

Dearomative oxyphosphorylation of indoles enables facile access to 2,2-disubstituted indolin-3-ones

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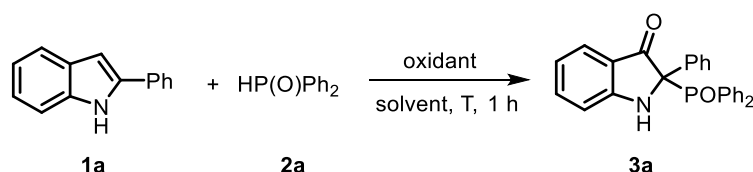
1. General Information

NMR spectra were recorded with BrukerAvance III HD500 and BrukerAvance III HD400 spectrometer at 500 MHz or 400 MHz. All ^1H , ^{19}F , ^{31}P and ^{13}C NMR spectra were recorded using CDCl_3 or $[\text{D}_6]$ DMSO as solvent. Tetramethylsilane (TMS) signals or residual solvent signals were used [TMS $\delta = 0.00$ (^1H NMR), CDCl_3 $\delta = 77.16$ (^{13}C NMR), DMSO- d_6 $\delta = 2.50$ (^1H NMR), 39.52 (^{13}C NMR)] as internal standards. The following abbreviations were used to describe peak splitting patterns when appropriate: s = singlet, d = doublet, dd = double doublet, t = triplet, q = quartet, m = multiplet, qd = quartet doublet. HRMS (ESI) Mass spectra were recorded on Thermo Fisher Scientific LTQ FT Ultra. All the reactions were monitored by thin layer chromatography (TLC), carried out on 0.25 mm silica gel plates using UV light as visualizing agent. Column chromatography was carried out on silica gel (particle size 300-400 mesh). The C2 substituted indoles and TEMPO oxoammonium salts were synthesized according to procedures described in the literature.^[1,2] Substrates **2** were prepared according to the literature method.^[3]

2. Experiment Section

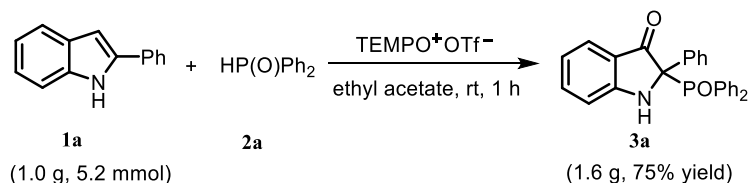
2.1 Experimental procedure for optimization of reaction conditions

(Table 1):



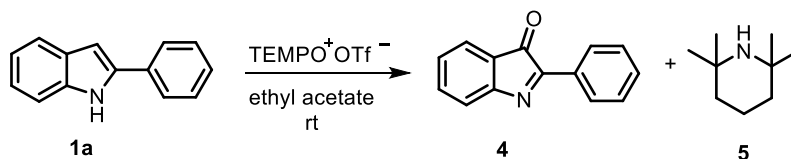
To a solution of 2-phenyl-1H-indole **1a** (0.1 mmol) in 1.5 mL solvent was added oxidant (0.1 mmol) at rt and stirred for 5 min. Then *H*-phosphine oxides **2a** (1.5 mmol) in ethyl acetate (0.5 mL) were added to the mixture. The mixture was stirred at room temperature for 1 h. After reaction, the solution was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate = 2:1) to give the product **3a**.

2.2 Experimental procedure for gram-scale reaction (Scheme 4):



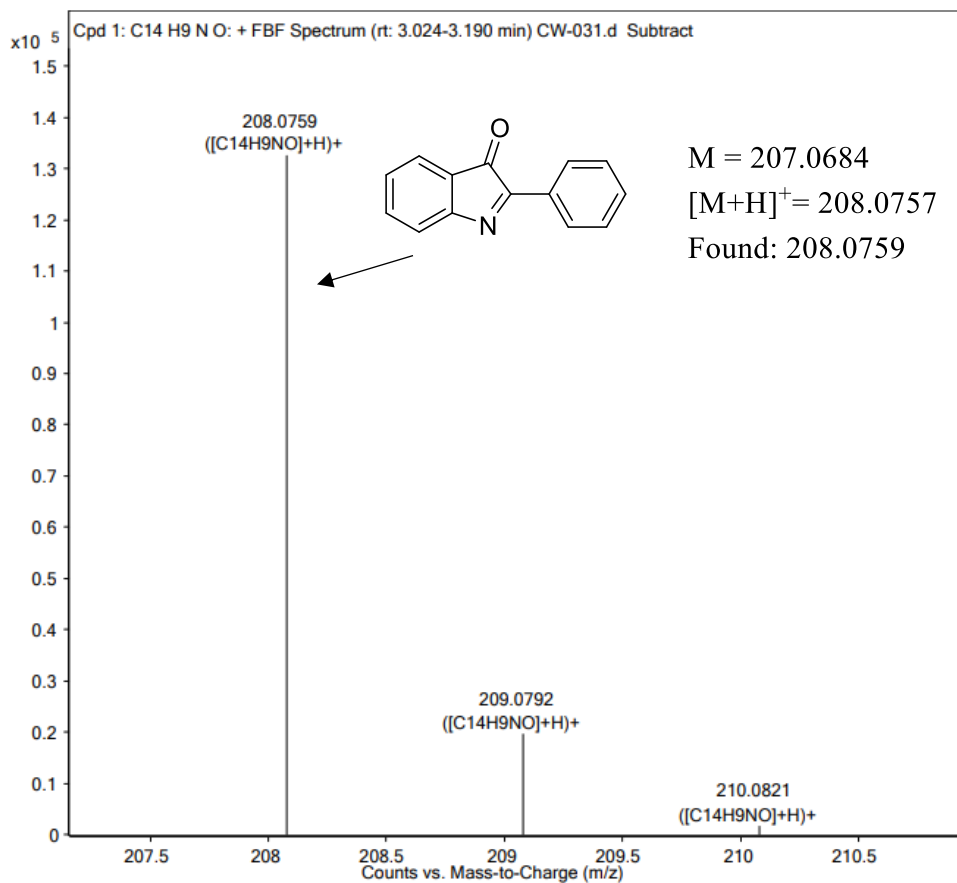
A solution of 2-phenyl-1H-indole **1a** (1.0 g, 5.2 mmol) and TEMPO⁺OTf⁻ (1.6 g, 5.2 mmol) in 80 mL ethyl acetate were added to 250 mL round bottom flask at rt until the disappearance of **1a**. H-phosphorus oxide **2a** (1.6 g, 7.8 mmol) in ethyl acetate (10 mL) were added to the mixture for 1h. After reaction, the solution was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate =2:1 to 1:1) to give the product **3a** (1.6 g) in 75% yield.

2.3 Mechanism studies (Scheme 5):

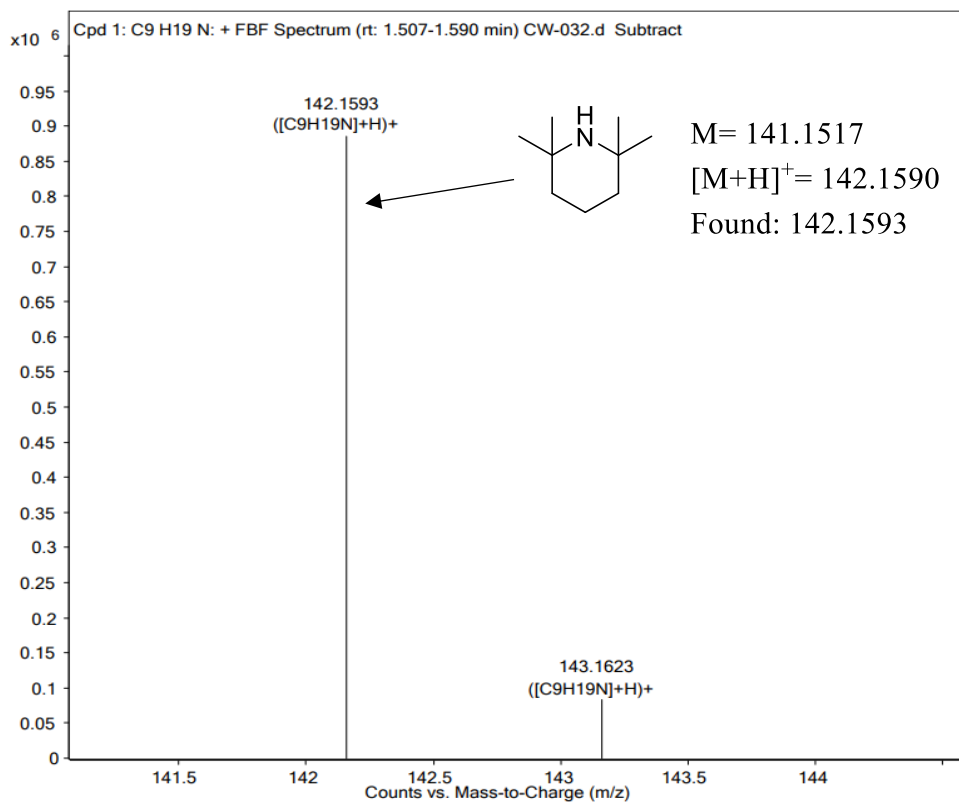


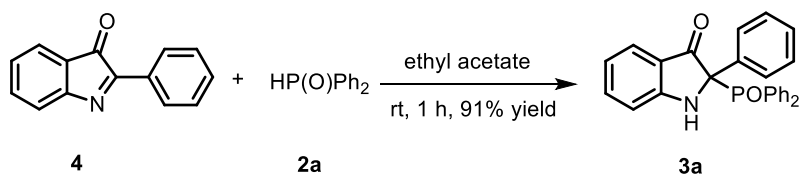
A solution of 2-phenyl-1H-indole **1a** (0.1 mmol) in 2 mL ethyl acetate was added TEMPO⁺OTf⁻ (0.1 mmol) at rt. The mixture was stirred until the disappearance of **1a**. Then, the solution was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate =50:1) to afford the red solid product **4**. The ¹H NMR of **4** was consistent with that reported previously.^[4] ESI-MS analysis was performed with 2,2,6,6-tetramethylpiperidine **5**.

2-phenyl-3H-indol-3-one (4), ¹H NMR (500 MHz, Chloroform-*d*) δ 8.40–8.34 (m, 2H), 7.57–7.51 (m, 3H), 7.50–7.45 (m, 2H), 7.41 (d, *J* = 7.4 Hz, 1H), 7.25–7.22 (m, 1H). HRMS (ESI) *m/z* calcd for: C₁₄H₉NO [M+H]⁺: 208.0757, found: 208.0759.



2,2,6,6-tetramethylpiperidine (5), HRMS (ESI) m/z calcd for: C₉H₁₉N [M+H]⁺ : 142.1590, found:142.1593.





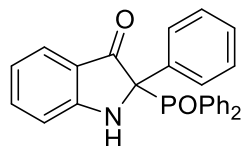
A solution of ketimines **4** (0.0207 g, 0.1 mmol) and *H*-phosphorus oxide **2a** (0.0303 g, 0.15 mmol) in ethyl acetate (2 mL) were added to centrifugal tube. The reaction mixture was stirred at room temperature for 1 h. After reaction, the solution was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate = 2:1) to give the product **3a** (0.0371 g) in 91% yield.

3. Reference

- [1] (a) S. K. Bhunia, A. Polley, R. Natarajan, R. Jana, *Chem.Eur.J.*, 2015, **21**,16786–16791; (b) N. Jacob, L. Guillemard, J. Wencel-Delord, *Synthesis*, 2020, **52**, 574-580.
- [2] X. Liu, X. Yan, Y. Tang, C. S. Jiang, J. H. Yu, K. Wang, H. Zhang, *Chem. Commun.*, 2019, **55**, 6535-6538.
- [3] C. A. Busacca, J. C. Lorenz, N. Grinberg, N. Haddad, M. Hrapchak, B. Latli, H. Lee, P. Sabila, A. Saha, M. Sarvestani, S. Shen, R. Varsolona, X. Wei, C. H. Senanayake, *Org. Lett.*, 2005, **7**, 4277.
- [4] (a) K. Q. Ling, *Synth. Commun.*, 1995, **25**, 3831; (b) J.-X. Liu, Q. Q. Zhou, J. G. Deng, Y. C. Chen, *Org. Biomol. Chem.*, 2013, **11**, 8175; (c) E. Najahi, A. Valentin, P.-L. Fabre, K. Reybier, F. Nepveu, *Eur. J. Med. Chem.*, 2014, **78**, 269.

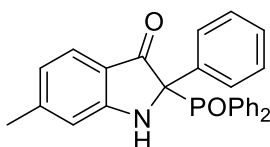
4. Characterization data of the compounds

2-(diphenylphosphoryl)-2-phenylindolin-3-one (3a). Yellow solid, 33.0 mg, 81%



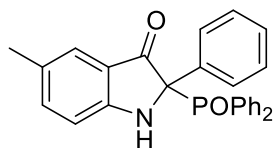
yield. $^1\text{H NMR}$ (500 MHz, $\text{DMSO-}d_6$) δ 8.65 (s, 1H), 7.83–7.74 (m, 4H), 7.65–7.58 (m, 2H), 7.51 (t, $J = 7.0$ Hz, 2H), 7.43–7.35 (m, 5H), 7.34–7.28 (m, 3H), 7.22 (d, $J = 7.7$ Hz, 1H), 6.96 (d, $J = 8.3$ Hz, 1H), 6.61 (t, $J = 7.4$ Hz, 1H); $^{13}\text{C NMR}$ (126 MHz, $\text{DMSO-}d_6$) δ 195.50, 160.82 (d, $J = 4.9$ Hz), 137.83, 133.85, 133.03 (d, $J = 8.3$ Hz), 132.72 (d, $J = 2.2$ Hz), 132.61 (d, $J = 2.4$ Hz), 132.09 (d, $J = 8.7$ Hz), 130.64 (d, $J = 97.4$ Hz), 129.31 (d, $J = 97.0$ Hz), 128.72 (d, $J = 11.5$ Hz), 128.48 (d, $J = 11.4$ Hz), 128.41, 128.33 (d, $J = 1.4$ Hz), 127.14 (d, $J = 3.5$ Hz), 124.32, 119.80, 118.73, 112.83, 74.32 (d, $J = 68.2$ Hz); $^{31}\text{P NMR}$ (202 MHz, $\text{DMSO-}d_6$) δ 25.82; **HRMS** (ESI) m/z calcd for: $\text{C}_{26}\text{H}_{20}\text{NO}_2\text{PNa}$ [$\text{M}+\text{Na}$] $^+$: 432.1124, found: 432.1124.

2-(diphenylphosphoryl)-6-methyl-2-phenylindolin-3-one (3b). Yellow solid, 35.9



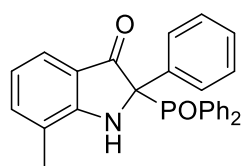
mg, 85% yield. $^1\text{H NMR}$ (500 MHz, $\text{Chloroform-}d$) δ 8.07–7.98 (m, 2H), 7.89 (dd, $J = 11.0, 7.8$ Hz, 2H), 7.77 (d, $J = 7.8$ Hz, 2H), 7.58–7.51 (m, 2H), 7.46–7.36 (m, 3H), 7.33 (d, $J = 8.0$ Hz, 1H), 7.28–7.23 (m, 2H), 7.23–7.14 (m, 3H), 6.64 (s, 1H), 6.49 (d, $J = 7.9$ Hz, 1H), 2.23 (s, 3H); $^{13}\text{C NMR}$ (126 MHz, $\text{Chloroform-}d$) δ 196.07 (d, $J = 1.2$ Hz), 160.70 (d, $J = 4.0$ Hz), 149.04, 133.35 (d, $J = 1.8$ Hz), 133.10 (d, $J = 9.2$ Hz), 132.28 (d, $J = 2.8$ Hz), 132.19 (d, $J = 9.1$ Hz), 132.12 (d, $J = 2.9$ Hz), 129.23 (d, $J = 96.6$ Hz), 128.86 (d, $J = 98.6$ Hz), 128.13–128.05 (m), 127.99 (d, $J = 1.5$ Hz), 127.89 (d, $J = 2.0$ Hz), 127.57 (d, $J = 4.0$ Hz), 124.22, 120.72, 119.11, 112.22, 75.13 (d, $J = 63.0$ Hz), 22.44; $^{31}\text{P NMR}$ (202 MHz, $\text{Chloroform-}d$) δ 29.65; **HRMS** (ESI) m/z calcd for: $\text{C}_{27}\text{H}_{22}\text{NO}_2\text{PNa}$ [$\text{M}+\text{Na}$] $^+$: 446.1280, found: 446.1280.

2-(diphenylphosphoryl)-5-methyl-2-phenylindolin-3-one (3c). Yellow solid, 37.3



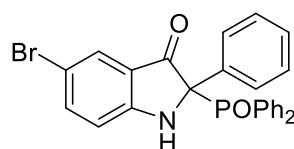
mg, 88% yield. $^1\text{H NMR}$ (500 MHz, $\text{Chloroform-}d$) δ 8.07–7.97 (m, 2H), 7.92–7.84 (m, 2H), 7.82–7.76 (m, 2H), 7.57 (s, 1H), 7.54–7.50 (m, 1H), 7.45–7.39 (m, 2H), 7.39–7.34 (m, 1H), 7.28–7.16 (m, 6H), 7.12 (dd, $J = 8.4, 1.8$ Hz, 1H), 6.78 (d, $J = 8.4$ Hz, 1H), 2.19 (s, 3H); $^{13}\text{C NMR}$ (126 MHz, $\text{Chloroform-}d$) δ 196.82, 158.74 (d, $J = 4.0$ Hz), 138.93, 133.36 (d, $J = 2.0$ Hz), 133.08 (d, $J = 9.1$ Hz), 132.27 (d, $J = 2.9$ Hz), 132.18 (d, $J = 9.1$ Hz), 132.07 (d, $J = 2.8$ Hz), 129.23 (d, $J = 96.5$ Hz), 128.84 (d, $J = 98.5$ Hz), 128.29, 128.09 (d, $J = 1.8$ Hz), 127.99 (d, $J = 1.5$ Hz), 127.95, 127.91 (d, $J = 2.3$ Hz), 127.58 (d, $J = 3.9$ Hz), 123.72, 121.44, 112.23, 75.26 (d, $J = 62.6$ Hz), 20.48; $^{31}\text{P NMR}$ (202 MHz, $\text{Chloroform-}d$) δ 29.44; **HRMS** (ESI) m/z calcd for: $\text{C}_{27}\text{H}_{22}\text{NO}_2\text{PNa}$ [$\text{M}+\text{Na}$] $^+$: 446.1280, found: 446.1280.

2-(diphenylphosphoryl)-7-methyl-2-phenylindolin-3-one (3d). Yellow solid, 34.5



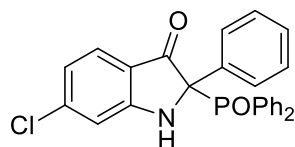
mg, 82% yield. $^1\text{H NMR}$ (500 MHz, Chloroform-*d*) δ 8.03–7.95 (m, 2H), 7.84–7.77 (m, 2H), 7.77–7.71 (m, 2H), 7.51 (t, $J = 7.3$ Hz, 1H), 7.44–7.38 (m, 2H), 7.30–7.35 (m, 2H), 7.24–7.18 (m, 5H), 7.09 (d, $J = 7.1$ Hz, 1H), 6.60 (t, $J = 7.5$ Hz, 1H), 6.39 (s, 1H), 2.19 (s, 3H); $^{13}\text{C NMR}$ (126 MHz, Chloroform-*d*) δ 196.83, 158.81 (d, $J = 2.4$ Hz), 137.20, 133.14, 133.04 (d, $J = 9.1$ Hz), 132.46 (d, $J = 2.2$ Hz), 132.23 (d, $J = 2.1$ Hz), 131.92 (d, $J = 8.8$ Hz), 128.13 (d, $J = 12.2$ Hz), 128.12 (d, $J = 1.4$ Hz), 128.05 (d, $J = 0.64$ Hz), 128.37 (d, $J = 91.50$ Hz), 127.90 (d, $J = 11.8$ Hz), 127.56 (d, $J = 91.09$ Hz), 127.53 (d, $J = 3.6$ Hz), 121.92, 121.39, 121.37, 119.56, 74.98 (d, $J = 60.0$ Hz), 15.79; $^{31}\text{P NMR}$ (202 MHz, Chloroform-*d*) δ 29.68; **HRMS** (ESI) m/z calcd for: $\text{C}_{27}\text{H}_{22}\text{NO}_2\text{PNa}$ [$\text{M}+\text{Na}$] $^+$: 446.1280, found: 446.1280.

5-bromo-2-(diphenylphosphoryl)-2-phenylindolin-3-one (3e). Yellow solid, 22.1



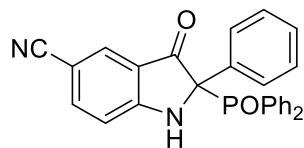
mg, 45% yield. $^1\text{H NMR}$ (500 MHz, Chloroform-*d*) δ 8.21 (s, 1H), 7.97 (dd, $J = 10.7, 8.1$ Hz, 2H), 7.91–7.82 (m, 2H), 7.72 (d, $J = 7.8$ Hz, 2H), 7.58–7.50 (m, 2H), 7.46–7.38 (m, 3H), 7.31 (dd, $J = 8.7, 2.0$ Hz, 1H), 7.29–7.27 (m, 2H), 7.21 (t, $J = 7.2$ Hz, 1H), 7.15 (t, $J = 7.5$ Hz, 2H), 6.76 (d, $J = 8.7$ Hz, 1H); $^{13}\text{C NMR}$ (126 MHz, Chloroform-*d*) δ 195.66, 158.88 (d, $J = 3.4$ Hz), 139.84, 133.03 (d, $J = 9.2$ Hz), 132.57, 132.54 (d, $J = 2.6$ Hz), 132.46 (d, $J = 1.9$ Hz), 132.11 (d, $J = 9.0$ Hz), 130.54 (d, $J = 93.6$ Hz), 129.24 (d, $J = 93.0$ Hz), 128.26 (d, $J = 9.0$ Hz), 128.18, 128.17 (d, $J = 9.1$ Hz), 128.02 (d, $J = 1.4$ Hz), 127.47 (d, $J = 3.5$ Hz), 126.83, 122.58, 113.95, 110.77, 75.80 (d, $J = 62.2$ Hz); $^{31}\text{P NMR}$ (202 MHz, Chloroform-*d*) δ 29.27; **HRMS** (ESI) m/z calcd for: $\text{C}_{26}\text{H}_{19}\text{NO}_2\text{PBrNa}$ [$\text{M}+\text{Na}$] $^+$: 510.0229 (^{79}Br), found: 510.0229.

6-chloro-2-(diphenylphosphoryl)-2-phenylindolin-3-one (3f). Yellow solid, 21.0 mg,



47% yield. $^1\text{H NMR}$ (500 MHz, DMSO-*d*₆) δ 8.92 (s, 1H), 7.84–7.77 (m, 2H), 7.73 (d, $J = 5.5$ Hz, 2H), 7.66–7.59 (m, 2H), 7.53 (q, $J = 6.7$ Hz, 2H), 7.48–7.39 (m, 4H), 7.38–7.28 (m, 3H), 7.24 (d, $J = 8.3$ Hz, 1H), 6.96 (s, 1H), 6.65–6.58 (m, 1H); $^{13}\text{C NMR}$ (126 MHz, DMSO-*d*₆) δ 194.33, 160.96 (d, $J = 4.5$ Hz), 142.61, 133.47, 133.05 (d, $J = 8.4$ Hz), 132.91 (d, $J = 2.3$ Hz), 132.79 (d, $J = 2.7$ Hz), 132.09 (d, $J = 8.7$ Hz), 130.31 (d, $J = 97.6$ Hz), 128.99 (d, $J = 96.8$ Hz), 128.81 (d, $J = 11.7$ Hz), 128.60 (d, $J = 11.2$ Hz), 128.57, 128.44 (d, $J = 1.5$ Hz), 127.05 (d, $J = 3.6$ Hz), 126.05, 119.11, 118.65, 112.03, 74.98 (d, $J = 66.6$ Hz); $^{31}\text{P NMR}$ (202 MHz, DMSO-*d*₆) δ 25.73; **HRMS** (ESI) m/z calcd for: $\text{C}_{26}\text{H}_{19}\text{NO}_2\text{PClNa}$ [$\text{M}+\text{Na}$] $^+$: 466.0734, found: 466.0734.

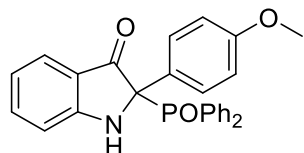
2-(diphenylphosphoryl)-3-oxo-2-phenylindoline-5-carbonitrile (3g). Yellow solid,



34.0 mg, 78% yield. $^1\text{H NMR}$ (500 MHz, Chloroform-*d*) δ 8.00 (dd, $J = 10.8, 8.0$ Hz, 2H), 7.91–7.82 (m, 2H), 7.80–7.73 (m, 2H), 7.72 (s, 1H), 7.54–7.48 (m, 1H), 7.44–7.39 (m, 3H), 7.34 (t, $J = 6.9$ Hz, 1H), 7.20 (dd, $J = 9.9, 4.0$ Hz, 3H), 7.16 (t, $J = 7.3$ Hz, 2H), 6.84 (d, $J = 8.3$ Hz, 1H), 6.63 (t, $J = 7.3$ Hz, 1H); ^{13}C

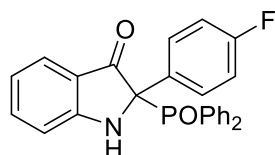
NMR (126 MHz, Chloroform-*d*) δ 196.88, 160.18 (d, $J = 3.7$ Hz), 137.36, 133.10 (d, $J = 2.2$ Hz), 133.08 (d, $J = 9.2$ Hz), 132.37 (d, $J = 2.8$ Hz), 132.18 (d, $J = 3.3$ Hz), 132.17 (d, $J = 9.0$ Hz), 128.99 (d, $J = 96.7$ Hz), 128.62 (d, $J = 98.6$ Hz), 128.12 (d, $J = 7.5$ Hz), 128.02, 128.02 (d, $J = 7.1$ Hz), 127.97 (d, $J = 2.1$ Hz), 127.56 (d, $J = 3.9$ Hz), 124.53, 121.29, 118.83, 112.31, 112.21, 75.01 (d, $J = 62.0$ Hz); **^{31}P NMR** (202 MHz, Chloroform-*d*) δ 29.50; **HRMS** (APCI) m/z calcd for: $\text{C}_{27}\text{H}_{19}\text{N}_2\text{O}_2\text{P}$ $[\text{M}+\text{H}]^+$: 435.1257, found: 435.1272.

