Supporting Information

$K_2S_2O_8$-mediated metal-free direct P-H/C-H functionalization: a convenient route to benzo[*b*]phosphole oxides from unactivated alkynes

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1. General information and experimental section

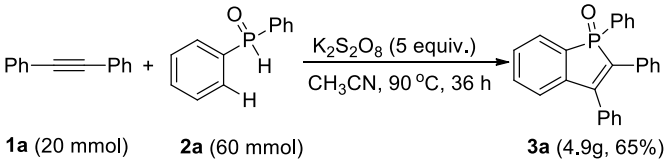
General information:

Substrates (**1b-1q** and **1u)** were prepared according to the known reference: *Org. Lett.* 2015, 17, 2522−2525. Other reagents were purchased from commercial sources and used without further purification. Spectroscopy data of the known compounds matches with the data reported in the corresponding references. ^{1}H, ^{13}C and ^{31}P NMR spectra were recorded on a Bruker Av500 spectrometer using tetramethylsilane (TMS) in $CDCl_3$ as the internal standard for ^{1}H, and ^{13}C NMR (1H NMR: TMS at 0.00 ppm, $CHCl_3$ at 7.26 ppm; ^{13}C NMR: $CDCl_3$ at 77.16 ppm) and 85% H_3PO_4 as external standard for ^{31}P NMR. Data are represented as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants in Hertz (Hz), integration. ESI mass spectra was aquired on a Bruker Dalton Esquire3000 Plus mass spectrometer. HRMS spectra of new compounds were recorded on a Waters Micromass LCT Premier TOF-MS apparatus. Infrared spectra were recorded on a Nicollet Avatar 330 spectrometer. Melting points were recorded on a WRS-1B Digital Melting-Point apparatus. The products were purified by column chromatography on silica gel 300-400 mesh.

Experimental section:

General procedure for the synthesis of **3**: $K_2S_2O_8$ (405 mg, 1.5 mmol), alkyne **1** (0.3 mmol) and diarylphosphine oxide **2** (0.9 mmol) were added to a sealed tube equipped with a magnetic stir bar under argon, and then CH_3CN (2 mL) were added to the sealed reaction tube by syringe. The resulting reaction mixture was kept stirring at 90 °C for 24 h. After required reaction time, the mixture was cooled down to room temperature and purified by flash chromatography (petroleum ether/ethyl acetate) to afford the corresponding product.

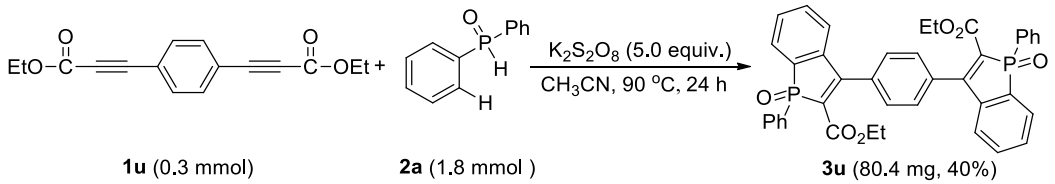
2. Gram-scale synthesis of 3a



Scheme 1 A Gram-Scale Synthesis of **3a**

$K_2S_2O_8$ (2.7 g, 100 mmol), **1a** (20 mmol, 3.6 g) and diphenylphosphine oxide **2a** (12 g, 100 mmol) were added to a round flask equipped with a magnetic stir bar under argon, and then CH_3CN (140 mL) were added by syringe. The resulting reaction mixture was kept stirring at 90 °C for 36 h. After 36 h, the mixture was cooled down to room temperature and purified by flash chromatography (petroleum ether/ethyl acetate) to afford the corresponding product.

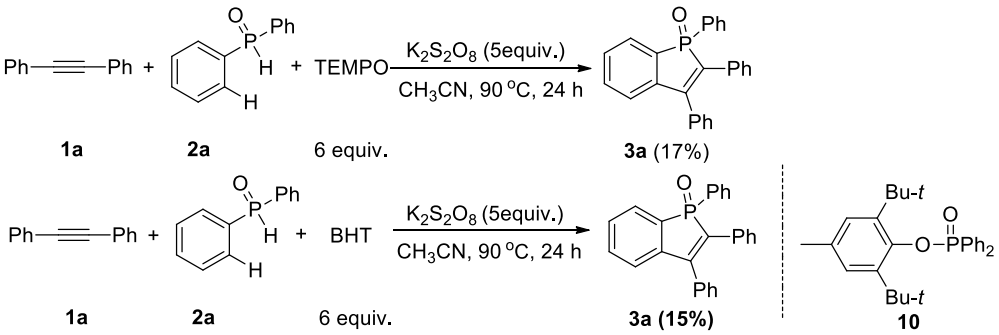
3. Application for the synthesis of 3u



Scheme 2 Application study

$K_2S_2O_8$ (810 mg, 3 mmol), **1u** (81 mg, 0.3 mmol) and diphenylphosphine oxide **2a** (362 mg, 1.8 mmol) were added to a sealed tube equipped with a magnetic stir bar under argon. Under argon, CH_3CN (4 mL) were added to the sealed reaction tube by syringe. The resulting reaction mixture was kept stirring at 90 °C for 24 h. After required reaction time, the mixture was cooled down to room temperature and purified by flash chromatography (petroleum ether/ethyl acetate) afforded the corresponding product.

4. Radical Trapping Study



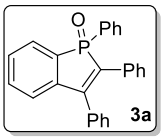
Scheme 3 Radical trapping study

The reaction was carried out according to the general procedure, except 6 equiv of TEMPO

(280 mg, 1.8 mmol) or BHT (396 mg, 1.8 mmol) was added. At the end of the reaction, the isolated yuield revealed formation of the target product **3a** decreased dramatically. The adduct **10** was detected by ESI-HRMS measurement of the crude reaction mixture (**HRMS (ESI**): $C_{27}H_{33}O_2PH^+$, Calcd: 421.2291, Found: 421.2296).

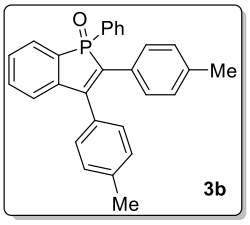
5. Spectral data and characterization of 3a-3u

1,2,3-Triphenyl-1H-phosphindole 1-oxide (3a, CAS 1478603-26-7)[1,2]



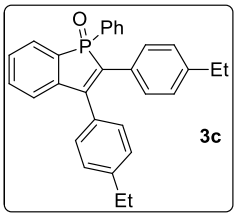
White solid, 90% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.80 (dd, J = 12.5, 7.2 Hz, 2 H), 7.69 (dd, J = 9.3, 7.9 Hz, 1 H), 7.45-7.32 (m, 10 H), 7.24-7.20 (m, 3 H), 7.08-7.07 (m, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 149.9 (d, $J_{C\text{-}P}$ = 21.3 Hz), 143.6 (d, $J_{C\text{-}P}$ = 26.8 Hz), 134.0 (d, $J_{C\text{-}P}$ = 14.9 Hz), 134.1 (d, $J_{C\text{-}P}$ = 95.8 Hz), 132.8 (d, $J_{C\text{-}P}$ = 1.4 Hz), 132.5 (d, $J_{C\text{-}P}$ = 9.8 Hz), 132.1 (d, $J_{C\text{-}P}$ = 2.6 Hz), 131.9 (d, $J_{C\text{-}P}$ = 121.4 Hz), 130.8 (d, $J_{C\text{-}P}$ = 10.6 Hz), 129.7 (d, $J_{C\text{-}P}$ = 99.7 Hz), 129.0 (d, $J_{C\text{-}P}$ = 11.0 Hz), 128.9 (d, $J_{C\text{-}P}$ = 2.7 Hz), 128.87, 128.84, 128.7 (d, $J_{C\text{-}P}$ = 12.5 Hz), 128.6, 128.1, 127.7, 123.9 (d, $J_{C\text{-}P}$ = 10.9 Hz). ^{31}P NMR (202 MHz, $CDCl_3$): δ 39.2. MS-ESI: *m/z* 379 $[M + H]^+$. IR (film) ν_{max}: 3057, 2921，1587, 1437, 1198 (P=O), 779, 634 cm^{-1}.

1-Phenyl-2,3-di-p-tolyl-1H-phosphindole 1-oxide (3b, CAS 1537189-01-7)[1]



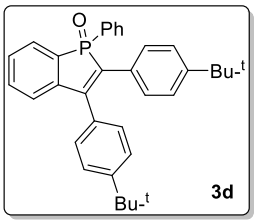
Pale yellow solid, 75% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.79-7.75 (m, 2 H), 7.68 (dd, J = 9.3, 7.4 Hz, 1 H), 7.45-7.32 (m, 5 H), 7.24-7.19 (m, 5 H), 7.15 (d, J = 7.8 Hz, 2 H), 6.90 (d, J = 8.1 Hz, 2 H), 2.40 (s, 3 H), 2.18 (s, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 148.5 (d, $J_{C\text{-}P}$ = 21.7 Hz), 143.1 (d, $J_{C\text{-}P}$ = 27.2 Hz), 137.6, 136.7, 132.8 (d, $J_{C\text{-}P}$ = 96.0 Hz), 131.9, 131.14 (d, $J_{C\text{-}P}$ = 2.6 Hz), 131.13 (d, $J_{C\text{-}P}$ = 105.5 Hz), 130.5, 130.02 (d, $J_{C\text{-}P}$ = 98.0 Hz), 130.03 (d, $J_{C\text{-}P}$ = 10.6 Hz), 128.9 (d, $J_{C\text{-}P}$ = 10.0 Hz), 128.8 (d, $J_{C\text{-}P}$ = 8.3 Hz), 128.7, 128.1, 128.01, 128.02, 127.9 (d, $J_{C\text{-}P}$ = 1.6 Hz), 127.88 (d, $J_{C\text{-}P}$ = 6.5 Hz), 123.0 (d, $J_{C\text{-}P}$ = 10.9 Hz), 20.5, 20.3. ^{31}P NMR (202MHz, $CDCl_3$,): δ 39.2. MS-ESI: *m/z* 407 $[M + H]^+$. IR (film) ν_{max}: 3054, 2921, 1586, 1437, 1197 (P=O), 732, 522 cm^{-1}.

2,3-Bis(4-ethylphenyl)-1-phenyl-1H-phosphindole 1-oxide (3c, new compound)



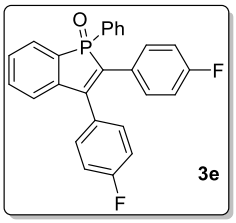
Yellow solid, 63% yield. mp 48.6-49.5 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7. 80-7.76 (m, 2 H), 7.68 (dd, J = 9.3, 7.6 Hz, 1 H), 7.47-7.30 (m, 7 H), 7.25-7.15 (m, 5 H), 6.91 (d, J = 8.2 Hz, 2 H), 2.71 (q, J = 7.6 Hz, 2 H), 2.49 (q, J = 7.5 Hz, 2 H), 1.28 (t, J = 7.6 Hz, 3 H), 1.10 (t, J = 7.6 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 149.9 (d, $J_{C\text{-}P}$ = 21.6 Hz), 144.8, 144.2 (d, $J_{C\text{-}P}$ = 27.3 Hz), 143.9, 133.7 (d, $J_{C\text{-}P}$ = 96.1 Hz), 132.9 (d, $J_{C\text{-}P}$ = 1.6 Hz), 132.13 (d, $J_{C\text{-}P}$ = 105.6 Hz), 132.14 (d, $J_{C\text{-}P}$ = 2.6 Hz), 131.7 (d, $J_{C\text{-}P}$ = 15.3 Hz), 131.0 (d, $J_{C\text{-}P}$ = 10.7 Hz), 130.3 (d, $J_{C\text{-}P}$ = 98.9 Hz), 130.1 (d, $J_{C\text{-}P}$ = 10.0 Hz), 129.1 (d, $J_{C\text{-}P}$ = 1.7 Hz), 129.0 (d, $J_{C\text{-}P}$ = 4.0 Hz), 128.97, 128.90 (d, $J_{C\text{-}P}$ = 1.5 Hz), 128.88 (d, $J_{C\text{-}P}$ = 2.6 Hz), 128.5, 127.8, 124.0 (d, $J_{C\text{-}P}$ = 10.9 Hz), 28.8, 28.6, 15.4, 15.1. ^{31}P NMR (202 MHz, $CDCl_3$): δ 39.2. IR (film) ν_{max}: 3055, 2964, 1587, 1437, 1196 (P=O), 732, 519 cm^{-1}. HRMS: $[M + H]^+$ *m/z* calcd for $C_{30}H_{27}OPH^+$: 435.1878, found: 435.1875.

2,3-Bis(4-(tert-butyl)phenyl)-1-phenyl-1H-phosphindole 1-oxide (3d, new compound)



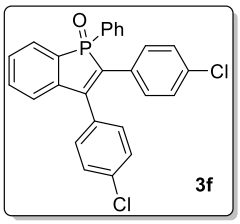
Yellow solid, 70% yield. mp 85.3-87.6 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7. 82-7.76 (m, 2 H), 7.67 (dd, J = 9.2, 7.4 Hz, 1 H), 7.50-7.39 (m, 6 H), 7.34-7.26 (m, 3 H), 7.20-7.14 (m, 3 H), 7.09 (d, J = 8.5 Hz, 2 H), 1.38 (m, 9 H), 1.19 (m, 9 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.8, 150.8, 149.4 (d, $J_{C\text{-}P}$ = 21.6 Hz), 144.4 (d, $J_{C\text{-}P}$ = 27.4 Hz), 133.3 (d, $J_{C\text{-}P}$ = 96.2 Hz), 132.9 (d, $J_{C\text{-}P}$ = 1.8 Hz), 132.2 (d, $J_{C\text{-}P}$ = 105.9 Hz), 132.1 (d, $J_{C\text{-}P}$ = 2.7 Hz), 131.7 (d, $J_{C\text{-}P}$ = 15.4 Hz), 131.1 (d, $J_{C\text{-}P}$ = 10.4 Hz), 130.2, 129.8, 129.4 (d, $J_{C\text{-}P}$ = 95.5 Hz), 129.0 (d, $J_{C\text{-}P}$ = 11.9 Hz), 128.9 (d, $J_{C\text{-}P}$ = 3.6 Hz), 128.8 (d, $J_{C\text{-}P}$ = 8.7 Hz), 128.78 (d, $J_{C\text{-}P}$ = 2.4 Hz), 126.0, 125.3, 124.1 (d, $J_{C\text{-}P}$ = 10.9 Hz), 34.9, 34.6, 31.5, 31.2. ^{31}P NMR (202 MHz, $CDCl_3$): δ 39.3. IR (film) ν_{max}: 3057, 2963, 1587, 1437, 1197 (P=O), 773, 523 cm^{-1}. HRMS: $[M + H]^+$ *m/z* calcd for $C_{34}H_{35}OPH^+$: 491.2504, found: 491.2500.

2,3-Bis(4-fluorophenyl)-1-phenyl-1H-phosphindole 1-oxide (3e, CAS 1478603-94-9)[2]



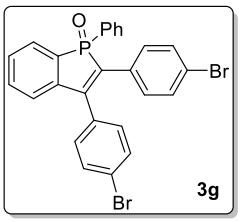
White solid, 51% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.74-7.68 (m, 3 H), 7.48-7.34 (m, 5 H), 7.30-7.27 (m, 2 H), 7.21-7.18 (m, 3 H), 7.14-7.10 (m, 2 H), 6.80-6.77 (m, 2 H).^{13}C NMR (125 MHz, $CDCl_3$): δ 162.7 (d, $J_{C\text{-}F}$ = 249.1 Hz), 162.1 (d, $J_{C\text{-}F}$ = 248.9 Hz), 148.7 (d, $J_{C\text{-}F}$ = 21.7 Hz), 143.3 (d, J = 26.6 Hz), 133.7 (d, $J_{C\text{-}P}$ = 96.0 Hz), 133.0 (d, J = 1.4 Hz), 132.3 (d, J = 2.6 Hz), 131.6 (d, $J_{C\text{-}P}$ = 106.0 Hz), 130.9 (d, J = 7.6 Hz), 130.8 (d, J = 9.2 Hz), 130.7 (dd, J = 6.2, 3.5 Hz), 130.1 (d, J = 103.9 Hz), 129.7 (dd, J = 15.2, 3.7 Hz), 129.2 (d, J = 24.9 Hz), 129.1 (d, J = 4.5 Hz), 128.8 (d, J = 12.5 Hz), 128.5 (dd, J = 10.0, 2.9 Hz) ,123.8 (d, J = 10.9 Hz), 116.2 (d, J = 21.6 Hz) , 115.4 (d, J = 21.5 Hz). ^{31}P NMR (202 MHz, $CDCl_3$): δ 38.9. MS-ESI: *m/z* 415 $[M + H]^+$. IR (film) ν_{max}: 3057, 2982， 1716, 1505, 1160 (P=O), 743, 561 cm^{-1}.

2,3-Bis(4-chlorophenyl)-1-phenyl-1H-phosphindole 1-oxide (3f, CAS 1537198-04-0)[1]



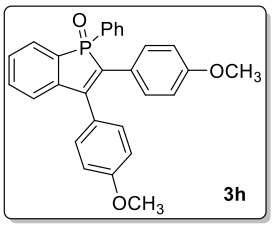
Pale yellow solid, 55% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.73-7.70 (m, 3 H), 7.51-7.47 (m, 2 H), 7.44-7.38 (m, 5 H), 7.26-7.25 (m, 2 H), 7.20-7.15 (m, 3 H),7.09 (d, J=8.6 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 149.2 (d, $J_{C\text{-}P}$ = 21.6 Hz), 143.3 (d, $J_{C\text{-}P}$ = 26.6 Hz), 135.1, 134.0 (d, $J_{C\text{-}P}$ = 95.4 Hz), 134.18, 134.19, 133.3 (d, $J_{C\text{-}P}$ = 1.8 Hz), 132.6 (d, $J_{C\text{-}P}$ = 2.6 Hz), 132.4 (d, $J_{C\text{-}P}$ = 15.0 Hz), 131.9 (d, $J_{C\text{-}P}$ = 107.5 Hz), 131.0 (d, $J_{C\text{-}P}$ = 10.7 Hz), 130.6, 130.3 (d, $J_{C\text{-}P}$ = 5.5 Hz), 129.65 (d, $J_{C\text{-}P}$ = 8.4 Hz), 129.61, 129.5 (d, $J_{C\text{-}P}$ = 9.6 Hz), 129.3 (d, $J_{C\text{-}P}$ = 100.5 Hz), 129.1 (d, J = 12.5 Hz), 128.9 , 124.0 (d, $J_{C\text{-}P}$ = 10.7 Hz). ^{31}P NMR (202 MHz, $CDCl_3$): δ 39.0. MS-ESI: *m/z* 446 $[M + H]^+$. IR (film) ν_{max}: 3048, 2941, 1585, 1483, 1197 (P=O), 773, 565 cm^{-1}.

2,3-Bis(4-bromophenyl)-1-phenyl-1H-phosphindole 1-oxide (3g, CAS 1537198-05-1)[1]



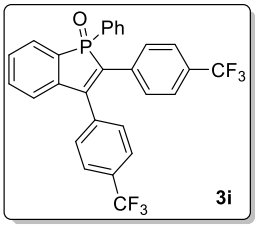
Pale yellow solid, 57% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.73-7.57 (m, 5 H), 7.47-7.39 (m, 5 H), 7.25-7.18 (m, 5 H), 7.10 (d, J =8.3 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 149.1 (d, $J_{C\text{-}P}$ = 21.6 Hz), 143.1 (d, $J_{C\text{-}P}$ = 26.4 Hz), 133.9 (d, $J_{C\text{-}P}$ = 95.5 Hz), 133.2 (d, $J_{C\text{-}P}$ = 1.4 Hz), 132.7 (d, $J_{C\text{-}P}$ = 14.7 Hz), 132.50 (d, $J_{C\text{-}P}$ = 12.8 Hz), 132.51, 131.8 (d, $J_{C\text{-}P}$ = 106.1 Hz), 131.7, 131.4 (d, $J_{C\text{-}P}$ = 9.9 Hz), 130.9 (d, $J_{C\text{-}P}$ = 10.6 Hz), 130.7, 130.5 (d, $J_{C\text{-}P}$ = 5.5 Hz), 129.6 (d, $J_{C\text{-}P}$ = 10.3 Hz), 129.4 (d, $J_{C\text{-}P}$ = 9.7 Hz), 129.2 (d, $J_{C\text{-}P}$ = 100.7 Hz), 129.0 (d, $J_{C\text{-}P}$ = 12.6 Hz), 124.0 (d, $J_{C\text{-}P}$ = 10.8 Hz) ,123.2, 122.5. ^{31}P NMR (202 MHz, $CDCl_3$): δ 38.9. MS-ESI: *m/z* 537 $[M + H]^+$. IR (film) ν_{max}: 3058, 2986, 1583, 1481, 1196 (P=O), 733, 540 cm^{-1}.

2,3-Bis(4-methoxyphenyl)-1-phenyl-1H-phosphindole 1-oxide (3h, CAS 1537189-02-8)[1]



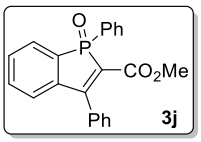
Yellow solid, 50% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.76 (dd, J = 12.3, 7.8 Hz, 2 H), 7.67 (dd, J = 8.4, 7.4 Hz, 1 H), 7.48-7.31 (m, 5 H), 7.28-7.20 (m, 5 H), 6.97 (d, J = 8.0 Hz, 2 H), 6.64 (d, J = 8.3 Hz, 2H), 3.86 (s, 3 H), 3.70 (s, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 159.7, 159.0, δ 148.1 (d, $J_{C\text{-}P}$ = 21.9 Hz), 144.2 (d, $J_{C\text{-}P}$ = 27.5 Hz), 133.1 (d, $J_{C\text{-}P}$ = 96.8 Hz), 132.8, 132.0 (d, $J_{C\text{-}P}$ = 2.7 Hz), 131.9 (d, $J_{C\text{-}P}$ = 106.0 Hz), 130.9 (d, $J_{C\text{-}P}$ = 10.7 Hz), 130.46, 130.4 (d, $J_{C\text{-}P}$ = 3.8 Hz), 130.38, 130.3 (d, $J_{C\text{-}P}$ = 99.6 Hz), 128.8 (d, $J_{C\text{-}P}$ = 34.7 Hz), 128.79 (d, $J_{C\text{-}P}$ = 12.5 Hz), 126.5 (d, $J_{C\text{-}P}$ = 15.5Hz), 125.3 (d, $J_{C\text{-}P}$ = 10.1 Hz), 123.7 (d, $J_{C\text{-}P}$ = 10.7 Hz), 114.5, 113.8, 55.3, 55.1. ^{31}P NMR (202 MHz, $CDCl_3$): δ 38.9. MS-ESI: *m/z* 439 $[M + H]^+$. IR (film) ν_{max}: 3058, 2930, 1606, 1501, 1195 (P=O), 736, 522 cm^{-1}.

1-Phenyl-2,3-bis(4-(trifluoromethyl)phenyl)-1H-phosphindole 1-oxide (3i, new compound)



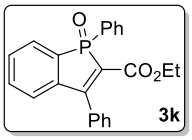
Pale yellow solid, 43% yield. mp 69.0-71.0 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.77-7.71 (m, 4 H), 7.53-7.40 (m, 8 H), 7.37 (d, J = 8.3, 2 H), 7.31 (d, J = 8.3 Hz, 2 H), 7.18-7.16 (m, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 150.0 (d, J = 21.4 Hz), 142.8 (d, J = 26.2 Hz), 137.5 (d, J = 14.9 Hz), 136.0 (d, J = 10.1 Hz), 134.7 (d, $J_{C\text{-}P}$ = 94.9 Hz), 133.4, 132.8 (d, $J_{C\text{-}P}$ = 2.5 Hz), 131.8 (d, $J_{C\text{-}P}$ = 106.9 Hz), 131.3 (q, $J_{C\text{-}F}$ = 33.2 Hz), 131.2 (d, J = 10.8 Hz), 130.1 (q, $J_{C\text{-}F}$ = 32.3 Hz), 129.7 (d, J = 10.1 Hz), 129.6, 129.3 (d, J = 1.8 Hz), 129.2 (d, J = 4.8 Hz), 128.9 (d, J = 101.2 Hz), 128.7 (d, J = 12.2 Hz), 126.3 (d, J = 3.5 Hz) , 125.6 (dd, J = 7.6, 3.4 Hz), 124.3 (d, J = 10.7 Hz), 123.9 (q, $J_{C\text{-}F}$ = 272.4 Hz), 123.7 (q, $J_{C\text{-}F}$ = 272.4 Hz). ^{31}P NMR (202 MHz, $CDCl_3$): δ 39.0. IR (film) ν_{max}: 3059, 2928, 1615, 1325, 1124 (P=O), 722, 562 cm^{-1}. HRMS: $[M + H]^+$ *m/z* calcd for $C_{28}H_{17}F_6OPH^+$: 515.0999, found: 515.1001.

Methyl 1,3-diphenyl-1H-phosphindole-2-carboxylate 1-oxide (3j, new compound)



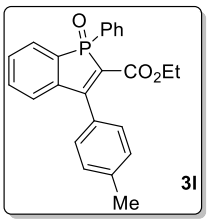
Yellow solid, 53% yield. mp 38.1-39.4 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.77-7.73 (m, 2 H), 7.71-7.67 (m, 1 H), 7.54-7.48 (m, 6 H), 7.46-7.35 (m, 4 H), 7.20-7.18 (m, 1 H), 3.55 (s, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 164.5 (d, $J_{C\text{-}P}$ = 18.3 Hz), 163.0 (d, $J_{C\text{-}P}$ = 12.0 Hz), 142.1 (d, $J_{C\text{-}P}$ = 25.2 Hz), 133.1 (d, $J_{C\text{-}P}$ = 1.4 Hz), 133.0, 132.49 (d, $J_{C\text{-}P}$ = 2.8 Hz), 132.46 (d, $J_{C\text{-}P}$ = 105.4 Hz), 131.9 (d, $J_{C\text{-}P}$ = 10.9 Hz), 130.9 (d, $J_{C\text{-}P}$ = 10.9 Hz), 129.5 (d, $J_{C\text{-}P}$ = 9.6 Hz), 129.4, 128.8 (d, $J_{C\text{-}P}$ = 13.1 Hz), 128.9 (d, $J_{C\text{-}P}$ = 106.2 Hz), 128.3, 127.9, 126.5 (d, $J_{C\text{-}P}$ = 10.5 Hz), 125.7 (d, $J_{C\text{-}P}$ = 98.0 Hz), 52.0. ^{31}P NMR (202 MHz, $CDCl_3$): δ 35.7. IR (film) ν_{max}: 3058, 2968, 1722, 1562, 1202 (P=O), 721, 549 cm^{-1}. HRMS: $[M + H]^+$ *m/z* calcd for $C_{22}H_{17}O_3PH^+$: 361.0994, found: 361.0999.

