz), 160.3, 141.1, 137.1 (d, *J* = 1.4 Hz), 135.3, 134.8, 130.4 (d, *J* = 1.5 Hz), 129.8 (d, *J* = 2.8 Hz), 129.1, 128.8, 128.4, 121.8, 101.9 (d, *J* = 9.0 Hz), 64.4 (d, *J* = 6.9 Hz), 21.7, 16.1 (d, *J* = 7.3 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.72. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{20}H_{20}ClNaO_5PS^+$: 461.0350, found: 461.0347.

S-(7-bromo-1-oxo-3-(*p*-tolyl)-1*H*-isochromen-4-yl) *O,O*-diethyl phosphorothioate (**4r**)



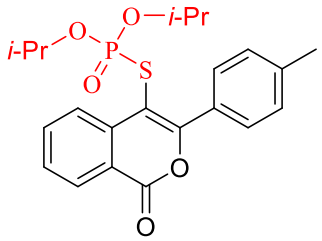
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 81 mg, 84%; Yellow Solid; m.p. 123.3-125.7 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.37 (d, *J* = 2.1 Hz, 1H), 8.04 (d, *J* = 8.7 Hz, 1H), 7.92 – 7.77 (m, 1H), 7.66 (d, *J* = 8.1 Hz, 2H), 7.21 (d, *J* = 7.8 Hz, 2H), 3.89 – 3.71 (m, 2H), 3.68 – 3.54 (m, 2H), 2.35 (s, 3H), 1.02 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.8 (d, *J* = 8.3 Hz), 160.1, 141.1, 138.1, 137.5 (d, *J* = 1.4 Hz), 132.1, 130.4 (d, *J* = 1.5 Hz), 129.8 (d, *J* = 2.8 Hz), 128.8, 128.4, 122.6, 122.0, 102.0 (d, *J* = 8.9 Hz), 64.4 (d, *J* = 6.9 Hz), 21.7, 16.1 (d, *J* = 7.3 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.69. HRMS: $[M+H]^+$ *m/z* calcd for $C_{20}H_{21}BrO_5PS^+$: 483.0025, found: 483.0033.

O,O-dimethyl *S*-(1-oxo-3-(*p*-tolyl)-1*H*-isochromen-4-yl) phosphorothioate (**4s**)



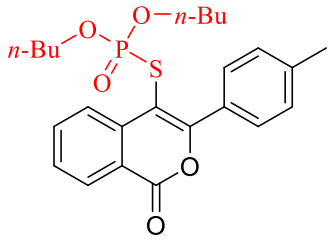
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 30.8 mg, 41%; Yellow Solid; m.p. 86.5-88.3 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.36 – 8.27 (m, 1H), 8.26 – 8.19 (m, 1H), 7.89 – 7.82 (m, 1H), 7.75 – 7.70 (m, 2H), 7.63 – 7.53 (m, 1H), 7.34 – 7.27 (m, 2H), 3.39 (s, 3H), 3.36 (s, 3H), 2.42 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.3, 160.7 (d, J = 8.3 Hz), 140.9, 138.5 (d, J = 1.4 Hz), 135.2, 130.5 (d, J = 1.8 Hz), 130.1 (d, J = 2.8 Hz), 129.9, 128.93, 128.91, 126.2, 120.7, 101.9 (d, J = 9.1 Hz), 54.2 (d, J = 6.5 Hz), 21.7. ^{31}P NMR (162 MHz, $CDCl_3$) δ 24.28. HRMS: $[M+H]^+$ *m/z* calcd for $C_{18}H_{18}O_5PS^+$: 377.0607, found: 377.0605.

O,O-diisopropyl *S*-(1-oxo-3-(*p*-tolyl)-1*H*-isochromen-4-yl) phosphorothioate (**4t**)



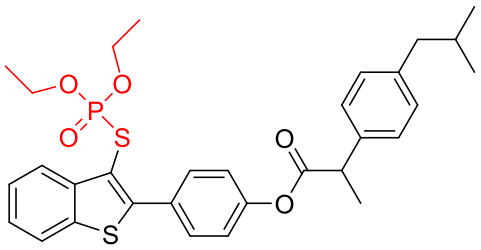
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 85.6 mg, 99%; Yellow Solid; m.p. 86.4-88.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 – 8.29 (m, 1H), 8.29 – 8.22 (m, 1H), 7.88 – 7.79 (m, 1H), 7.75 (d, J = 8.2 Hz, 2H), 7.64 – 7.52 (m, 1H), 7.26 (d, J = 8.0 Hz, 2H), 4.53 – 4.33 (m, 2H), 2.41 (s, 3H), 1.17 (d, J = 6.2 Hz, 6H), 1.06 (d, J = 6.2 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.5, 160.5 (d, J = 8.8 Hz), 140.5, 138.7 (d, J = 1.4 Hz), 135.0, 130.4 (d, J = 1.7 Hz), 130.3 (d, J = 2.8 Hz), 129.7, 128.8, 128.7, 126.9, 120.7, 103.2 (d, J = 8.9 Hz), 73.8 (d, J = 7.5 Hz), 23.9 (d, J = 4.1 Hz), 23.6 (d, J = 5.8 Hz), 21.6. ^{31}P NMR (162 MHz, $CDCl_3$) δ 19.44, 19.44. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{22}H_{25}NaO_5PS^+$: 455.1053, found: 455.1050.

O,O-dibutyl *S*-(1-oxo-3-(*p*-tolyl)-1*H*-isochromen-4-yl) phosphorothioate (**4u**)



Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 87.4 mg, 95%; Yellow Oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.36 – 8.23 (m, 1H), 8.20 – 8.12 (m, 1H), 7.86 – 7.71 (m, 1H), 7.68 (d, *J* = 8.3 Hz, 2H), 7.58 – 7.44 (m, 1H), 7.27 – 7.13 (m, 2H), 3.78 – 3.61 (m, 2H), 3.55 – 3.38 (m, 2H), 2.35 (s, 3H), 1.41 – 1.21 (m, 4H), 1.16 – 1.00 (m, 4H), 0.74 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.4, 160.4 (d, *J* = 8.4 Hz), 140.7, 138.7 (d, *J* = 1.4 Hz), 135.0, 130.5 (d, *J* = 1.5 Hz), 130.2 (d, *J* = 2.7 Hz), 129.7, 128.8 (2C, overlap), 126.6, 120.7, 102.5 (d, *J* = 9.1 Hz), 67.9 (d, *J* = 7.2 Hz), 32.1 (d, *J* = 7.1 Hz), 21.6, 18.6, 13.7. ^{31}P NMR (162 MHz, $CDCl_3$) δ 21.14. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{24}H_{29}NaO_5PS^+$: 483.1366, found: 483.1349.

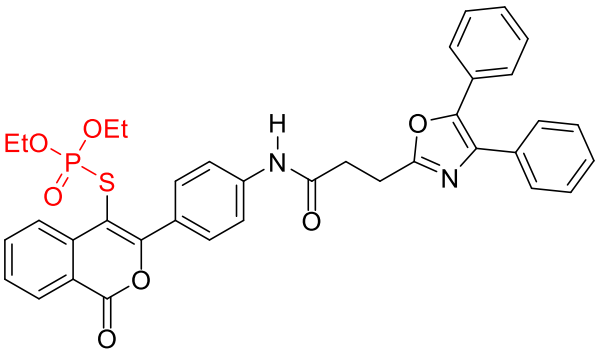
4-(3-((Diethoxyphosphoryl)thio)benzo[*b*]thiophen-2-yl)phenyl 2-(4-isobutylphenyl)propanoate (**5a**)



Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 79.2 mg, 68%; Yellow Oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (d, *J* = 8.1 Hz, 1H), 7.86 – 7.71 (m, 3H), 7.53 – 7.44 (m, 1H), 7.43 – 7.36 (m, 1H), 7.32 (d, *J* = 8.1 Hz, 2H), 7.22 – 7.08 (m, 4H), 4.05 – 3.86 (m, 3H), 3.80 – 3.67 (m, 2H), 2.49 (d, *J* = 7.2 Hz, 2H), 1.96 – 1.82 (m, 1H), 1.63 (d, *J* = 7.1 Hz, 3H), 1.06 (t, *J* = 7.1 Hz, 6H), 0.93 (d, *J* = 6.6 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.2, 151.6, 149.0 (d, *J* = 8.0 Hz), 141.5 (d, *J* = 1.8 Hz), 141.1, 138.0 (d, *J* = 1.2 Hz), 137.2, 131.4 (d, *J* = 1.5 Hz), 131.0 (d, *J* = 2.5 Hz), 129.7, 127.3, 125.5, 125.1, 124.3, 122.2, 121.8, 112.5 (d, *J* = 8.9 Hz), 64.3 (d, *J* = 6.7 Hz), 45.3 (d, *J* = 22.0 Hz), 30.3, 22.5, 18.7, 16.0 (d, *J* = 7.2 Hz). ^{31}P

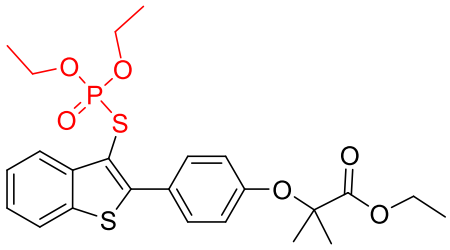
NMR (162 MHz, $CDCl_3$) δ 20.93. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{31}H_{35}NaO_5PS_2^+$: 605.1556, found: 605.1550.

S-(3-(4-(3-(4,5-diphenyloxazol-2-yl)propanamido)phenyl)-1-oxo-1*H*-isochromen-4-yl) *O,O*-diethyl phosphorothioate (**5b**)



Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 46.2 mg, 68%; Yellow Solid; m.p. 184.7-186.9 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.29 (d, *J* = 3.4 Hz, 1H), 8.42 – 8.28 (m, 1H), 8.22 (d, *J* = 8.1 Hz, 1H), 7.87 – 7.73 (m, 3H), 7.69 (d, *J* = 8.5 Hz, 2H), 7.64 – 7.60 (m, 2H), 7.58 – 7.47 (m, 3H), 7.42 – 7.28 (m, 6H), 3.98 – 3.78 (m, 2H), 3.76 – 3.59 (m, 2H), 3.28 (t, *J* = 7.0 Hz, 2H), 2.99 (t, *J* = 7.1 Hz, 2H), 1.03 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.4, 162.6, 161.6, 159.8 (d, *J* = 8.3 Hz), 145.8, 140.5, 138.7, 135.2, 135.0, 132.4, 131.4, 129.8, 128.9, 128.9, 128.8, 128.8, 128.4, 128.0, 127.9 (d, *J* = 2.6 Hz), 126.6, 126.5, 120.5, 118.7, 102.3 (d, *J* = 9.1 Hz), 64.5 (d, *J* = 6.8 Hz), 34.2, 24.1, 16.0 (d, *J* = 7.4 Hz). HRMS: $[M+H]^+$ *m/z* calcd for $C_{37}H_{34}N_2O_7PS^+$: 681.1819, found: 681.1802.

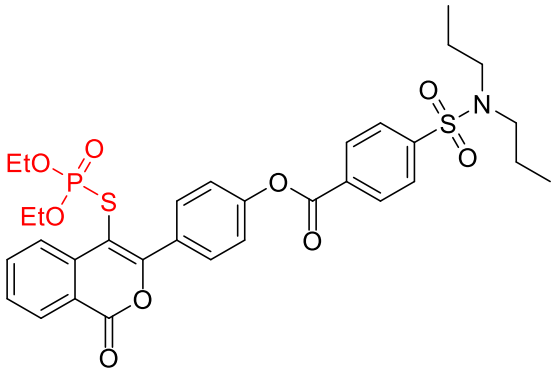
Mthyl 2-(4-(3-((diethoxyphosphoryl)thio)benzo[*b*]thiophen-2-yl)phenoxy)-2-methylpropanoate (**5c**)



Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 93.5 mg, 92%; Yellow Solid; m.p. 64.5-66.3 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 8.1 Hz, 1H), 7.78 (d, *J* = 8.0 Hz, 1H), 7.69 (d, *J* = 8.8 Hz, 2H), 7.49 – 7.41 (m, 1H), 7.40 – 7.30 (m, 1H), 6.91 (d, *J* = 8.7 Hz, 2H), 4.25 (q, *J* = 7.1 Hz, 2H),

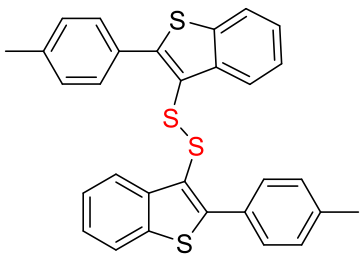
3.99 – 3.82 (m, 2H), 3.79 – 3.64 (m, 2H), 1.63 (s, 6H), 1.26 (t, J = 7.1 Hz, 3H), 1.03 (t, J = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.0, 156.3, 149.8 (d, J = 8.0 Hz), 141.7 (d, J = 1.8 Hz), 137.8 (d, J = 1.2 Hz), 131.3 (d, J = 1.5 Hz), 126.9 (d, J = 2.6 Hz), 125.2, 125.0, 124.1, 122.1, 118.6, 111.3 (d, J = 8.9 Hz), 79.3, 64.1 (d, J = 6.6 Hz), 61.7, 25.5, 16.0 (d, J = 7.2 Hz), 14.2. ^{31}P NMR (162 MHz, $CDCl_3$) δ 21.12. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{24}H_{29}NaO_6PS_2^+$: 531.1035, found: 531.1029.

4-(4-((Diethoxyphosphoryl)thio)-1-oxo-1*H*-isochromen-3-yl) phenyl 4-(*N,N*-dipropylsulfamoyl) benzoate (**5d**)



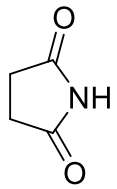
Purification by flash column chromatography(eluent/petroleum ether/EtOAc = 3:1); Yield: 37 mg, 55%; Yellow Solid; m.p. 130.5-132.7 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 – 8.29 (m, 3H), 8.26 (d, J = 8.1 Hz, 1H), 7.96 (d, J = 8.6 Hz, 4H), 7.91 – 7.82 (m, 1H), 7.72 – 7.54 (m, 1H), 7.40 – 7.30 (m, 2H), 3.99 – 3.81 (m, 2H), 3.80 – 3.63 (m, 2H), 3.28 – 2.86 (m, 4H), 1.62 – 1.44 (m, 4H), 1.11 (t, J = 7.1 Hz, 6H), 0.88 (t, J = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.7, 161.1, 159.3 (d, J = 8.4 Hz), 152.1, 145.3, 138.4, 135.2, 132.6, 132.0 (d, J = 1.7 Hz), 131.1 (d, J = 1.5 Hz), 131.0, 129.9, 129.2, 127.4, 126.6, 121.5, 120.8, 103.4 (d, J = 9.0 Hz), 64.5 (d, J = 6.7 Hz), 50.1, 22.1, 16.1 (d, J = 7.1 Hz), 11.4. ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.60. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{32}H_{36}NNaO_9PS_2^+$: 696.1461, found: 696.1454.

1,2-Bis(2-(p-tolyl)benzo[*b*]thiophen-3-yl)disulfane (**6**)



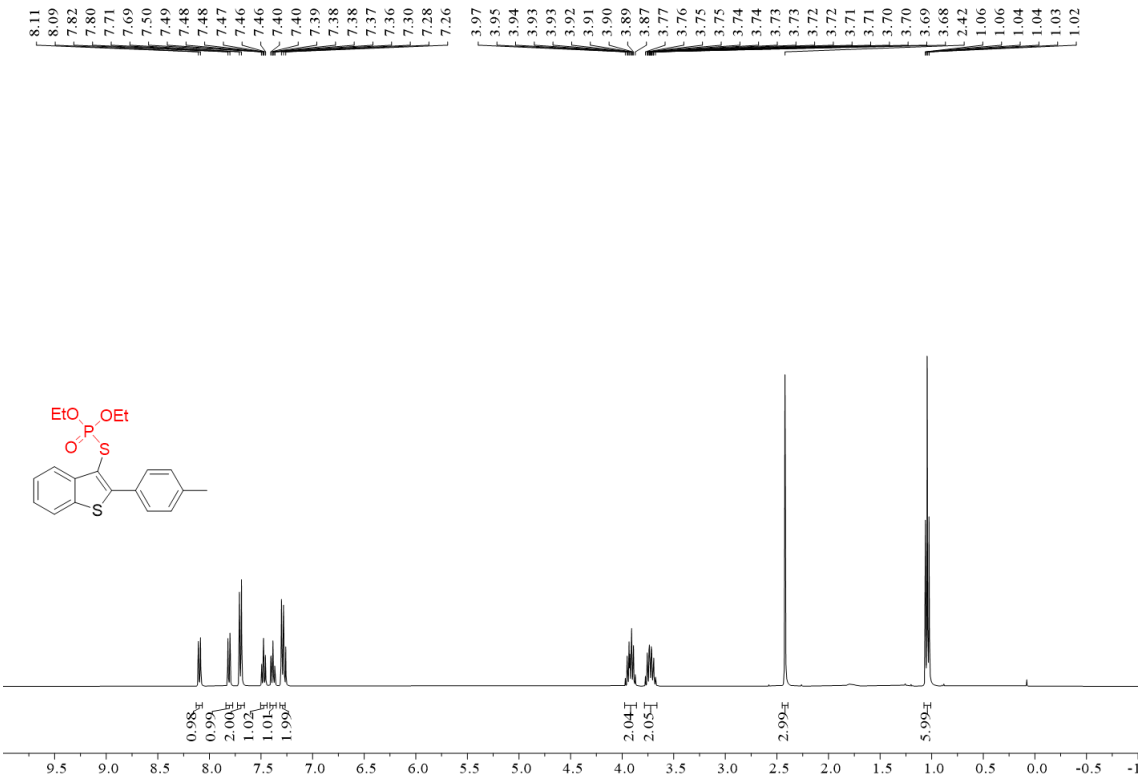
Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 3:1); Yield: 34.7 mg, 68%; Yellow Solid; m.p. 113.5-115.2 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.78 (m, 2H), 7.76 – 7.70 (m, 2H), 7.41 – 7.30 (m, 4H), 6.96 (d, *J* = 8.1 Hz, 4H), 6.78 (d, *J* = 8.0 Hz, 4H), 2.22 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.8, 140.7, 138.5, 138.2, 129.8, 129.5, 128.6, 125.0 (d, *J* = 3.8 Hz), 124.9, 123.8, 122.1, 121.4, 21.4. HRMS: $[M+Na]^+$ *m/z* calcd for $C_{30}H_{22}NaS_4^+$: 533.0497, found: 533.0488.

Pyrrolidine-2,5-dione (**7**)

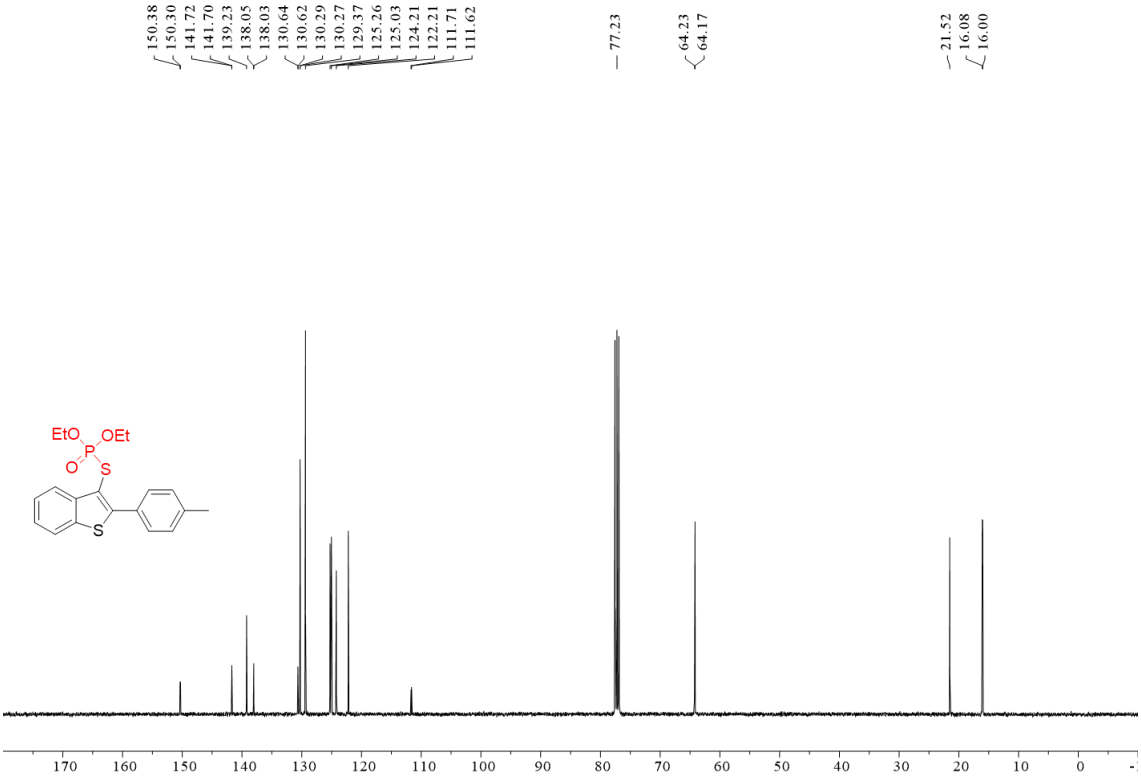


Purification by flash column chromatography (eluent/petroleum ether/EtOAc = 2:1); Yield: 29.7 mg, 75%; ^{1}H NMR (400 MHz, CDCl3) δ 8.78 (s, 1H), 2.75 (s, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.1, 29.8.

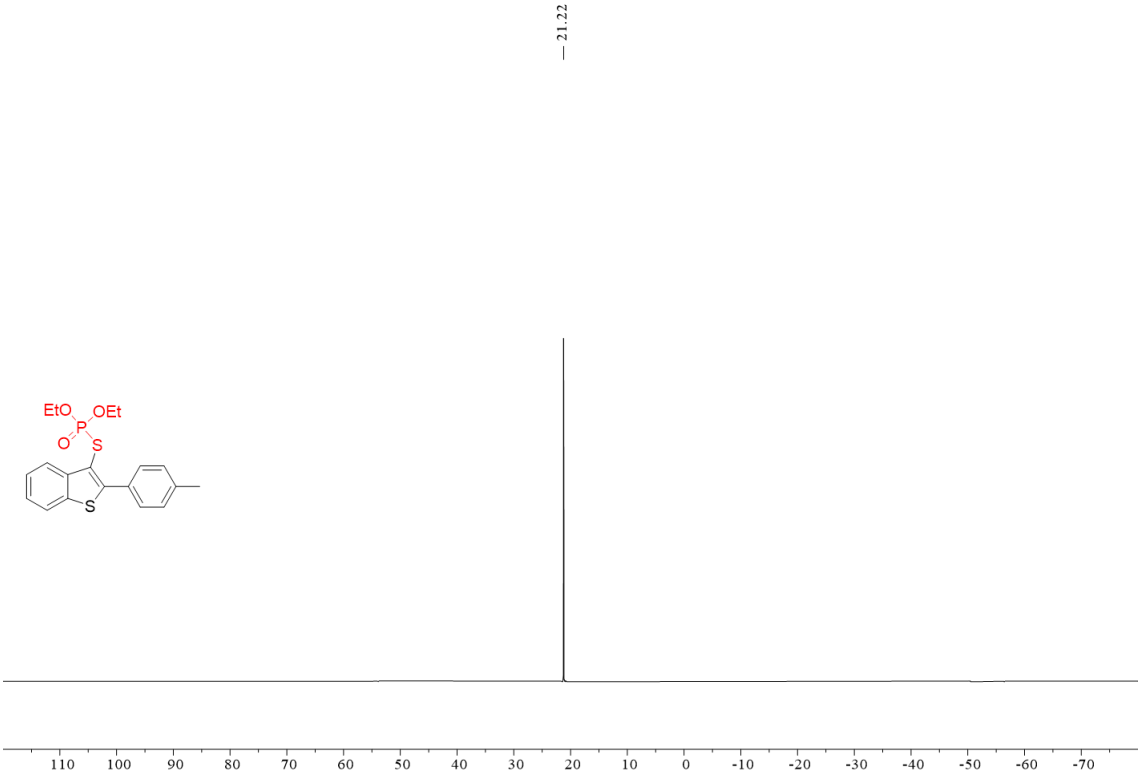
^{1}H NMR (400 MHz) Spectrum of **3a** in $CDCl_3$



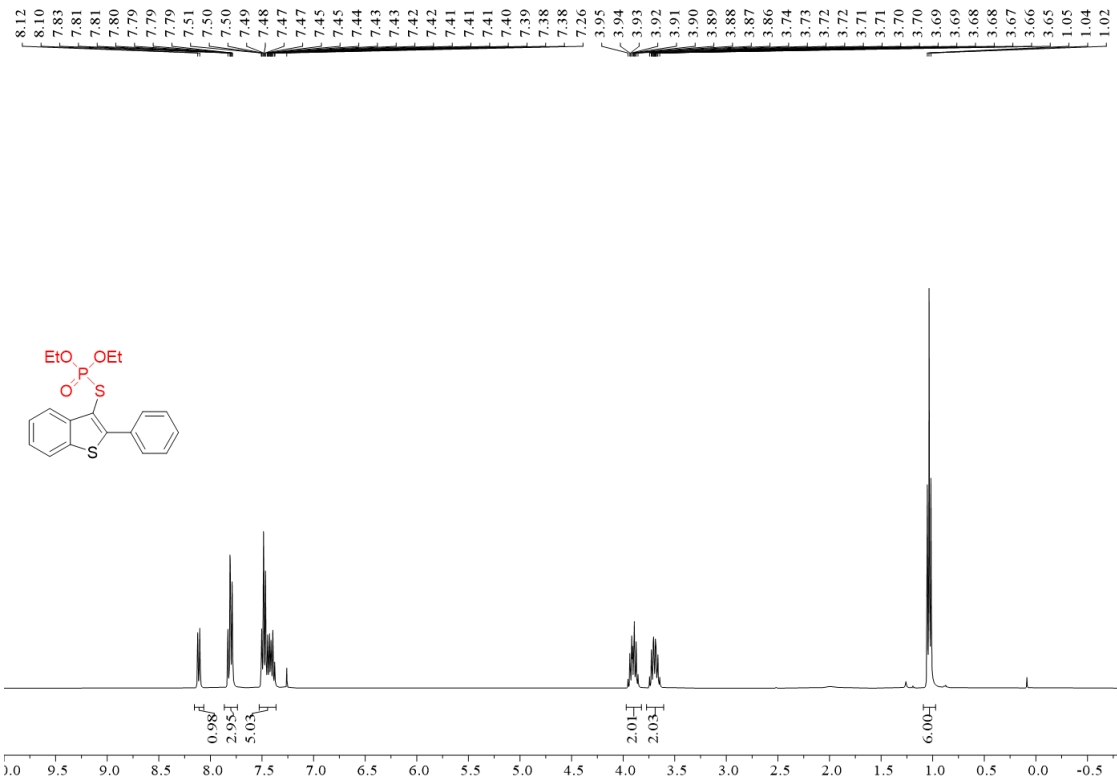
^{13}C NMR (101 MHz) Spectrum of **3a** in $CDCl_3$



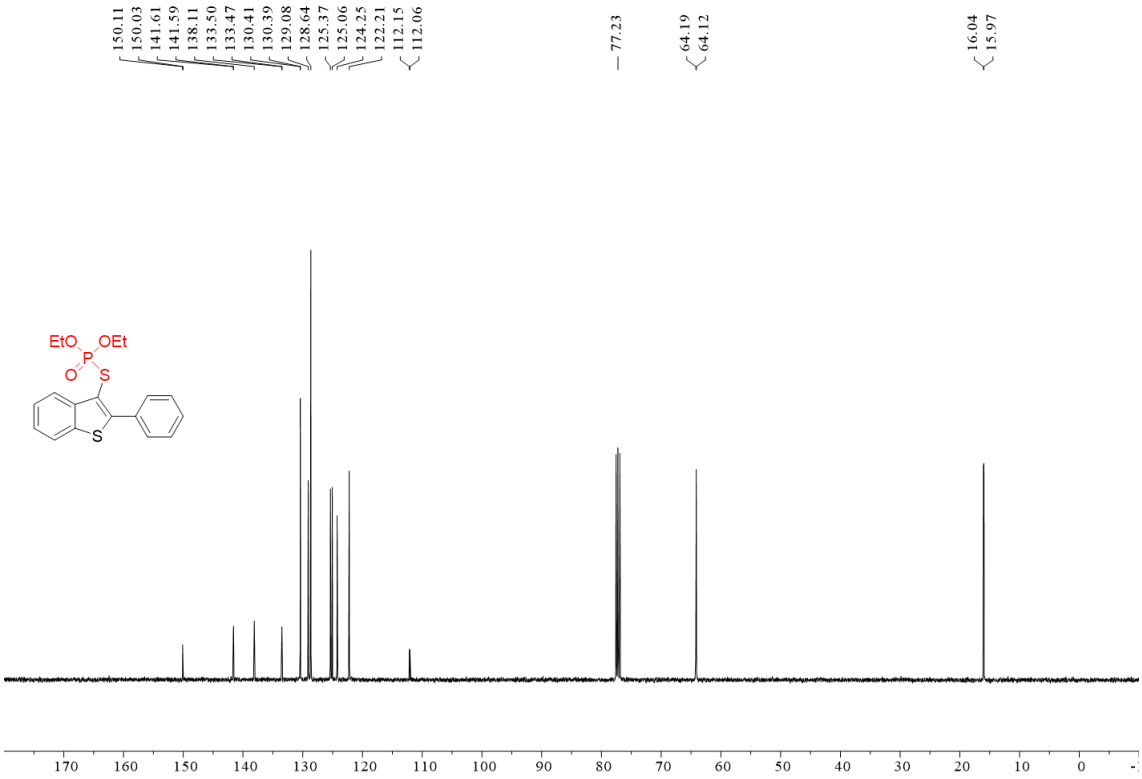
^{31}P NMR (162 MHz) Spectrum of **3a** in $CDCl_3$



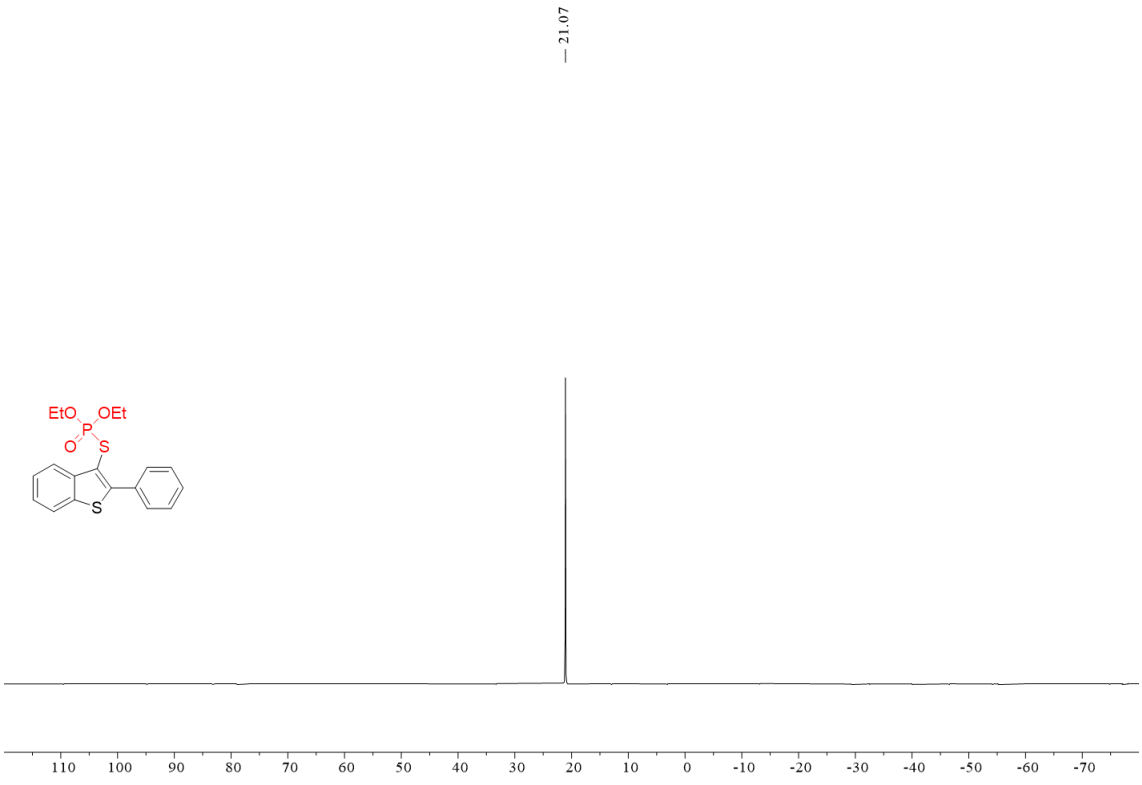
^{1}H NMR (400 MHz) Spectrum of **3b** in $CDCl_3$



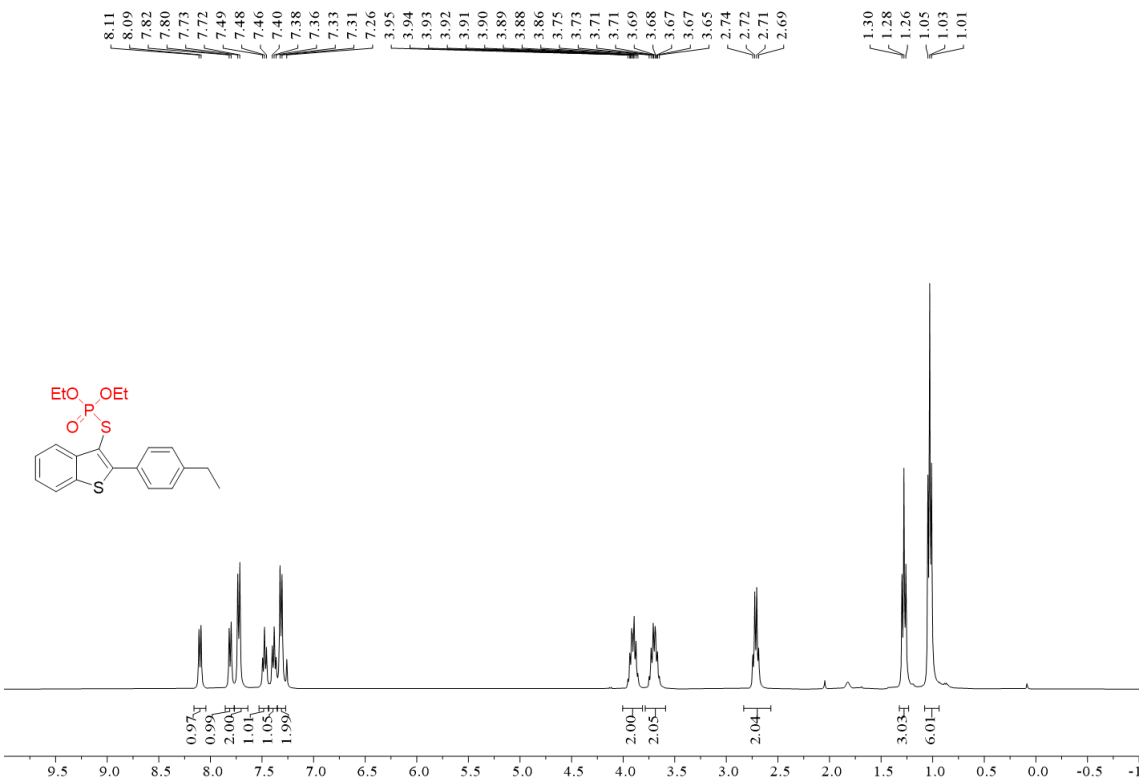
^{13}C NMR (101 MHz) Spectrum of **3b** in $CDCl_3$



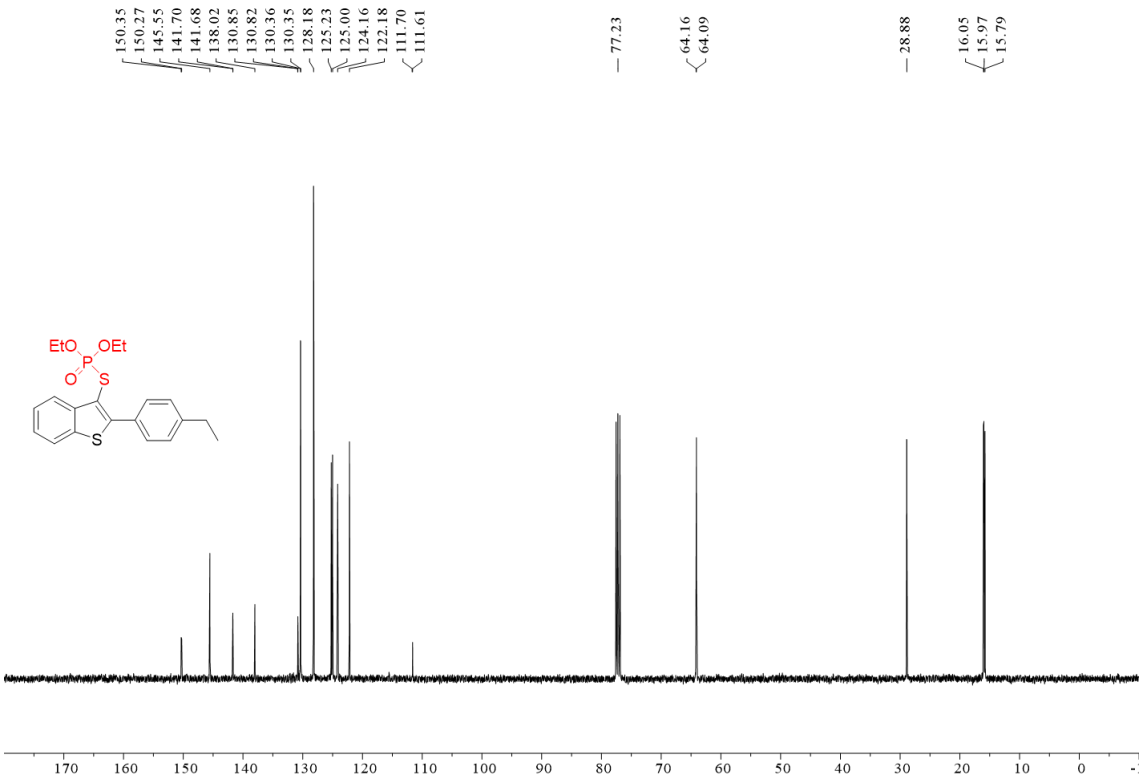
^{31}P NMR (162 MHz) Spectrum of **3b** in $CDCl_3$



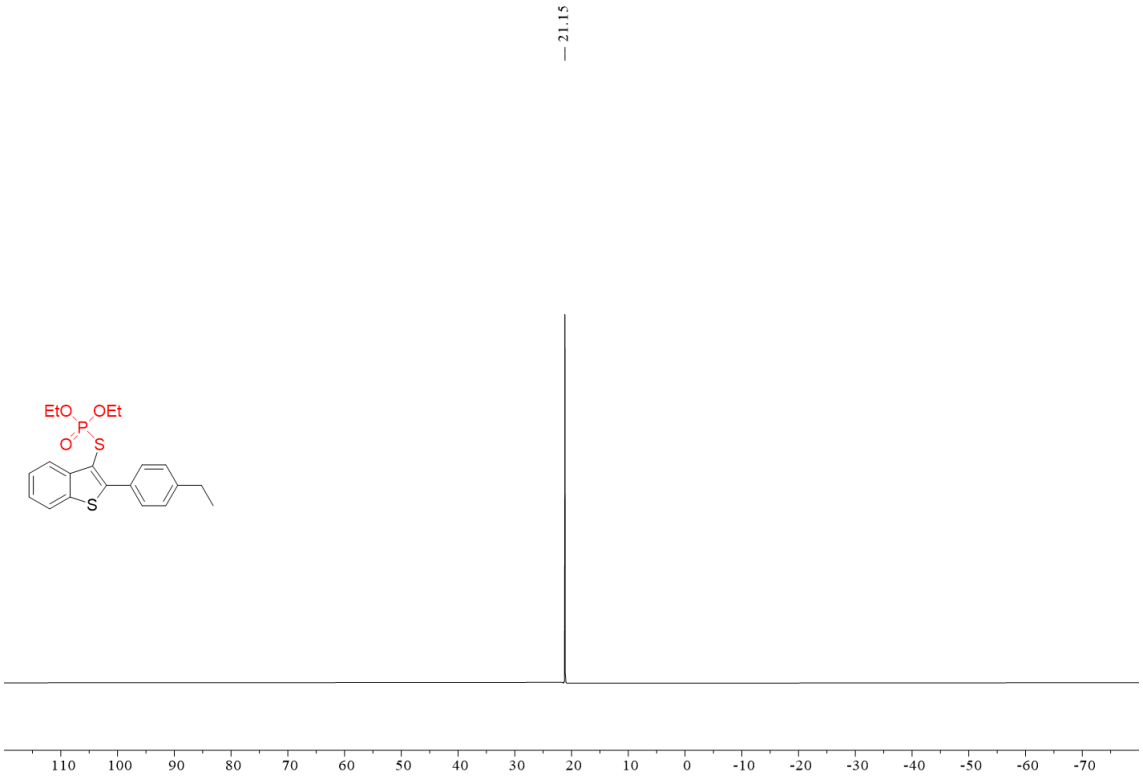
^{1}H NMR (400 MHz) Spectrum of **3c** in $CDCl_3$



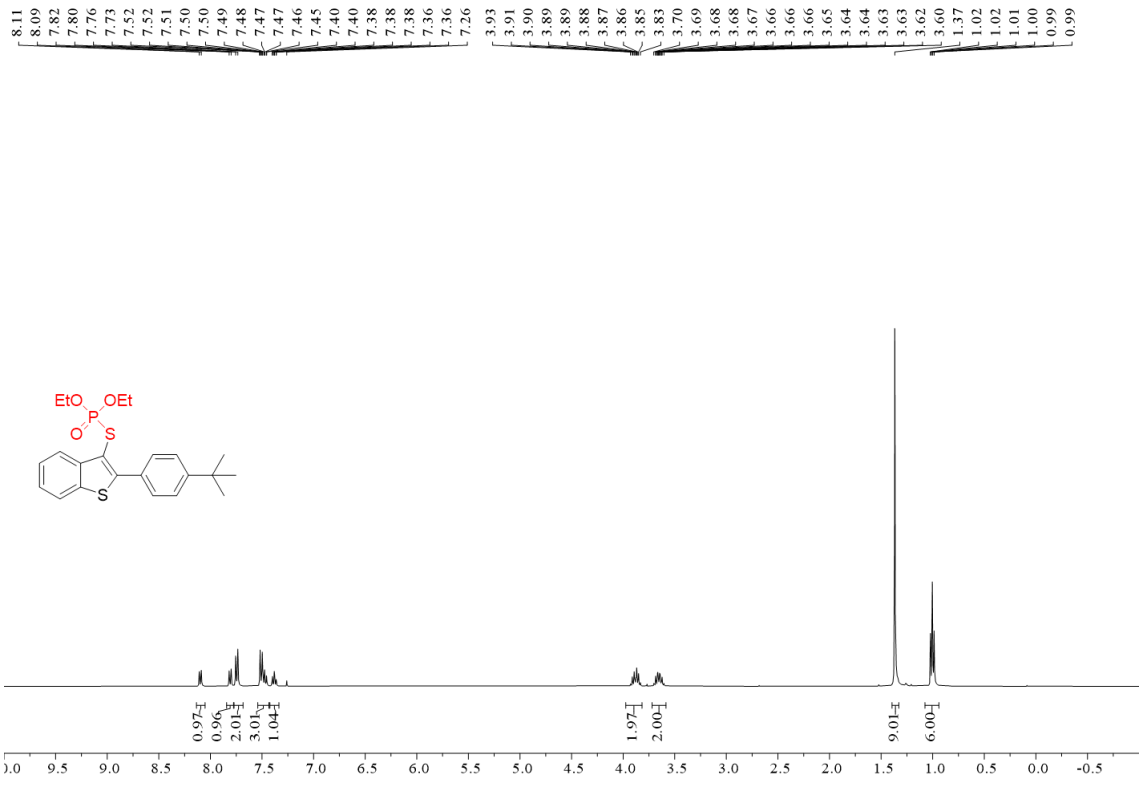
^{13}C NMR (101 MHz) Spectrum of **3c** in $CDCl_3$



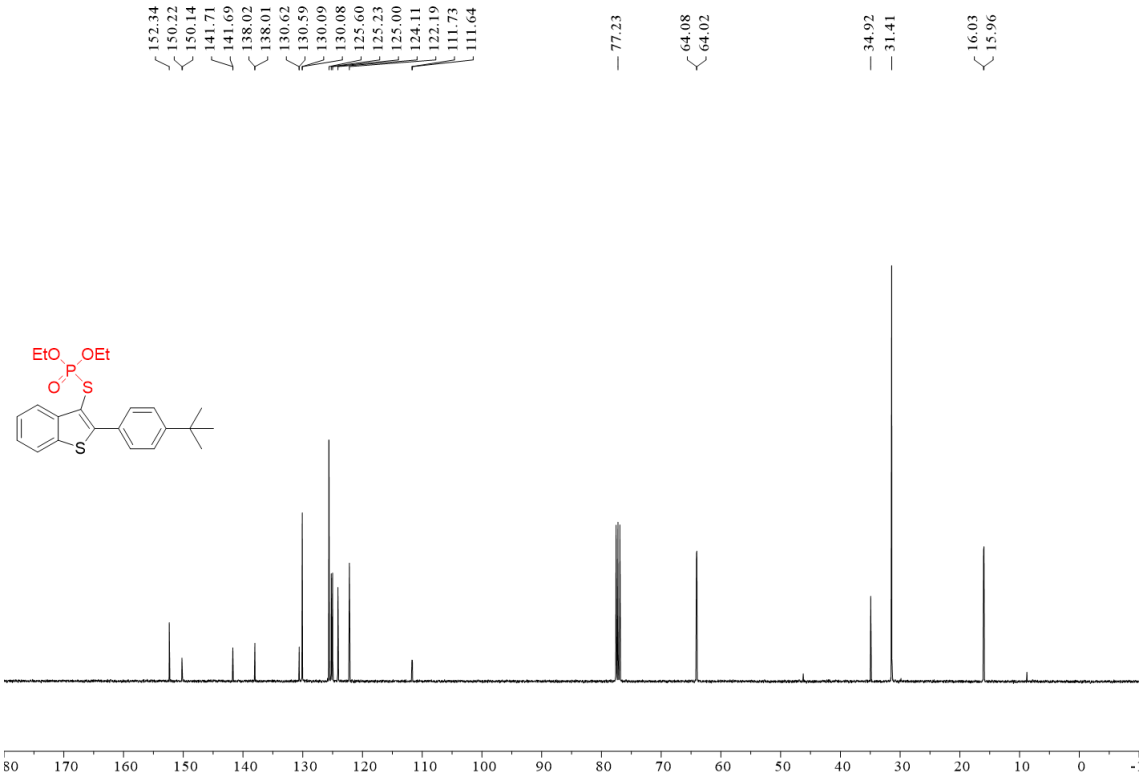
^{31}P NMR (162 MHz) Spectrum of **3c** in $CDCl_3$



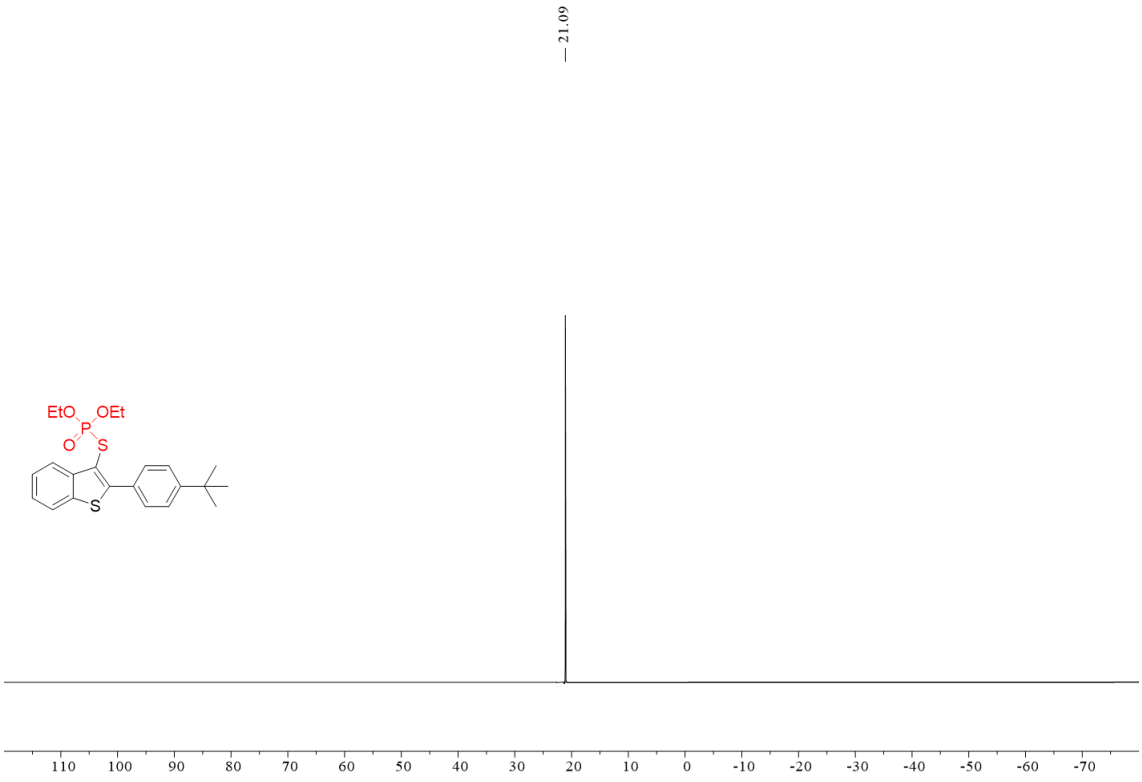
^{1}H NMR (400 MHz) Spectrum of **3d** in $CDCl_3$



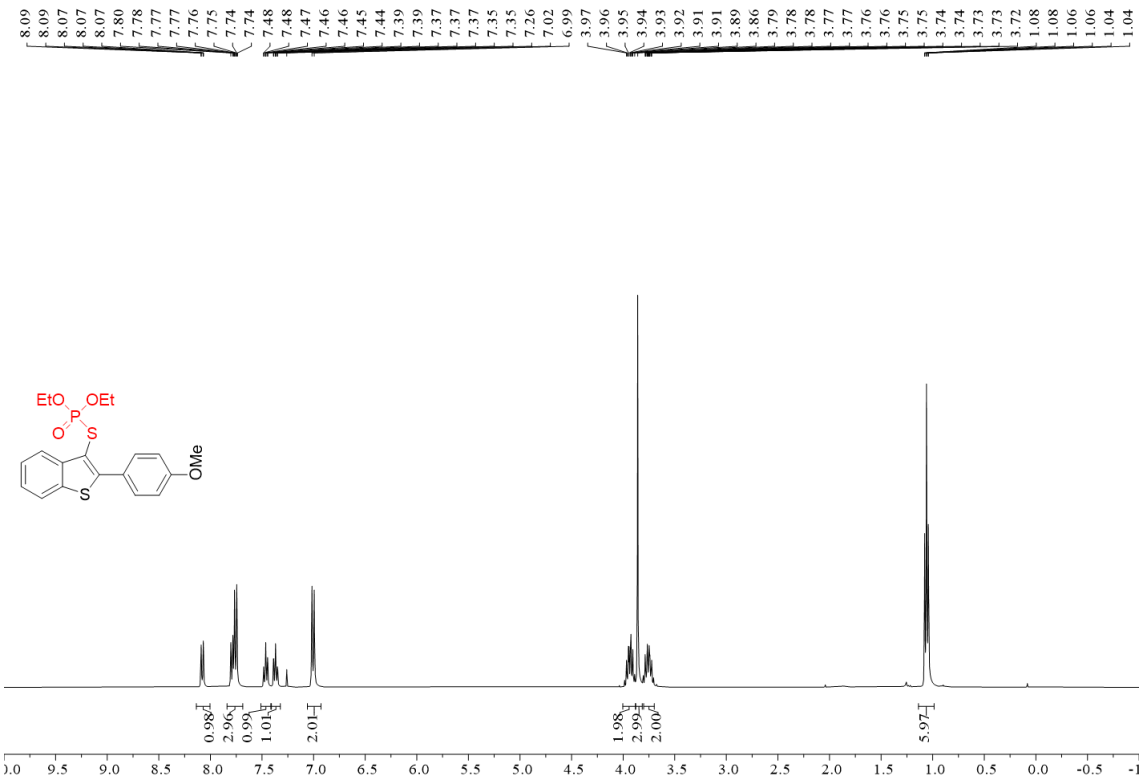
^{13}C NMR (101 MHz) Spectrum of **3d** in $CDCl_3$



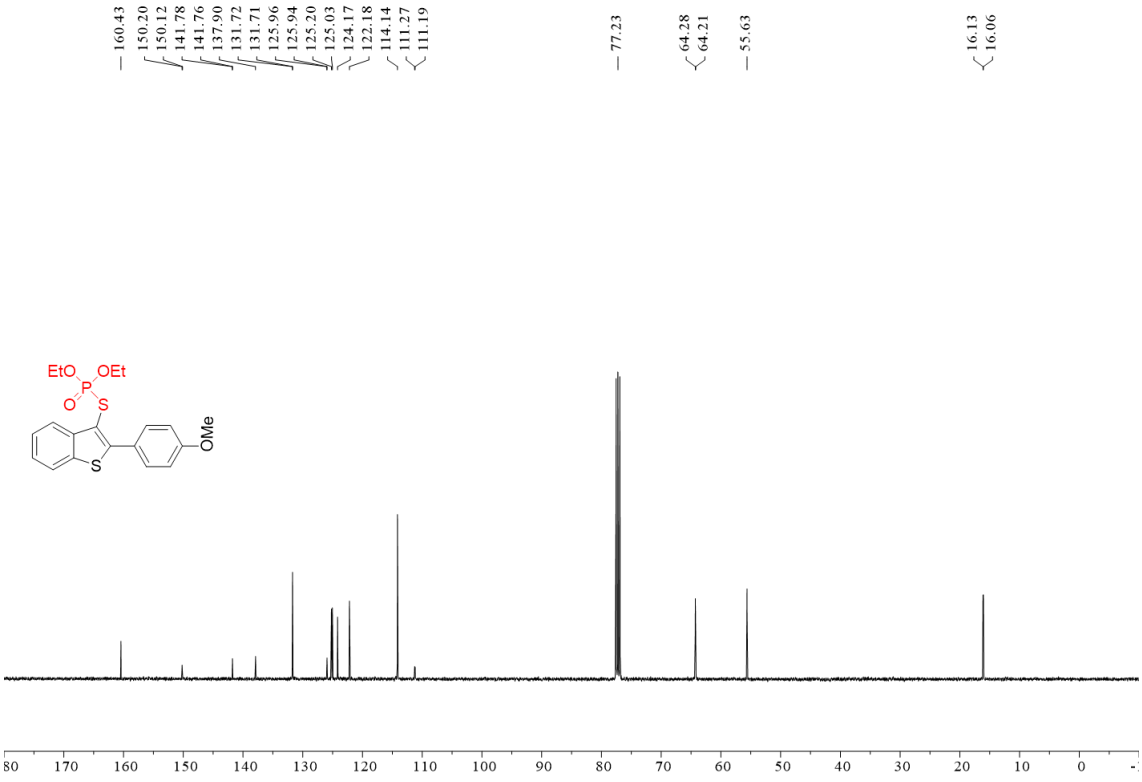
^{31}P NMR (162 MHz) Spectrum of **3d** in $CDCl_3$