2-(diphenylphosphoryl)-2-(4-methoxyphenyl)indolin-3-one (3h). Yellow solid, 35.2



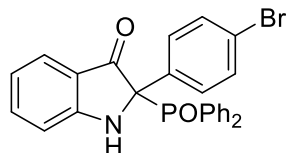
mg, 80% yield. **^1H NMR** (500 MHz, Chloroform-*d*) δ 8.04–7.95 (m, 2H), 7.88–7.80 (m, 2H), 7.68 (d, $J = 7.5$ Hz, 2H), 7.60 (s, 1H), 7.51 (t, $J = 7.3$ Hz, 1H), 7.41 (t, $J = 6.8$ Hz, 3H), 7.34 (t, $J = 7.3$ Hz, 1H), 7.28–7.18 (m, 3H), 6.84 (d, $J = 8.3$ Hz, 1H), 6.69 (d, $J = 8.8$ Hz, 2H), 6.63 (t, $J = 7.4$ Hz, 1H), 3.71 (s, 3H); **^{13}C NMR** (126 MHz, Chloroform-*d*) δ 197.16, 160.11 (d, $J = 2.7$ Hz), 159.35, 137.33, 133.08 (d, $J = 8.6$ Hz), 132.34, 132.15 (d, $J = 5.7$ Hz), 132.11, 129.04 (d, $J = 94.2$ Hz), 128.82 (d, $J = 3.0$ Hz), 128.11 (d, $J = 10.4$ Hz), 128.02 (d, $J = 10.0$ Hz), 127.92 (d, $J = 94.6$ Hz), 125.02, 124.51, 121.30, 118.81, 113.46 – 113.36 (m), 112.36, 74.54 (d, $J = 63.9$ Hz), 55.18; **^{31}P NMR** (202 MHz, Chloroform-*d*) δ 29.74; **HRMS** (ESI) m/z calcd for: $\text{C}_{27}\text{H}_{22}\text{NO}_3\text{PNa}$ $[\text{M}+\text{Na}]^+$: 462.1230, found: 462.1230.

2-(diphenylphosphoryl)-2-(4-fluorophenyl)indolin-3-one (3i). Yellow solid, 32.1



mg, 75% yield. **^1H NMR** (500 MHz, Chloroform-*d*) δ 8.12 (s, 1H), 8.00 (dd, $J = 11.2, 7.7$ Hz, 2H), 7.94–7.82 (m, 2H), 7.83–7.73 (m, 2H), 7.58–7.50 (m, 1H), 7.49–7.40 (m, 3H), 7.39–7.33 (m, 1H), 7.30–7.20 (m, 3H), 6.88–6.77 (m, 3H), 6.64 (t, $J = 7.4$ Hz, 1H); **^{13}C NMR** (126 MHz, Chloroform-*d*) δ 196.89, , 162.54 (dd, $J = 247.2, 2.3$ Hz), 160.28 (d, $J = 3.7$ Hz), 137.53, 133.04 (d, $J = 9.1$ Hz), 132.54 (d, $J = 2.8$ Hz), 132.31 (d, $J = 2.7$ Hz), 132.09 (d, $J = 9.1$ Hz), 129.45 (dd, $J = 8.2, 3.7$ Hz), 128.86 (d, $J = 3.0$ Hz), 128.78 (d, $J = 96.5$ Hz), 128.47 (d, $J = 95.4$ Hz), 128.21 (d, $J = 9.8$ Hz), 128.11 (d, $J = 9.5$ Hz), 124.55, 121.11, 118.87, 114.73 (dd, $J = 21.3, 2.1$ Hz), 112.32, 74.52 (d, $J = 61.6$ Hz); **^{31}P NMR** (202 MHz, Chloroform-*d*) δ 29.53; **^{19}F NMR** (471 MHz, Chloroform-*d*) δ -114.10; **HRMS** (ESI) m/z calcd for: $\text{C}_{26}\text{H}_{19}\text{NO}_2\text{PFNa}$ $[\text{M}+\text{Na}]^+$: 450.1030 found: 450.1031.

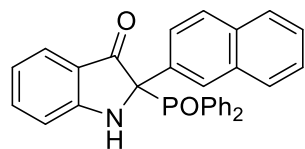
2-(4-bromophenyl)-2-(diphenylphosphoryl)indolin-3-one (3j). Yellow solid, 33.9



mg, 70% yield. **^1H NMR** (500 MHz, Chloroform-*d*) δ 8.05–7.98 (m, 2H), 7.95 (s, 1H), 7.90–7.84 (m, 2H), 7.63 (dd, $J = 8.7, 1.9$ Hz, 2H), 7.59–7.53 (m, 1H), 7.49–7.44 (m, 2H), 7.42 (d, $J = 7.8$ Hz, 1H), 7.40–7.34 (m, 1H), 7.29–7.26 (m, 2H), 7.26–7.22 (m, 3H), 6.80 (d, $J = 8.3$ Hz, 1H), 6.65 (t, $J = 7.4$ Hz, 1H); **^{13}C NMR** (126 MHz, Chloroform-*d*) δ 196.52 (d, $J = 1.0$ Hz), 160.22 (d, $J = 3.6$ Hz), 137.57, 133.04 (d, $J = 9.2$ Hz), 132.63 (d, $J = 2.9$ Hz), 132.36 (d, $J = 2.8$ Hz), 132.24 (d, $J = 2.15$ Hz), 132.06 (d, $J = 9.1$ Hz), 130.97 (d, $J = 2.1$ Hz), 129.29 (d, $J =$

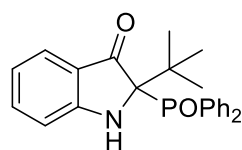
3.8 Hz), 128.71 (d, $J = 99.9$ Hz), 128.36 (d, $J = 98.4$ Hz), 128.28 (d, $J = 12.2$ Hz), 128.14 (d, $J = 11.9$ Hz), 124.58, 122.48 (d, $J = 3.2$ Hz), 121.08, 118.98, 112.29, 74.64 (d, $J = 61.0$ Hz); ^{31}P NMR (202 MHz, Chloroform- d) δ 29.27; HRMS (ESI) m/z calcd for: $\text{C}_{26}\text{H}_{19}\text{NO}_2\text{PBrNa}$ $[\text{M}+\text{Na}]^+$: 510.0229 (^{79}Br), found: 510.0229.

2-(diphenylphosphoryl)-2-(naphthalen-2-yl)indolin-3-one (3k). Yellow solid, 33.1



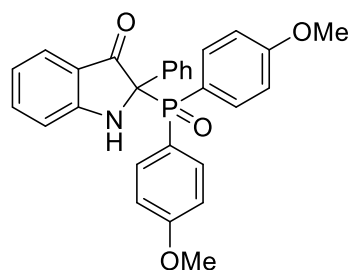
mg, 72% yield. ^1H NMR (500 MHz, Chloroform- d) δ 8.19 (s, 1H), 8.05–7.99 (m, 2H), 7.93–7.86 (m, 3H), 7.71 (d, $J = 8.1$ Hz, 1H), 7.59 (dd, $J = 23.7, 8.5$ Hz, 3H), 7.52–7.46 (m, 1H), 7.46–7.40 (m, 2H), 7.40–7.31 (m, 4H), 7.26–7.21 (m, 3H), 6.85 (d, $J = 8.3$ Hz, 1H), 6.68–6.63 (m, 1H); ^{13}C NMR (126 MHz, Chloroform- d) δ 196.79, 160.13 (d, $J = 3.9$ Hz), 137.45, 133.10 (d, $J = 9.2$ Hz), 132.81 (d, $J = 1.5$ Hz), 132.75 (d, $J = 2.1$ Hz), 132.48 (d, $J = 2.9$ Hz), 132.30 (d, $J = 2.7$ Hz), 132.22 (d, $J = 9.1$ Hz), 130.53 (d, $J = 2.2$ Hz), 128.83 (d, $J = 96.5$ Hz), 128.58, 128.35 (d, $J = 99.0$ Hz), 128.19 (d, $J = 5.3$ Hz), 128.09 (d, $J = 5.0$ Hz), 127.62 (d, $J = 1.2$ Hz), 127.31, 127.09 (d, $J = 5.1$ Hz), 126.28, 125.89, 125.05 (d, $J = 2.8$ Hz), 124.60, 121.33, 119.04, 112.41, 75.17 (d, $J = 61.9$ Hz); ^{31}P NMR (202 MHz, Chloroform- d) δ 29.92; HRMS (ESI) m/z calcd for: $\text{C}_{30}\text{H}_{22}\text{NO}_2\text{PNa}$ $[\text{M}+\text{Na}]^+$: 482.1280, found: 482.1281.

2-(tert-butyl)-2-(diphenylphosphoryl)indolin-3-one (3l). Yellow solid, 25.1 mg, 65%



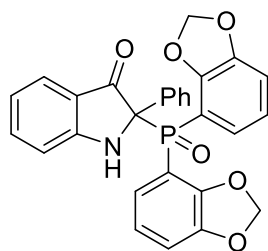
yield. ^1H NMR (500 MHz, Chloroform- d) δ 8.50–8.44 (m, 2H), 7.82–7.75 (m, 2H), 7.60–7.50 (m, 3H), 7.25 (d, $J = 7.8$ Hz, 1H), 7.20–7.12 (m, 2H), 7.11–7.06 (m, 2H), 6.67 (d, $J = 8.3$ Hz, 1H), 6.52 (t, $J = 7.3$ Hz, 1H), 6.44 (s, 1H), 1.10 (s, 9H); ^{13}C NMR (126 MHz, Chloroform- d) δ 200.00 (d, $J = 2.4$ Hz), 160.45 (d, $J = 3.2$ Hz), 136.83, 133.01 (d, $J = 9.1$ Hz), 132.66 (d, $J = 91.5$ Hz), 132.18 (d, $J = 3.0$ Hz), 131.90 (d, $J = 8.6$ Hz), 131.39 (d, $J = 2.8$ Hz), 130.52 (d, $J = 97.3$ Hz), 128.42 (d, $J = 12.0$ Hz), 127.48 (d, $J = 11.7$ Hz), 123.93, 122.81, 118.40, 111.43, 78.48 (d, $J = 60.5$ Hz), 40.72 (d, $J = 1.6$ Hz), 27.00 (d, $J = 4.2$ Hz); ^{31}P NMR (202 MHz, Chloroform- d) δ 22.82; HRMS (ESI) m/z calcd for: $\text{C}_{24}\text{H}_{24}\text{NO}_3\text{PNa}$ $[\text{M}+\text{Na}]^+$: 412.1437, found: 412.1437.

2-(bis(4-methoxyphenyl)phosphoryl)-2-phenylindolin-3-one (3m). Yellow solid,



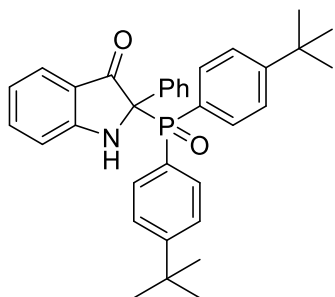
36.6 mg, 78% yield. ^1H NMR (500 MHz, Chloroform- d) δ 7.84 (t, $J = 9.3$ Hz, 2H), 7.72 (d, $J = 7.7$ Hz, 3H), 7.70 (s, 1H), 7.42 (dd, $J = 7.9, 1.2$ Hz, 1H), 7.29 (d, $J = 7.5$ Hz, 1H), 7.19 (d, $J = 6.8$ Hz, 4H), 6.90 (d, $J = 7.8$ Hz, 2H), 6.85 (d, $J = 8.2$ Hz, 1H), 6.71 (d, $J = 7.6$ Hz, 2H), 6.65 (t, $J = 7.4$ Hz, 1H), 3.82 (s, 3H), 3.71 (s, 3H); ^{13}C NMR (126 MHz, Chloroform- d) δ 197.06, 162.74, 162.50, 160.07, 137.27, 134.93 (d, $J = 10.2$ Hz), 134.05 (d, $J = 9.1$ Hz), 133.37, 127.93, 127.92, 127.50 (d, $J = 1.8$ Hz), 124.54, 124.09 (d, $J = 98.2$ Hz), 122.71 (d, $J = 99.3$ Hz), 121.49, 118.90, 113.70 (d, $J = 9.5$ Hz), 113.60 (d, $J = 9.6$ Hz), 112.34, 75.07 (d, $J = 41.2$ Hz), 55.30, 55.25; ^{31}P NMR (202 MHz, Chloroform- d) δ 30.32; HRMS (ESI) m/z calcd for: $\text{C}_{28}\text{H}_{25}\text{NO}_4\text{P}$ $[\text{M}+\text{H}]^+$: 470.1516, found: 470.1516.

2-(bis(benzo[d][1,3]dioxol-4-yl)phosphoryl)-2-phenylindolin-3-one (3n). Yellow



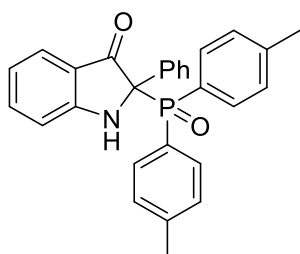
solid 27.2 mg, 55% yield. $^1\text{H NMR}$ (500 MHz, Chloroform-*d*) δ 7.80–7.73 (m, 2H), 7.48 (dd, $J = 11.6, 8.3$ Hz, 1H), 7.46–7.40 (m, 2H), 7.32 (q, $J = 6.4, 5.6$ Hz, 2H), 7.25–7.17 (m, 4H), 6.88 (d, $J = 8.3$ Hz, 1H), 6.82 (dd, $J = 8.0, 2.3$ Hz, 1H), 6.69 (t, $J = 7.4$ Hz, 1H), 6.66–6.61 (m, 1H), 6.03–5.97 (m, 2H), 5.91–5.84 (m, 2H), $^{13}\text{C NMR}$ (126 MHz, Chloroform-*d*) δ 196.90, 160.04 (d, $J = 3.3$ Hz), 151.17 (d, $J = 2.6$ Hz), 150.94 (d, $J = 2.7$ Hz), 147.65 (d, $J = 9.6$ Hz), 147.50 (d, $J = 8.9$ Hz), 137.44, 133.24, 128.83 (d, $J = 9.8$ Hz), 128.11 (d, $J = 1.9$ Hz), 128.08, 127.84 (d, $J = 10.1$ Hz), 127.48 (d, $J = 3.8$ Hz), 124.64, 121.96 (d, $J = 98.1$ Hz), 121.41, 121.13 (d, $J = 99.0$ Hz), 119.05, 112.36 (d, $J = 12.2$ Hz), 112.34, 111.67 (d, $J = 12.2$ Hz), 108.46 (d, $J = 15.4$ Hz), 108.29 (d, $J = 15.0$ Hz), 101.58, 101.50, 75.16 (d, $J = 62.6$ Hz); $^{31}\text{P NMR}$ (202 MHz, Chloroform-*d*) δ 30.32; **HRMS** (ESI) m/z calcd for: $\text{C}_{28}\text{H}_{21}\text{NO}_6\text{P}$ $[\text{M}+\text{H}]^+$: 498.1101, found: 498.1101.

2-(bis(4-(tert-butyl)phenyl)phosphoryl)-2-phenylindolin-3-one (3o). Yellow solid



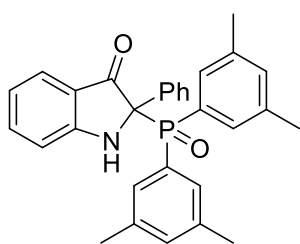
37.9 mg, 73% yield. $^1\text{H NMR}$ (400 MHz, Chloroform-*d*) δ 7.89 (t, $J = 9.3$ Hz, 2H), 7.76 (m, 4H), 7.63 (s, 1H), 7.42 (d, $J = 7.4$ Hz, 2H), 7.37 (d, $J = 7.7$ Hz, 1H), 7.18 (dd, $J = 21.4, 7.1$ Hz, 6H), 6.81 (d, $J = 8.1$ Hz, 1H), 6.59 (t, $J = 7.3$ Hz, 1H), 1.31 (s, 9H), 1.18 (s, 9H); $^{13}\text{C NMR}$ (101 MHz, Chloroform-*d*) δ 196.92, 160.24, 157.11 – 154.58 (m), 136.99, 133.43 – 133.37 (m), 132.97 (d, $J = 9.5$ Hz), 132.17 (d, $J = 9.4$ Hz), 127.88 (d, $J = 1.6$ Hz), 127.83 (d, $J = 1.9$ Hz), 127.62 (d, $J = 3.7$ Hz), 125.73 (d, $J = 99.5$ Hz), 125.44 (d, $J = 101.0$ Hz), 125.10 (d, $J = 12.4$ Hz), 124.93 (d, $J = 12.0$ Hz), 124.49, 121.46, 118.61, 112.41, 75.19 (d, $J = 60.8$ Hz), 35.02, 34.85, 31.09, 30.92; $^{31}\text{P NMR}$ (162 MHz, Chloroform-*d*) δ 30.03; **HRMS** (ESI) m/z calcd for: $\text{C}_{34}\text{H}_{36}\text{NO}_2\text{PBr}_2$ $[\text{M}+\text{H}]^+$: 522.2556, found: 522.2551.

2-(di-*p*-tolylphosphoryl)-2-phenylindolin-3-one (3p). Yellow solid, 35.1 mg, 80%



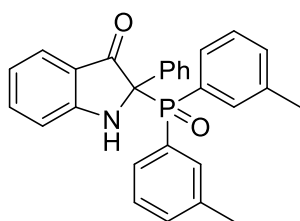
yield. $^1\text{H NMR}$ (500 MHz, Chloroform-*d*) δ 7.83 (dd, $J = 11.0, 7.9$ Hz, 2H), 7.76 (d, $J = 7.6$ Hz, 2H), 7.71 (dd, $J = 11.0, 7.9$ Hz, 2H), 7.57 (s, 1H), 7.44 (d, $J = 7.8$ Hz, 1H), 7.29 (dd, $J = 15.3, 1.3$ Hz, 1H), 7.26–7.15 (m, 5H), 7.03 (dd, $J = 8.0, 2.7$ Hz, 2H), 6.85 (d, $J = 8.3$ Hz, 1H), 6.66 (t, $J = 7.4$ Hz, 1H), 2.39 (s, 3H), 2.27 (s, 3H); $^{13}\text{C NMR}$ (126 MHz, Chloroform-*d*) δ 196.94, 160.13 (d, $J = 3.4$ Hz), 142.81 (d, $J = 2.5$ Hz), 142.57 (d, $J = 2.4$ Hz), 137.21, 133.32, 133.02 (d, $J = 9.4$ Hz), 132.13 (d, $J = 9.3$ Hz), 128.85 (d, $J = 7.9$ Hz), 128.75 (d, $J = 7.7$ Hz), 127.90, 127.88, 127.56 (d, $J = 3.5$ Hz), 125.86 (d, $J = 99.9$ Hz), 125.44 (d, $J = 100.8$ Hz), 124.53, 121.38, 118.70, 112.32, 75.03 (d, $J = 61.1$ Hz), 21.67, 21.47; $^{31}\text{P NMR}$ (202 MHz, Chloroform-*d*) δ 30.35; **HRMS** (ESI) m/z calcd for: $\text{C}_{28}\text{H}_{25}\text{NO}_2\text{P}$ $[\text{M}+\text{H}]^+$: 438.1617, found: 438.1623.

2-((2,4-dimethylphenyl)(3,5-dimethylphenyl)phosphoryl)-2-phenylindolin-3-one



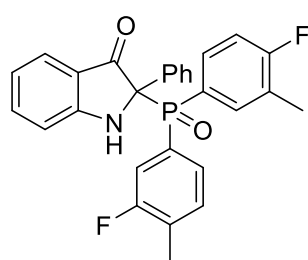
(3q). Yellow solid, 37.0 mg, 80% yield. $^1\text{H NMR}$ (400 MHz, Chloroform-*d*) δ 7.78–7.67 (m, 2H), 7.45 (d, $J = 9.7$ Hz, 3H), 7.38 (d, $J = 11.5$ Hz, 2H), 7.27 (d, $J = 9.4$ Hz, 3H), 7.11 (s, 2H), 7.04 (s, 1H), 6.95 (s, 1H), 6.83 (d, $J = 8.0$ Hz, 1H), 6.66 (t, $J = 7.0$ Hz, 1H), 2.28 (s, 6H), 2.13 (s, 6H); $^{13}\text{C NMR}$ (101 MHz, Chloroform-*d*) δ 196.59, 159.87, 137.72 (d, $J = 8.3$ Hz), 137.59 (d, $J = 8.8$ Hz), 137.06, 134.09 (d, $J = 2.4$ Hz), 133.83 (d, $J = 2.5$ Hz), 133.24, 130.65 (d, $J = 9.1$ Hz), 129.83 (d, $J = 8.9$ Hz), 128.47 (d, $J = 94.8$ Hz), 127.91 (d, $J = 97.8$ Hz), 127.92 (d, $J = 1.8$ Hz), 127.84–127.76 (m), 127.61 (d, $J = 3.4$ Hz), 124.40, 121.92, 119.02, 112.28, 74.85 (d, $J = 59.8$ Hz), 21.14, 21.10; $^{31}\text{P NMR}$ (162 MHz, Chloroform-*d*) δ 31.24; **HRMS** (ESI) m/z calcd for: $\text{C}_{30}\text{H}_{29}\text{NO}_2\text{P}$ $[\text{M}+\text{H}]^+$: 466.1930, found: 466.1932.

2-(di-*m*-tolylphosphoryl)-2-phenylindolin-3-one (3r). Yellow solid, 34.8 mg, 80%



yield. $^1\text{H NMR}$ (500 MHz, Chloroform-*d*) δ 7.78 (dd, $J = 8.3$, 2.9 Hz, 1H), 7.76–7.70 (m, 2H), 7.66 (dd, $J = 11.6$, 5.6 Hz, 2H), 7.62–7.53 (m, 1H), 7.43 (d, $J = 7.7$ Hz, 1H), 7.34–7.29 (m, 3H), 7.28 (s, 1H), 7.19 (q, $J = 7.9$, 6.5 Hz, 3H), 7.16–7.09 (m, 2H), 6.83 (d, $J = 8.3$ Hz, 1H), 6.65 (t, $J = 7.4$ Hz, 1H), 2.31 (s, 3H), 2.16 (s, 3H); $^{13}\text{C NMR}$ (126 MHz, Chloroform-*d*) δ 196.72, 160.00 (d, $J = 3.5$ Hz), 137.91 (d, $J = 1.7$ Hz), 137.82 (d, $J = 1.6$ Hz), 137.19, 133.59 (d, $J = 8.6$ Hz), 133.17 (d, $J = 2.0$ Hz), 133.13 (d, $J = 3.0$ Hz), 132.91 (d, $J = 2.9$ Hz), 132.79 (d, $J = 8.6$ Hz), 130.06 (d, $J = 9.8$ Hz), 129.16 (d, $J = 9.7$ Hz), 128.73 (d, $J = 95.9$ Hz), 128.25 (d, $J = 96.4$ Hz), 127.95 (d, $J = 2.2$ Hz), 127.87 (d, $J = 2.0$ Hz), 127.84, 127.57 (d, $J = 3.9$ Hz), 124.48, 121.62, 118.92, 112.27, 74.92 (d, $J = 60.8$ Hz), 21.40, 21.22; $^{31}\text{P NMR}$ (202 MHz, Chloroform-*d*) δ 30.31; **HRMS** (ESI) m/z calcd for: $\text{C}_{28}\text{H}_{25}\text{NO}_2\text{P}$ $[\text{M}+\text{H}]^+$: 438.1617, found: 438.1621.

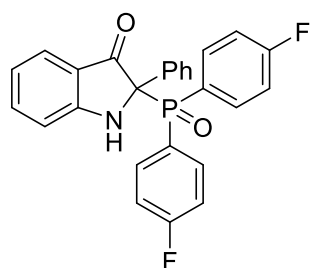
2-((4-fluoro-3-methylphenyl)(3-fluoro-4-methylphenyl)phosphoryl)-2-



phenylindolin-3-one (3s). Yellow solid 36.0 mg, 76% yield. $^1\text{H NMR}$ (500 MHz, Chloroform-*d*) δ 7.84 (s, 1H), 7.78 (d, $J = 7.7$ Hz, 2H), 7.70–7.59 (m, 2H), 7.52–7.43 (m, 3H), 7.30 (t, $J = 7.3$ Hz, 1H), 7.25–7.17 (m, 4H), 7.05 (q, $J = 7.3$ Hz, 1H), 6.86 (d, $J = 8.3$ Hz, 1H), 6.67 (t, $J = 7.4$ Hz, 1H), 2.30 (s, 3H), 2.18 (s, 3H); $^{13}\text{C NMR}$ (126 MHz, Chloroform-*d*) δ 196.73, 161.71 (dd, $J = 17.3$, 13.3 Hz), 160.25 (d, $J = 4.1$ Hz), 159.74 (dd, $J = 17.3$, 14.0 Hz), 137.59, 132.75 (d, $J = 2.0$ Hz), 131.57 (t, $J = 4.9$ Hz), 131.46 (t, $J = 4.7$ Hz), 130.21 (dd, $J = 17.1$, 2.5 Hz), 130.00 (dd, $J = 17.2$, 2.6 Hz), 128.58 (d, $J = 3.6$ Hz), 128.51 (d, $J = 3.6$ Hz), 128.28 (dd, $J = 97.8$, 6.0 Hz), 128.24 (d, $J = 2.1$ Hz), 128.10 (d, $J = 1.9$ Hz), 127.78 (dd, $J = 100.6$, 6.1 Hz), 127.50 (d, $J = 3.8$ Hz), 124.60, 121.09, 119.46 (dd, $J = 24.5$, 10.4 Hz), 118.95, 118.72 (dd, $J = 24.1$, 10.1 Hz), 112.36, 74.96 (d, $J = 64.9$ Hz), 14.84 (d, $J = 3.6$ Hz), 14.63 (d, $J = 3.4$ Hz); $^{31}\text{P NMR}$ (202 MHz, Chloroform-*d*) δ 27.70; $^{19}\text{F NMR}$ (471 MHz, Chloroform-*d*) δ -

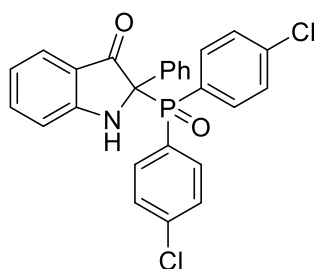
115.64, -115.72; **HRMS** (ESI) m/z calcd for: $C_{28}H_{23}NO_2PF_2 [M+H]^+$: 474.1429, found: 474.1428.