Ethyl 1,3-diphenyl-1H-phosphindole-2-carboxylate 1-oxide (3K, CAS 1478604-03-3)[2]



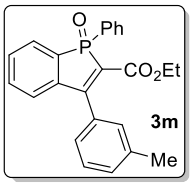
White solid, 60% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.79-7.74 (m, 2 H), 7.73-7.69 (m, 1 H), 7.55-7.47 (m, 6 H), 7. 45-7.36 (m, 4 H), 7.22-7.19 (m, 1 H), 4.08-3.94 (m, 2 H), 0.95 (t, J =7.1 Hz, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 164.0 (d, $J_{C\text{-}P}$ = 18.3 Hz), 162.6 (d, $J_{C\text{-}P}$ = 12.5 Hz), 142.3 (d, $J_{C\text{-}P}$ = 24.8 Hz), 133.2, 133.1, 132.6 (d, $J_{C\text{-}P}$ = 106.1 Hz), 132.5 (d, $J_{C\text{-}P}$ = 2.9 Hz), 131.8 (d, $J_{C\text{-}P}$ = 10.6 Hz), 131.1 (d, $J_{C\text{-}P}$ = 11.2 Hz), 129.6 (d, $J_{C\text{-}P}$ = 9.3 Hz), 129.4, 129.1 (d, $J_{C\text{-}P}$ = 106.4 Hz), 128.8 (d, $J_{C\text{-}P}$ = 12.8 Hz), 128.3, 128.0, 126.5 (d, $J_{C\text{-}P}$ = 10.7 Hz), 126.4 (d, $J_{C\text{-}P}$ = 98.3 Hz), 60.9, 13.7. ^{31}P NMR (202 MHz, $CDCl_3$): δ 35.8. MS-ESI: *m/z* 375 $[M + H]^+$. IR (film) ν_{max}: 3048, 2981, 1716, 1562, 1208 (P=O), 752, 550 cm^{-1}.

Ethyl 1-phenyl-3-(p-tolyl)-1H-phosphindole-2-carboxylate 1-oxide (3l, new compound)



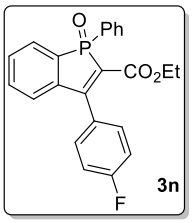
White solid, 61% yield. mp 159.0-159.8 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.78-7.74 (m, 2 H), 7. 73-7.69 (m, 1 H), 7.56-7.49 (m, 3 H), 7.46-7.42 (m, 2 H), 7.32-7.25 (m, 5 H), 4.11-3.96 (m, 2 H), 2.44 (s, 3 H), 0.97 (t, J =7.1 Hz, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 164.5 (d, $J_{C\text{-}P}$ = 18.6 Hz), 162.7 (d, $J_{C\text{-}P}$ = 12.0 Hz), 142.5 (d, $J_{C\text{-}P}$ = 25.0 Hz), 139.7, 133.1, 132.5 (d, $J_{C\text{-}P}$ = 2.7 Hz), 131.9 (d, $J_{C\text{-}P}$ = 104.6 Hz), 131.8 (d, $J_{C\text{-}P}$ = 10.7 Hz), 131.2 (d, $J_{C\text{-}P}$ = 11.0 Hz), 130.1 (d, $J_{C\text{-}P}$ = 12.8 Hz), 129.3 (d, $J_{C\text{-}P}$ = 105.6 Hz), 129.6 (d, $J_{C\text{-}P}$ = 9.7 Hz), 129.1, 128.8 (d, $J_{C\text{-}P}$ = 12.8 Hz), 128.2, 126.6 (d, $J_{C\text{-}P}$ = 10.4 Hz), 125.9 (d, $J_{C\text{-}P}$ = 98.3 Hz), 60.9, 21.6, 13.8. ^{31}P NMR (202 MHz, $CDCl_3$): δ 35.7. IR (film) ν_{max}: 3038, 2981, 1719, 1562, 1209 (P=O), 768, 562 cm^{-1}. HRMS: $[M + H]^+$ *m/z* calcd for $C_{24}H_{21}O_3PH^+$: 389.1307, found: 389.1310

Ethyl 1-phenyl-3-(m-tolyl)-1H-phosphindole-2-carboxylate 1-oxide (3m, new compound)



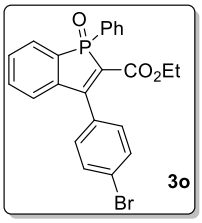
Yellow solid, 48% yield. mp 158.2-160.0 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.80-7.78 (m, 2 H), 7.73-7.71 (m, 1 H), 7.57-7.51 (m, 3 H), 7.47-7.38 (m, 3 H), 7.30-7.29 (m, 1 H), 7.23-7.18 (m, 3 H), 4.11-3.96 (m, 2 H), 2.43 (s, 3 H), 0.97 (t, J =7.1 Hz, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 164.5 (d, $J_{C\text{-}P}$ = 18.3 Hz), 162.7 (d, $J_{C\text{-}P}$ = 12.3 Hz), 142.5 (d, $J_{C\text{-}P}$ = 25.2 Hz), 139.1, 133.21, 133.19 (d, $J_{C\text{-}P}$ = 1.5 Hz), 132.7 (d, $J_{C\text{-}P}$ = 106.6 Hz), 132.5 (d, $J_{C\text{-}P}$ = 3.4 Hz), 131.8 (d, $J_{C\text{-}P}$ = 10.8 Hz), 131.2 (d, $J_{C\text{-}P}$ = 10.9 Hz), 130.2, 129.6 (d, $J_{C\text{-}P}$ = 9.5 Hz), 129.3 (d, $J_{C\text{-}P}$ = 106.2 Hz), 128.8 (d, $J_{C\text{-}P}$ = 12.9 Hz), 128.3, 126.6 (d, $J_{C\text{-}P}$ = 10.7 Hz), 126.3 (d, $J_{C\text{-}P}$ = 98.2 Hz), 60.9, 21.6, 13.8. ^{31}P NMR (202 MHz, $CDCl_3$): δ 35.8. IR (film) ν_{max}: 3038, 2920, 1718, 1559, 1211 (P=O), 751, 554 cm^{-1}. HRMS: $[M + H]^+$ *m/z* calcd for $C_{24}H_{21}O_3PH^+$: 389.1307, found: 389.1300.

Ethyl 3-(4-fluorophenyl)-1-phenyl-1H-phosphindole-2-carboxylate 1-oxide (3n, new compound)



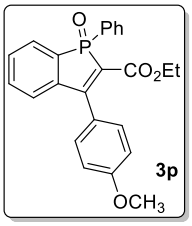
Yellow solid, 51% yield. mp 54.0-55.7 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.77-7.72 (m, 3 H), 7.56-7.51 (m, 3 H), 7.45-7.38 (m, 4 H), 7.21-7.18 (m, 3 H), 4.10-3.95 (m, 2 H), 0.98 (t, J= 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 163.4 (d, $J_{C\text{-}F}$ = 249.7 Hz), 163.0 (d, J = 18.9 Hz),162.5 (d, J = 12.1 Hz), 142.2 (d, J = 25.0 Hz), 133.3 (d, J = 1.2 Hz), 132.57 (d, $J_{C\text{-}P}$ = 105.8 Hz), 132.58 (d, J = 2.6 Hz), 132.0 (d, J = 10.7Hz), 131.2 (d, J = 11.0 Hz), 130.2, 129.8 (d, J = 9.6 Hz), 129.0 (d, $J_{C\text{-}P}$ = 106.6 Hz), 128.9 (dd, J = 11.3, 3.5 Hz), 128.8 (d, J = 12.9 Hz), 126.7 (d, $J_{C\text{-}P}$ = 96.0 Hz), 126.3 (d, J = 10.5 Hz), 115.6 (d, J = 21.8 Hz), 61.1, 13.7. ^{31}P NMR (202 MHz, $CDCl_3$): δ 35.8. IR (film) ν_{max}: 3057, 2982, 1716, 1505, 1160 (P=O), 743, 561 cm^{-1}. HRMS: $[M+H]^+$ *m/z* calcd for $C_{23}H_{18}FO_3PH^+$: 393.1056, found: 393.1052.

Ethyl 3-(4-bromophenyl)-1-phenyl-1H-phosphindole-2-carboxylate 1-oxide (3o, new compound)



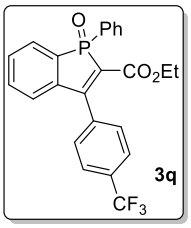
White solid, 33% yield. mp 154.2-155.1 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.76-7.71 (m, 3 H), 7.62-7.61 (m, 2 H), 7.53-7.43 (m, 5 H), 7.26-7.17 (m, 3 H), 4.07-3.96 (m, 2 H), 0.97 (t, J = 6.8 Hz, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 162.6 (d, $J_{C\text{-}P}$ = 18.7 Hz), 162.3 (d, $J_{C\text{-}P}$ = 11.8 Hz), 141.8 (d, $J_{C\text{-}P}$ = 24.7 Hz), 133.2, 132.4 (d, $J_{C\text{-}P}$ = 103.6 Hz), 132.6 (d, $J_{C\text{-}P}$ = 2.2 Hz), 132.0 (d, $J_{C\text{-}P}$ = 10.6 Hz), 131.9 (d, $J_{C\text{-}P}$ = 13.4 Hz), 131.6, 131.1 (d, $J_{C\text{-}P}$ = 11.0 Hz), 129.74, 129.70 (d, $J_{C\text{-}P}$ = 9.2 Hz), 128.8 (d, $J_{C\text{-}P}$ = 106.4 Hz), 128.7 (d, $J_{C\text{-}P}$ = 12.8 Hz), 126.8 (d, $J_{C\text{-}P}$ = 97.4 Hz), 126.2 (d, $J_{C\text{-}P}$ = 10.4 Hz), 123.7, 61.0, 13.7. ^{31}P NMR (202 MHz, $CDCl_3$): δ 35.9. IR (film) ν_{max}: 3057, 2981, 1716, 1557, 1207 (P=O), 744, 550 cm^{-1}. HRMS: $[M + H]^+$ *m/z* calcd for $C_{23}H_{18}BrO_3PH^+$: 453.0255, found: 453.0260.

Ethyl 3-(4-methoxyphenyl)-1-phenyl-1H-phosphindole-2-carboxylate 1-oxide (3p, new compound)



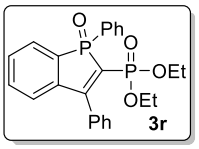
Yellow solid, 53% yield. mp 137.0-137.7 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.76-7.69 (m, 3 H), 7.51-7.26 (m, 8 H), 7.01 (m, J = 7.8 Hz, 2 H), 4.09-3.95 (m, 2 H), 3.85 (s, 3 H), 0.96 (t, J = 6.5 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 164.0 (d, $J_{C\text{-}P}$ = 18.6 Hz), 162.7 (d, $J_{C\text{-}P}$ = 12.0 Hz), 160.7, 142.4 (d, $J_{C\text{-}P}$ = 25.2 Hz), 133.0, 132.7 (d, $J_{C\text{-}P}$ = 105.2 Hz), 132.4 (d, $J_{C\text{-}P}$ = 2.5 Hz), 131.7 (d, $J_{C\text{-}P}$ = 10.6 Hz), 131.09 (d, $J_{C\text{-}P}$ = 11.0 Hz), 131.05, 129.5 (d, $J_{C\text{-}P}$ = 9.6 Hz), 129.3 (d, $J_{C\text{-}P}$ = 106.4 Hz), 128.7 (d, $J_{C\text{-}P}$ = 12.9 Hz), 126.5 (d, $J_{C\text{-}P}$ = 10.5 Hz), 125.3 (d, $J_{C\text{-}P}$ = 98.8 Hz), 124.8 (d, $J_{C\text{-}P}$ = 13.1 Hz), 113.7, 60.8, 55.4, 13.7. ^{31}P NMR (202 MHz, $CDCl_3$): δ 35.4. IR (film) ν_{max}: 3028, 2980, 1716, 1508, 1180 (P=O), 768, 552 cm^{-1}. HRMS: $[M + H]^+$ *m/z* calcd for $C_{24}H_{21}O_4PH^+$: 405.1256, found: 405.1251.

Ethyl 1-phenyl-3-(4-(trifluoromethyl)phenyl)-1H-phosphindole-2-carboxylate 1-oxide (3q, new compound)



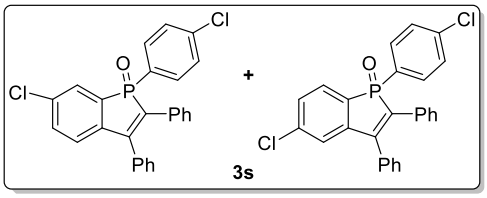
White solid, 38% yield. Mp 177.6-178.8 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.78-7.71 (m, 5 H), 7.56-7.43 (m, 7 H), 7.11-7.10 (m, 1 H), 4.08-3.94 (m, 2 H), 0.95 (t, J = 7.1 Hz, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 162.3 (d, J = 4.2 Hz), 162.2 (d, J = 2.0 Hz), 141.7 (d, J = 24.8 Hz), 137.0 (d, J = 13.1 Hz), 133.4 (d, J = 1.3 Hz), 132.4 (d, $J_{C\text{-}P}$ = 106.1 Hz), 132.7 (d, J = 2.7 Hz), 132.2 (d, J = 10.9 Hz), 131.3 (q, $J_{C\text{-}F}$ = 32.4 Hz), 131.1 (d, J = 10.9 Hz), 129.9 (d, J = 9.4 Hz), 128.8 (d, J = 12.9 Hz), 128.7 (d, $J_{C\text{-}P}$ = 106.6 Hz), 128.5, 127.5 (d, $J_{C\text{-}P}$ = 96.9 Hz), 126.2 (d, J = 10.6 Hz), 125.4 (d, J = 2.9 Hz), 123.9 (d, $J_{C\text{-}F}$ = 272.4 Hz), 61.2, 13.6. ^{31}P NMR (202 MHz, $CDCl_3$): δ 35.8. IR (film) ν_{max}: 3028, 2968, 1716, 1324, 1206 (P=O), 768, 549 cm^{-1}. HRMS: $[M + H]^+$ *m/z* calcd for $C_{24}H_{18}F_3O_3PH^+$: 443.1024, found: 443.1028.

Diethyl (1-oxido-1,3-diphenyl-1H-phosphindol-2-yl)phosphonate (3r, CAS 1537189-12-0)[1]



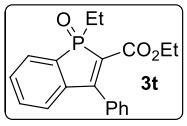
Colorless solid, 57% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.80 (dd, J =12.9, 7.6 Hz ,2 H), 7.67-7.64 (m, 1 H), 7.52-7.42 (m, 10 H), 7.17-7.16 (m, 1 H), 3.93-3.78 (m, 3 H), 3.69-3.61 (m, 1 H), 1.05 (t, J = 7.0 Hz, 3 H), 0.93 (t, J = 7.0 Hz, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 167.0 (dd, $J_{C\text{-}P}$ = 13.1, 7.2 Hz), 142.5 (dd, $J_{C\text{-}P}$ = 27.5, 20.8 Hz), 133.9 (dd, $J_{C\text{-}P}$ = 104.8, 7.4 Hz), 133.4 (dd, $J_{C\text{-}P}$ = 11.6, 3.3 Hz), 133.0, 132.4 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.3 (d, $J_{C\text{-}P}$ = 10.6 Hz), 131.2 (d, $J_{C\text{-}P}$ = 11.0 Hz), 129.7, 129.2 (d, $J_{C\text{-}P}$ = 9.8 Hz), 129.0 (d, $J_{C\text{-}P}$ = 105.6 Hz), 128.7 (d, $J_{C\text{-}P}$ = 12.8 Hz), 128.4, 128.2, 125.9 (d, $J_{C\text{-}P}$ = 11.3 Hz), 125.4 (dd, $J_{C\text{-}P}$ = 188.7, 80.5 Hz), 62.3 (d, $J_{C\text{-}P}$ = 6.0Hz), 61.9 (d, $J_{C\text{-}P}$ = 5.8 Hz), 16.0 (d, $J_{C\text{-}P}$ = 7.2 Hz), 15.9(d, $J_{C\text{-}P}$ = 6.4 Hz). ^{31}P NMR (202 MHz, $CDCl_3$): δ 40.8 (d, J =39.7 Hz), 11.2 (d,J =39.7 Hz). MS-ESI: *m/z* 439 $[M + H]^+$. IR (film) ν_{max}: 3068, 2982, 1546, 1438, 1206 (P=O), 1111 (P=O), 721, 575 cm^{-1}.

5-Chloro-1-(4-chlorophenyl)-2,3-diphenyl-1H-phosphindole 1-oxide compound with 6-chloro-1-(4-chlorophenyl)-2,3-diphenyl-1H-phosphindole 1-oxide (3s, 1:1, CAS 1478603-76-7, 1478603-73-4)[2]



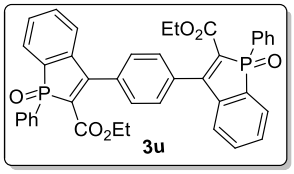
White solid, 61% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.71 (m, 5 H), 7.45-7.35 (m, 9 H), 7.31-7.30 (m, 3 H), 7.22-7.14 (m, 5 H), 7.11-7.09 (m, 4 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 149.6 (d, $J_{C\text{-}P}$ = 20.8 Hz), 149.1 (d, $J_{C\text{-}P}$ = 20.7 Hz), 145.8 (d, $J_{C\text{-}P}$ = 28.8 Hz), 142.0 (d, $J_{C\text{-}P}$ = 27.2 Hz), 140.0 (d, $J_{C\text{-}P}$ = 2.7 Hz), 139.3 (d, $J_{C\text{-}P}$ = 3.3 Hz), 139.2 (d, $J_{C\text{-}P}$ = 3.1 Hz), 135.8, 135.7, 135.6 (d, $J_{C\text{-}P}$ = 95.8 Hz), 134.4-133.4 (m), 133.1 (d, $J_{C\text{-}P}$ = 1.8 Hz), 132.4 (d, $J_{C\text{-}P}$ = 11.6 Hz), 132.2 (d, $J_{C\text{-}P}$ = 10.0 Hz), 132.1, 130.2 (d, $J_{C\text{-}P}$ = 10.8 Hz), 127.7 (d, $J_{C\text{-}P}$ =101.6 Hz), 125.4 (d, $J_{C\text{-}P}$ = 11.8 Hz), 124.7 (d, $J_{C\text{-}P}$= 11.7 Hz). ^{31}P NMR (202 MHz, $CDCl_3$): δ 37.3 (s), 37.0.

Ethyl 1-ethyl-3-phenyl-1H-phosphindole-2-carboxylate 1-oxide (3t, new compound)



White solid, 63% yield. Mp 139.7-140.0 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.83 (t, J = 8.0 Hz, 1 H), 7.55-7.51 (m, 1 H), 7.48-7.47 (m, 4H), 7.33-7.27 (m, 2 H), 7.14-7.12 (m, 1 H), 4.23-4.10 (m, 2 H), 2.49-2.22 (m, 2 H), 1.16-1.04 (m, 6 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 163.2 (d, $J_{C\text{-}P}$ = 3.1 Hz), 163.1 (d, $J_{C\text{-}P}$ = 7.4 Hz), 141.9 (d, $J_{C\text{-}P}$ = 23.6 Hz), 133.3 (d, $J_{C\text{-}P}$ = 11.9 Hz), 133.0, 131.4 (d, $J_{C\text{-}P}$ = 10.1 Hz), 131.1 (d, $J_{C\text{-}P}$ = 99.5 Hz), 129.2, 129.0 (d, $J_{C\text{-}P}$ = 9.3 Hz), 128.3, 128.0, 126.4 (d, $J_{C\text{-}P}$ = 9.8 Hz), 124.7 (d, $J_{C\text{-}P}$ = 93.5 Hz), 61.1, 22.1 (d, $J_{C\text{-}P}$ = 71.6 Hz), 14.0, 6.2 (d, $J_{C\text{-}P}$ = 3.7 Hz). ^{31}P NMR (202 MHz, $CDCl_3$): δ 49.9. IR (film) ν_{max} : 3392, 2919, 1720, 1645, 1195 (P=O), 766, 700 cm^{-1}. HRMS: $[M + H]^+$ m/z calcd for $C_{19}H_{19}O_3PH^+$: 372.1150, found: 372.1145.

Diethyl 3,3'-(1,4-phenylene)bis(1-phenyl-1H-phosphindole-2-carboxylate 1-oxide) (3u, CAS 1478604-30-6)[2]



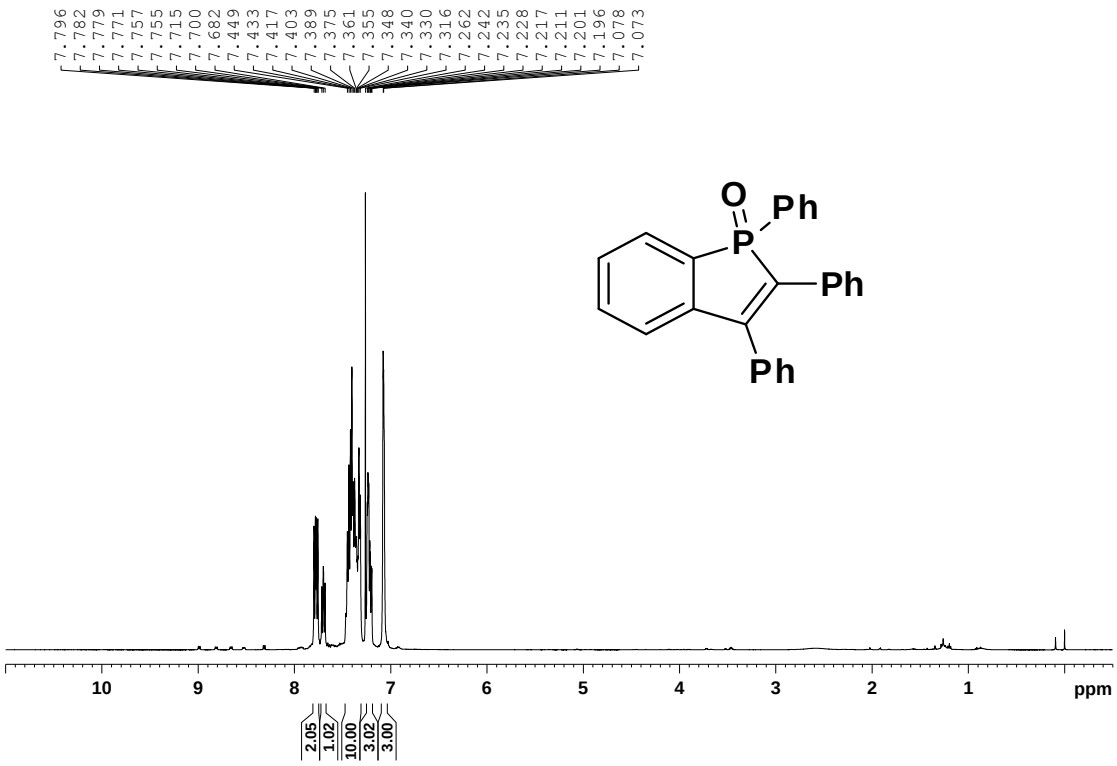
White solid, 40 % yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.80-7.74 (m, 6 H), 7.58-7.55 (m, 10 H), 7.48-7.45 (m, 4 H), 7.33-7.30 (m, 2 H), 4.14-4.01 (m, 4 H), 1.02 (td, J = 7.1, 3.7 Hz, 6 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 163.2 (d, $J_{C\text{-}P}$ = 18.7 Hz), 162.6 (dd, $J_{C\text{-}P}$ = 11.7, 8.2 Hz), 142.0 (dd, $J_{C\text{-}P}$ = 24.7, 3.9 Hz), 134.3 (dd, $J_{C\text{-}P}$ = 25.2, 2.6 Hz), 133.4, 132.65 (d, $J_{C\text{-}P}$ = 2.4 Hz), 132.62 (d, $J_{C\text{-}P}$ = 106.1 Hz), 132.1 (d, $J_{C\text{-}P}$ = 11.0 Hz), 131.2 (d, $J_{C\text{-}P}$ = 11.0 Hz), 129.9 (dd, $J_{C\text{-}P}$ = 9.2, 1.7 Hz), 129.0 (d, $J_{C\text{-}P}$ = 106.4 Hz), 128.9 (dd, $J_{C\text{-}P}$ = 12.9, 1.7 Hz), 128.2, 127.4 (d, $J_{C\text{-}P}$ = 7.6 Hz), 127.0 (d, $J_{C\text{-}P}$ = 90.1 Hz), 126.6 (dd, $J_{C\text{-}P}$ = 11.0, 5.1Hz), 61.2, 13.8. ^{31}P NMR (202 MHz, $CDCl_3$): δ 35.7. MS-ESI: *m/z* 671 $[M + H]^+$. IR (film) ν_{max}: 3050, 2982, 1719, 1438, 1206 (P=O), 1113 (P=O), 724, 551 cm^{-1}.

6. References

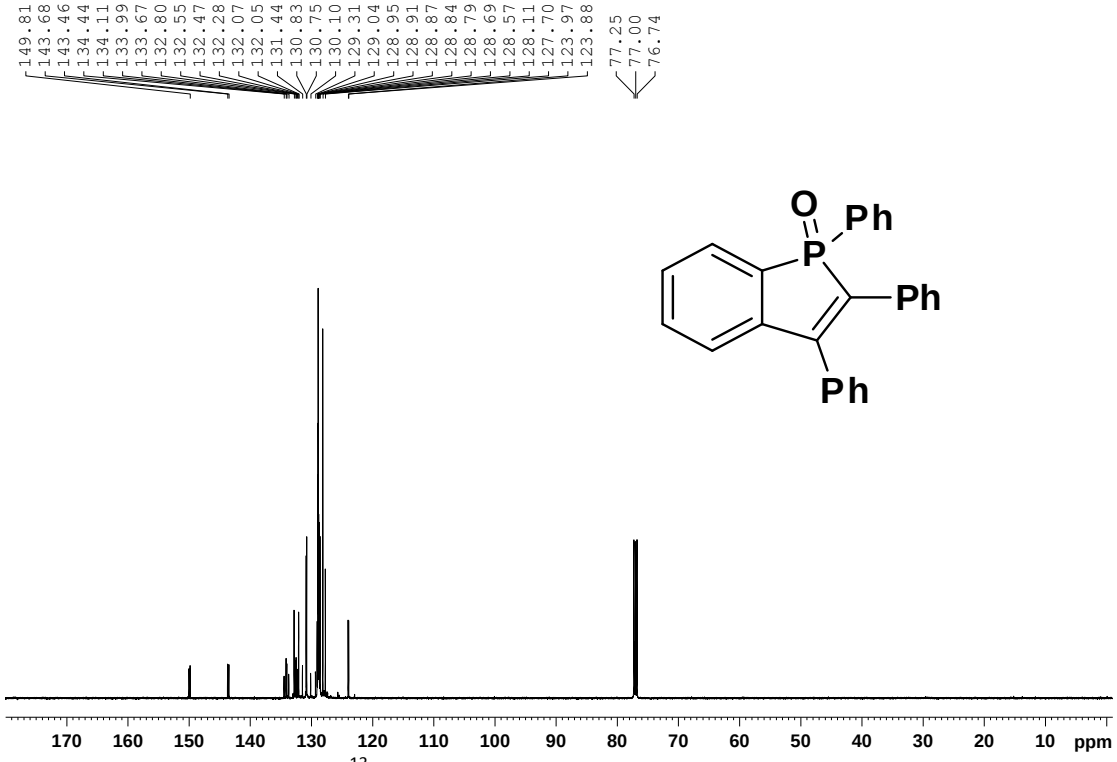
1. Y. Unoh, K. Hirano, T. Satoh and M. Miura, *Angew. Chem. Int. Ed.,* 2013, **52**, 12975.