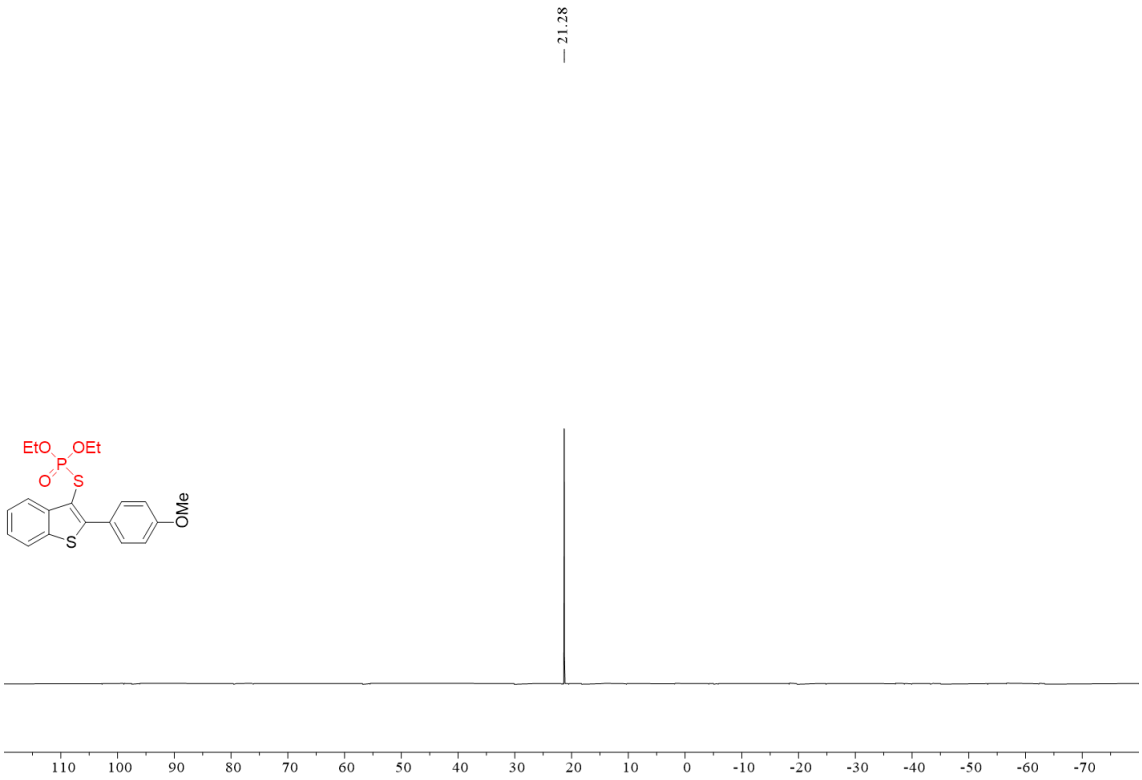
^{1}H NMR (400 MHz) Spectrum of **3e** in $CDCl_3$



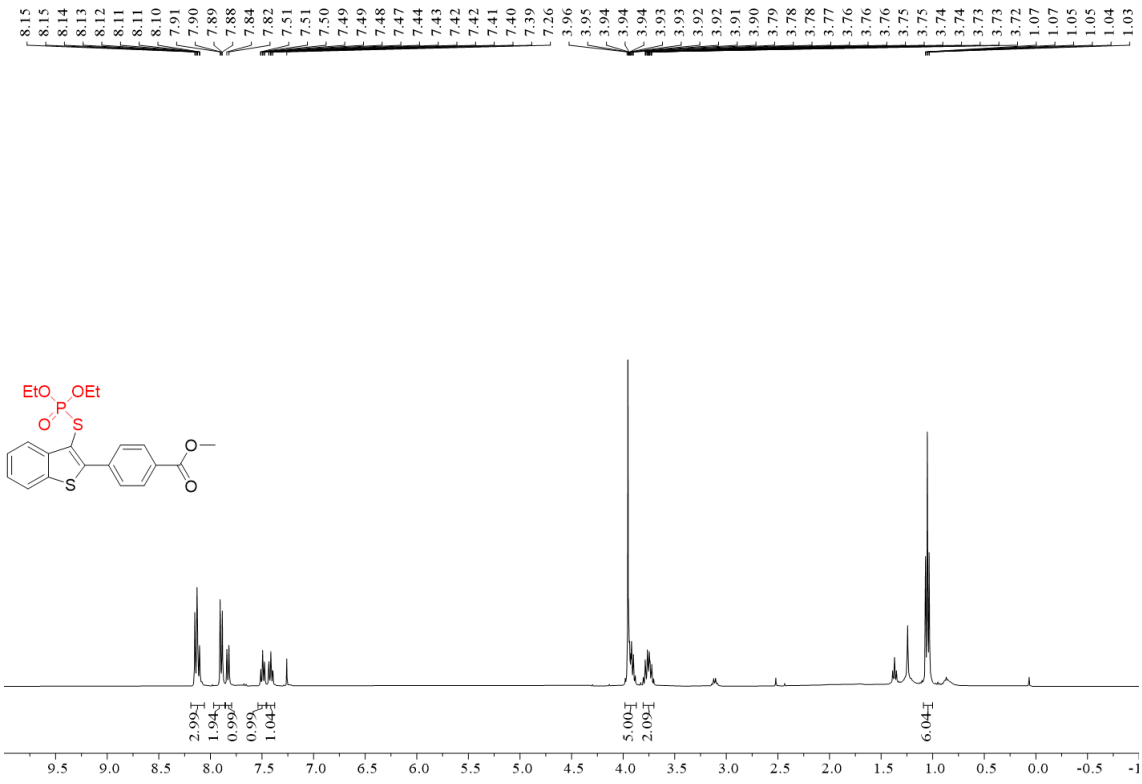
^{13}C NMR (101 MHz) Spectrum of **3e** in $CDCl_3$



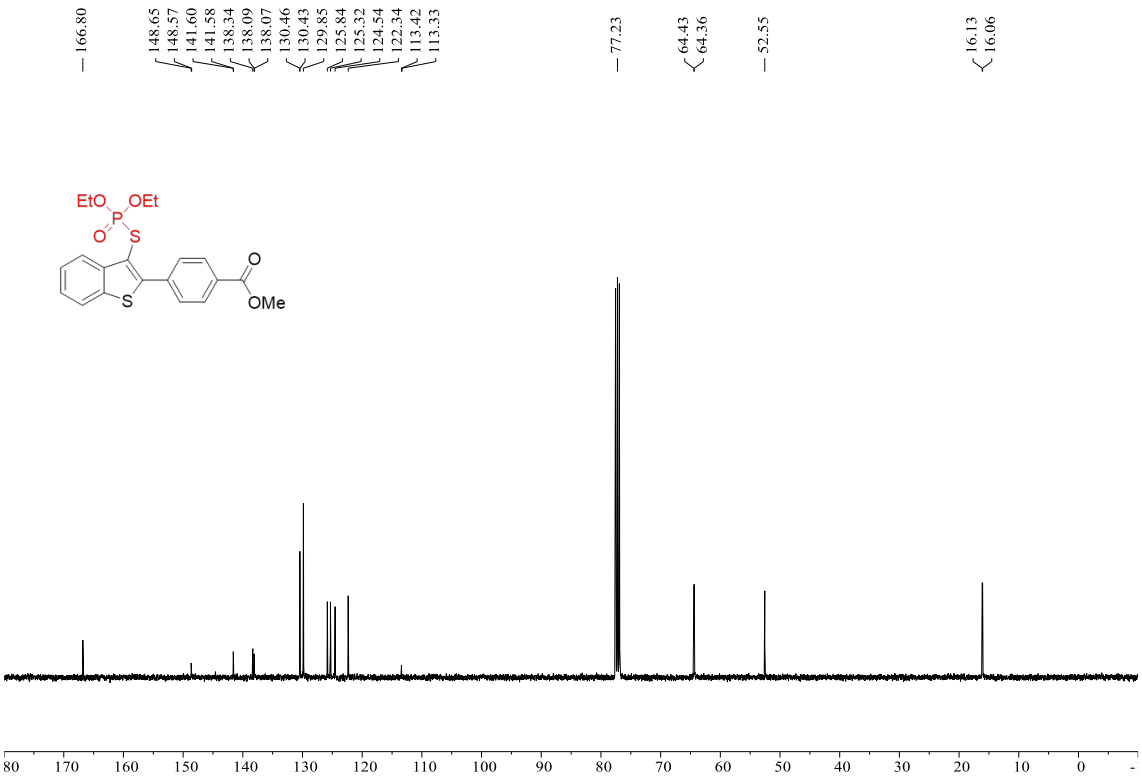
^{31}P NMR (162 MHz) Spectrum of **3e** in $CDCl_3$



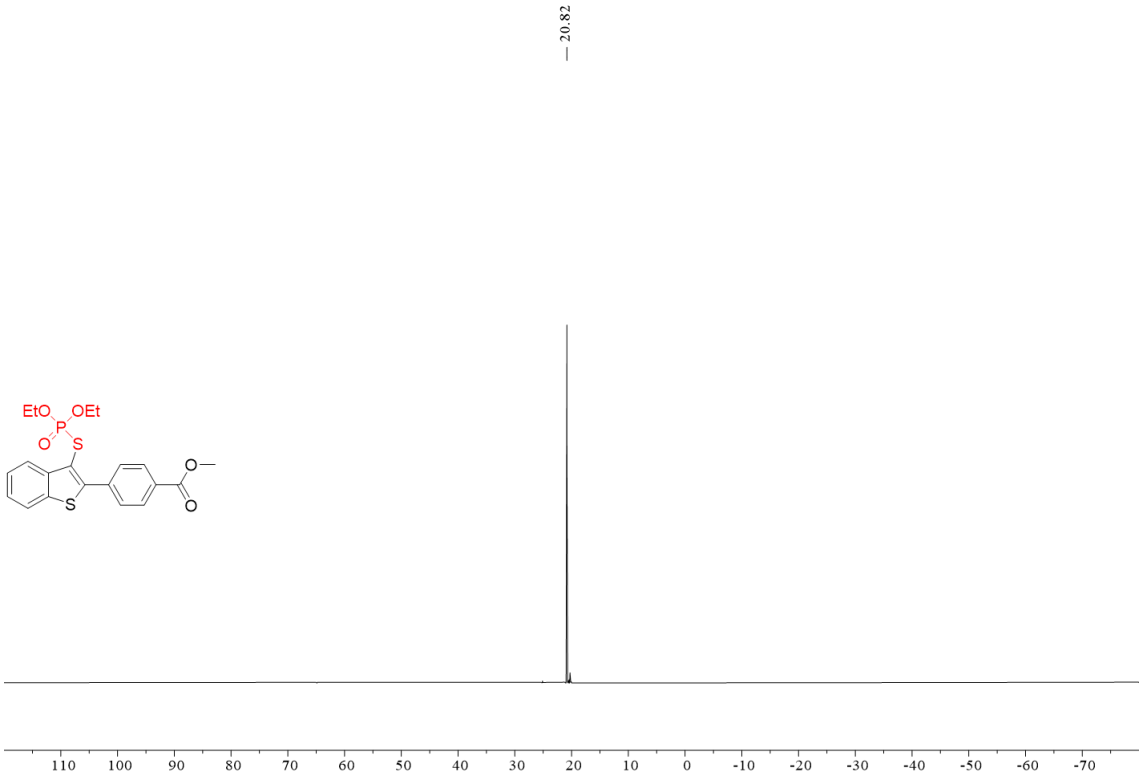
^{1}H NMR (400 MHz) Spectrum of **3f** in $CDCl_3$



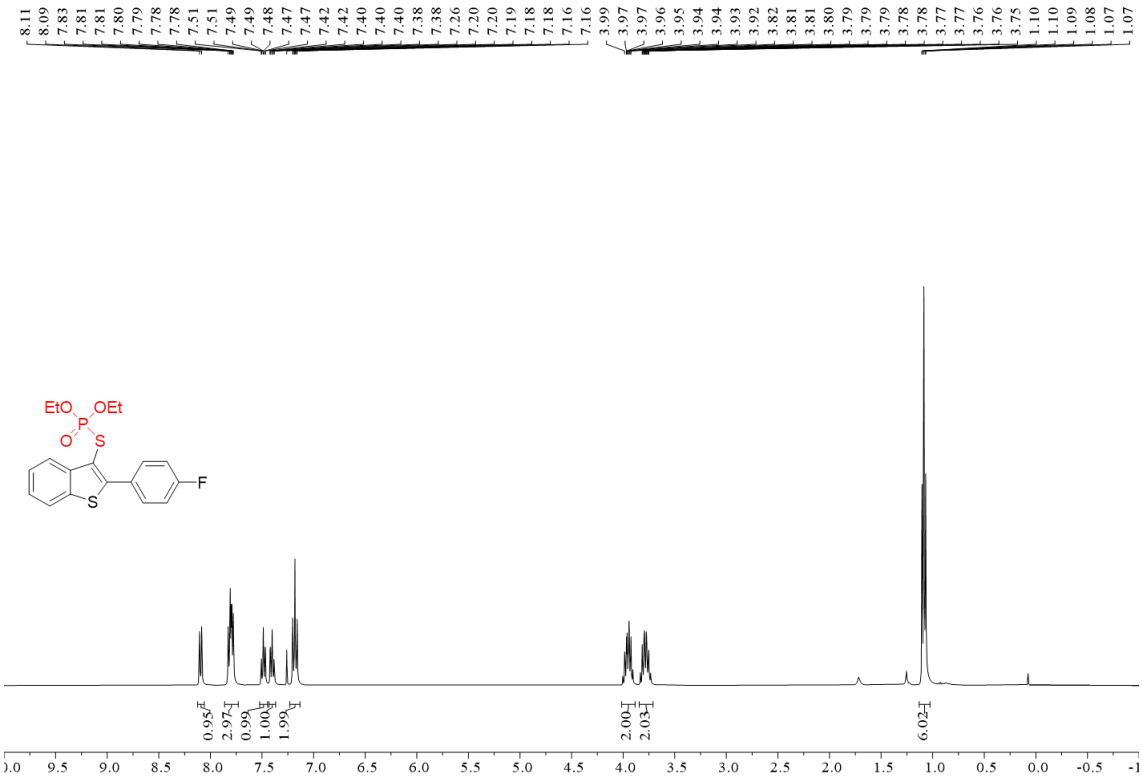
^{13}C NMR (101 MHz) Spectrum of **3f** in $CDCl_3$



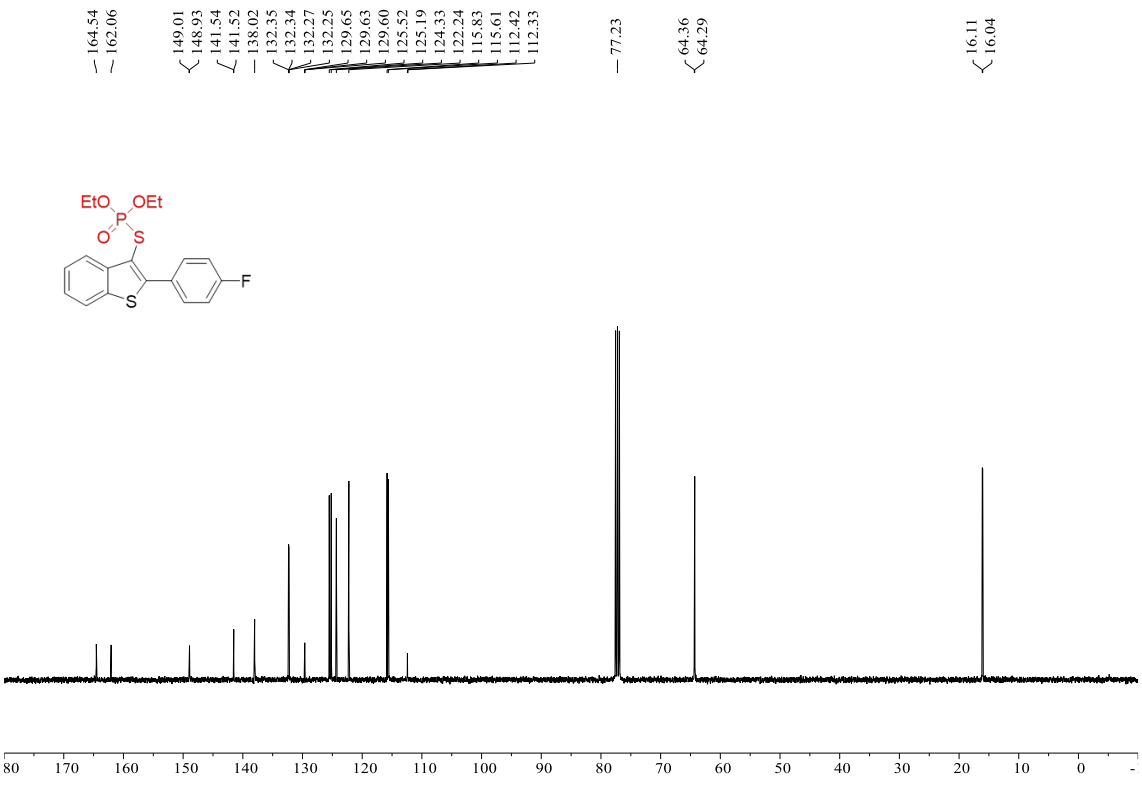
^{31}P NMR (162 MHz) Spectrum of **3f** in $CDCl_3$



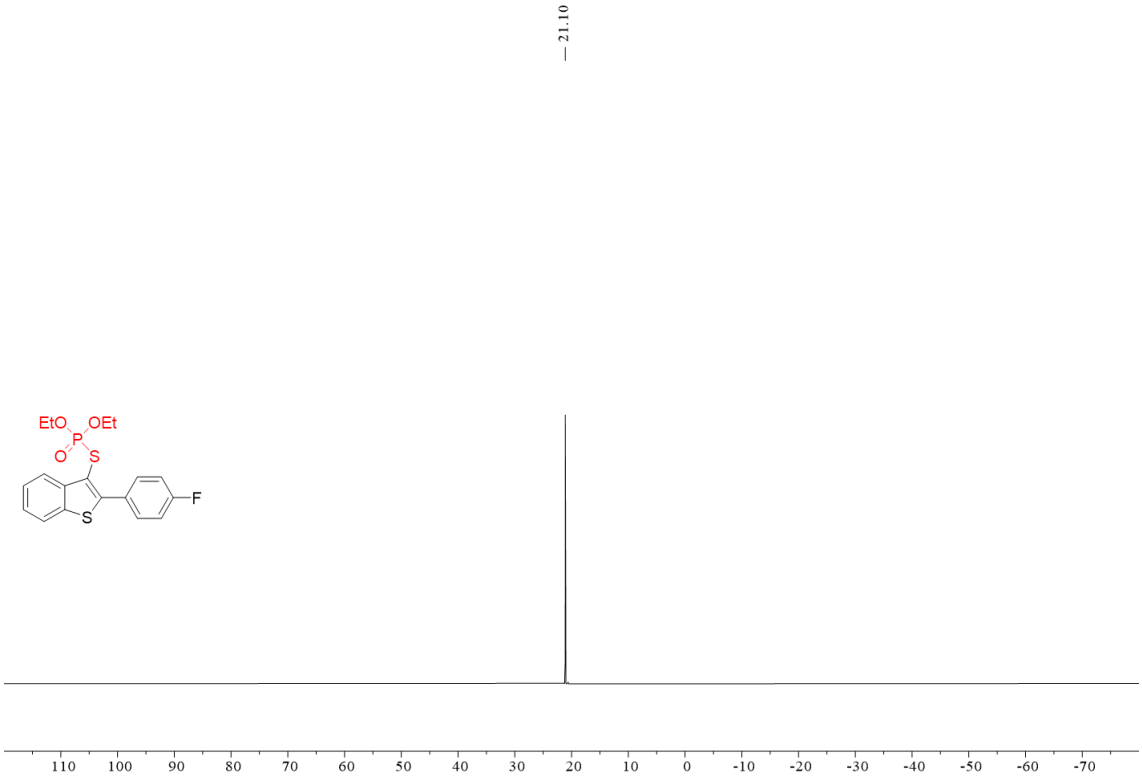
^{1}H NMR (400 MHz) Spectrum of **3g** in $CDCl_3$



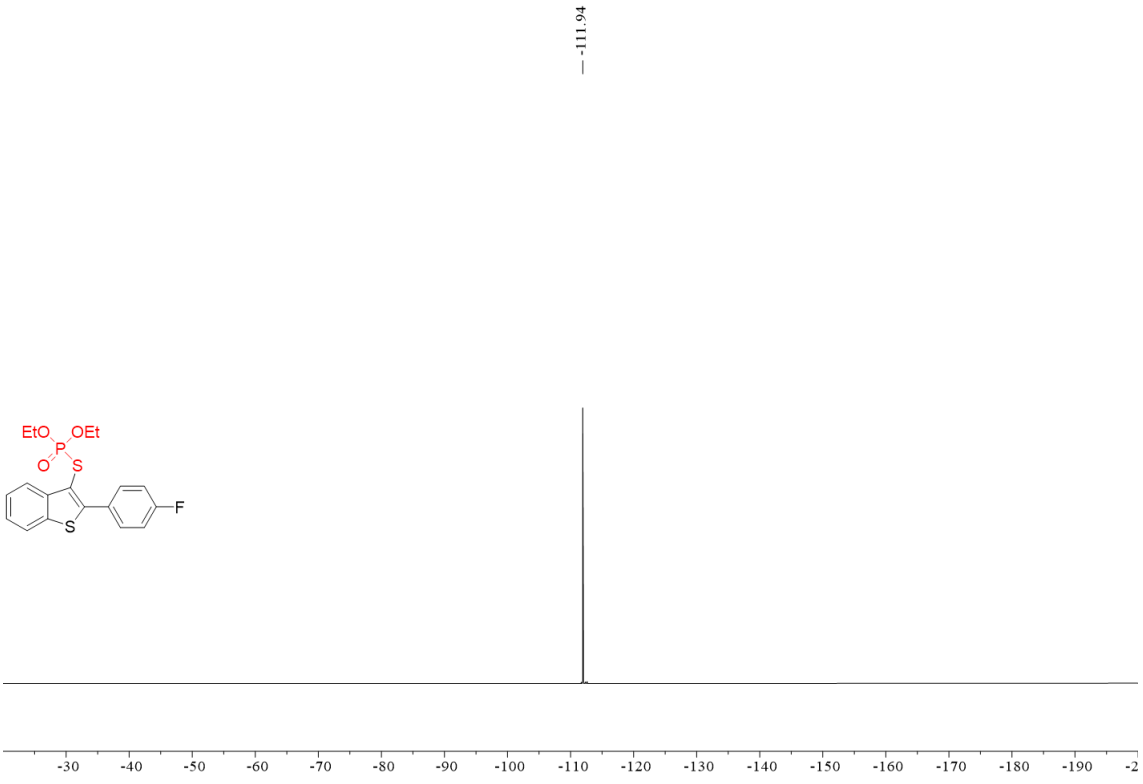
^{13}C NMR (101 MHz) Spectrum of **3g** in $CDCl_3$



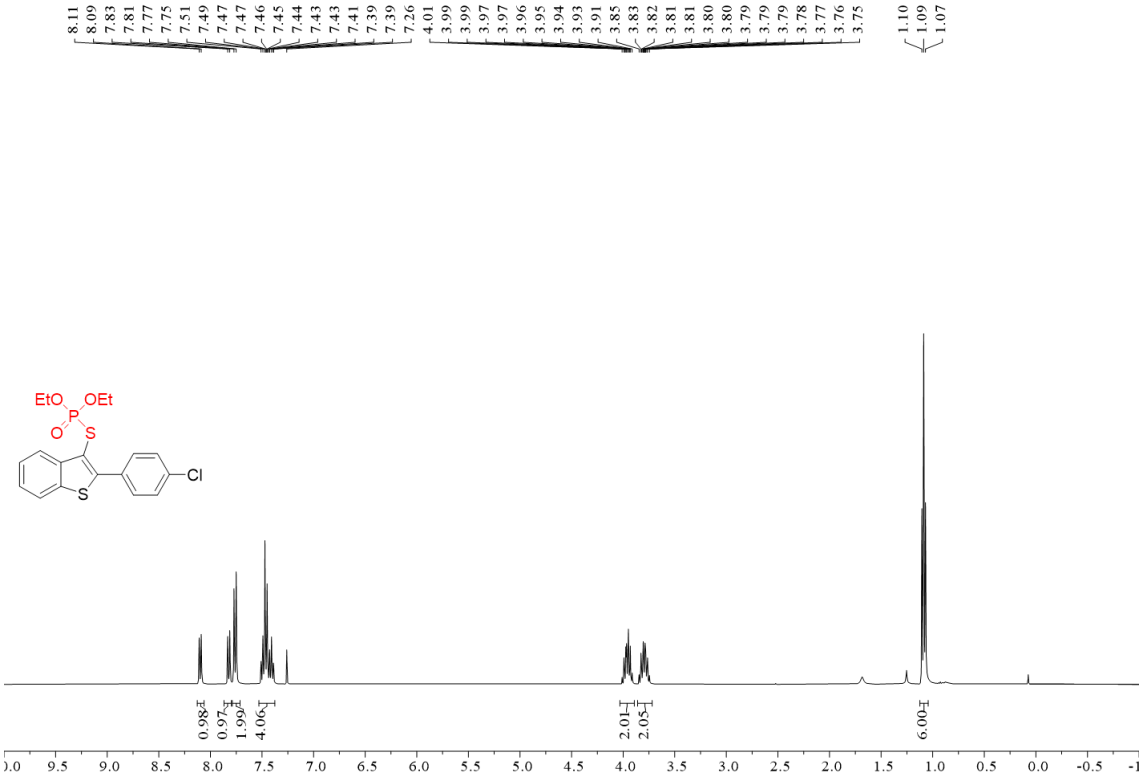
^{31}P NMR (162 MHz) Spectrum of **3g** in $CDCl_3$



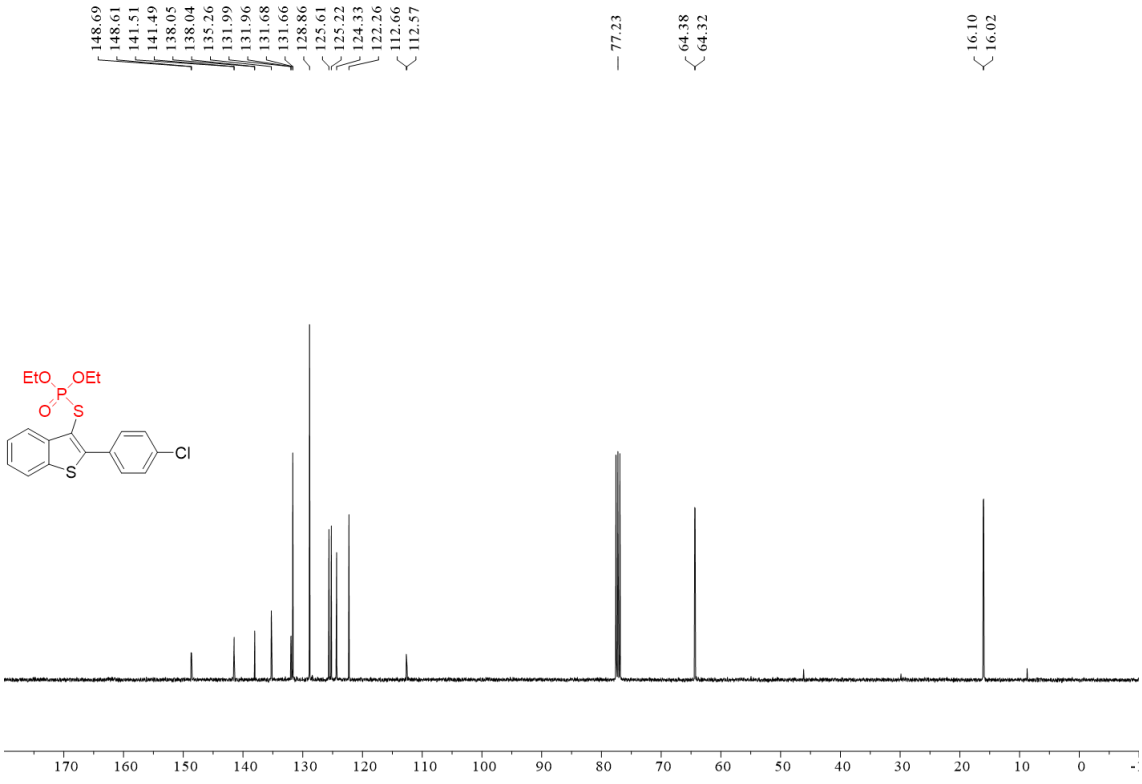
^{19}F NMR (376 MHz) Spectrum of **3g** in $CDCl_3$



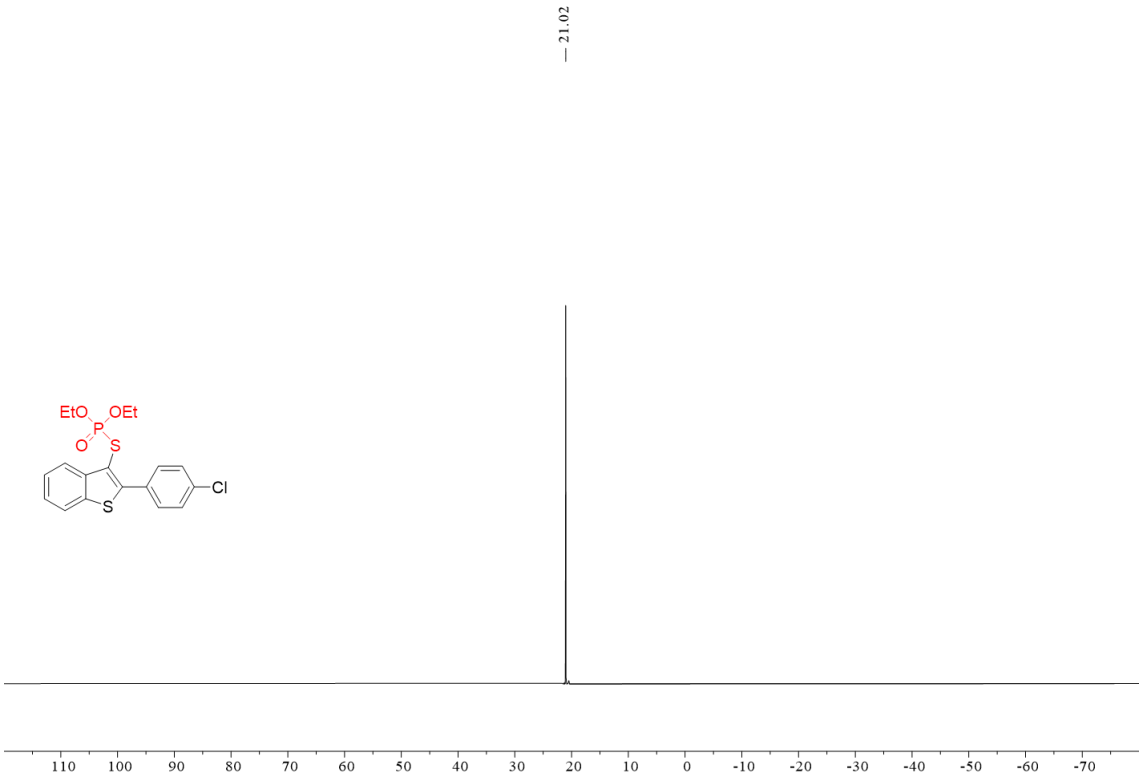
^{1}H NMR (400 MHz) Spectrum of **3h** in $CDCl_3$



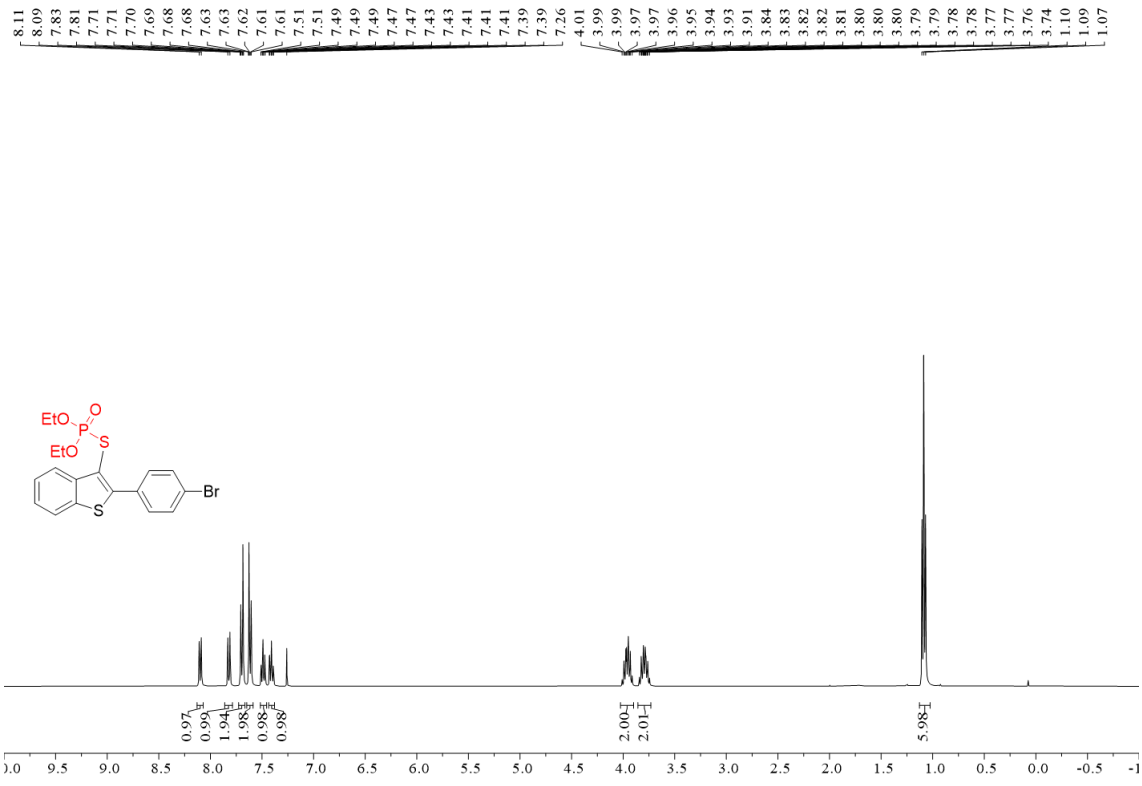
^{13}C NMR (101 MHz) Spectrum of **3h** in $CDCl_3$



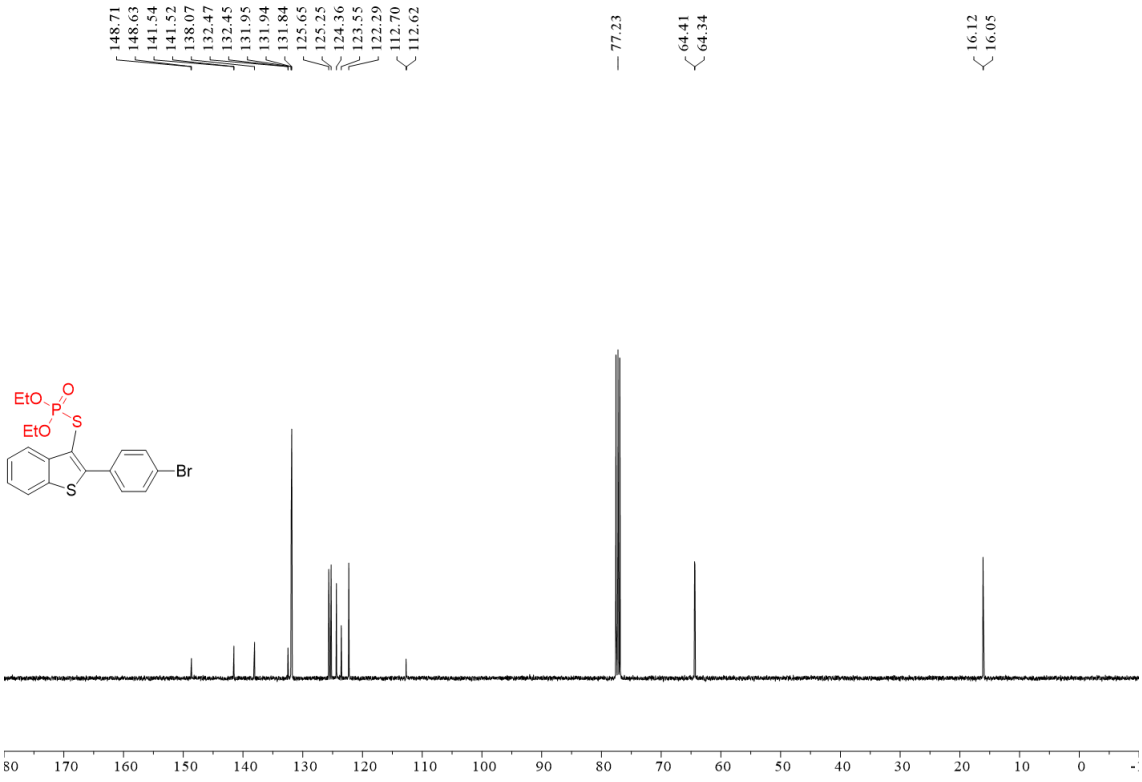
^{31}P NMR (162 MHz) Spectrum of **3h** in $CDCl_3$



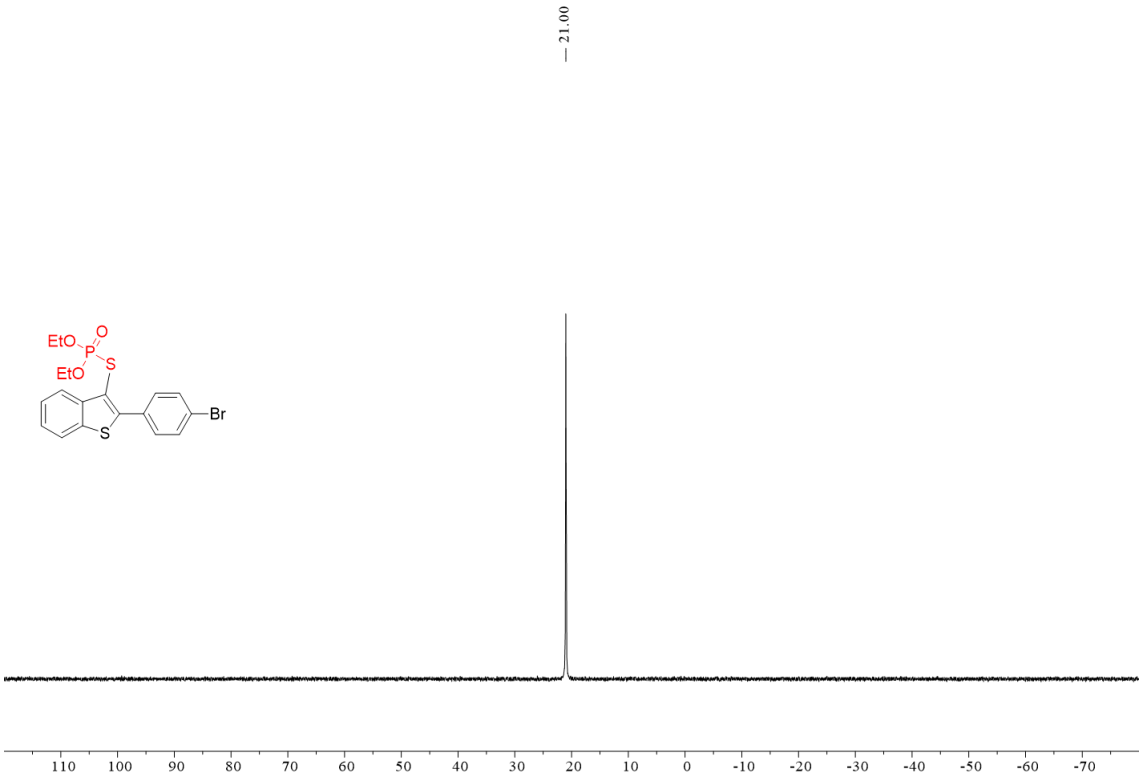
^{1}H NMR (400 MHz) Spectrum of **3i** in $CDCl_3$



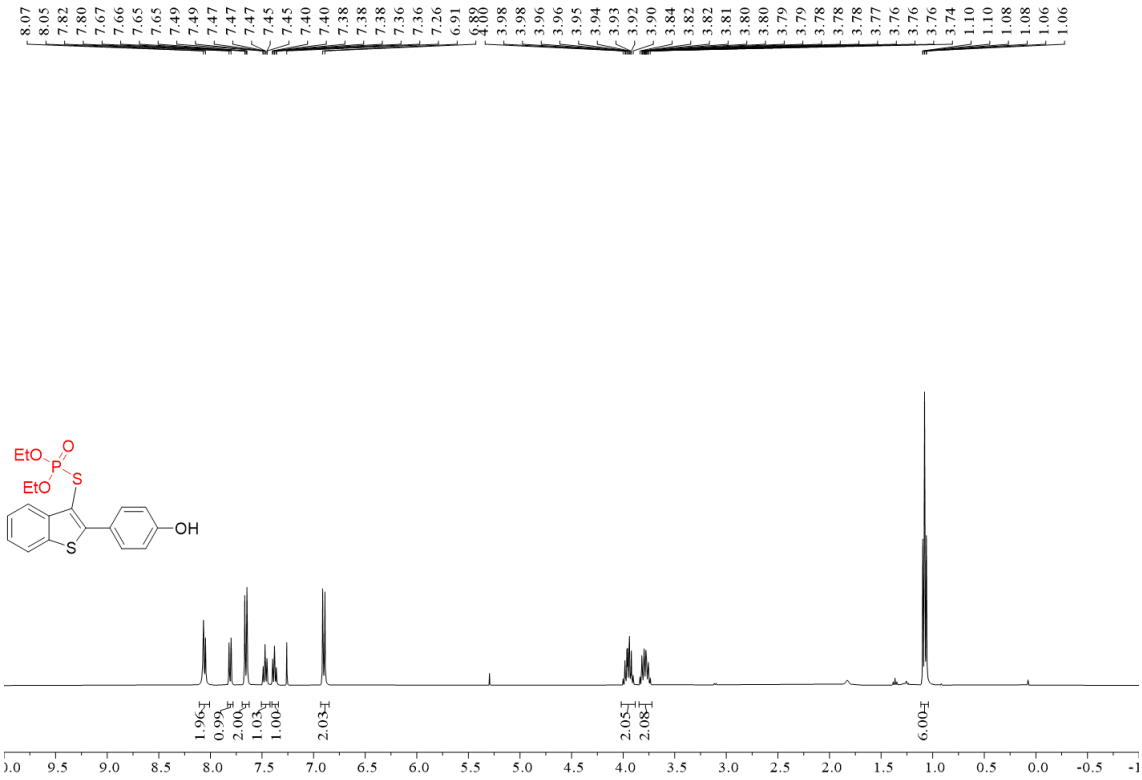
^{13}C NMR (101 MHz) Spectrum of **3i** in $CDCl_3$



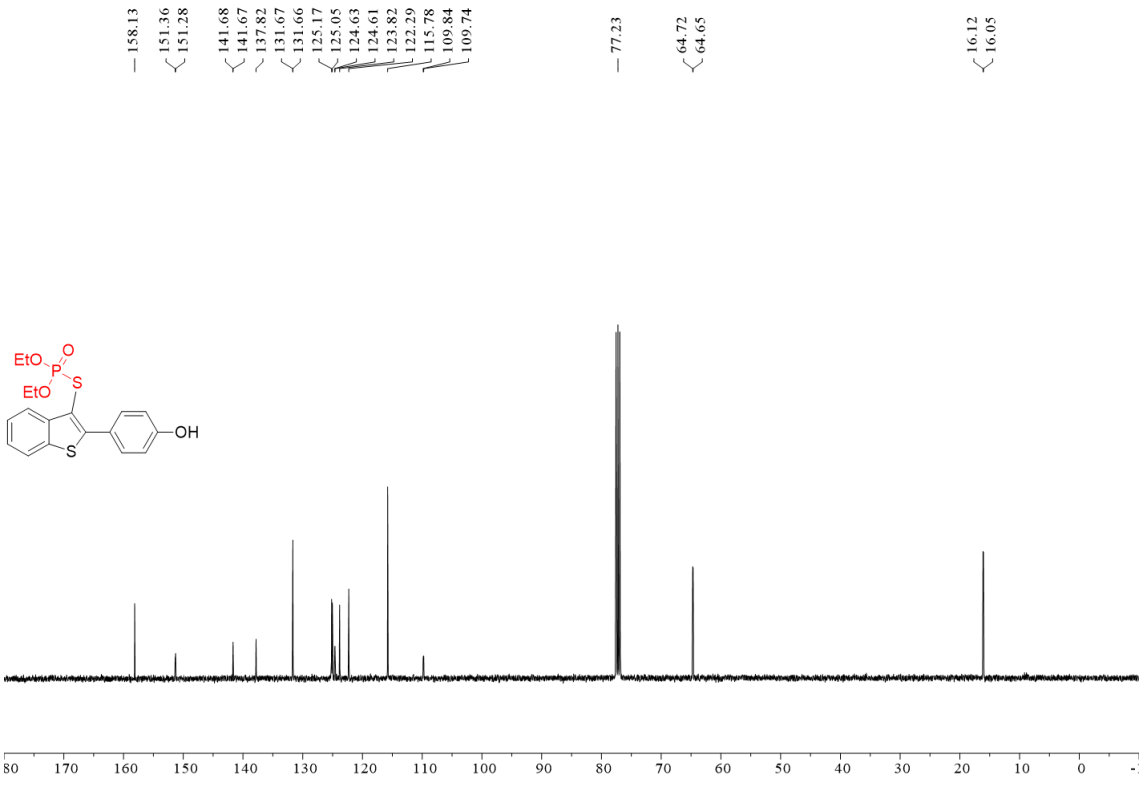
^{31}P NMR (162 MHz) Spectrum of **3i** in $CDCl_3$



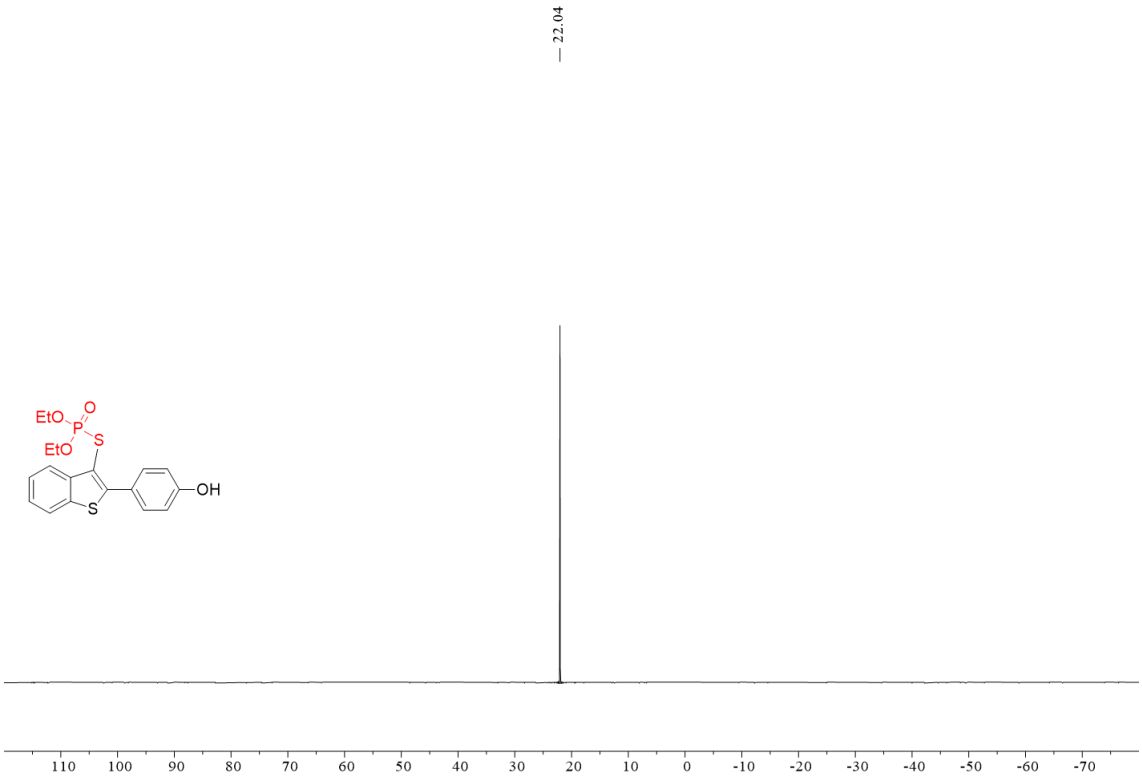
^{1}H NMR (400 MHz) Spectrum of **3j** in $CDCl_3$



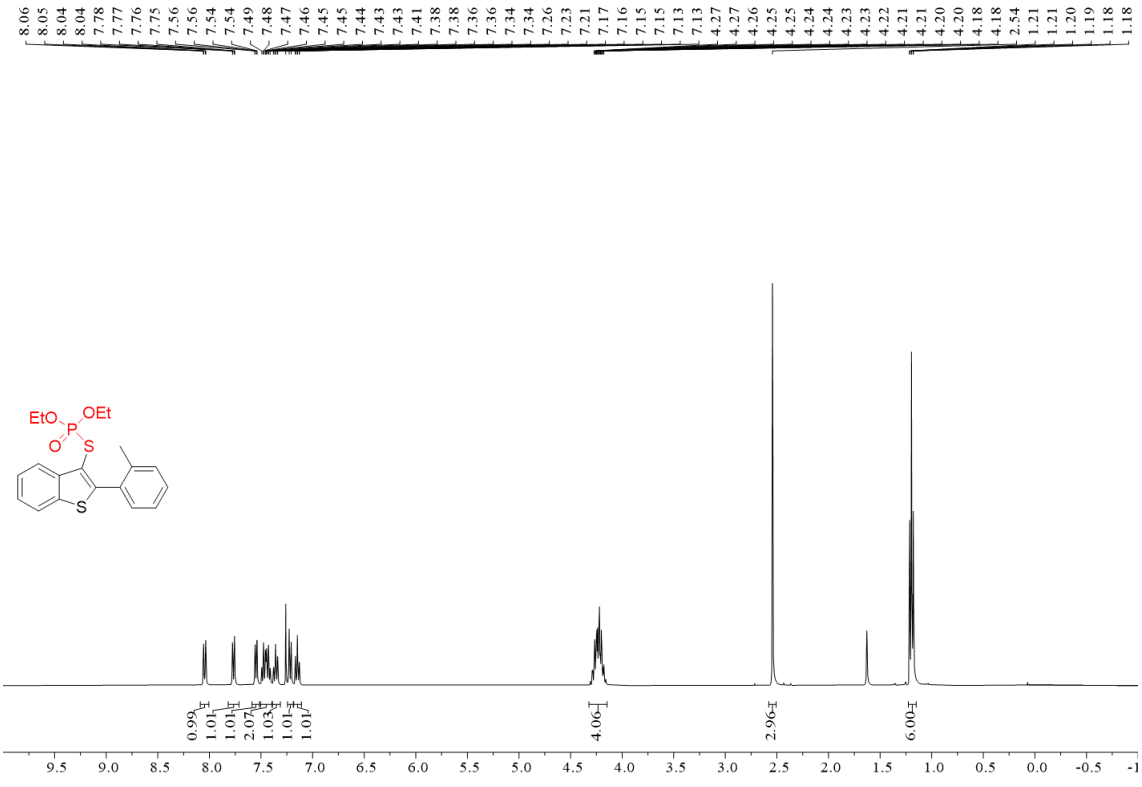
^{13}C NMR (101 MHz) Spectrum of **3j** in $CDCl_3$



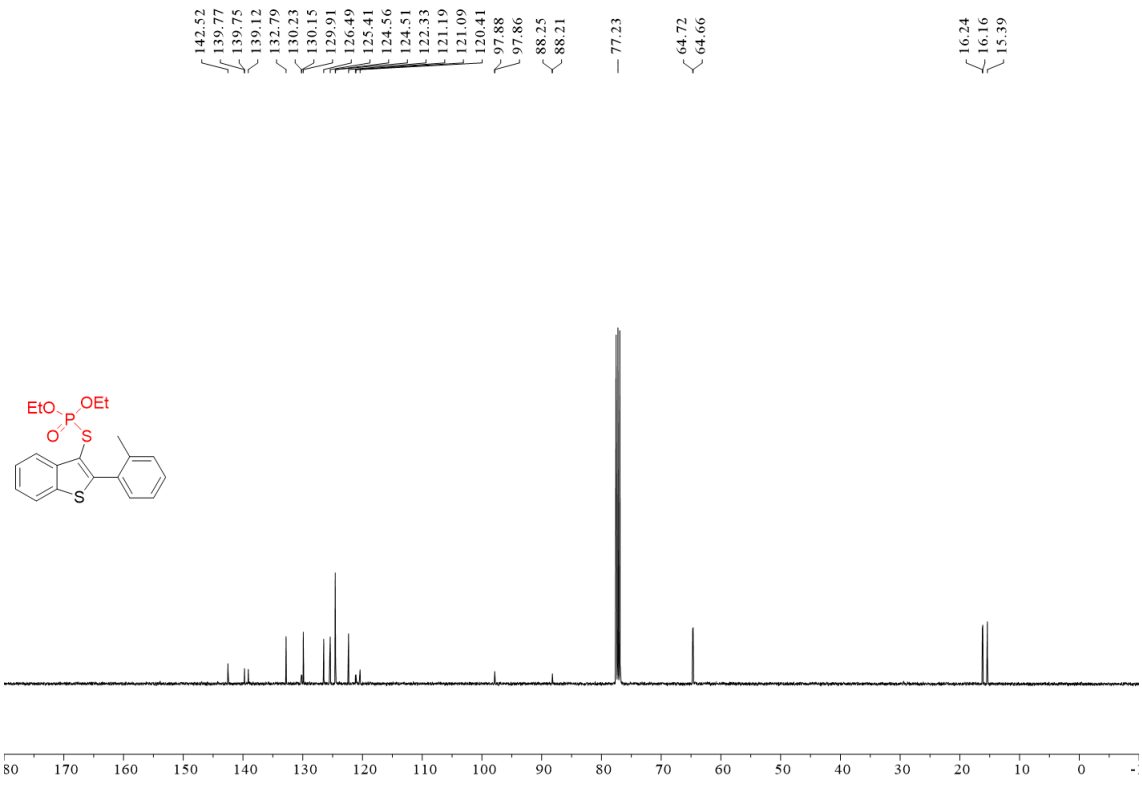
^{31}P NMR (162 MHz) Spectrum of **3j** in $CDCl_3$