2-(bis(4-fluorophenyl)phosphoryl)-2-phenylindolin-3-one (3t). Yellow solid 31.5



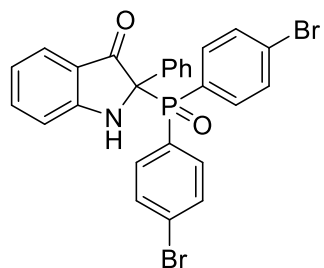
mg, 71% yield. 1H NMR (500 MHz, Chloroform-*d*) δ 8.09 – 8.00 (m, 2H), 7.91 – 7.81 (m, 2H), 7.77 (d, $J = 7.2$ Hz, 3H), 7.45 (d, $J = 7.8$ Hz, 1H), 7.33 (t, $J = 7.7$ Hz, 1H), 7.27 – 7.18 (m, 2H), 7.17 – 7.10 (m, 2H), 6.94 (t, $J = 8.4$ Hz, 2H), 6.86 (d, $J = 8.3$ Hz, 1H), 6.70 (t, $J = 7.4$ Hz, 1H); ^{13}C NMR (126 MHz, Chloroform-*d*) δ 197.00 (d, $J = 1.2$ Hz), 166.30 (dd, $J = 30.4, 3.4$ Hz), 164.28 (dd, $J = 30.4, 3.4$ Hz), 160.23 (d, $J = 4.0$ Hz), 137.74, 135.61 (dd, $J = 10.6, 9.0$ Hz), 134.69 (dd, $J = 10.4, 9.1$ Hz), 132.80 (d, $J = 2.1$ Hz), 128.28 (d, $J = 2.4$ Hz), 128.13 (d, $J = 2.0$ Hz), 127.48 (d, $J = 4.1$ Hz), 124.77 (dd, $J = 100.0, 3.3$ Hz), 124.58, 124.34 (dd, $J = 102.5, 3.3$ Hz), 121.07, 119.09, 115.84 – 115.61 (m), 115.60 – 115.37 (m), 112.24, 75.05 (d, $J = 64.1$ Hz); ^{31}P NMR (202 MHz, Chloroform-*d*) δ 28.45; ^{19}F NMR (471 MHz, Chloroform-*d*) δ -105.29, -105.52; **HRMS** (ESI) m/z calcd for: $C_{26}H_{18}NO_2PBr_2 [M+H]^+$: 446.1116, found: 446.1118.

2-(bis(4-chlorophenyl)phosphoryl)-2-phenylindolin-3-one (3u). Yellow solid, 34.2



mg, 72% yield. 1H NMR (400 MHz, Chloroform-*d*) δ 8.22 (s, 1H), 7.99–7.90 (m, 2H), 7.76 (t, $J = 6.6$ Hz, 4H), 7.48–7.38 (m, 3H), 7.36–7.28 (m, 1H), 7.27–7.20 (m, 3H), 7.17 (d, $J = 7.6$ Hz, 2H), 6.85 (d, $J = 8.3$ Hz, 1H), 6.68 (t, $J = 7.4$ Hz, 1H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 196.90, 160.42 (d, $J = 4.2$ Hz), 139.36 (d, $J = 3.5$ Hz), 139.17 (d, $J = 3.5$ Hz), 137.88, 134.41 (d, $J = 10.1$ Hz), 133.46 (d, $J = 10.0$ Hz), 132.63 (d, $J = 1.7$ Hz), 128.66 (d, $J = 8.5$ Hz), 128.54 (d, $J = 8.3$ Hz), 128.40 (d, $J = 2.0$ Hz), 128.19 (d, $J = 1.6$ Hz), 127.56 (d, $J = 4.0$ Hz), 127.31 (d, $J = 98.4$ Hz), 126.98 (d, $J = 100.5$ Hz), 124.64, 120.91, 119.08, 112.34, 75.05 (d, $J = 64.9$ Hz); ^{31}P NMR (162 MHz, Chloroform-*d*) δ 28.72; **HRMS** (ESI) m/z calcd for: $C_{26}H_{19}NO_2PCl_2 [M+H]^+$: 478.0525, found: 478.0521.

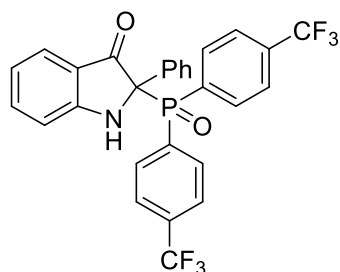
2-(bis(4-bromophenyl)phosphoryl)-2-phenylindolin-3-one (3v). Yellow solid 44.1



mg, 78% yield. 1H NMR (500 MHz, Chloroform-*d*) δ 7.83 (dd, $J = 10.7, 8.5$ Hz, 2H), 7.73 (d, $J = 7.8$ Hz, 2H), 7.70 (s, 1H), 7.65 (dd, $J = 10.7, 8.5$ Hz, 2H), 7.56 (dd, $J = 8.4, 2.3$ Hz, 2H), 7.44 (d, $J = 7.7$ Hz, 1H), 7.36 (d, $J = 6.5$ Hz, 2H), 7.35–7.29 (m, 1H), 7.24 (d, $J = 7.0$ Hz, 1H), 7.20 (t, $J = 7.4$ Hz, 2H), 6.84 (d, $J = 8.3$ Hz, 1H), 6.70 (t, $J = 7.4$ Hz, 1H); ^{13}C NMR (126 MHz, Chloroform-*d*) δ 196.73, 160.16 (d, $J = 3.8$ Hz), 137.89, 134.43 (d, $J = 10.0$ Hz), 133.48 (d, $J = 9.8$ Hz), 132.57 (d, $J = 1.8$ Hz), 131.58 (d, $J = 12.6$ Hz), 131.47 (d, $J = 12.1$ Hz), 128.44 (d, $J = 2.5$ Hz), 128.24 (d, $J = 1.6$ Hz), 128.10 (d, $J = 3.5$ Hz), 127.86 (d, $J = 3.5$ Hz), 127.67 (d, $J = 99.1$ Hz),

127.46 (d, $J = 4.0$ Hz), 127.32 (d, $J = 99.7$ Hz), 124.67, 121.04, 119.29, 112.30, 74.85 (d, $J = 64.4$ Hz); **^{31}P NMR** (202 MHz, Chloroform-*d*) δ 28.98; **HRMS** (ESI) m/z calcd for: $\text{C}_{26}\text{H}_{19}\text{NO}_2\text{PBr}_2$ [$\text{M}+\text{H}$] $^+$: 565.9515 (^{79}Br), found: 565.9520.

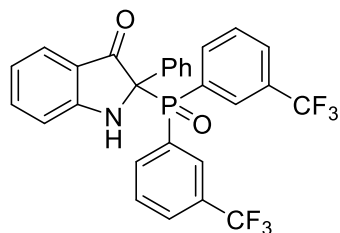
2-(bis(4-(trifluoromethyl)phenyl)phosphoryl)-2-phenylindolin-3-one (3w). Yellow



solid 40.4 mg, 74% yield. **^1H NMR** (500 MHz, Chloroform-*d*) δ 8.17 (dd, $J = 10.7, 8.5$ Hz, 2H), 7.94 (dd, $J = 10.6, 8.5$ Hz, 2H), 7.77–7.72 (m, 2H), 7.70 (dd, $J = 8.3, 2.1$ Hz, 3H), 7.49 (d, $J = 6.4$ Hz, 2H), 7.44 (d, $J = 7.8$ Hz, 1H), 7.37–7.30 (m, 1H), 7.25–7.18 (m, 2H), 6.85 (d, $J = 8.3$ Hz, 1H), 6.71 (t, $J = 7.4$ Hz, 1H); **^{13}C NMR** (126 MHz, Chloroform-*d*) δ 196.52 (d, $J = 1.6$ Hz), 160.09 (d, $J = 4.3$

Hz), 138.09, 134.75–134.34 (m), 133.82–133.22 (m), 133.53 (d, $J = 9.7$ Hz), 132.75 (d, $J = 94.4$ Hz), 132.61 (d, $J = 9.5$ Hz), 132.48 (d, $J = 96.4$ Hz), 132.25 (d, $J = 2.1$ Hz), 128.70 (d, $J = 2.4$ Hz), 128.39 (d, $J = 2.2$ Hz), 127.60 – 127.29 (m), 125.32 – 124.83 (m), 124.73, 123.34 (qd, $J = 272.3$ Hz), 121.00, 119.57, 112.29, 74.84 (d, $J = 64.9$ Hz); **^{31}P NMR** (202 MHz, Chloroform-*d*) δ 27.66; **^{19}F NMR** (471 MHz, Chloroform-*d*) δ -63.33, -63.54; **HRMS** (ESI) m/z calcd for: $\text{C}_{28}\text{H}_{19}\text{NO}_2\text{PF}_6$ [$\text{M}+\text{H}$] $^+$: 546.1052, found: 546.1048.

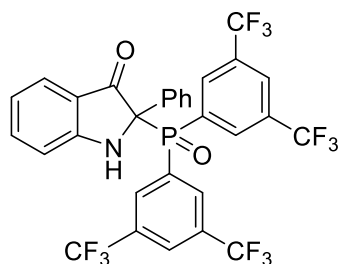
2-(bis(3-(trifluoromethyl)phenyl)phosphoryl)-2-phenylindolin-3-one (3x). Yellow



solid 42.5 mg, 78% yield. **^1H NMR** (500 MHz, Chloroform-*d*) δ 8.29–8.22 (m, 2H), 8.16 (d, $J = 11.3$ Hz, 1H), 7.95–7.88 (m, 1H), 7.80 (s, 1H), 7.76–7.70 (m, 2H), 7.66–7.57 (m, 2H), 7.4 – 7.38 (m, 2H), 7.35 (s, 1H), 7.35–7.31 (m, 1H), 7.25–7.17 (m, 3H), 6.85 (d, $J = 8.3$ Hz, 1H), 6.72–6.66 (m, 1H); **^{13}C NMR** (126 MHz, Chloroform-*d*) δ

196.47 (d, $J = 1.3$ Hz), 159.90 (d, $J = 4.1$ Hz), 138.03, 136.13 (d, $J = 9.6$ Hz), 135.13 (d, $J = 9.4$ Hz), 132.09 (d, $J = 2.1$ Hz), 130.11 (dd, $J = 9.8, 3.8$ Hz), 131.02 (d, $J = 12.4$ Hz), 130.76 (d, $J = 12.4$ Hz), 129.84 (d, $J = 96.0$ Hz), 129.41 – 129.30 (m), 129.30 (d, $J = 96.0$ Hz), 129.25 – 129.17 (m), 129.08 (dd, $J = 9.5, 3.9$ Hz), 128.87 (d, $J = 5.6$ Hz), 128.78 (d, $J = 5.2$ Hz), 128.64 (d, $J = 2.5$ Hz), 128.37 (d, $J = 2.1$ Hz), 127.30 (d, $J = 4.3$ Hz), 123.42 (qd, $J = 273.2, 27.2$ Hz), 124.66, 121.15, 119.64, 112.28, 74.78 (d, $J = 64.5$ Hz); **^{31}P NMR** (202 MHz, Chloroform-*d*) δ 27.51; **^{19}F NMR** (471 MHz, Chloroform-*d*) δ -62.94, -62.98; **HRMS** (ESI) m/z calcd for: $\text{C}_{28}\text{H}_{19}\text{NO}_2\text{PF}_6$ [$\text{M}+\text{H}$] $^+$: 546.1052, found: 546.1050.

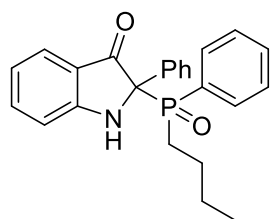
2-(bis(3,5-bis(trifluoromethyl)phenyl)phosphoryl)-2-phenylindolin-3-one (3y).



Yellow solid 55.4 mg, 81% yield. **^1H NMR** (500 MHz, Chloroform-*d*) δ 8.51 (d, $J = 11.0$ Hz, 2H), 8.29 (d, $J = 10.9$ Hz, 2H), 8.07 (s, 1H), 7.90 (s, 2H), 7.79–7.72 (m, 2H), 7.48 (d, $J = 7.8$ Hz, 1H), 7.43–7.34 (m, 1H), 7.30–7.27 (m, 1H), 7.20 (t, $J = 7.7$ Hz, 2H), 6.85 (d, $J = 8.3$ Hz, 1H), 6.75 (t, $J = 7.4$ Hz, 1H); **^{13}C NMR** (126 MHz, Chloroform-*d*) δ

196.19 (d, $J = 2.0$ Hz), 160.08 (d, $J = 4.8$ Hz), 138.63, 133.21 (dd, $J = 9.5, 3.2$ Hz), 132.73 – 131.59 (m), 131.15(d, $J = 95.2$ Hz), 131.07 (d, $J = 2.3$ Hz), 130.60 (d, $J = 98.0$ Hz), 129.22 (d, $J = 2.6$ Hz), 128.70 (d, $J = 2.2$ Hz), 127.17 (d, $J = 4.5$ Hz), 126.80 – 126.38 (m), 124.83, 122.53 (qd, $J = 272.9, 33.2$ Hz), 120.82, 120.17, 112.33, 74.79 (d, $J = 68.0$ Hz); ^{31}P NMR (202 MHz, Chloroform- d) δ 27.61; ^{19}F NMR (471 MHz, Chloroform- d) δ -63.14, -63.20; HRMS (ESI) m/z calcd for: $\text{C}_{30}\text{H}_{17}\text{NO}_2\text{PF}_{12}$ $[\text{M}+\text{H}]^+$: 682.0800, found: 682.0798.

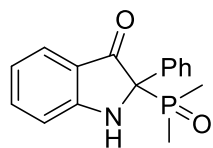
2-phenyl-2-(phenyl(propyl)phosphoryl)indolin-3-one (3z). Yellow solid 20.7 mg, 53%



yield, mixture of diastereomer (1:1 dr). ^1H NMR (500 MHz, Chloroform- d) δ 8.02 (d, $J = 7.8$ Hz, 2H), 7.70–7.64 (m, 2H), 7.62 (d, $J = 7.7$ Hz, 1H), 7.57 (d, $J = 8.1$ Hz, 2H), 7.53 (d, $J = 8.4$ Hz, 1H), 7.48 (q, $J = 7.2$ Hz, 4H), 7.42 (t, $J = 7.5$ Hz, 2H), 7.40–7.34 (m, 4H), 7.29 (s, 2H), 7.22–7.18 (m, 2H), 7.16 (t, 2H), 7.11 (t, $J = 7.4$ Hz, 2H), 7.07 (d, $J = 8.2$ Hz, 1H), 6.84 (t, $J = 7.4$

Hz, 1H), 6.71 (d, $J = 8.3$ Hz, 1H), 6.54 (t, $J = 7.4$ Hz, 1H), 2.07–1.88 (m, 4H), 1.40–1.29 (m, 4H), 1.29–1.20 (m, 4H), 0.80 (t, $J = 7.1$ Hz, 3H), 0.73 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (126 MHz, Chloroform- d) δ 196.76, 196.71, 160.18 (d, $J = 3.1$ Hz), 159.97 (d, $J = 2.9$ Hz), 137.69, 137.10, 132.92 (d, $J = 1.9$ Hz), 132.77 (d, $J = 1.8$ Hz), 132.51 (d, $J = 8.7$ Hz), 132.39 (d, $J = 1.9$ Hz), 132.12 (d, $J = 8.8$ Hz), 132.07 (d, $J = 1.4$ Hz), 128.52 (d, $J = 1.4$ Hz), 128.30 (d, $J = 1.8$ Hz), 128.09 (d, $J = 82.6$ Hz), 128.03 (d, $J = 11.9$ Hz), 127.89 (d, $J = 11.4$ Hz), 127.74 (d, $J = 1.8$ Hz), 127.68 (d, $J = 1.1$ Hz), 127.37 (d, $J = 80.5$ Hz), 127.35 (d, $J = 3.2$ Hz), 126.75 (d, $J = 2.8$ Hz), 124.82, 124.24, 121.32, 121.02, 119.17, 118.61, 112.80, 112.11, 73.99 (d, $J = 65.7$ Hz), 73.92 (d, $J = 64.8$ Hz), 24.08 (d, $J = 8.5$ Hz), 23.96 (d, $J = 7.5$ Hz), 23.95 (d, $J = 66.2$ Hz), 23.40 (d, $J = 4.2$ Hz), 22.84 (d, $J = 4.2$ Hz), 22.71 (d, $J = 67.6$ Hz), 13.57, 13.53; ^{31}P NMR (202 MHz, Chloroform- d) δ 42.95, 41.86; HRMS (ESI) m/z calcd for: $\text{C}_{24}\text{H}_{25}\text{NO}_2\text{P}$ $[\text{M}+\text{H}]^+$: 390.1617, found: 390.1616.

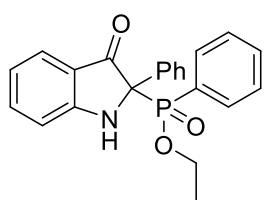
2-(dimethylphosphoryl)-2-phenylindolin-3-one (3aa). Yellow solid 23.1 mg, 81%



yield. ^1H NMR (500 MHz, DMSO- d_6) δ 8.84 (s, 1H), 7.85–7.80 (m, 2H), 7.5–7.51 (m, 1H), 7.48 (d, $J = 7.8$ Hz, 1H), 7.42 (t, $J = 7.6$ Hz, 2H), 7.38–7.33 (m, 1H), 7.09 (d, $J = 8.3$ Hz, 1H), 6.82–6.75 (m, 1H), 1.35 (d, $J = 13.0$ Hz, 3H), 1.26 (d, $J = 12.5$ Hz, 3H); ^{13}C NMR (126

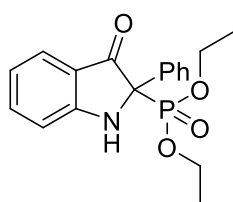
MHz, DMSO- d_6) δ 195.99, 160.89 (d, $J = 4.0$ Hz), 138.25, 133.79 (d, $J = 1.1$ Hz), 128.60 (d, $J = 1.8$ Hz), 128.36 (d, $J = 2.1$ Hz), 126.83 (d, $J = 3.5$ Hz), 124.63, 119.73, 118.68, 112.73, 73.75 (d, $J = 60.8$ Hz), 12.90 (d, $J = 90.4$ Hz), 12.37 (d, $J = 89.4$ Hz); ^{31}P NMR (162 MHz, Chloroform- d) δ 48.58; HRMS (ESI) m/z calcd for: $\text{C}_{16}\text{H}_{16}\text{NO}_2\text{P}$ $[\text{M}+\text{H}]^+$: 286.0991, found: 286.0993.

ethyl (3-oxo-2-phenylindolin-2-yl)(phenyl)phosphinate (3ab). Yellow solid 24.6 mg,



65% yield, mixture of diastereomer (1:1 dr). $^1\text{H NMR}$ (500 MHz, Chloroform-*d*) δ 8.01 (d, $J = 7.8$ Hz, 2H), 7.71 (dd, $J = 10.9, 7.9$ Hz, 2H), 7.59 (q, $J = 5.6, 4.2$ Hz, 6H), 7.53 (t, $J = 7.5$ Hz, 1H), 7.45–7.40 (m, 1H), 7.40–7.31 (m, 7H), 7.25–7.15 (m, 6H), 6.98 (d, $J = 8.2$ Hz, 1H), 6.83–6.77 (m, 3H), 6.59 (t, $J = 7.3$ Hz, 1H), 4.07–3.98 (m, 4H), 1.29 (t, $J = 7.0$ Hz, 3H), 1.08 (t, $J = 7.0$ Hz, 3H); $^{13}\text{C NMR}$ (126 MHz, Chloroform-*d*) δ 195.49, 195.08, 159.84 (d, $J = 3.3$ Hz), 159.63 (d, $J = 3.9$ Hz), 137.09, 136.97, 133.66 (d, $J = 9.4$ Hz), 133.18 (d, $J = 9.4$ Hz), 133.02 (d, $J = 2.7$ Hz), 132.87, 132.83, 132.73 (d, $J = 2.6$ Hz), 128.21 (d, $J = 2.1$ Hz), 128.15 (d, $J = 2.4$ Hz), 128.01 (d, $J = 1.8$ Hz), 127.93 (d, $J = 3.1$ Hz), 127.90 (d, $J = 1.8$ Hz), 127.88 (d, $J = 2.3$ Hz), 127.43 (d, $J = 85.4$ Hz), 127.31 (d, $J = 3.7$ Hz), 126.90 (d, $J = 4.2$ Hz), 126.41 (d, $J = 83.5$ Hz), 124.90, 124.59, 121.24, 120.87, 119.24, 118.93, 112.44, 112.07, 73.49 (d, $J = 94.8$ Hz), 73.29 (d, $J = 91.8$ Hz), 63.05 (d, $J = 7.6$ Hz), 62.65 (d, $J = 7.3$ Hz), 16.44 (d, $J = 5.4$ Hz), 16.30 (d, $J = 5.7$ Hz); $^{31}\text{P NMR}$ (202 MHz, Chloroform-*d*) δ 36.21, 30.45; **HRMS** (ESI) m/z calcd for: $\text{C}_{22}\text{H}_{21}\text{NO}_3\text{P}$ $[\text{M}+\text{H}]^+$: 378.1254, found: 378.1255.

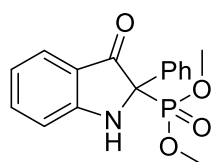
diethyl (3-oxo-2-phenylindolin-2-yl)phosphonate(3ac). Yellow solid 22.5 mg, 65%



yield. $^1\text{H NMR}$ (500 MHz, Chloroform-*d*) δ 8.00–7.87 (m, 2H), 7.62 (d, $J = 7.7$ Hz, 1H), 7.50–7.42 (m, 1H), 7.37 (t, $J = 7.5$ Hz, 2H), 7.34–7.29 (m, 1H), 6.99 (d, $J = 8.2$ Hz, 1H), 6.84 (t, $J = 7.4$ Hz, 1H), 6.17 (s, 1H), 4.13–4.02 (m, 2H), 4.01–3.86 (m, 2H), 1.19 (t, $J = 7.1$ Hz, 3H), 1.07 (t, $J = 7.0$ Hz, 3H); $^{13}\text{C NMR}$ (126 MHz, Chloroform-*d*) δ 194.67 (d, $J = 4.0$ Hz), 159.80 (d, $J = 4.5$

Hz), 137.29, 133.44 (d, $J = 2.9$ Hz), 128.34 (d, $J = 2.6$ Hz), 128.26 (d, $J = 3.0$ Hz), 126.80 (d, $J = 4.9$ Hz), 125.19, 120.62, 119.56, 112.53, 71.52 (d, $J = 144.6$ Hz), 64.59 (d, $J = 7.7$ Hz), 64.33 (d, $J = 7.3$ Hz), 16.23 (d, $J = 5.9$ Hz), 16.18 (d, $J = 5.9$ Hz); $^{31}\text{P NMR}$ (202 MHz, Chloroform-*d*) δ 15.86; **HRMS** (ESI) m/z calcd for: $\text{C}_{18}\text{H}_{20}\text{NO}_4\text{P}$ $[\text{M}+\text{H}]^+$: 346.1203, found: 346.1204.

dimethyl (3-oxo-2-phenylindolin-2-yl)phosphonate(3ad). Yellow solid 20.3 mg, 64%

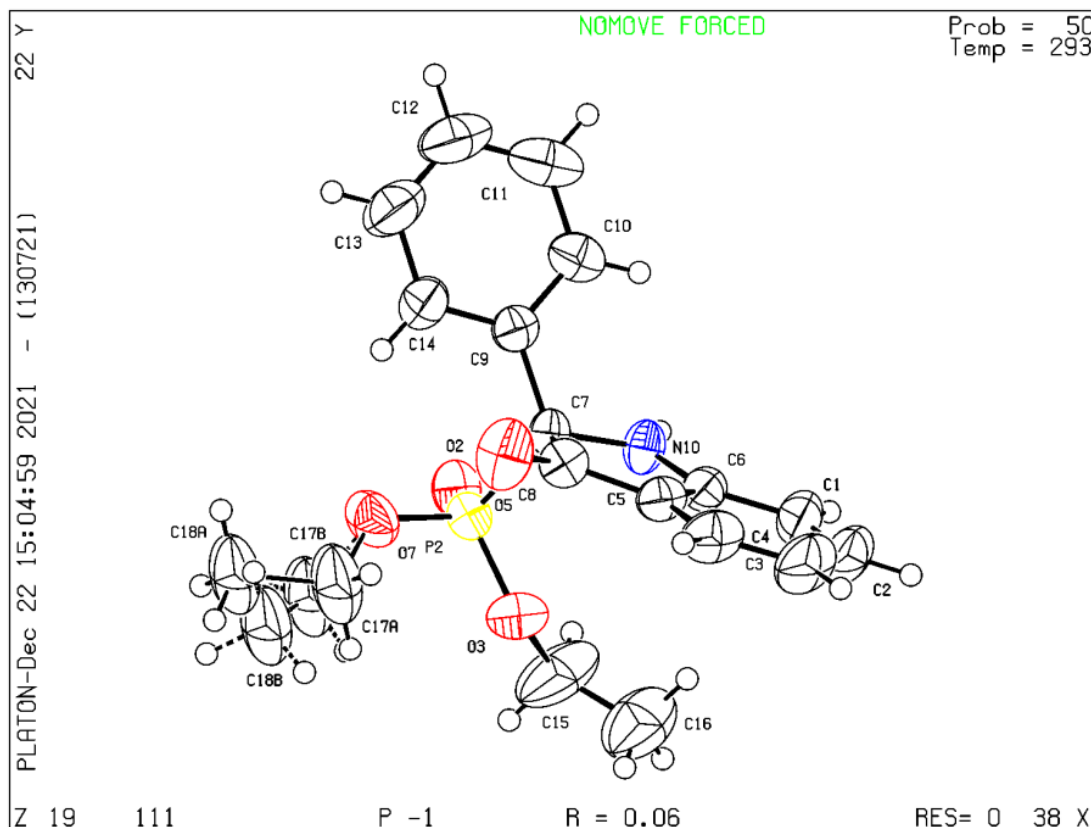


yield. $^1\text{H NMR}$ (500 MHz, Chloroform-*d*) δ 7.95–7.89 (m, 2H), 7.67–7.62 (m, 1H), 7.51–7.46 (m, 1H), 7.39 (t, $J = 7.6$ Hz, 2H), 7.36–7.31 (m, 1H), 7.00 (d, $J = 8.2$ Hz, 1H), 6.87 (t, $J = 7.4$ Hz, 1H), 3.66 (dd, $J = 17.4, 10.7$ Hz, 6H); $^{13}\text{C NMR}$ (126 MHz, Chloroform-*d*) δ 194.53 (d, $J = 3.8$ Hz), 159.69 (d, $J = 4.9$ Hz), 137.50, 133.18

(d, $J = 3.1$ Hz), 128.53 (d, $J = 2.7$ Hz), 128.46 (d, $J = 3.0$ Hz), 126.67 (d, $J = 4.9$ Hz), 125.33, 120.35 (d, $J = 1.9$ Hz), 119.75, 112.60, 71.27 (d, $J = 146.7$ Hz), 54.96 (d, $J = 7.5$ Hz), 54.71 (d, $J = 7.3$ Hz), 50.85 (s, MeOH); $^{31}\text{P NMR}$ (202 MHz, Chloroform-*d*) δ 18.46; **HRMS** (ESI) m/z calcd for: $\text{C}_{16}\text{H}_{17}\text{NO}_4\text{P}$ $[\text{M}+\text{H}]^+$: 318.0890, found: 318.0887.