2. Y.-R. Chen and W.-L. Duan, *J. Am. Chem. Soc.,* 2013, **135**, 16754.

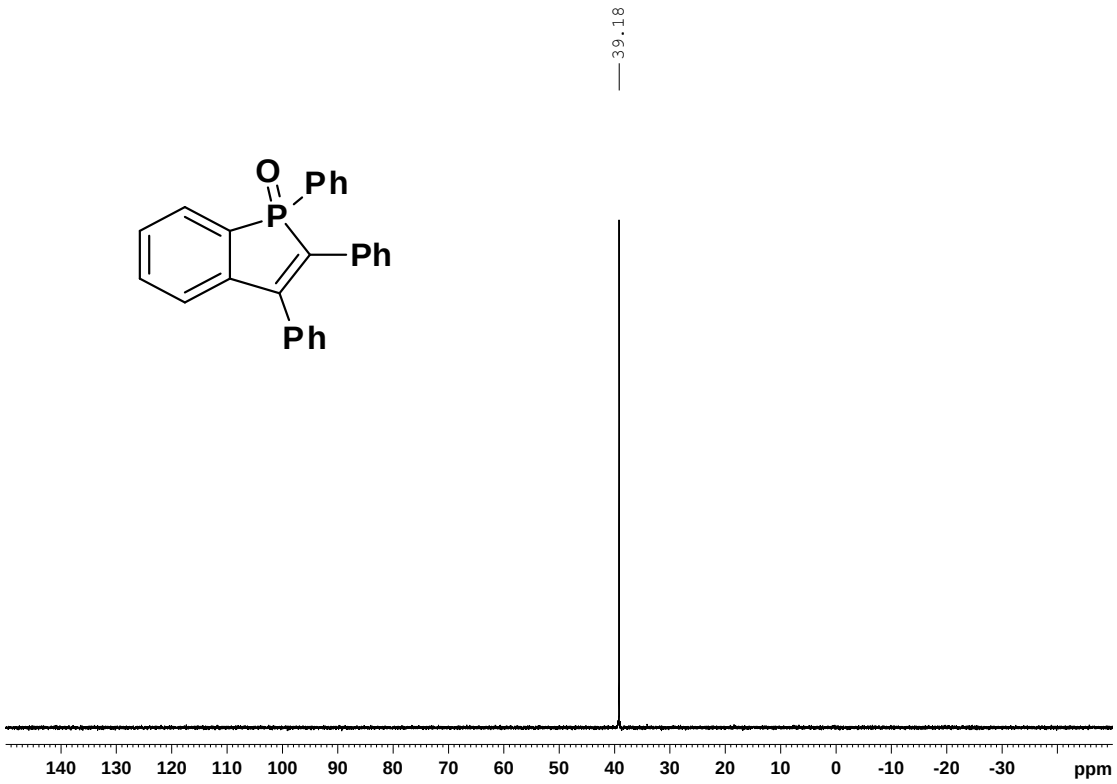
7. NMR Spectra of the Compounds of 3a-3u



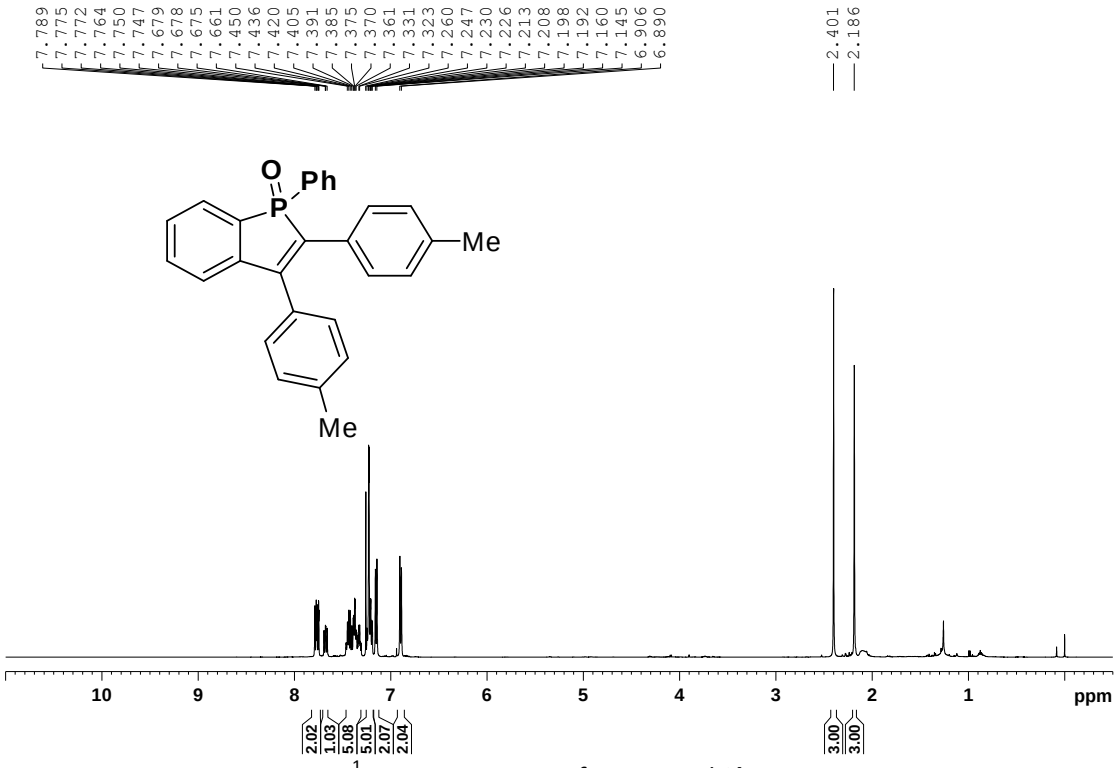
^{1}H NMR spectrum of compound **3a**.



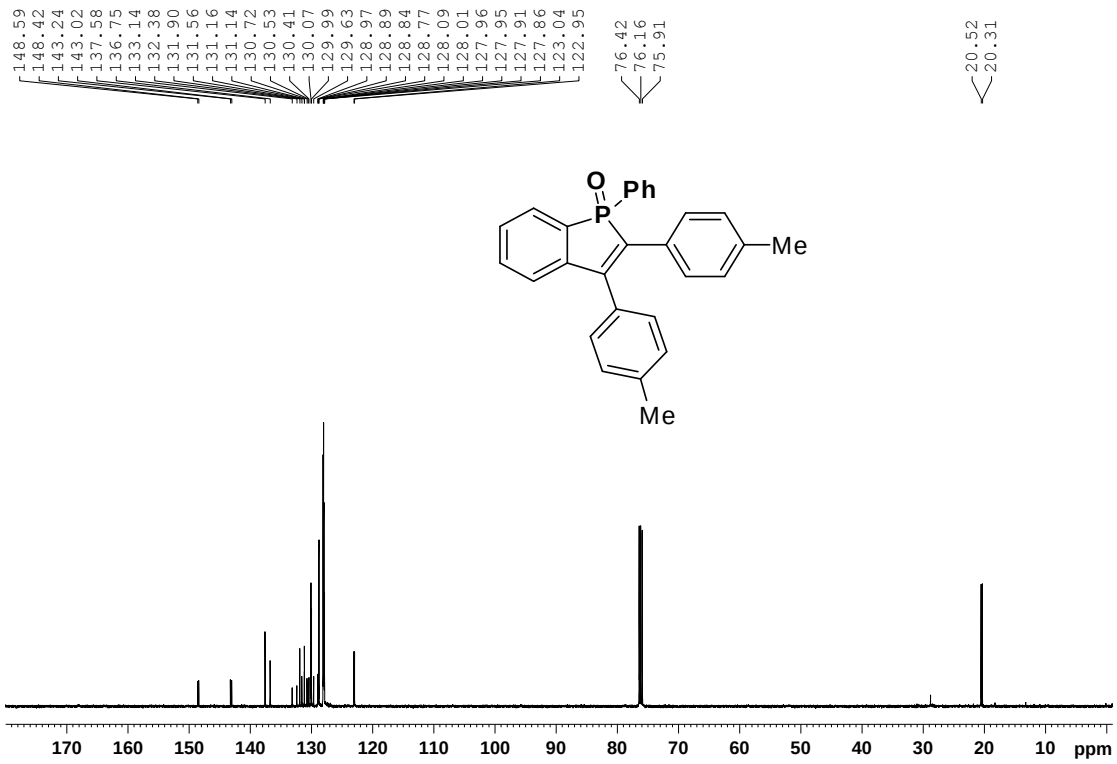
^{13}C NMR spectrum of compound **3a**.



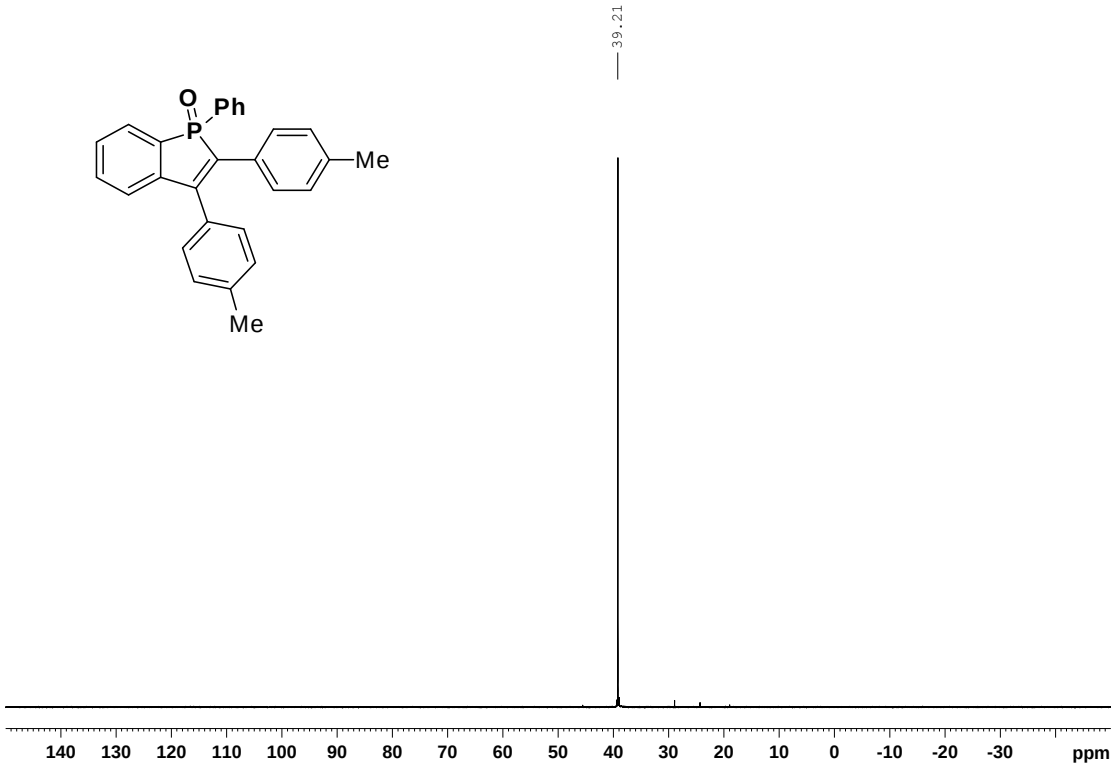
^{31}P NMR spectrum of compound **3a**.



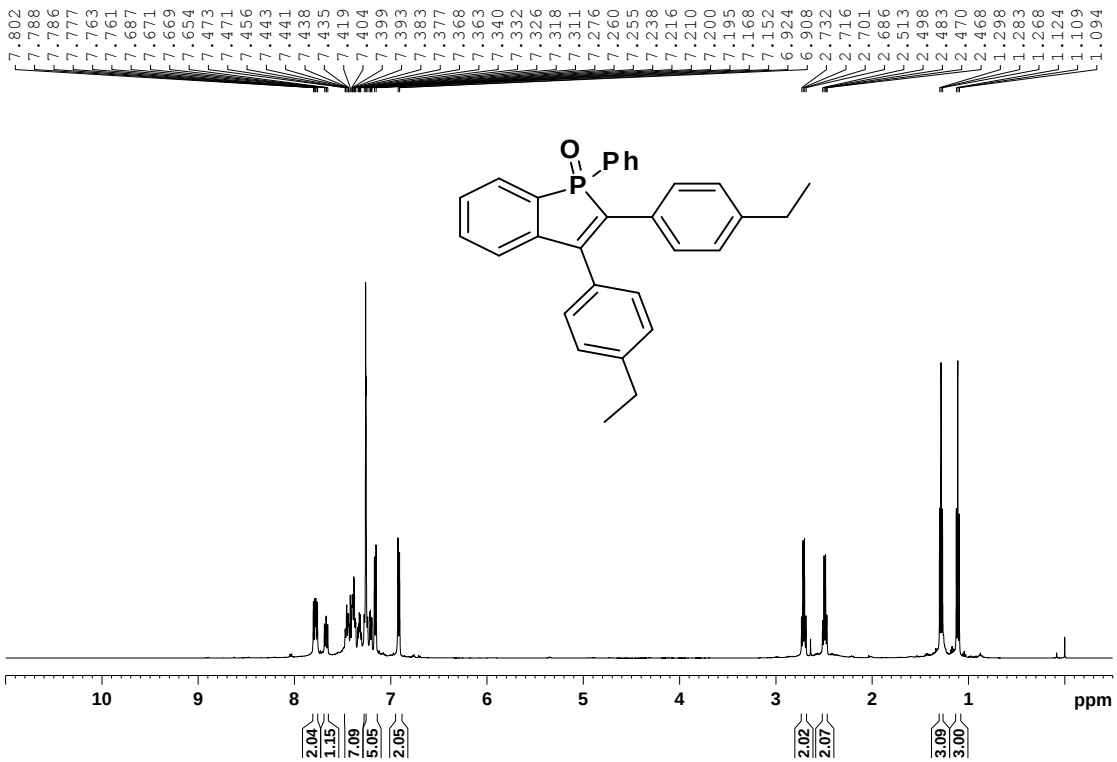
^{1}H NMR spectrum of compound **3b**.



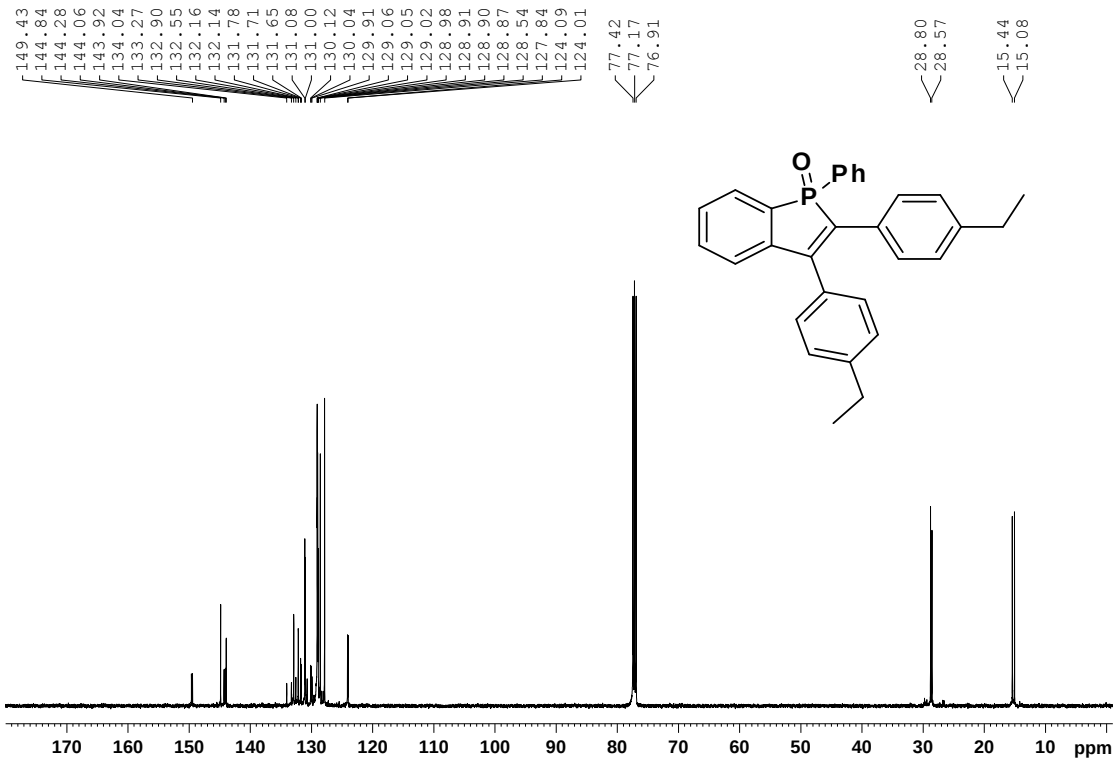
^{13}C NMR spectrum of compound **3b**.



^{31}P NMR spectrum of compound **3b**.

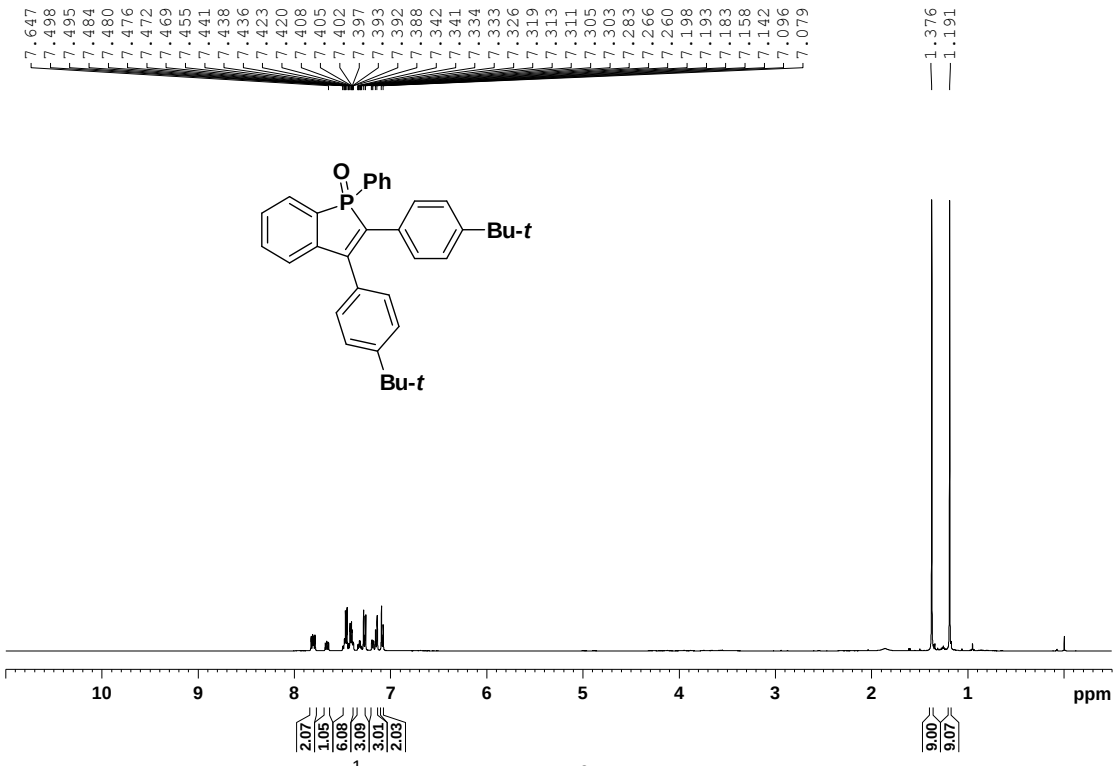


^{1}H NMR spectrum of compound **3c**.

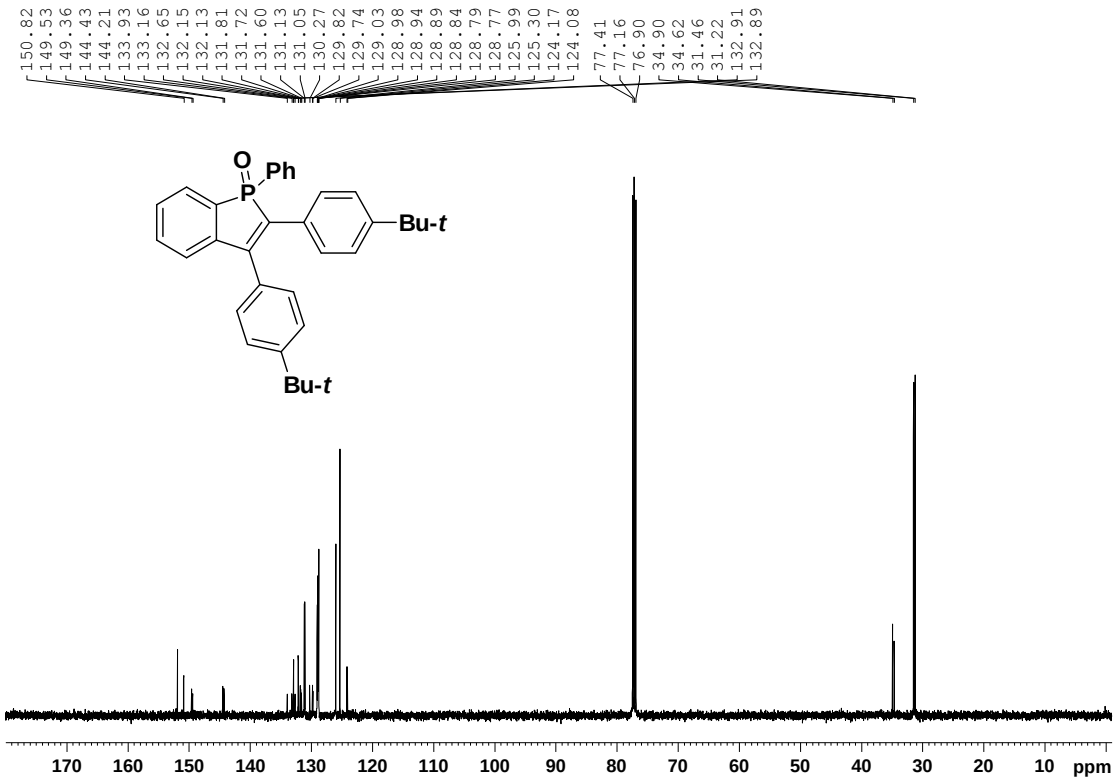


^{13}C NMR spectrum of compound **3c.**

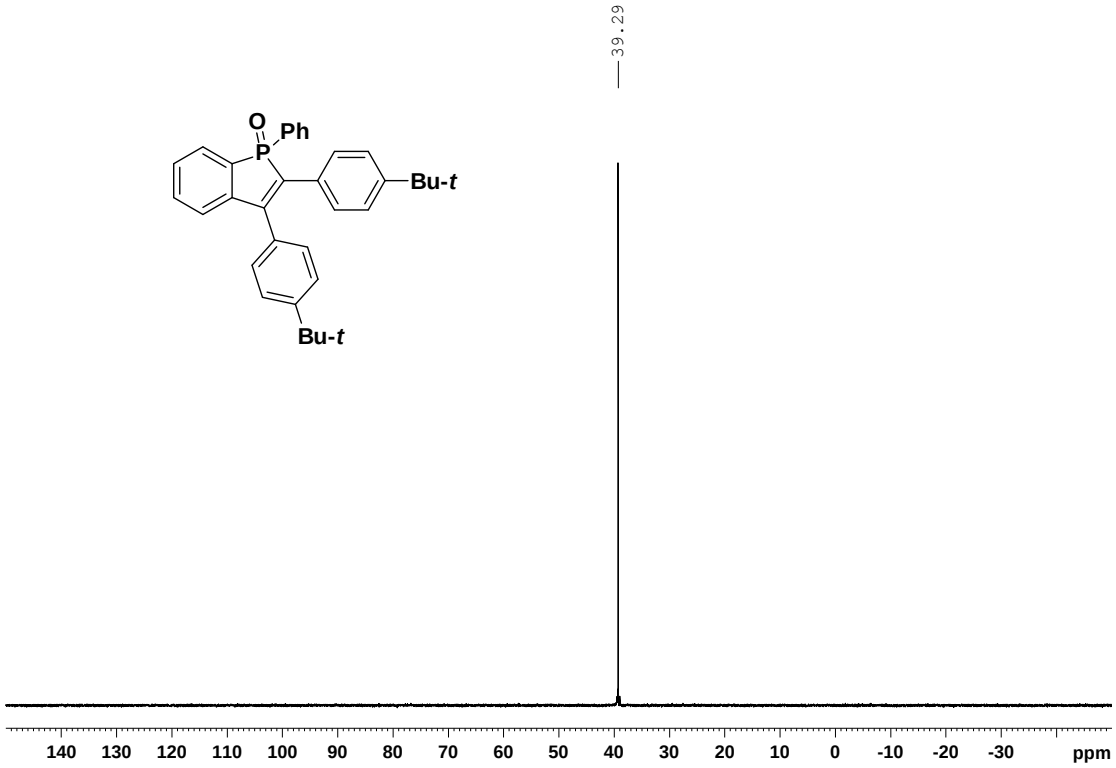
^{31}P NMR spectrum of compound 3c.



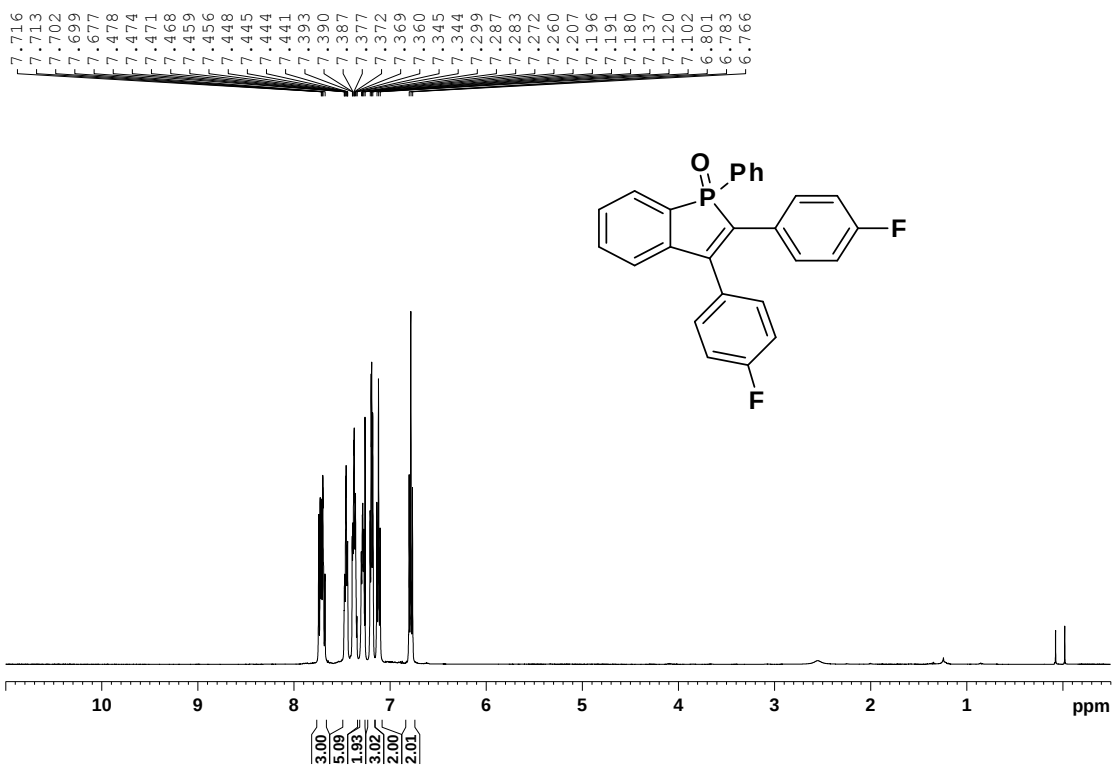
^{1}H NMR spectrum of compound **3d**.