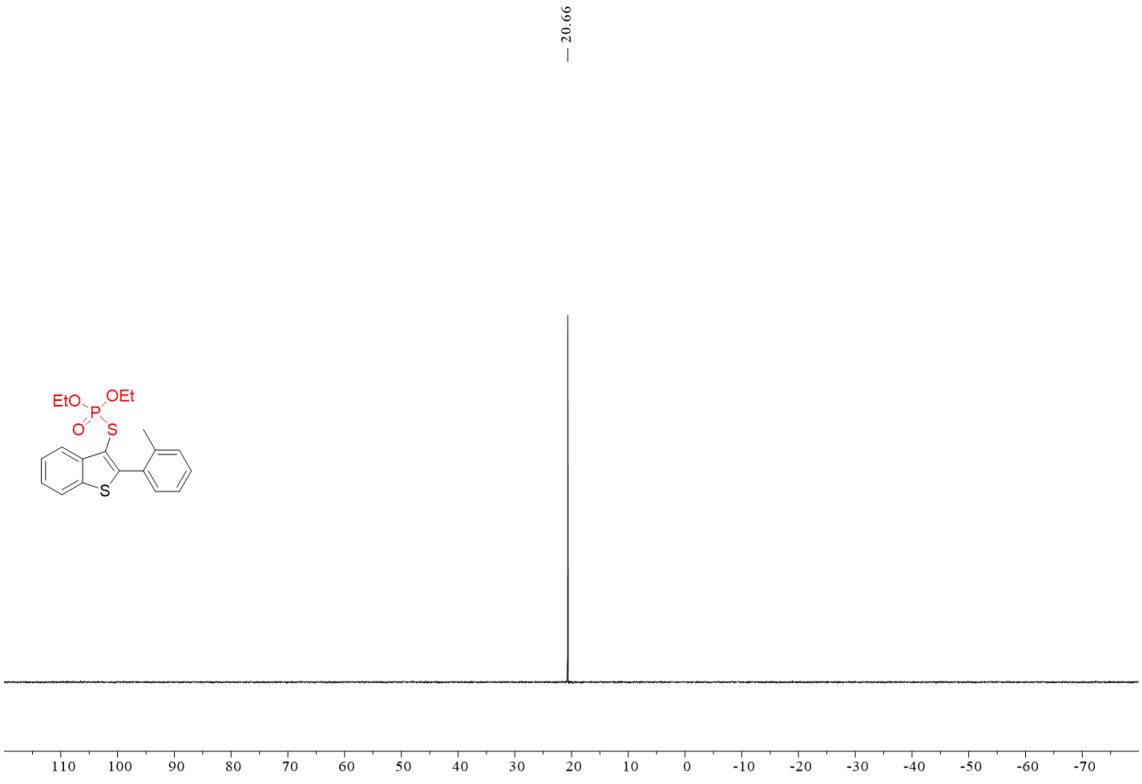
^{1}H NMR (400 MHz) Spectrum of **3k** in $CDCl_3$



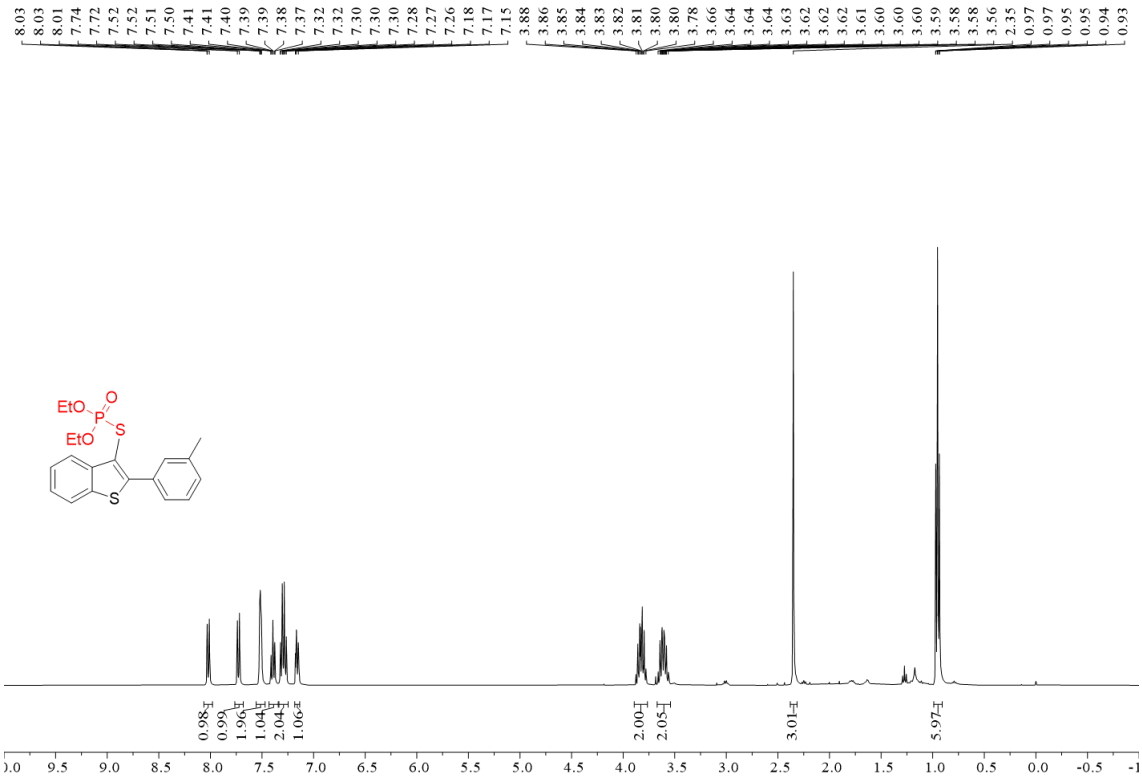
^{13}C NMR (101 MHz) Spectrum of **3k** in $CDCl_3$



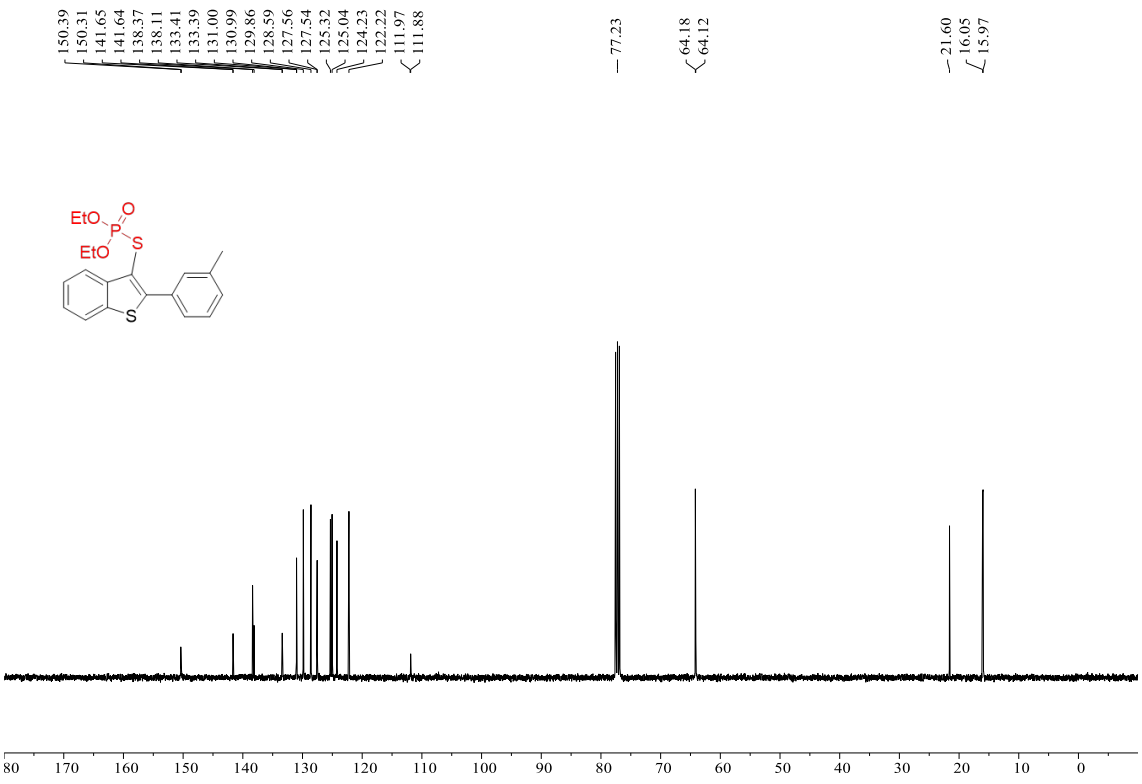
^{31}P NMR (162 MHz) Spectrum of **3k** in $CDCl_3$



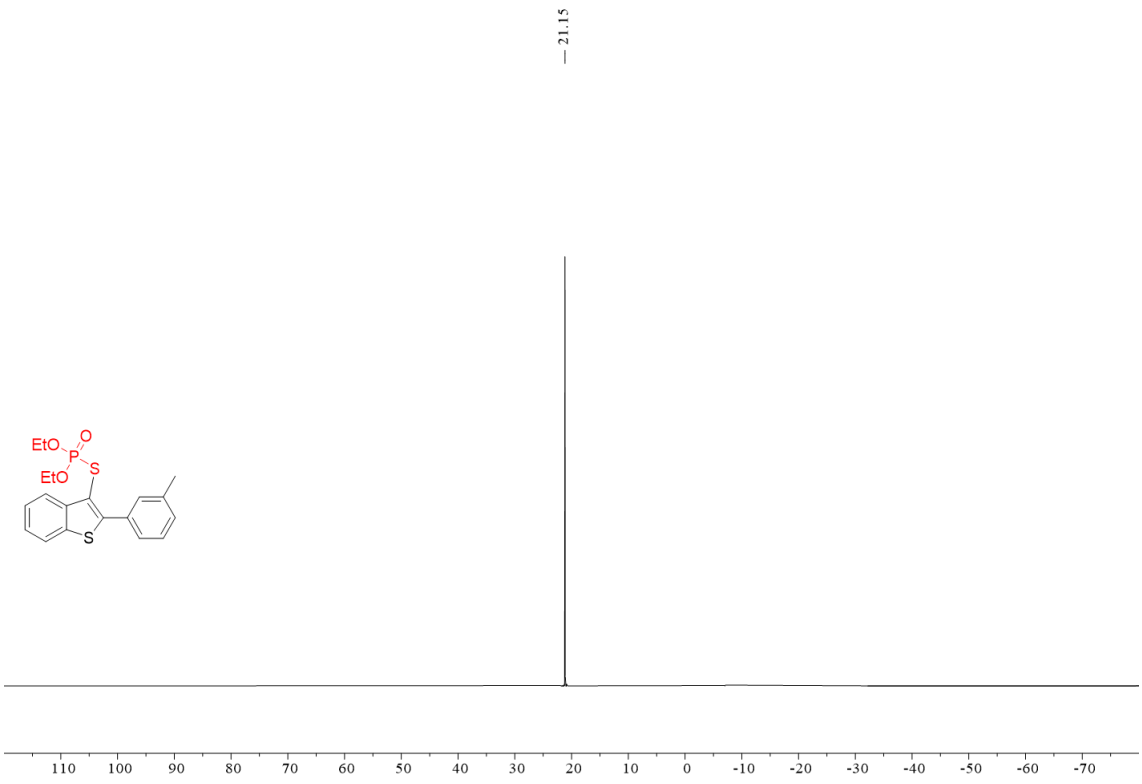
^{1}H NMR (400 MHz) Spectrum of **3l** in $CDCl_3$



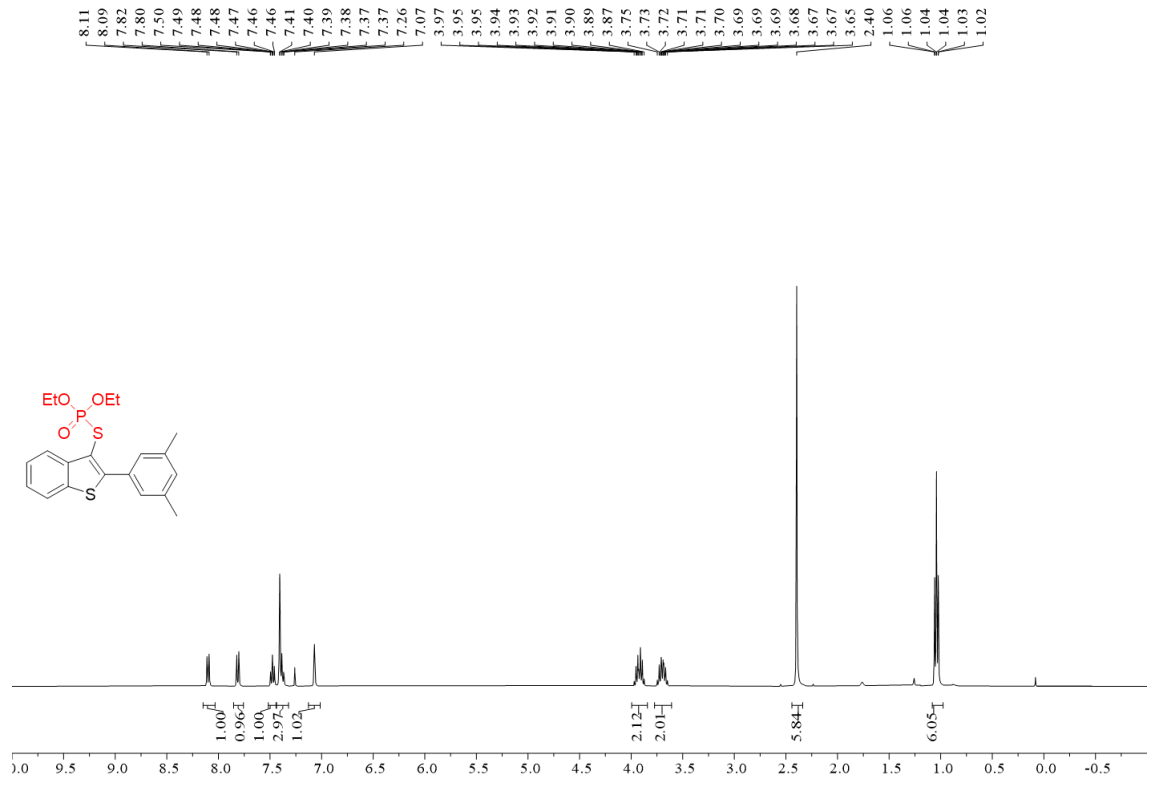
^{13}C NMR (101 MHz) Spectrum of **3l** in $CDCl_3$



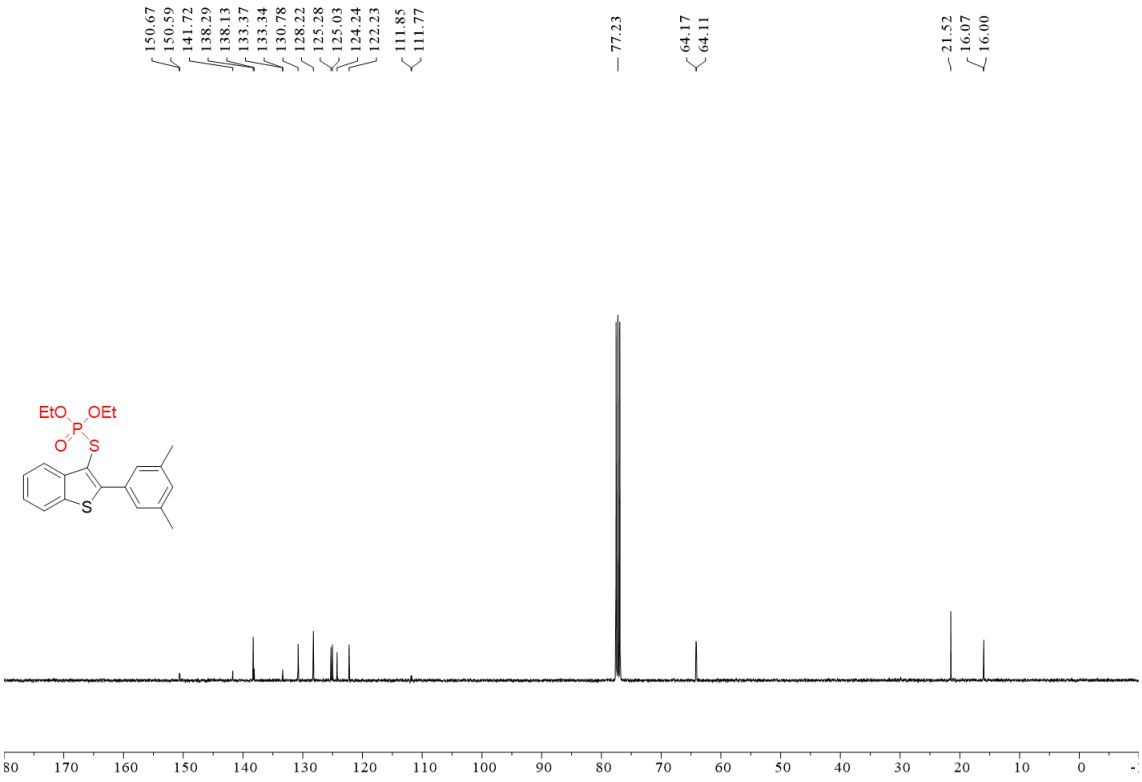
^{31}P NMR (162 MHz) Spectrum of **3l** in $CDCl_3$



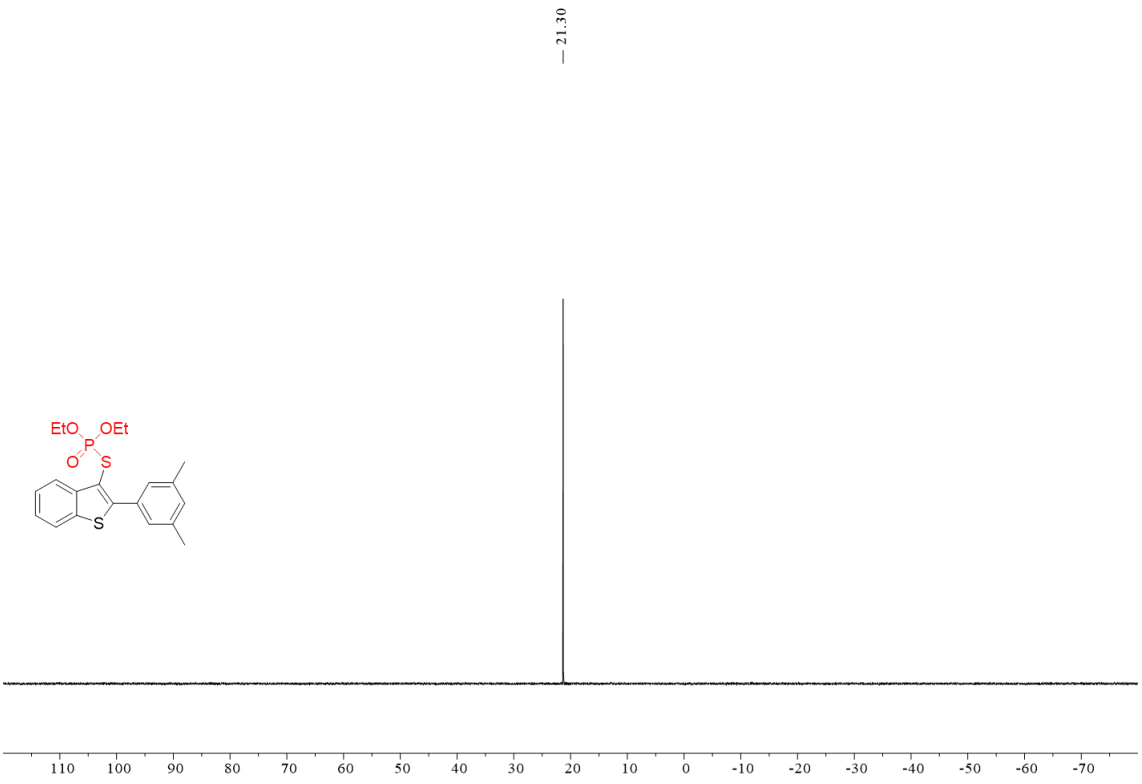
^{1}H NMR (400 MHz) Spectrum of **3m** in $CDCl_3$



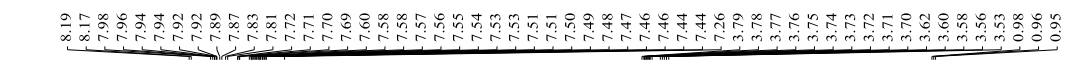
^{13}C NMR (101 MHz) Spectrum of **3m** in $CDCl_3$

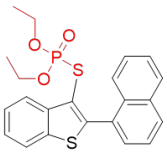


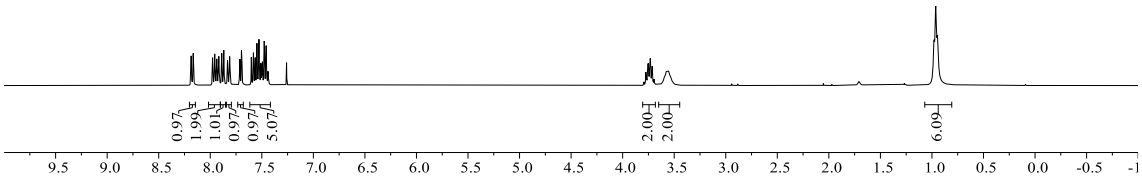
^{31}P NMR (162 MHz) Spectrum of **3m** in $CDCl_3$



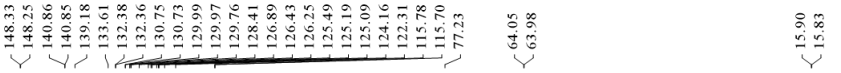
^{1}H NMR (400 MHz) Spectrum of **3n** in $CDCl_3$

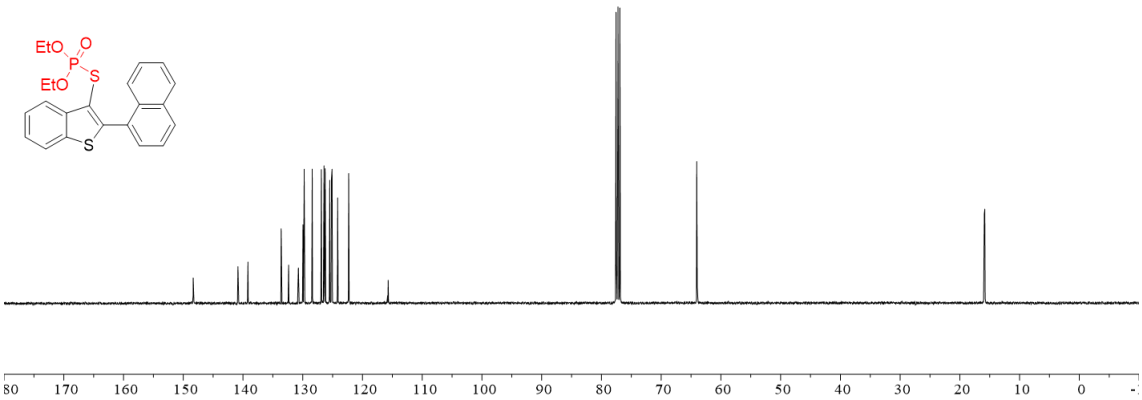




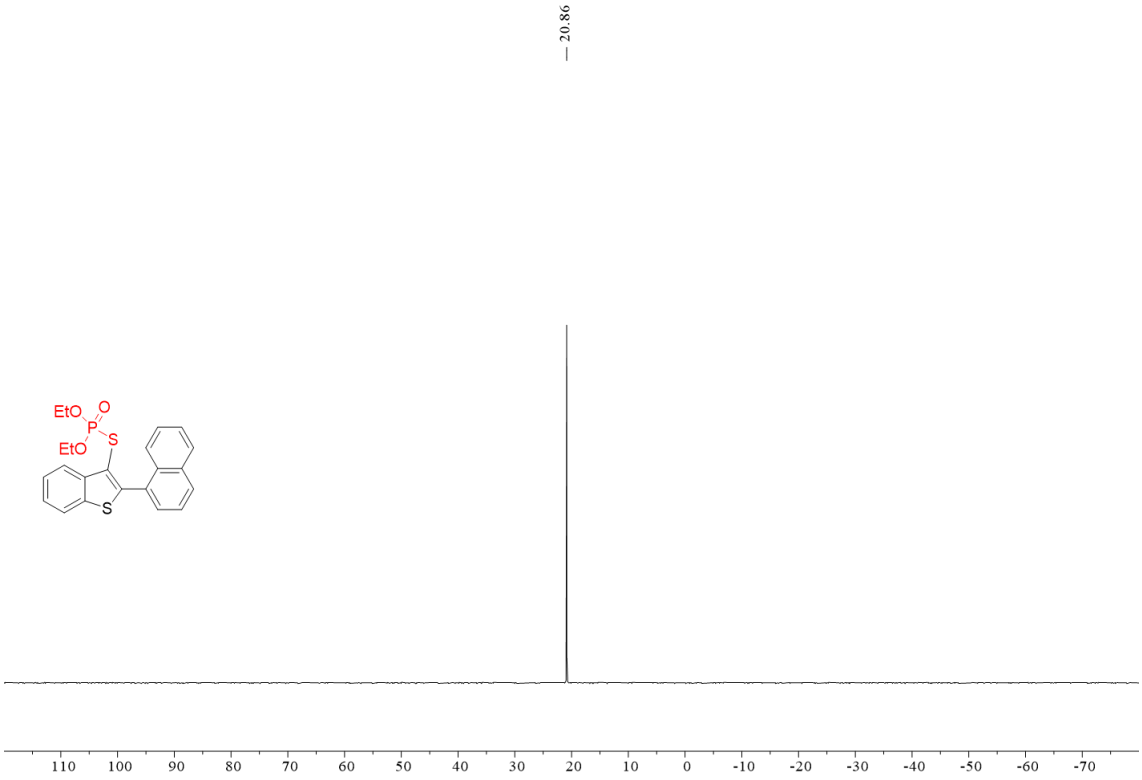


^{13}C NMR (101 MHz) Spectrum of **3n** in $CDCl_3$

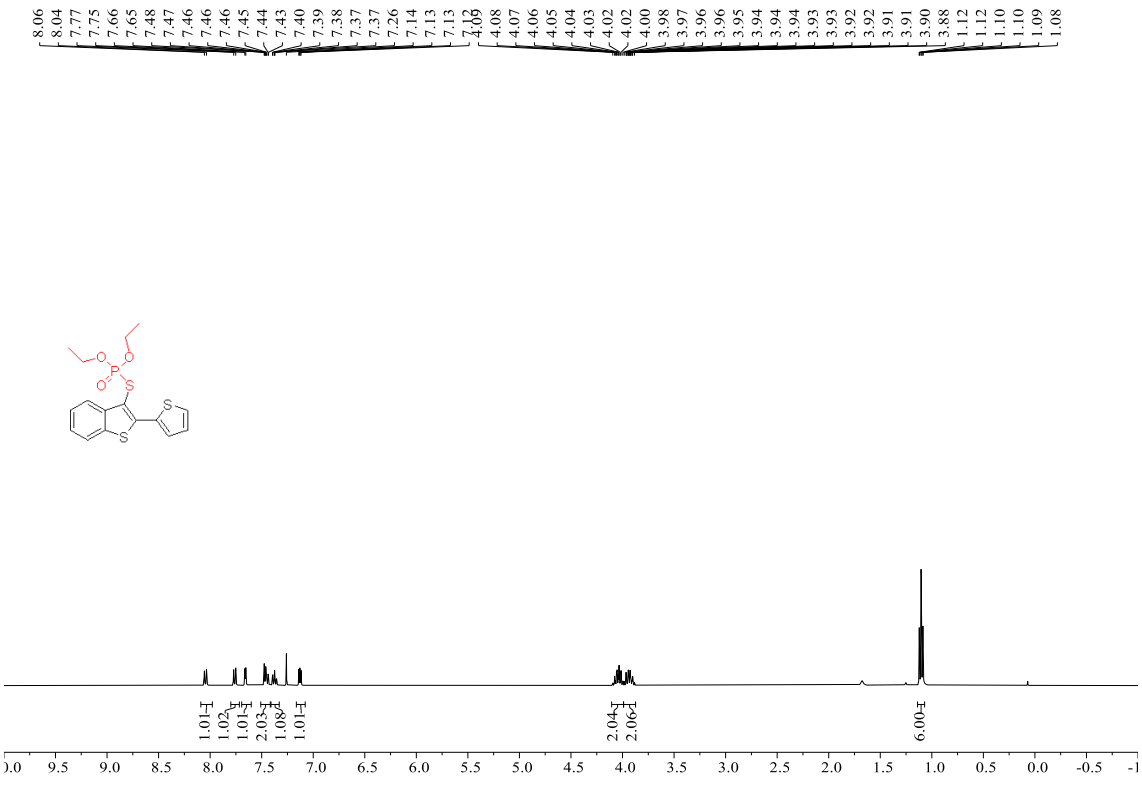




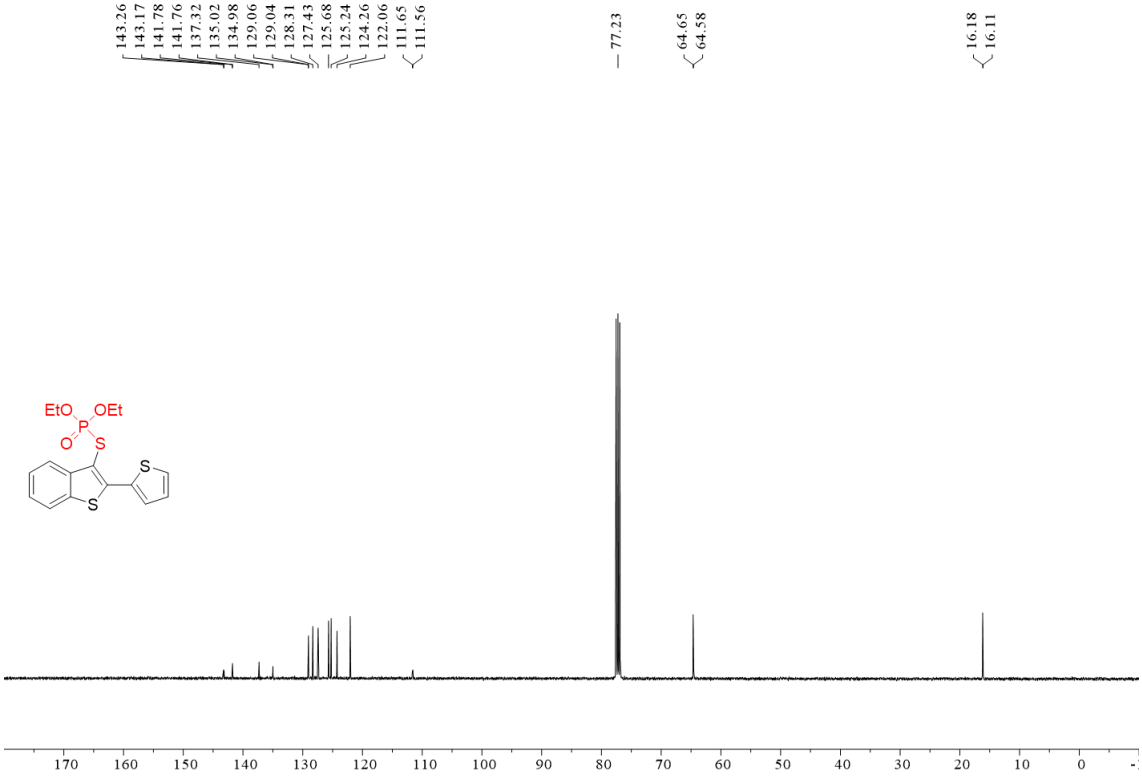
^{31}P NMR (162 MHz) Spectrum of **3n** in $CDCl_3$



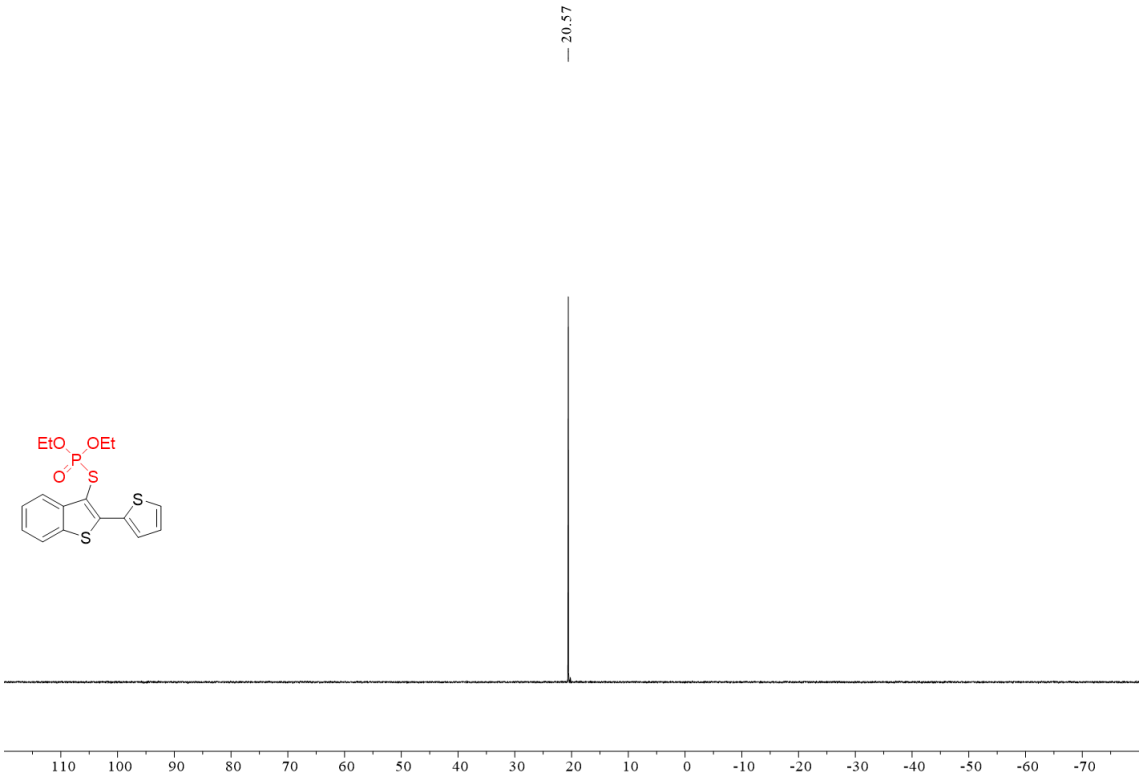
^{1}H NMR (400 MHz) Spectrum of **3o** in $CDCl_3$



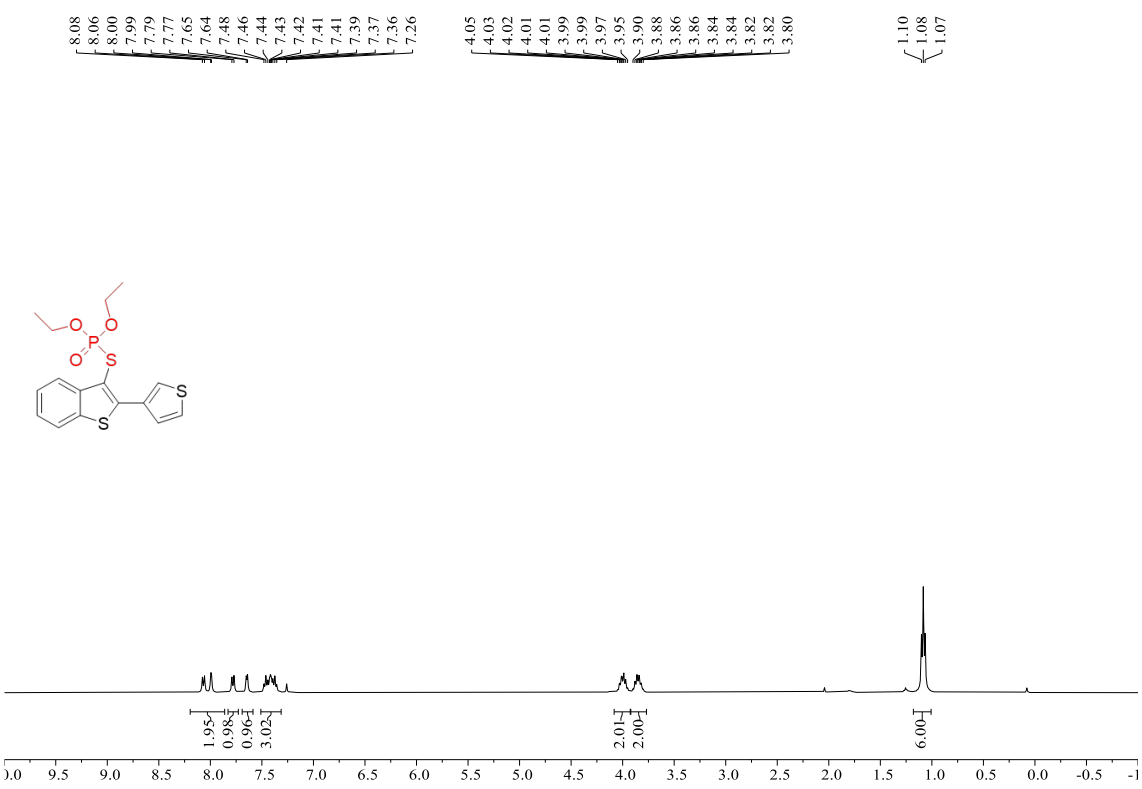
^{13}C NMR (101 MHz) Spectrum of **3o** in $CDCl_3$



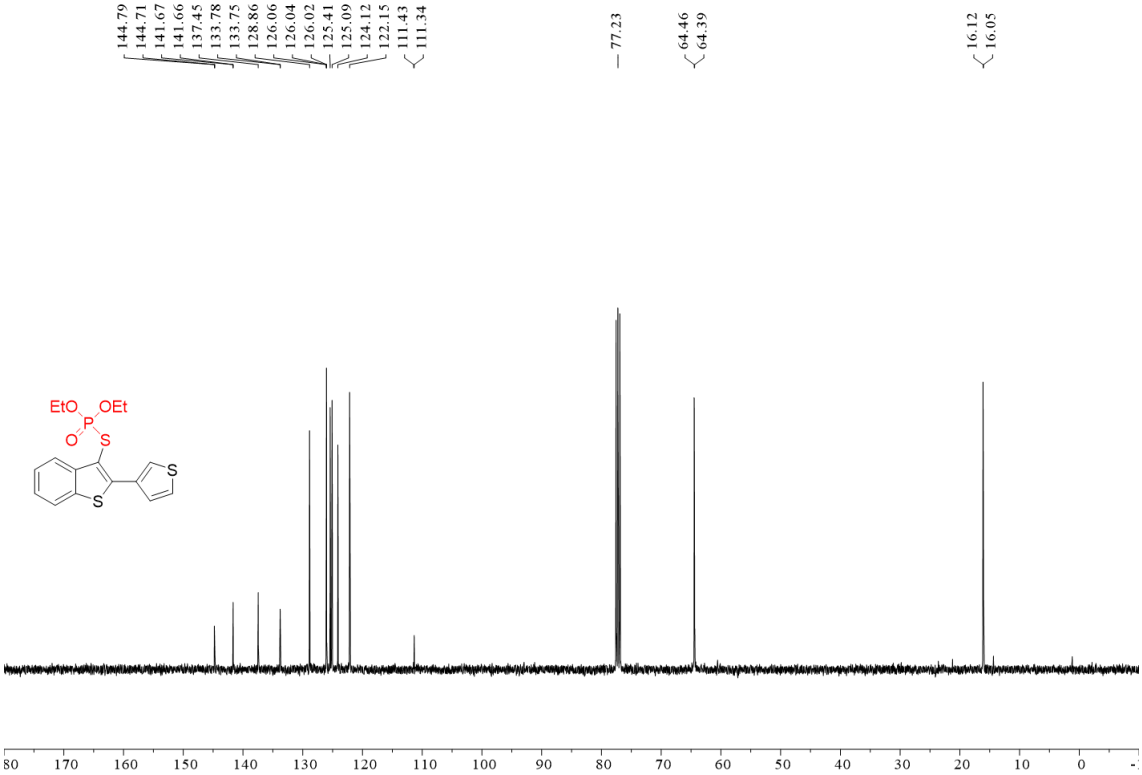
^{31}P NMR (162 MHz) Spectrum of **3o** in $CDCl_3$



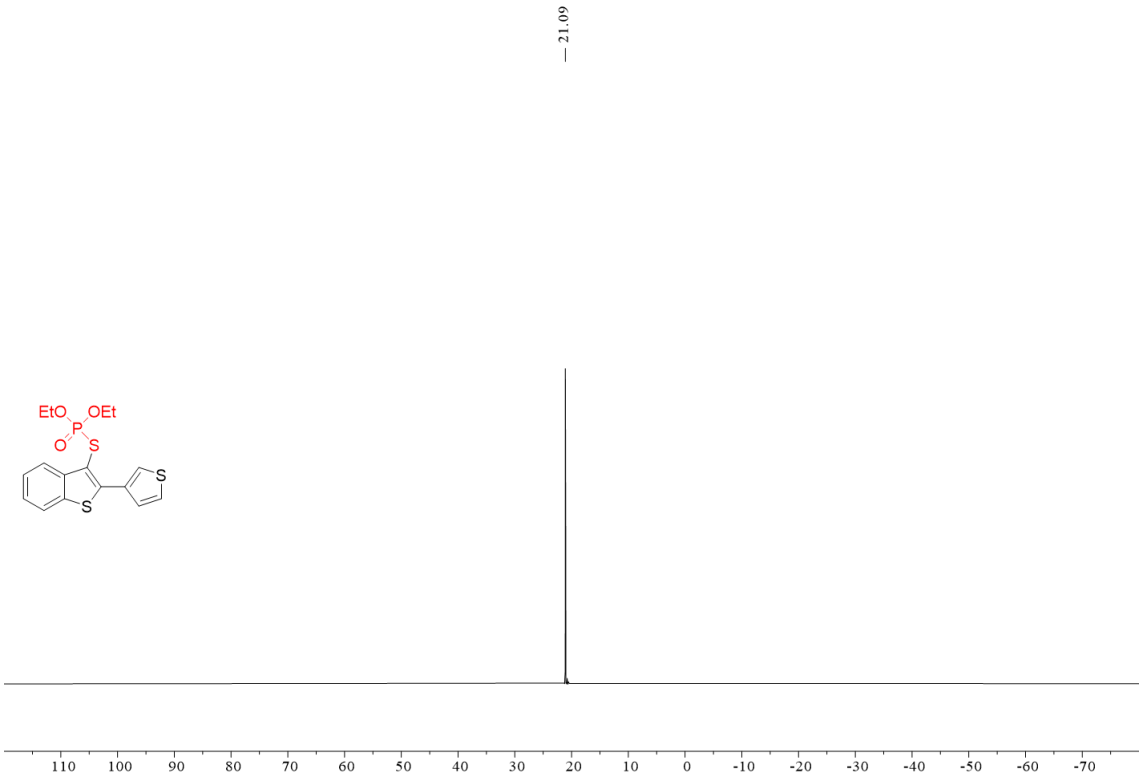
^{1}H NMR (400 MHz) Spectrum of **3p** in $CDCl_3$



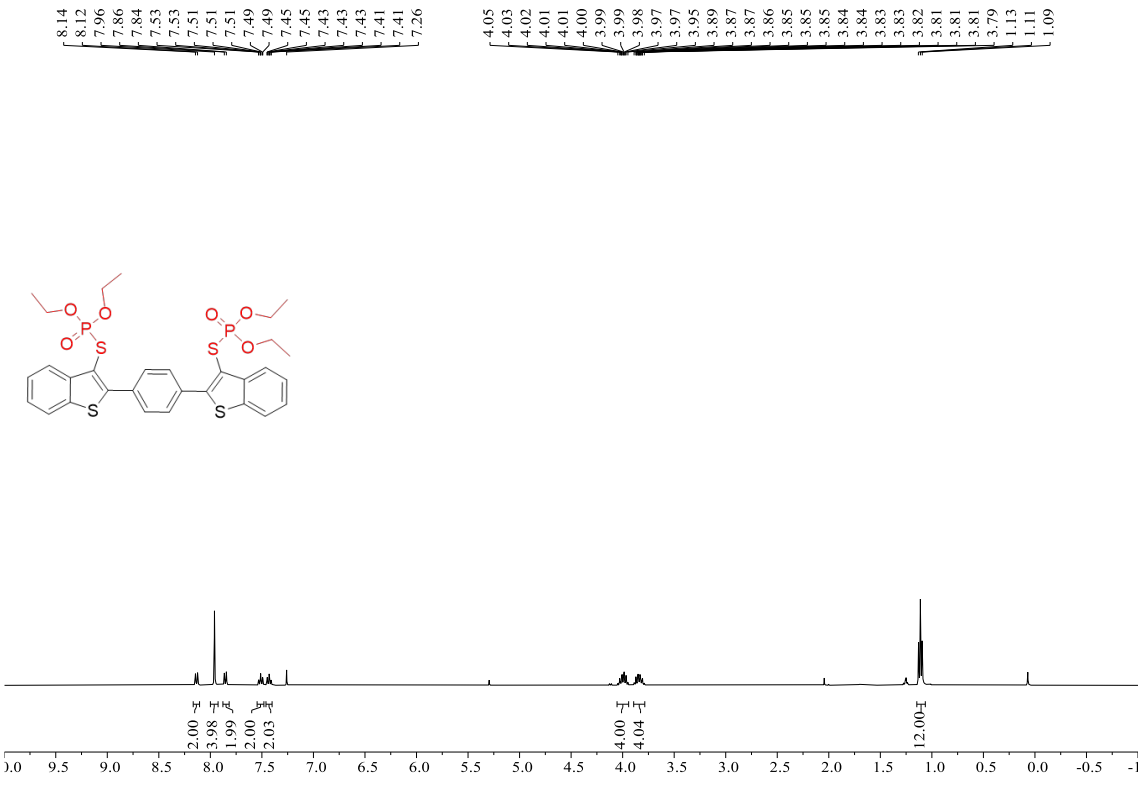
^{13}C NMR (101 MHz) Spectrum of **3p** in $CDCl_3$



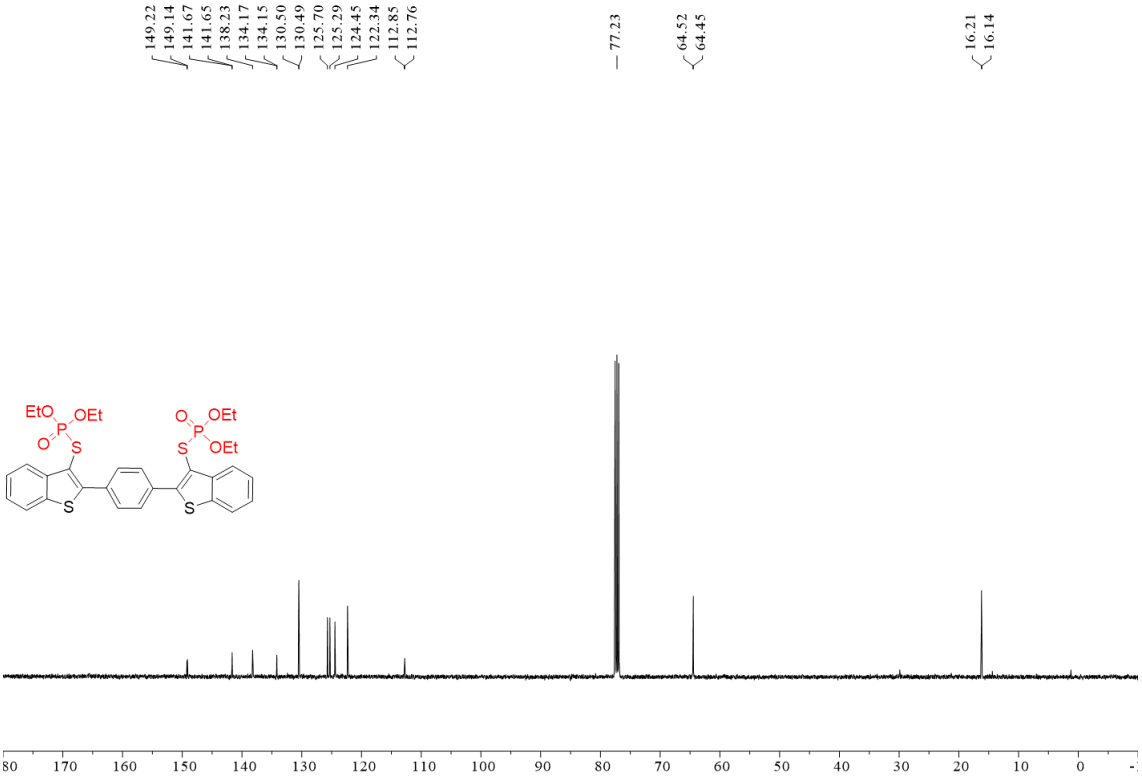
^{31}P NMR (162 MHz) Spectrum of **3p** in $CDCl_3$



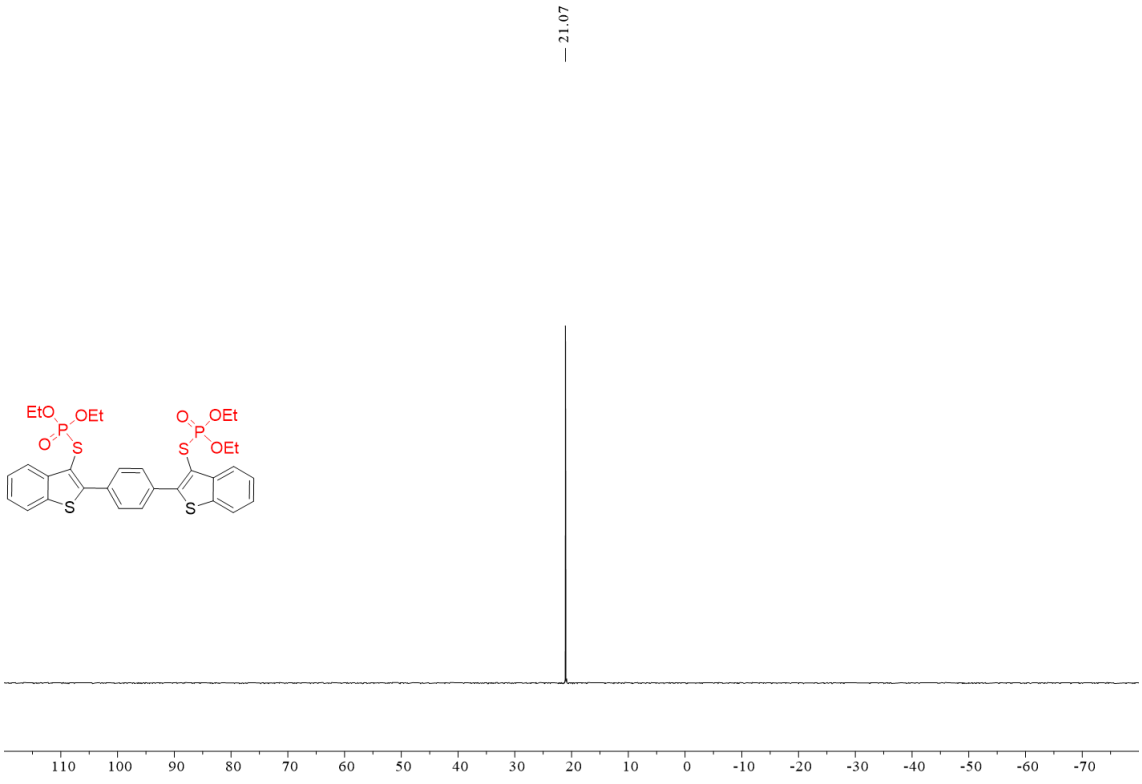
^{1}H NMR (400 MHz) Spectrum of **3q** in $CDCl_3$



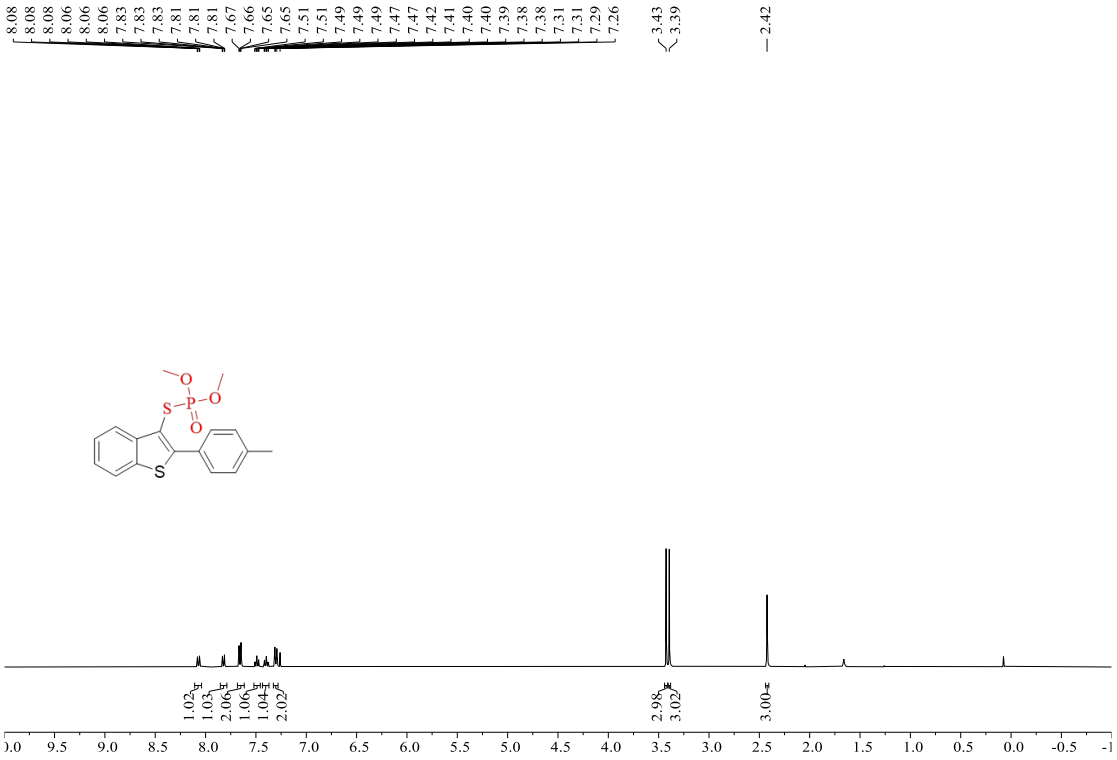
^{13}C NMR (101 MHz) Spectrum of **3q** in $CDCl_3$