5. Single Crystal X-ray Structure Determinations of Compounds

Single crystal X-ray structure determinations of compound 3ac (CCDC: 2159691)

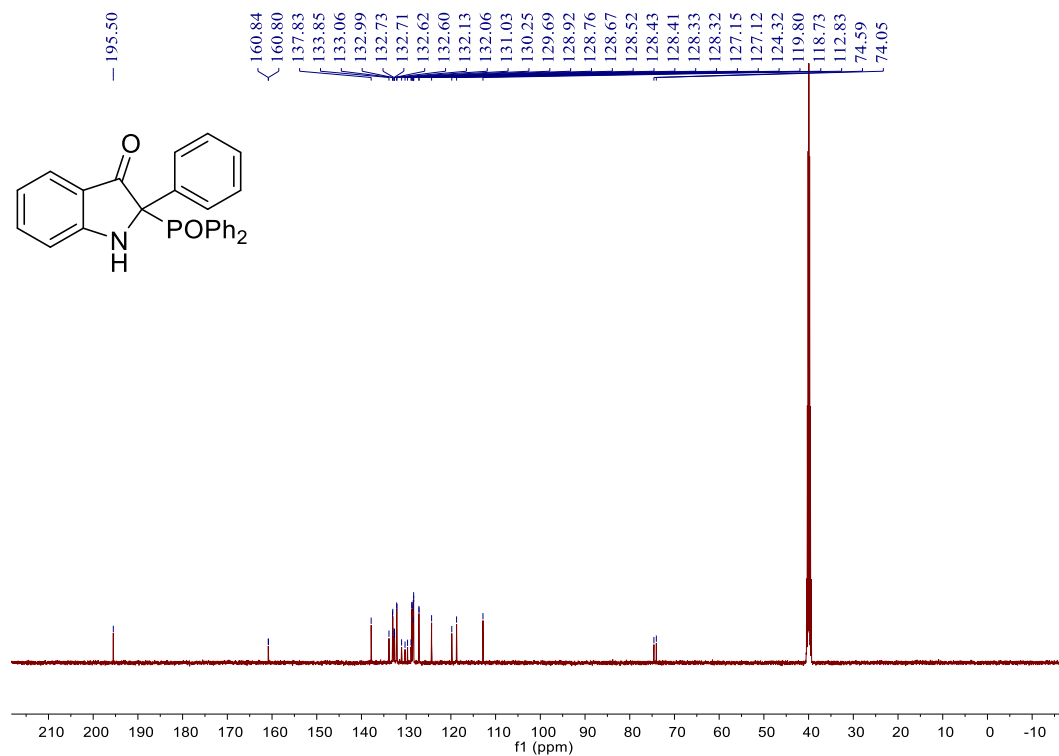
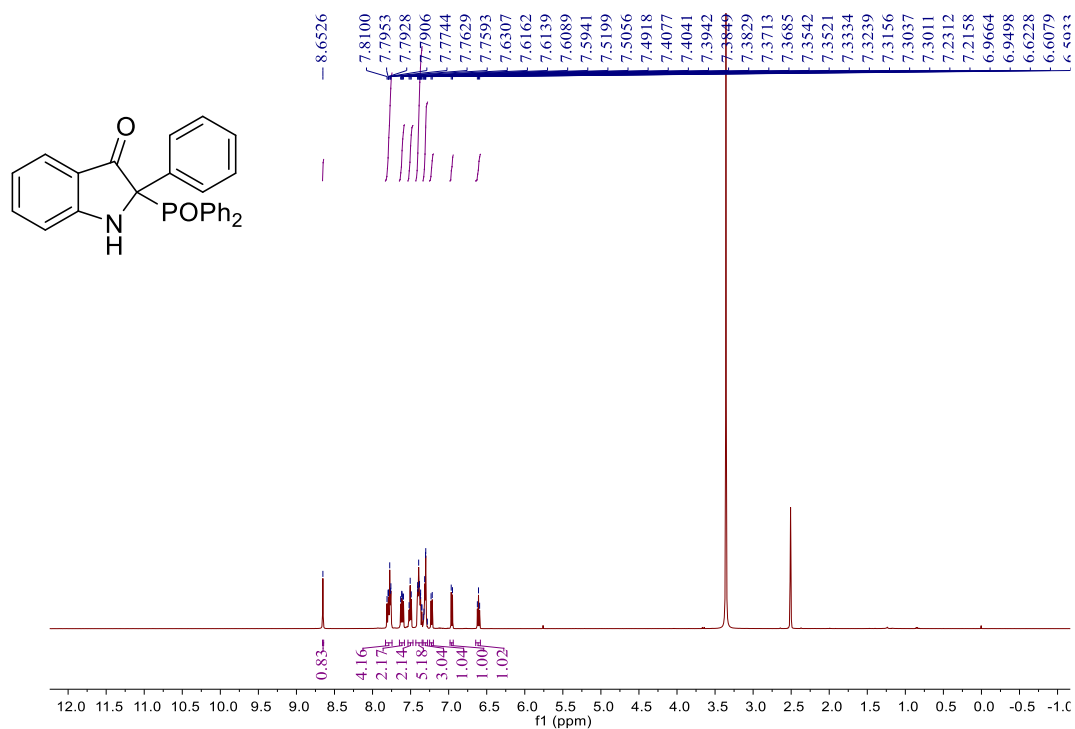


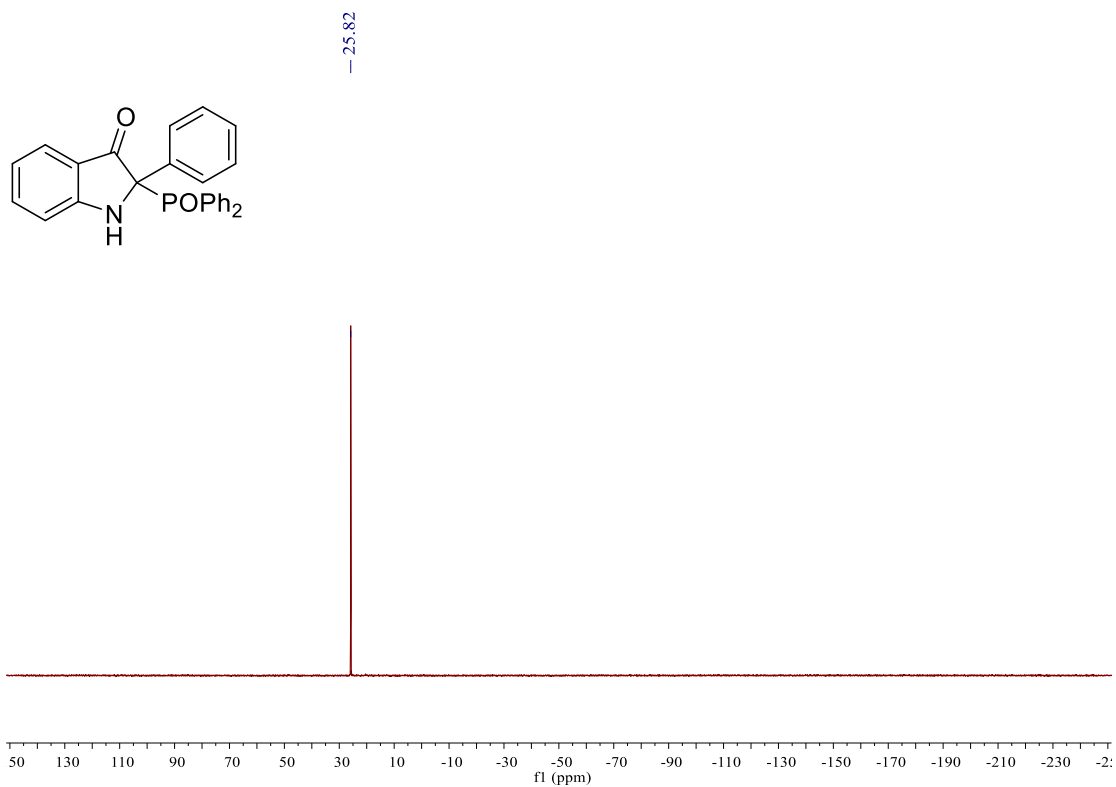
Bond precision:	C-C = 0.0045 Å	Wavelength=0.71073
Cell:	a=9.8688(7) b=9.9088(7) c=10.7278(8)	
	alpha=73.336 (2) beta=75.766 (2)	
	gamma=60.403 (2)	
Temperature:	293 K	
	Calculated	Reported
Volume	866.99 (11)	866.99 (11)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C18 H20 N O4 P	C18 H20 N O4 P
Sum formula	C18 H20 N O2 P	C18 H20 N O4 P
Mr	345.32	345.32
Dx,g cm-3	1.323	1.323
Z	2	2
Mu (mm-1)	0.180	0.180
F000	364.0	364.0
F000'	364.37	
h,k,lmax	12,12,13	12,12,13

Nref	3991	3970
Tmin,Tmax	0.945,0.972	0.689,0.746
Tmin'	0.937	
Correction method= # Reported T Limits: Tmin=0.689 Tmax=0.746		
AbsCorr = MULTI-SCAN		
Data completeness= 0.995		Theta(max)= 27.551
R(reflections)= 0.0589(3309)		wR2(reflections)= 0.1673(3970)
S = 1.026		Npar= 221

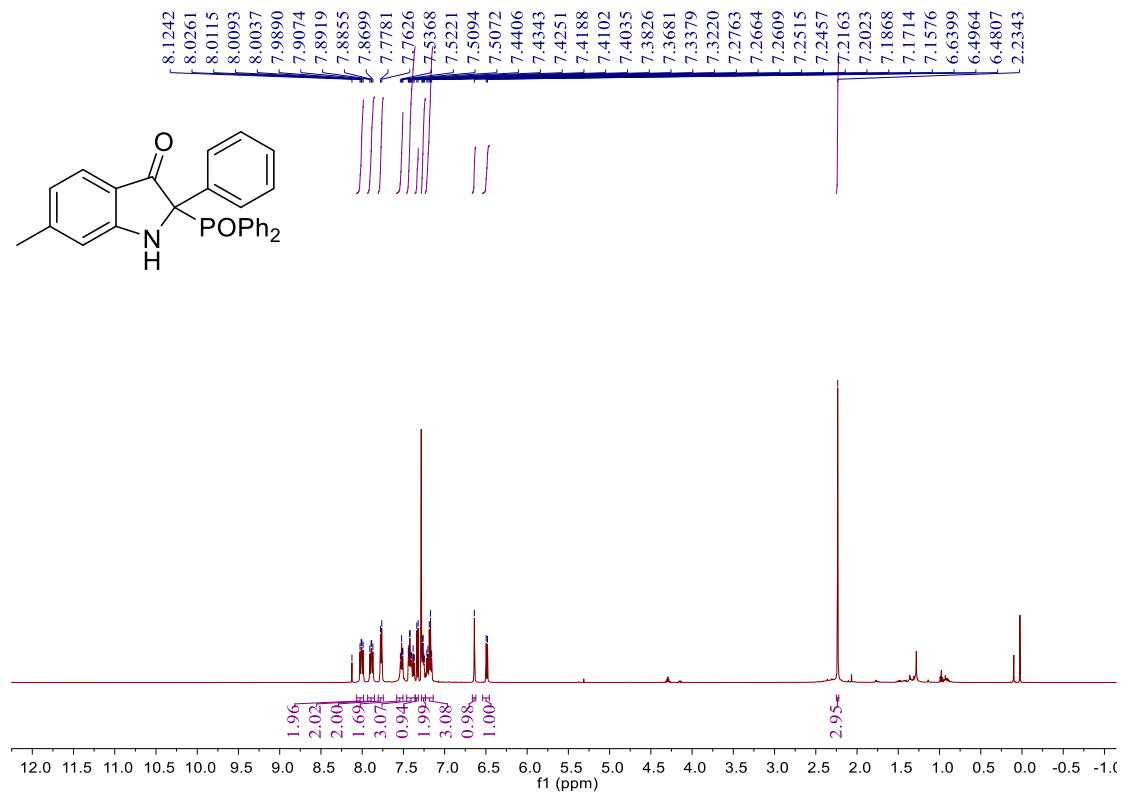
6. ^1H NMR, ^{13}C NMR, ^{31}P NMR and ^{19}F NMR spectra for compounds

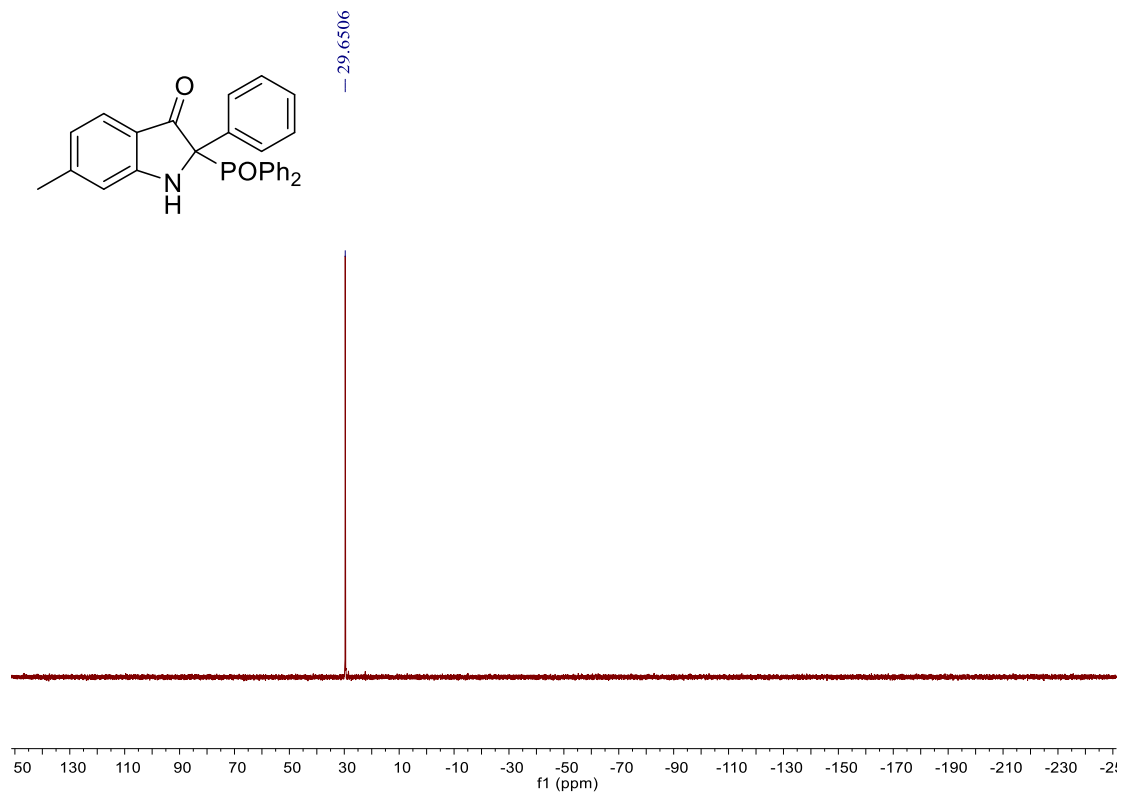
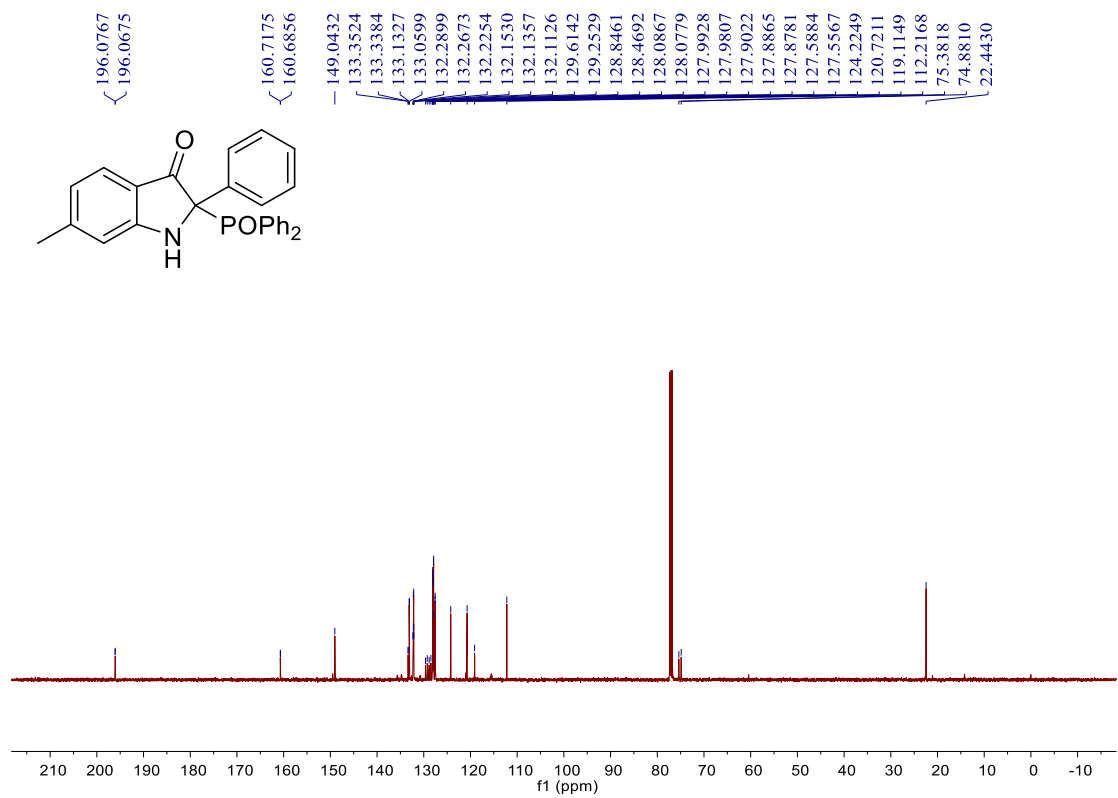
2-(diphenylphosphoryl)-2-phenylindolin-3-one (3a)



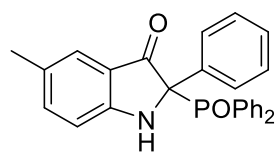
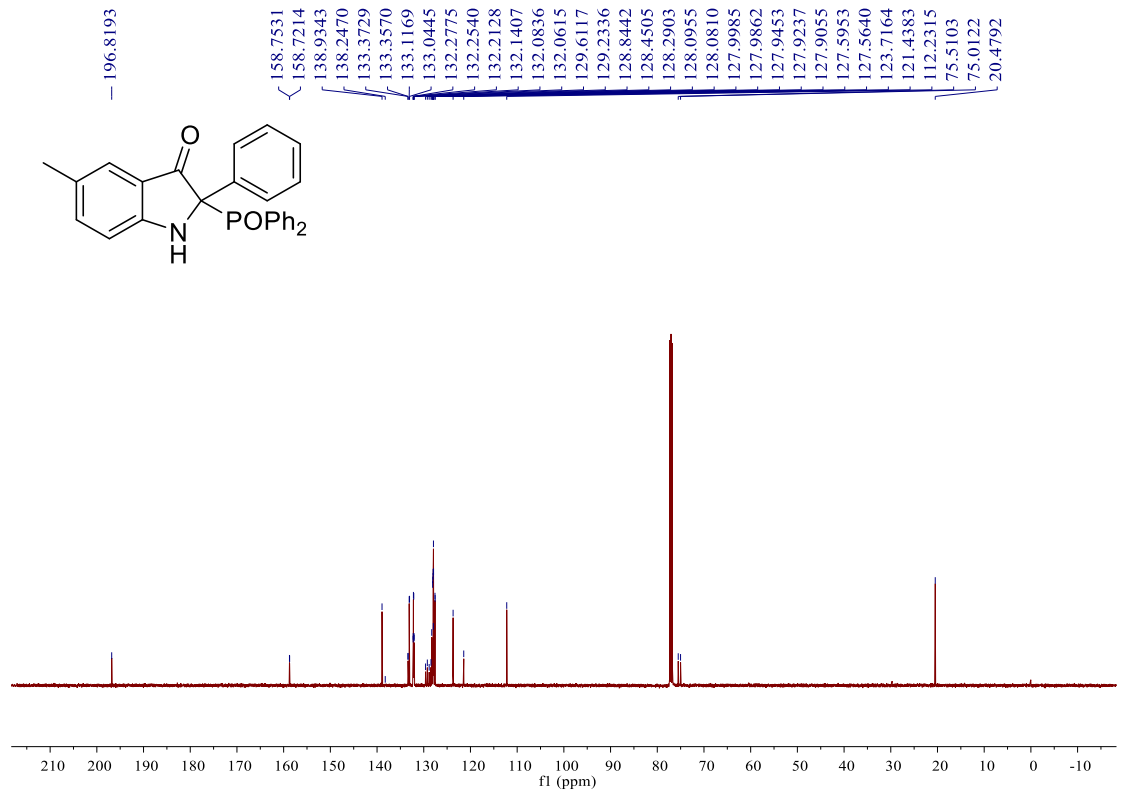
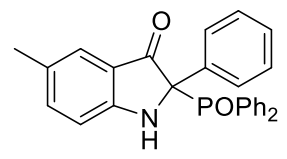
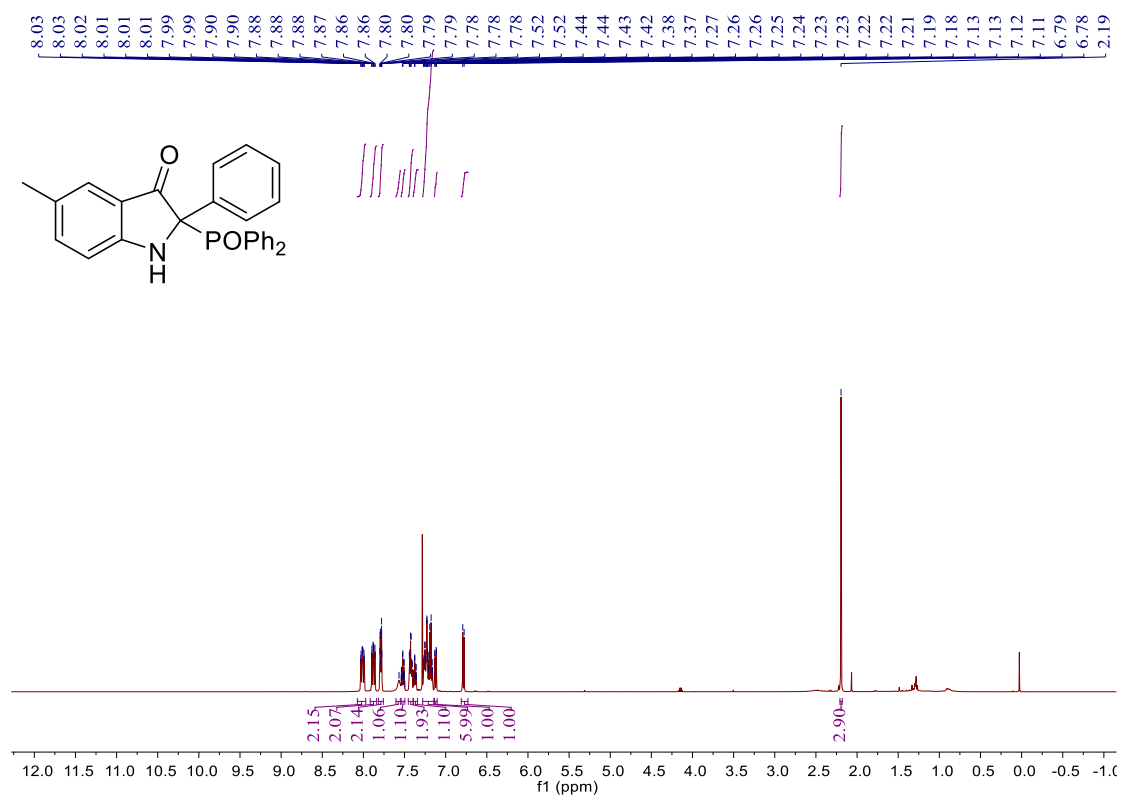