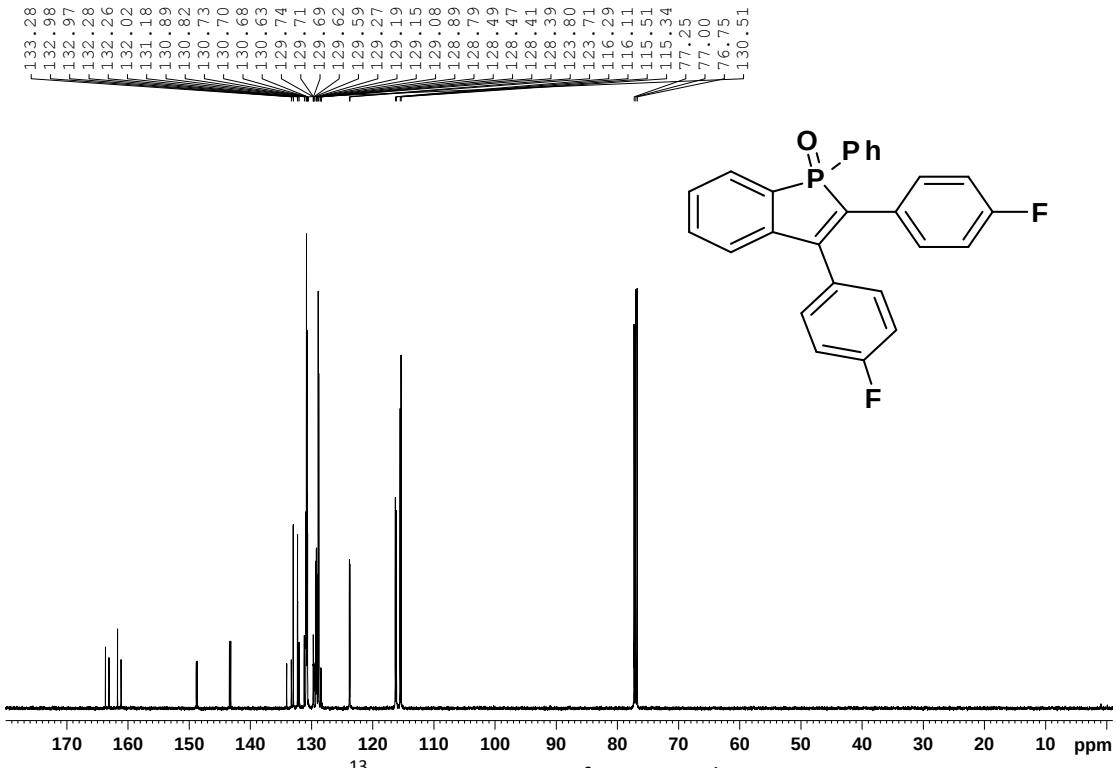
^{13}C NMR spectrum of compound 3d.



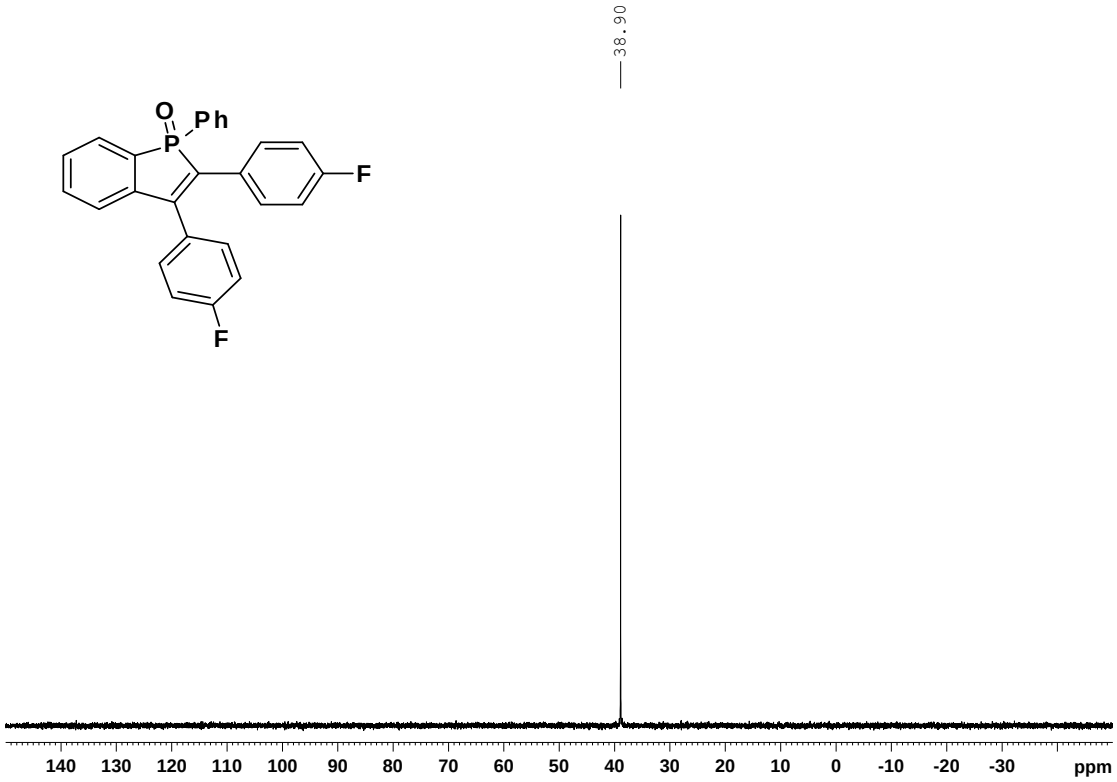
^{31}P NMR spectrum of compound **3d**.



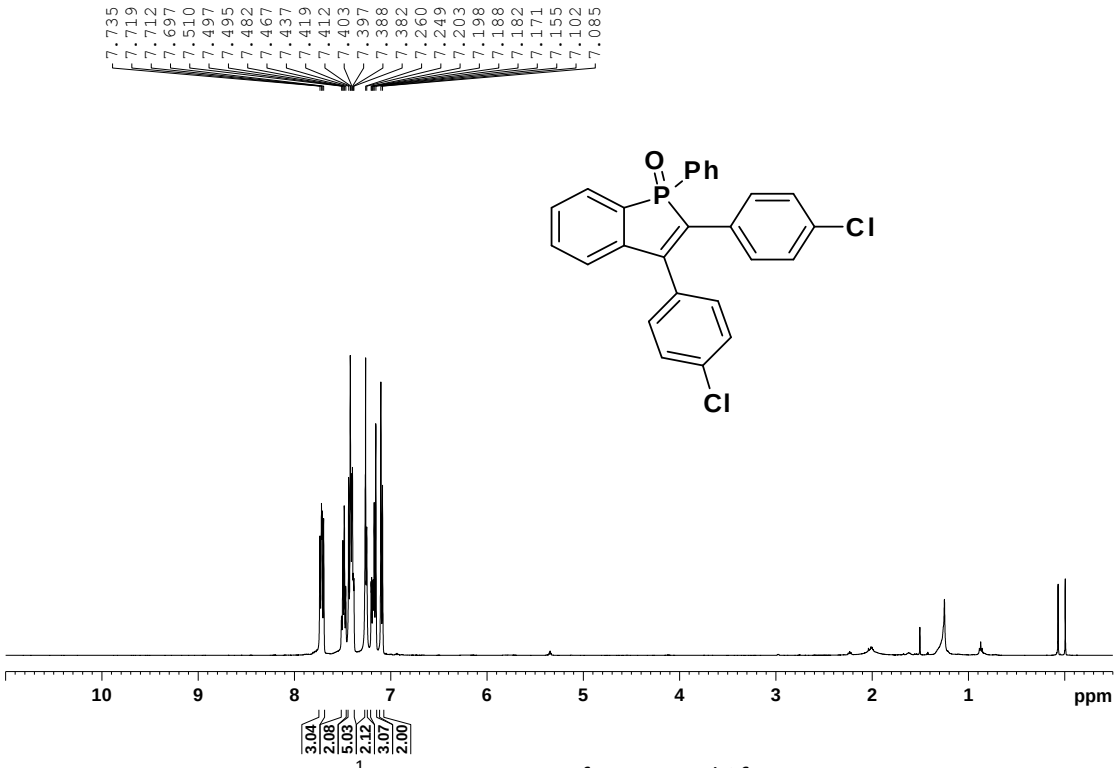
^{1}H NMR spectrum of compound 3e.



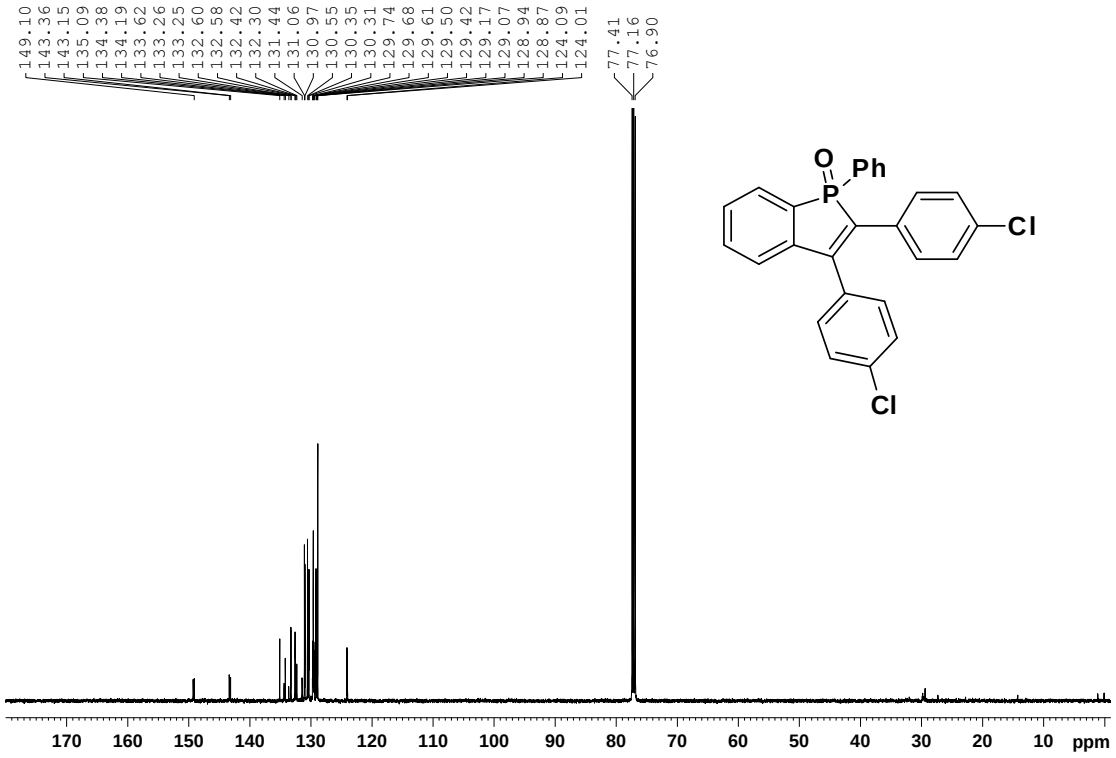
^{13}C NMR spectrum of compound **3e**.



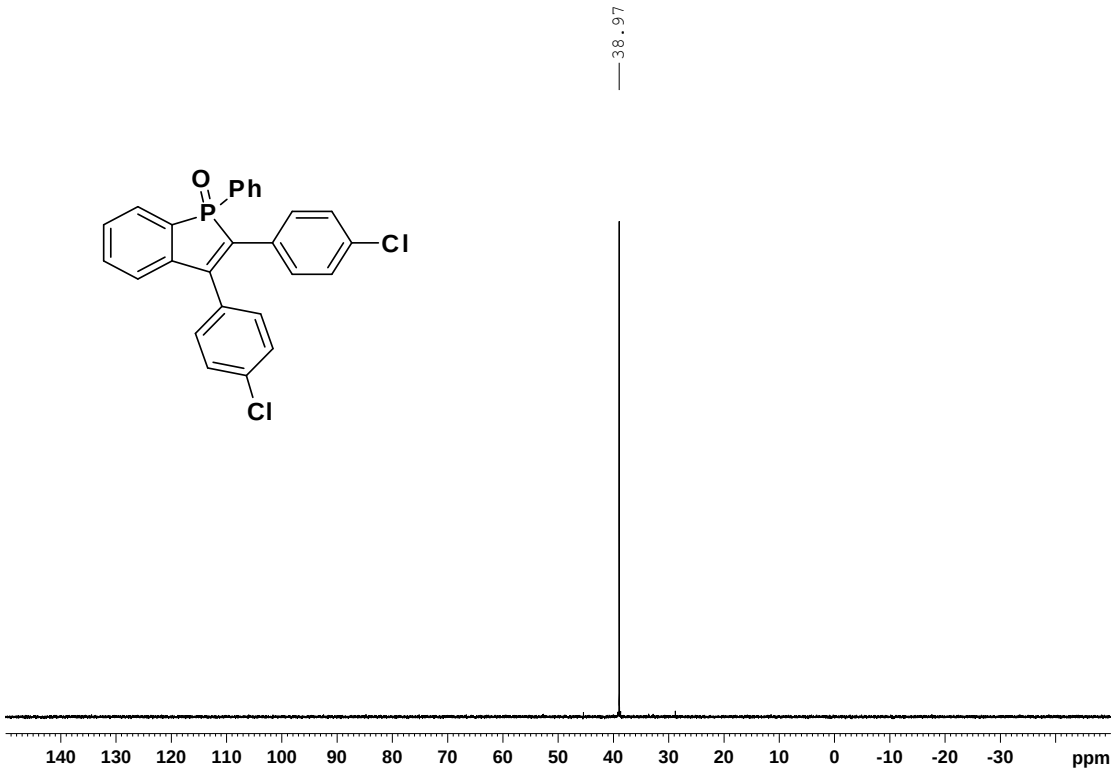
^{31}P NMR spectrum of compound **3e**.



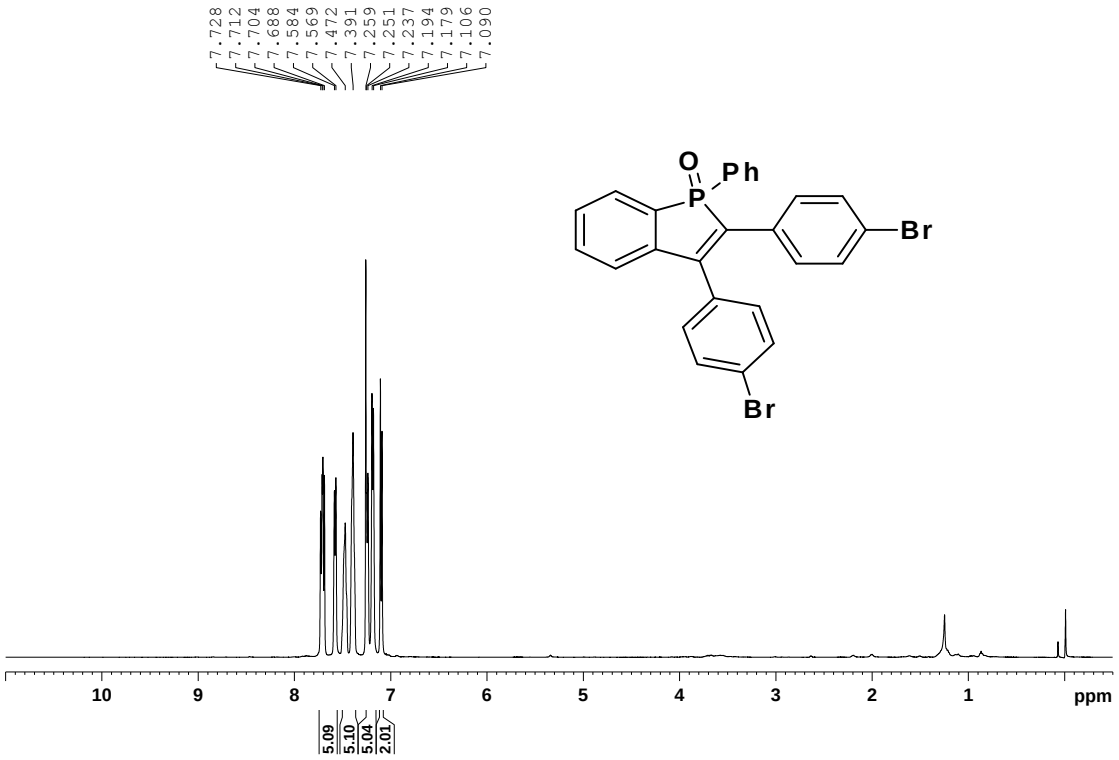
^{1}H NMR spectrum of compound **3f**.



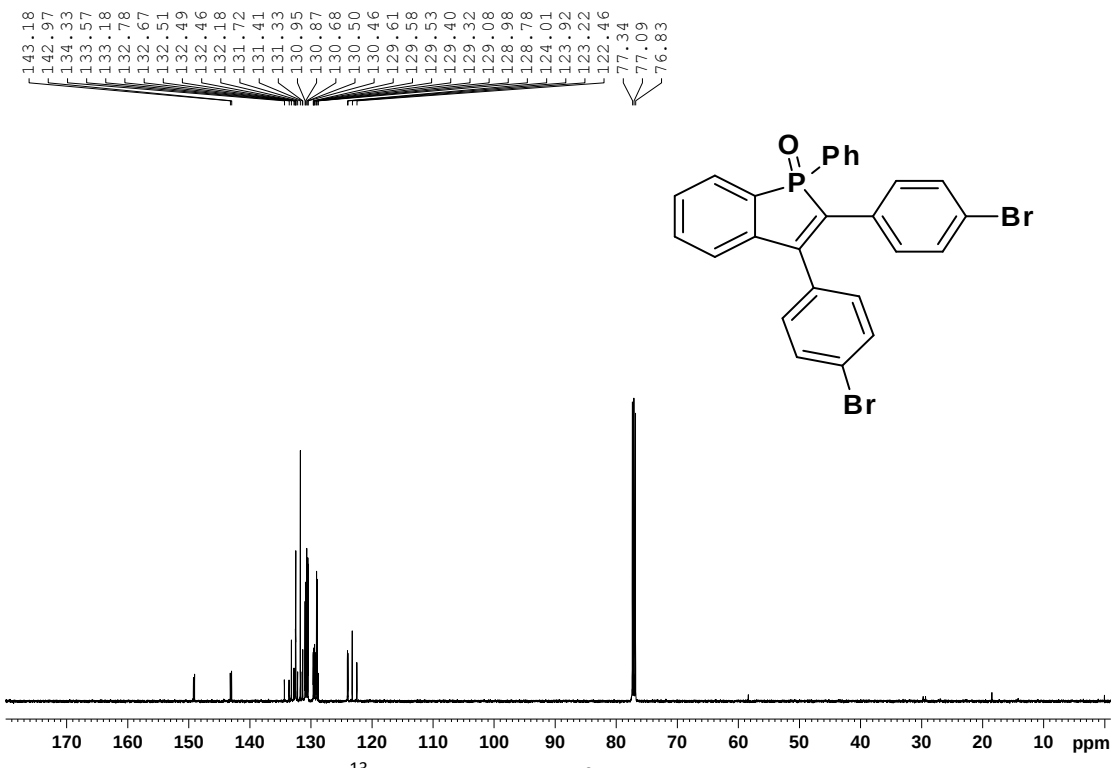
^{13}C NMR spectrum of compound **3f**.



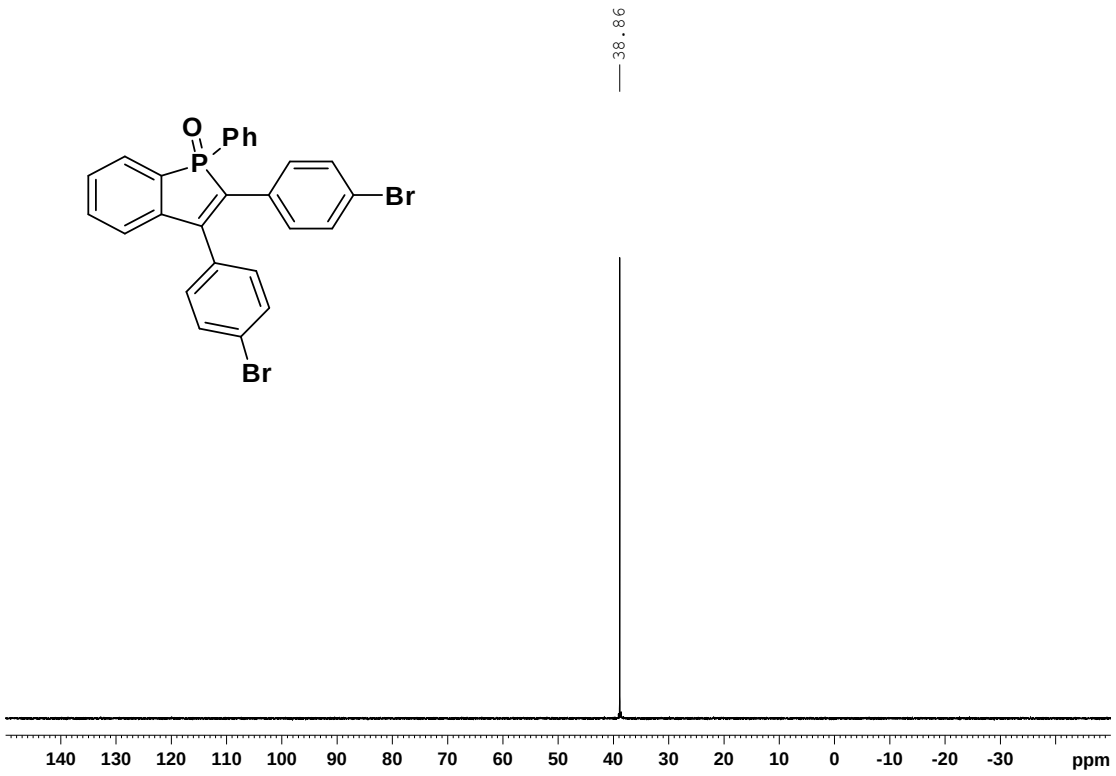
^{31}P NMR spectrum of compound **3f**.



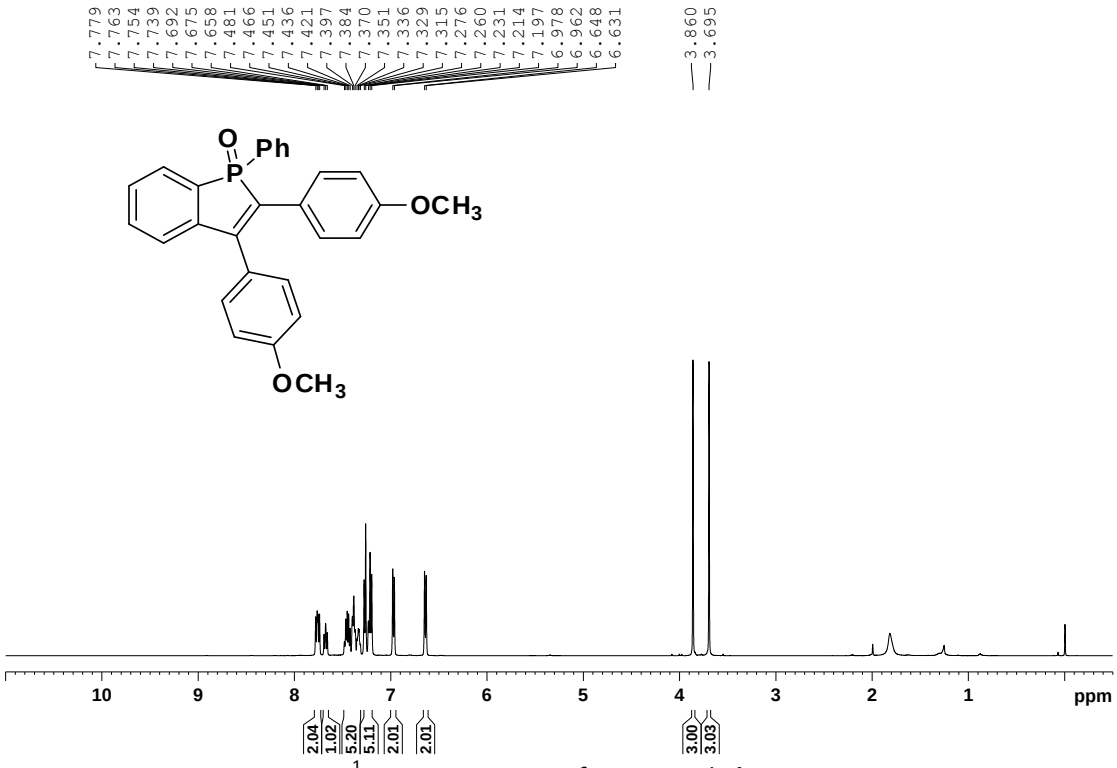
^{1}H NMR spectrum of compound **3g**.



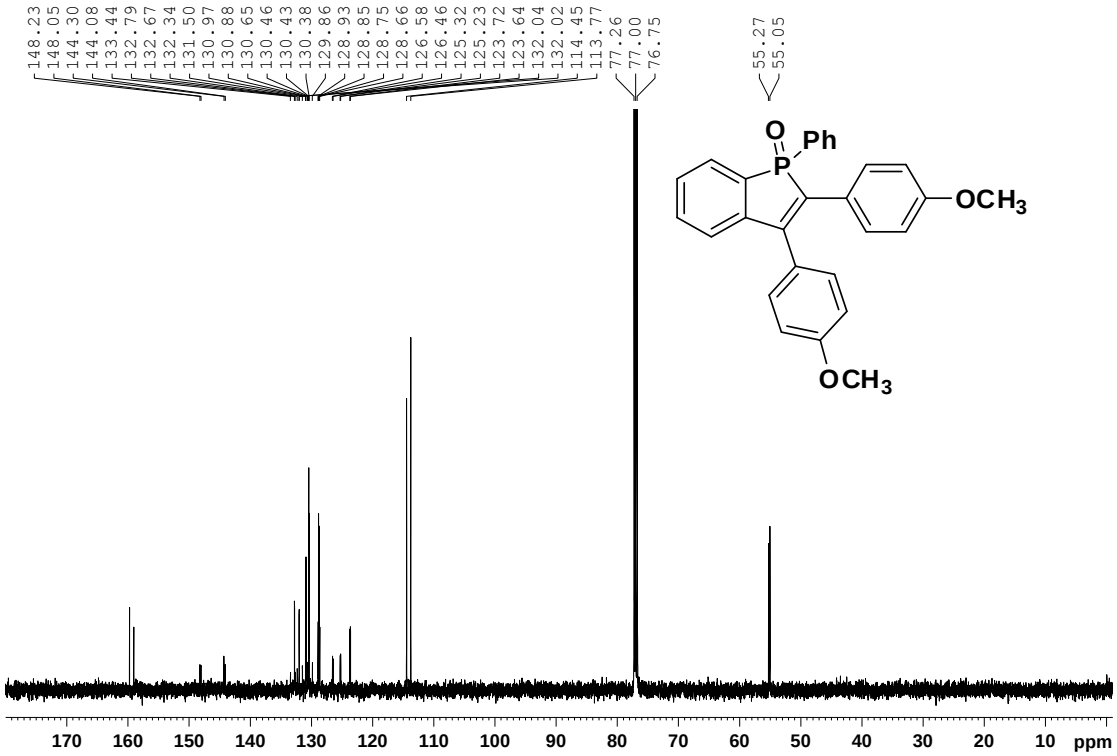
^{13}C NMR spectrum of compound **3g**.