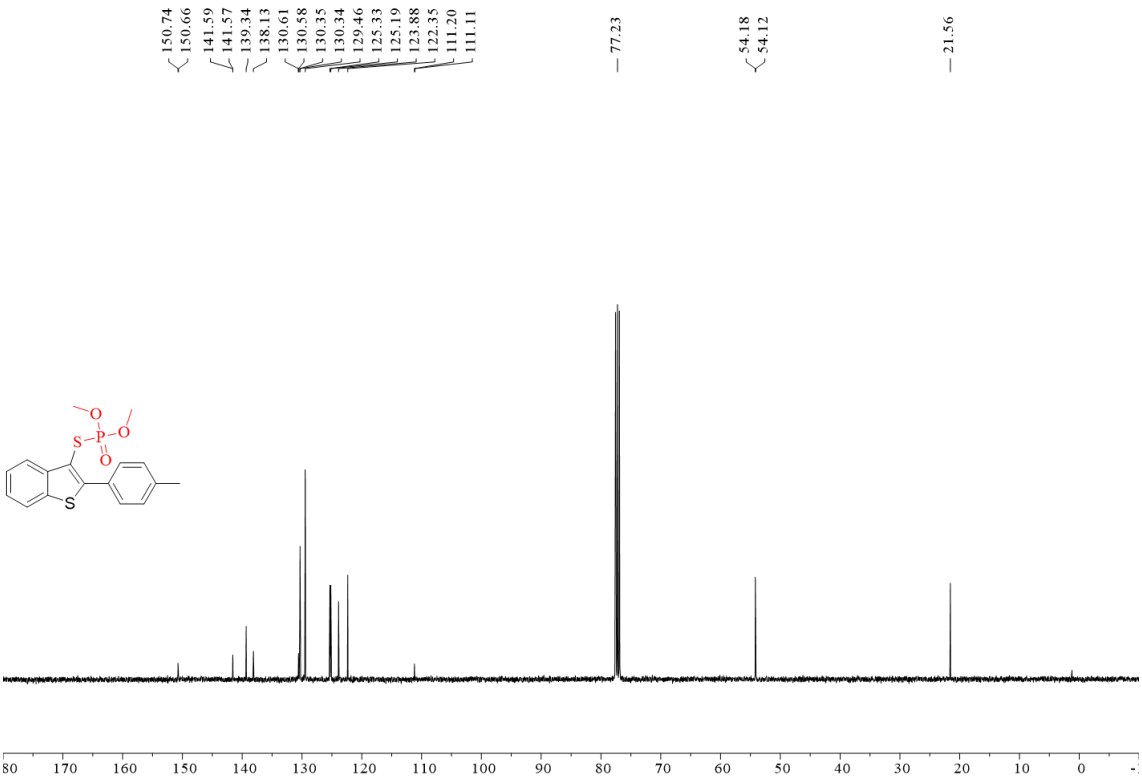
^{31}P NMR (162 MHz) Spectrum of **3q** in $CDCl_3$



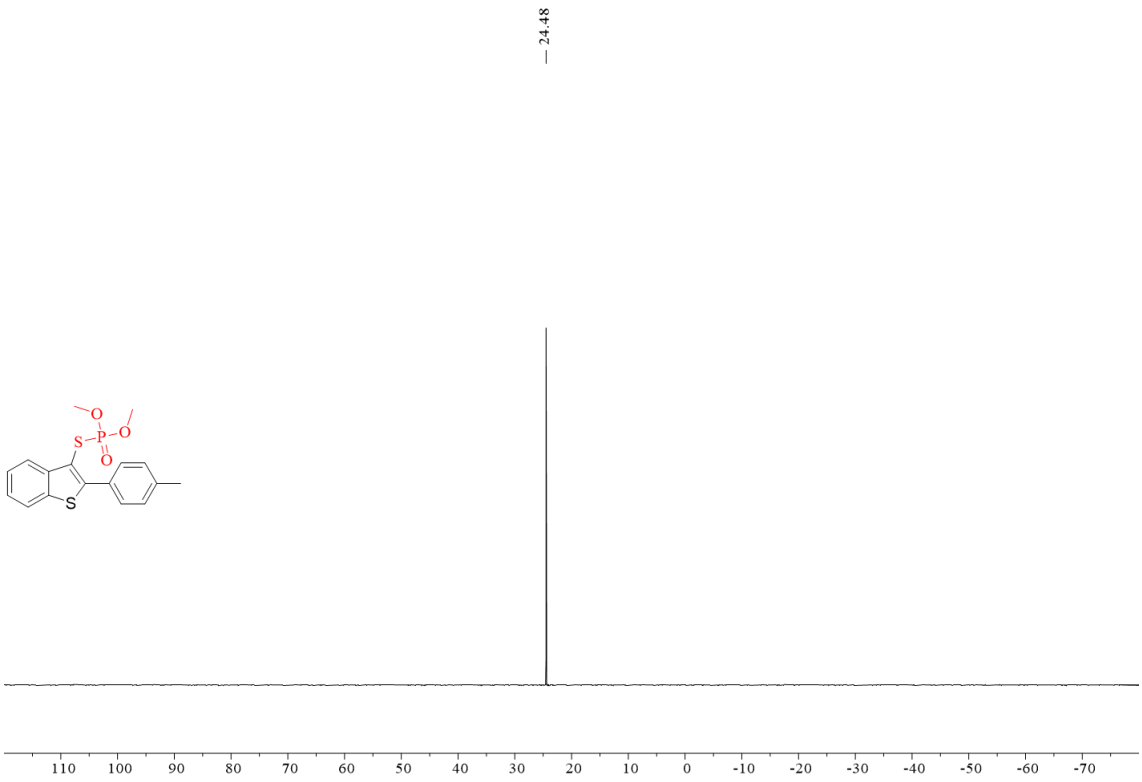
^{1}H NMR (400 MHz) Spectrum of **3r** in $CDCl_3$



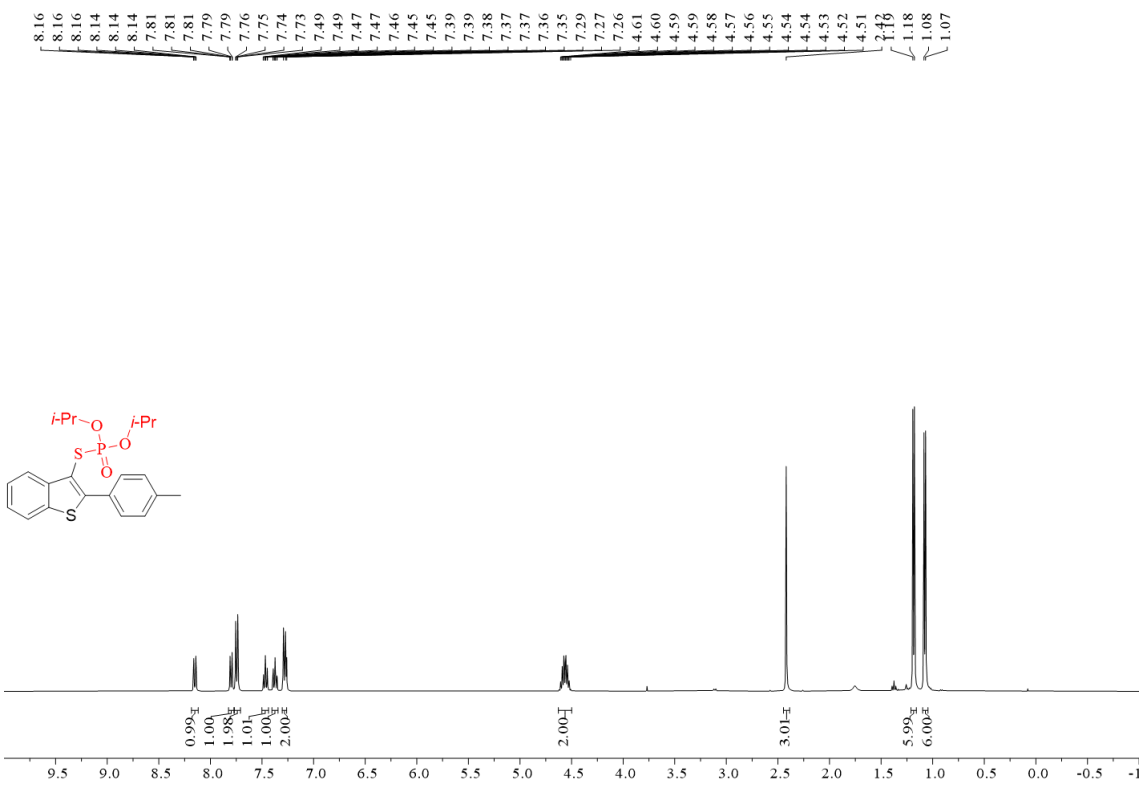
^{13}C NMR (101 MHz) Spectrum of **3r** in $CDCl_3$



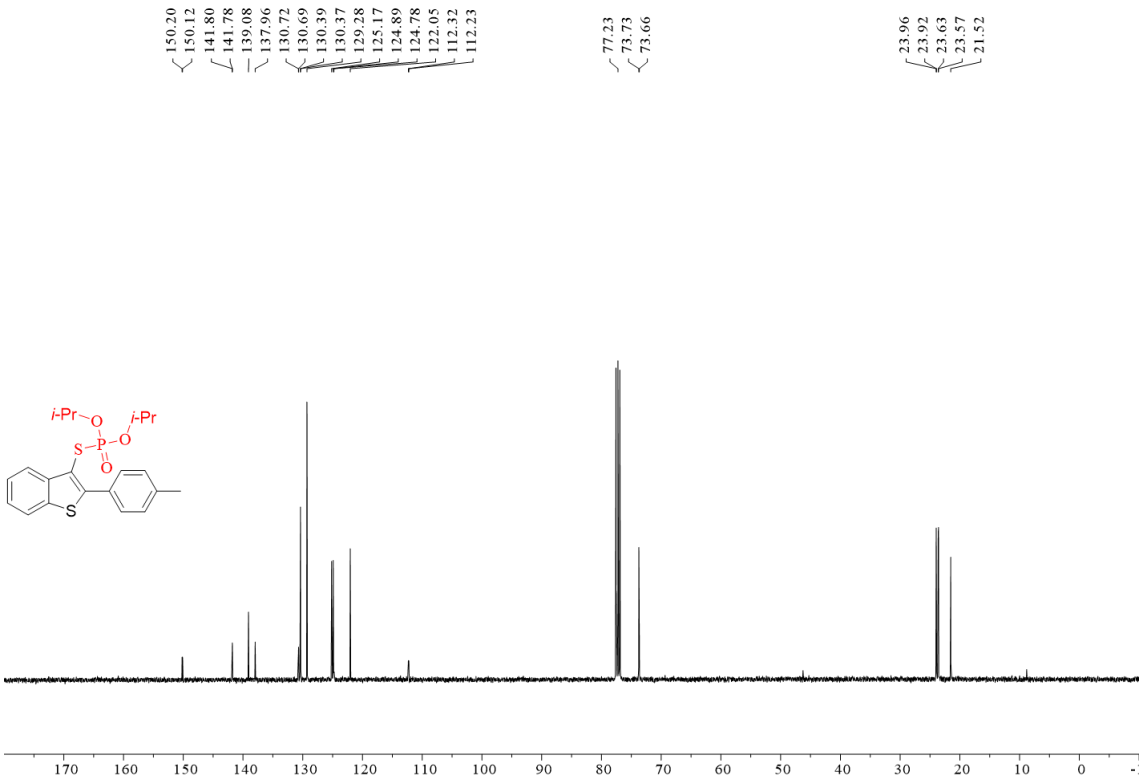
^{31}P NMR (162 MHz) Spectrum of **3r** in $CDCl_3$



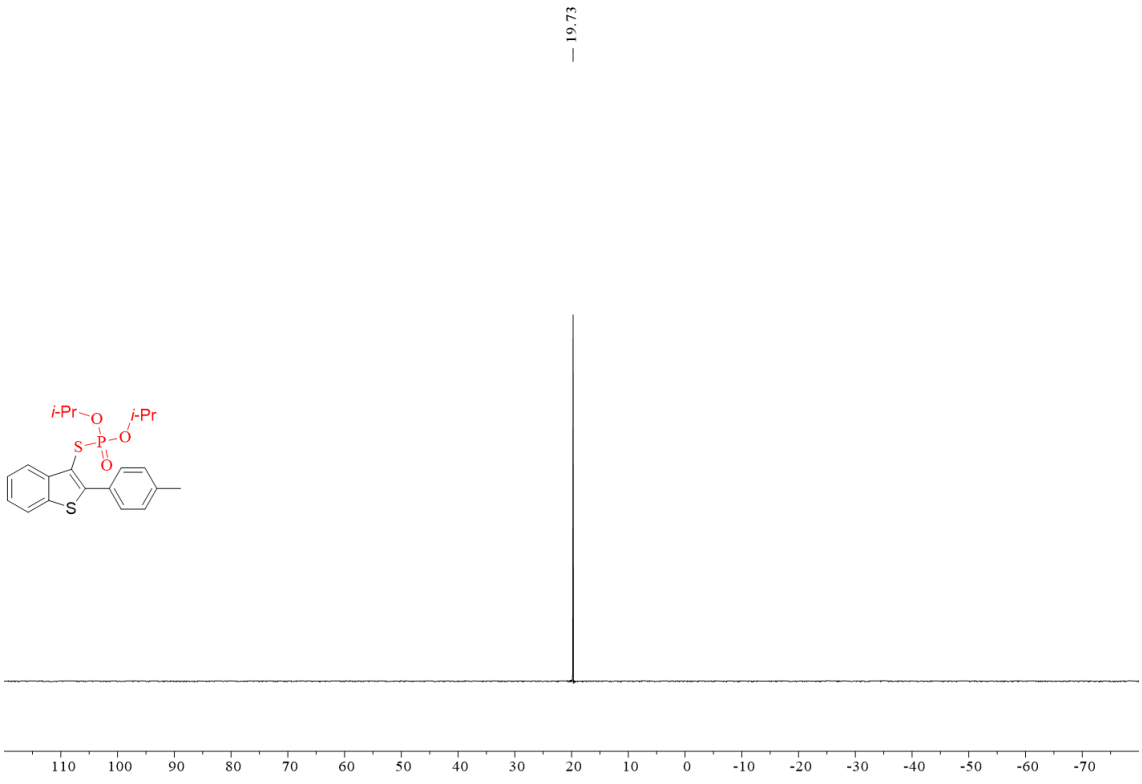
^{1}H NMR (400 MHz) Spectrum of **3s** in $CDCl_3$



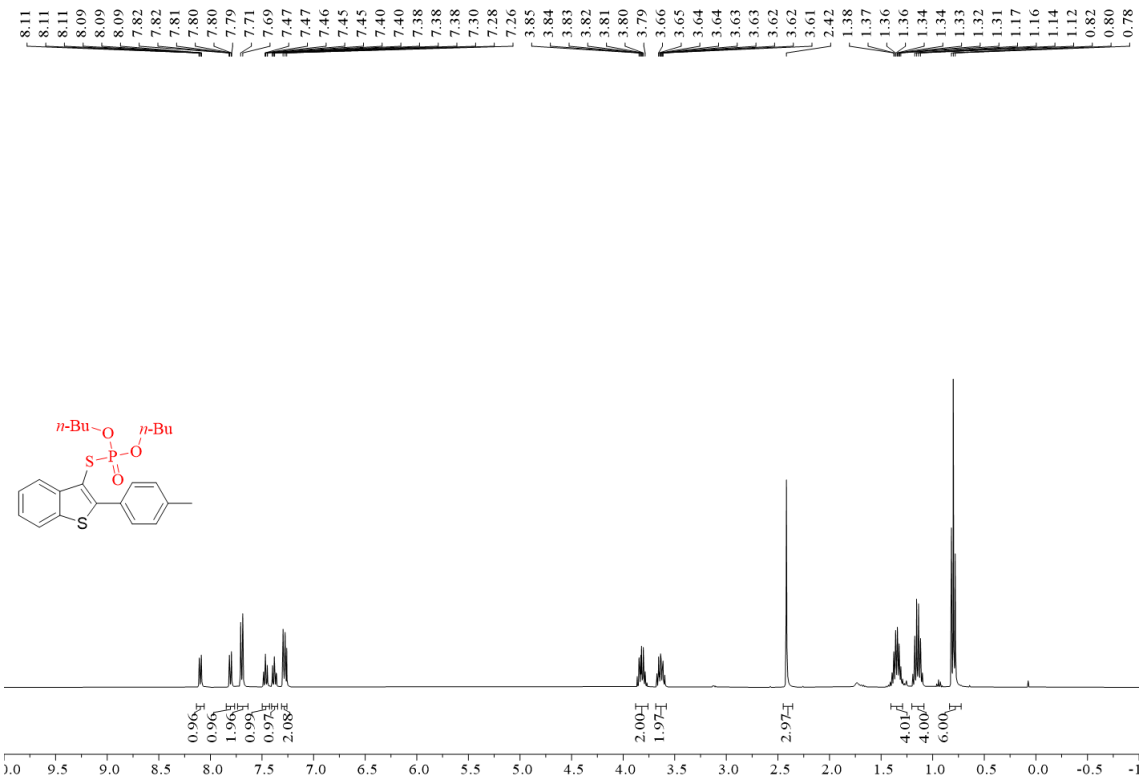
^{13}C NMR (101 MHz) Spectrum of **3s** in $CDCl_3$



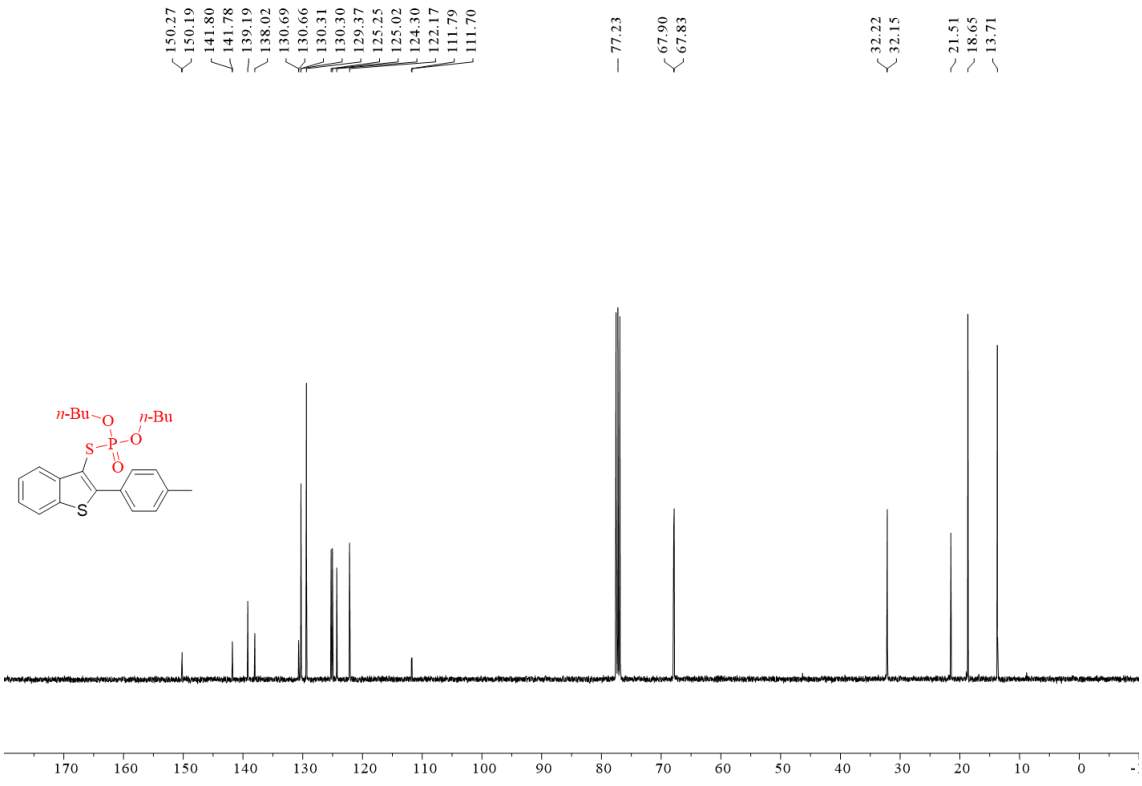
^{31}P NMR (162 MHz) Spectrum of **3s** in $CDCl_3$



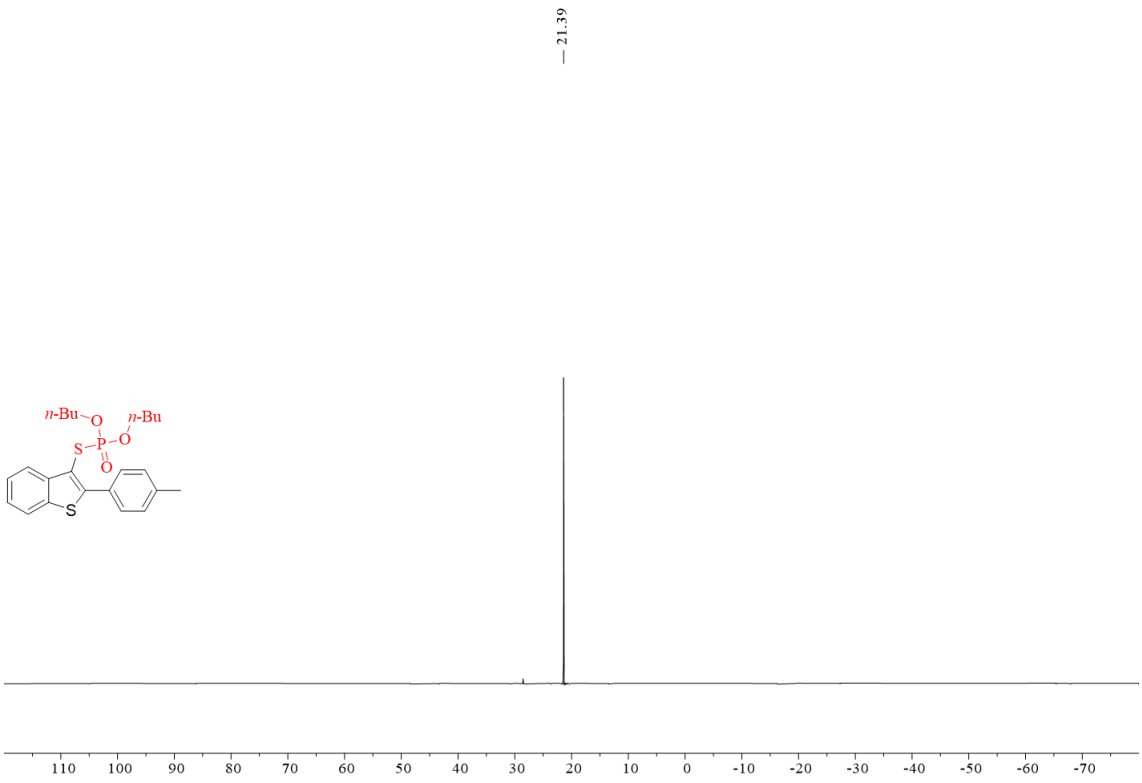
^{1}H NMR (400 MHz) Spectrum of **3t** in $CDCl_3$



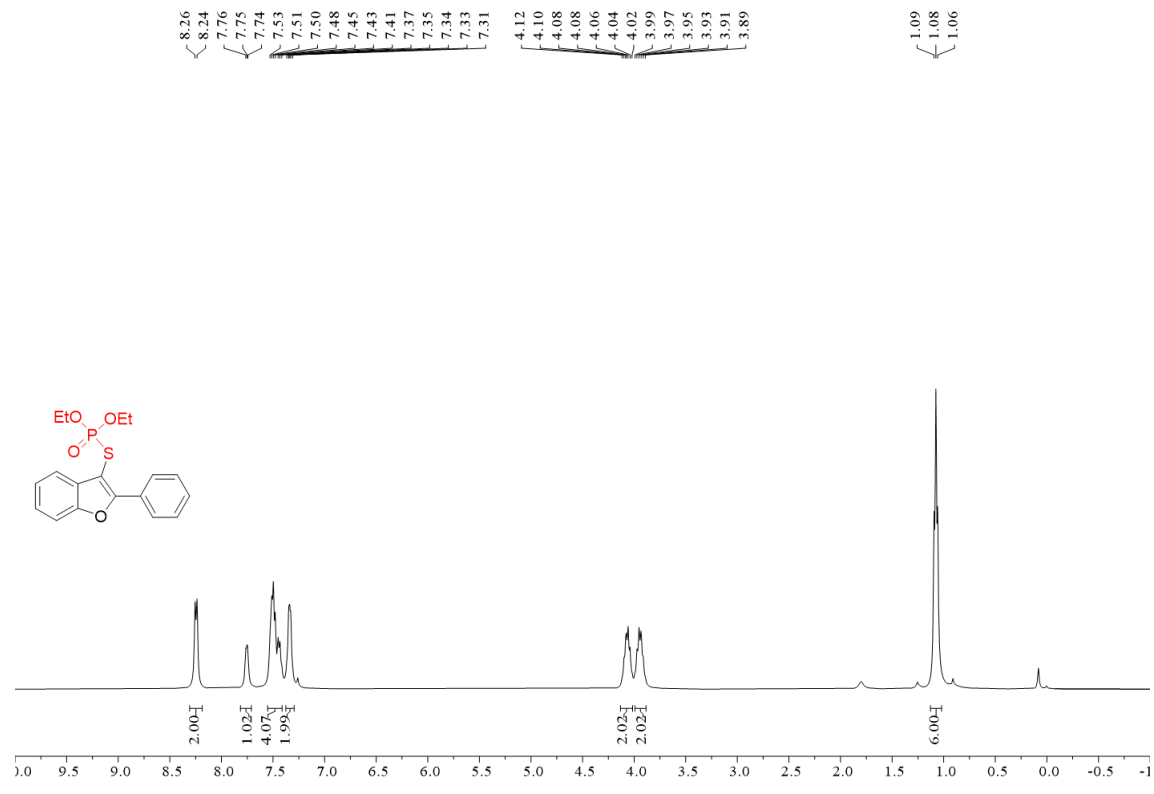
^{13}C NMR (101 MHz) Spectrum of **3t** in $CDCl_3$



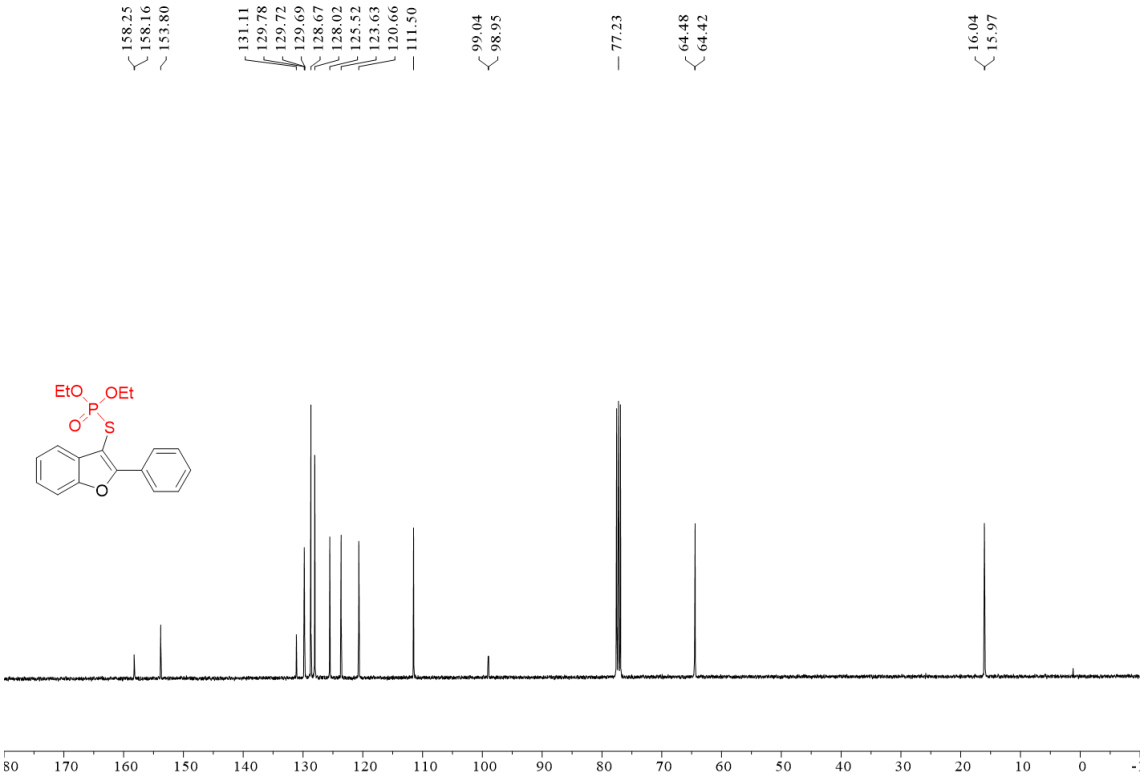
^{31}P NMR (162 MHz) Spectrum of **3t** in $CDCl_3$



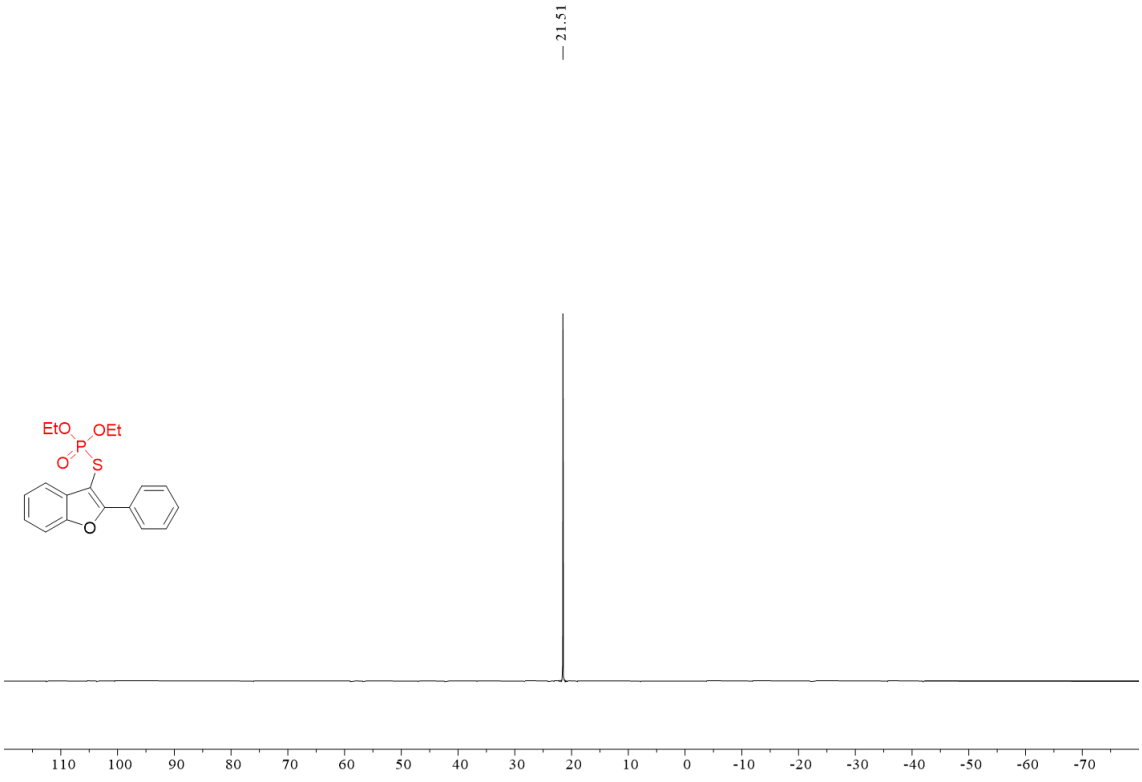
^{1}H NMR (400 MHz) Spectrum of **3u** in $CDCl_3$



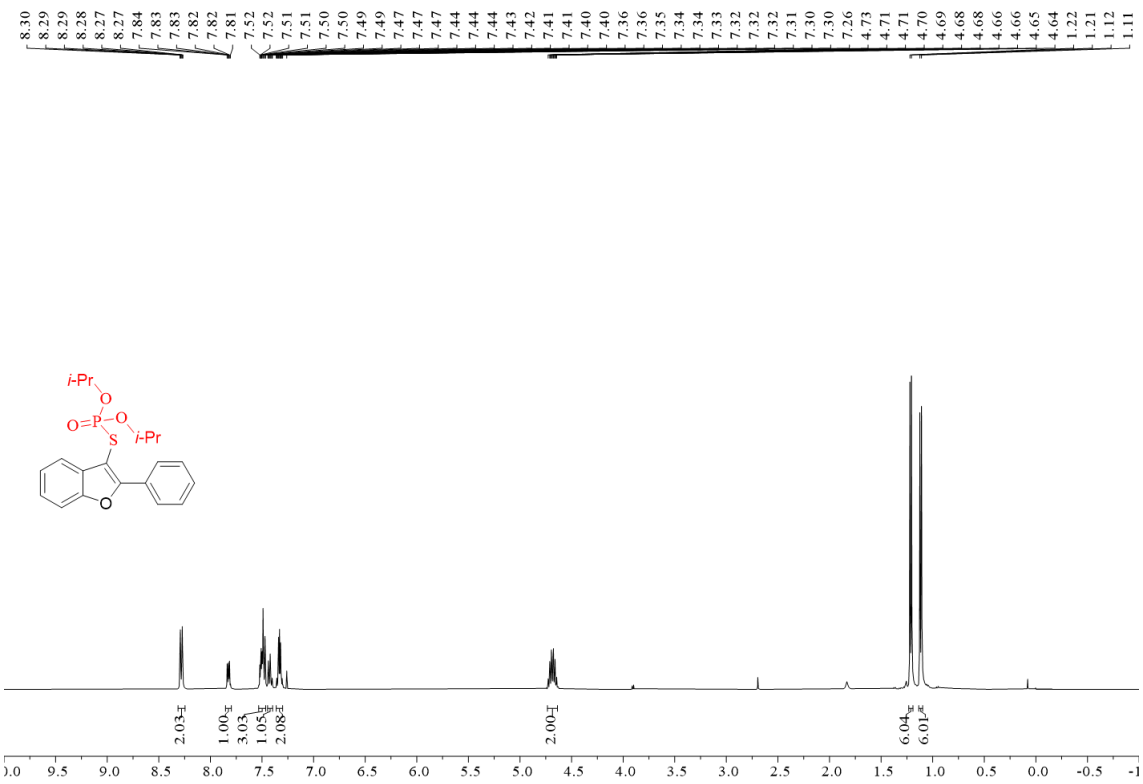
^{13}C NMR (101 MHz) Spectrum of **3u** in CDCl



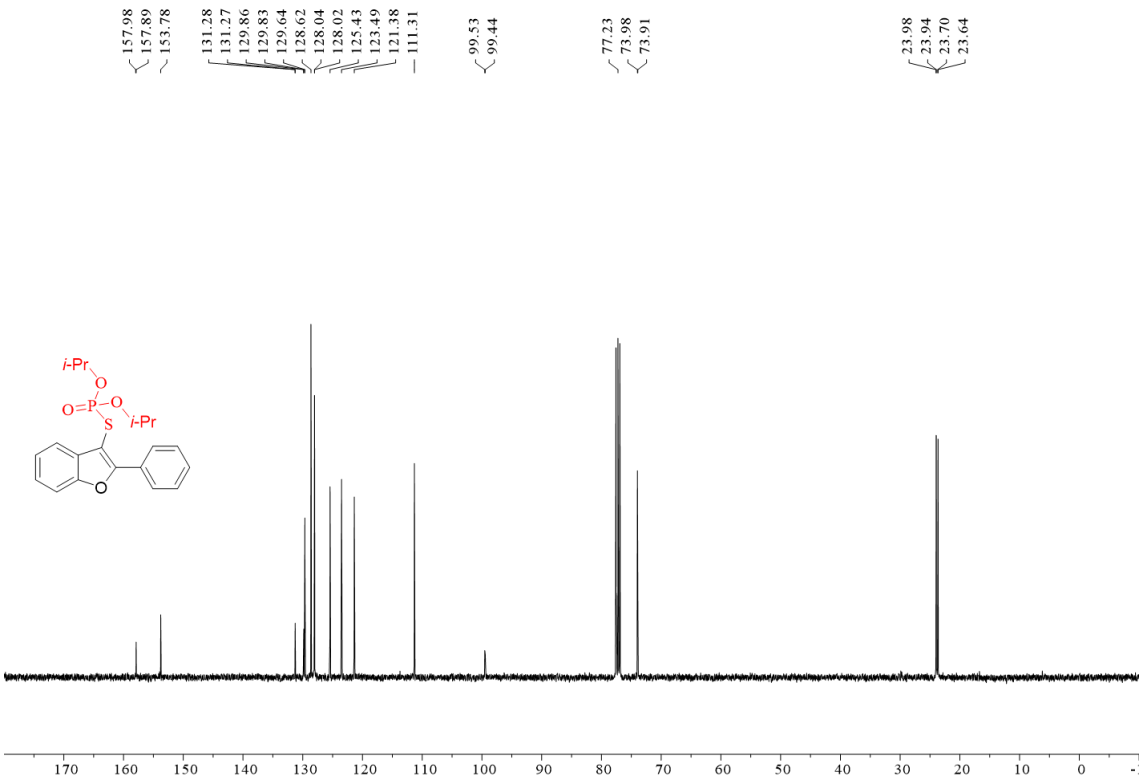
^{31}P NMR (162 MHz) Spectrum of **3u** in $CDCl_3$



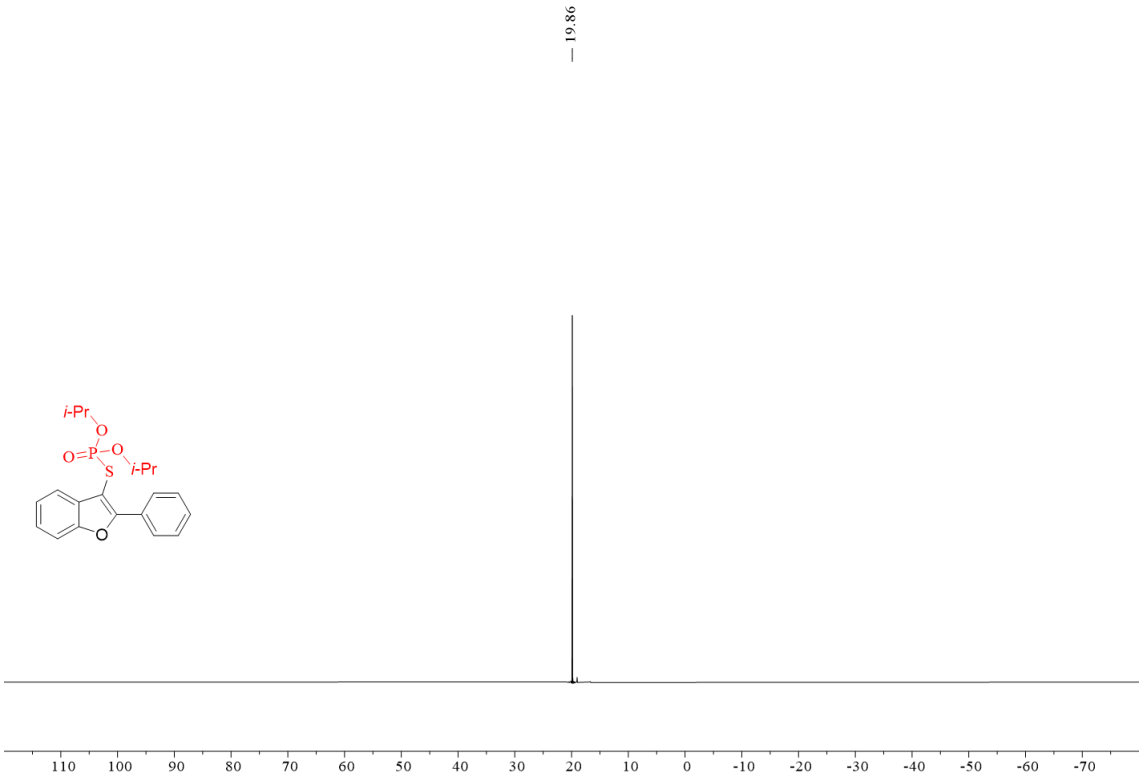
^{1}H NMR (400 MHz) Spectrum of **3v** in $CDCl_3$



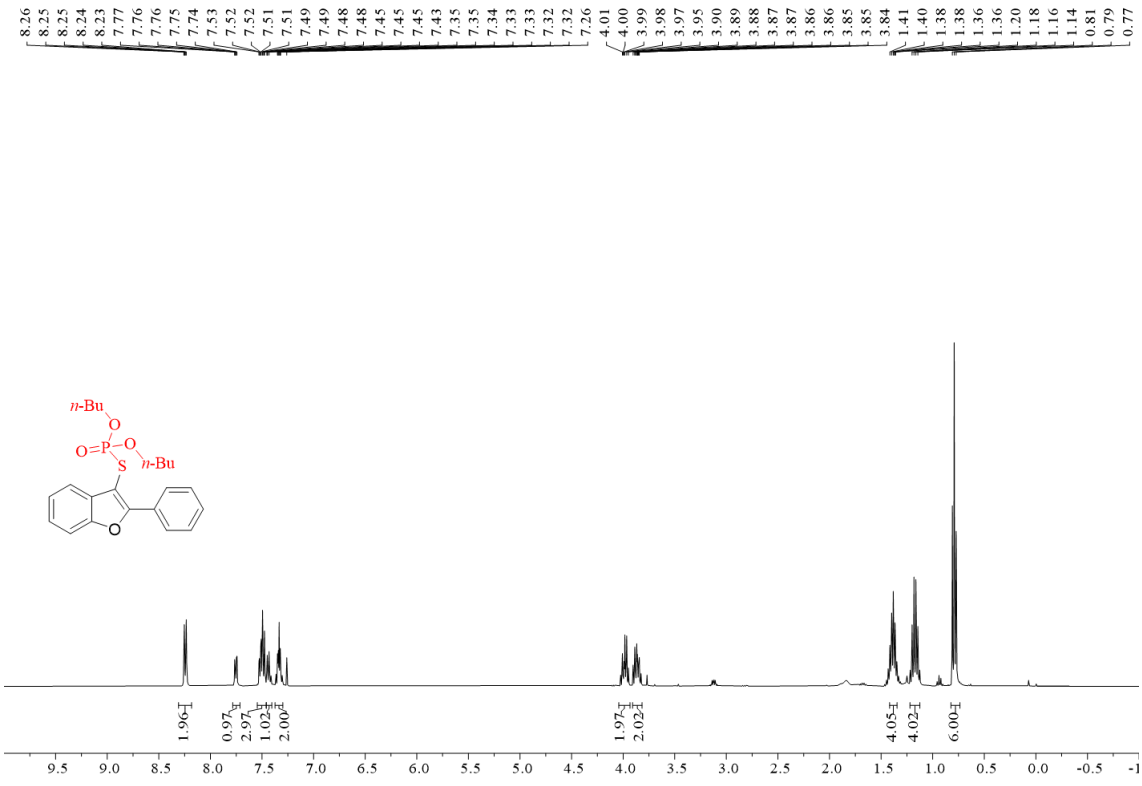
^{13}C NMR (101 MHz) Spectrum of **3v** in $CDCl_3$



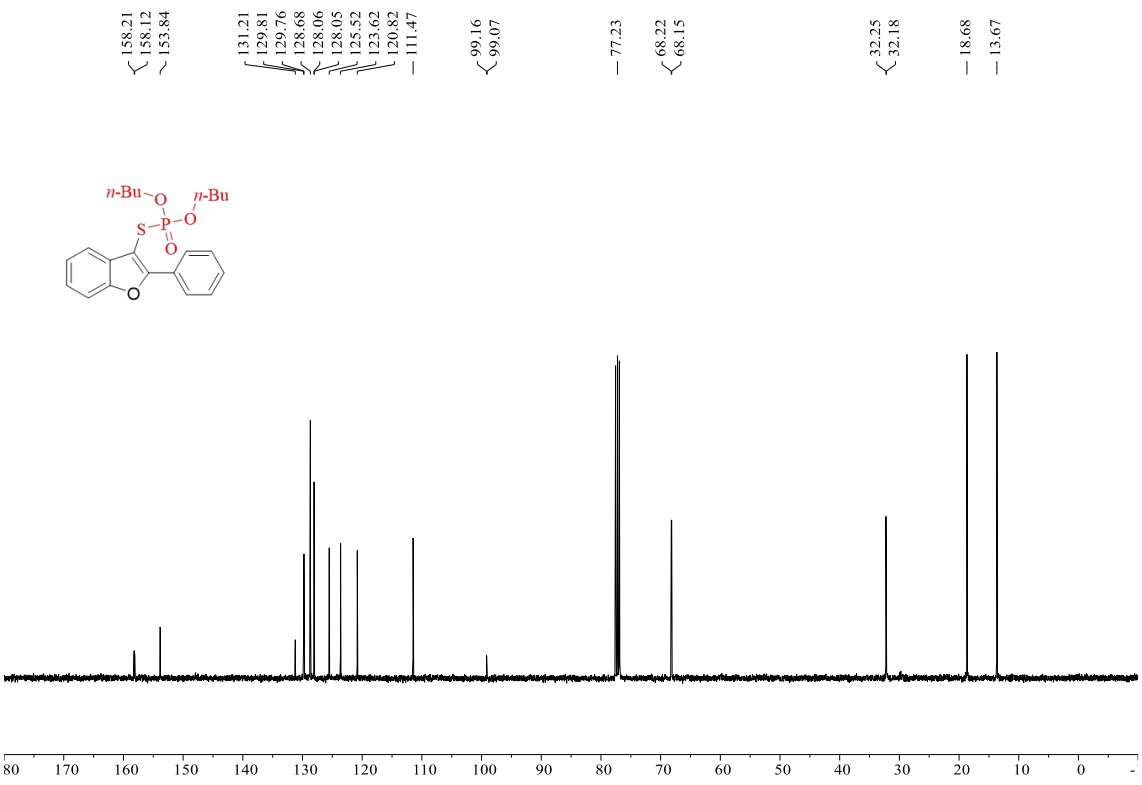
^{31}P NMR (162 MHz) Spectrum of **3v** in $CDCl_3$



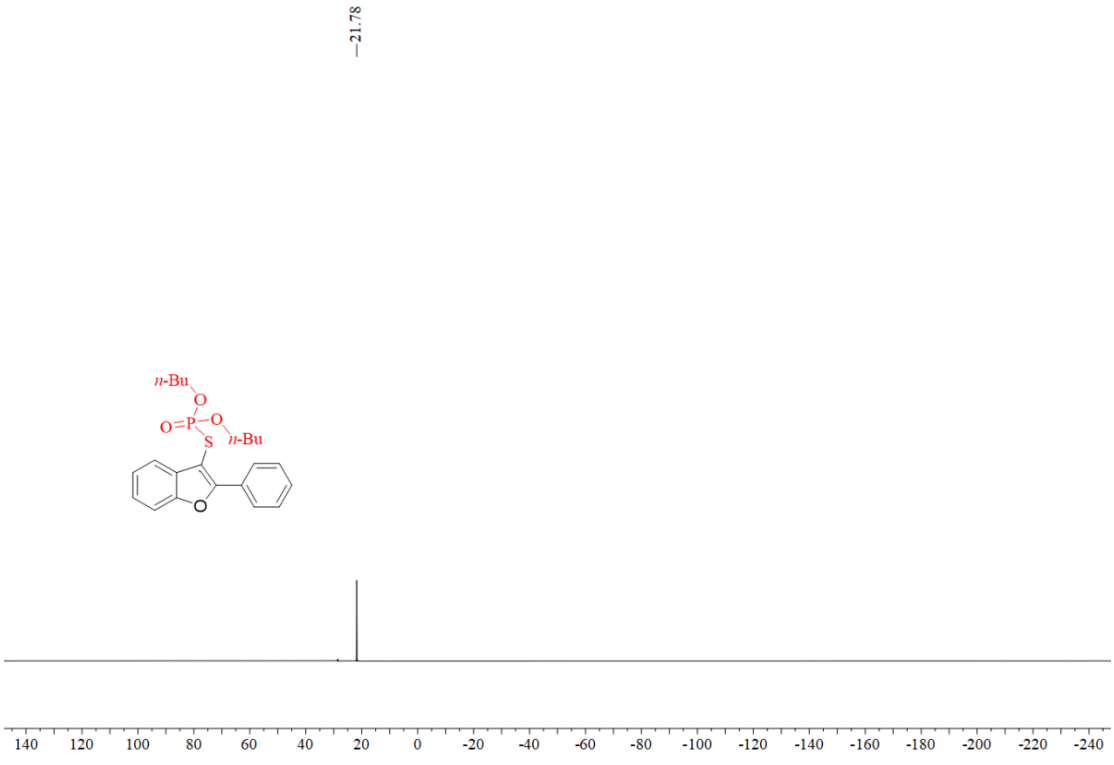
^{1}H NMR (400 MHz) Spectrum of **3w** in $CDCl_3$



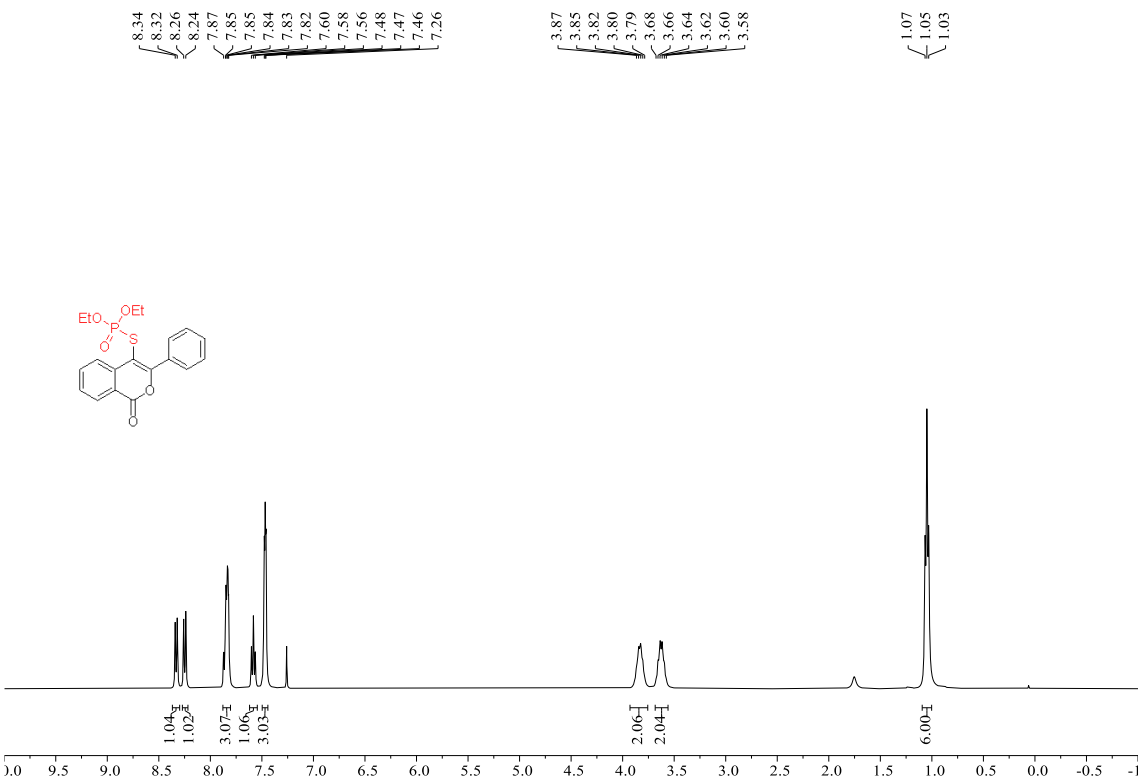
^{13}C NMR (101 MHz) Spectrum of **3w** in $CDCl_3$



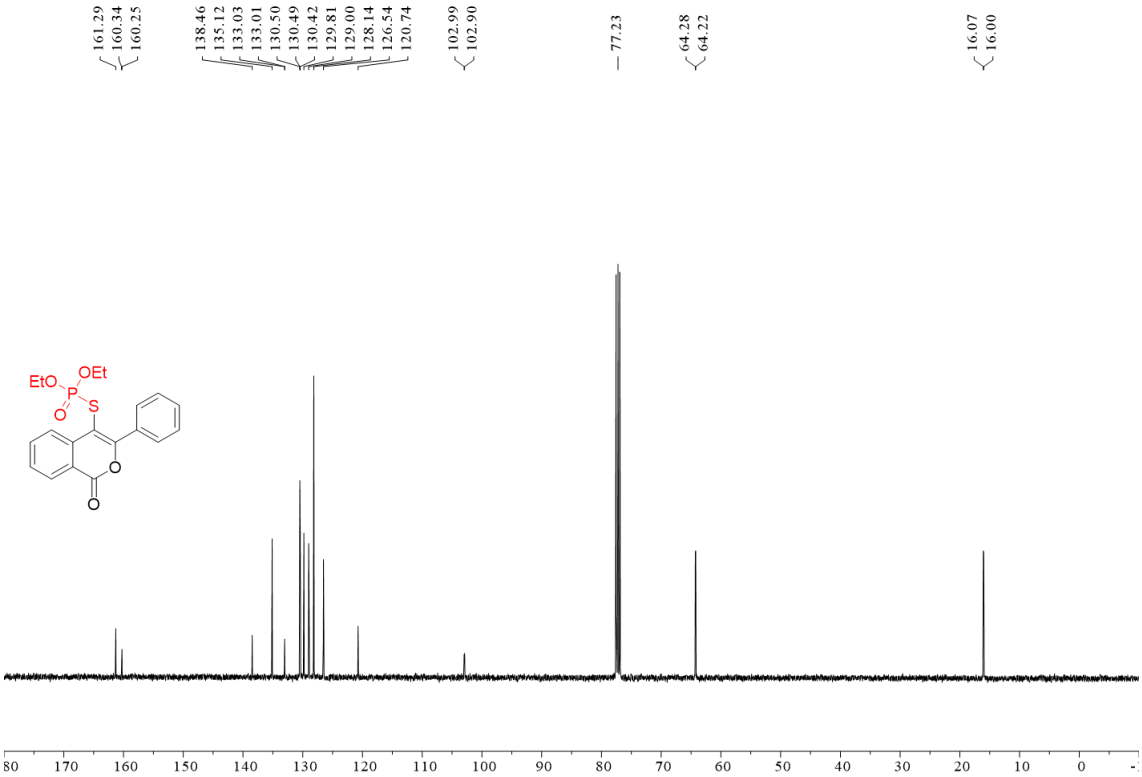
^{31}P NMR (162 MHz) Spectrum of **3w** in $CDCl_3$