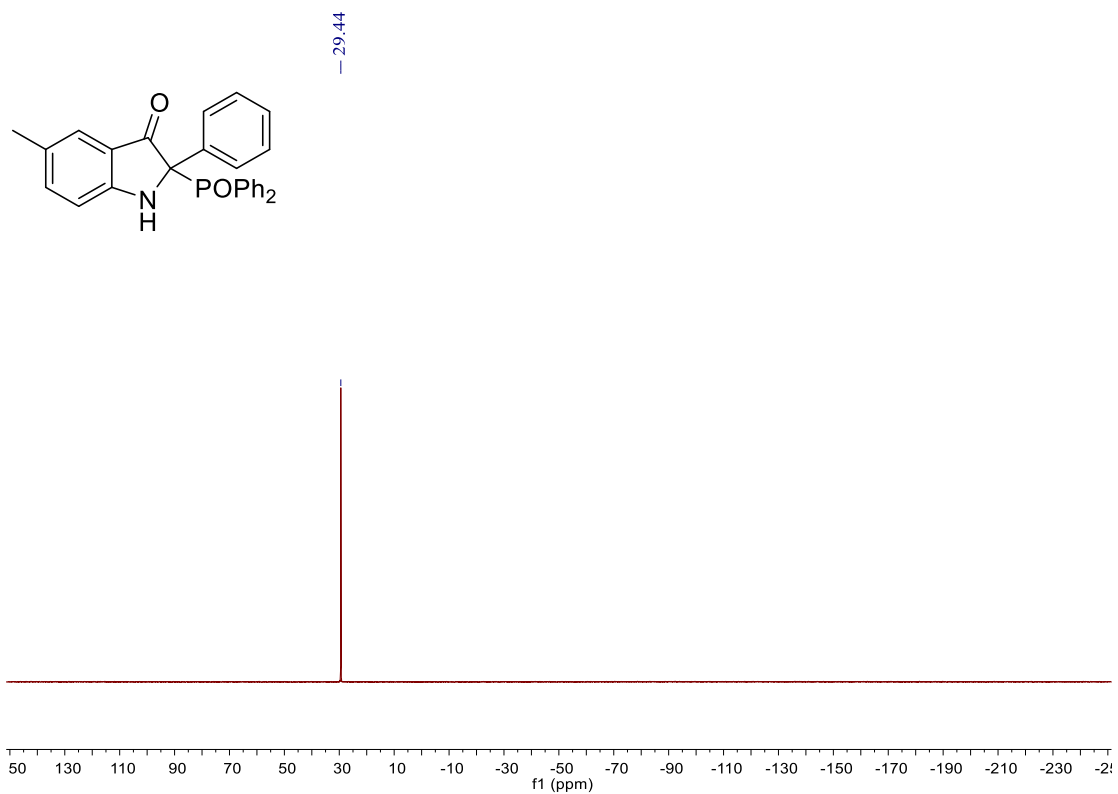
2-(diphenylphosphoryl)-6-methyl-2-phenylindolin-3-one (3b)



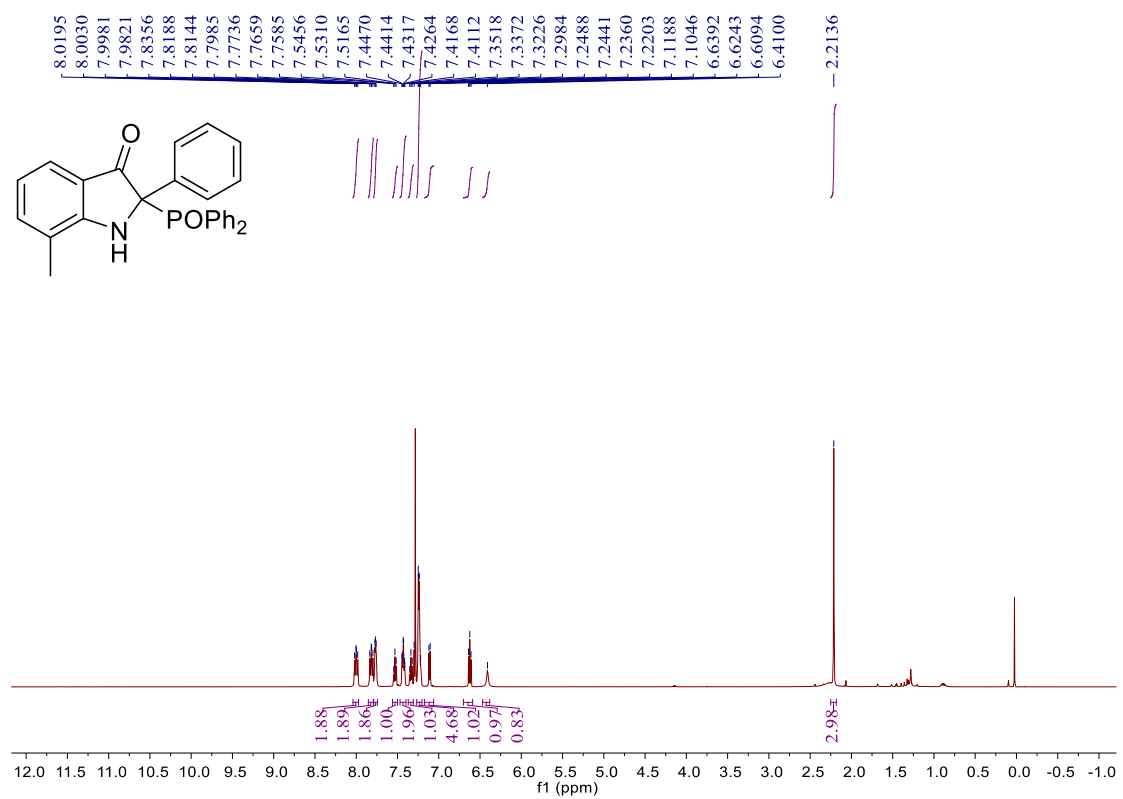


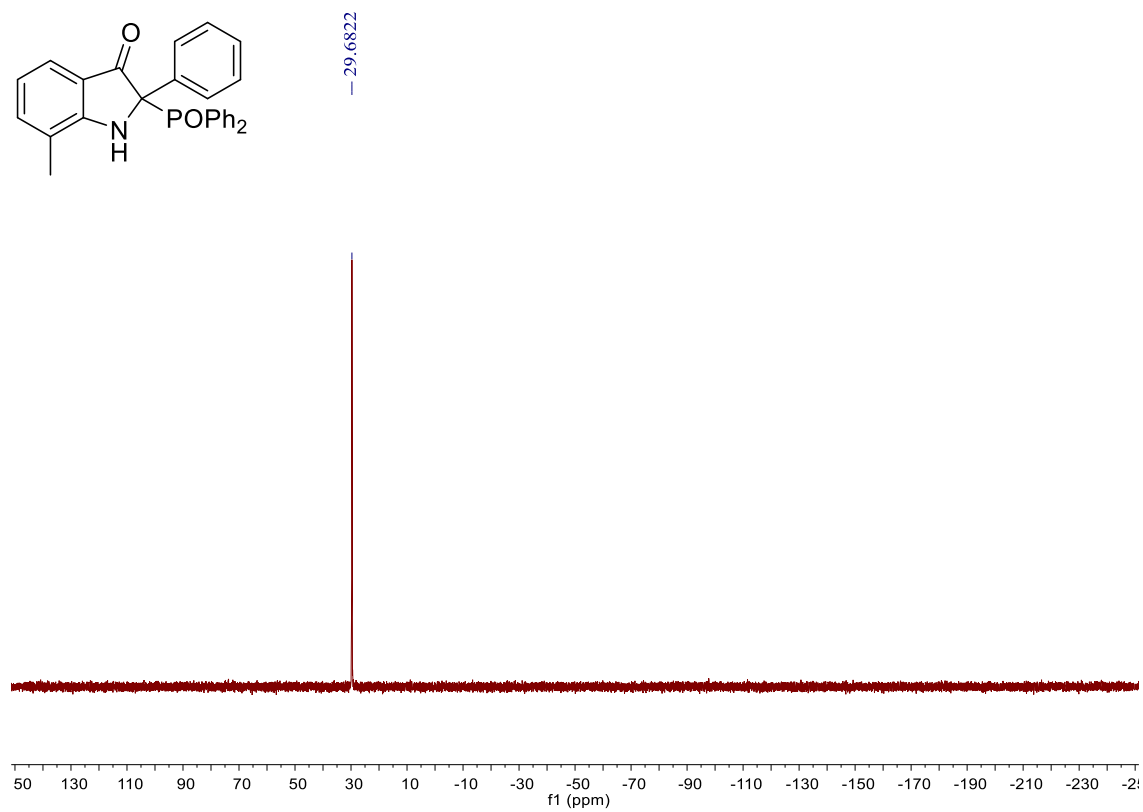
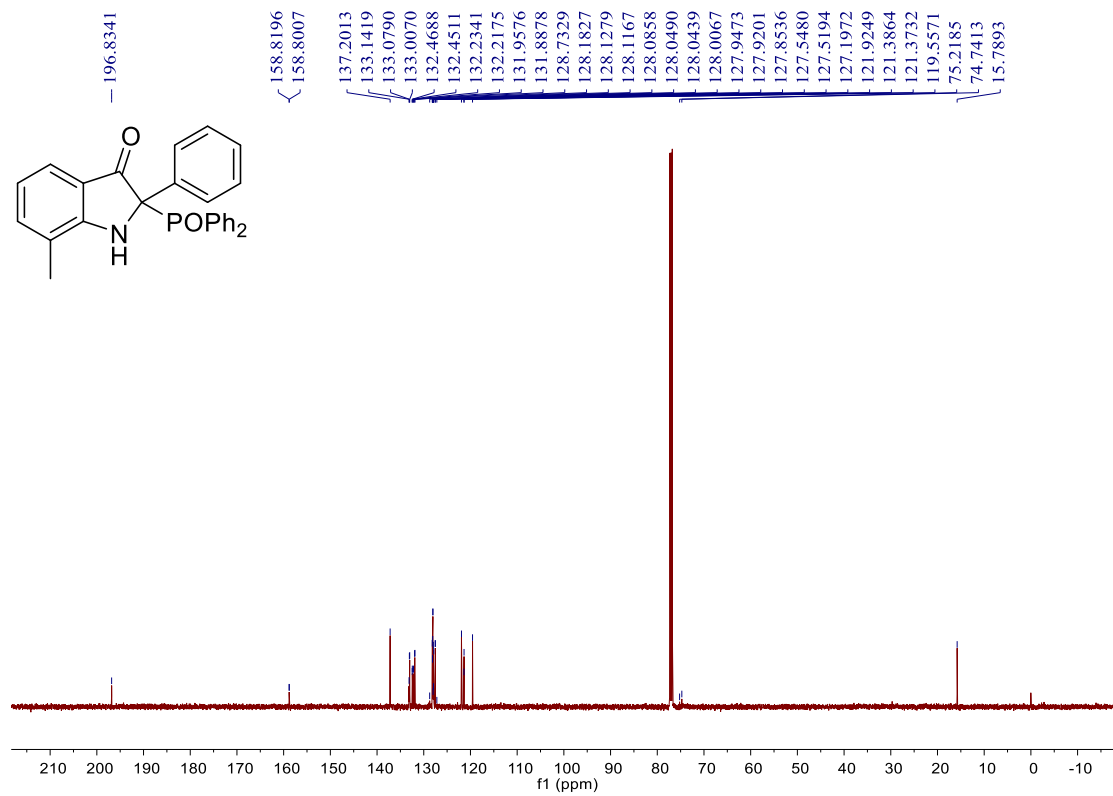
2-(diphenylphosphoryl)-5-methyl-2-phenylindolin-3-one (3c)



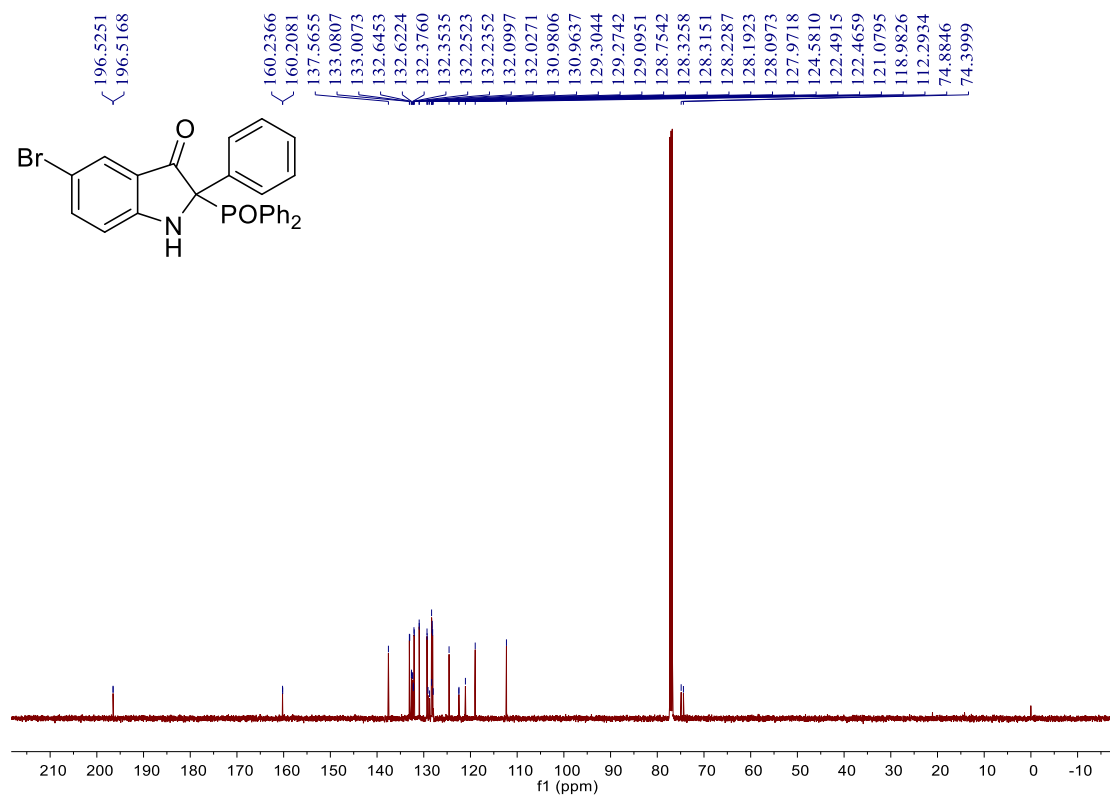
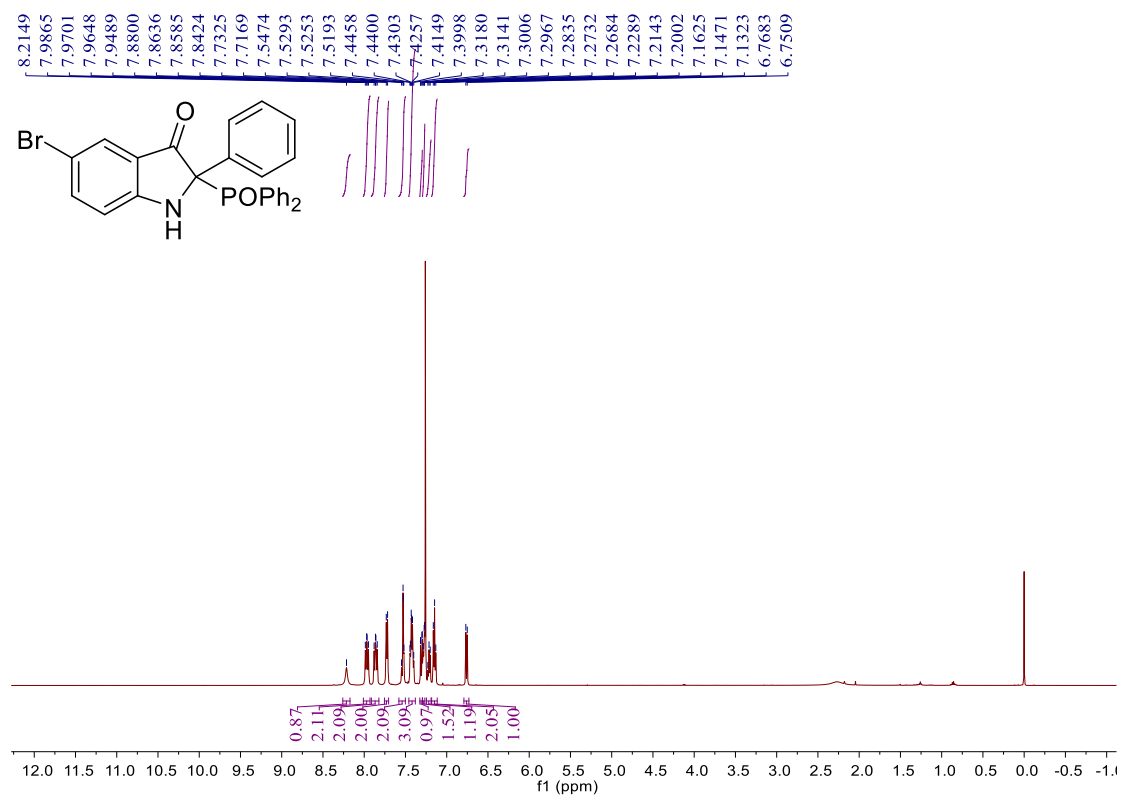


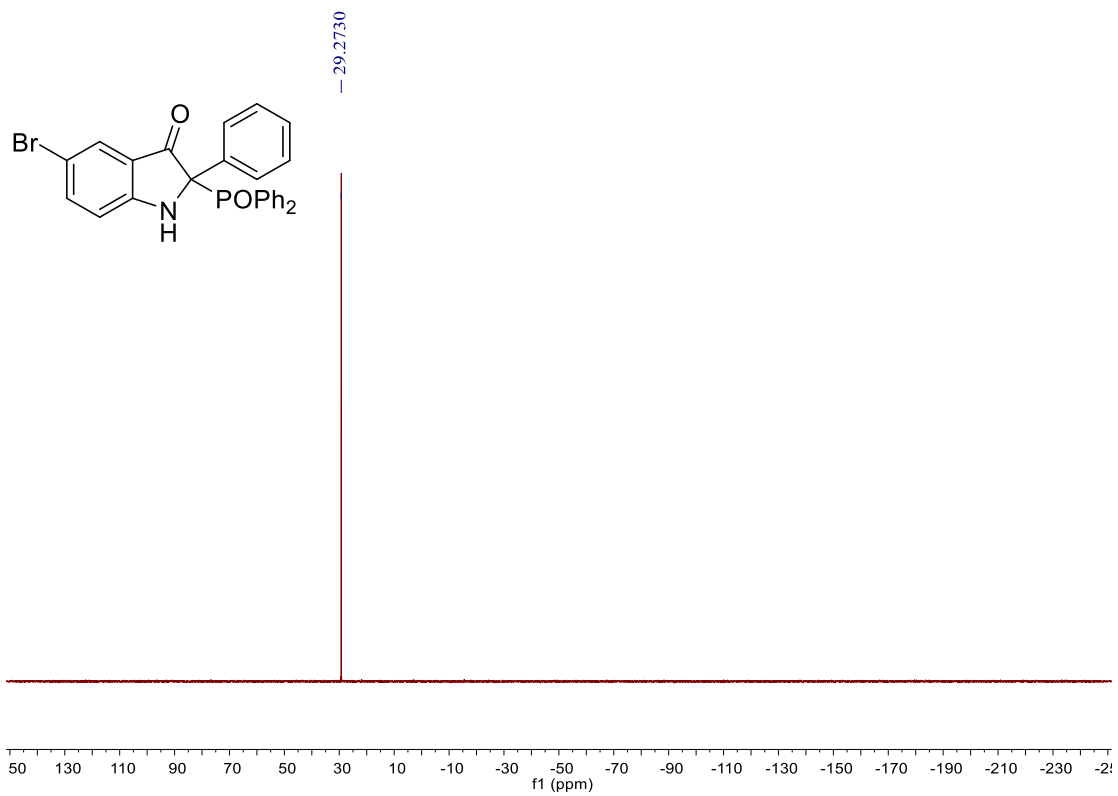
2-(diphenylphosphoryl)-7-methyl-2-phenylindolin-3-one (3d)



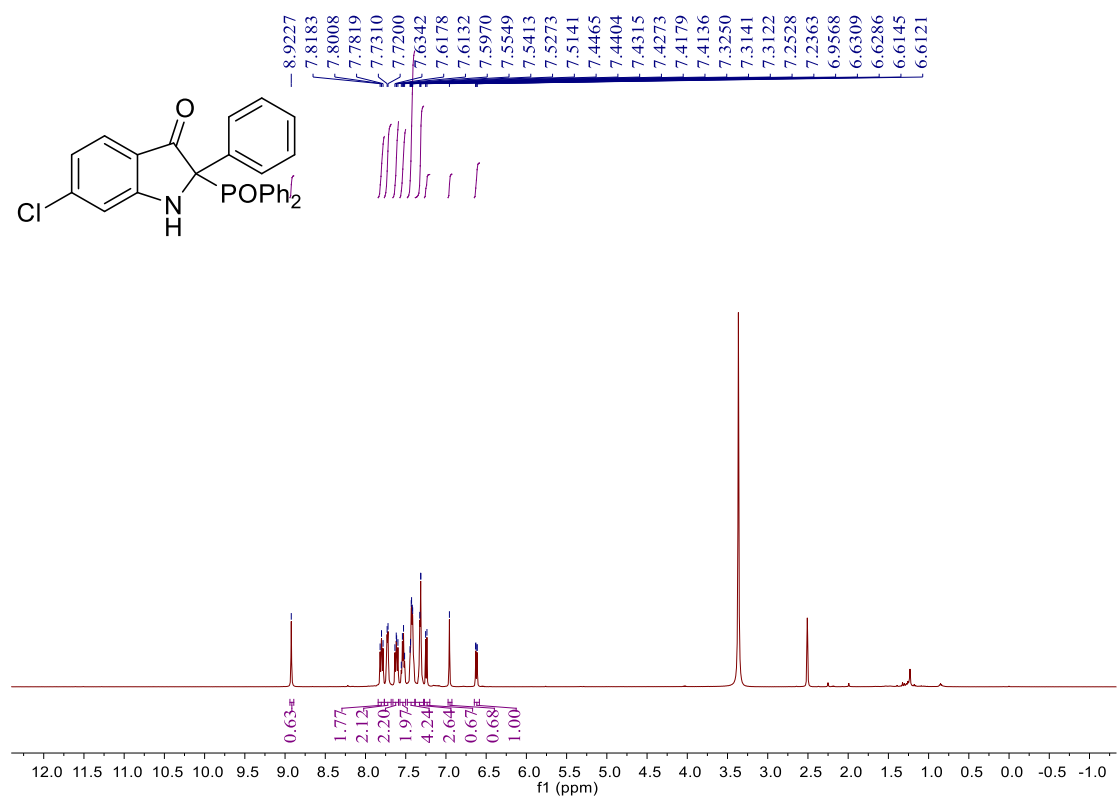


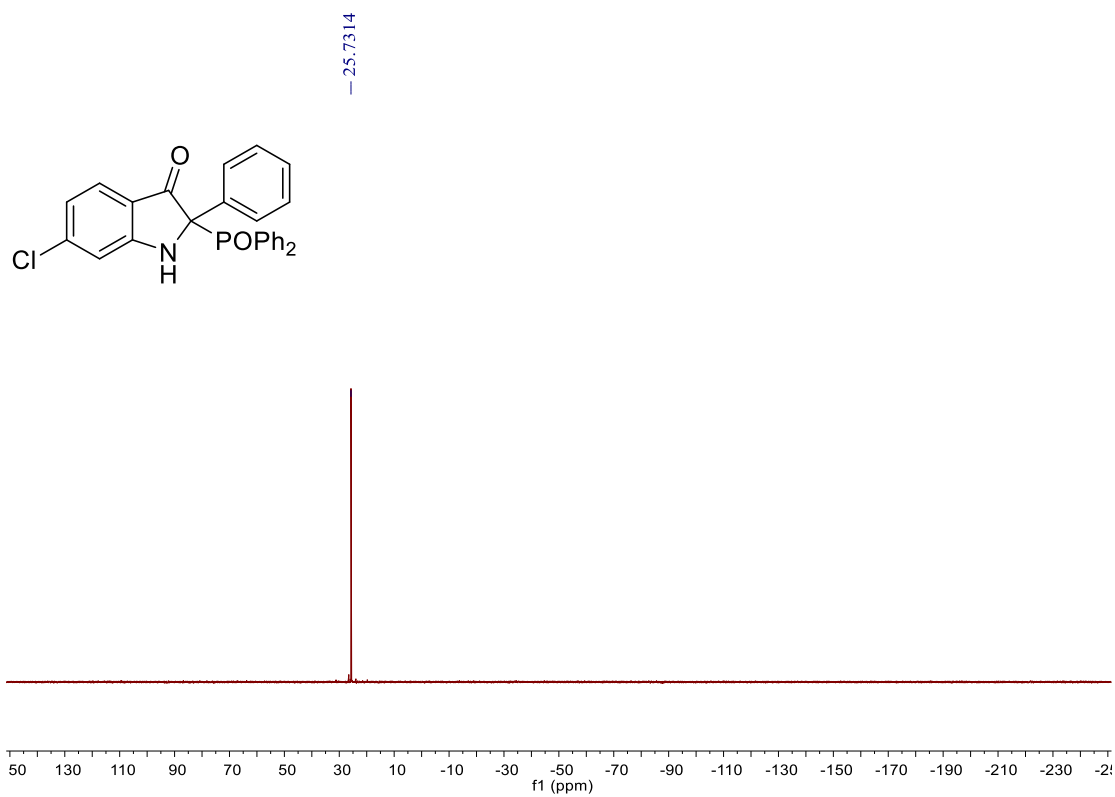
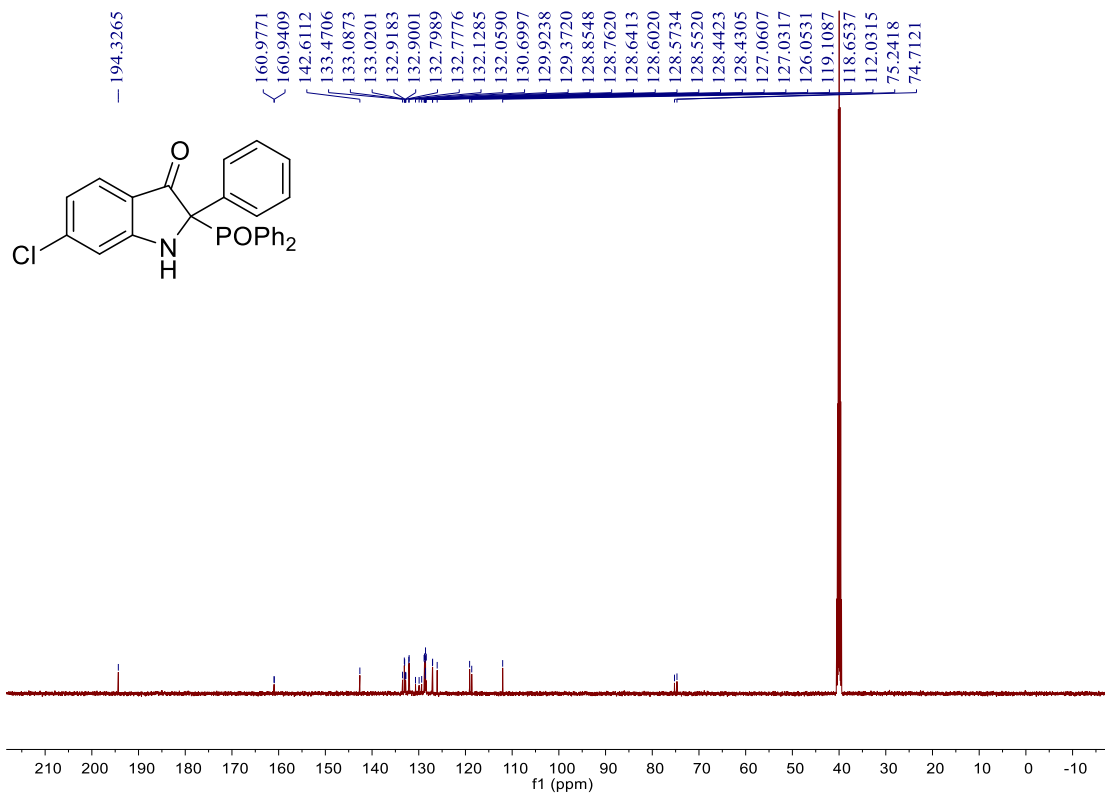
5-bromo-2-(diphenylphosphoryl)-2-phenylindolin-3-one (3e)