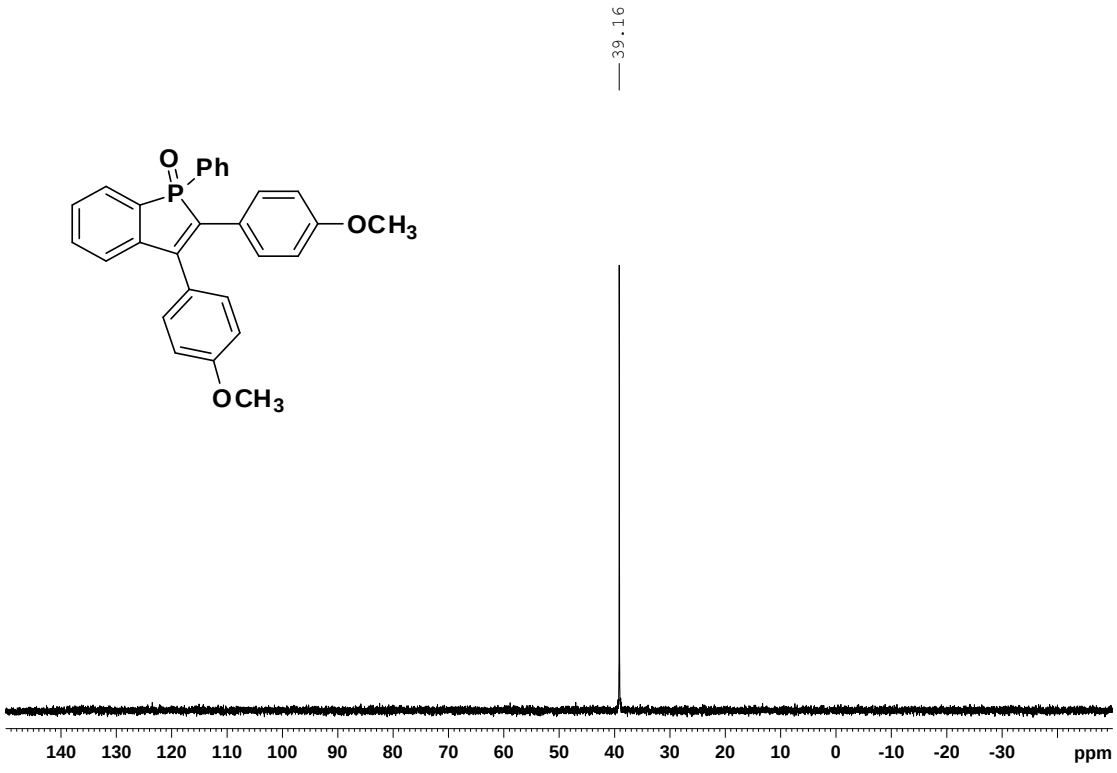
^{31}P NMR spectrum of compound **3g**.



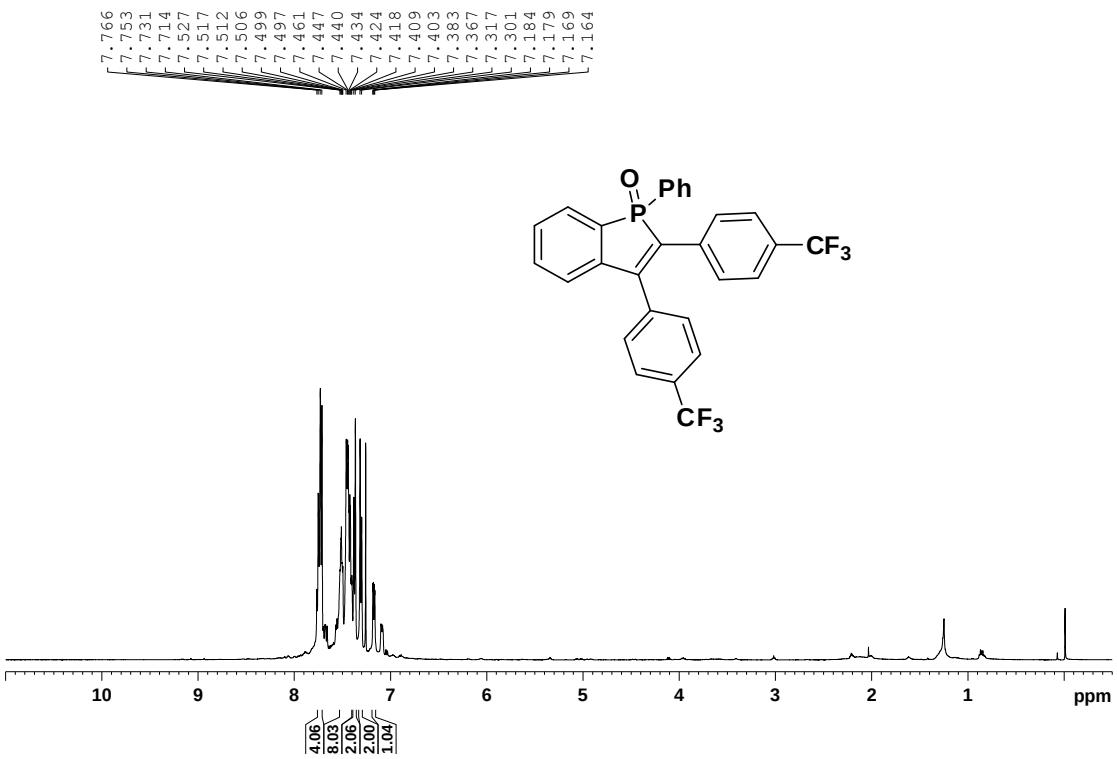
^{1}H NMR spectrum of compound **3h**.



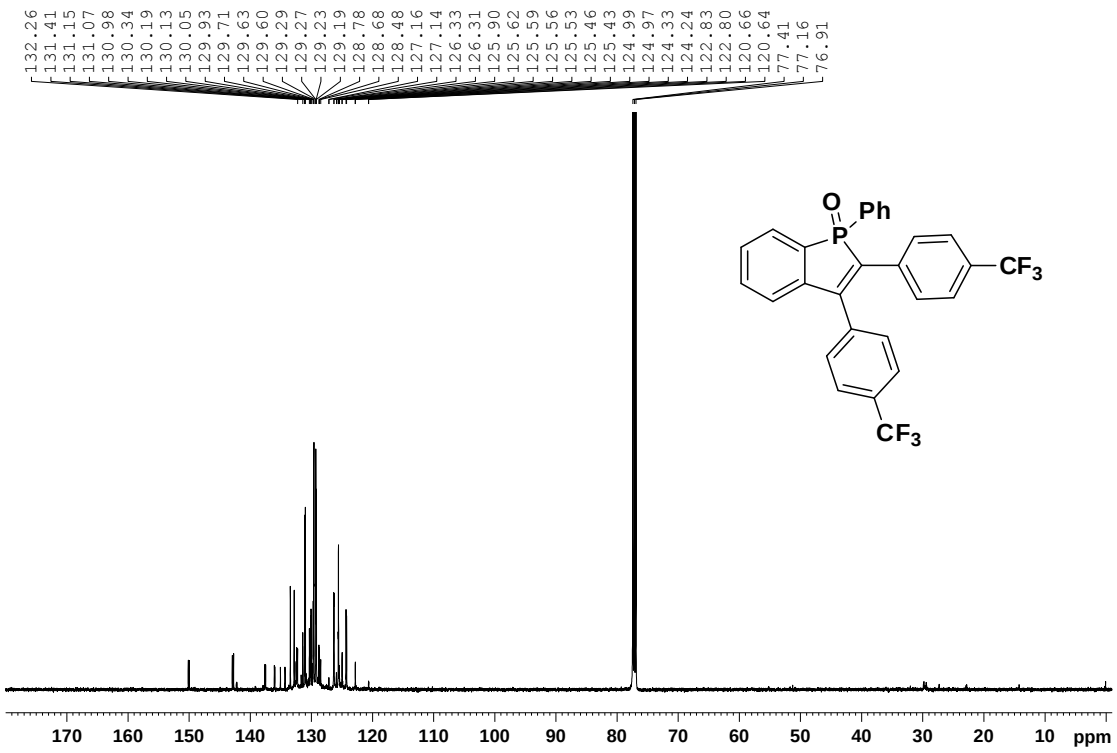
^{13}C NMR spectrum of compound **3h**.



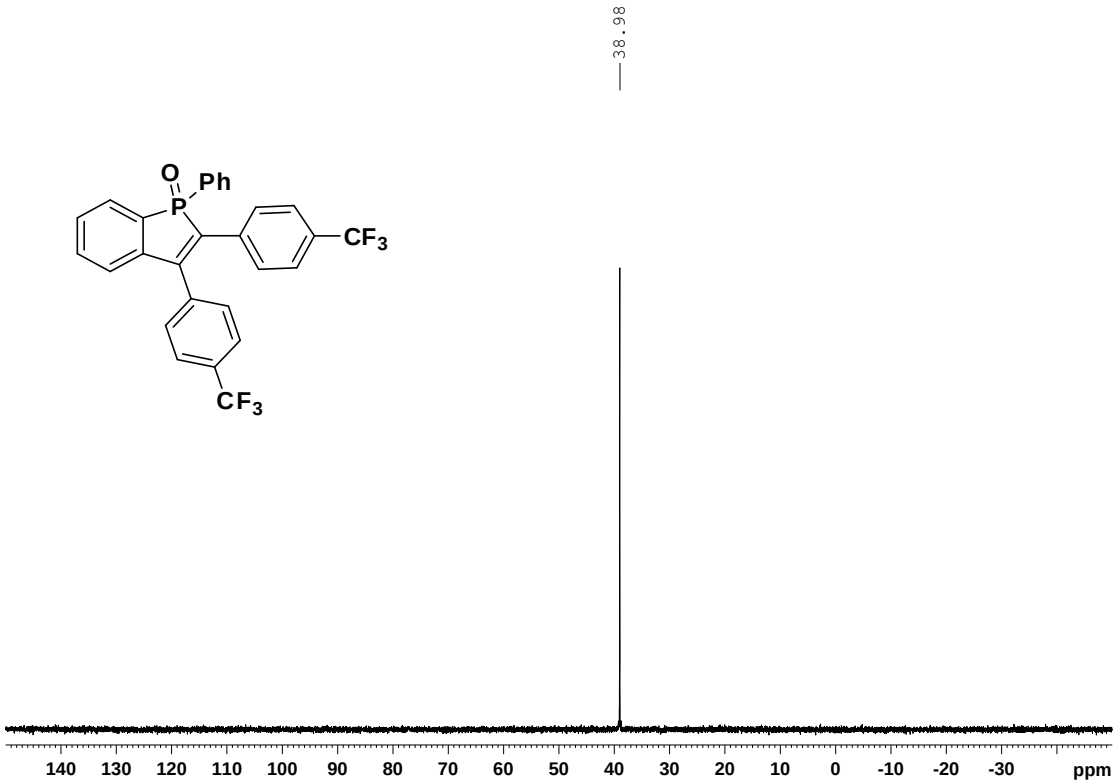
^{31}P NMR spectrum of compound **3h**.



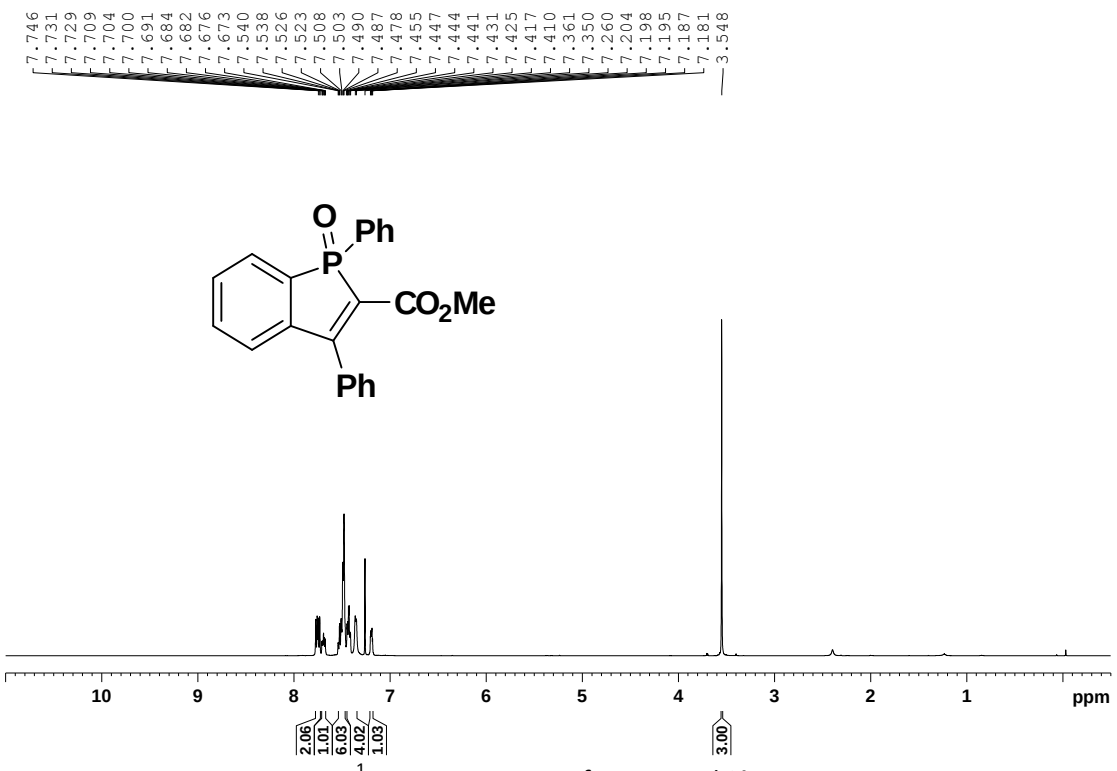
^{1}H NMR spectrum of compound **3i**.



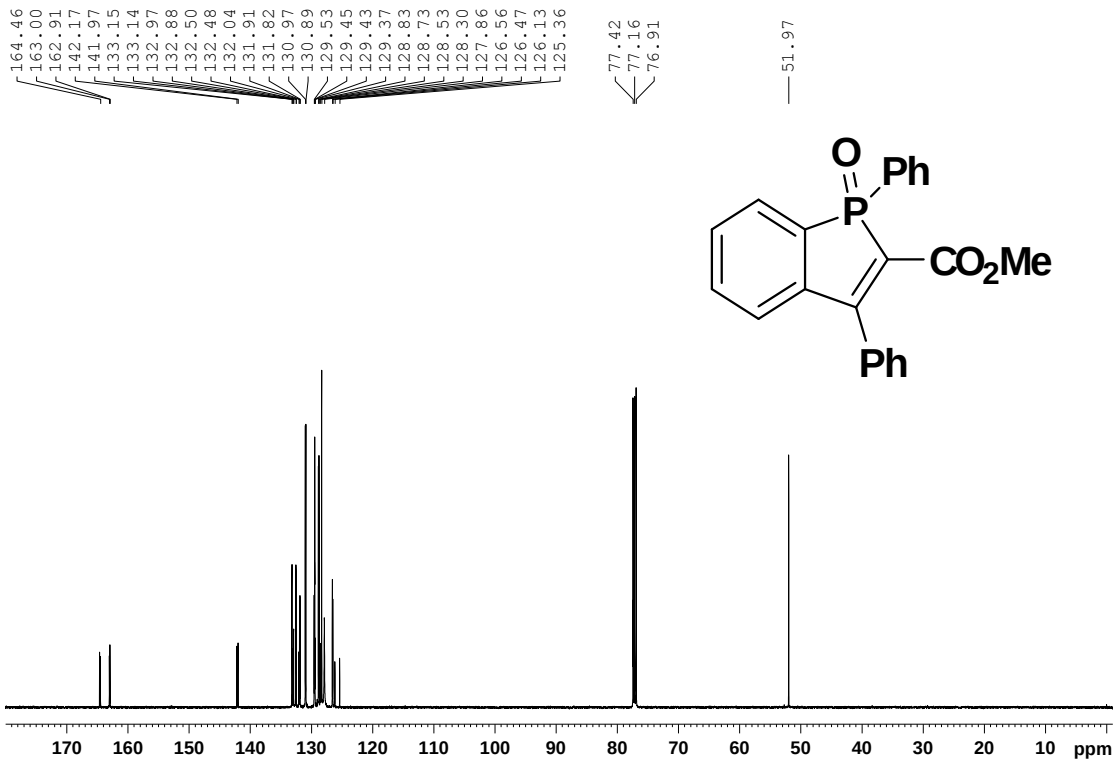
^{13}C NMR spectrum of compound **3i**.



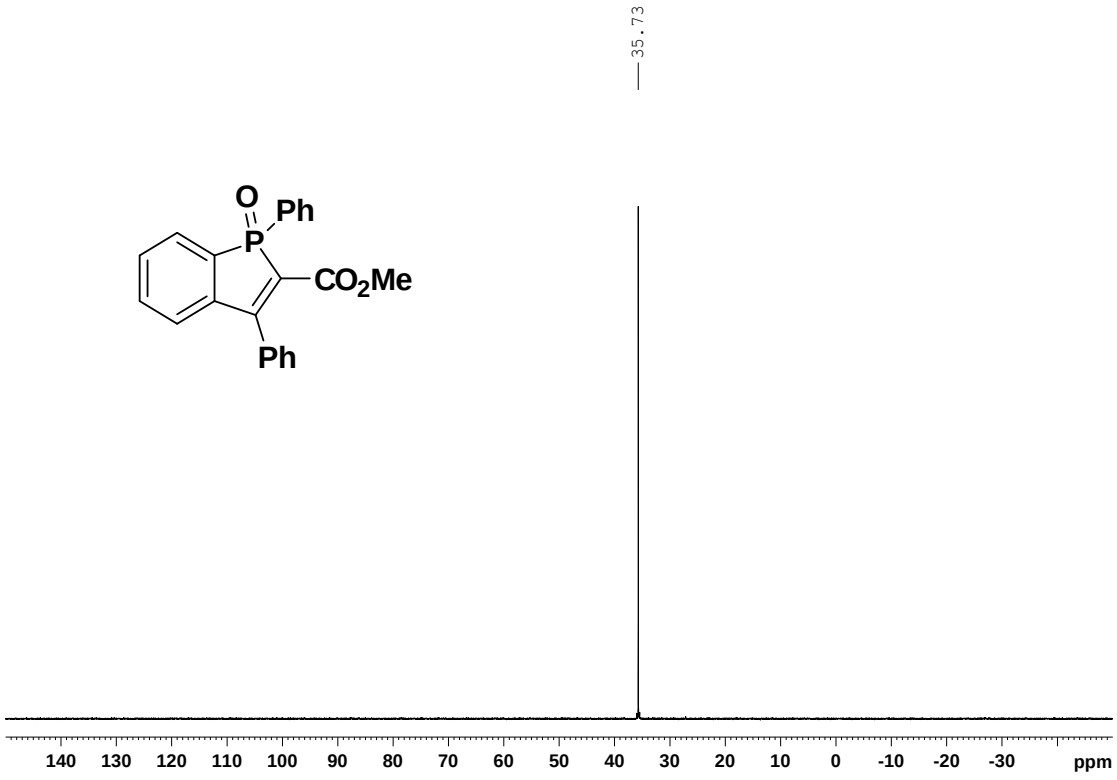
^{31}P NMR spectrum of compound **3i**.



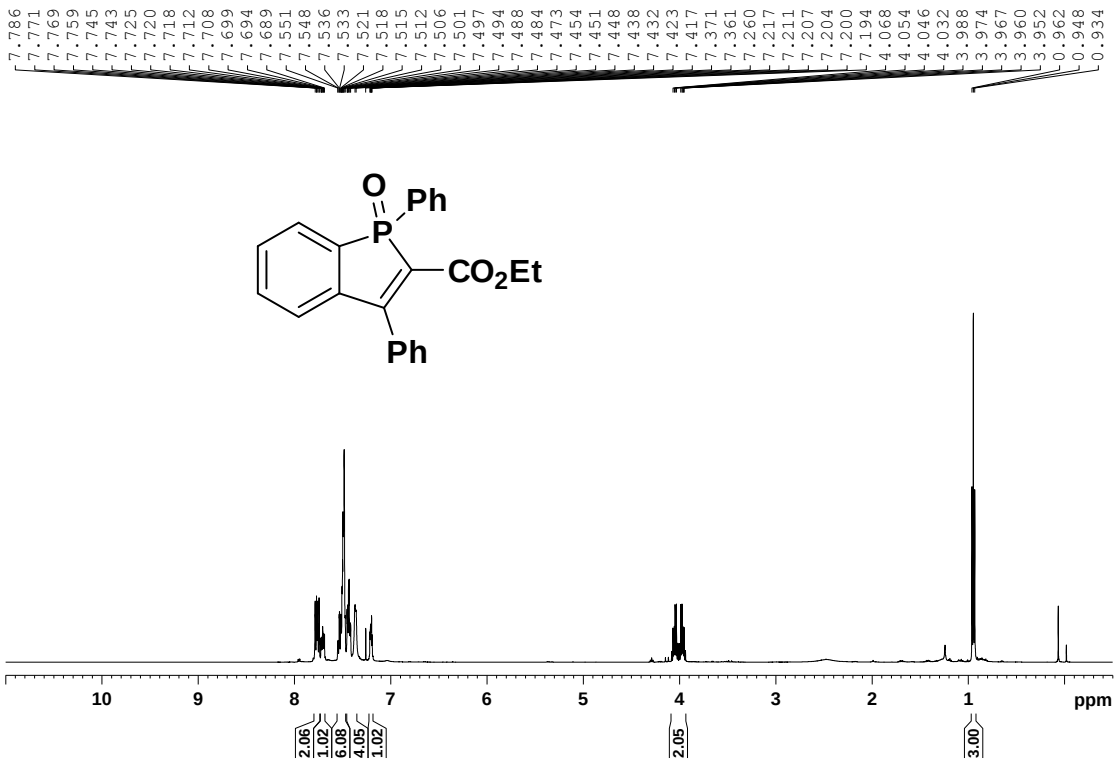
^{1}H NMR spectrum of compound **3j**.



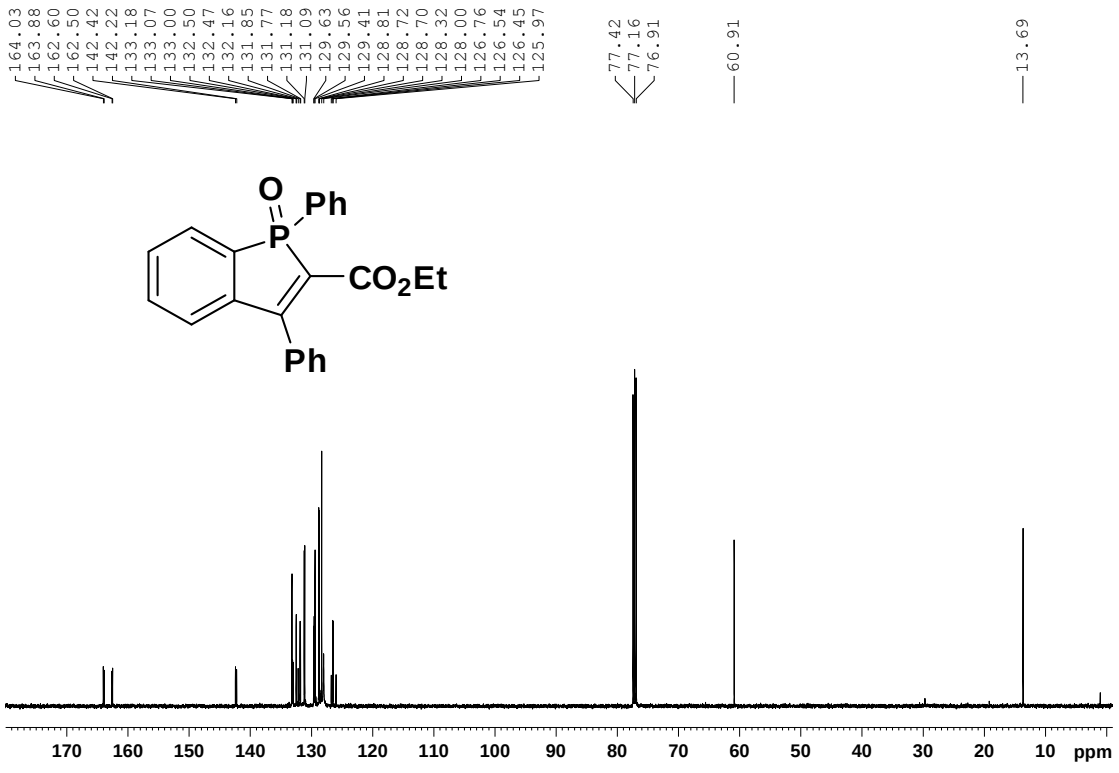
^{13}C NMR spectrum of compound **3j**.



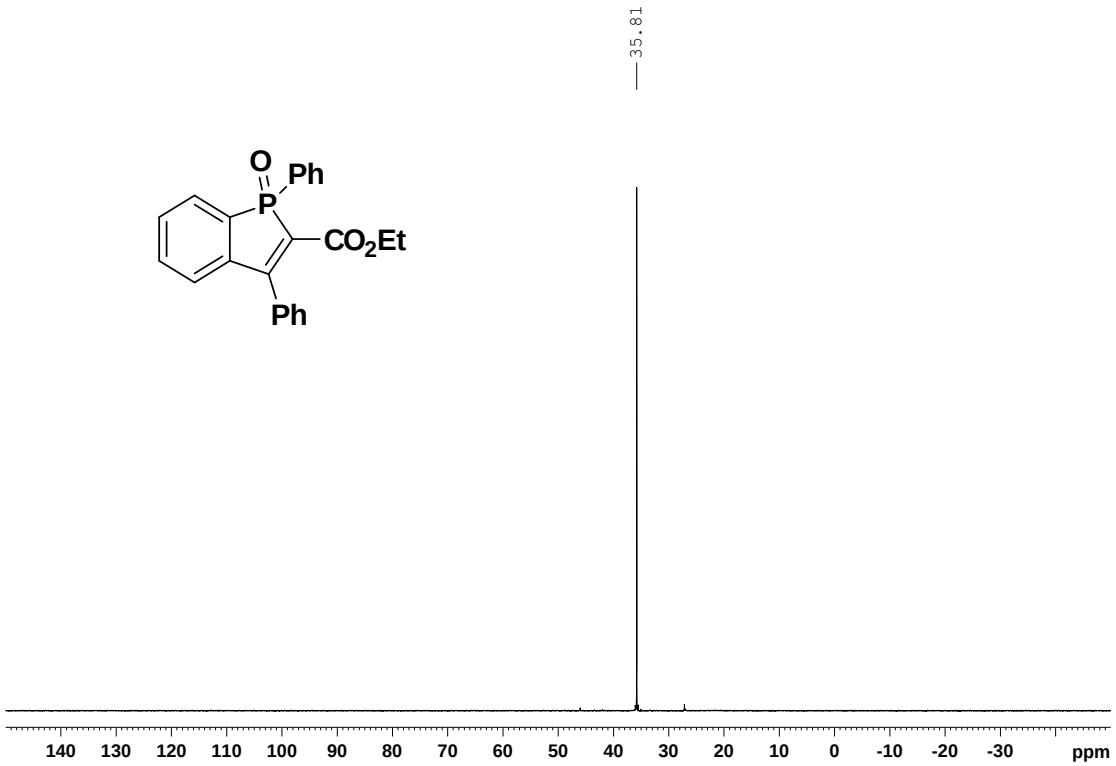
^{31}P NMR spectrum of compound **3j**.