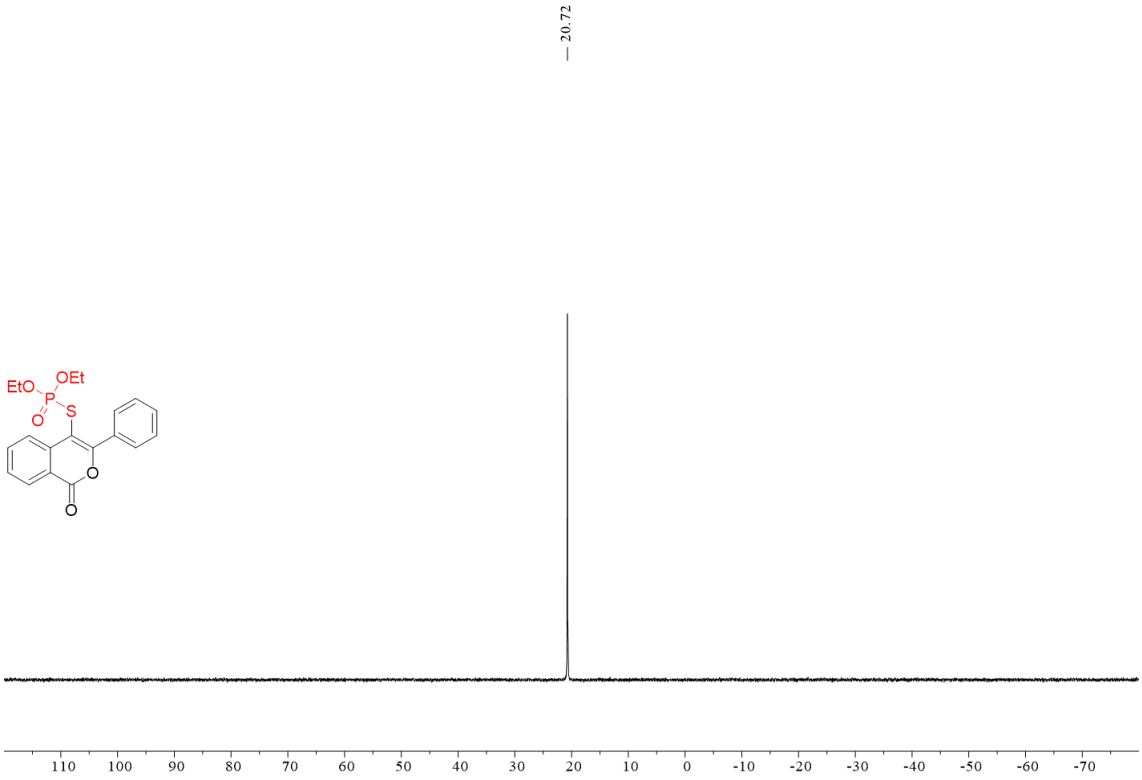
^{1}H NMR (400 MHz) Spectrum of **4a** in $CDCl_3$



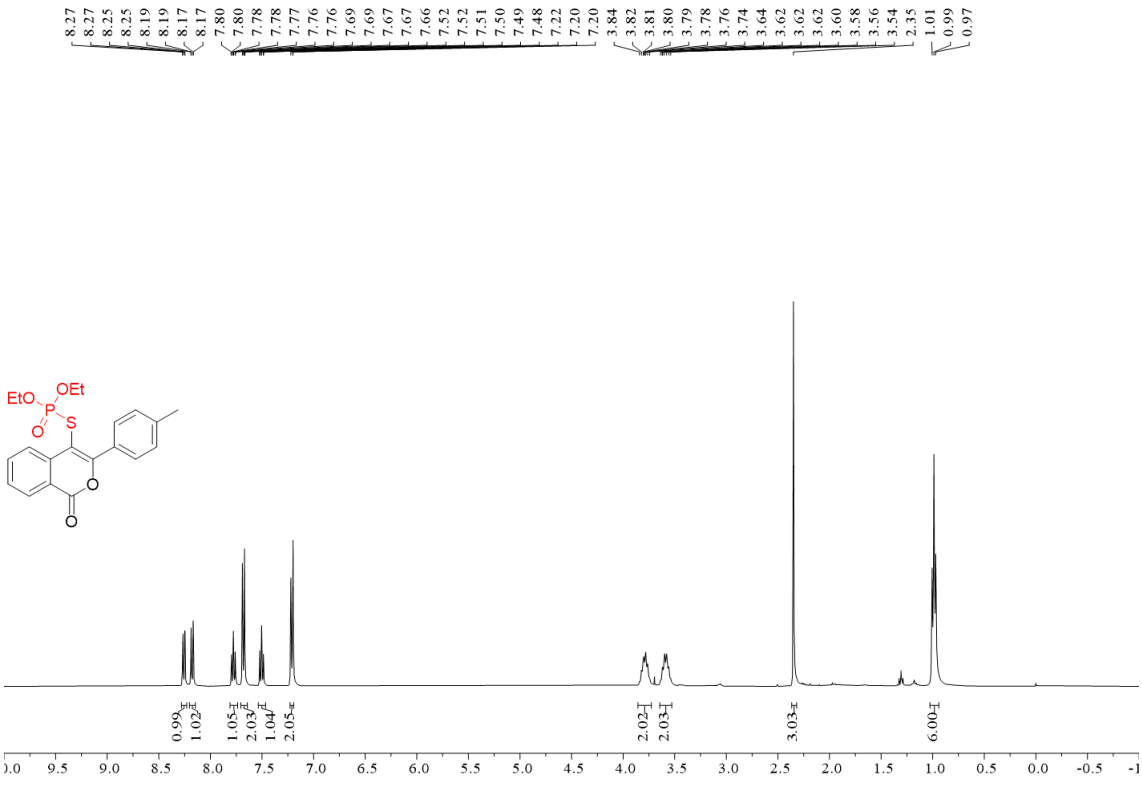
^{13}C NMR (101 MHz) Spectrum of **4a** in $CDCl_3$



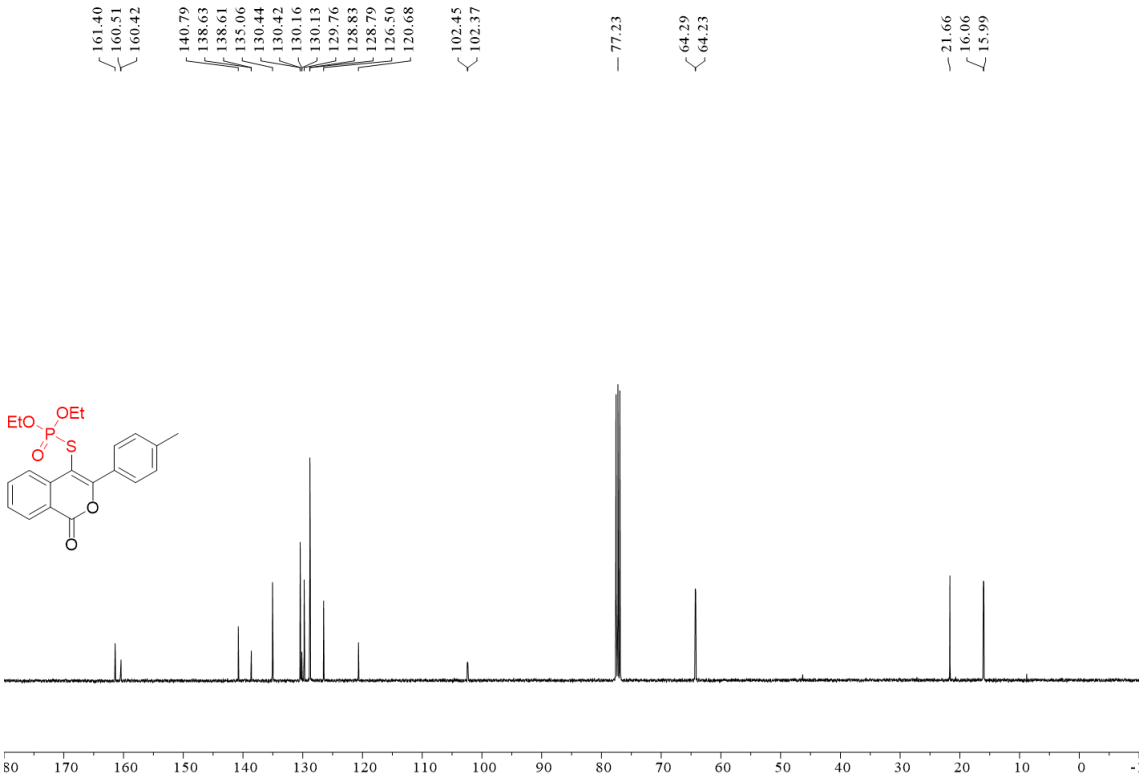
^{31}P NMR (162 MHz) Spectrum of **4a** in $CDCl_3$



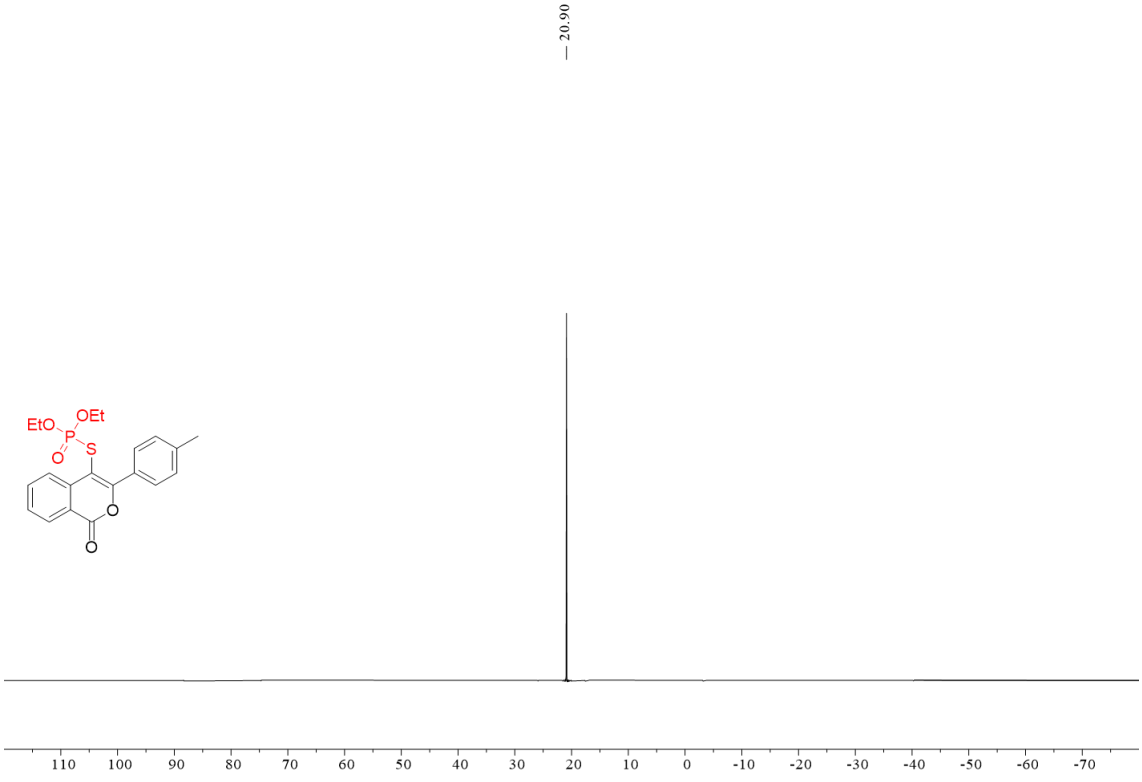
^{1}H NMR (400 MHz) Spectrum of **4b** in $CDCl_3$



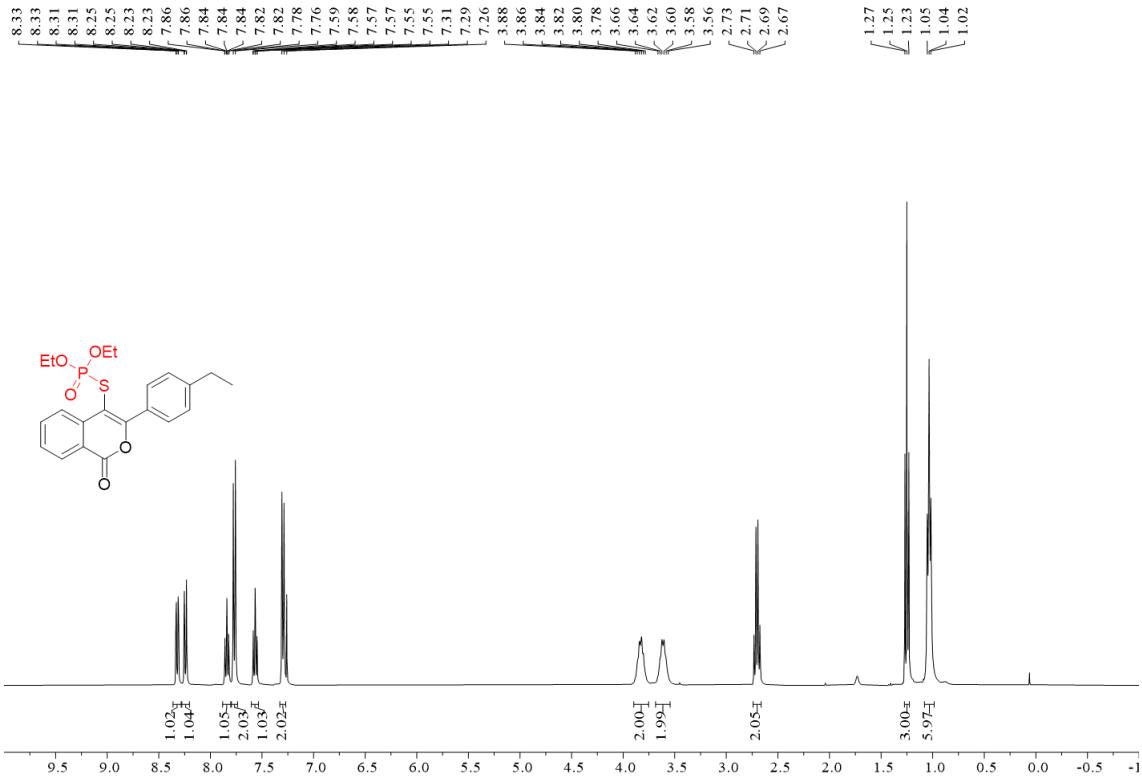
^{13}C NMR (101 MHz) Spectrum of **4b** in $CDCl_3$



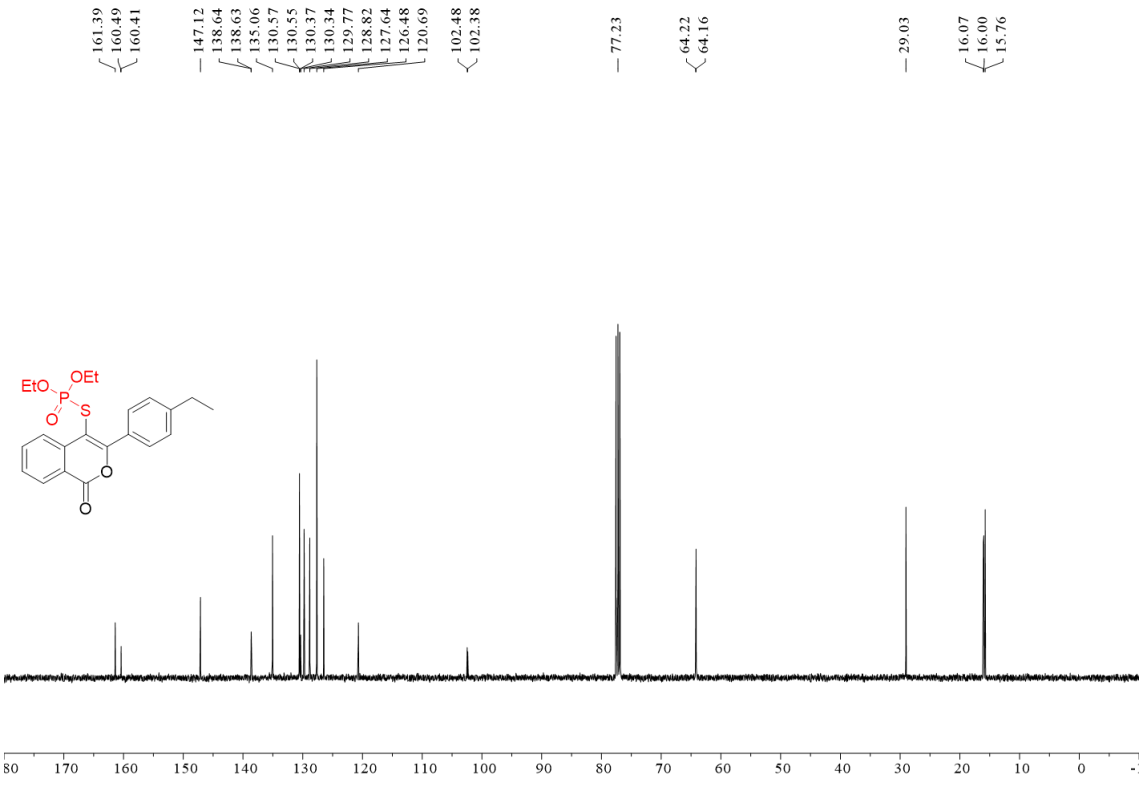
^{31}P NMR (162 MHz) Spectrum of **4b** in $CDCl_3$



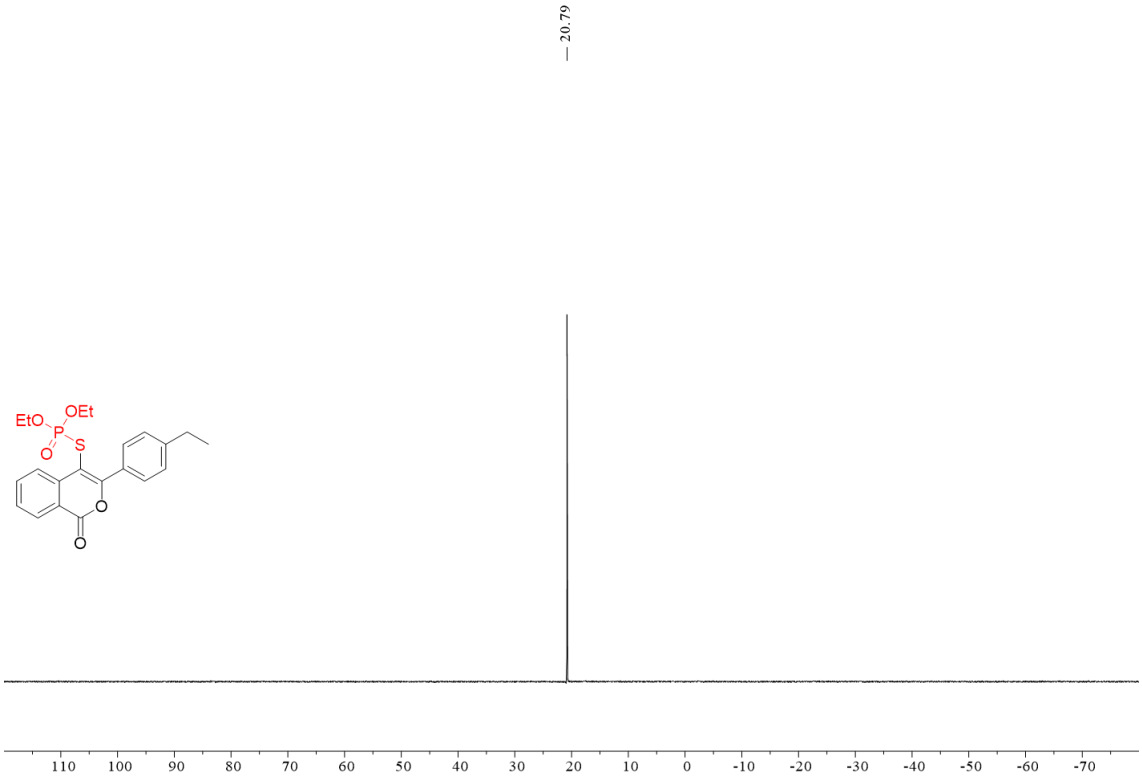
^{1}H NMR (400 MHz) Spectrum of **4c** in $CDCl_3$



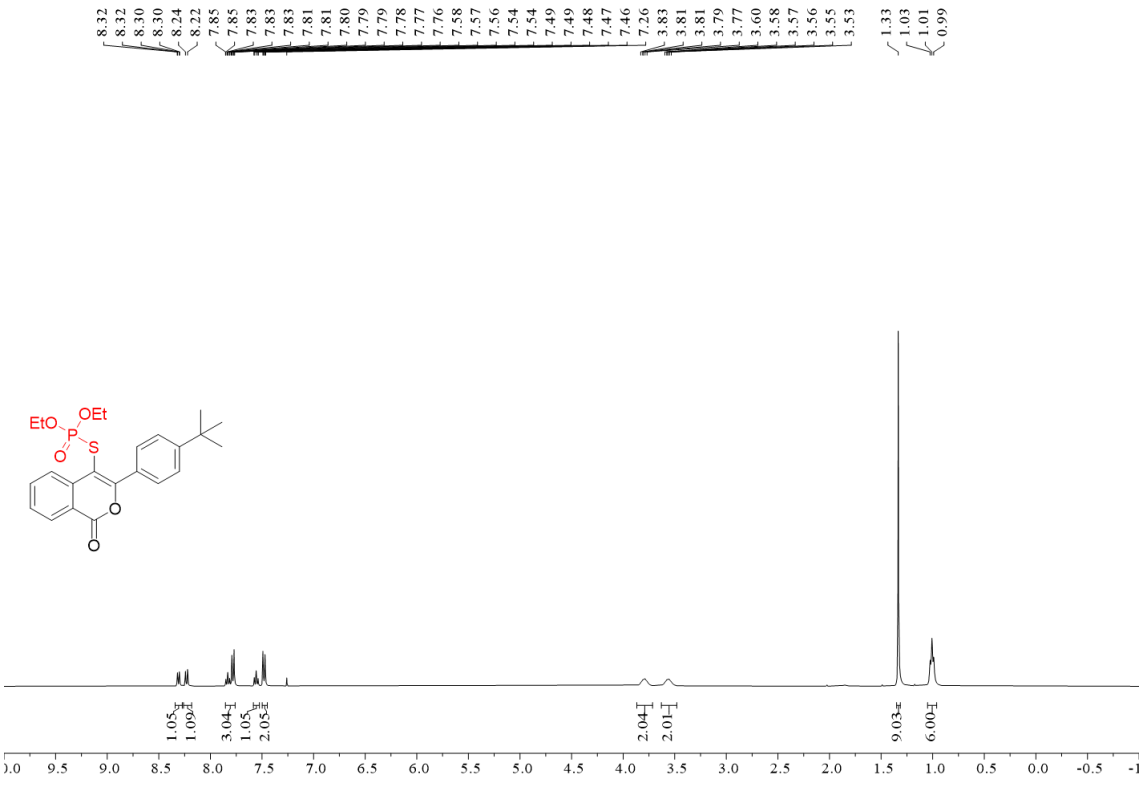
^{13}C NMR (101 MHz) Spectrum of **4c** in $CDCl_3$



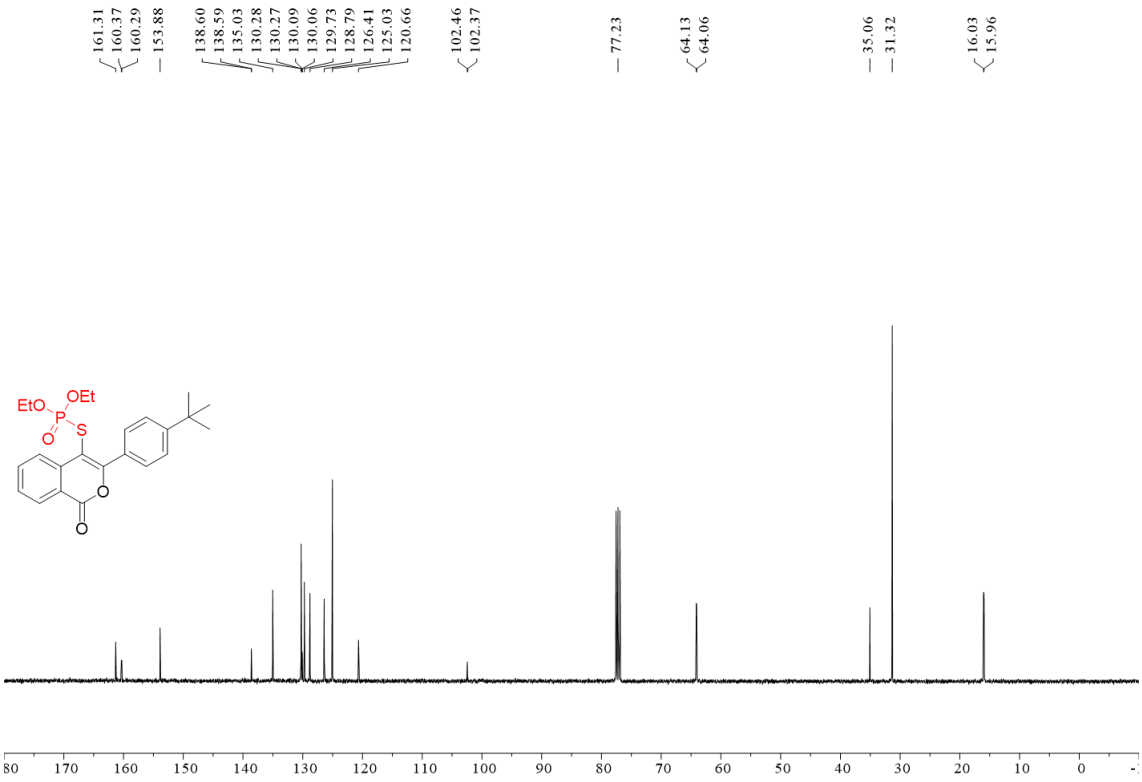
^{31}P NMR (162 MHz) Spectrum of **4c** in $CDCl_3$



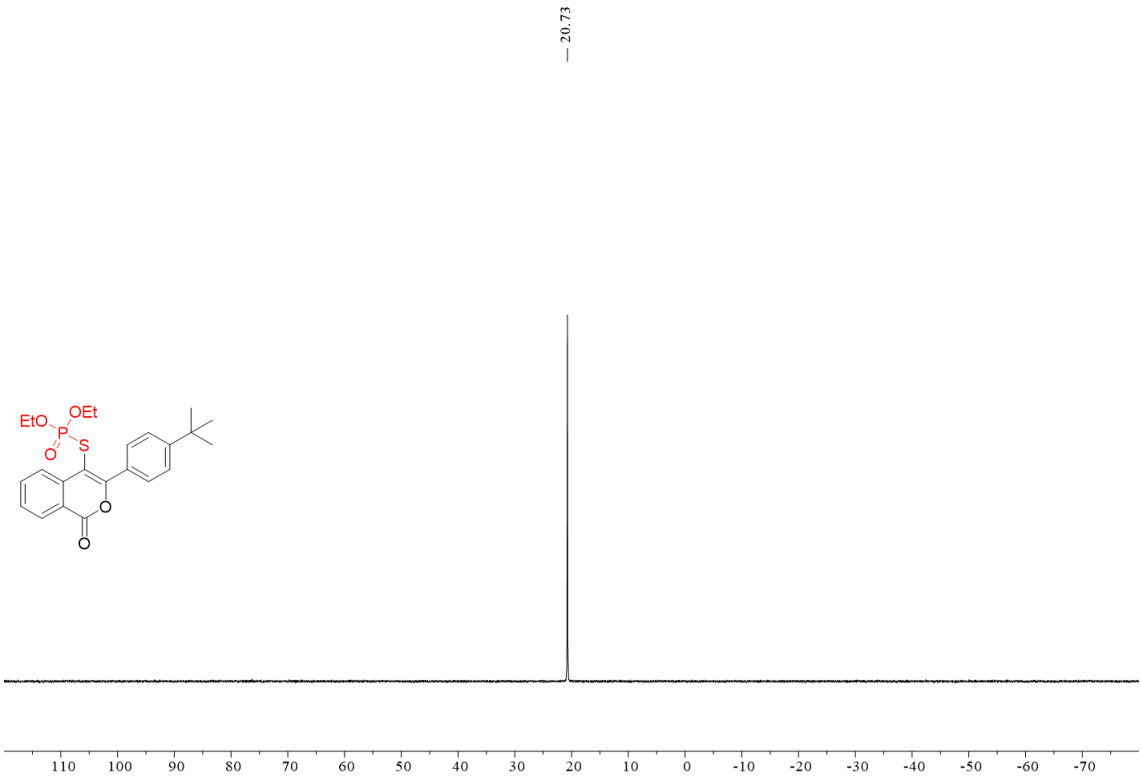
^{1}H NMR (400 MHz) Spectrum of **4d** in $CDCl_3$



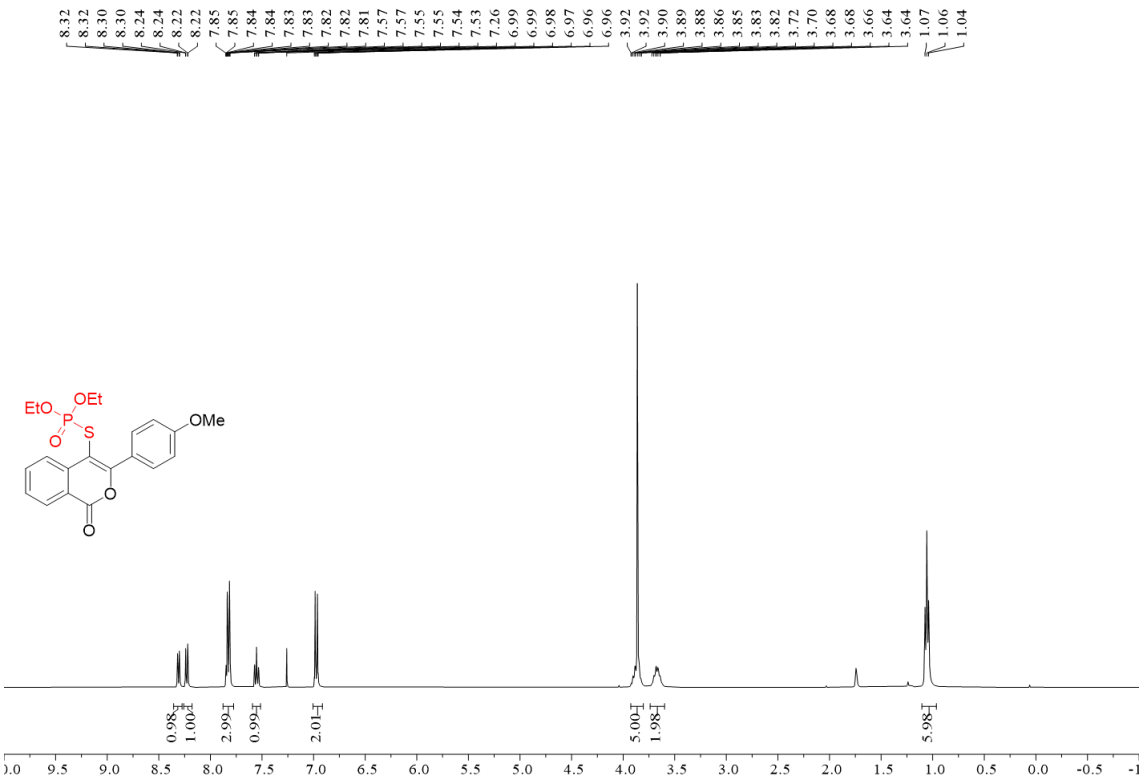
^{13}C NMR (101 MHz) Spectrum of **4d** in $CDCl_3$



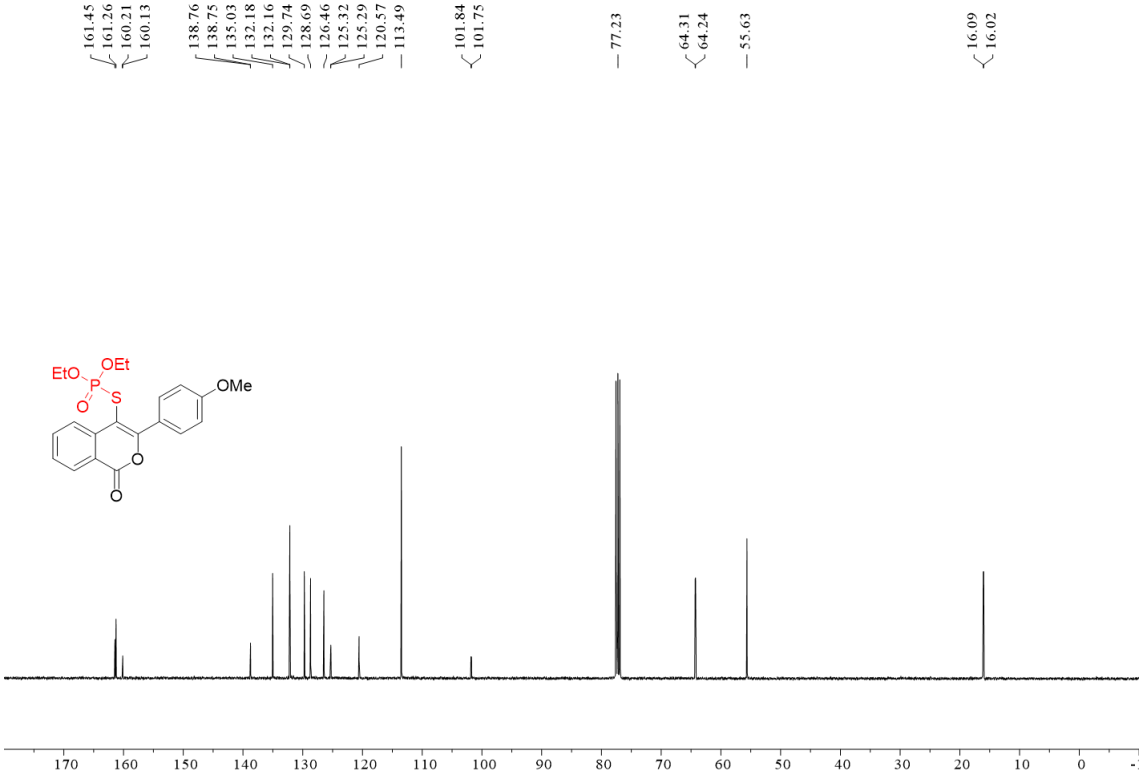
^{31}P NMR (162 MHz) Spectrum of **4d** in $CDCl_3$



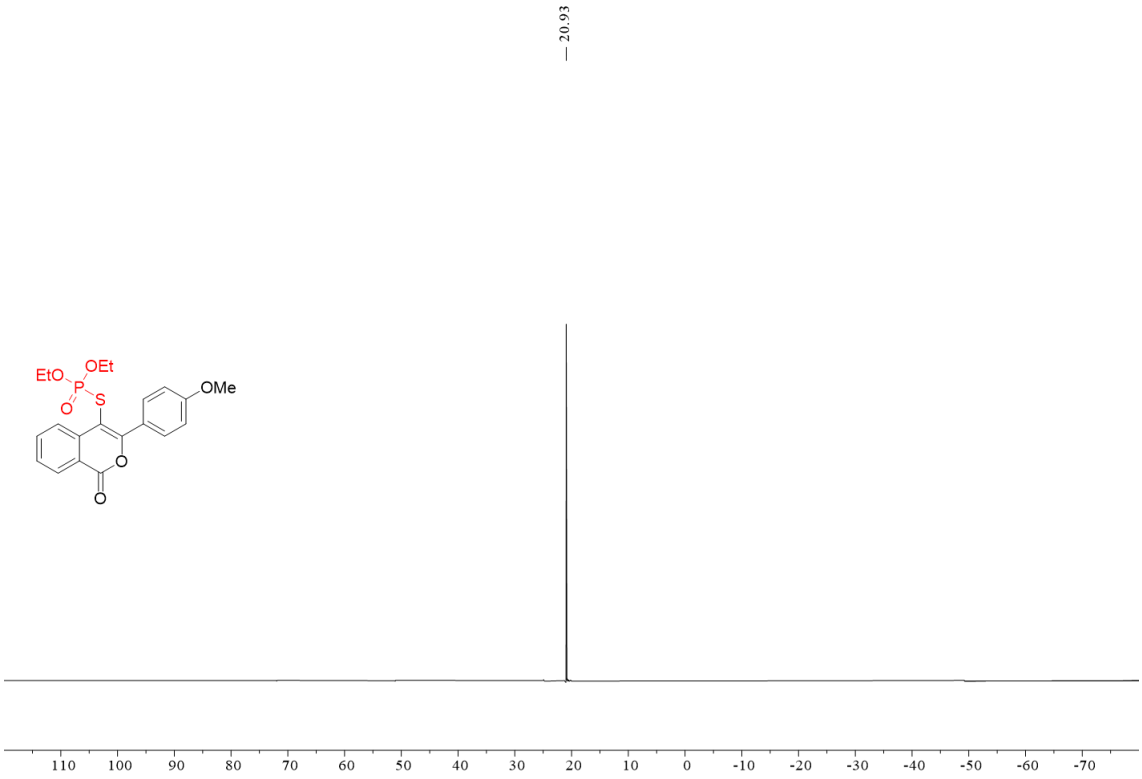
^{1}H NMR (400 MHz) Spectrum of **4e** in $CDCl_3$



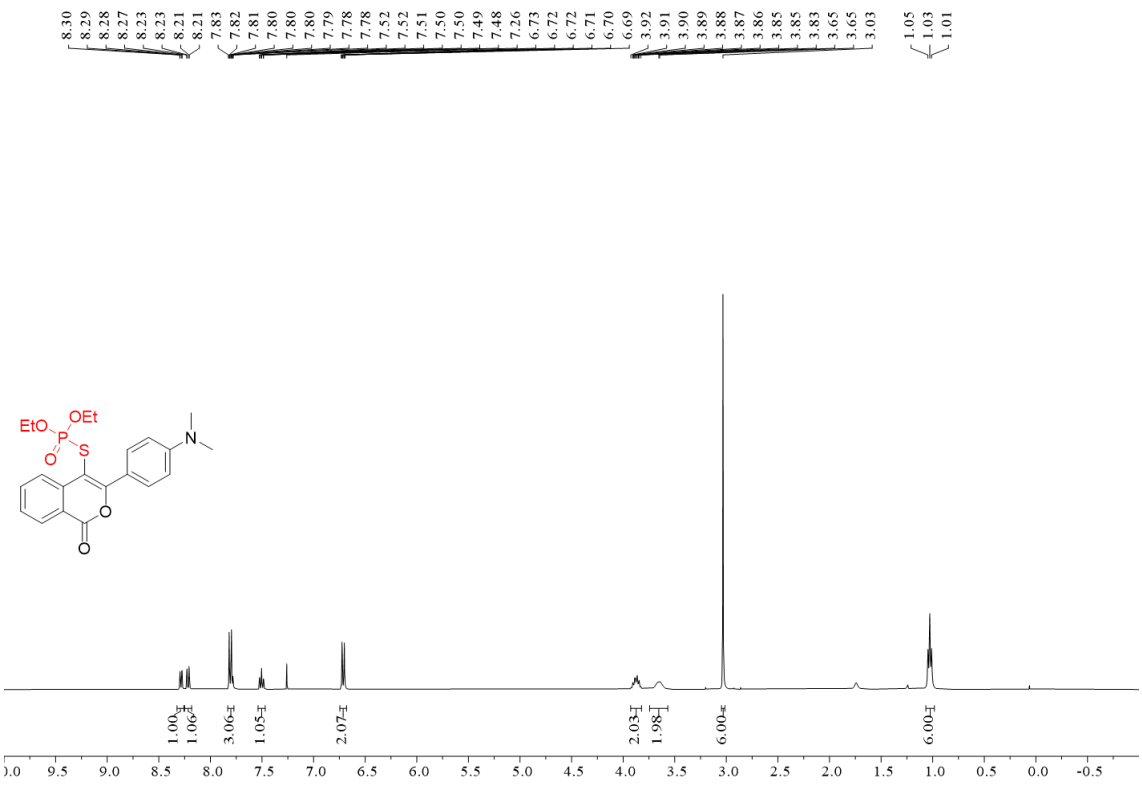
^{13}C NMR (101 MHz) Spectrum of **4e** in $CDCl_3$



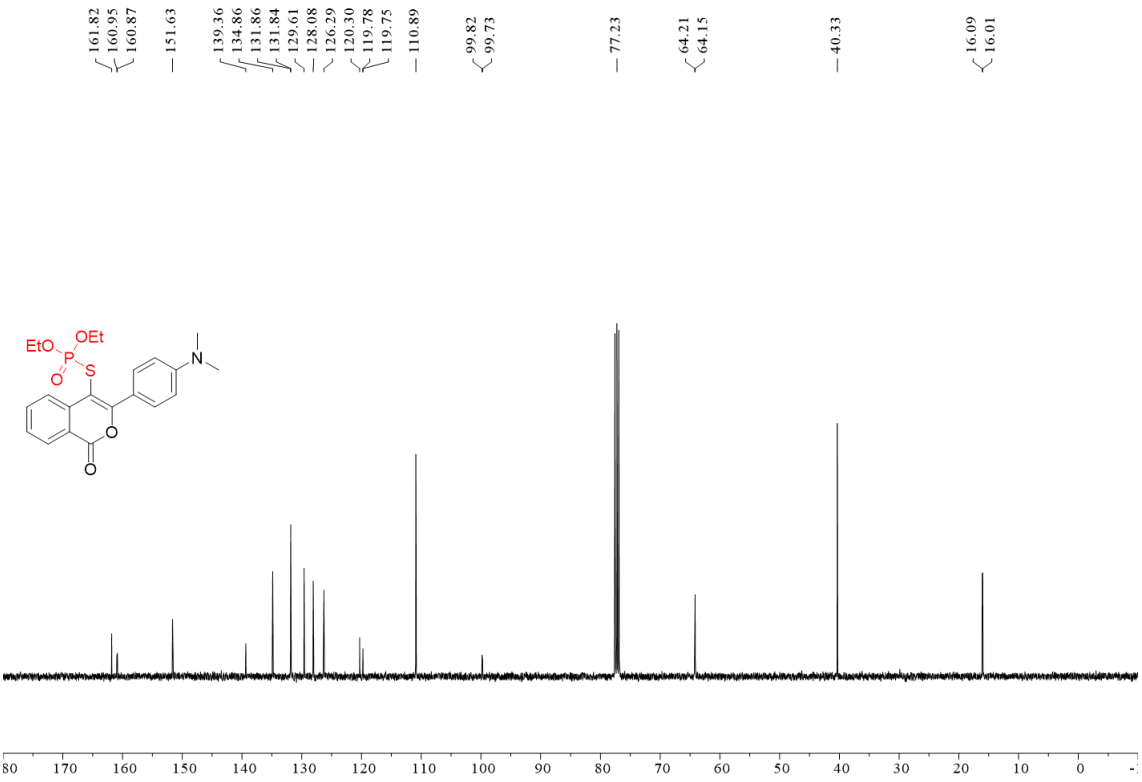
^{31}P NMR (162 MHz) Spectrum of **4e** in $CDCl_3$



^{1}H NMR (400 MHz) Spectrum of **4f** in $CDCl_3$

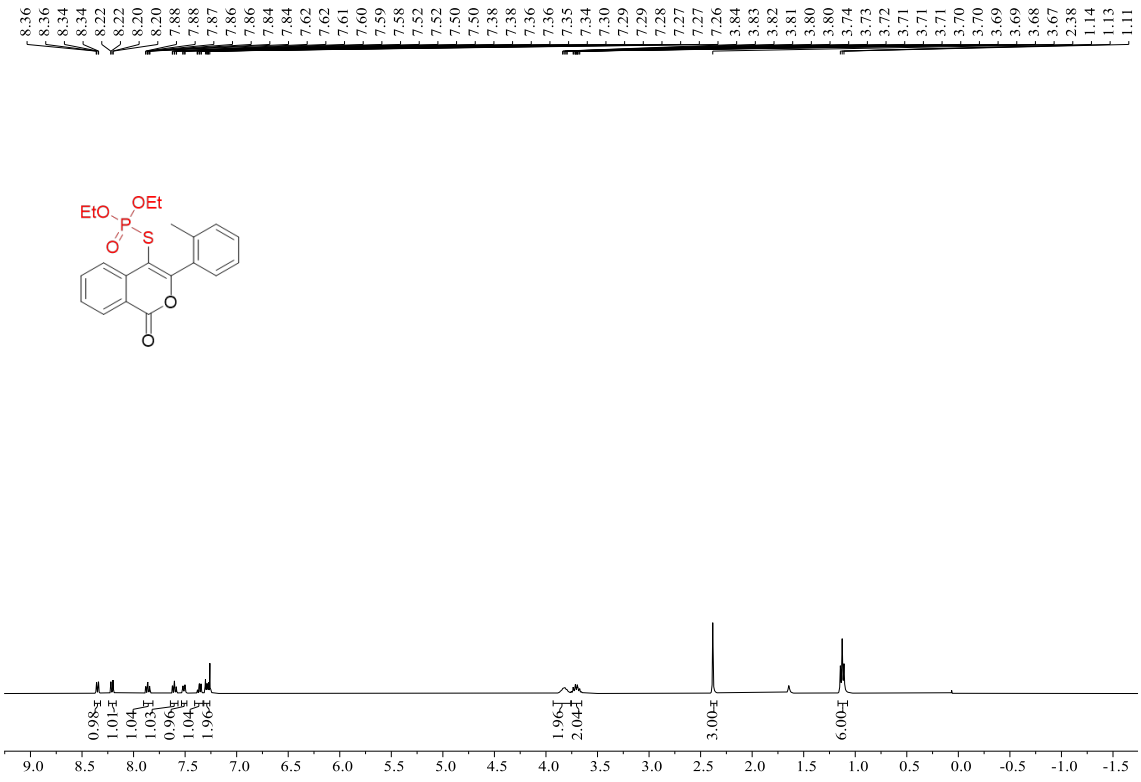


^{13}C NMR (101 MHz) Spectrum of **4f** in $CDCl_3$

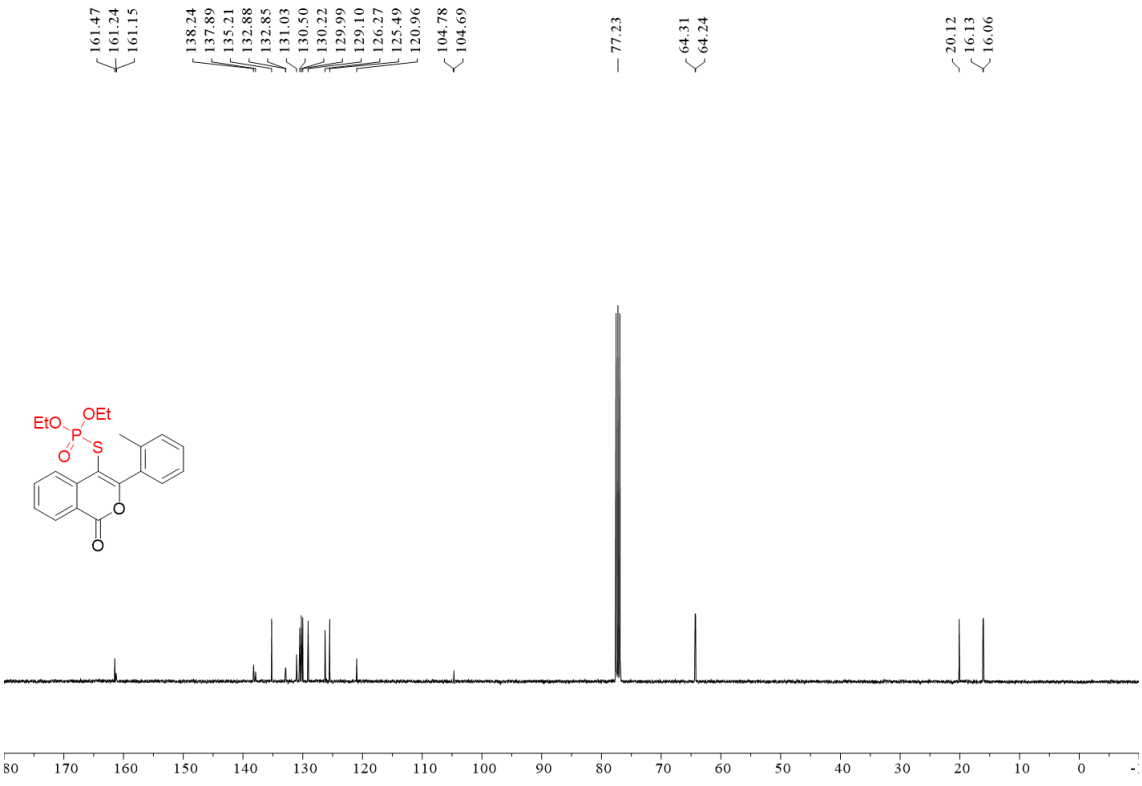


^{31}P NMR (162 MHz) Spectrum of **4f** in $CDCl_3$

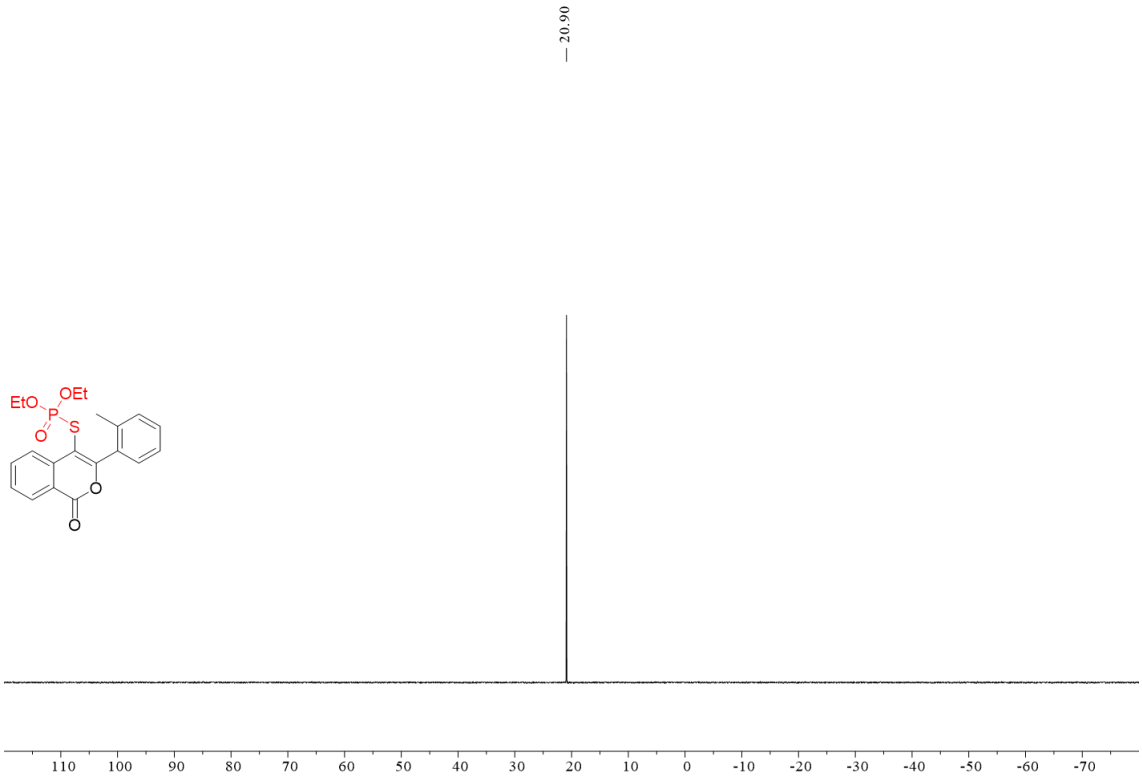
^{1}H NMR (400 MHz) Spectrum of **4g** in $CDCl_3$



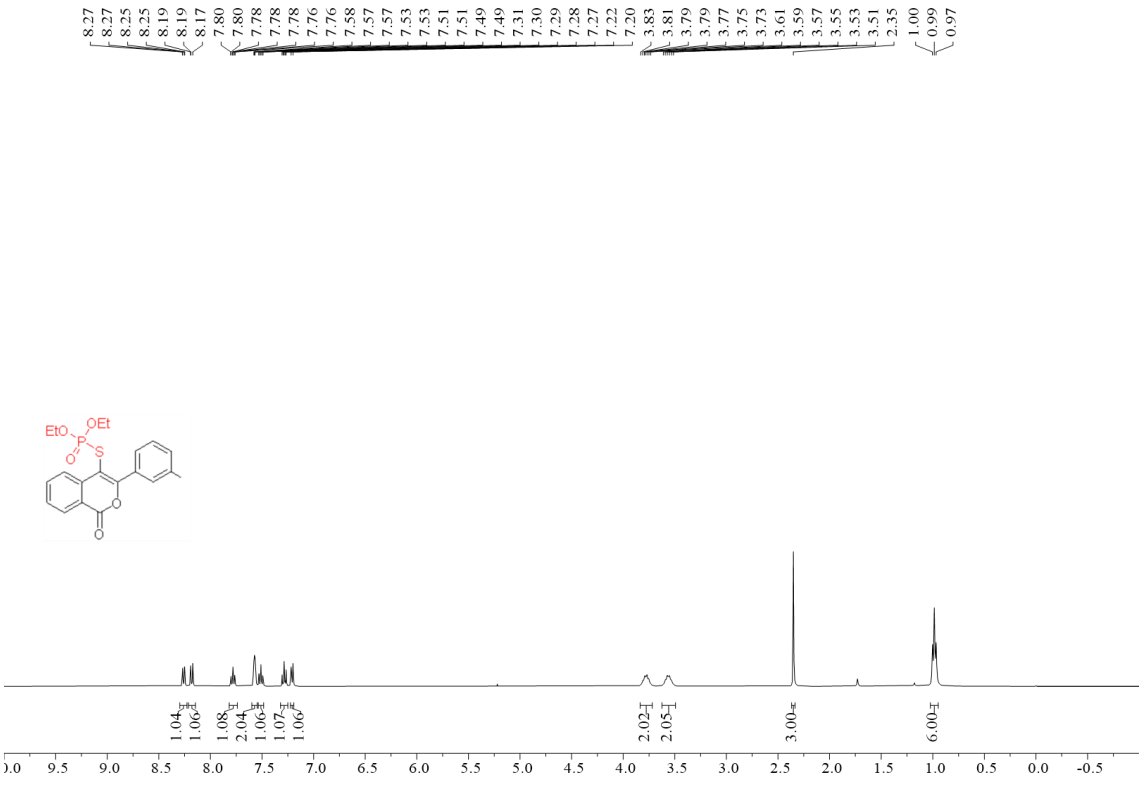
^{13}C NMR (101 MHz) Spectrum of **4g** in $CDCl_3$