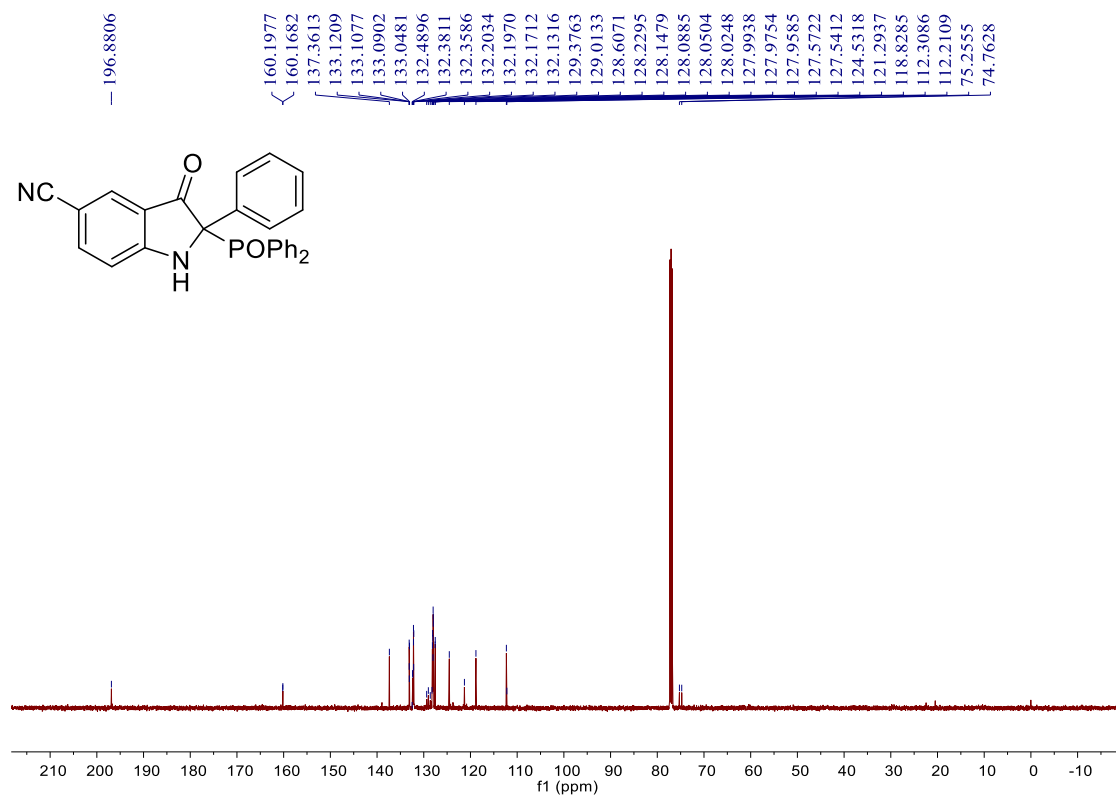
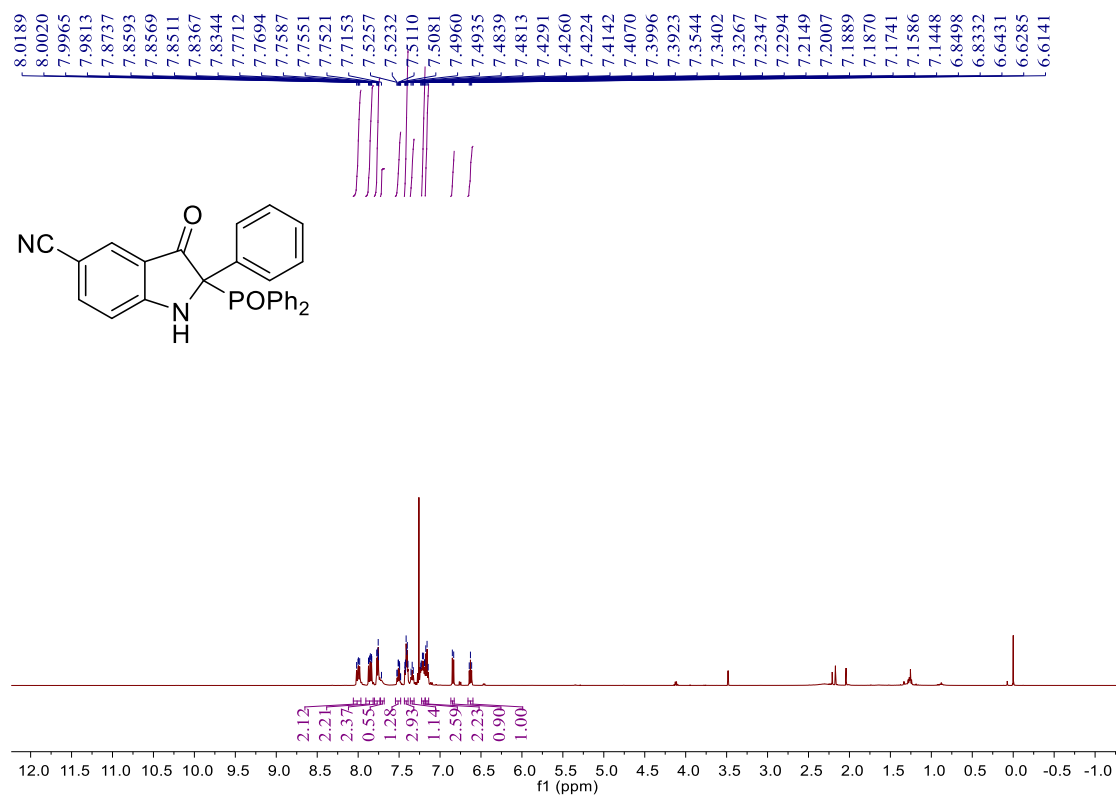


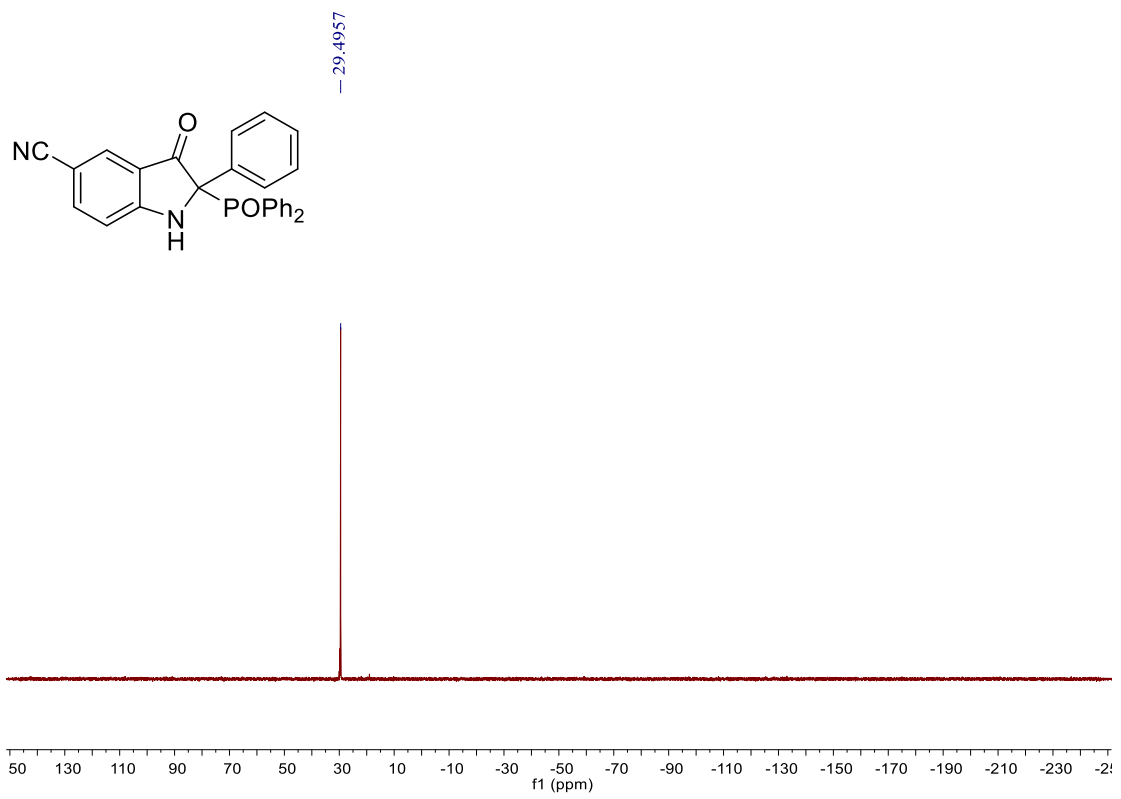
6-chloro-2-(diphenylphosphoryl)-2-phenylindolin-3-one (3f)



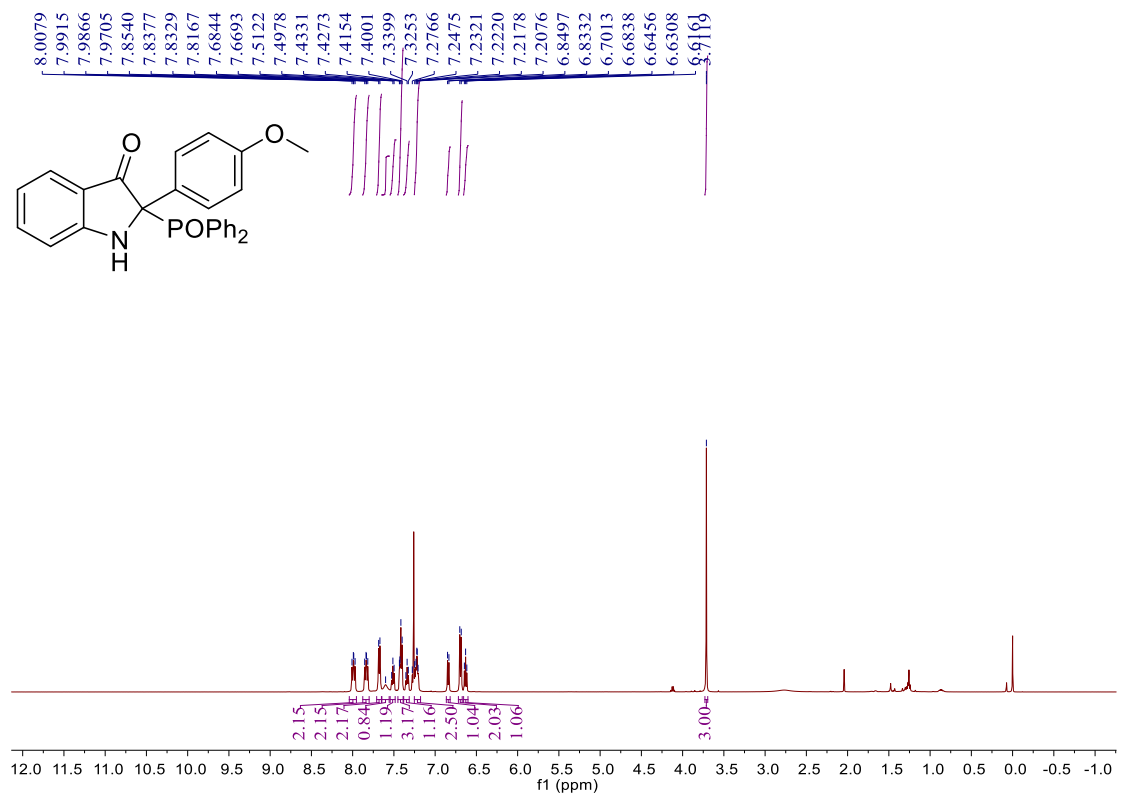


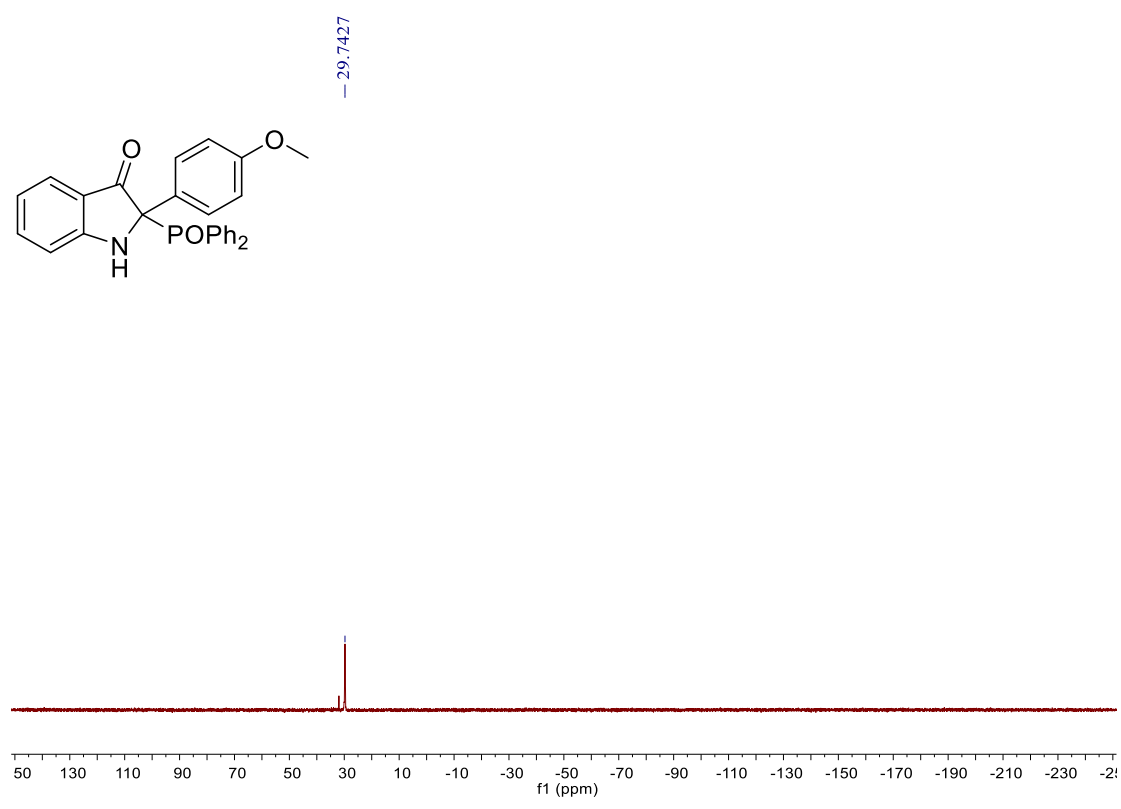
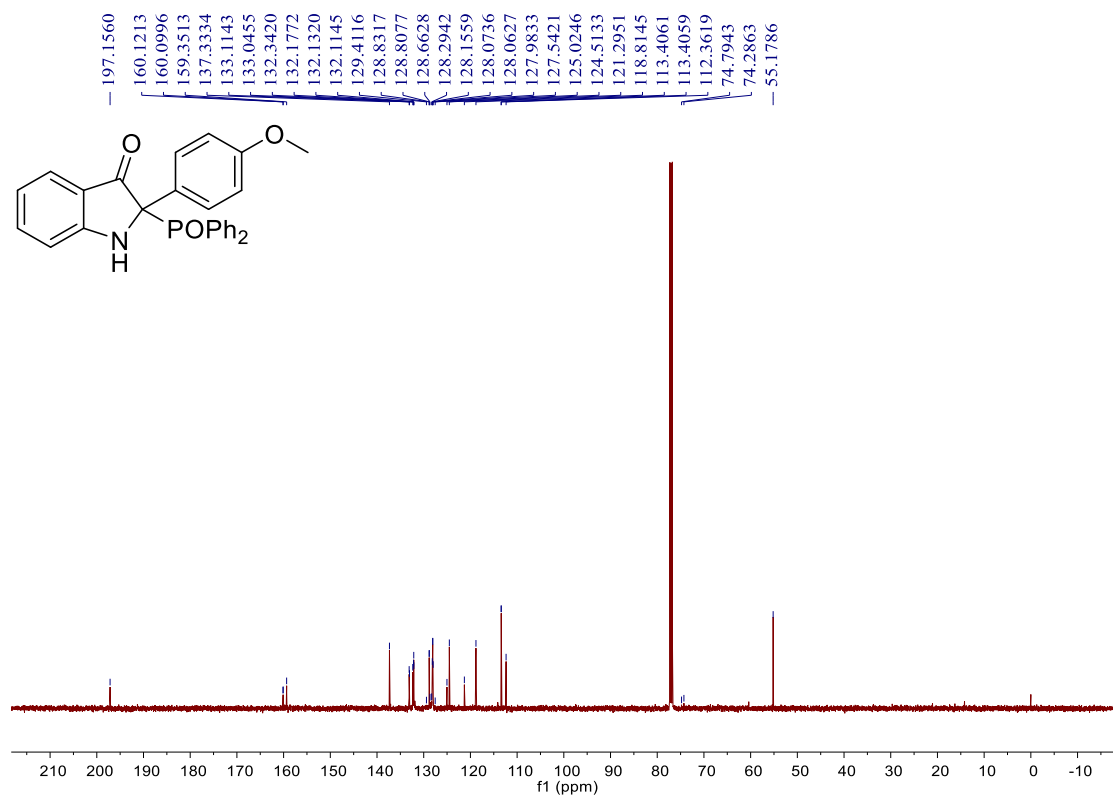
2-(diphenylphosphoryl)-3-oxo-2-phenylindoline-5-carbonitrile (3g)



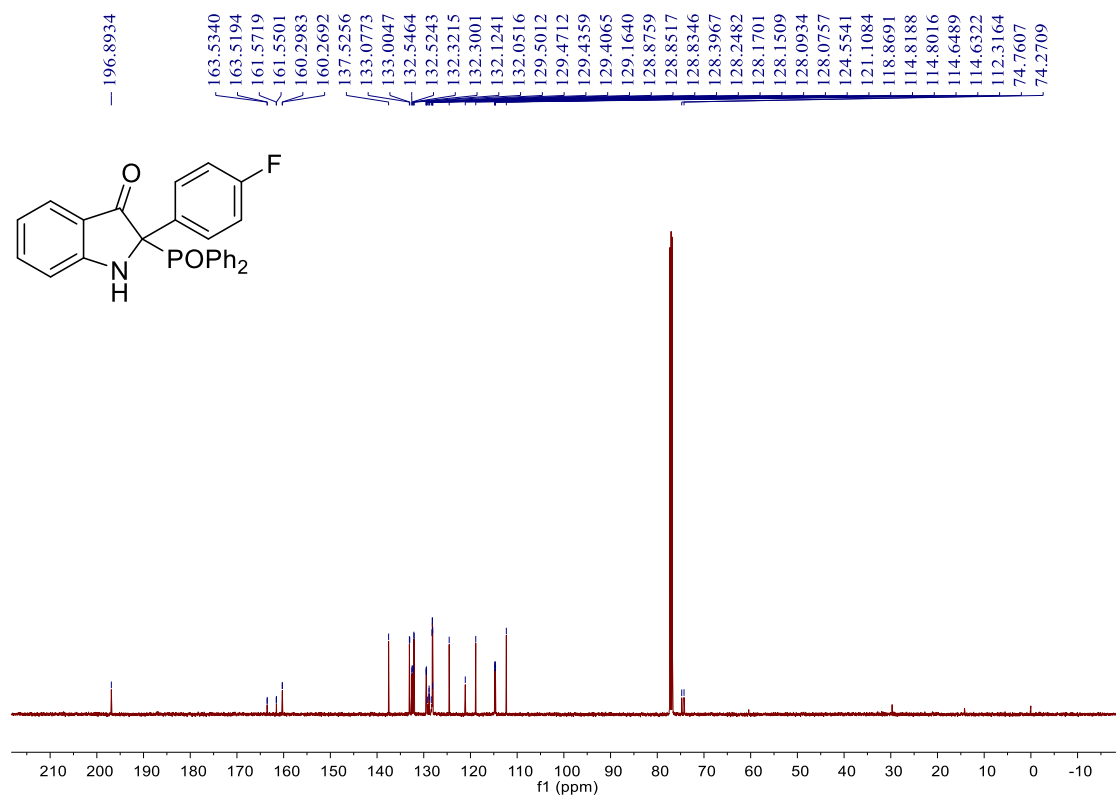
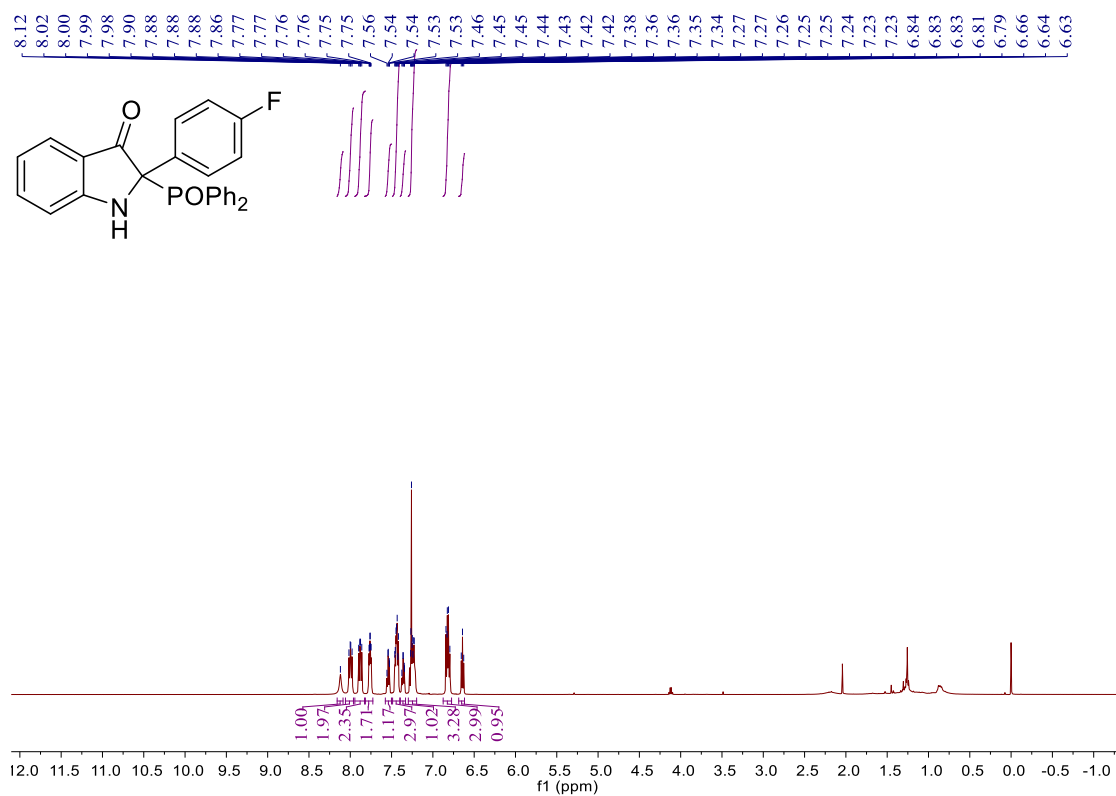