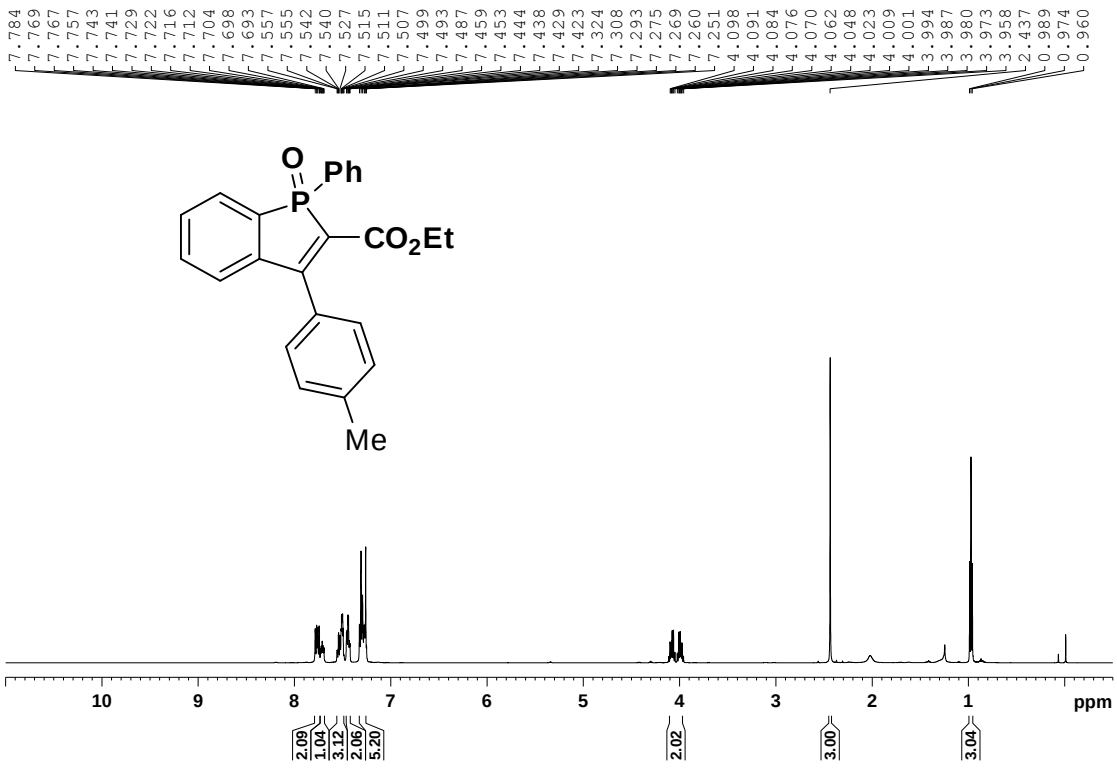
^{1}H NMR spectrum of compound **3k**.



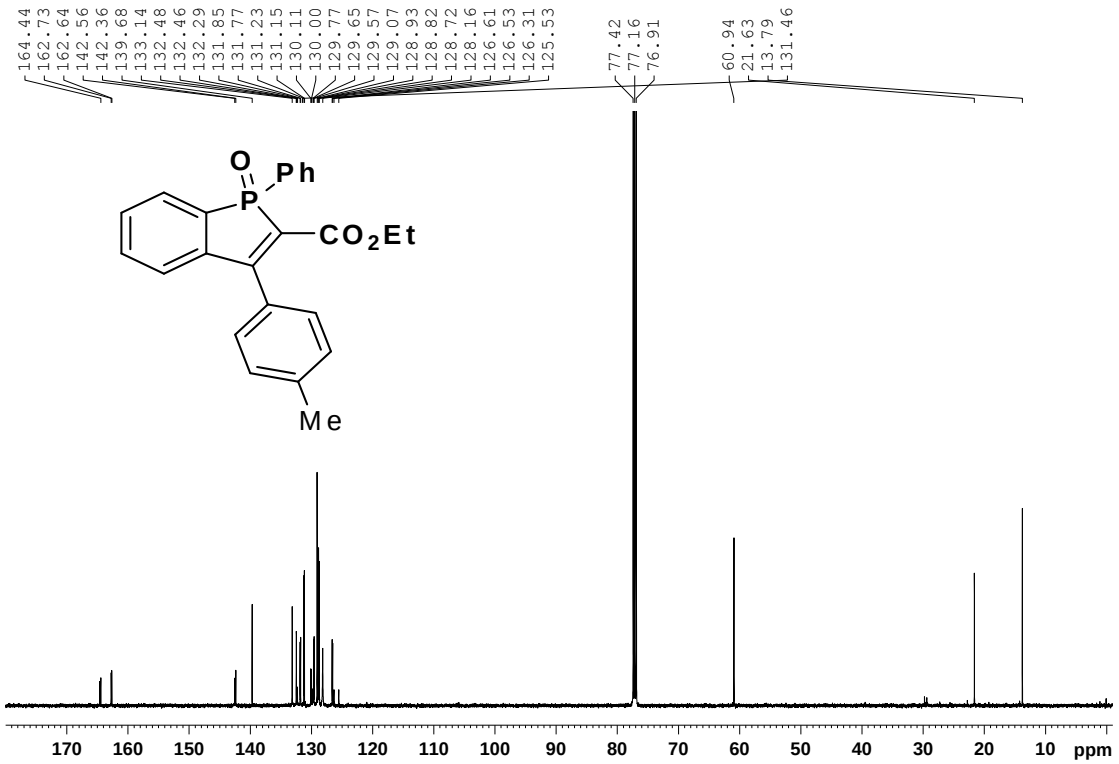
^{13}C NMR spectrum of compound **3k**.



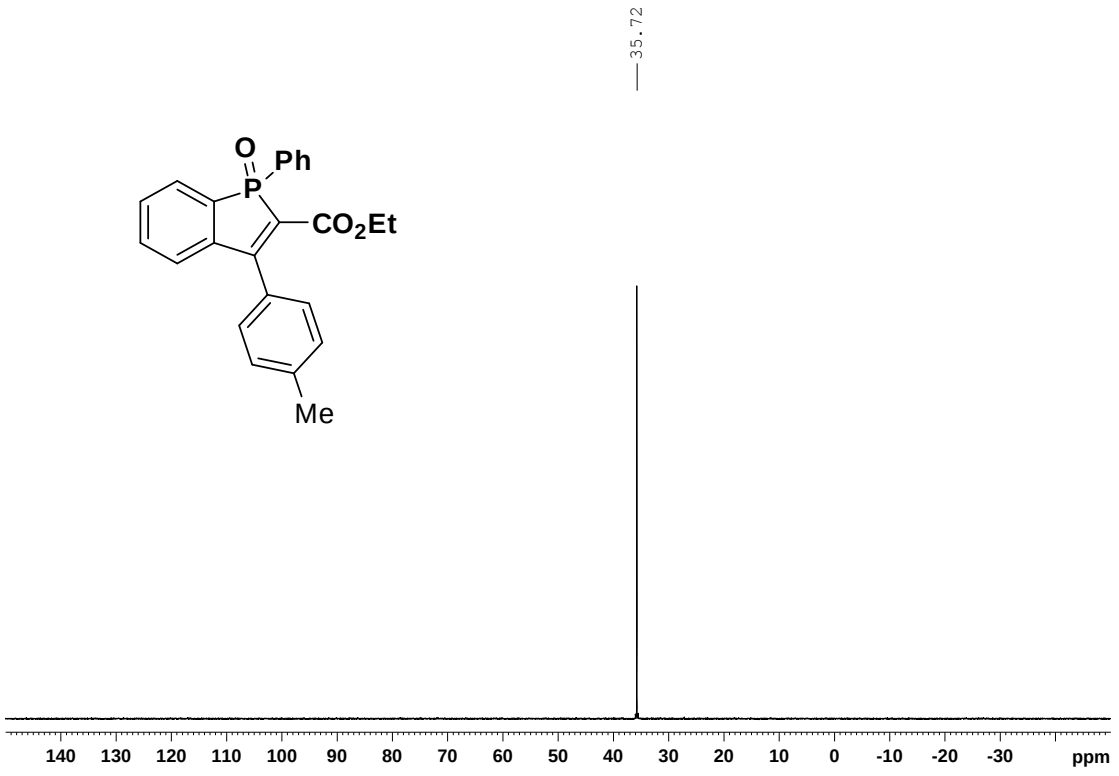
^{31}P NMR spectrum of compound **3k**.



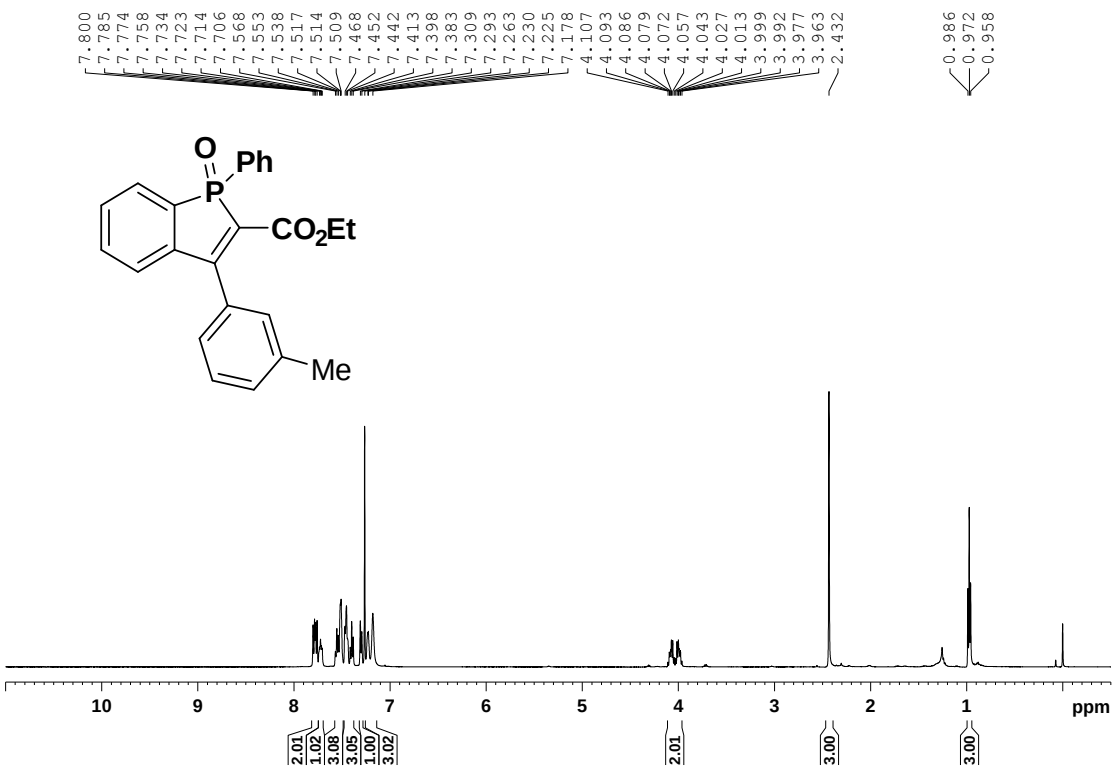
^{1}H NMR spectrum of compound **3l**.



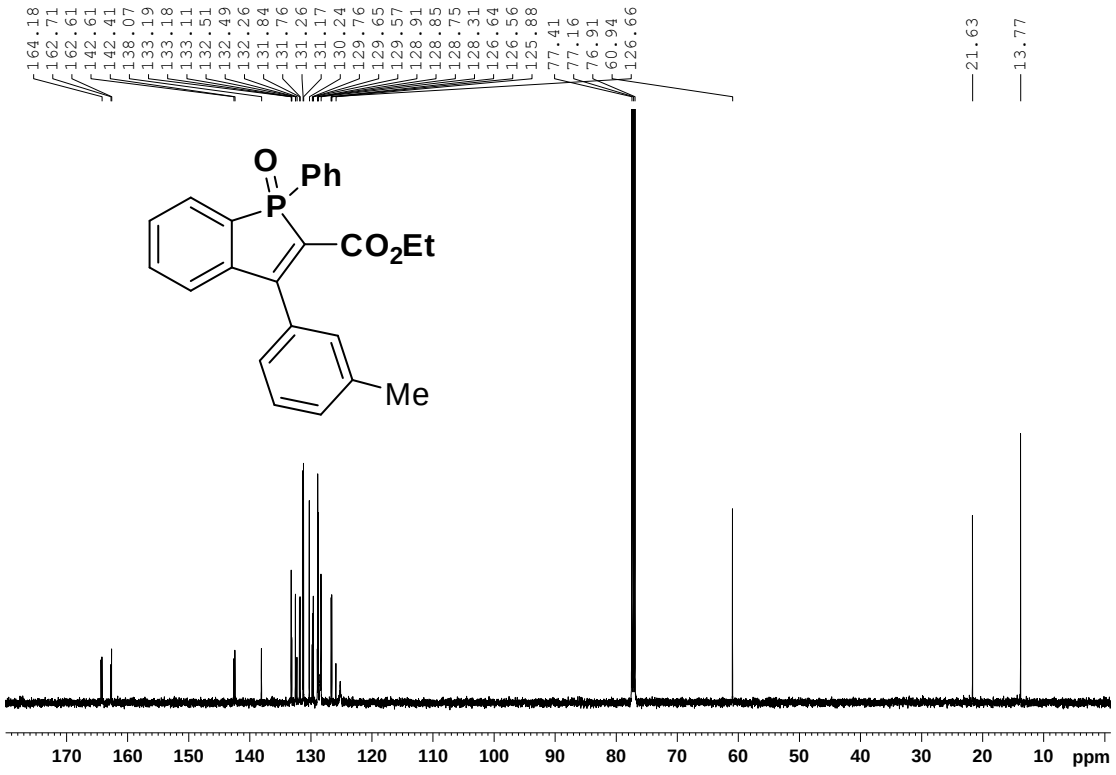
^{13}C NMR spectrum of compound **3l**.



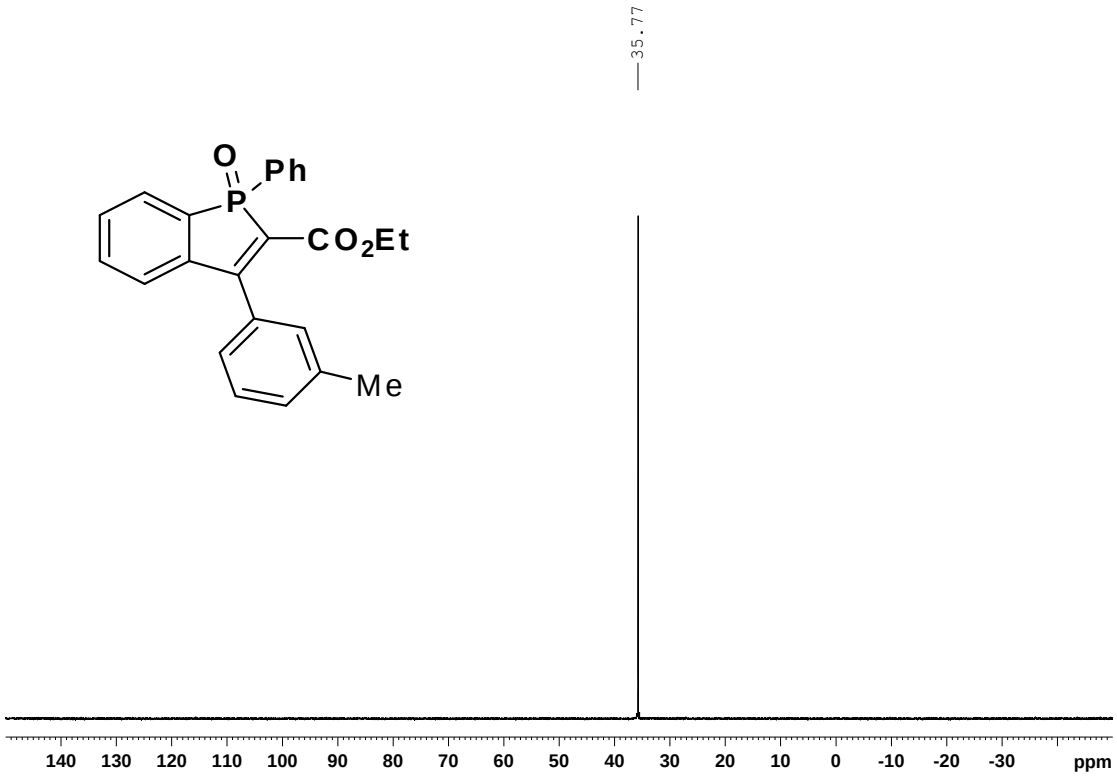
^{31}P NMR spectrum of compound **3l**.



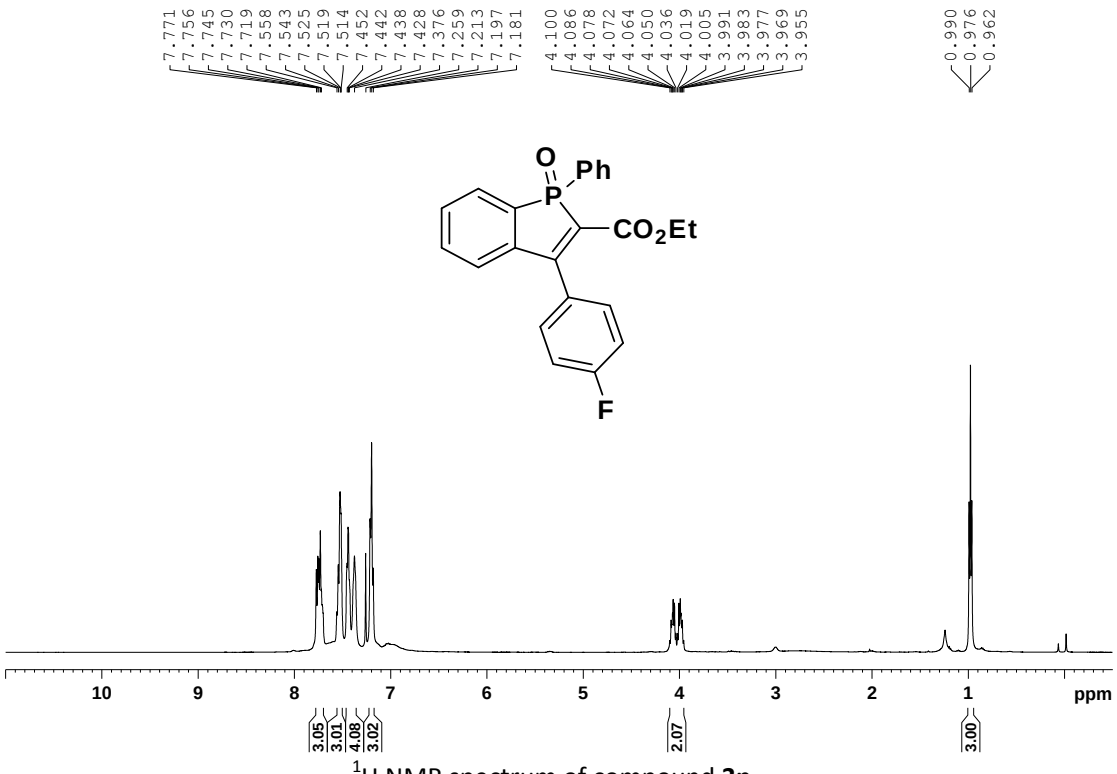
^{1}H NMR spectrum of compound **3m**.



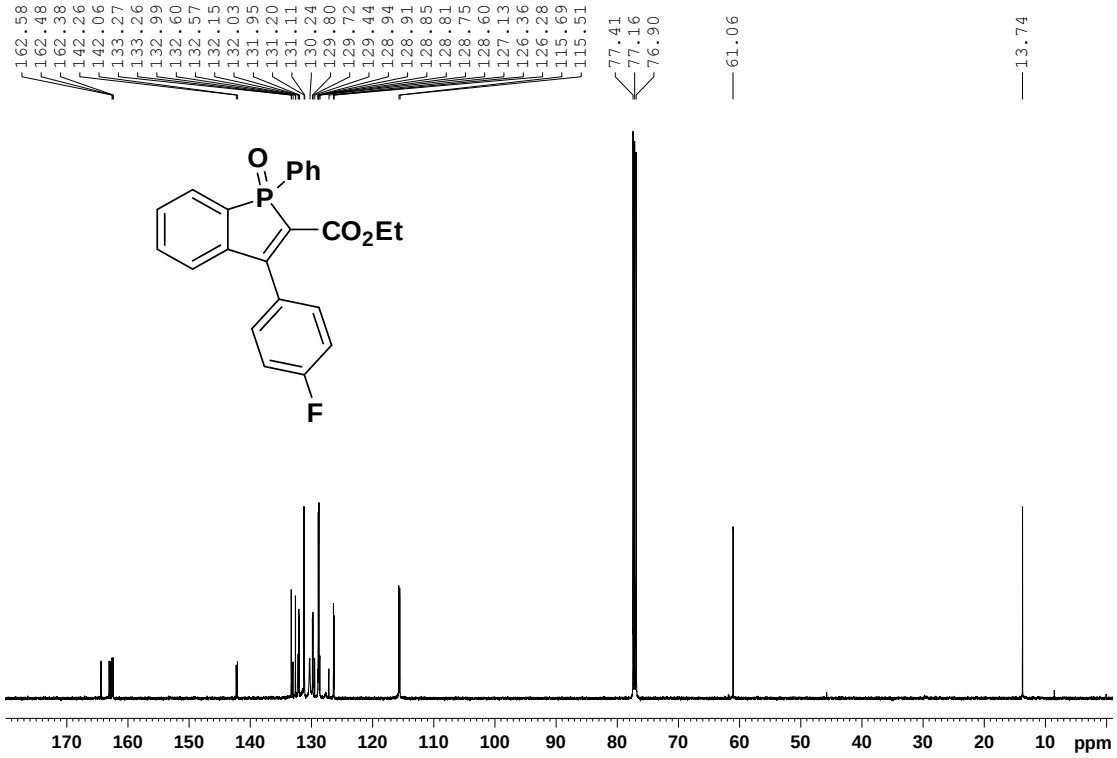
^{13}C NMR spectrum of compound **3m**.



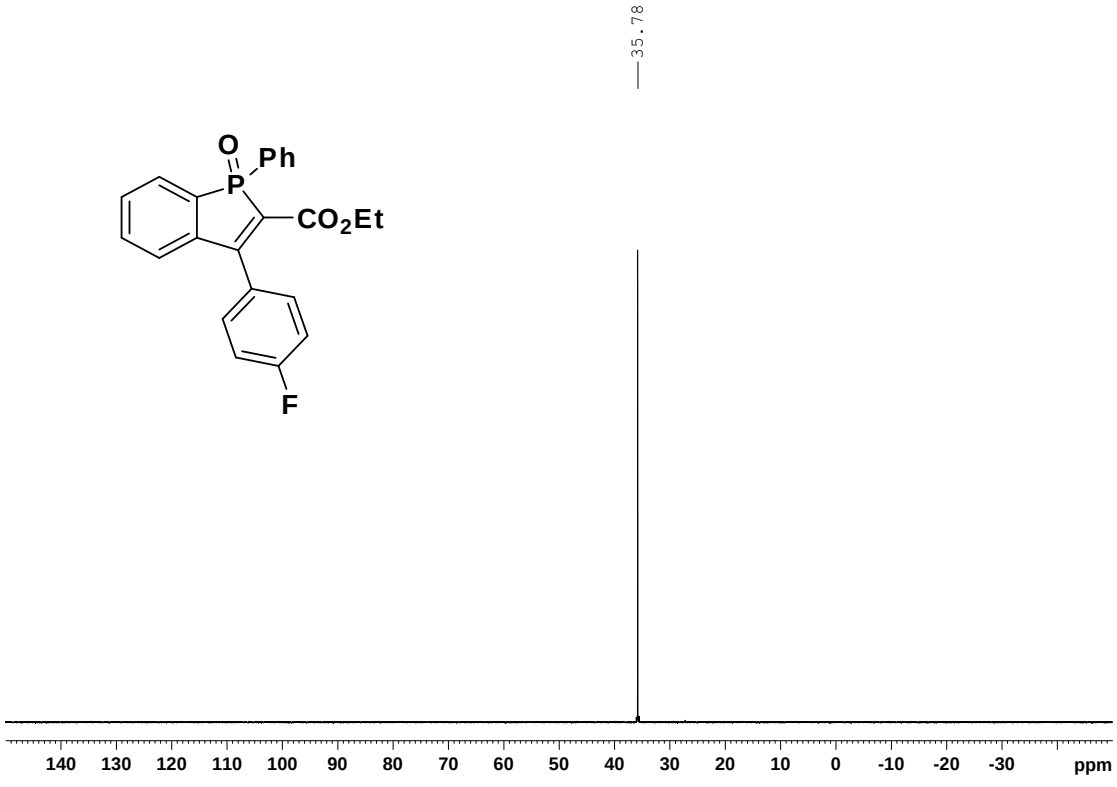
^{31}P NMR spectrum of compound **3m**.



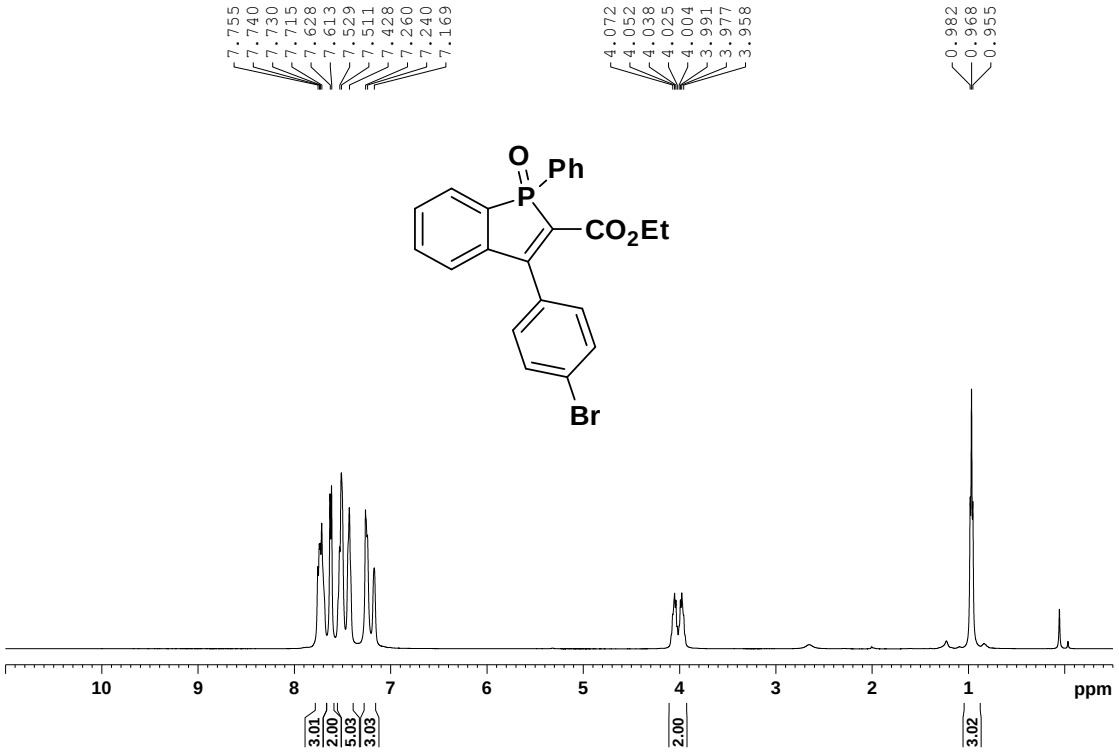
^{1}H NMR spectrum of compound **3n**.