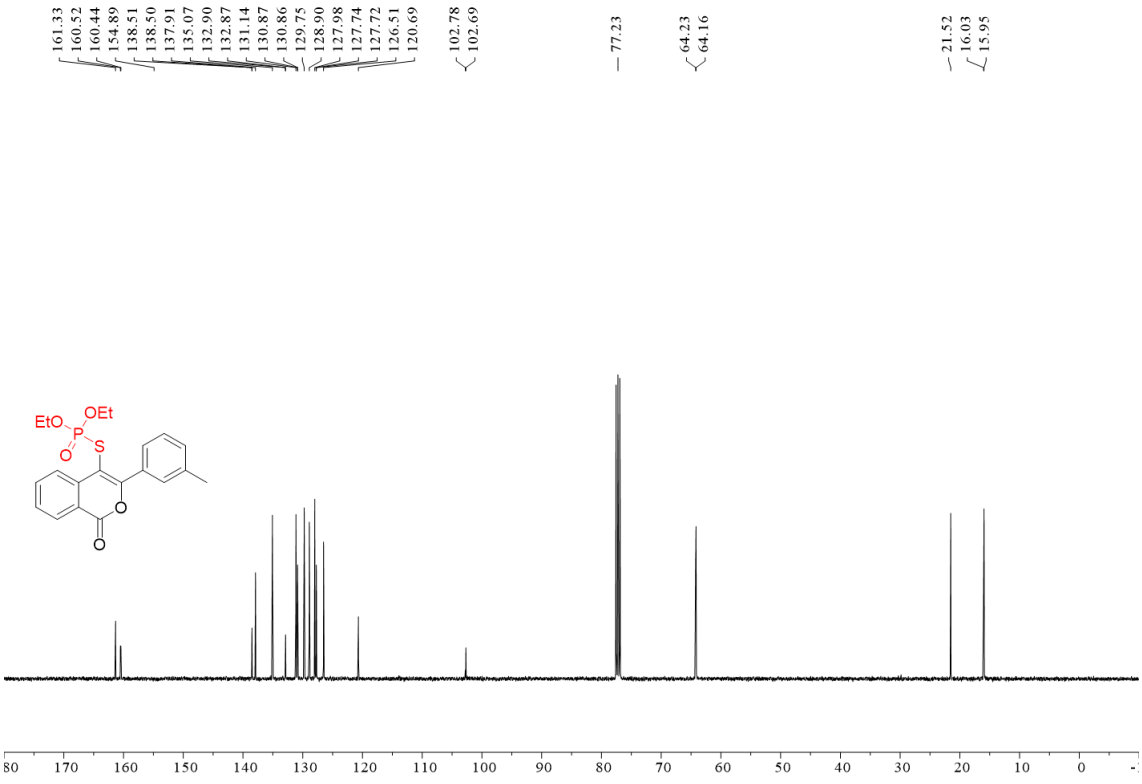
^{31}P NMR (162 MHz) Spectrum of **4g** in $CDCl_3$



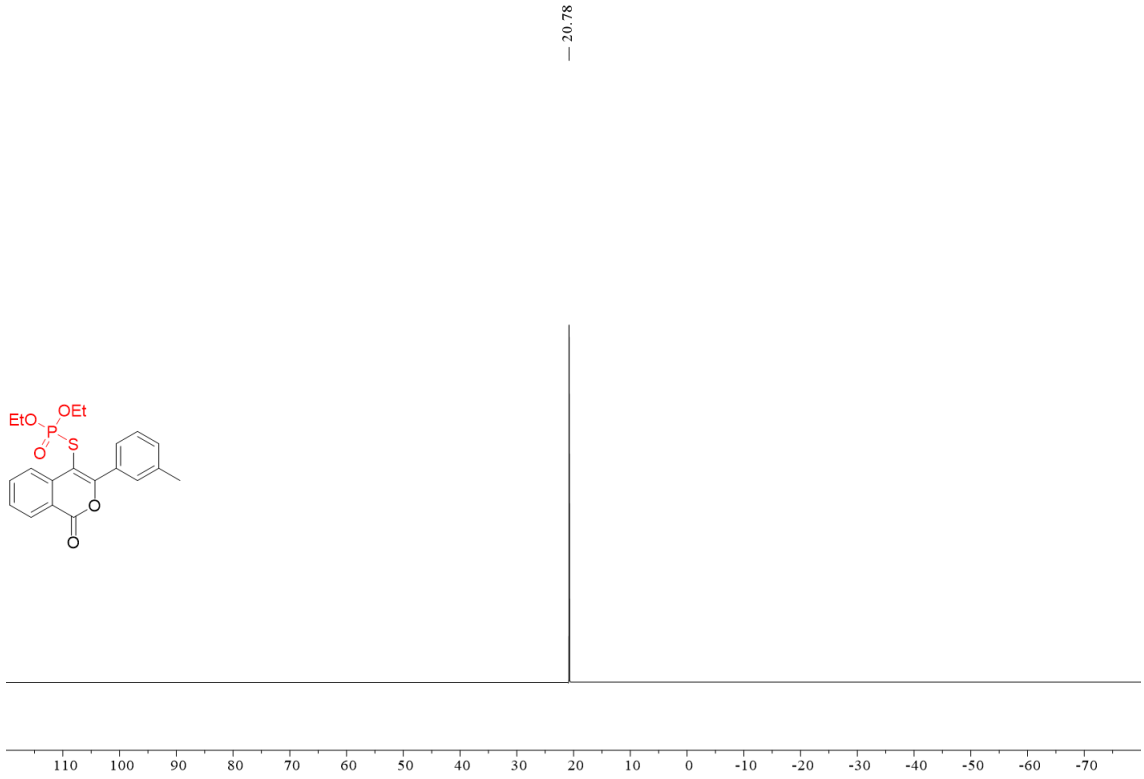
^{1}H NMR (400 MHz) Spectrum of **4h** in $CDCl_3$



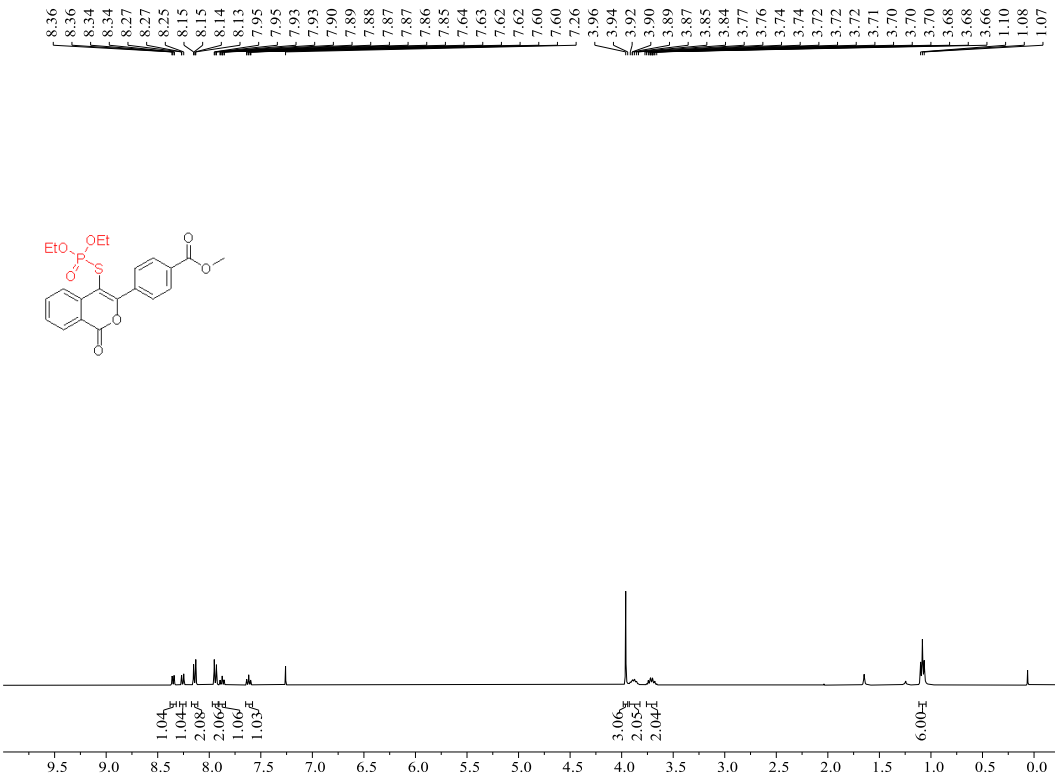
^{13}C NMR (101 MHz) Spectrum of **4h** in $CDCl_3$



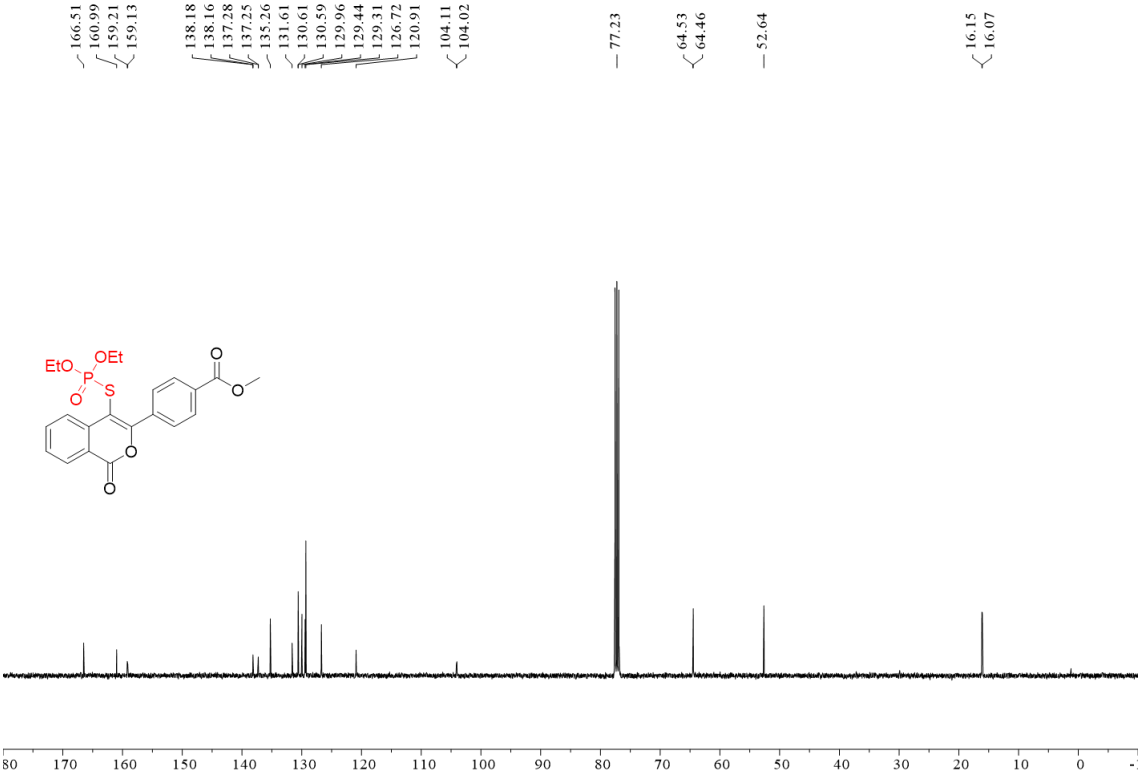
^{31}P NMR (162 MHz) Spectrum of **4h** in $CDCl_3$



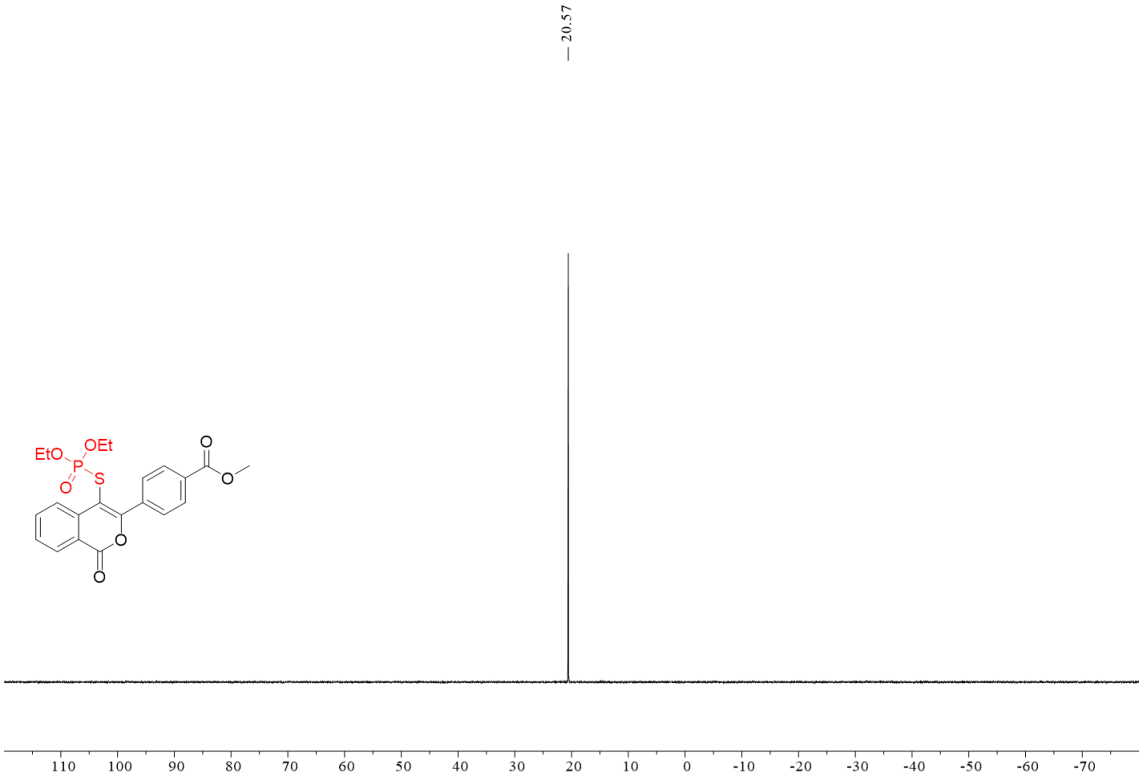
^{1}H NMR (400 MHz) Spectrum of **4i** in $CDCl_3$



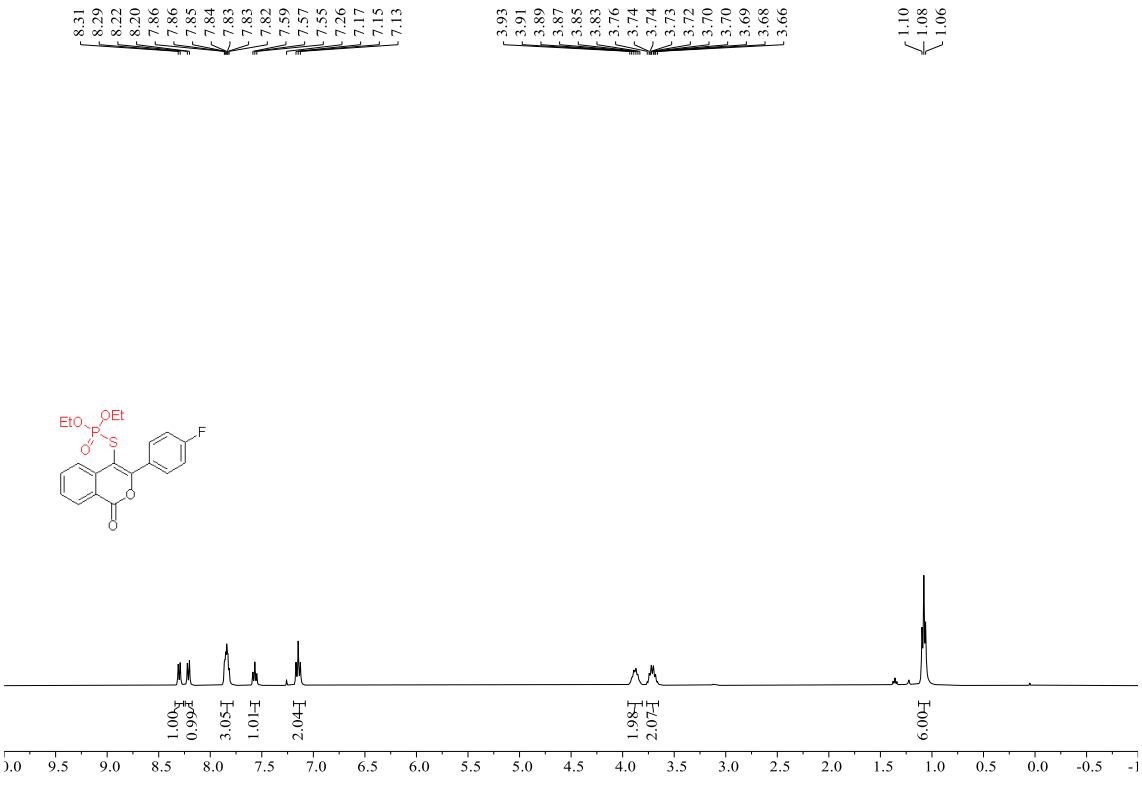
^{13}C NMR (101 MHz) Spectrum of **4i** in $CDCl_3$



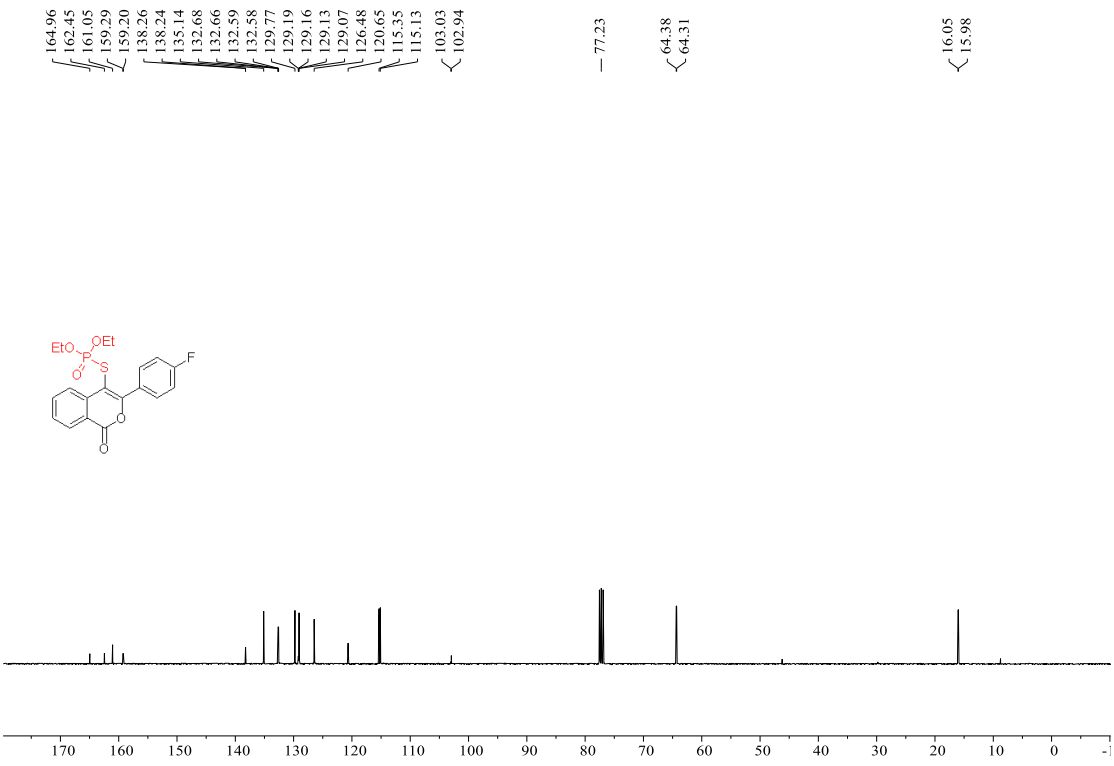
^{31}P NMR (162 MHz) Spectrum of **4i** in $CDCl_3$



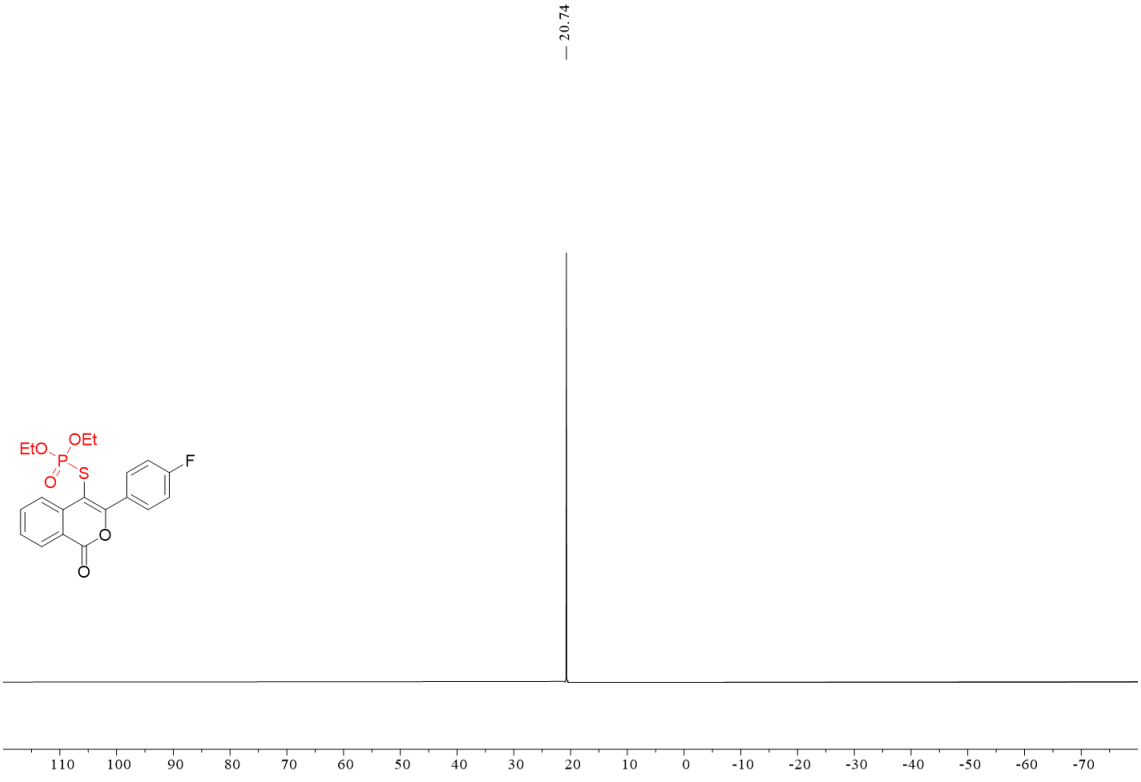
^{1}H NMR (400 MHz) Spectrum of **4j** in $CDCl_3$



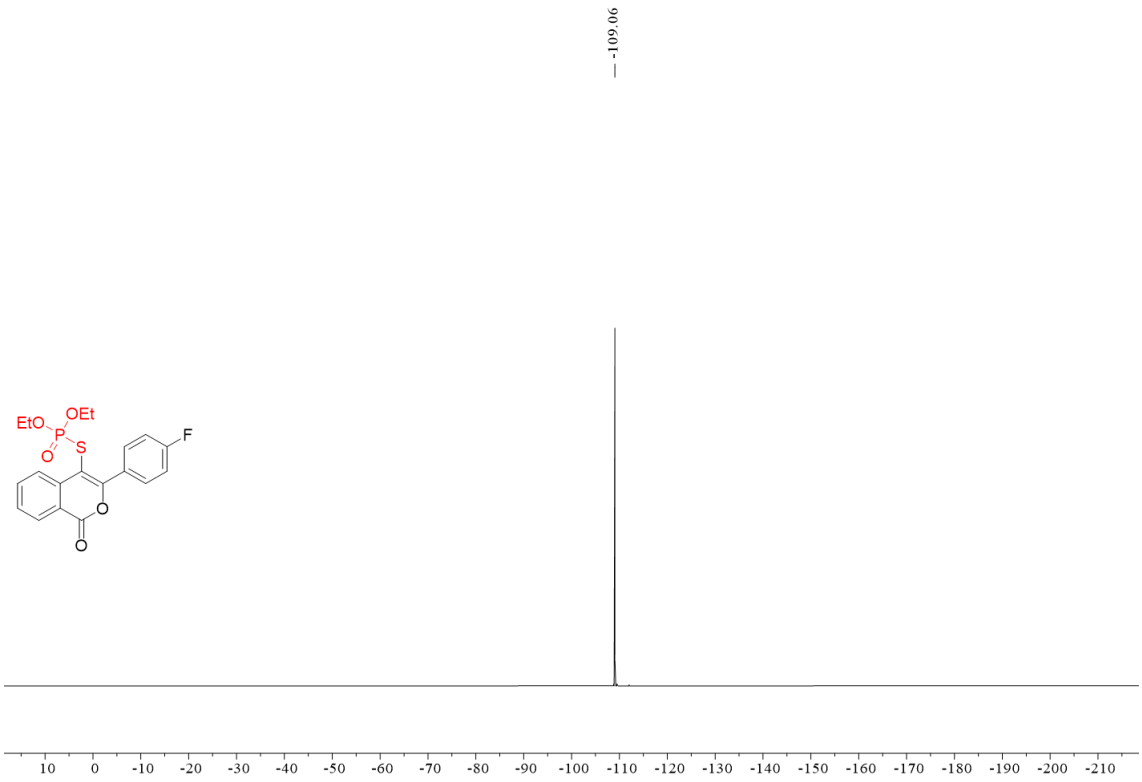
^{13}C NMR (101 MHz) Spectrum of **4j** in $CDCl_3$



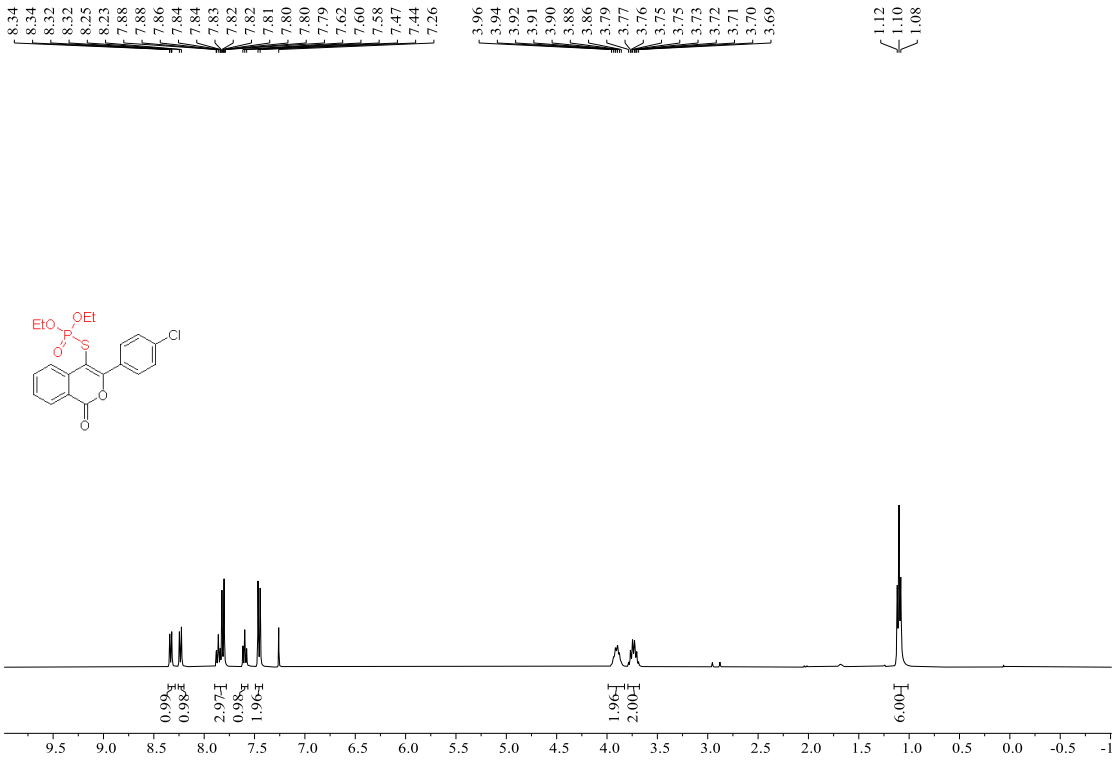
^{31}P NMR (162 MHz) Spectrum of **4j** in $CDCl_3$



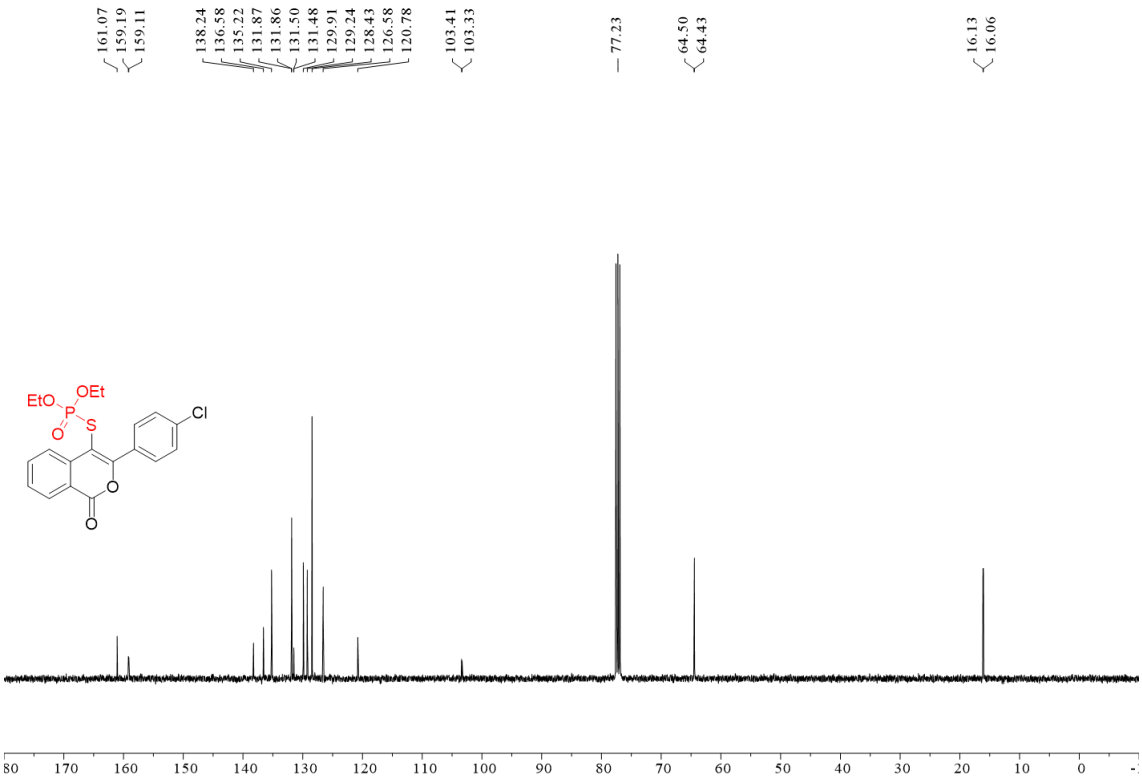
^{19}F NMR (376 MHz) Spectrum of **4j** in $CDCl_3$



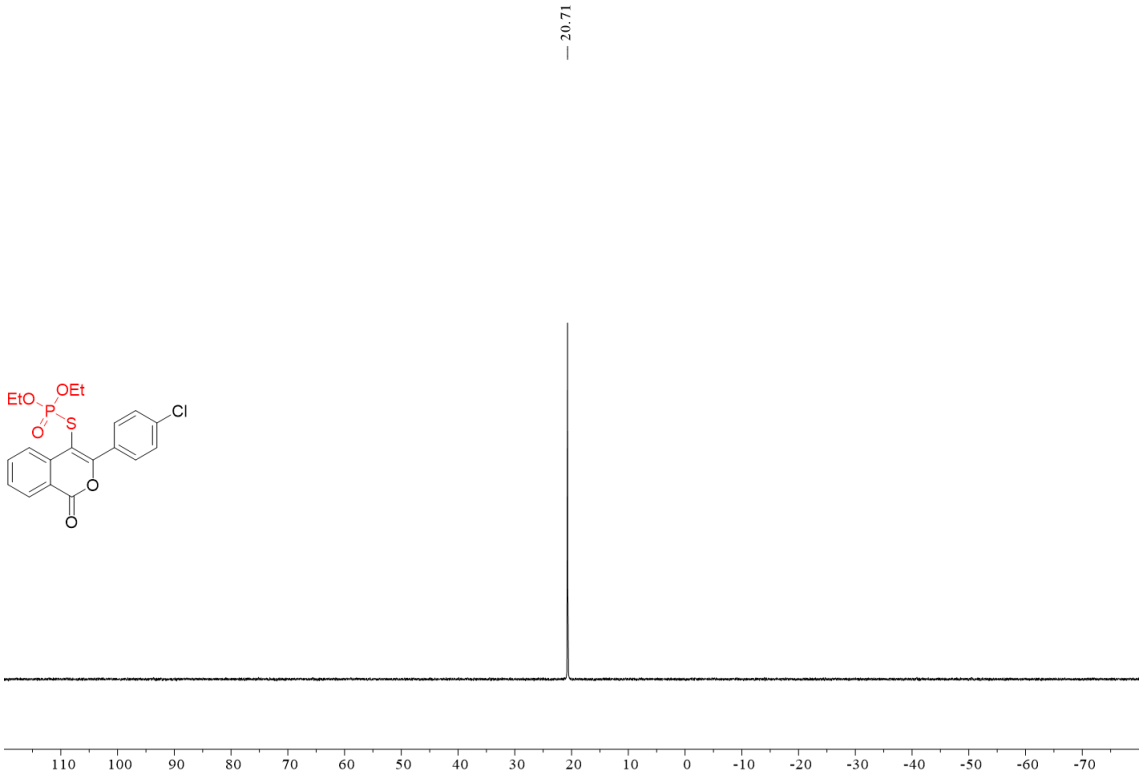
^{1}H NMR (400 MHz) Spectrum of **4k** in $CDCl_3$



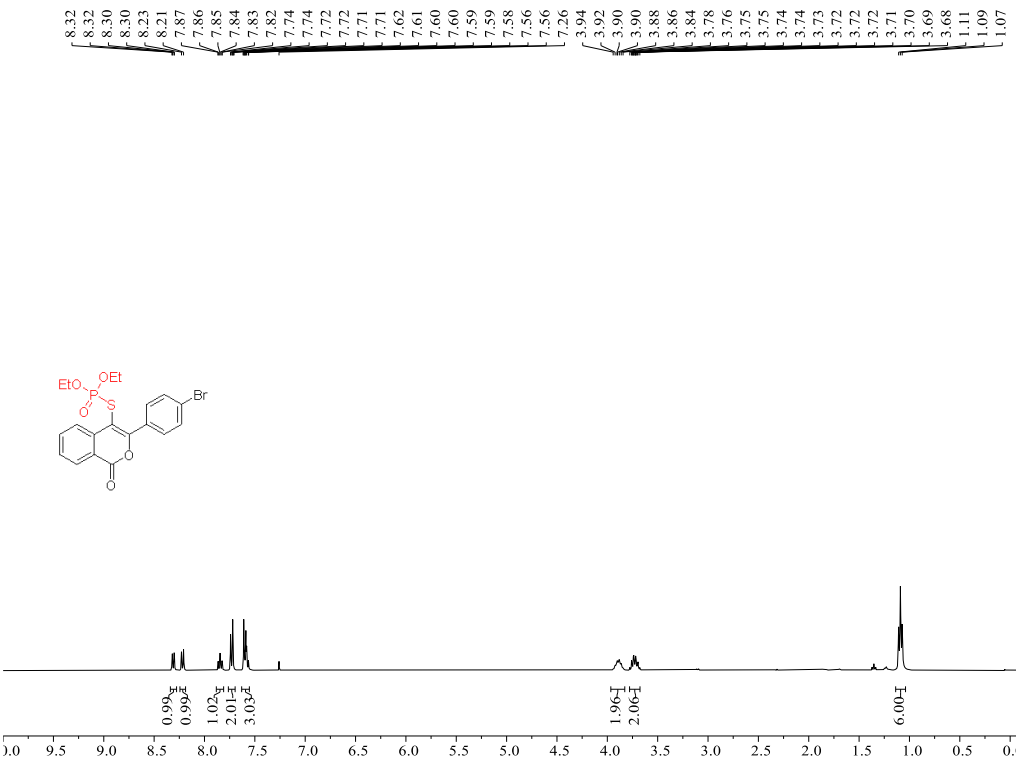
^{13}C NMR (101 MHz) Spectrum of **4k** in $CDCl_3$



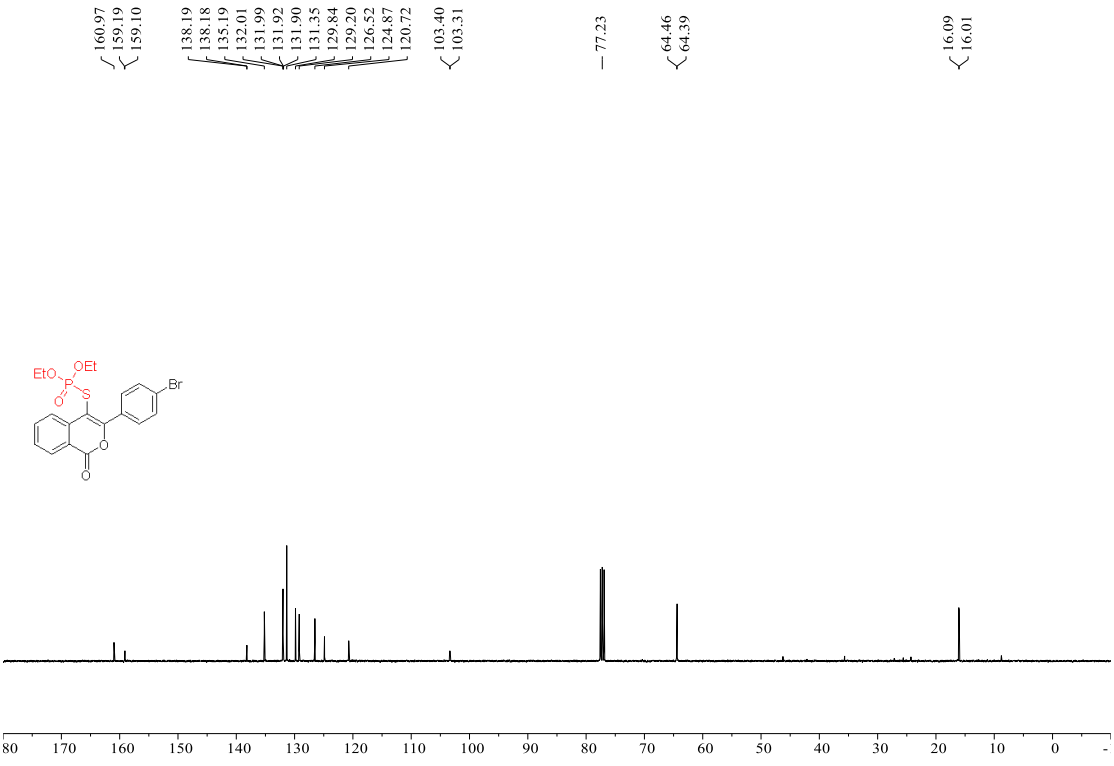
^{31}P NMR (162 MHz) Spectrum of **4k** in $CDCl_3$



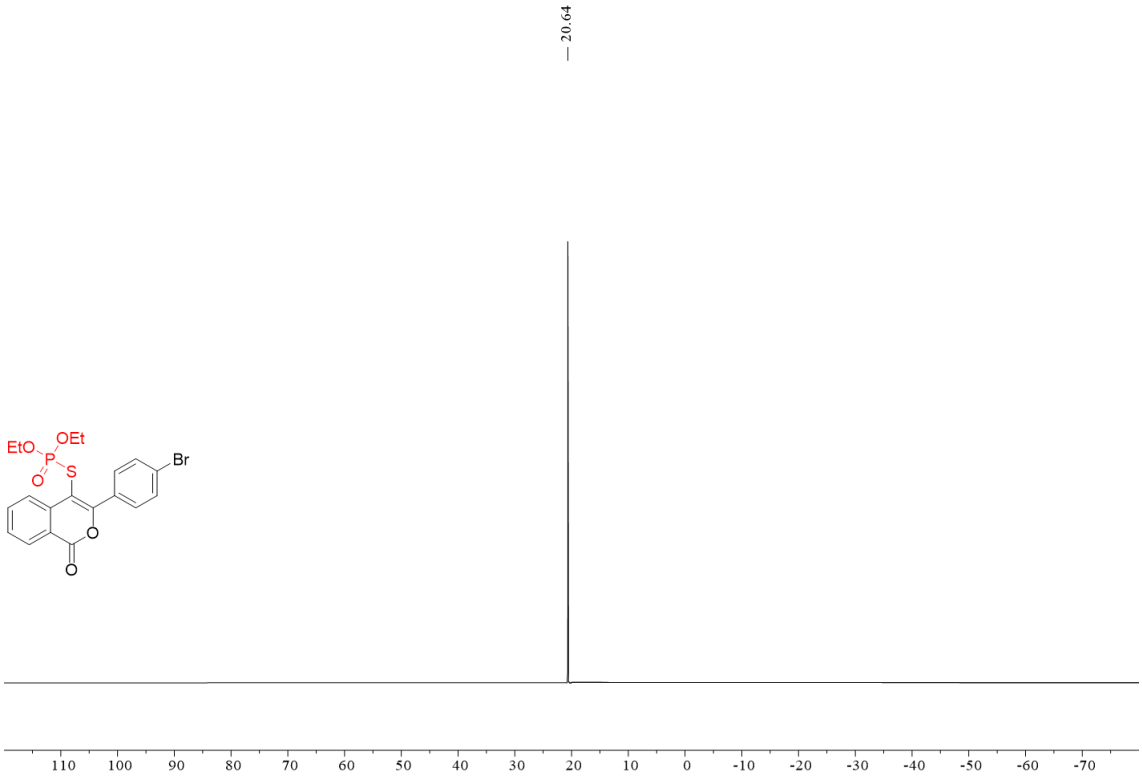
^{1}H NMR (400 MHz) Spectrum of **4l** in $CDCl_3$



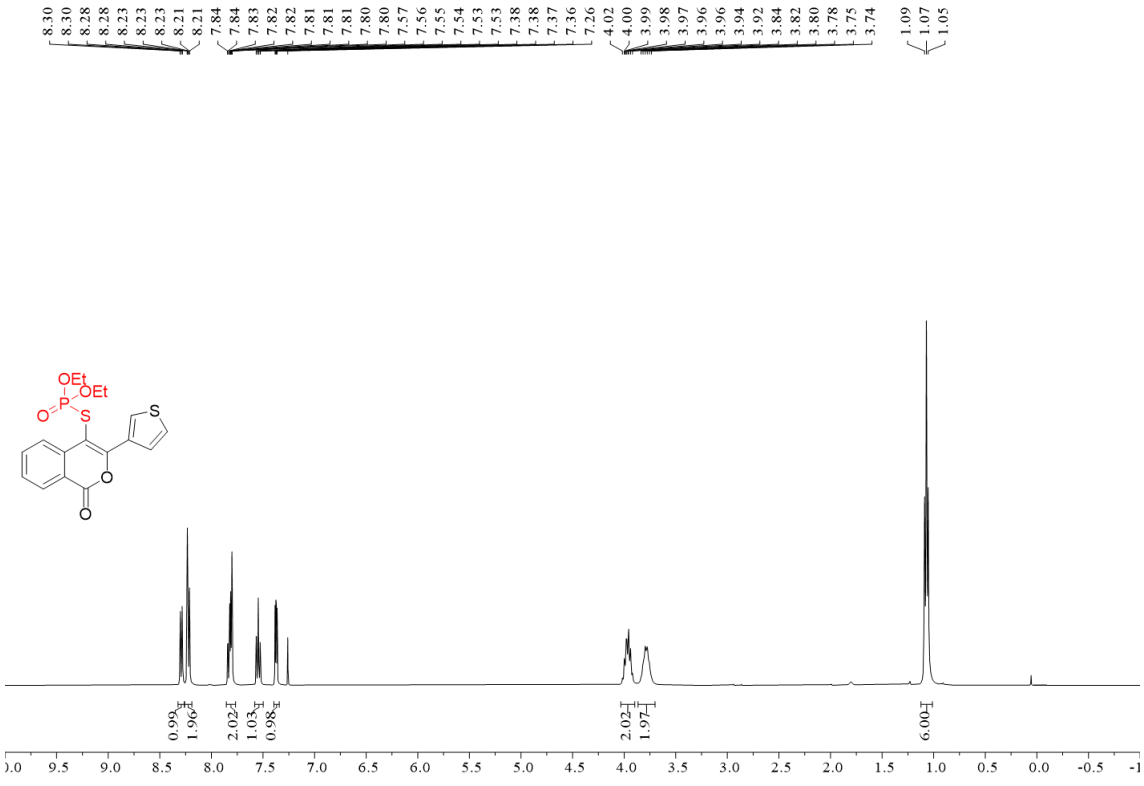
^{13}C NMR (101 MHz) Spectrum of **4l** in $CDCl_3$



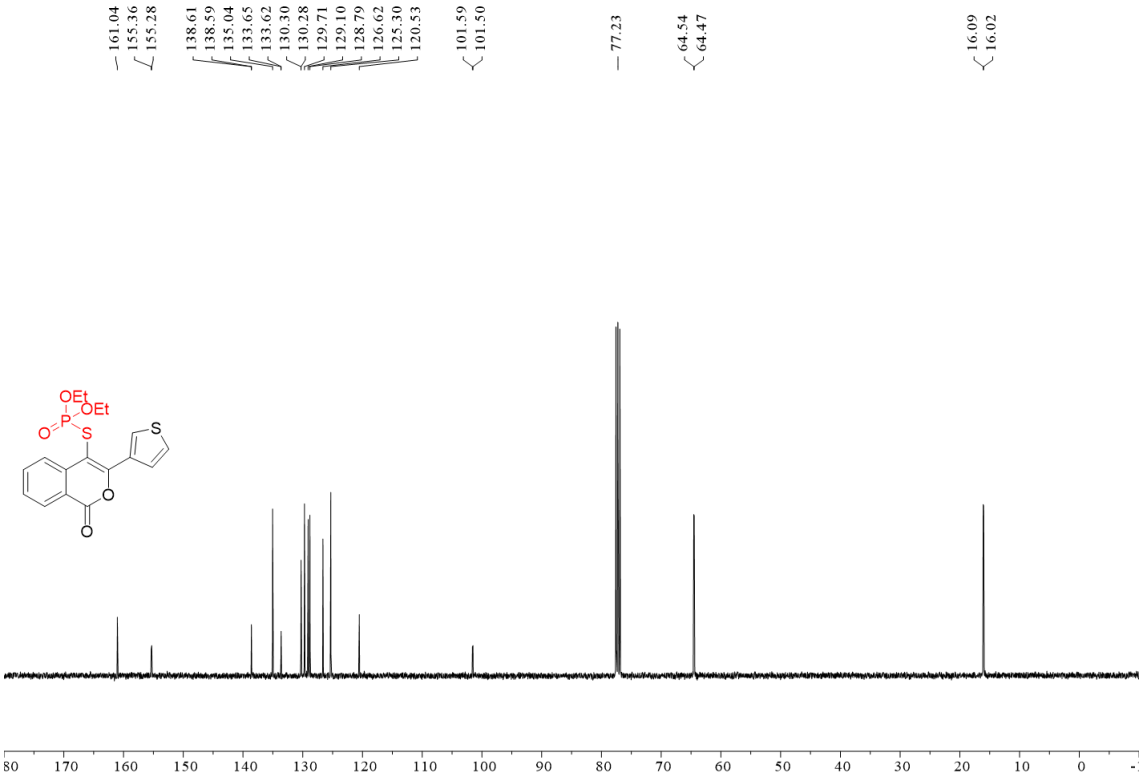
^{31}P NMR (162 MHz) Spectrum of **4l** in $CDCl_3$



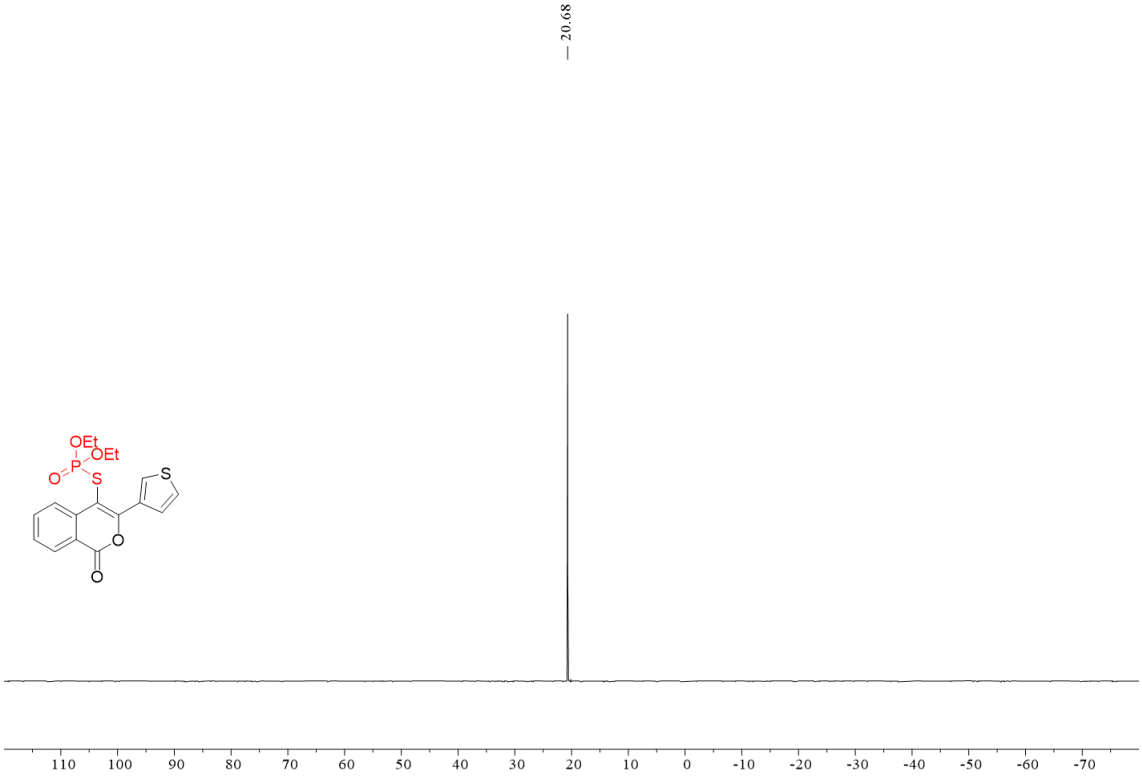
^{1}H NMR (400 MHz) Spectrum of **4m** in $CDCl_3$



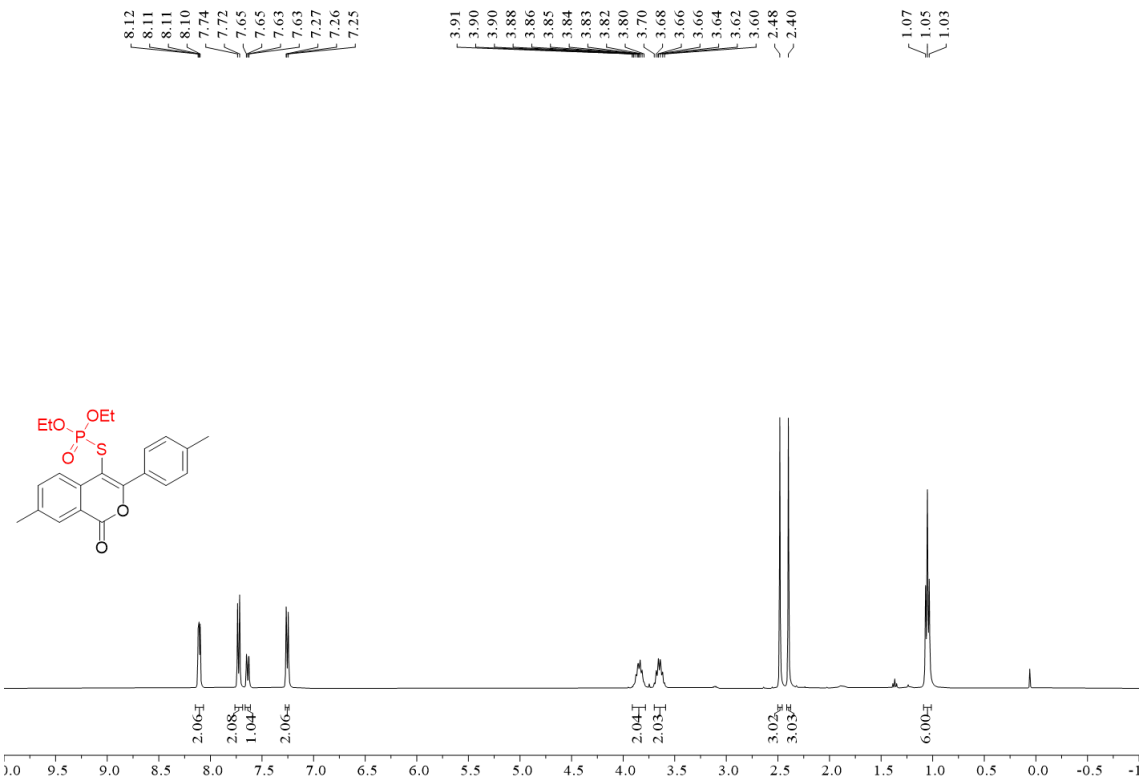
^{13}C NMR (101 MHz) Spectrum of **4m** in $CDCl_3$