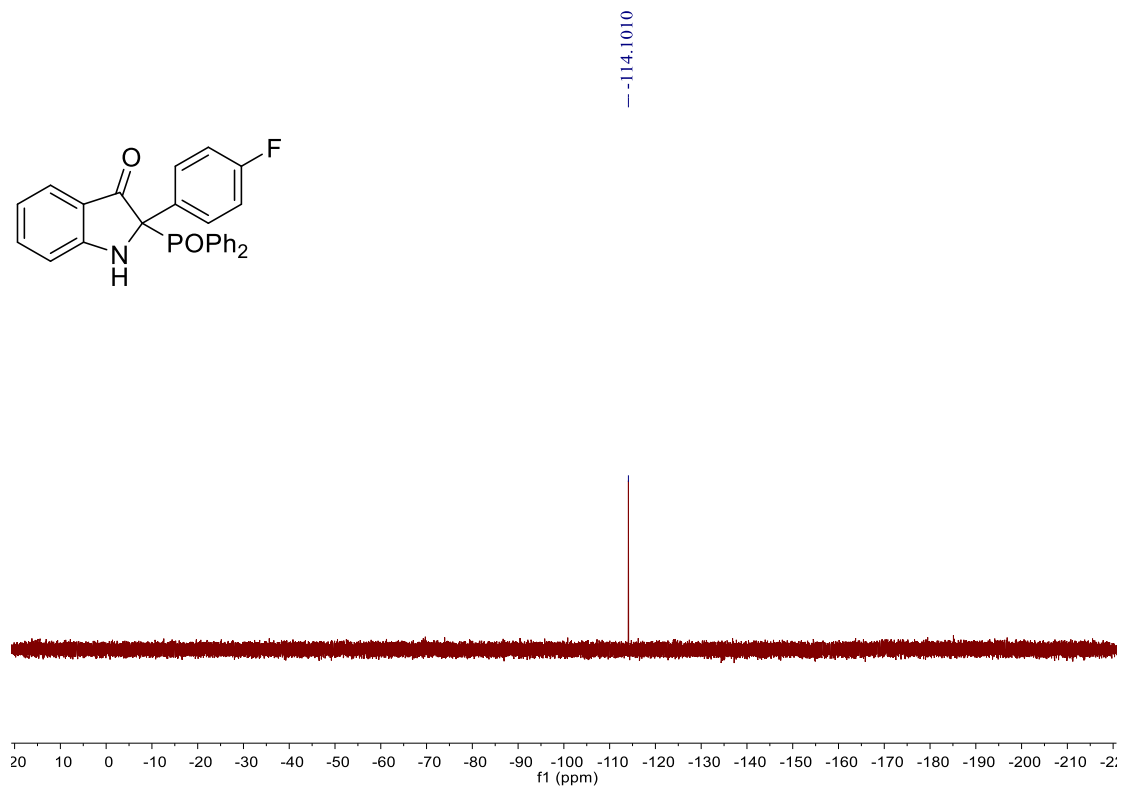
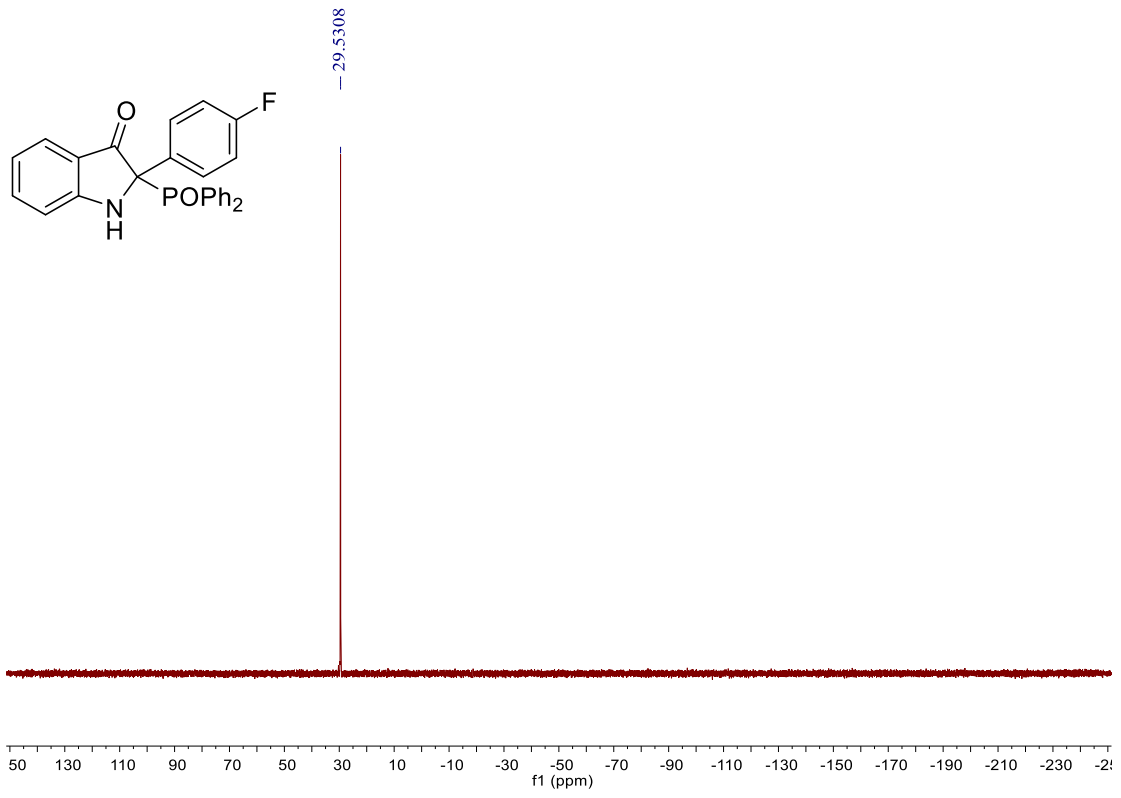
2-(diphenylphosphoryl)-2-(4-methoxyphenyl)indolin-3-one (3h)



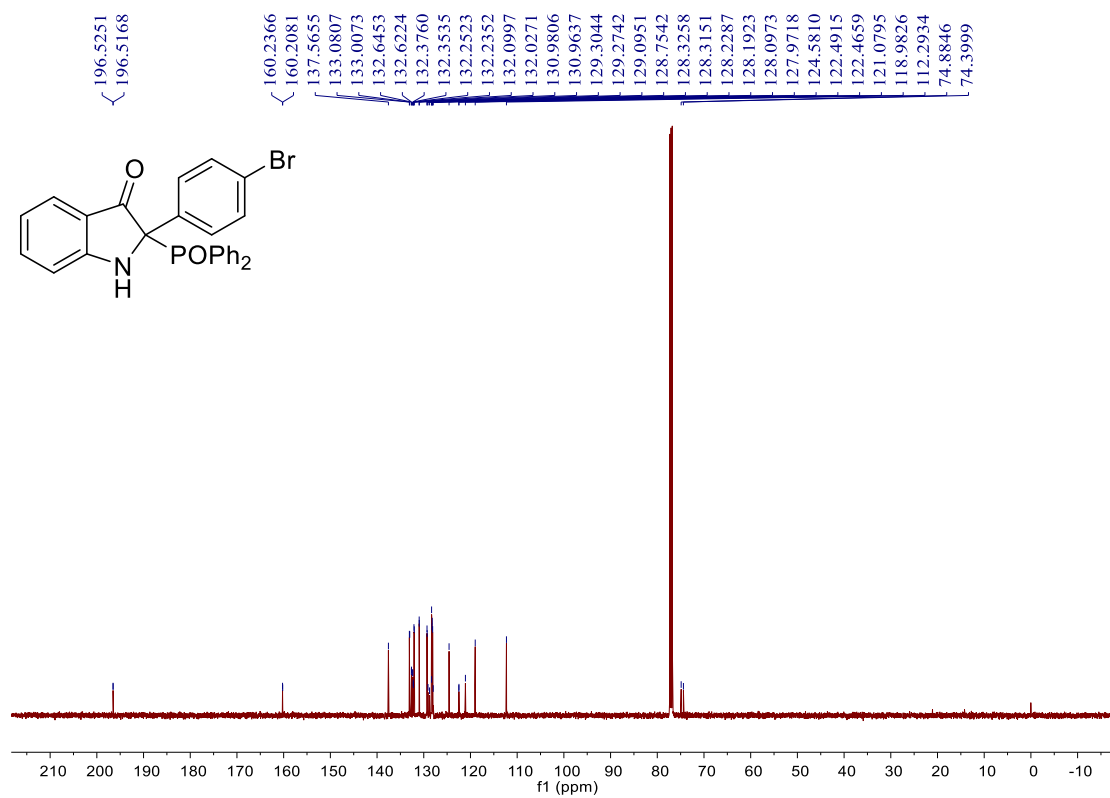
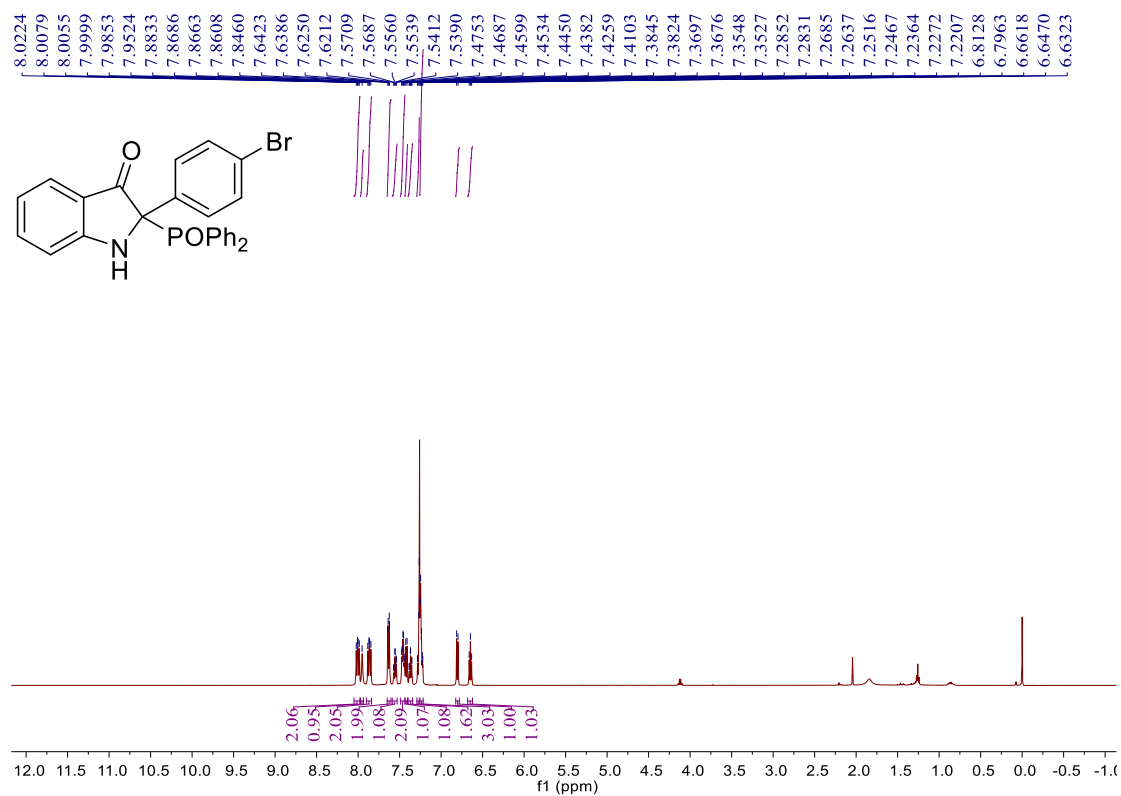


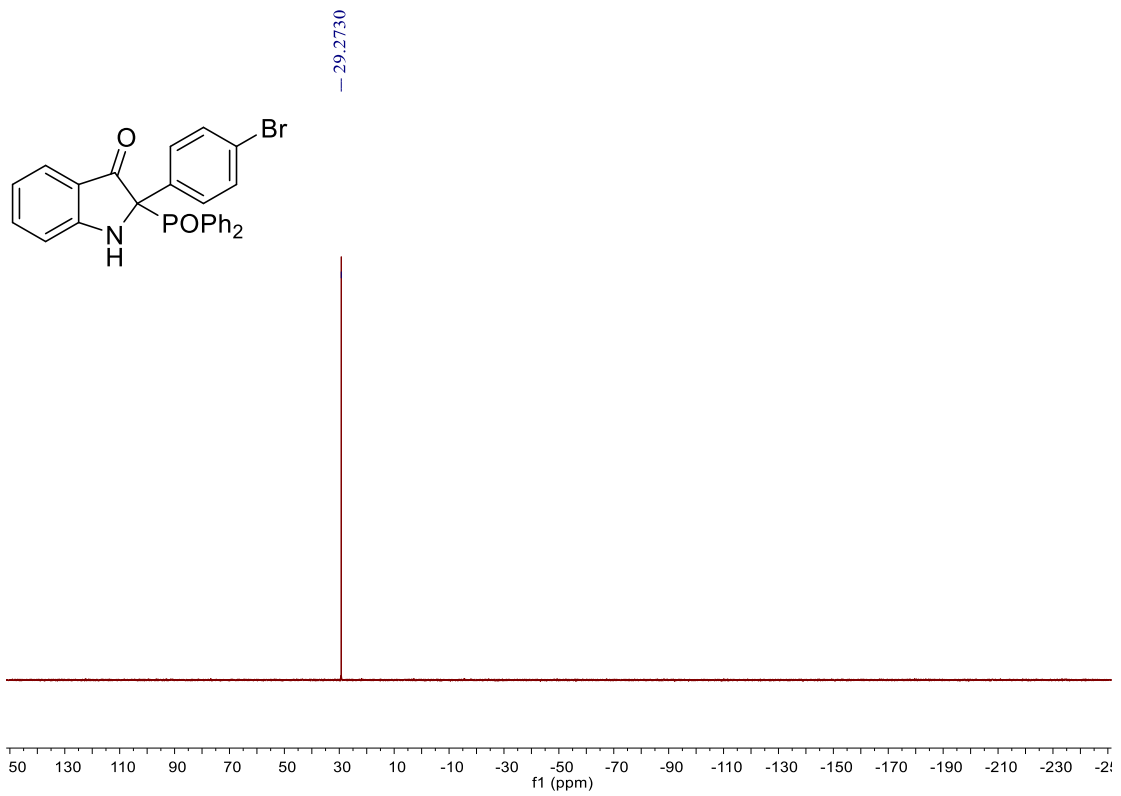
2-(diphenylphosphoryl)-2-(4-fluorophenyl)indolin-3-one (3i)



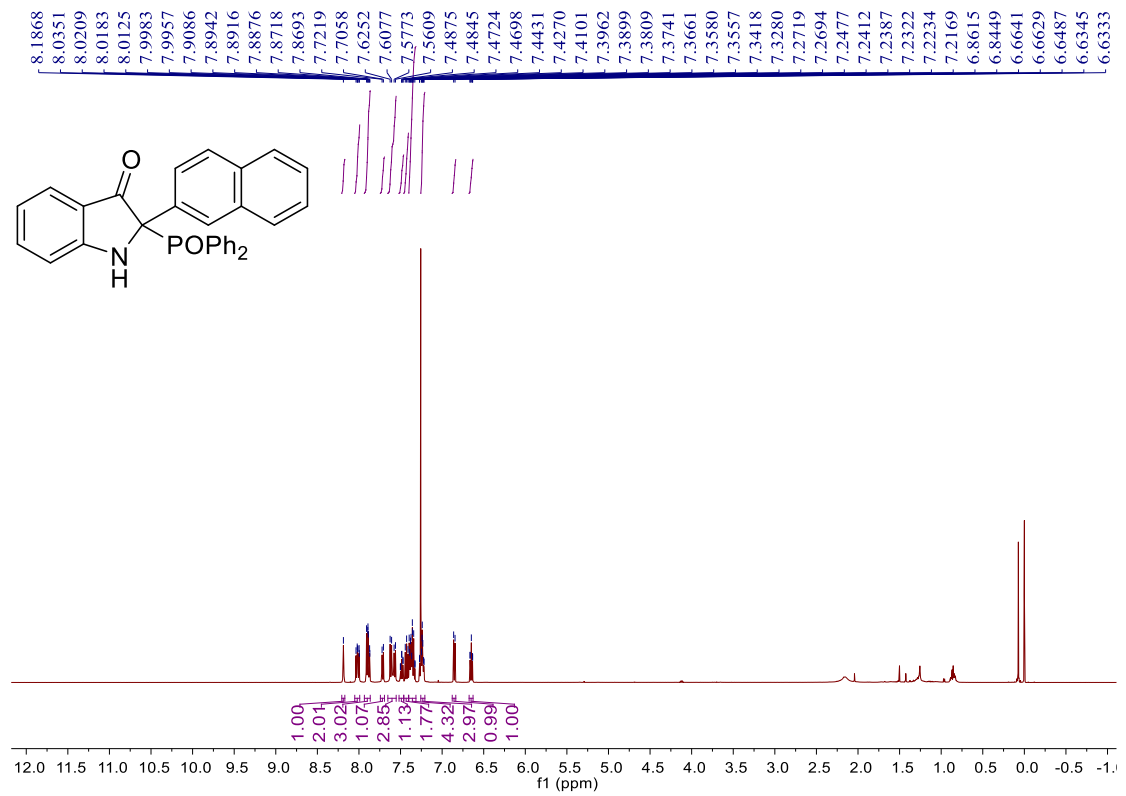


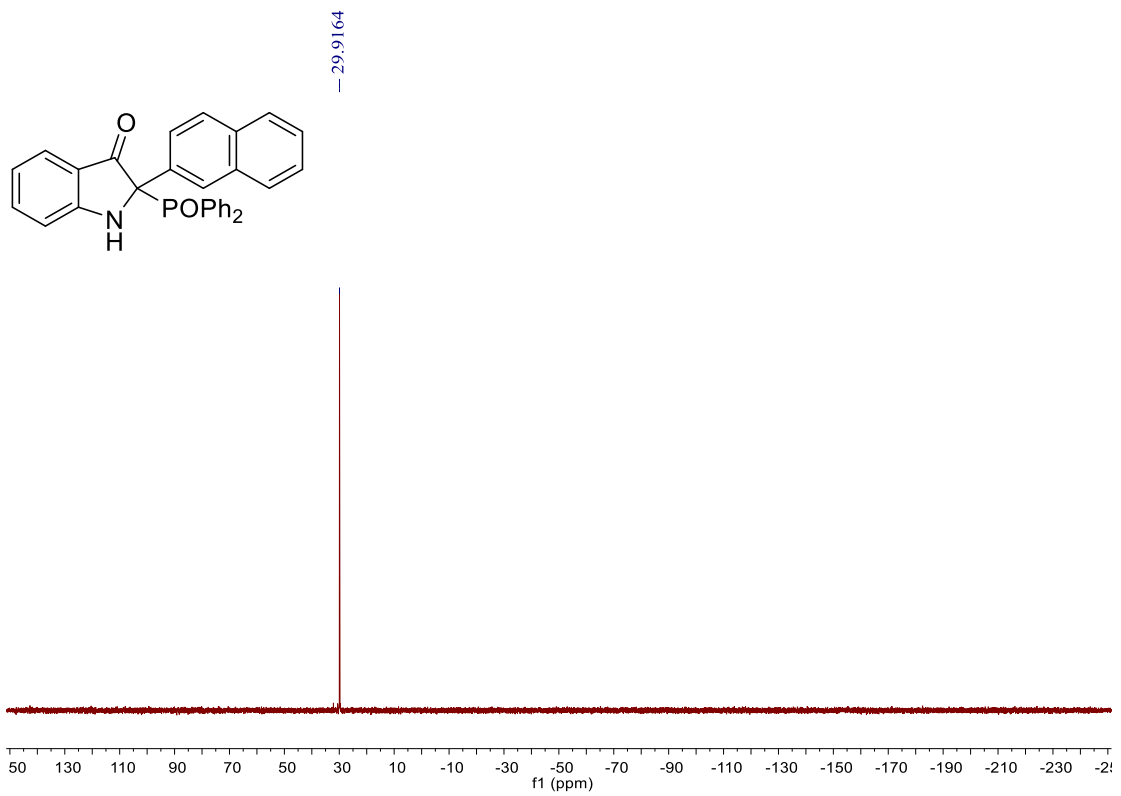
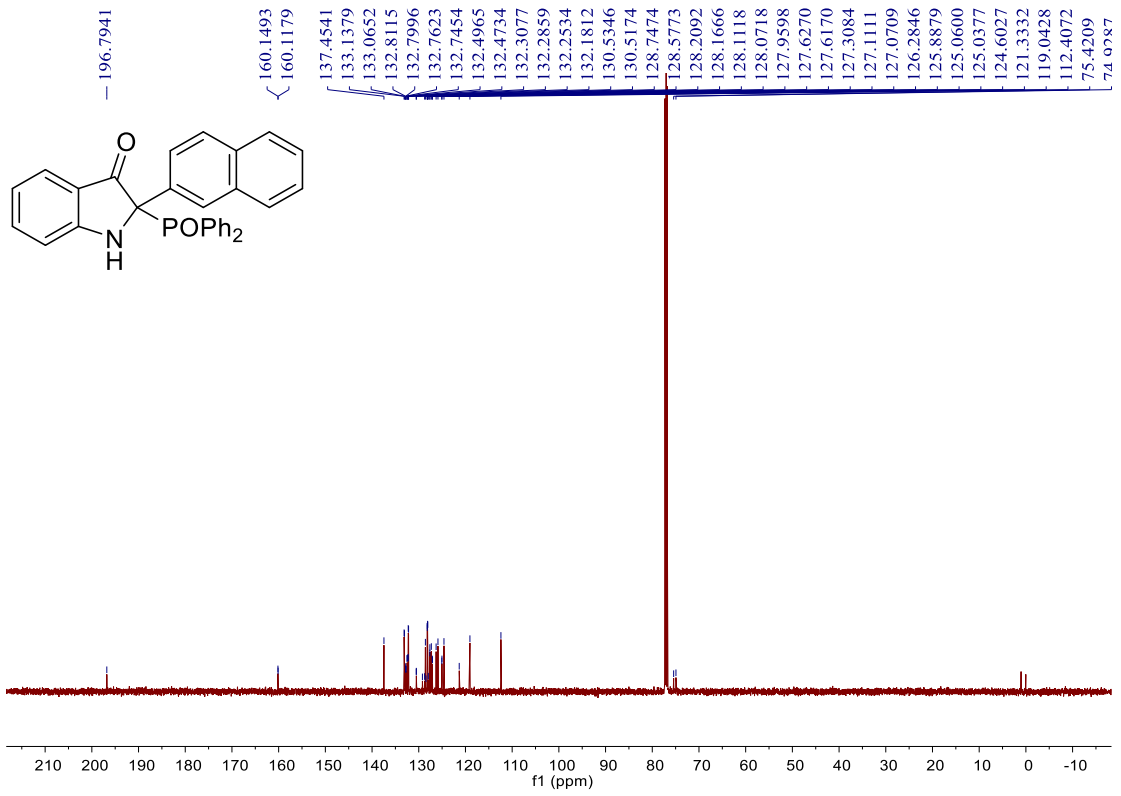
2-(4-bromophenyl)-2-(diphenylphosphoryl)indolin-3-one (3j)



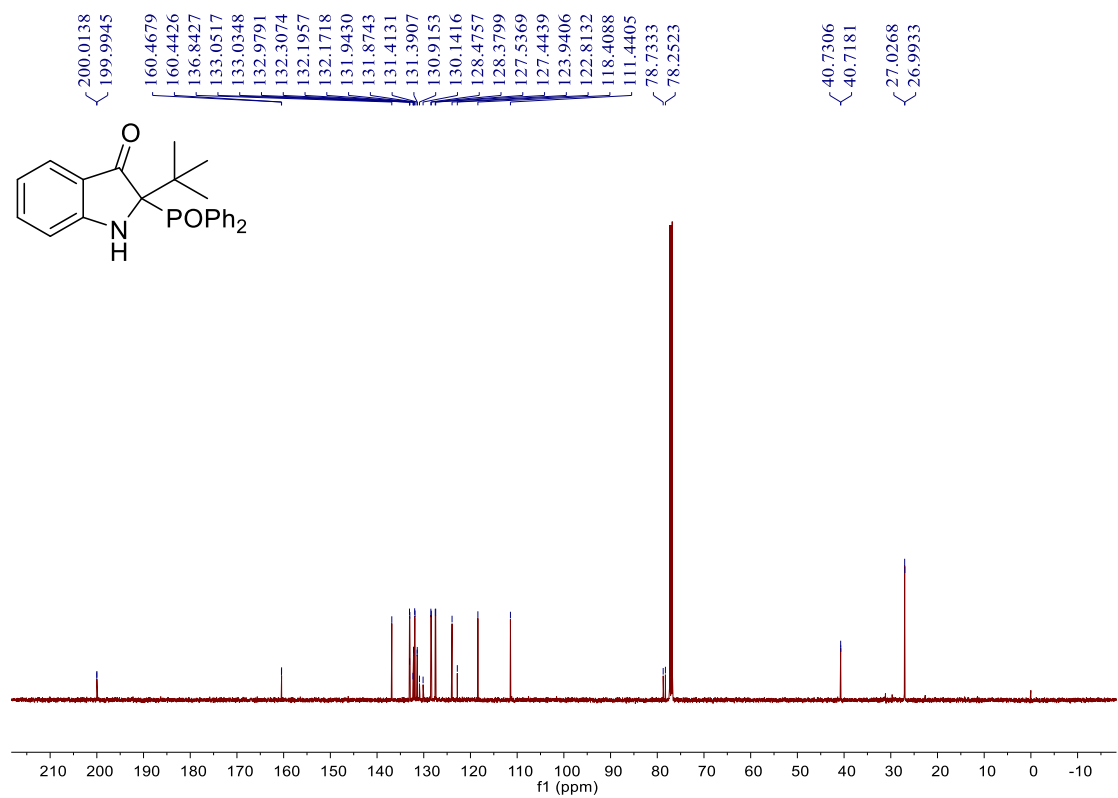
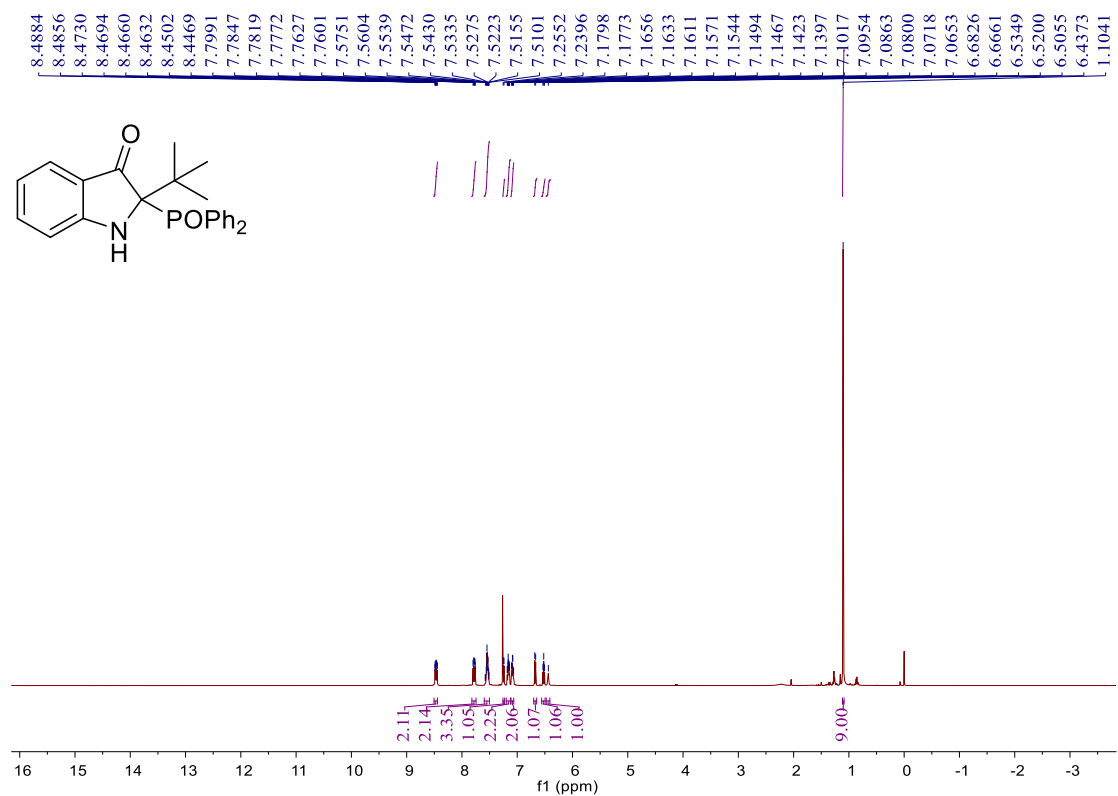