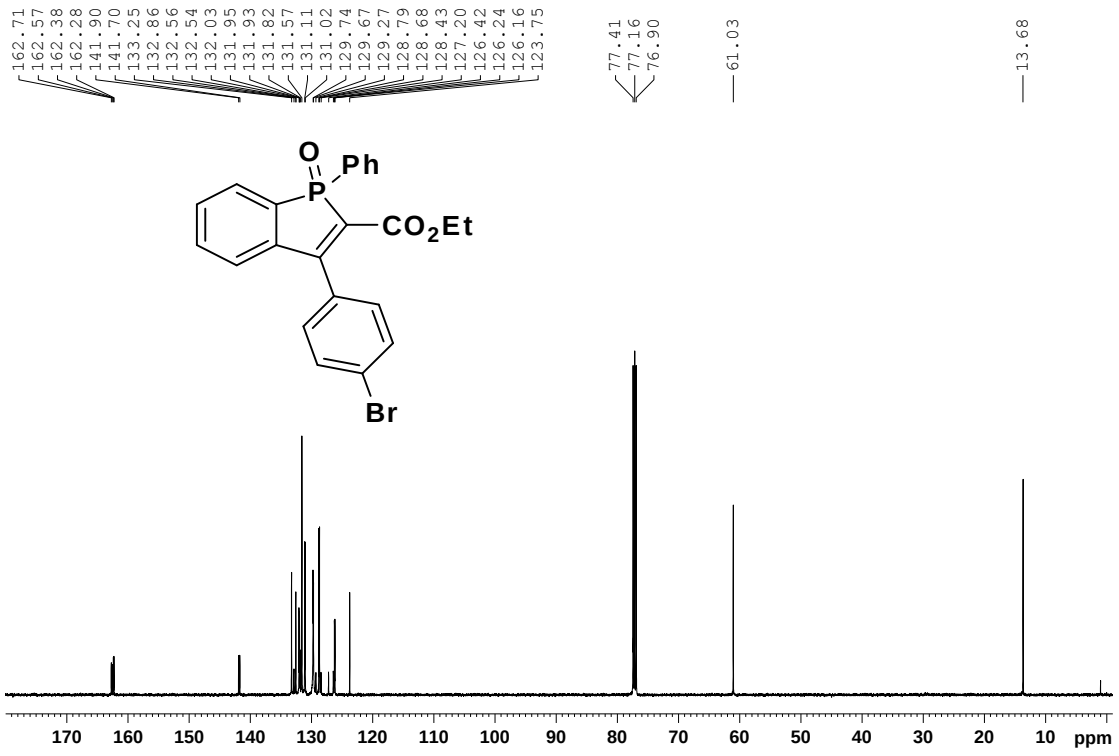
^{13}C NMR spectrum of compound **3n**.



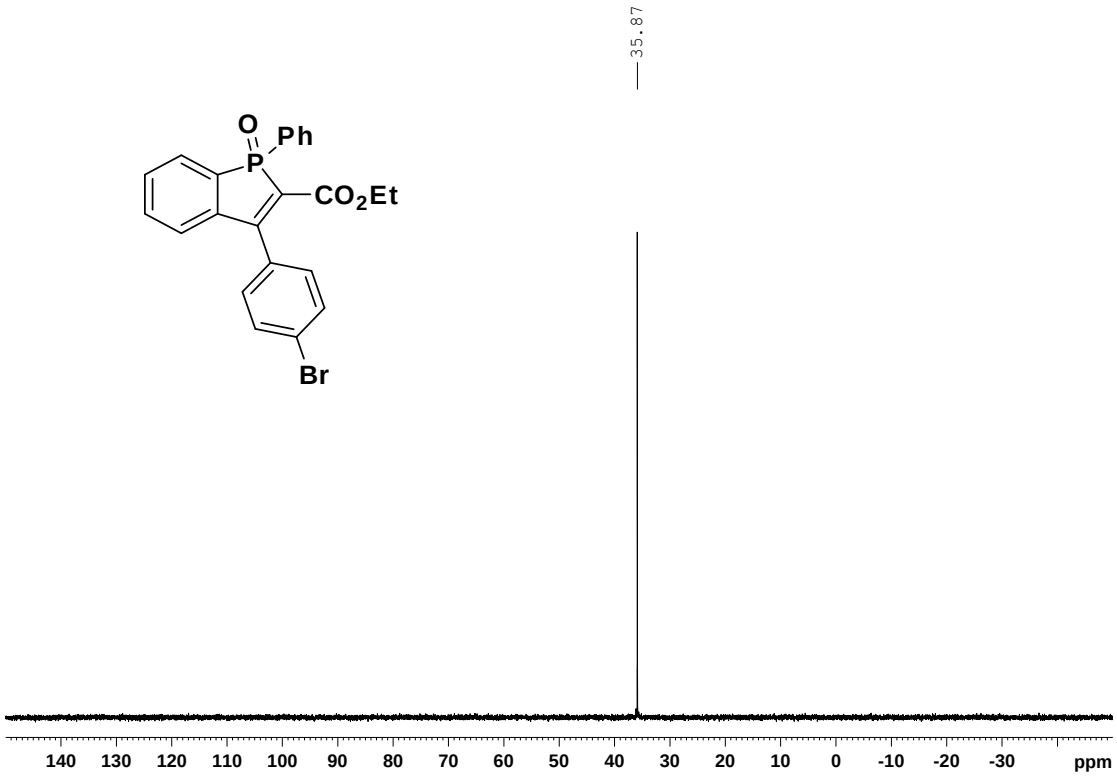
^{31}P NMR spectrum of compound **3n**.



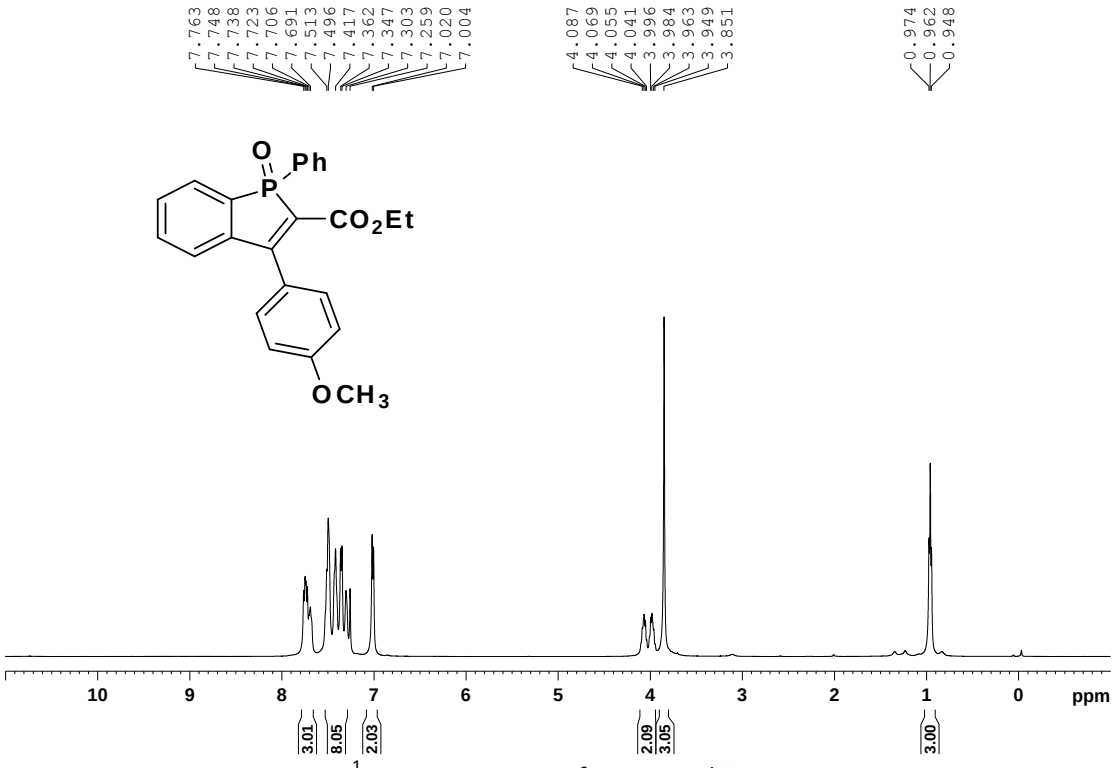
^{1}H NMR spectrum of compound **3o**.



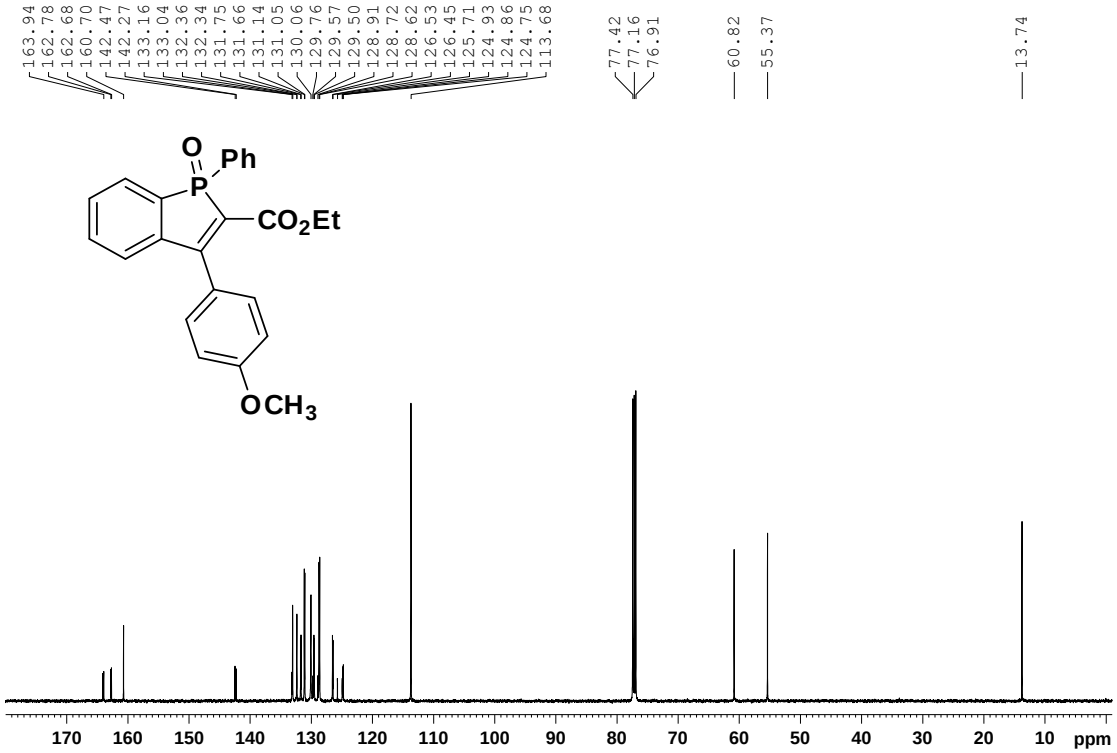
^{13}C NMR spectrum of compound **3o**.



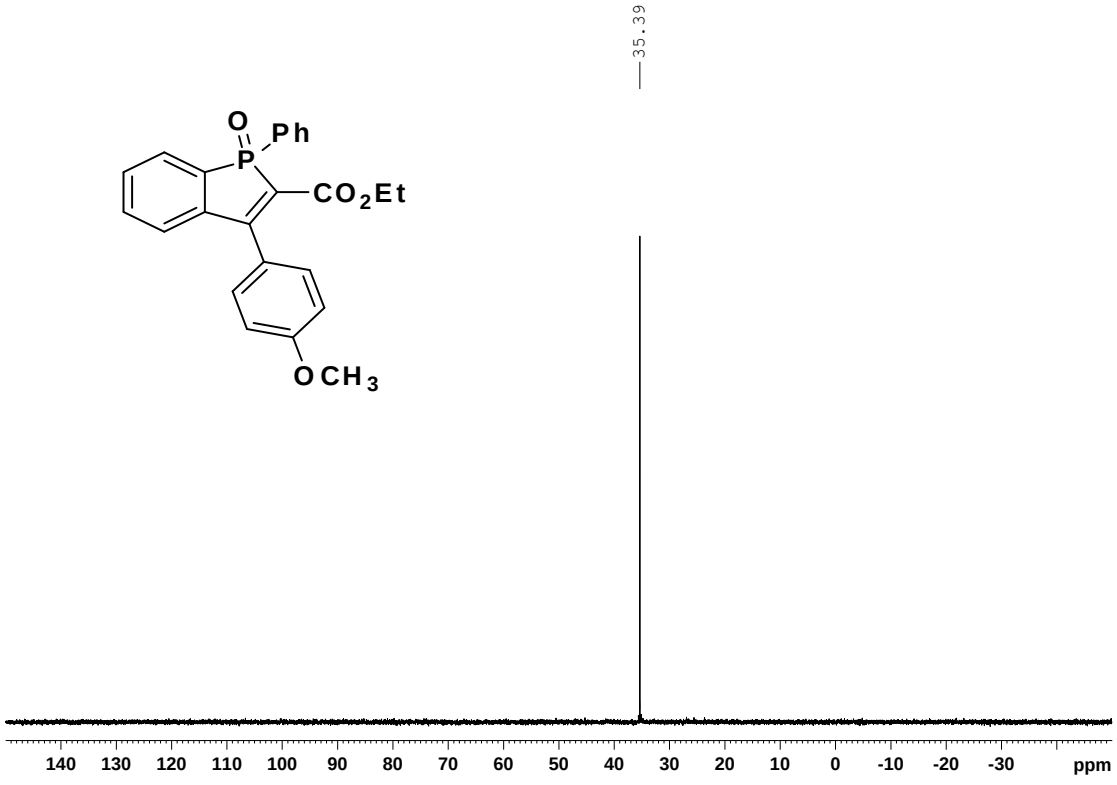
^{31}P NMR spectrum of compound **3o**.



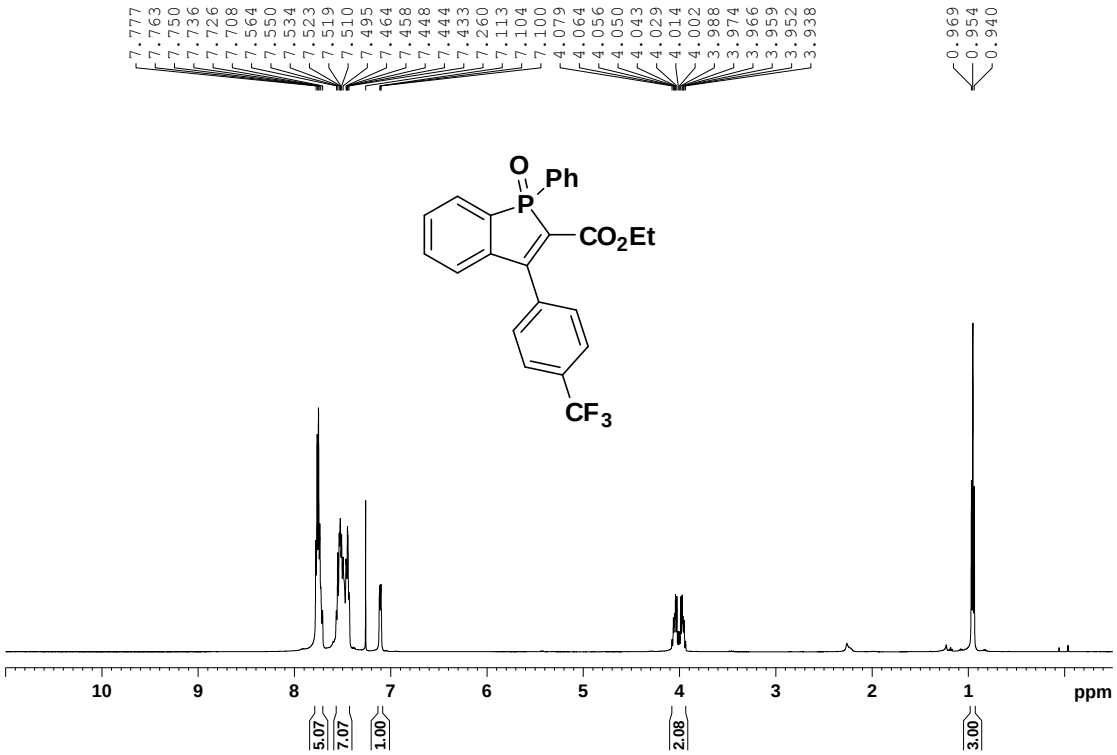
^{1}H NMR spectrum of compound **3p**.



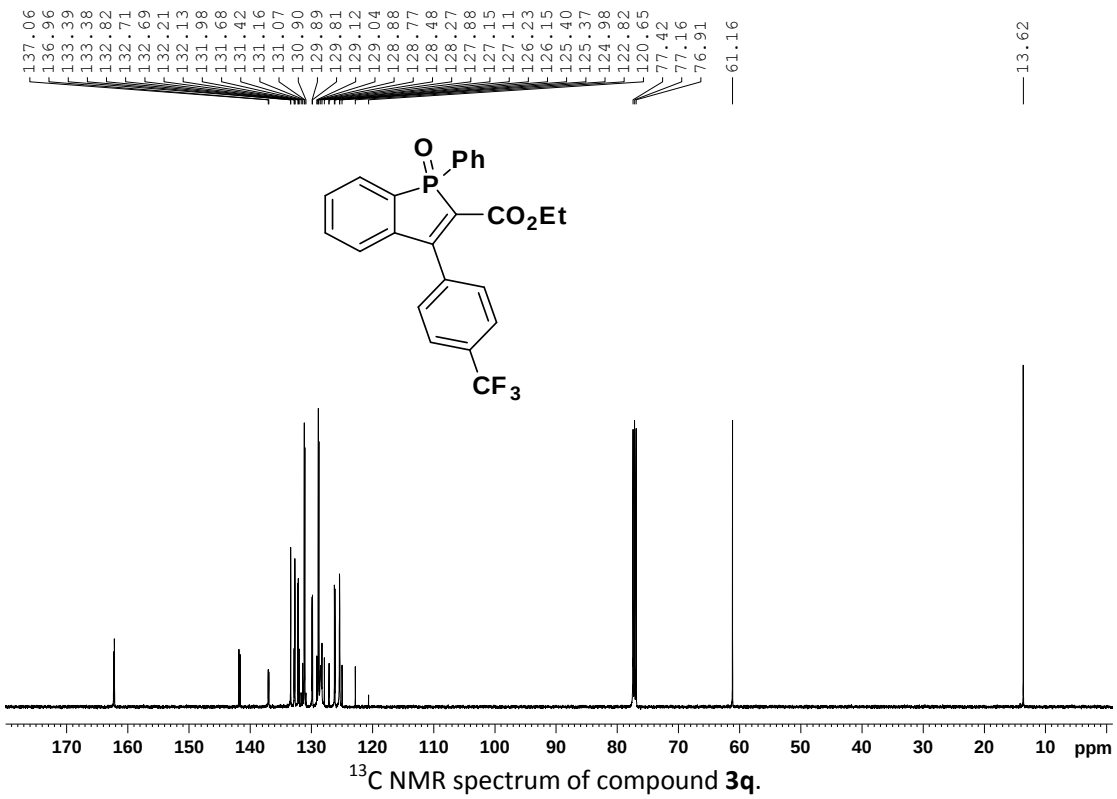
^{13}C NMR spectrum of compound **3p**.



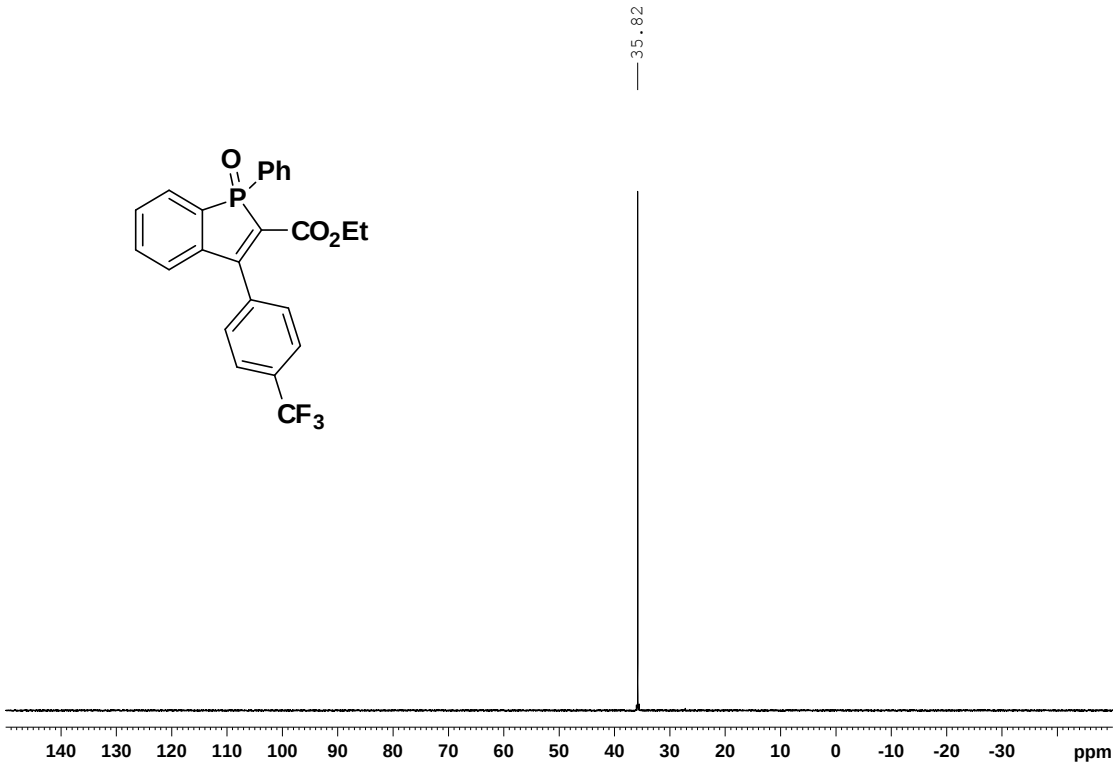
^{31}P NMR spectrum of compound **3p**.



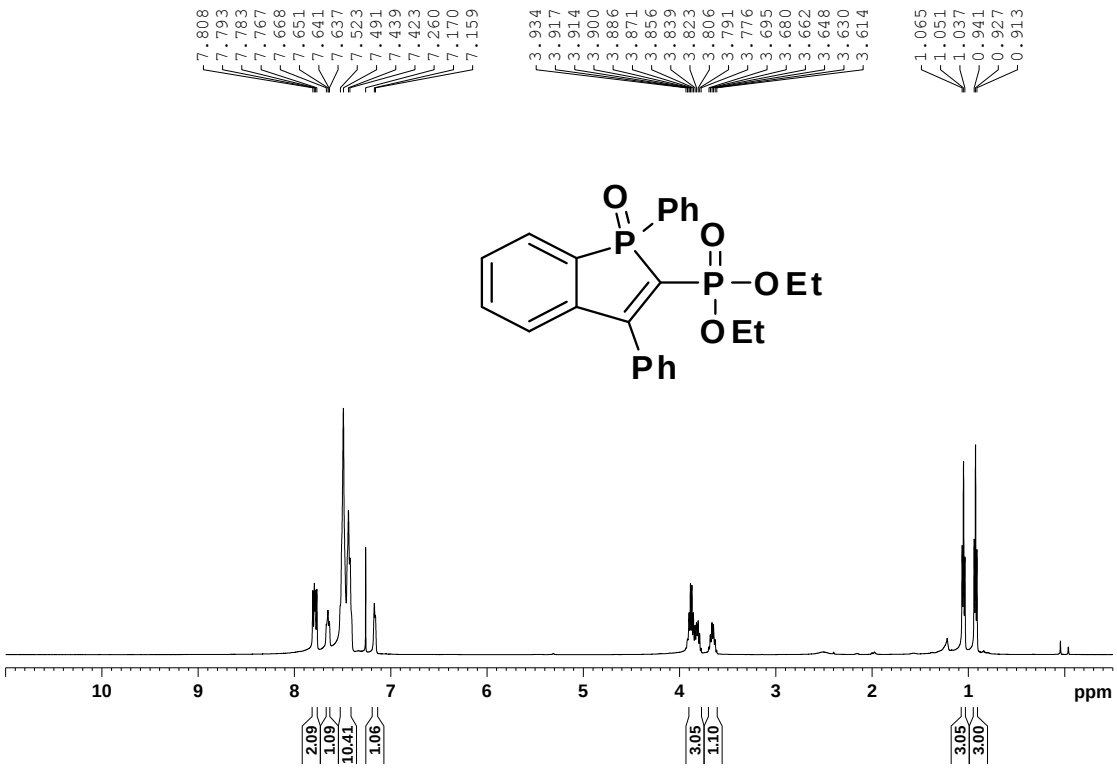
^{1}H NMR spectrum of compound **3q**.



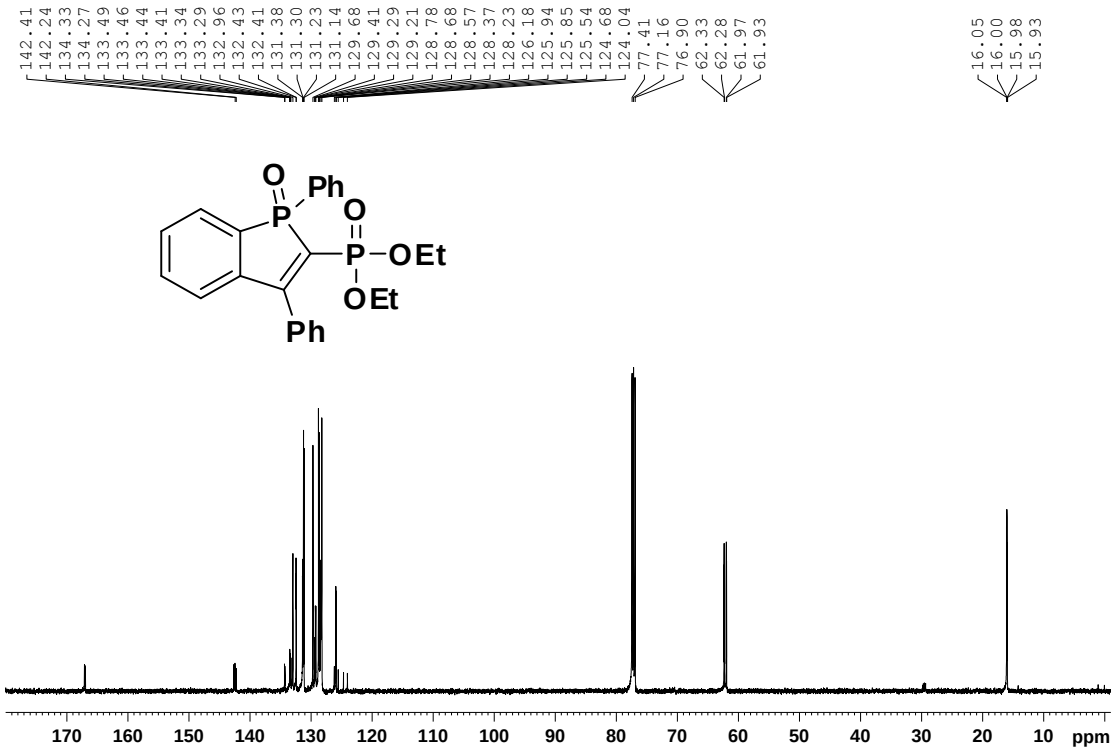
^{13}C NMR spectrum of compound **3q**.