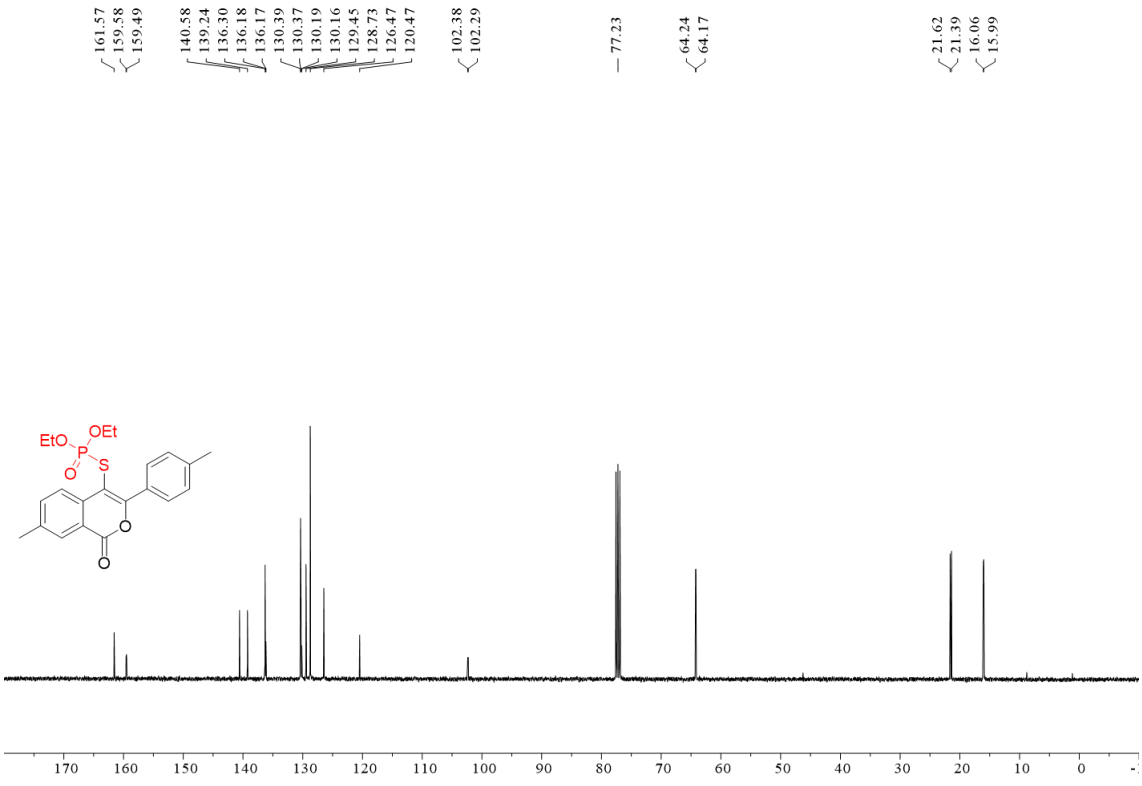
^{31}P NMR (162 MHz) Spectrum of **4m** in $CDCl_3$



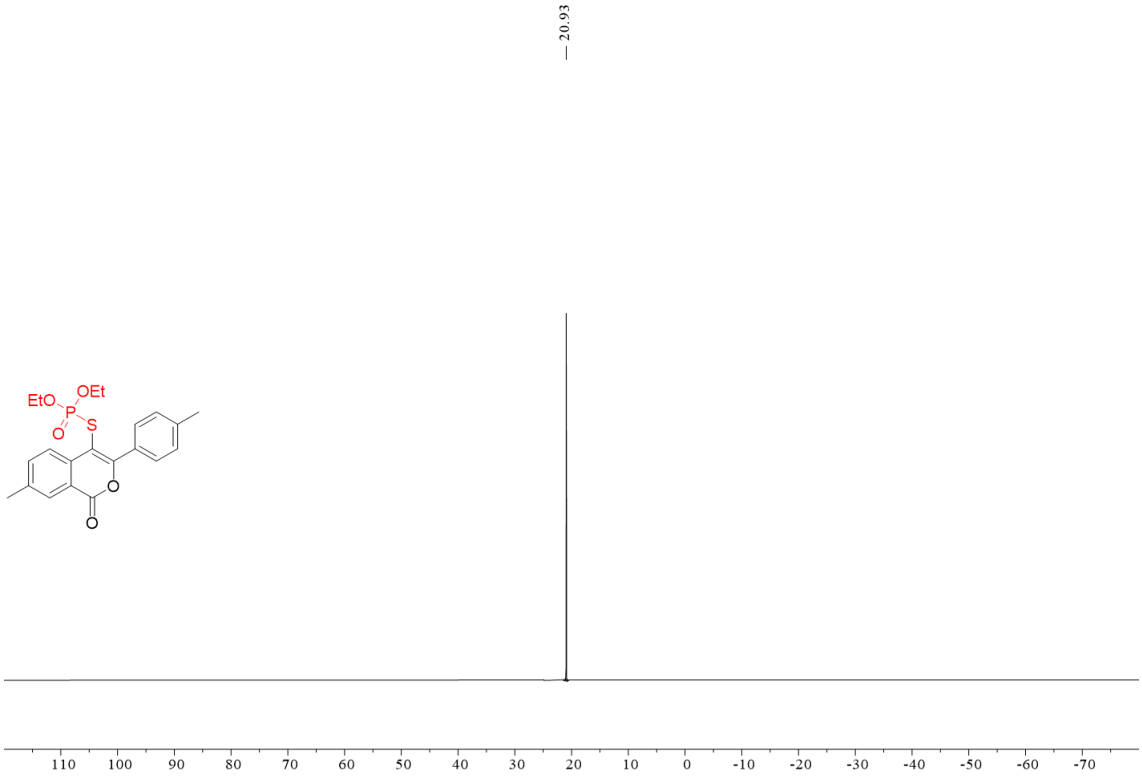
^{1}H NMR (400 MHz) Spectrum of **4n** in $CDCl_3$



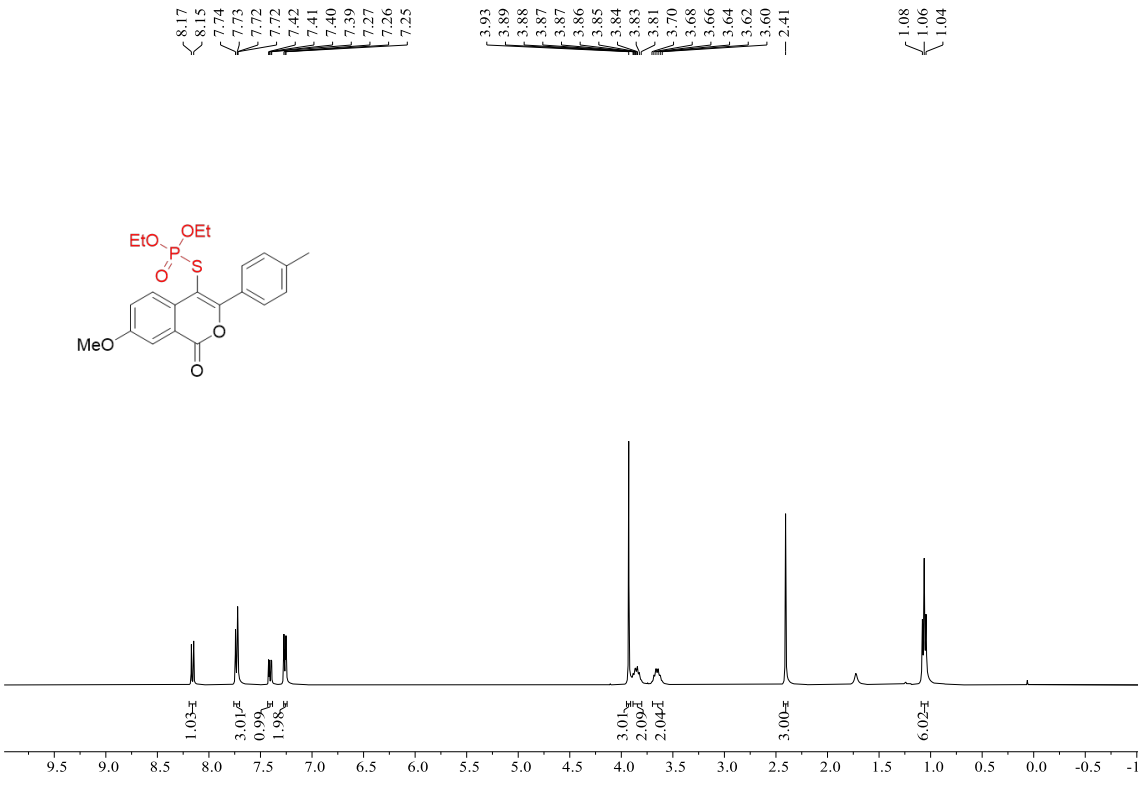
^{13}C NMR (101 MHz) Spectrum of **4n** in $CDCl_3$



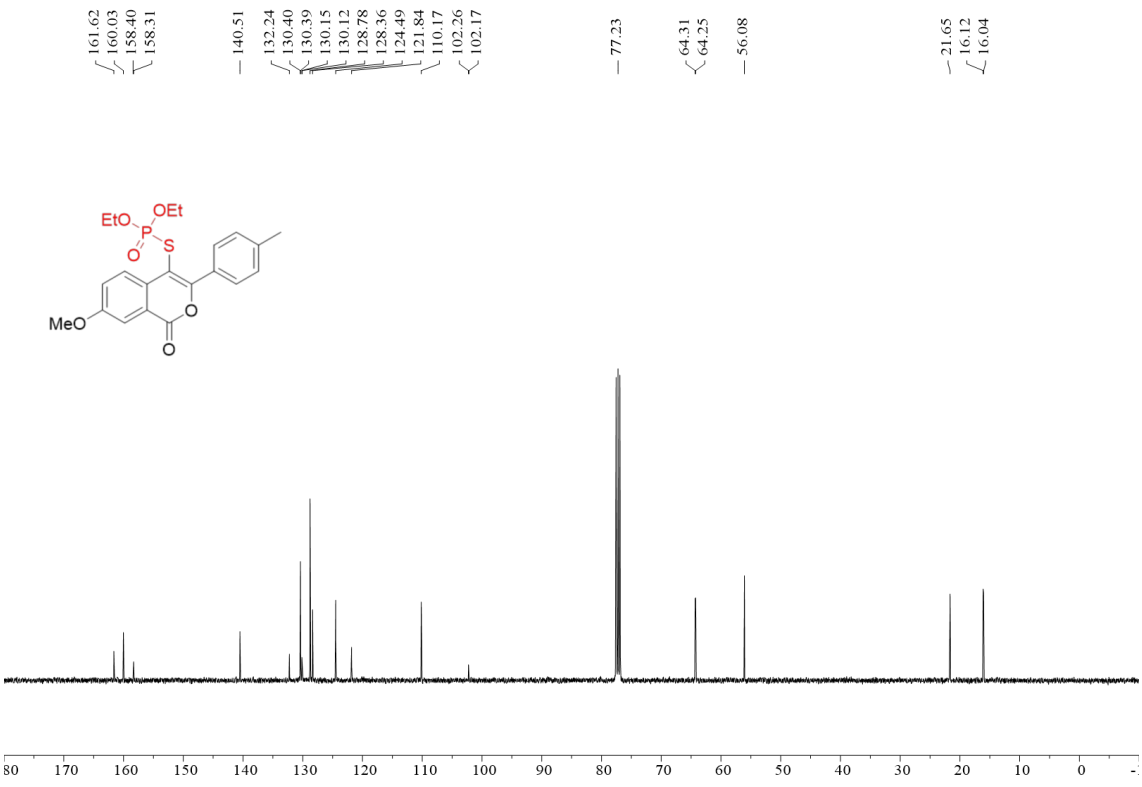
^{31}P NMR (162 MHz) Spectrum of **4n** in $CDCl_3$



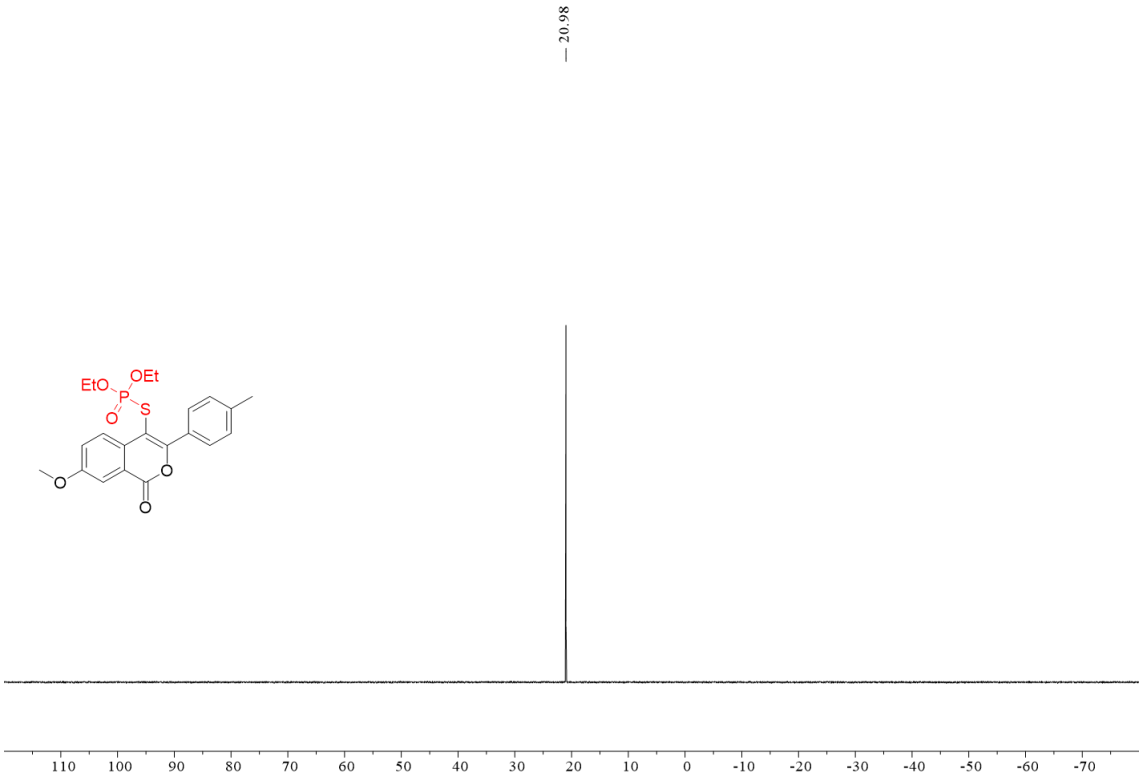
^{1}H NMR (400 MHz) Spectrum of **4o** in $CDCl_3$



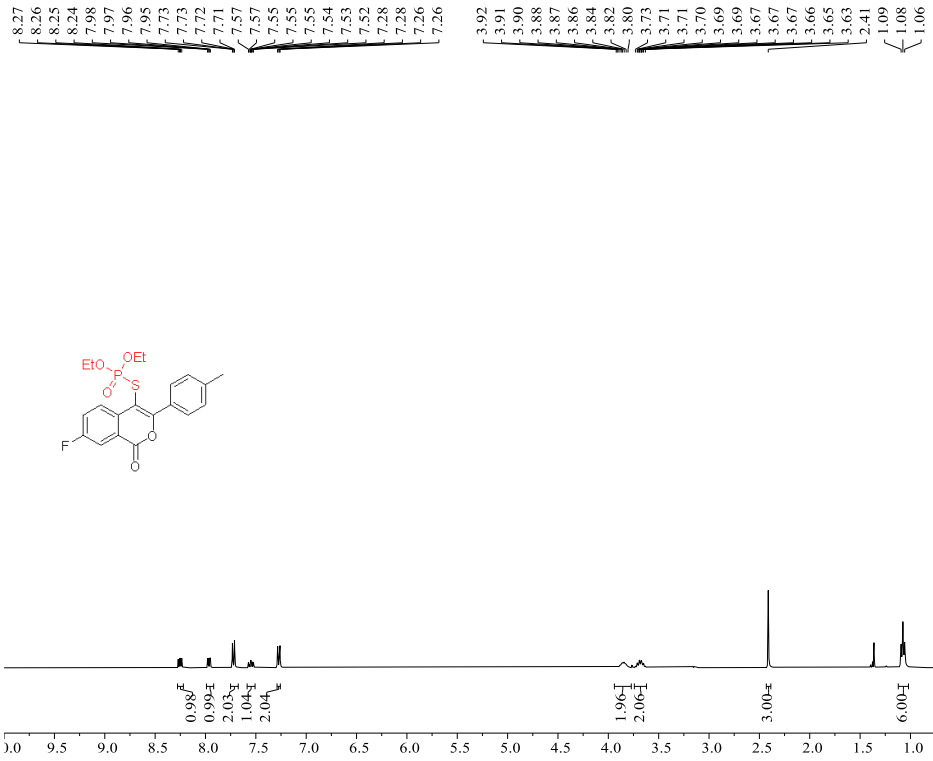
^{13}C NMR (101 MHz) Spectrum of **4o** in $CDCl_3$



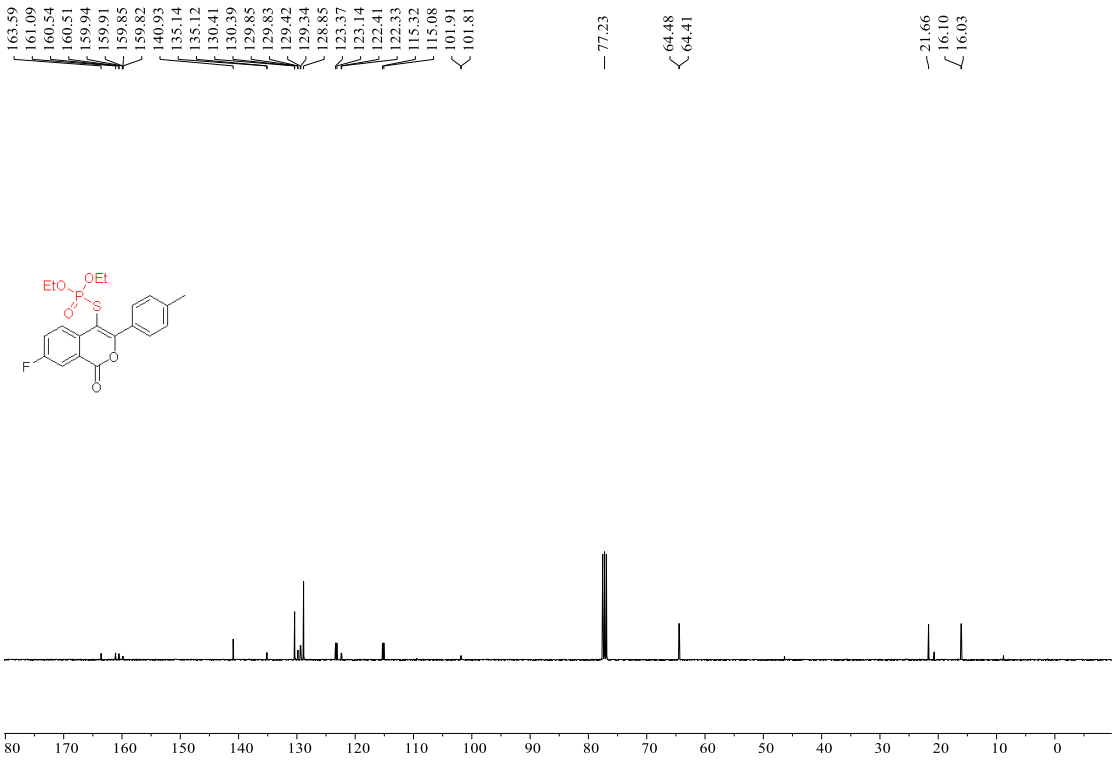
^{31}P NMR (162 MHz) Spectrum of **4o** in $CDCl_3$



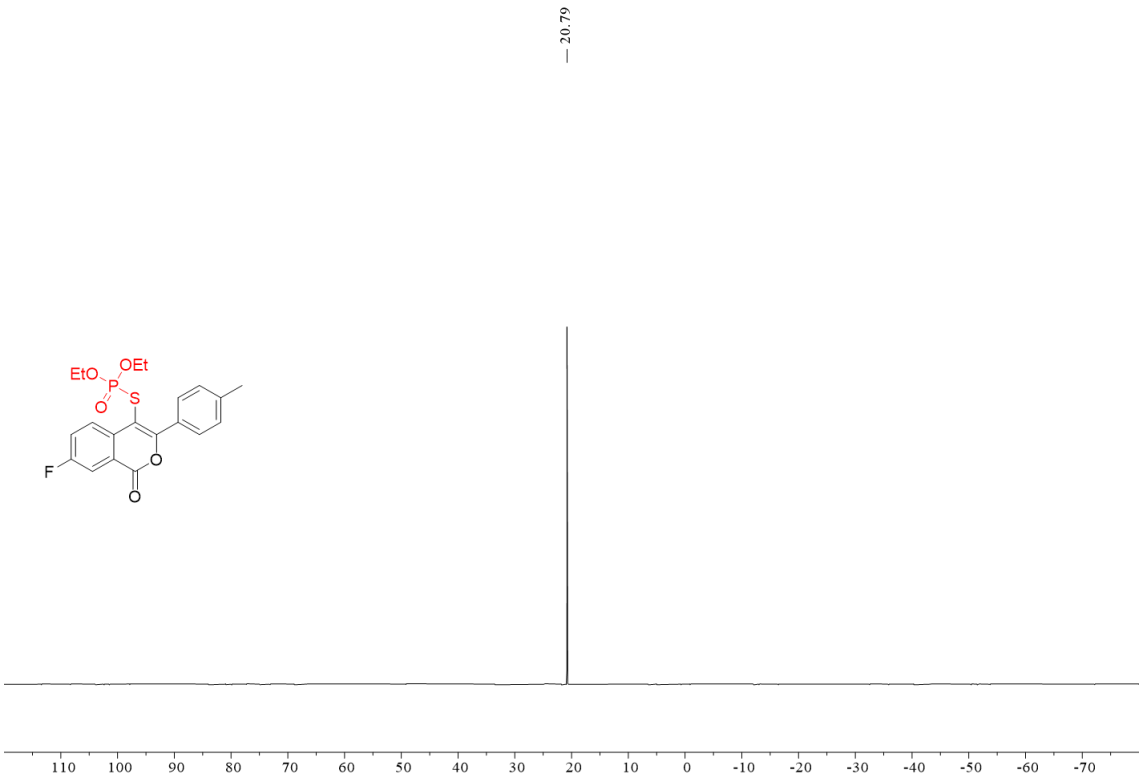
^{1}H NMR (400 MHz) Spectrum of **4p** in $CDCl_3$



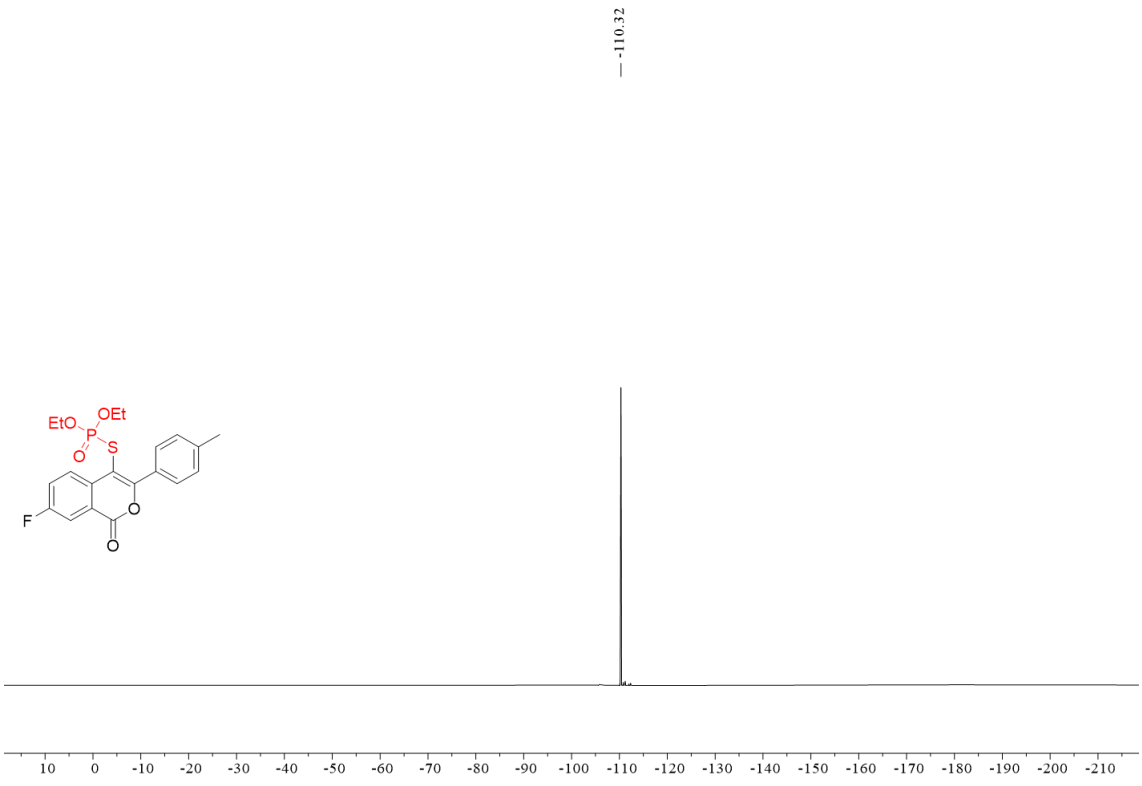
^{13}C NMR (101 MHz) Spectrum of **4p** in $CDCl_3$



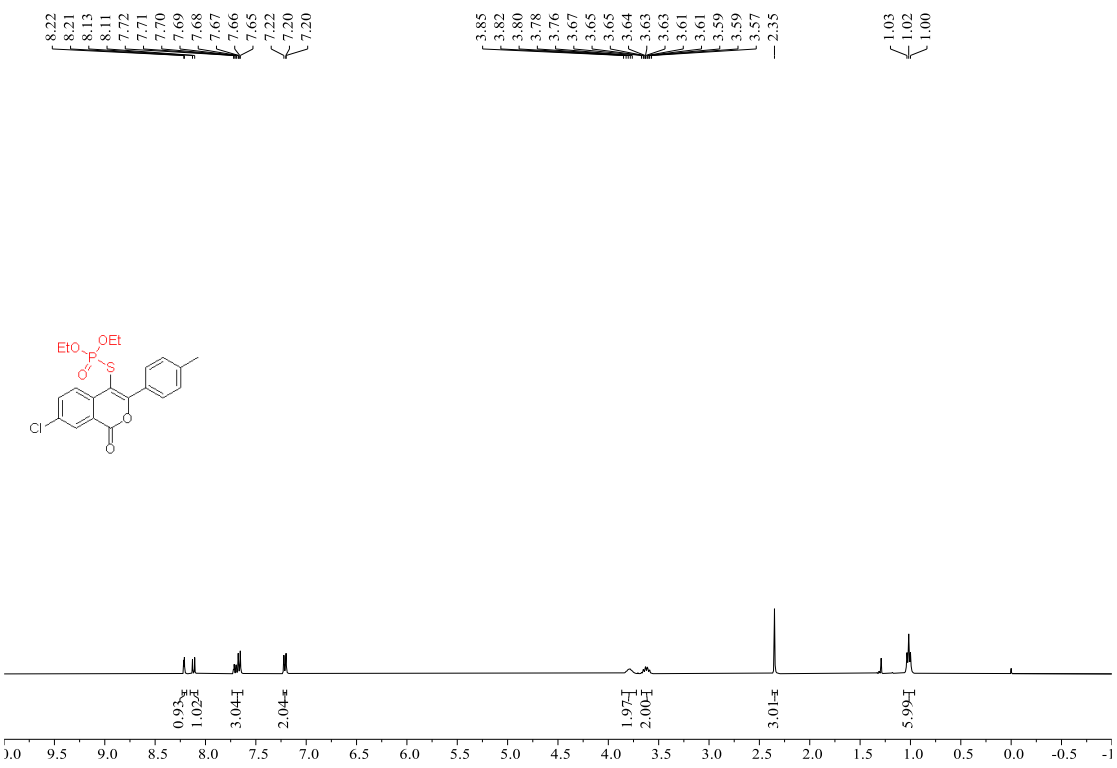
^{31}P NMR (162 MHz) Spectrum of **4p** in $CDCl_3$



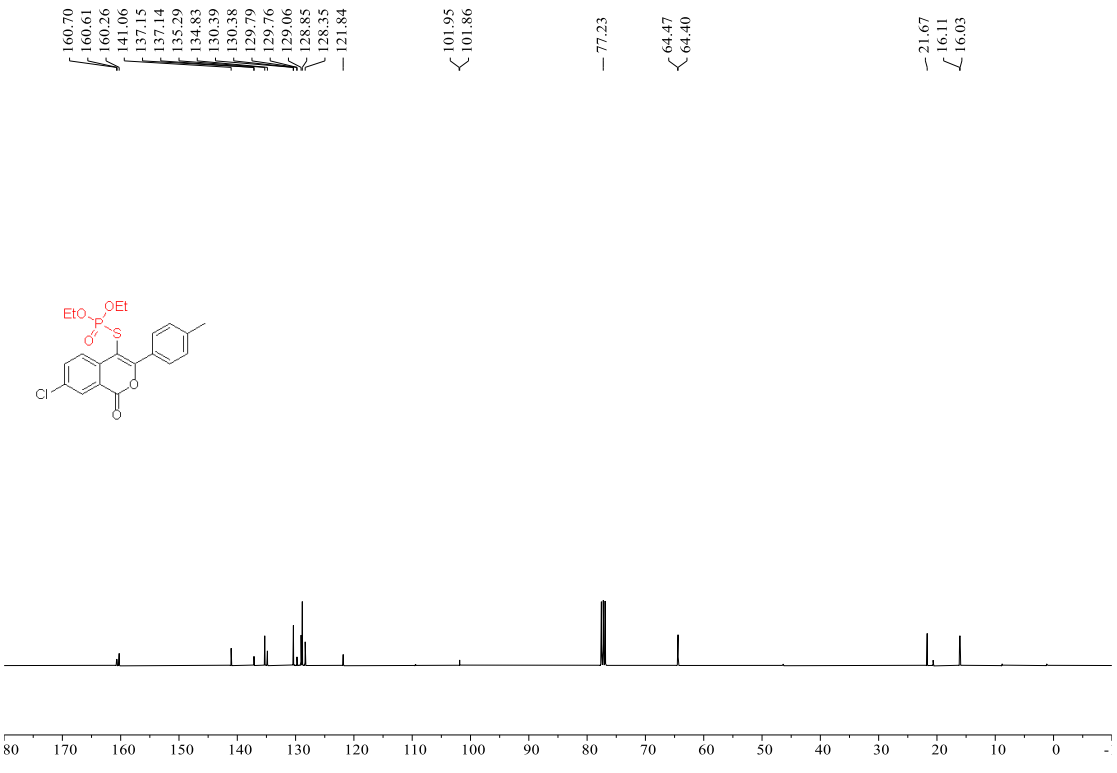
^{19}F NMR (376 MHz) Spectrum of **4p** in $CDCl_3$



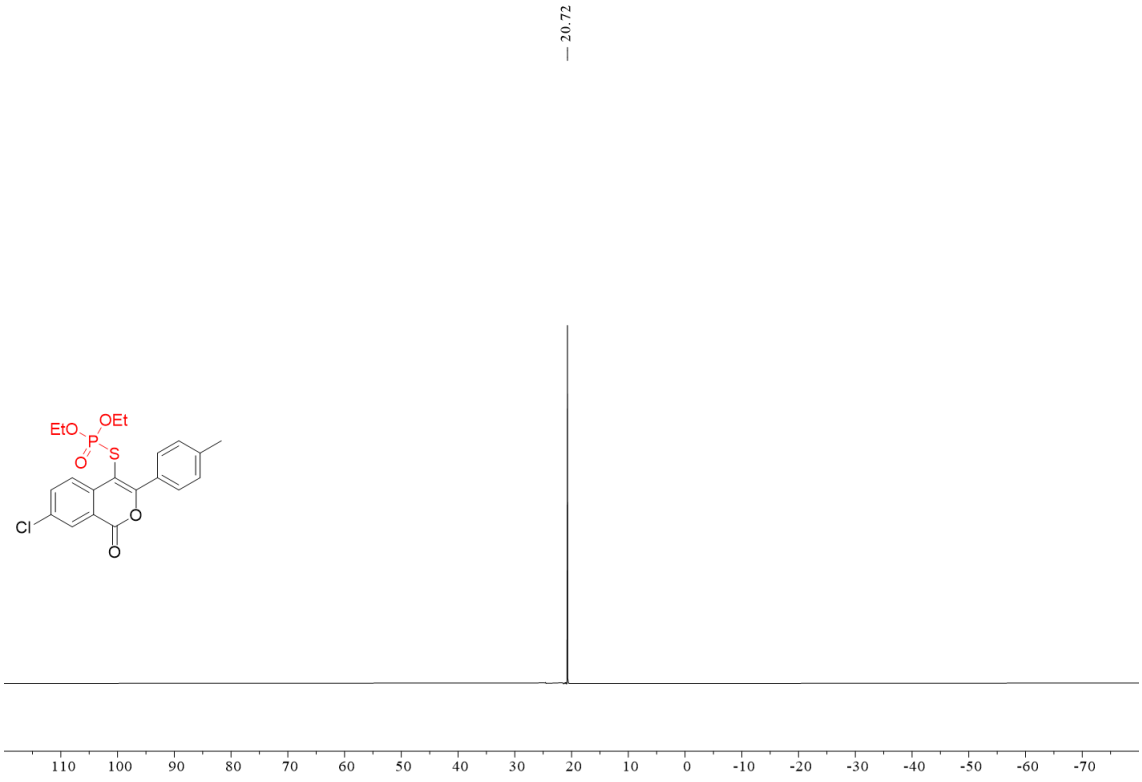
^{1}H NMR (400 MHz) Spectrum of **4q** in $CDCl_3$



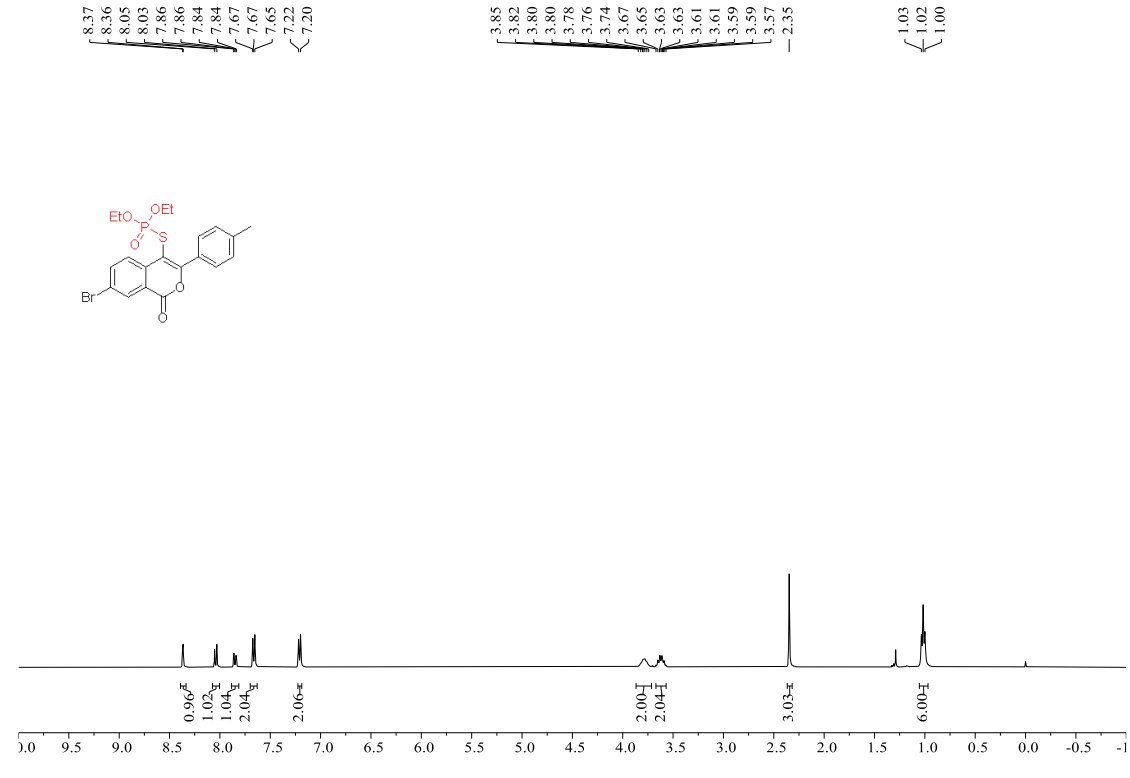
^{13}C NMR (101 MHz) Spectrum of **4q** in $CDCl_3$



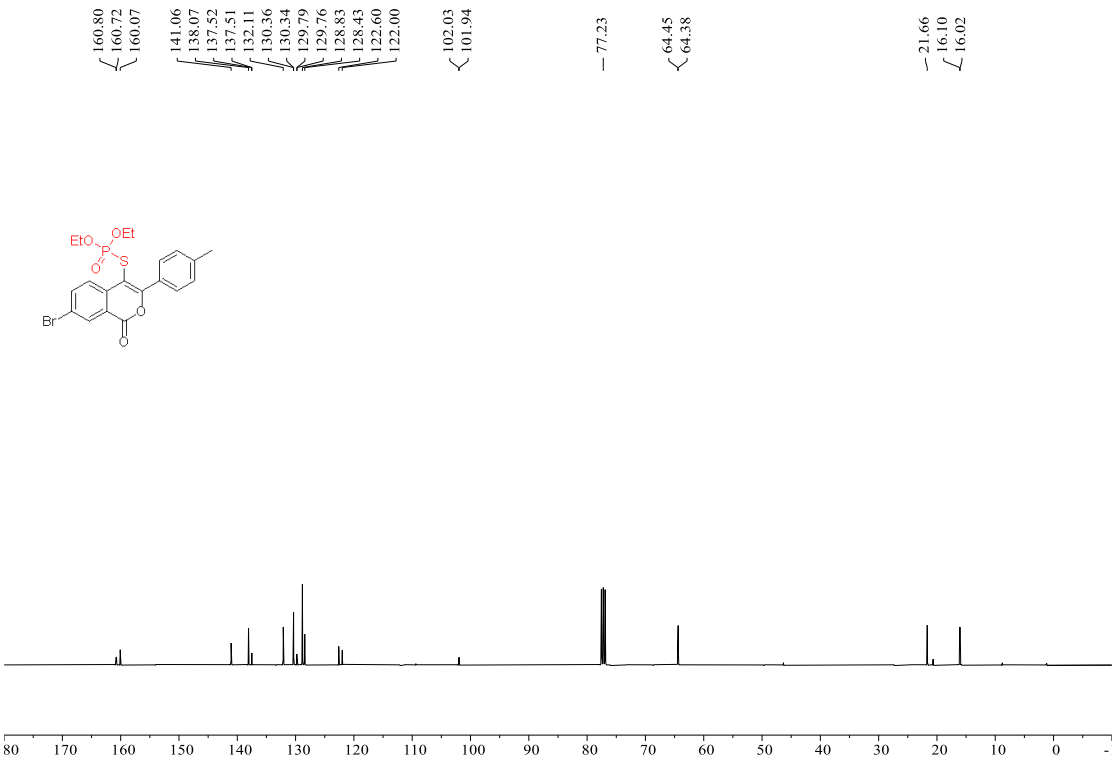
^{31}P NMR (162 MHz) Spectrum of **4q** in $CDCl_3$



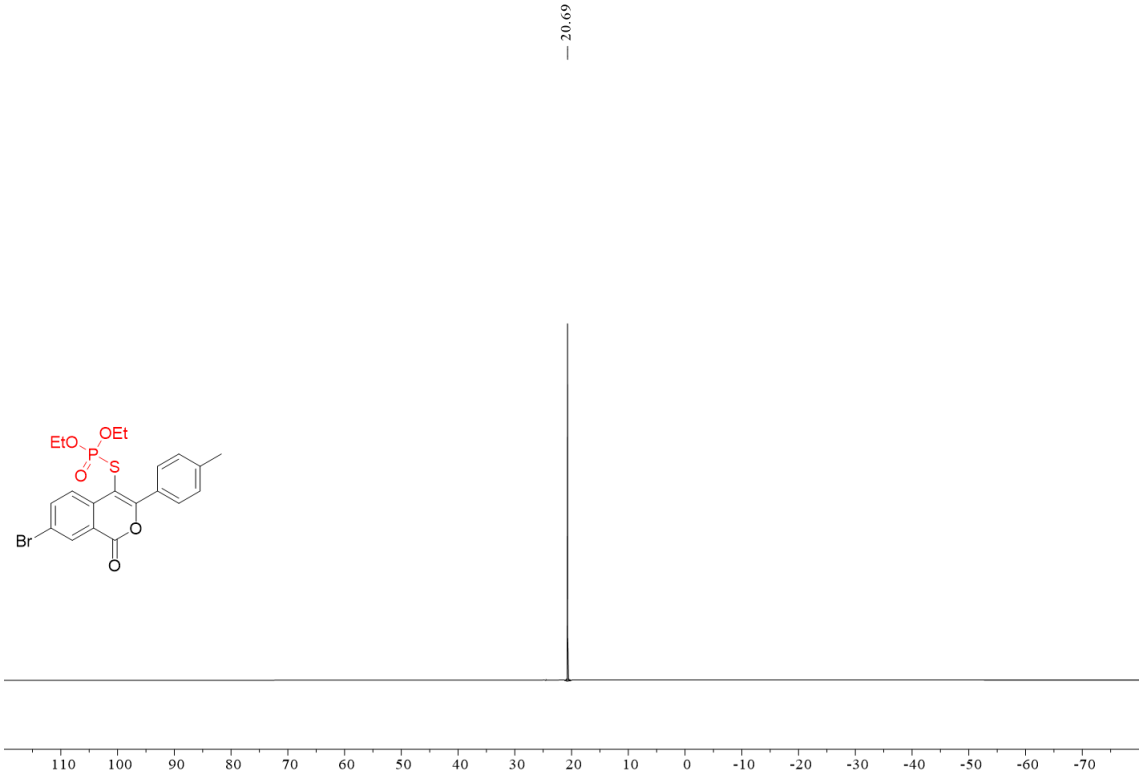
^{1}H NMR (400 MHz) Spectrum of **4r** in $CDCl_3$



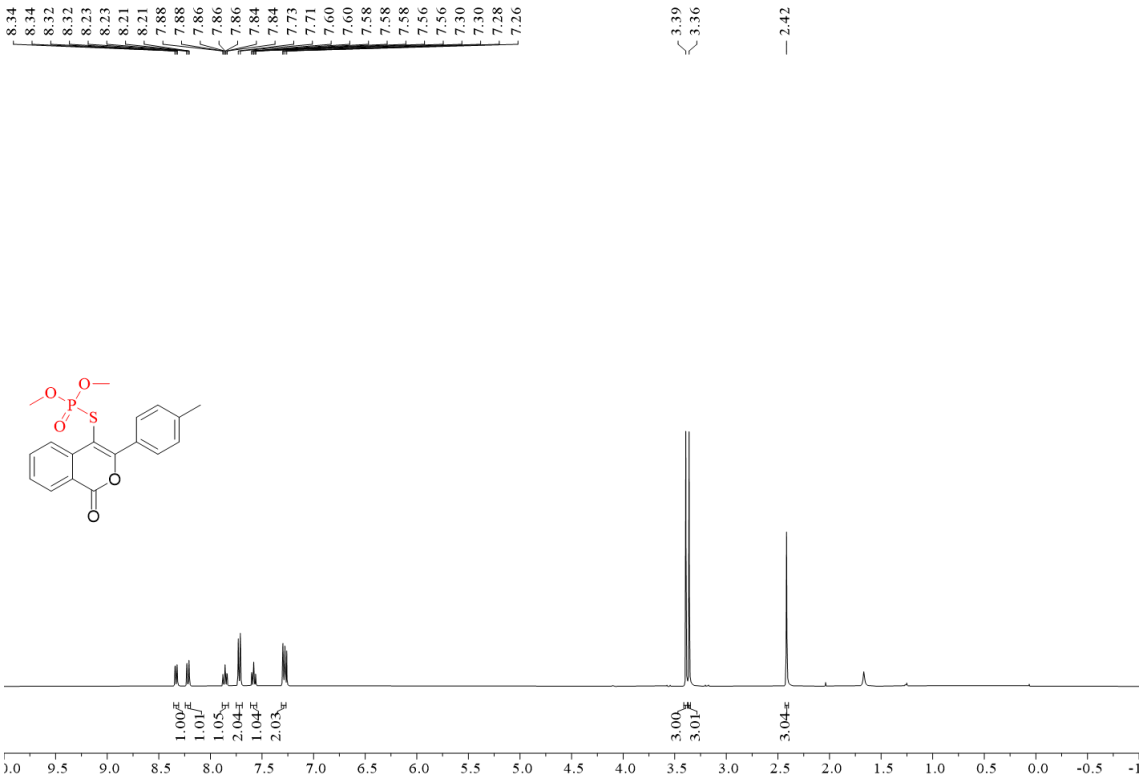
^{13}C NMR (101 MHz) Spectrum of **4r** in $CDCl_3$



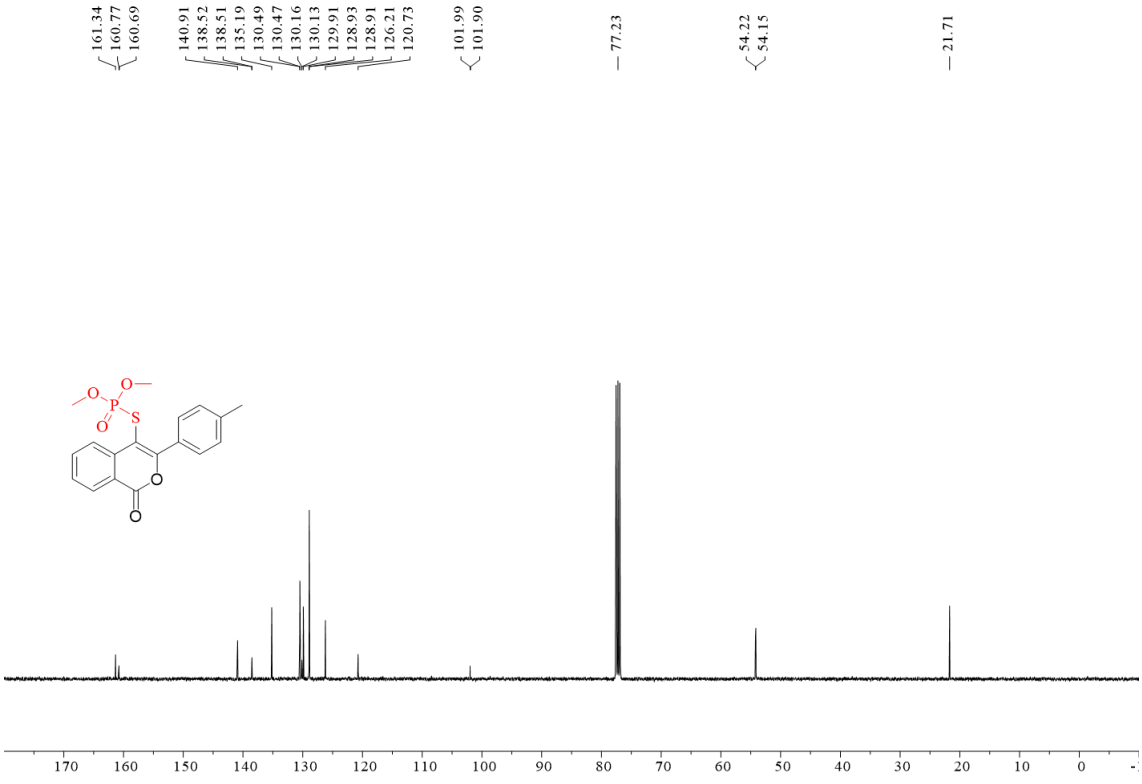
^{31}P NMR (162 MHz) Spectrum of **4r** in $CDCl_3$



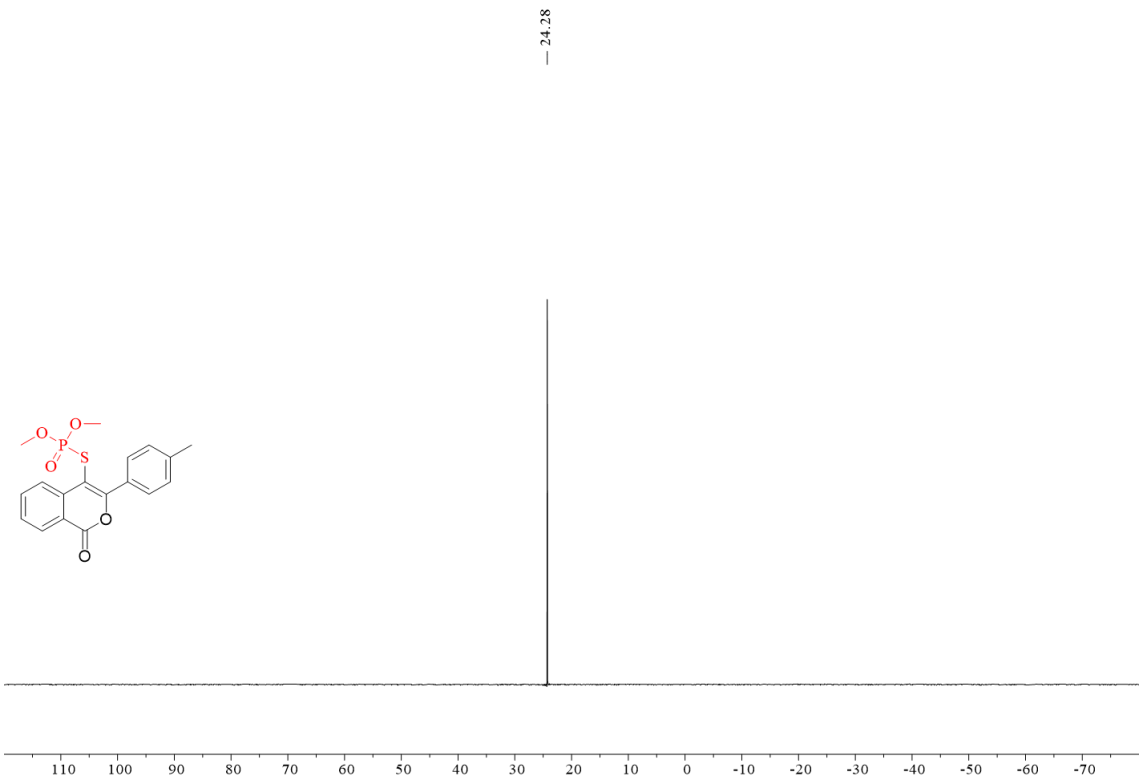
^{1}H NMR (400 MHz) Spectrum of **4s** in $CDCl_3$



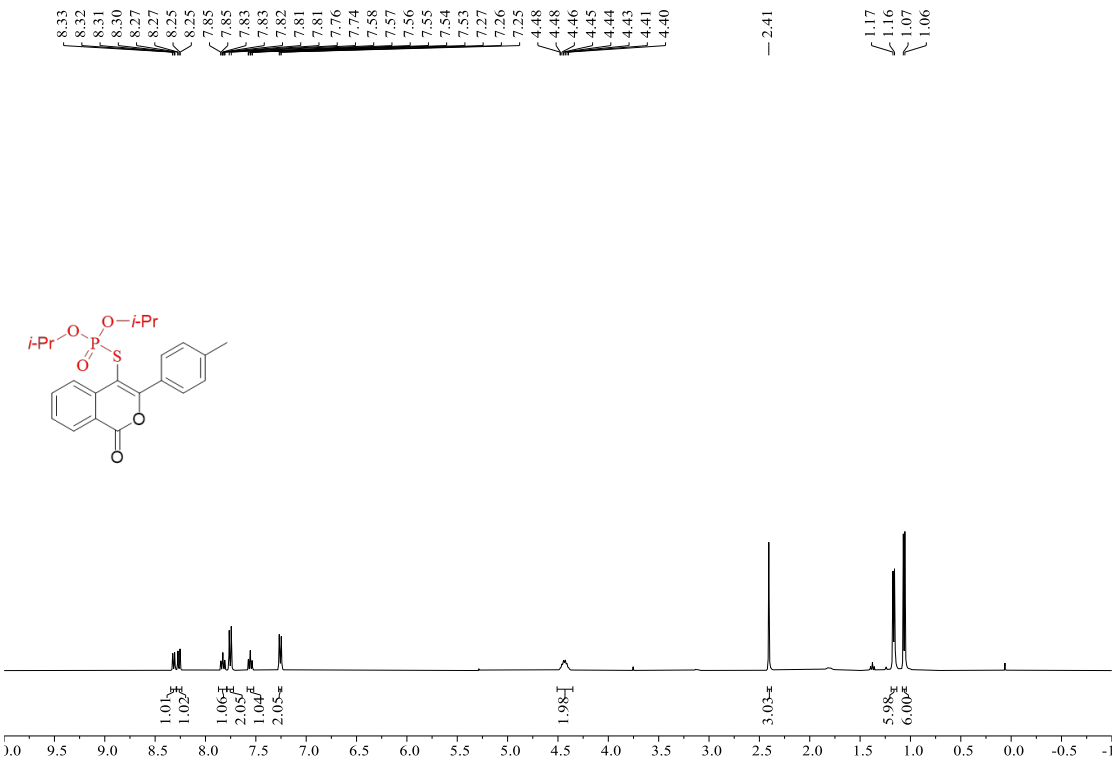
^{13}C NMR (101 MHz) Spectrum of **4s** in $CDCl_3$



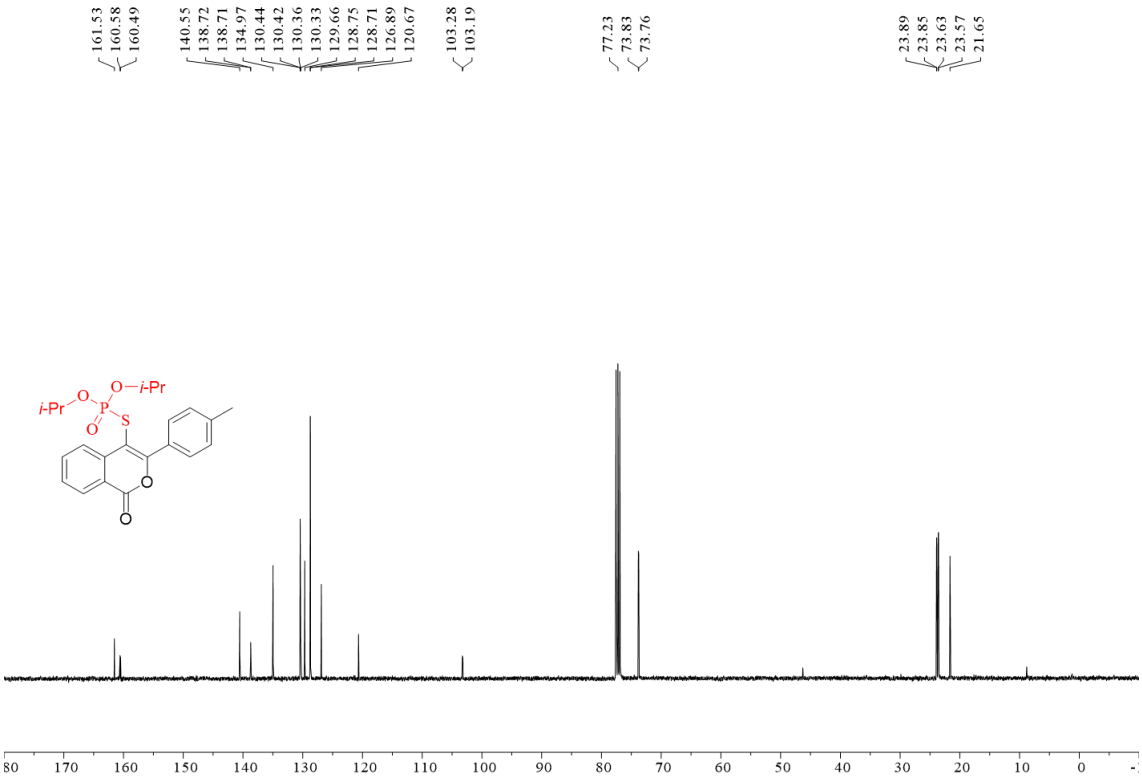
^{31}P NMR (162 MHz) Spectrum of **4s** in $CDCl_3$



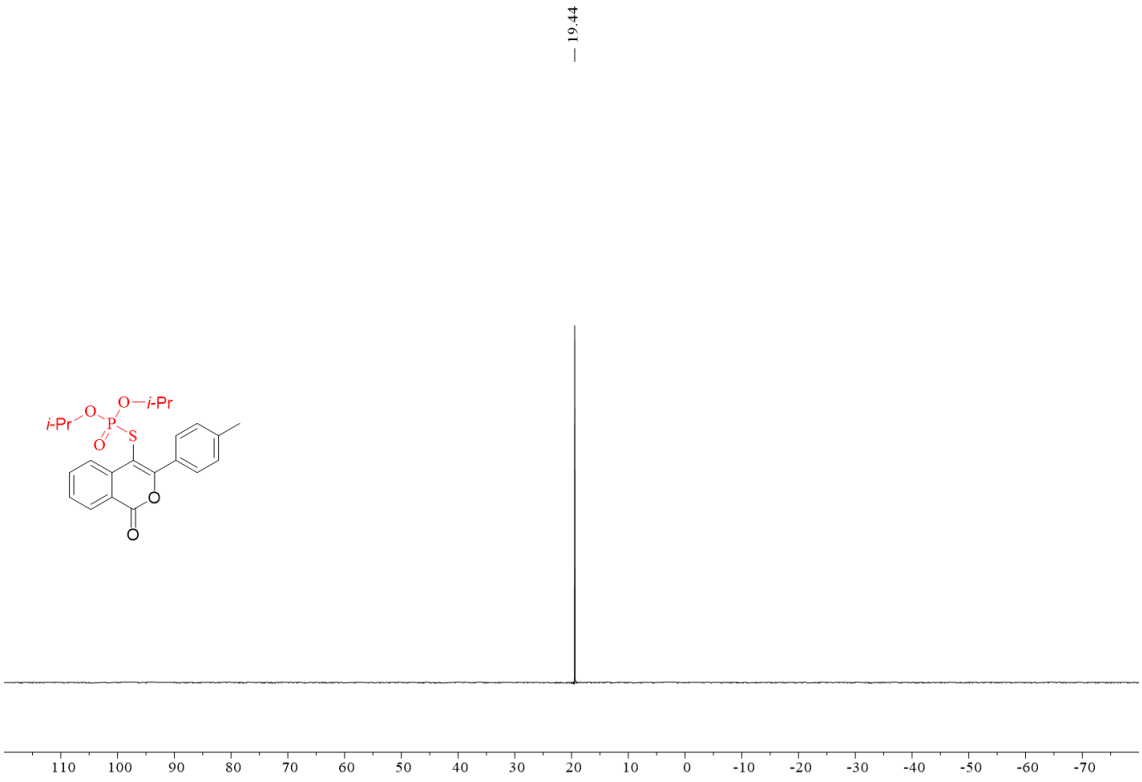
^{1}H NMR (400 MHz) Spectrum of **4t** in $CDCl_3$



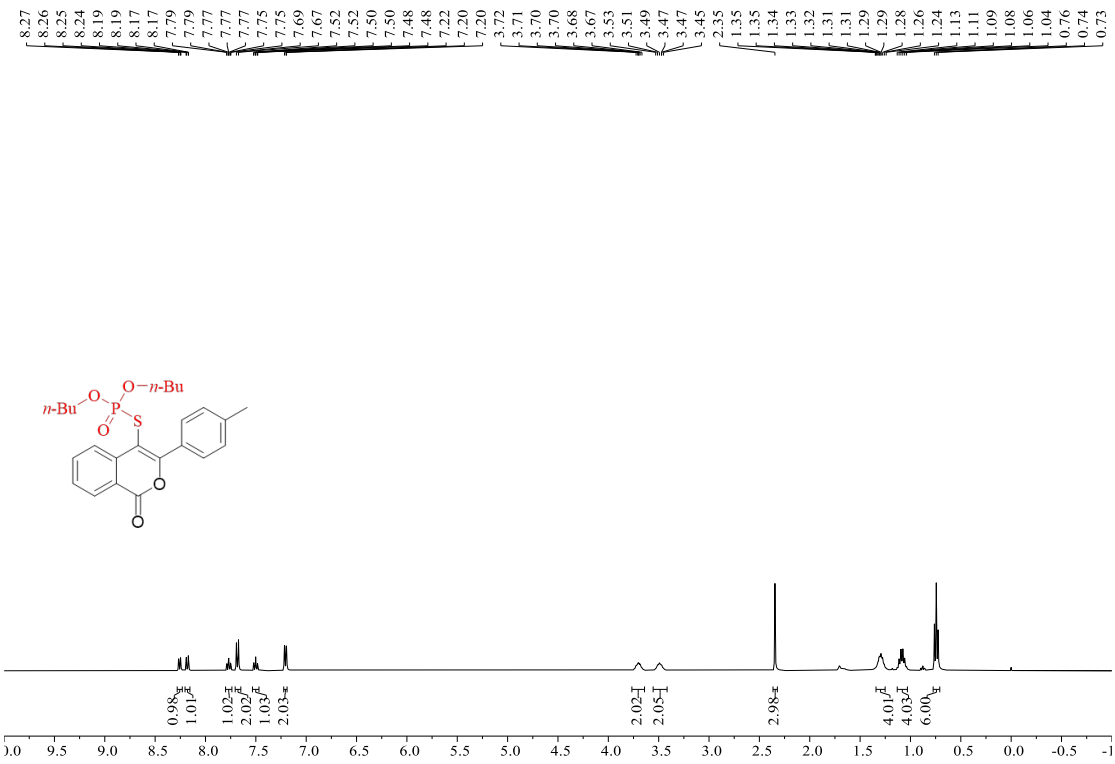
^{13}C NMR (101 MHz) Spectrum of **4t** in $CDCl_3$



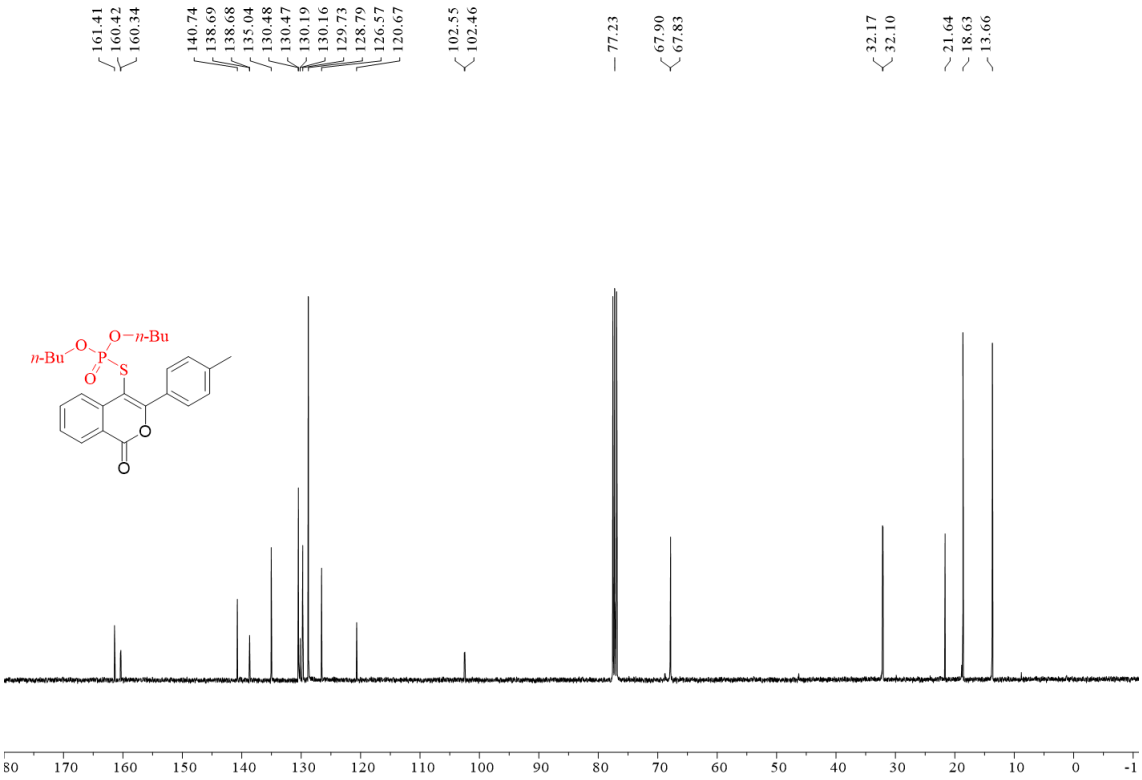
^{31}P NMR (162 MHz) Spectrum of **4t** in $CDCl_3$



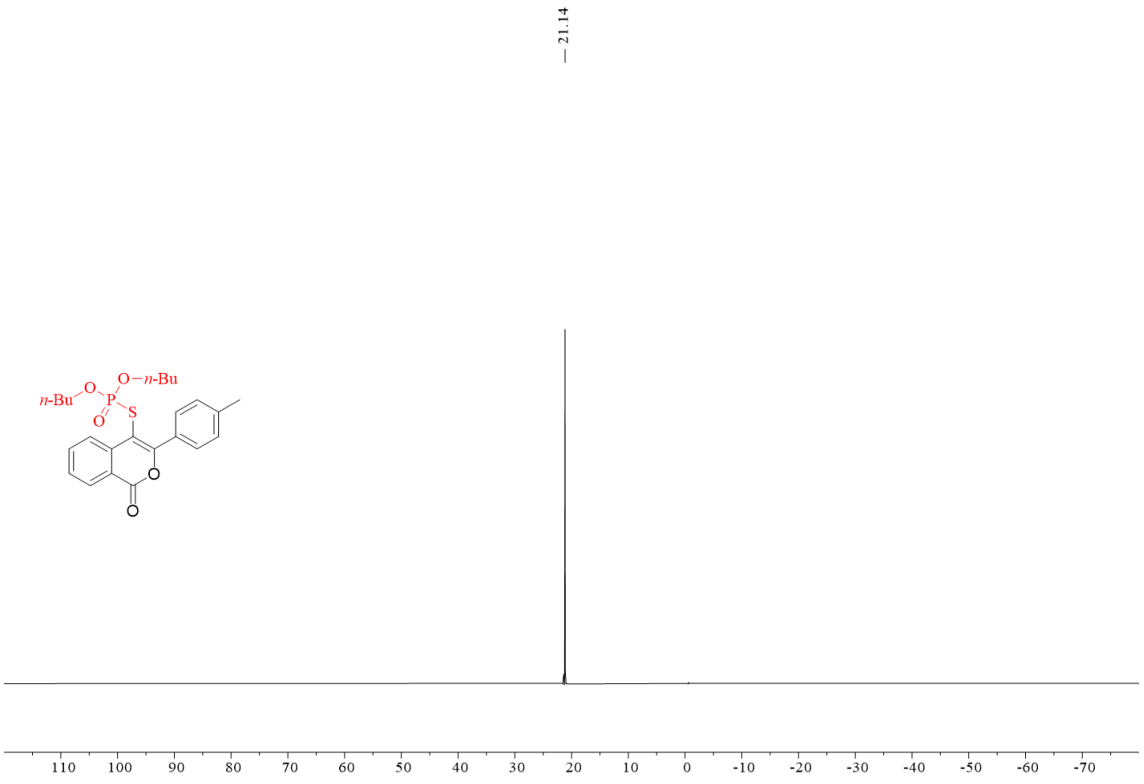
^{1}H NMR (400 MHz) Spectrum of **4u** in $CDCl_3$



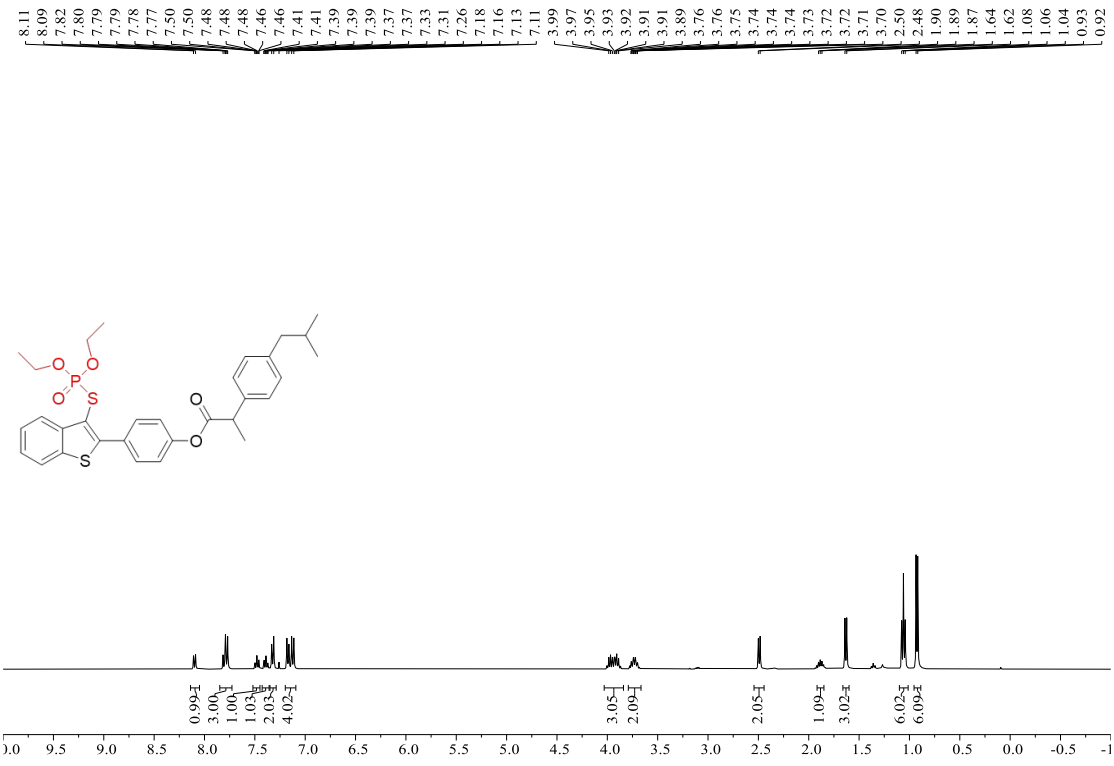
^{13}C NMR (101 MHz) Spectrum of **4u** in $CDCl_3$



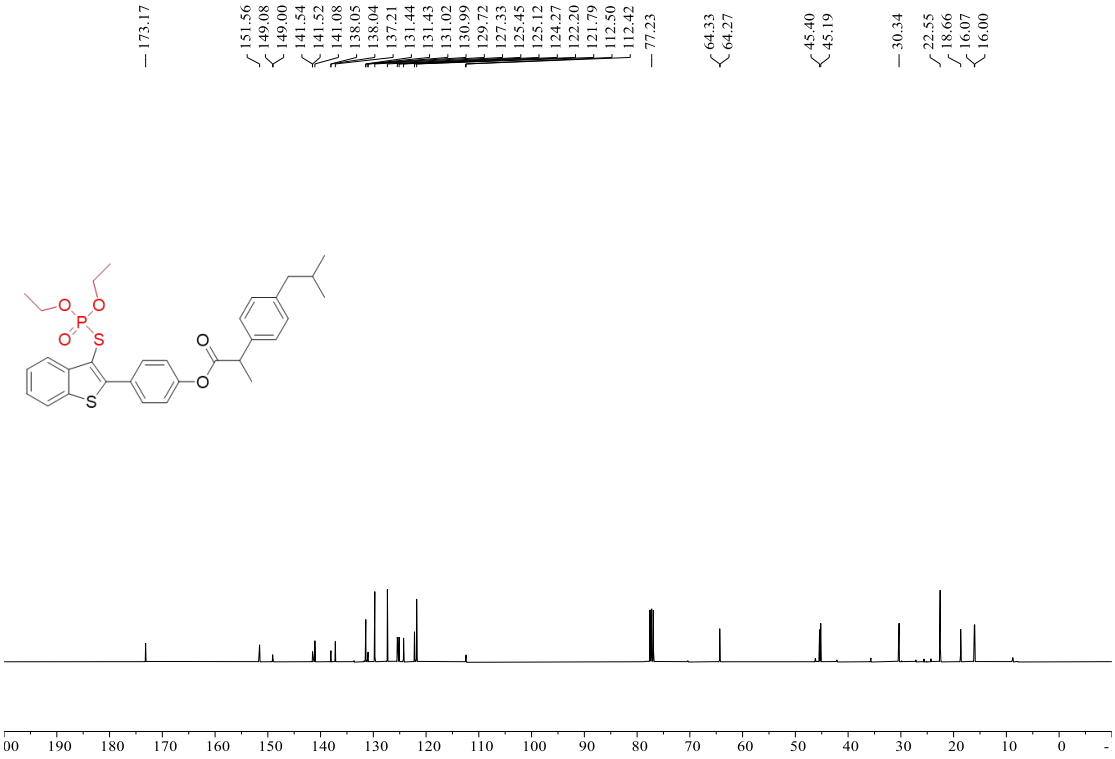
^{31}P NMR (162 MHz) Spectrum of **4u** in $CDCl_3$



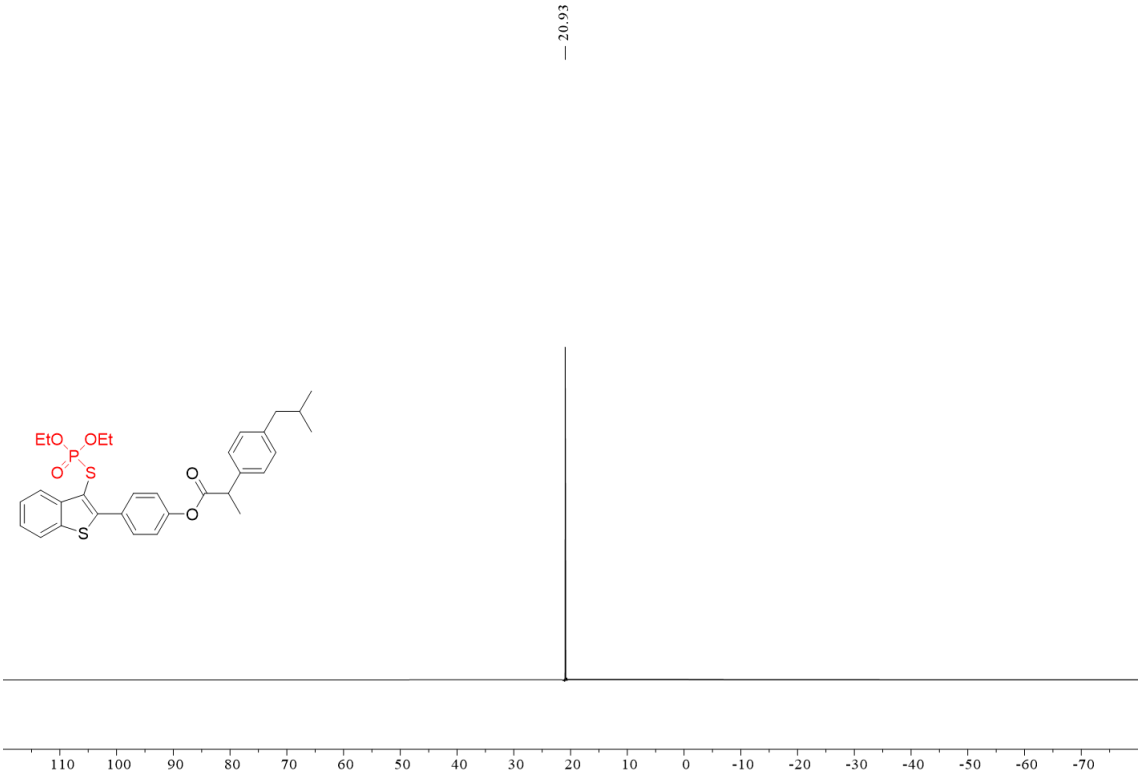
^{1}H NMR (400 MHz) Spectrum of **5a** in $CDCl_3$



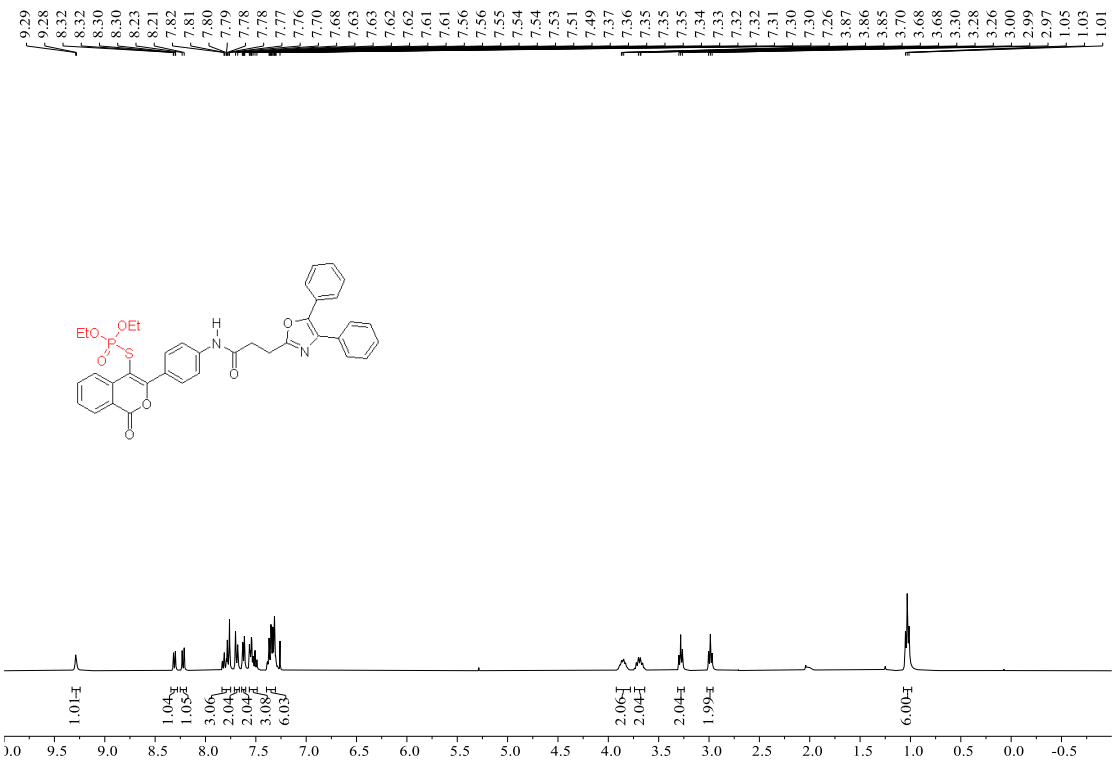
^{13}C NMR (101 MHz) Spectrum of **5a** in $CDCl_3$



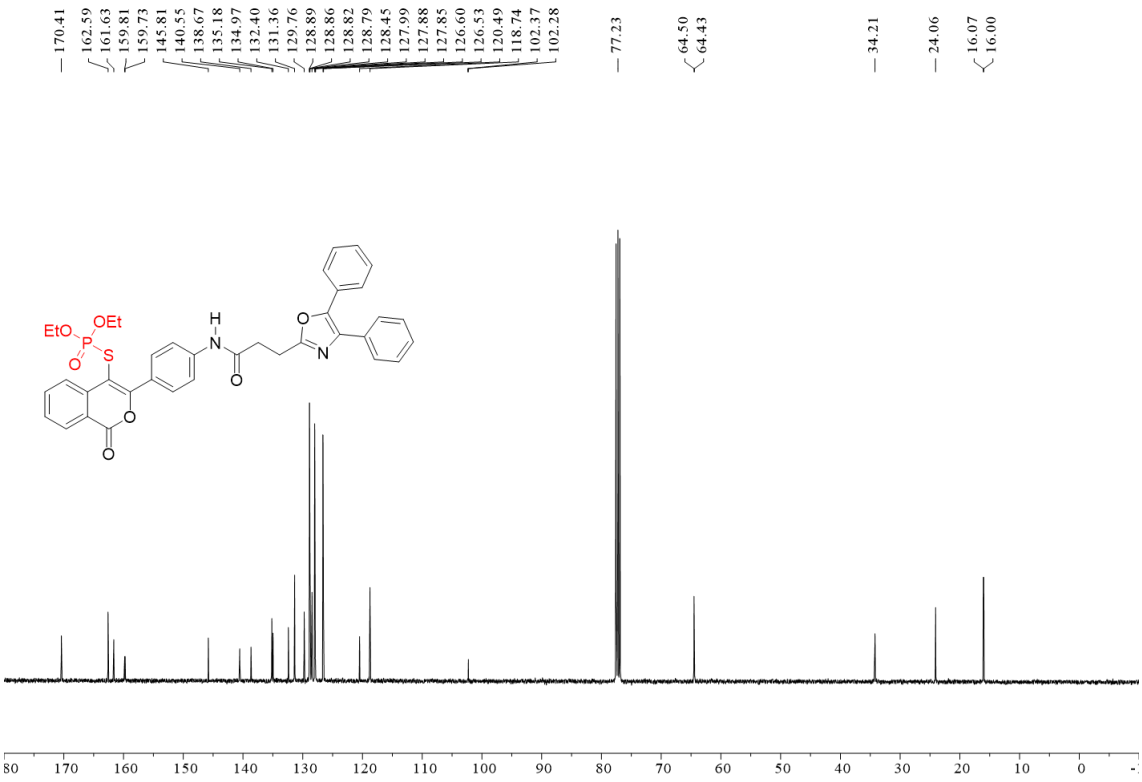
^{31}P NMR (162 MHz) Spectrum of **5a** in $CDCl_3$



^{1}H NMR (400 MHz) Spectrum of **5b** in $CDCl_3$

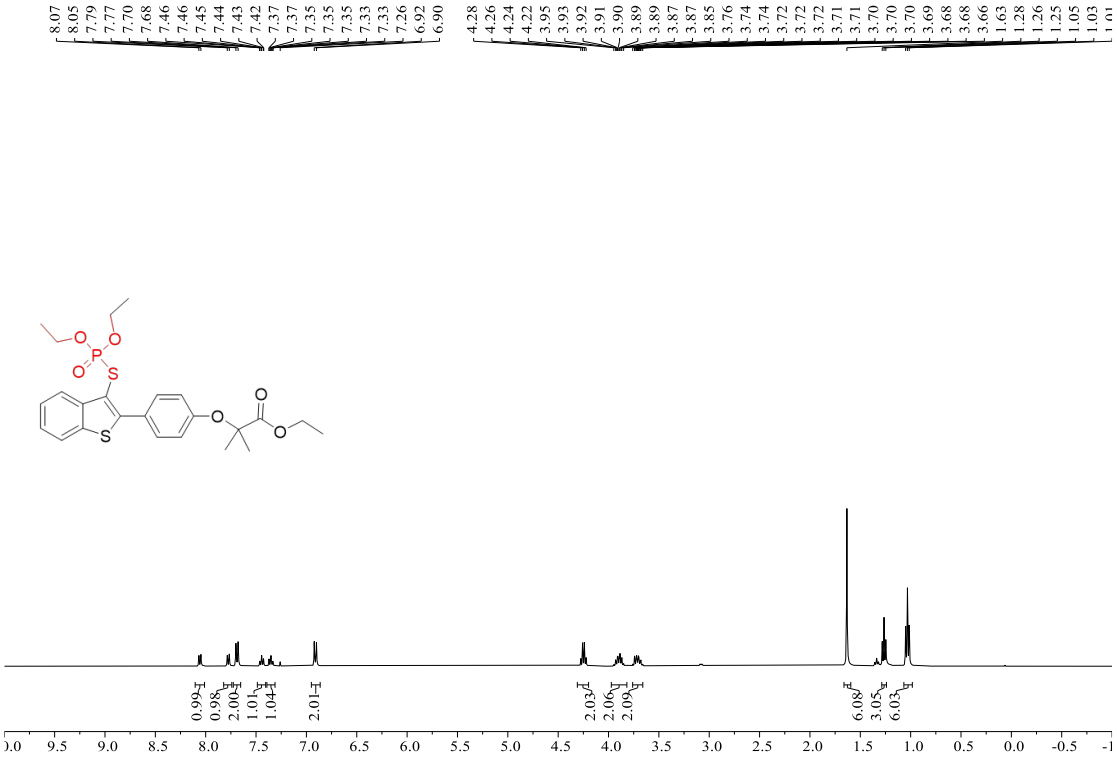


^{13}C NMR (101 MHz) Spectrum of **5b** in $CDCl_3$

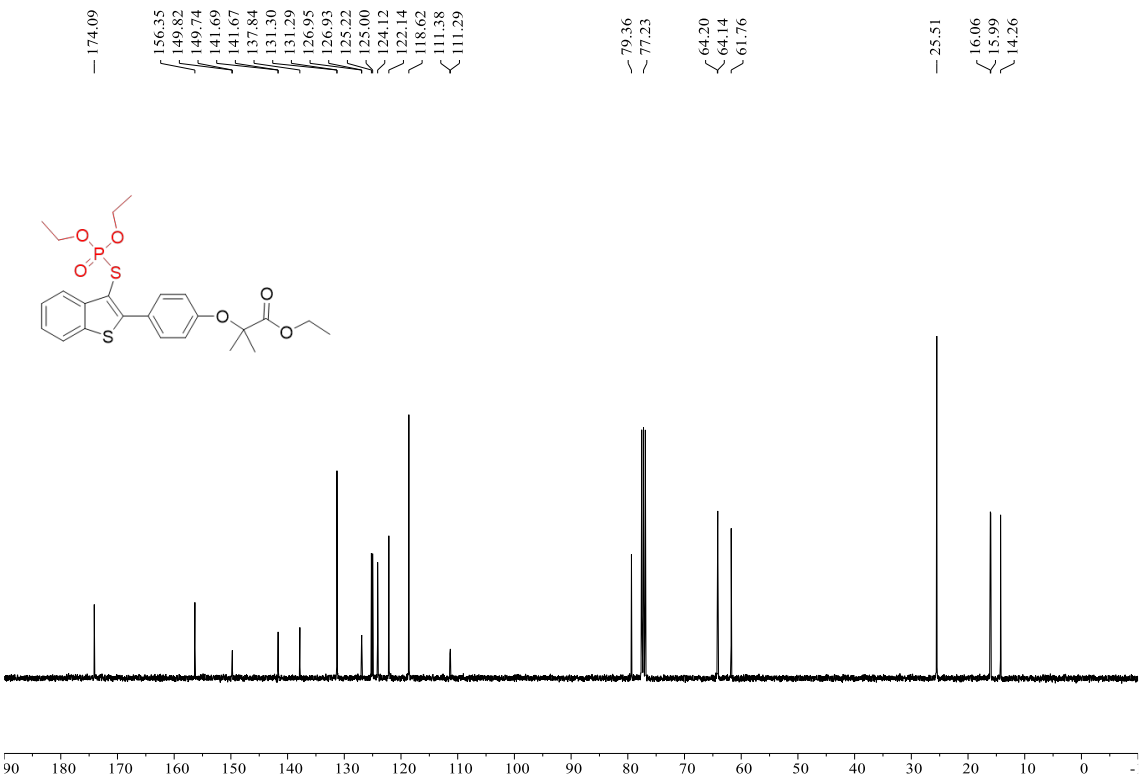


^{31}P NMR (162 MHz) Spectrum of **5b** in $CDCl_3$

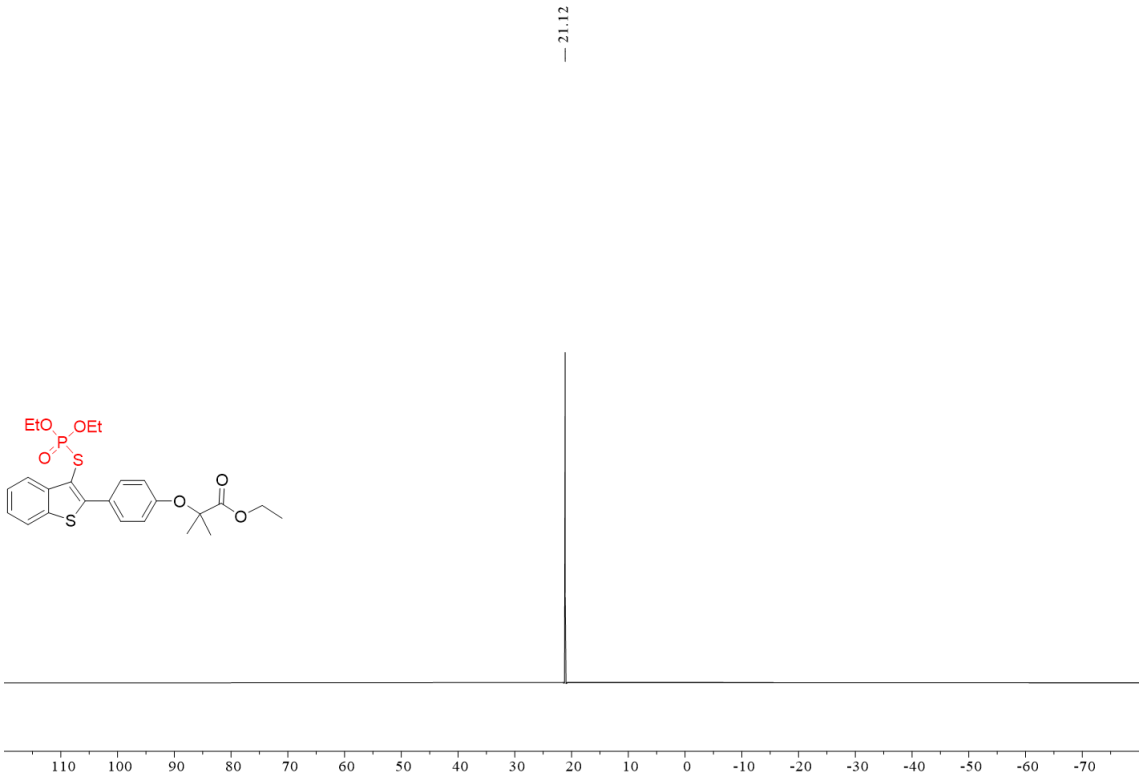
^{1}H NMR (400 MHz) Spectrum of **5c** in $CDCl_3$



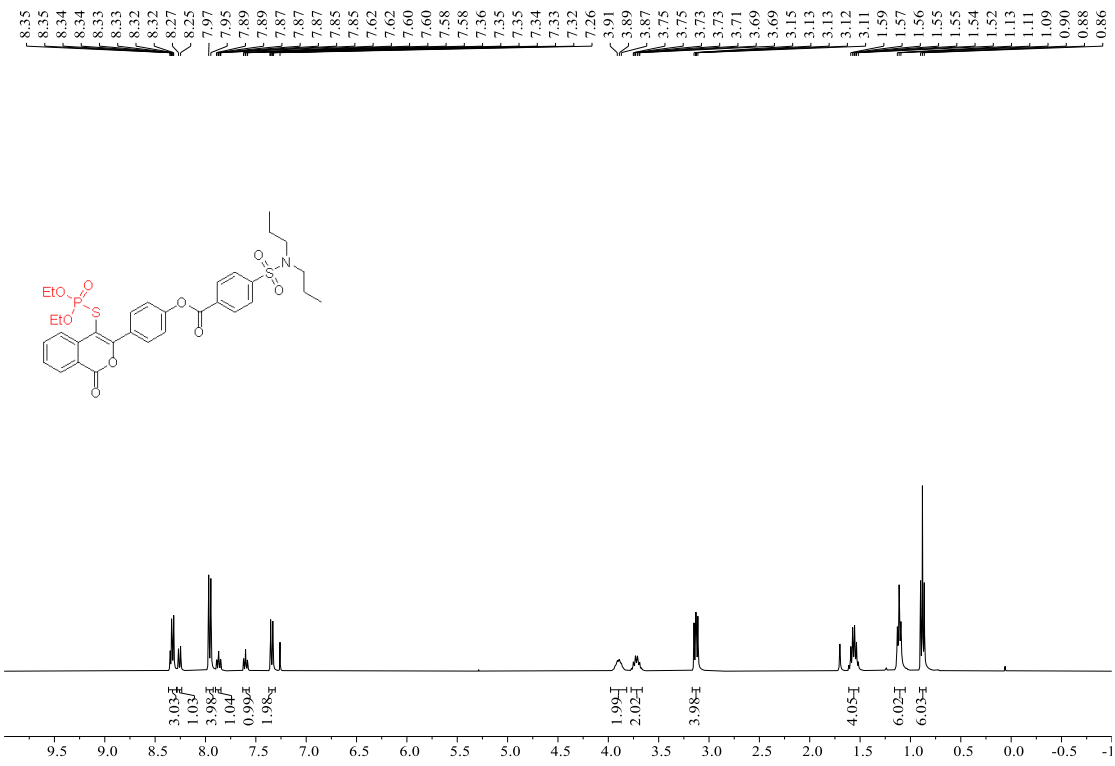
^{13}C NMR (101 MHz) Spectrum of **5c** in $CDCl_3$



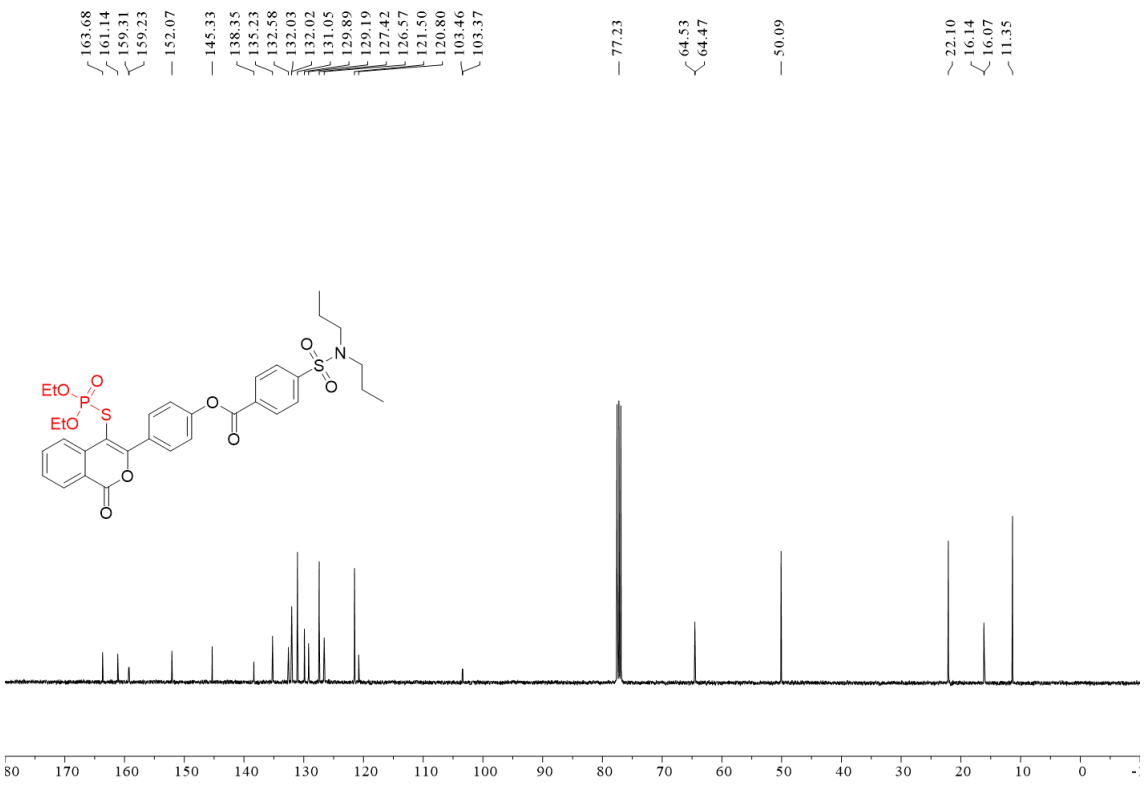
^{31}P NMR (162 MHz) Spectrum of **5c** in $CDCl_3$



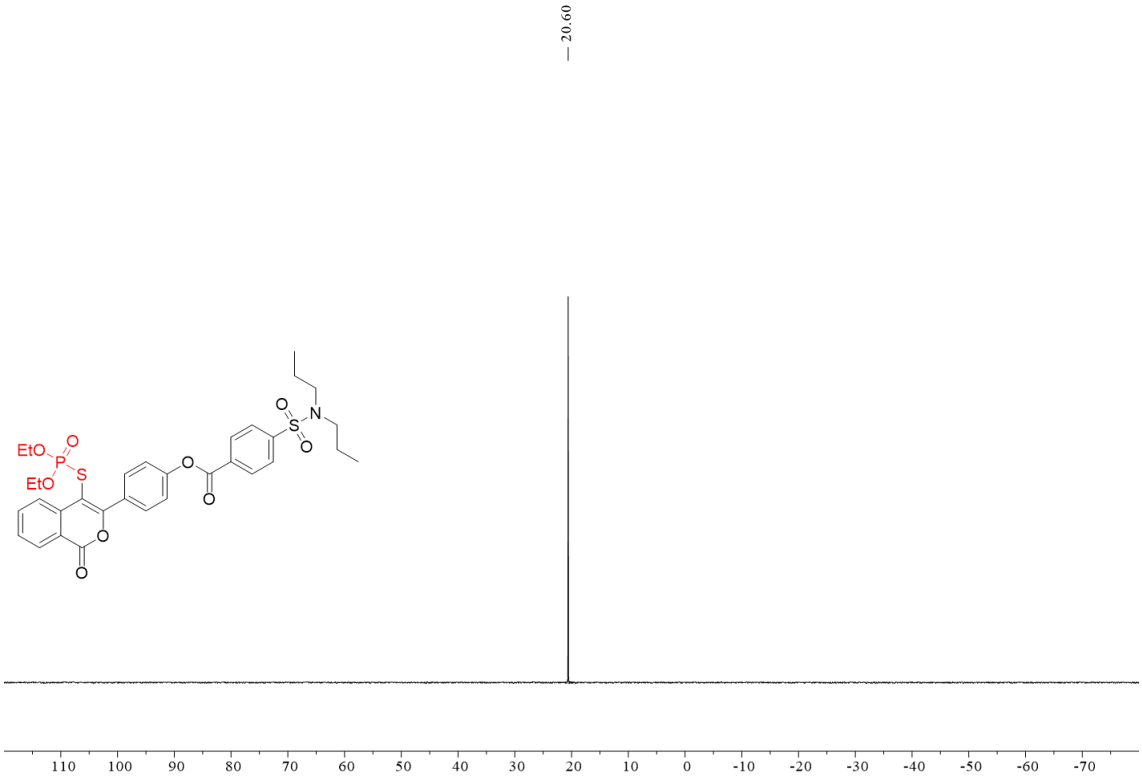
^{1}H NMR (400 MHz) Spectrum of **5d** in $CDCl_3$



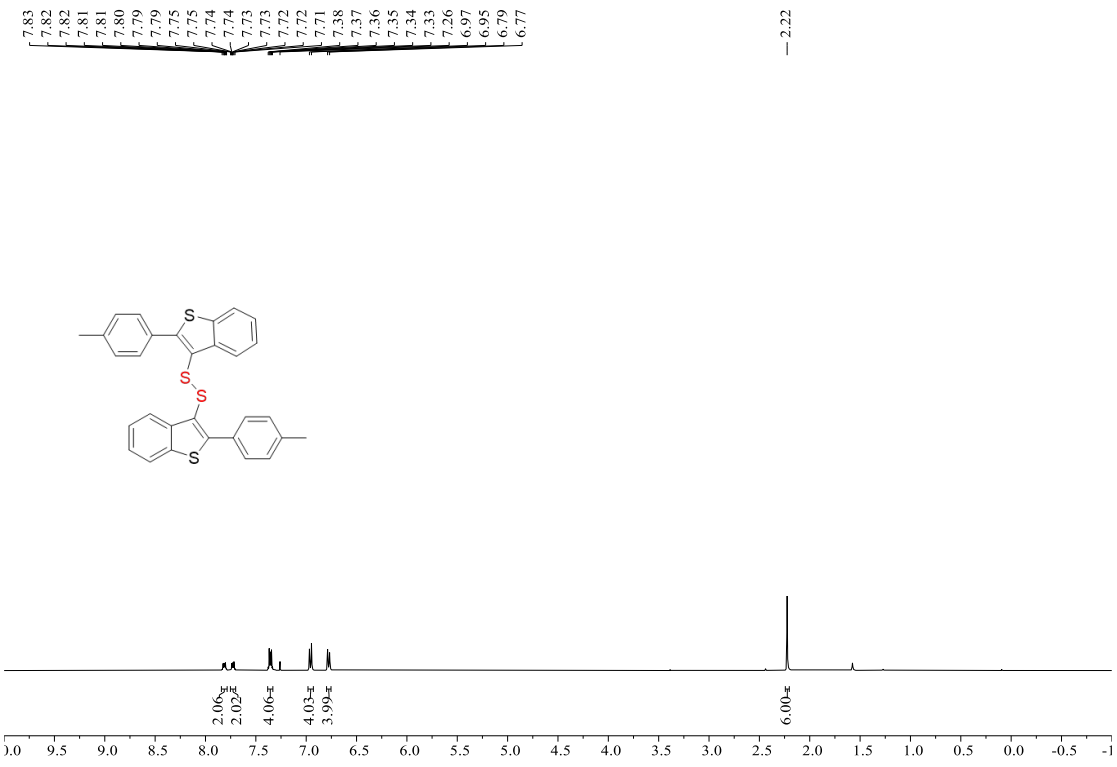
^{13}C NMR (101 MHz) Spectrum of **5d** in $CDCl_3$



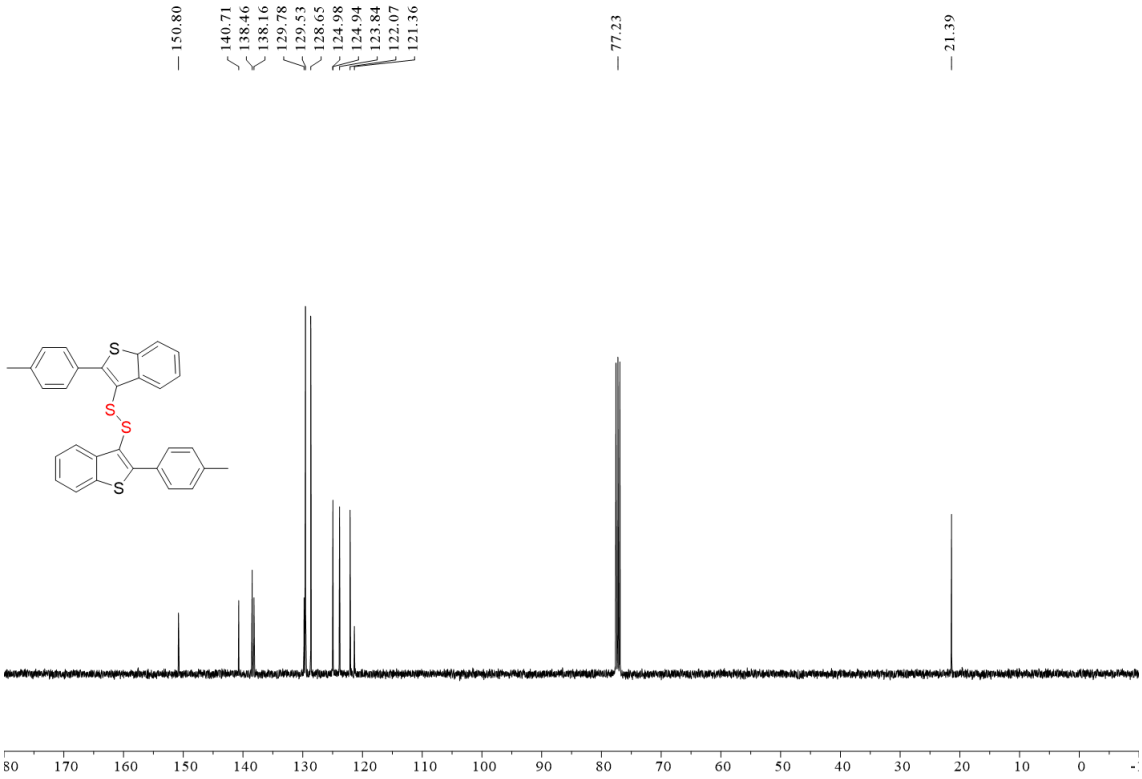
^{31}P NMR (162 MHz) Spectrum of **5d** in $CDCl_3$



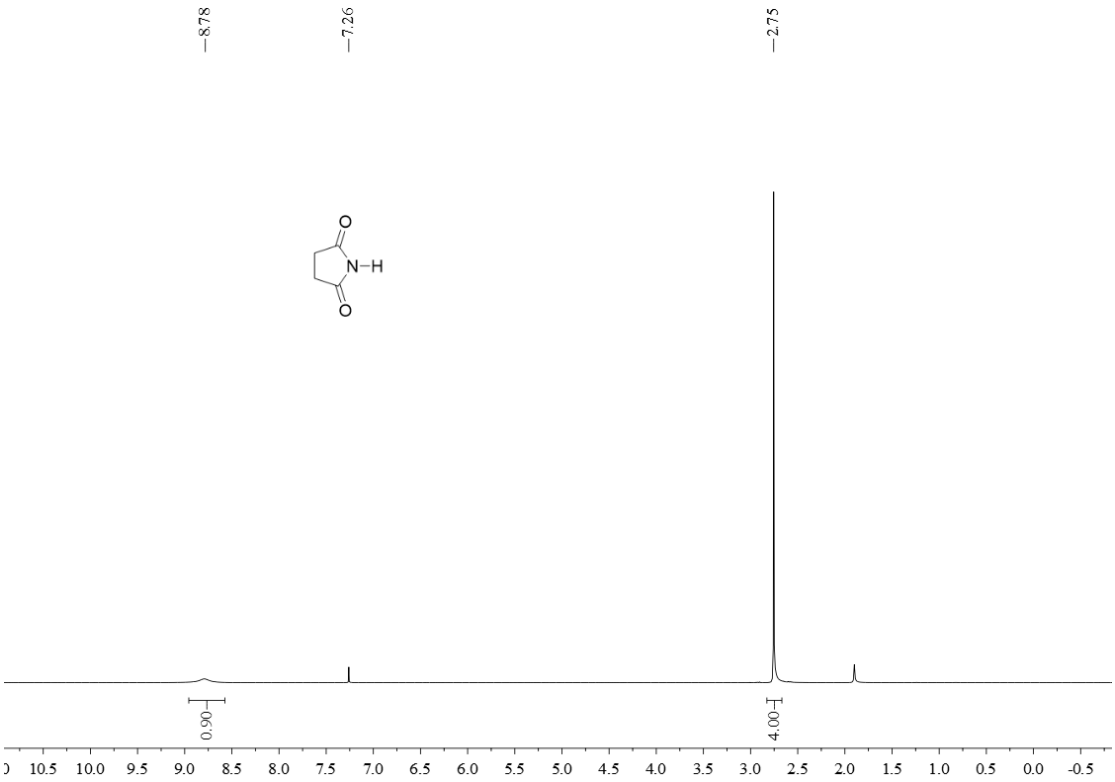
^{1}H NMR (400 MHz) Spectrum of **6** in $CDCl_3$



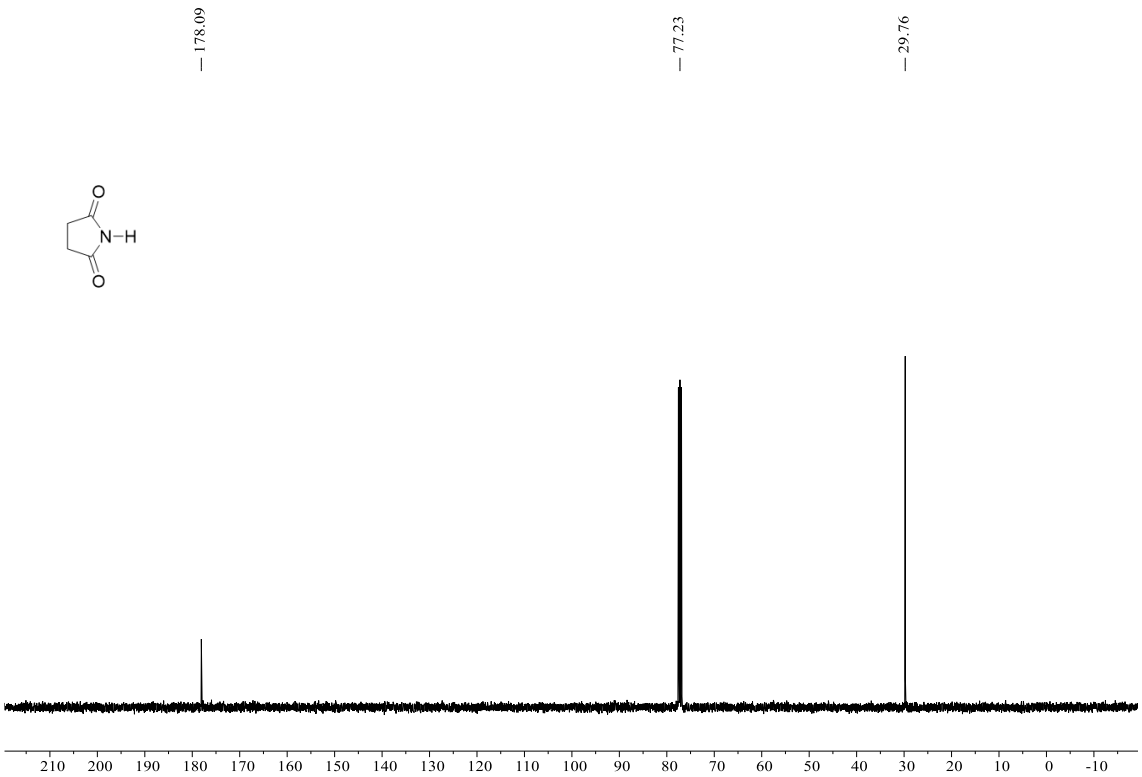
^{13}C NMR (101 MHz) Spectrum of **6** in $CDCl_3$



^{1}H NMR (400 MHz) Spectrum of **7** in $CDCl_3$



^{13}C NMR (101 MHz) Spectrum of **7** in $CDCl_3$



^{31}P NMR (162 MHz) Spectrum of the crude reaction mixture of **1a** with NCS.