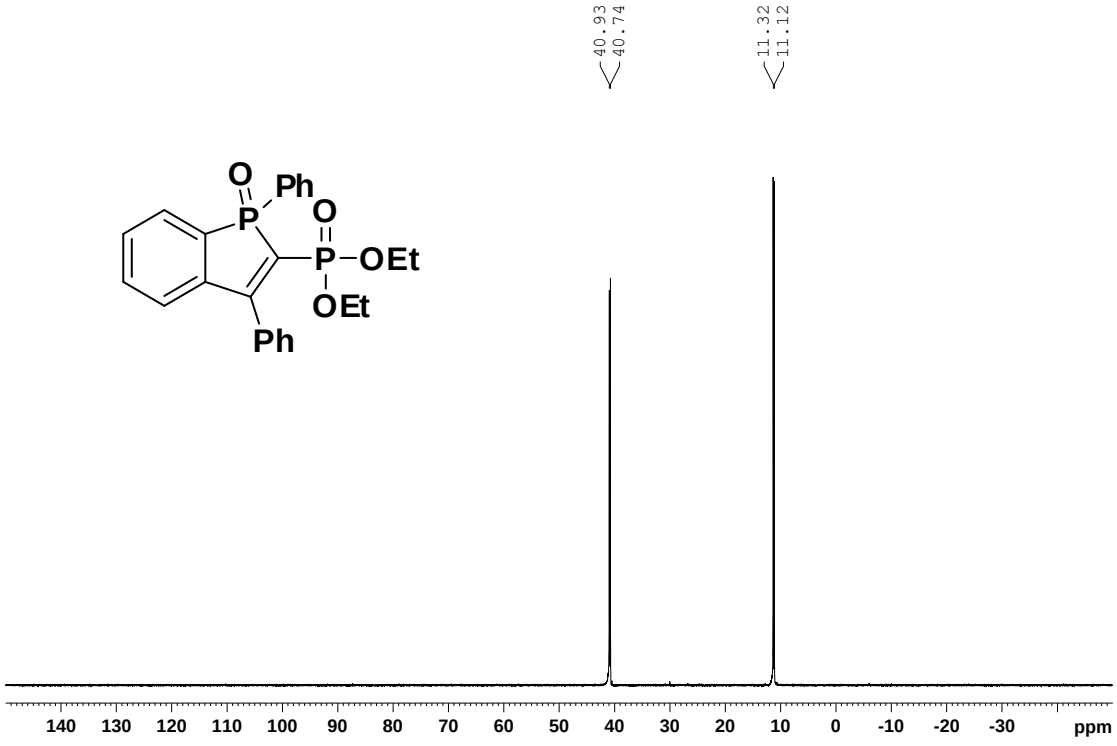
^{31}P NMR spectrum of compound **3q**.



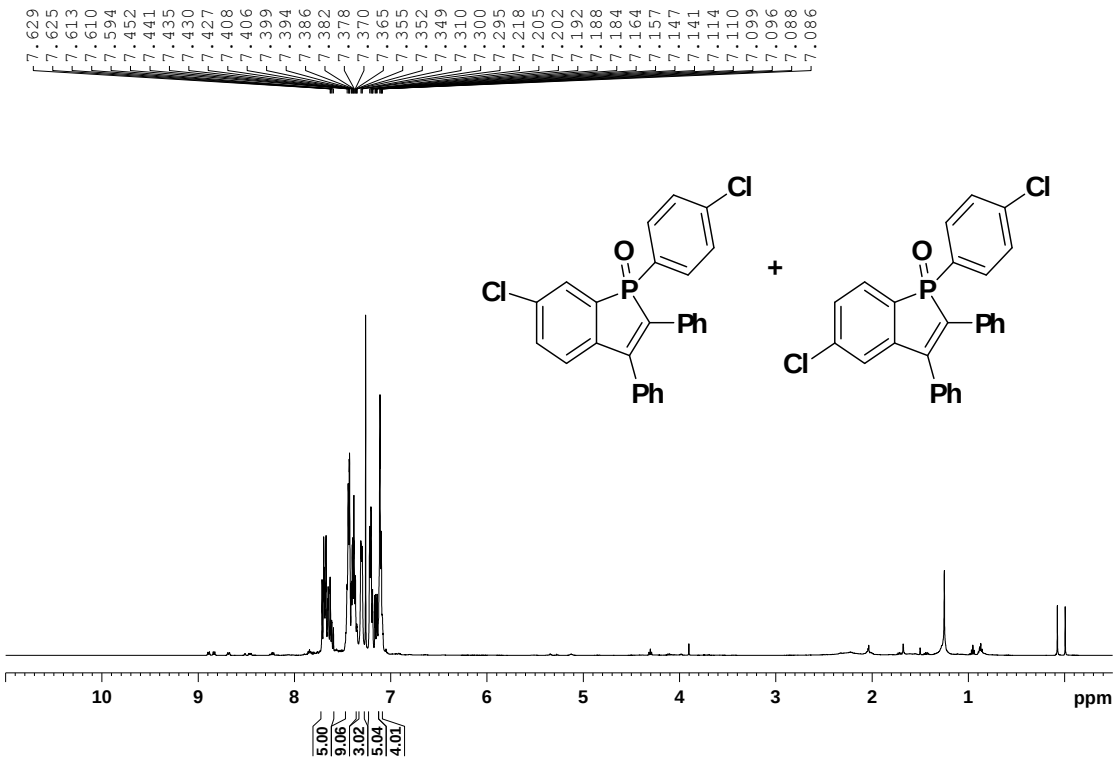
^{1}H NMR spectrum of compound **3r**.



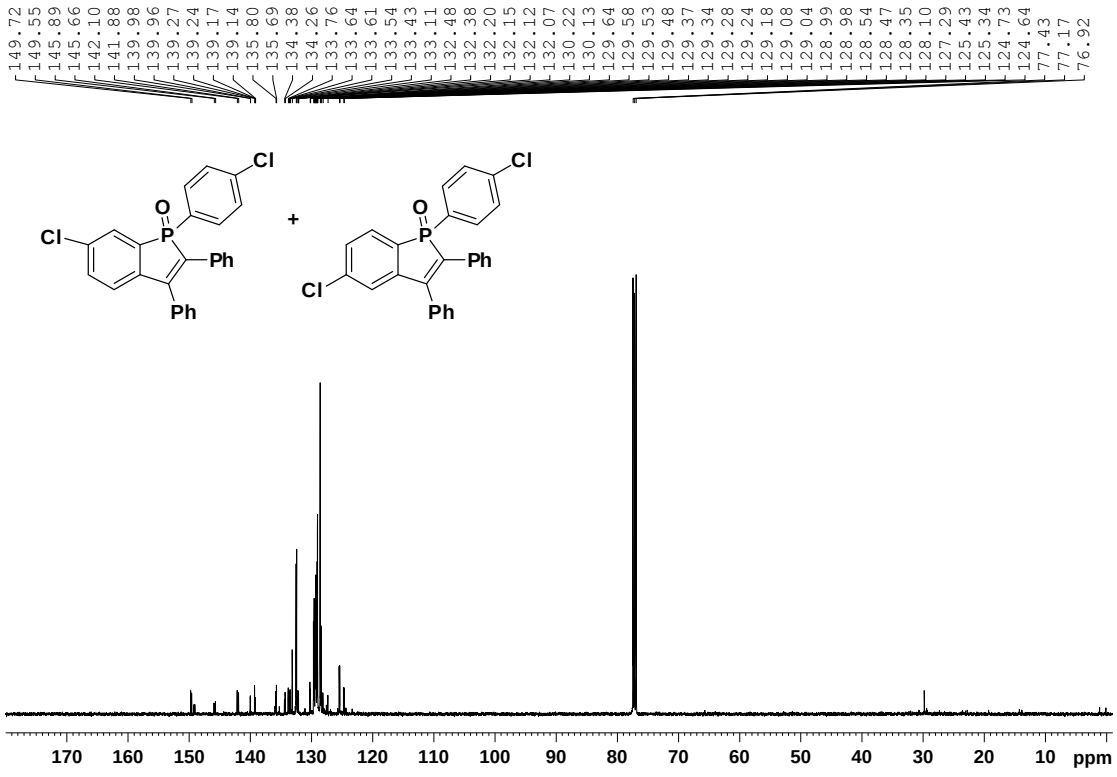
^{13}C NMR spectrum of compound **3r**.



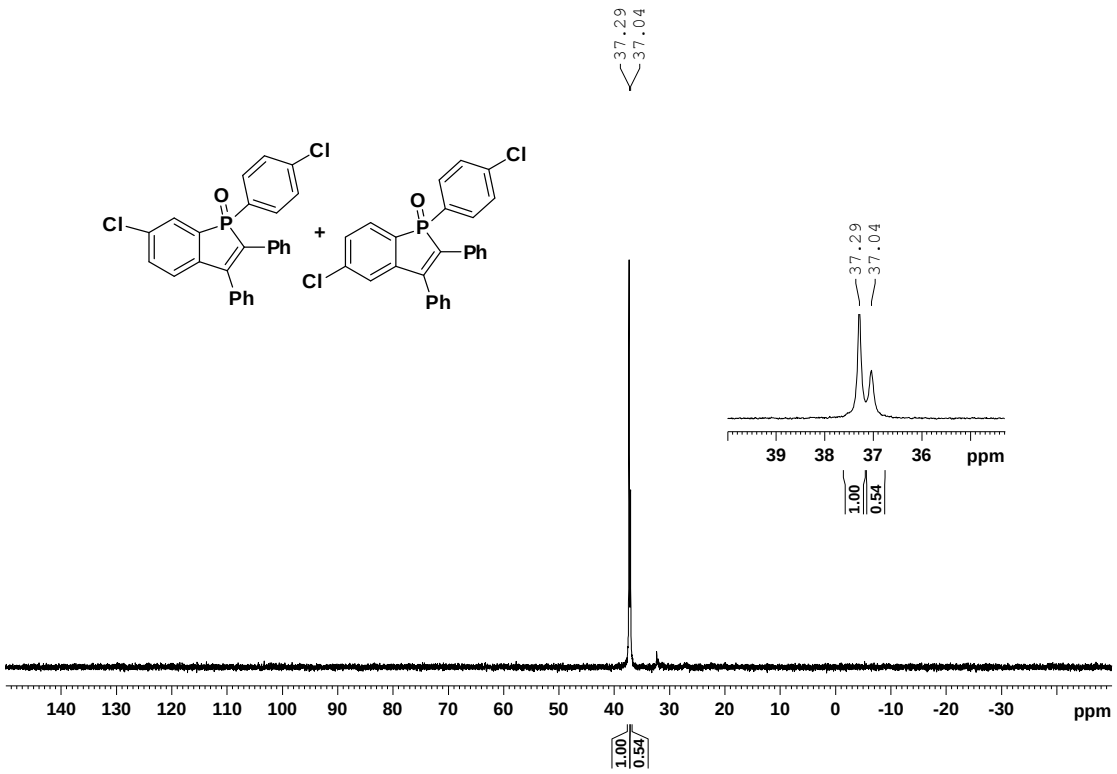
^{31}P NMR spectrum of compound **3r**.



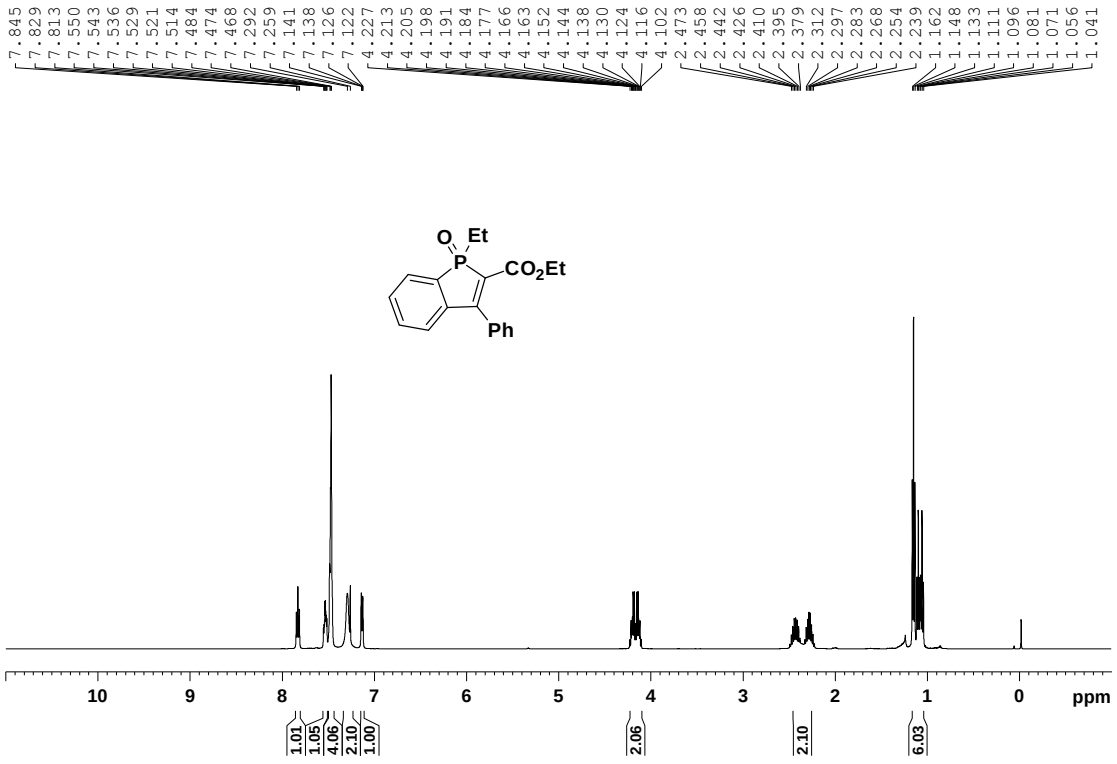
^{1}H NMR spectrum of compound **3s**.



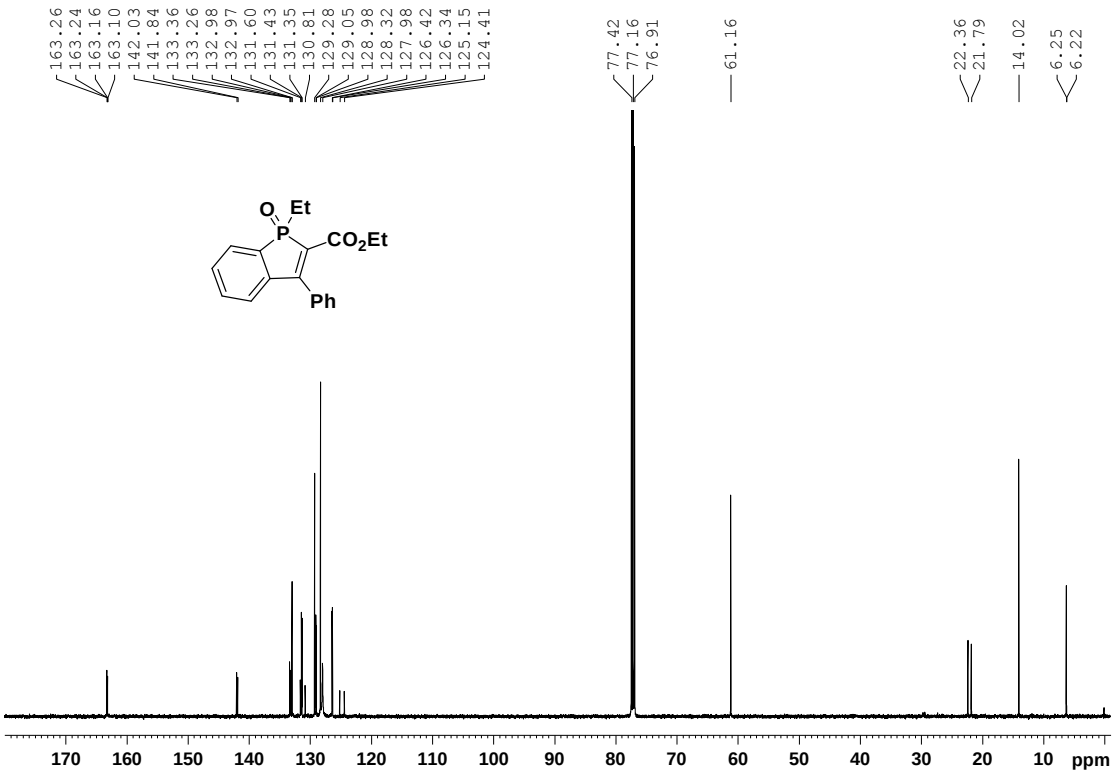
^{13}C NMR spectrum of compound **3s**.



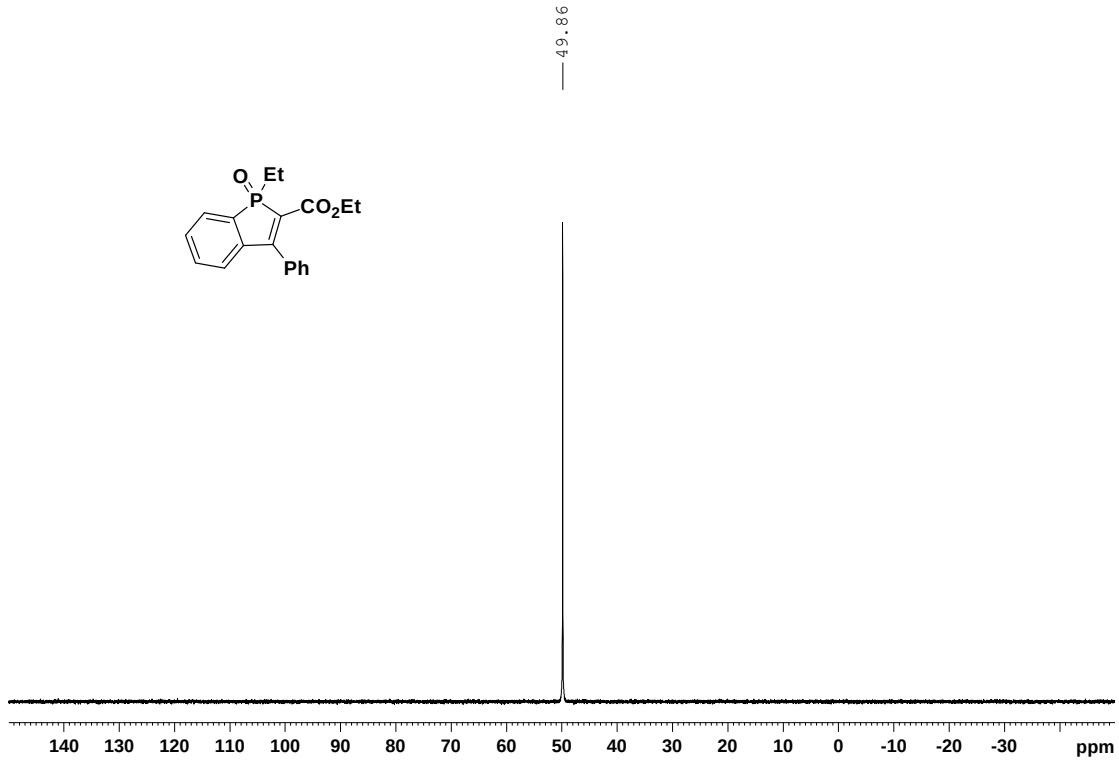
^{31}P NMR spectrum of compound **3s**.



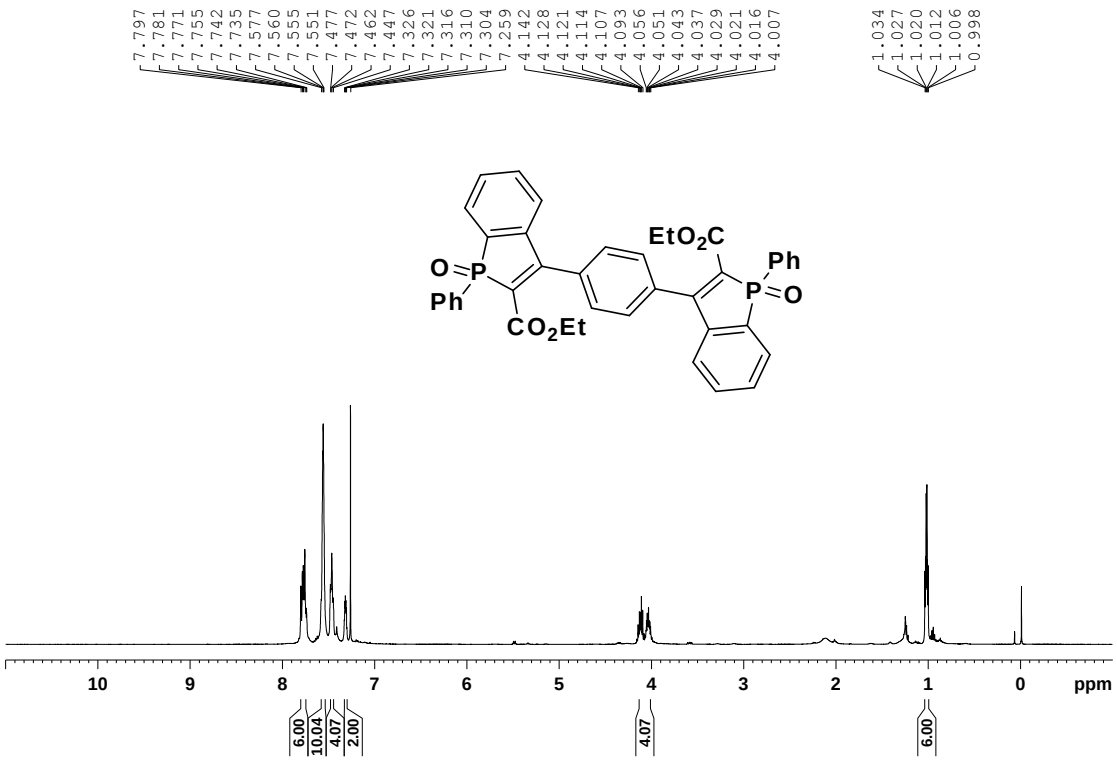
^{1}H NMR spectrum of compound **3t**.



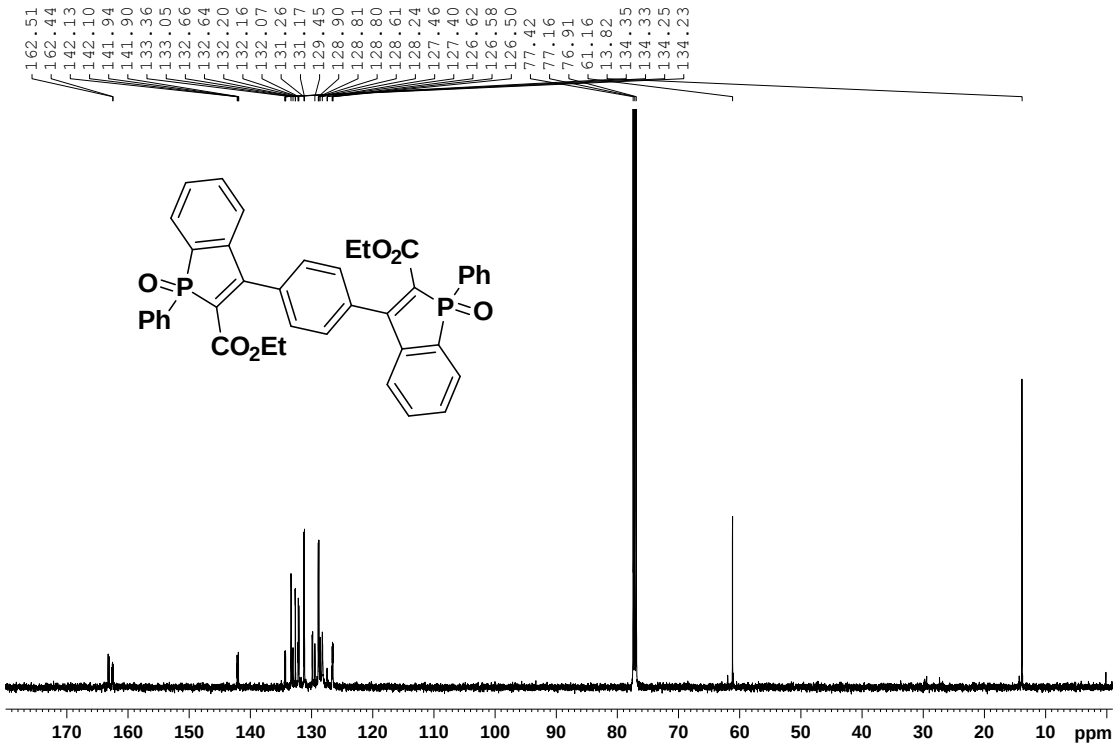
^{13}C NMR spectrum of compound **3t**.



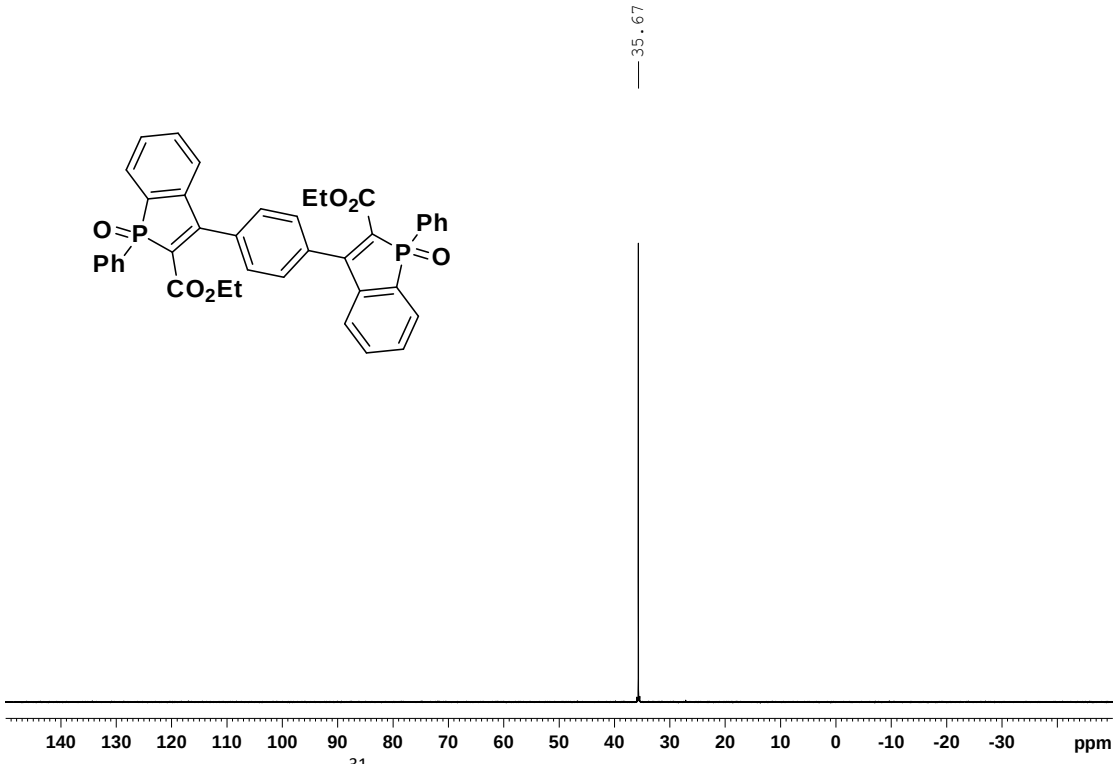
^{31}P NMR spectrum of compound **3t**.



^{1}H NMR spectrum of compound **3u**.



^{13}C NMR spectrum of compound **3u**.



^{31}P NMR spectrum of compound **3u**.