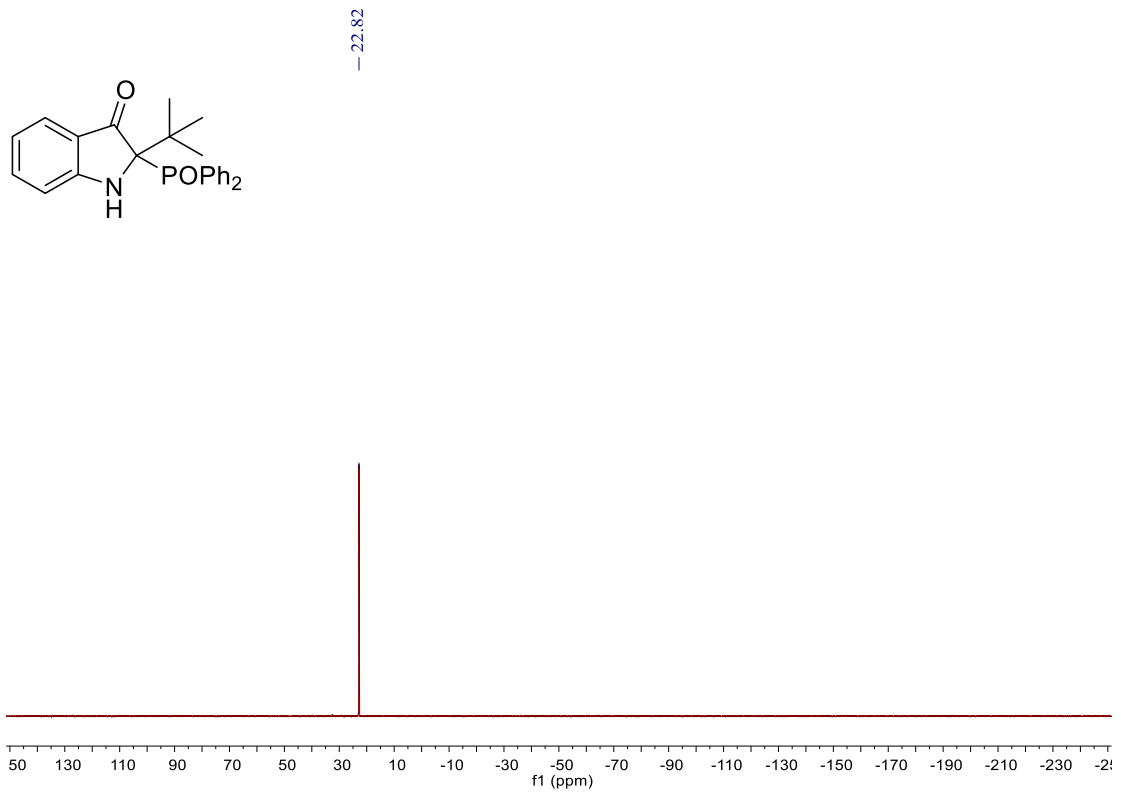
2-(diphenylphosphoryl)-2-(naphthalen-2-yl)indolin-3-one (3k)



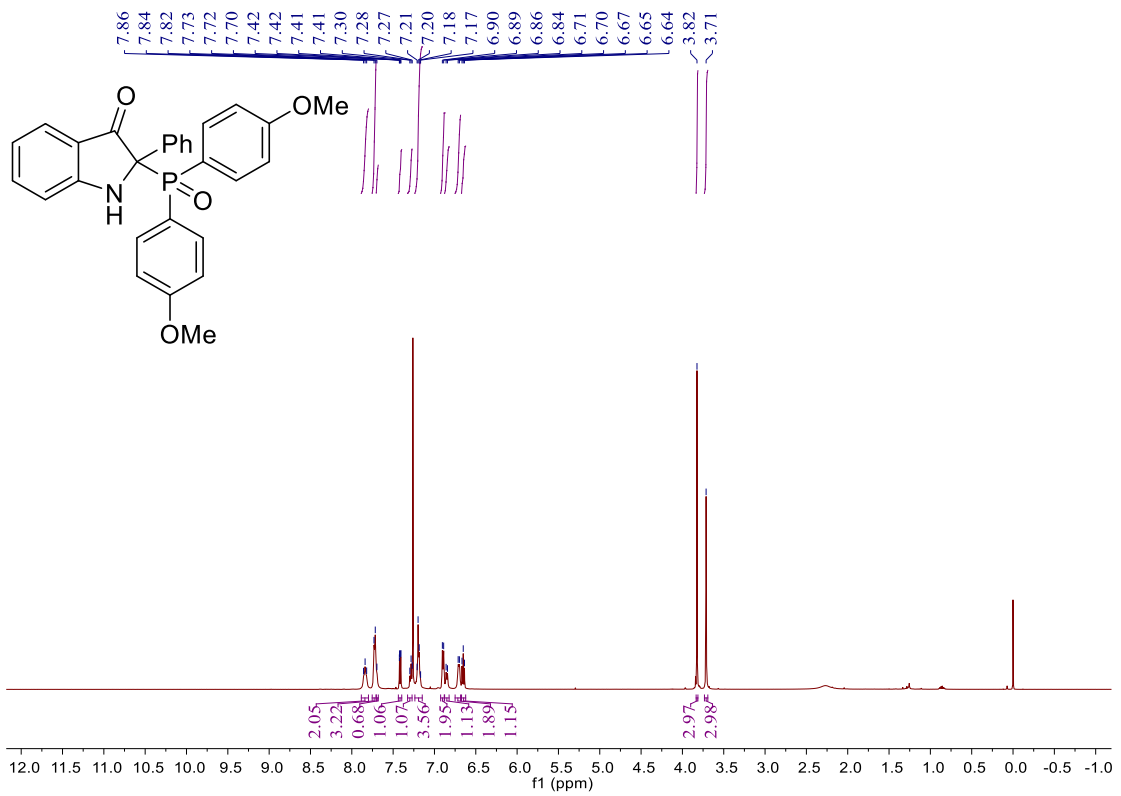


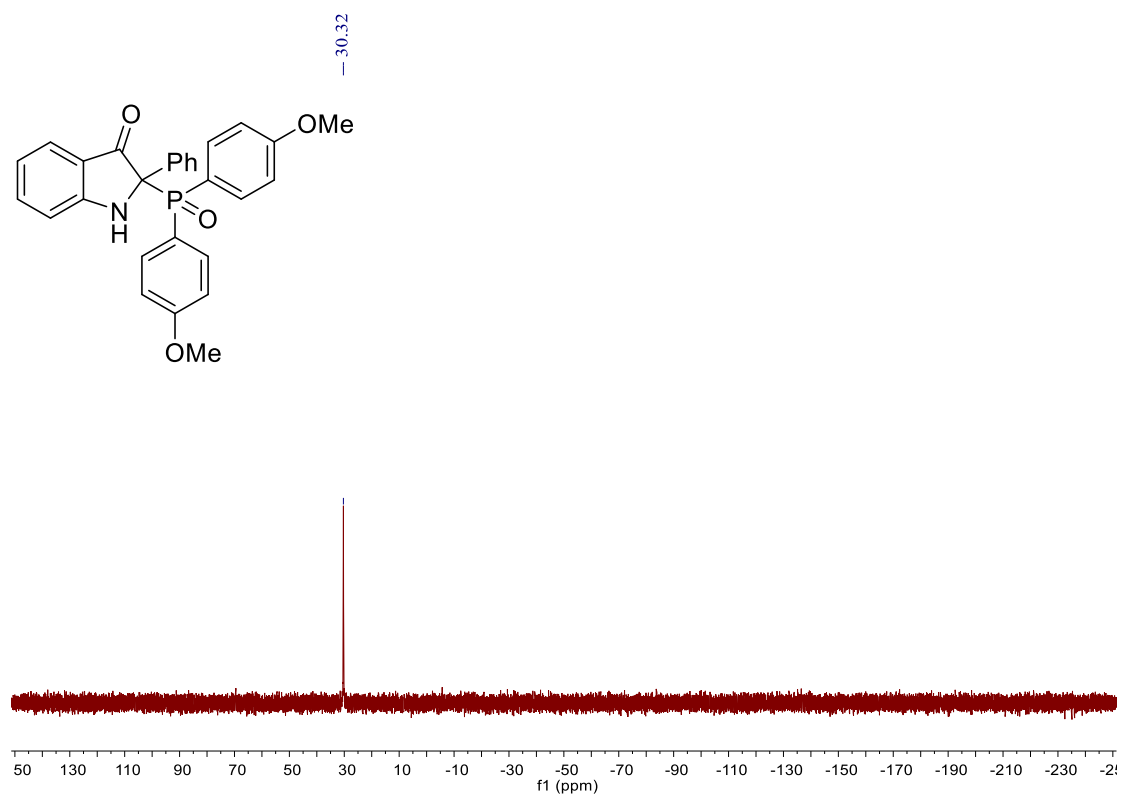
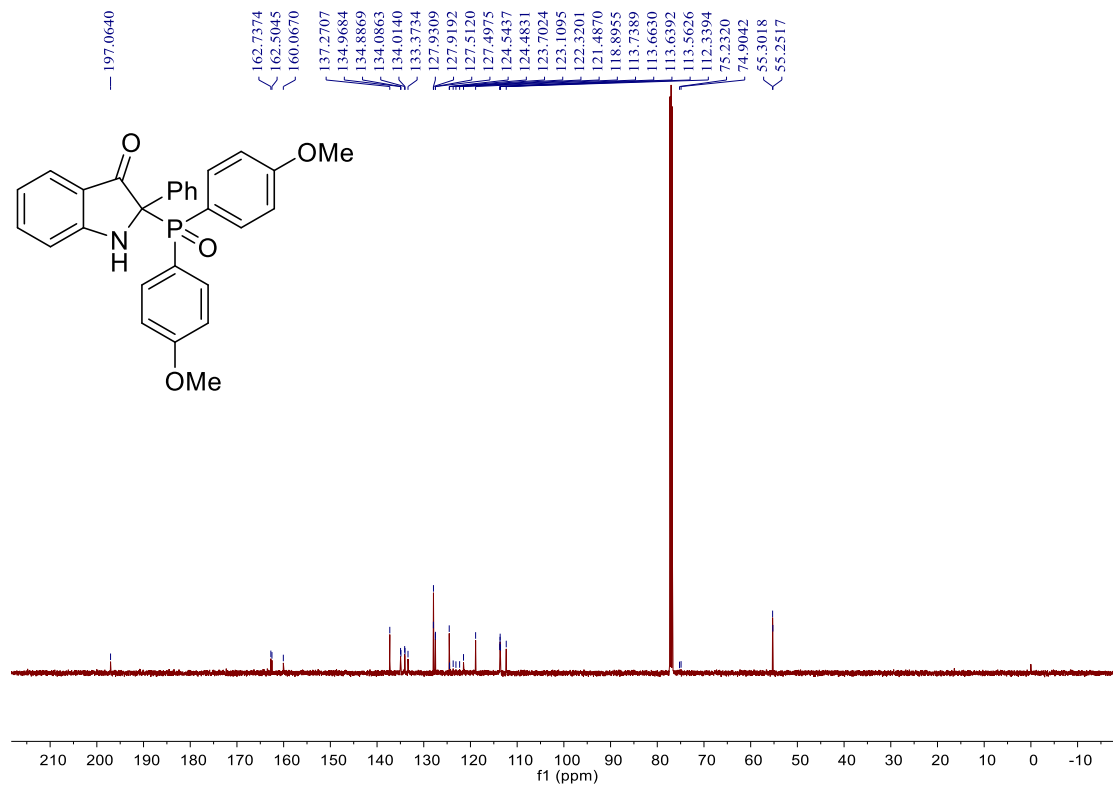
2-(tert-butyl)-2-(diphenylphosphoryl)indolin-3-one (3l).



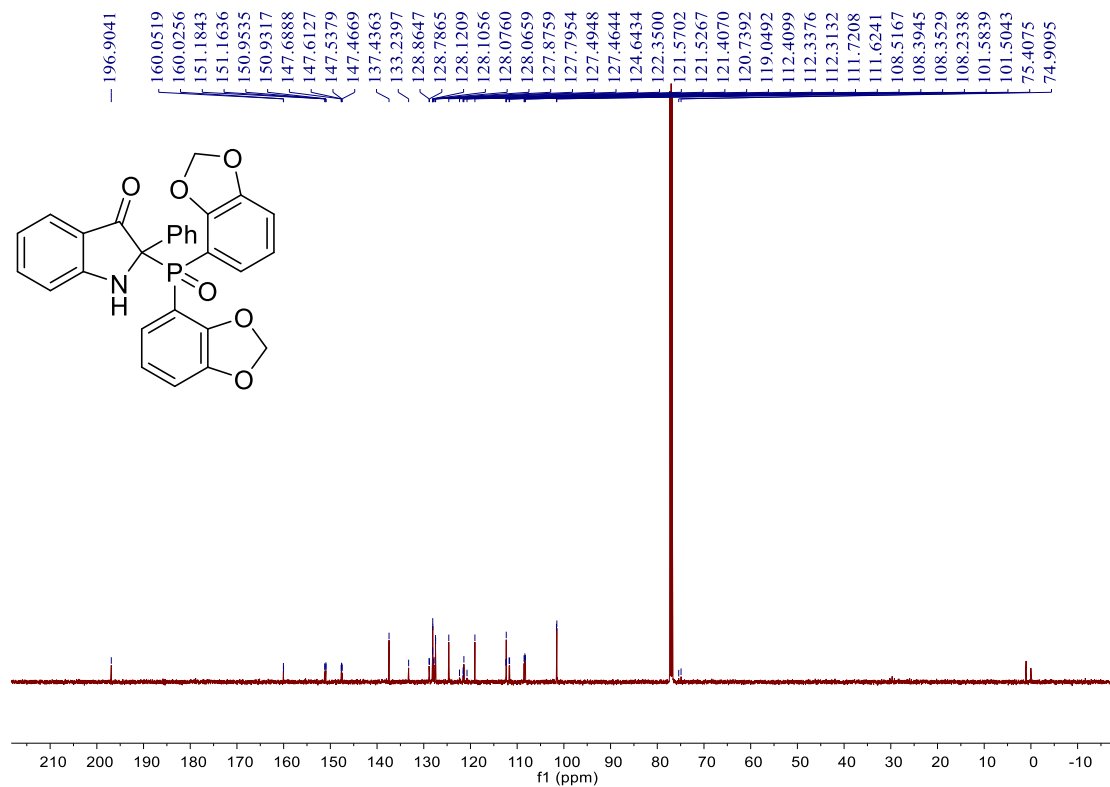
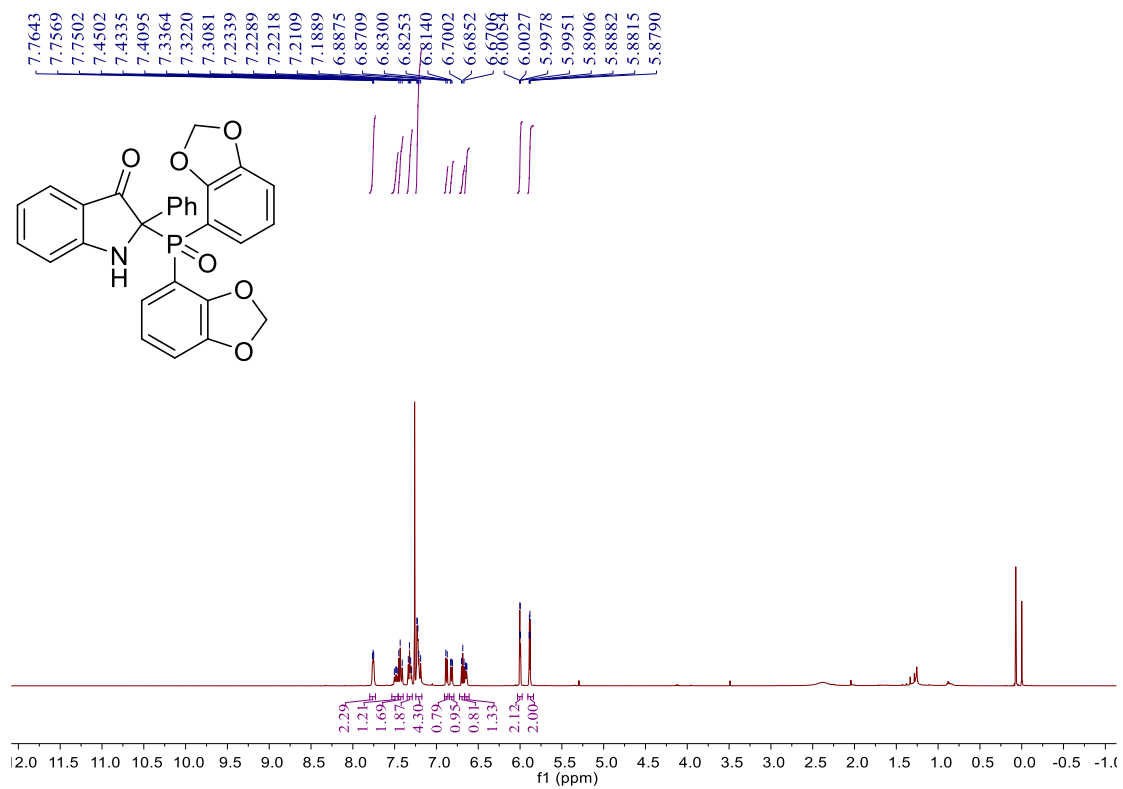


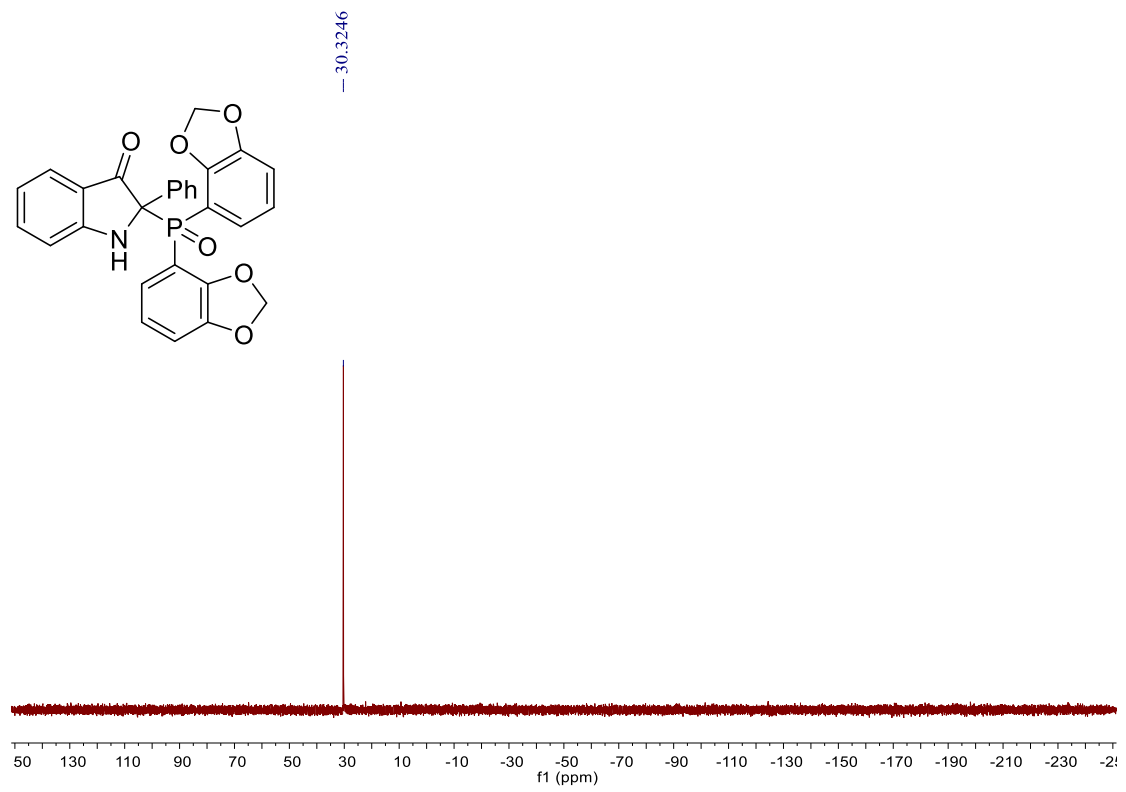
2-(bis(4-methoxyphenyl)phosphoryl)-2-phenylindolin-3-one (3m)



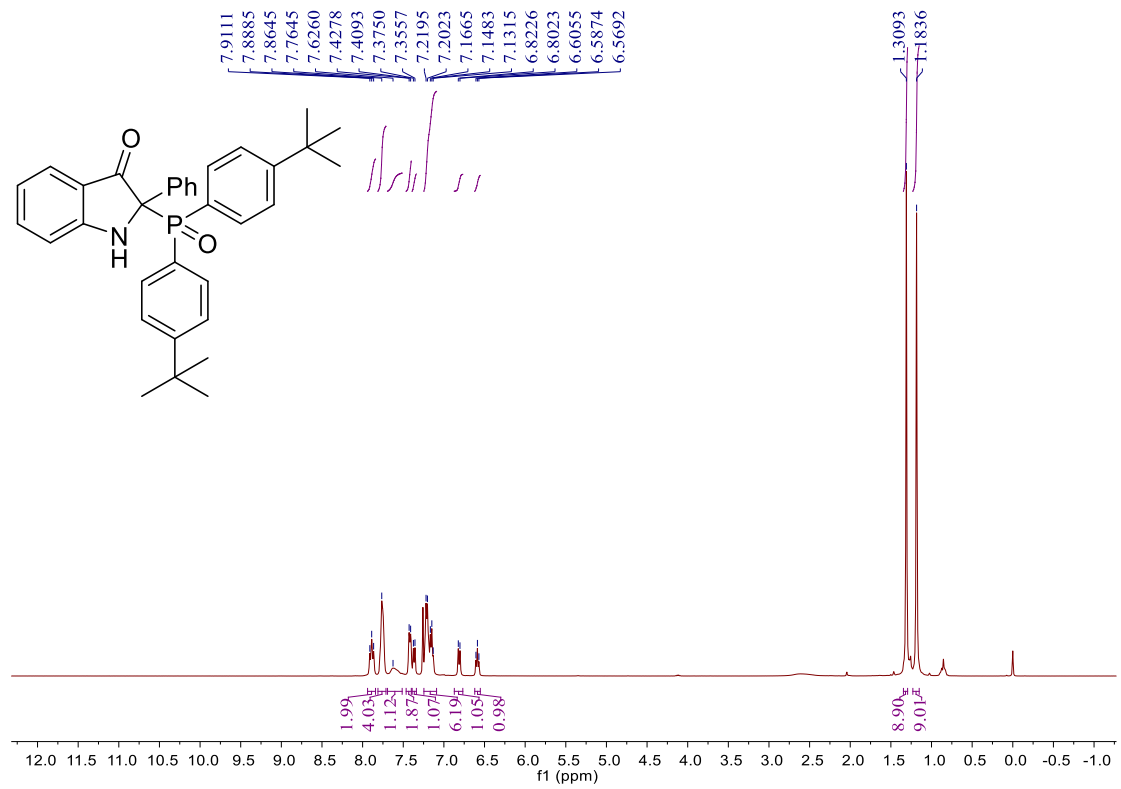


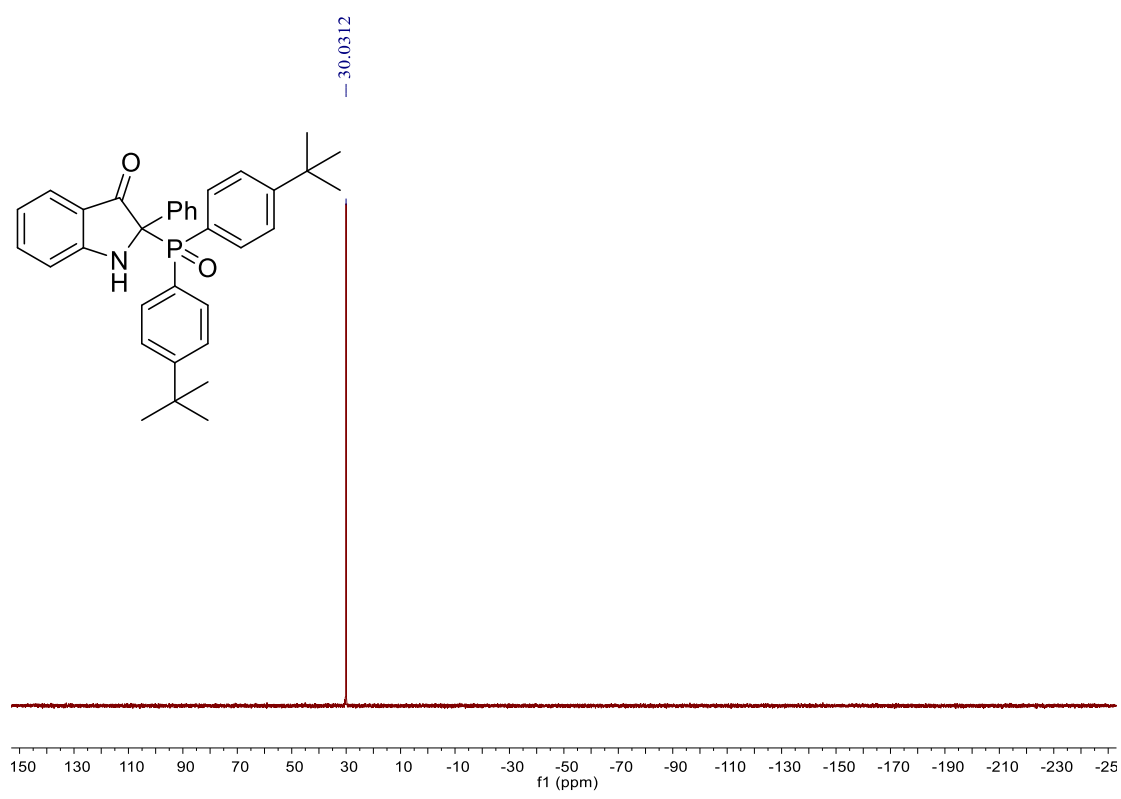
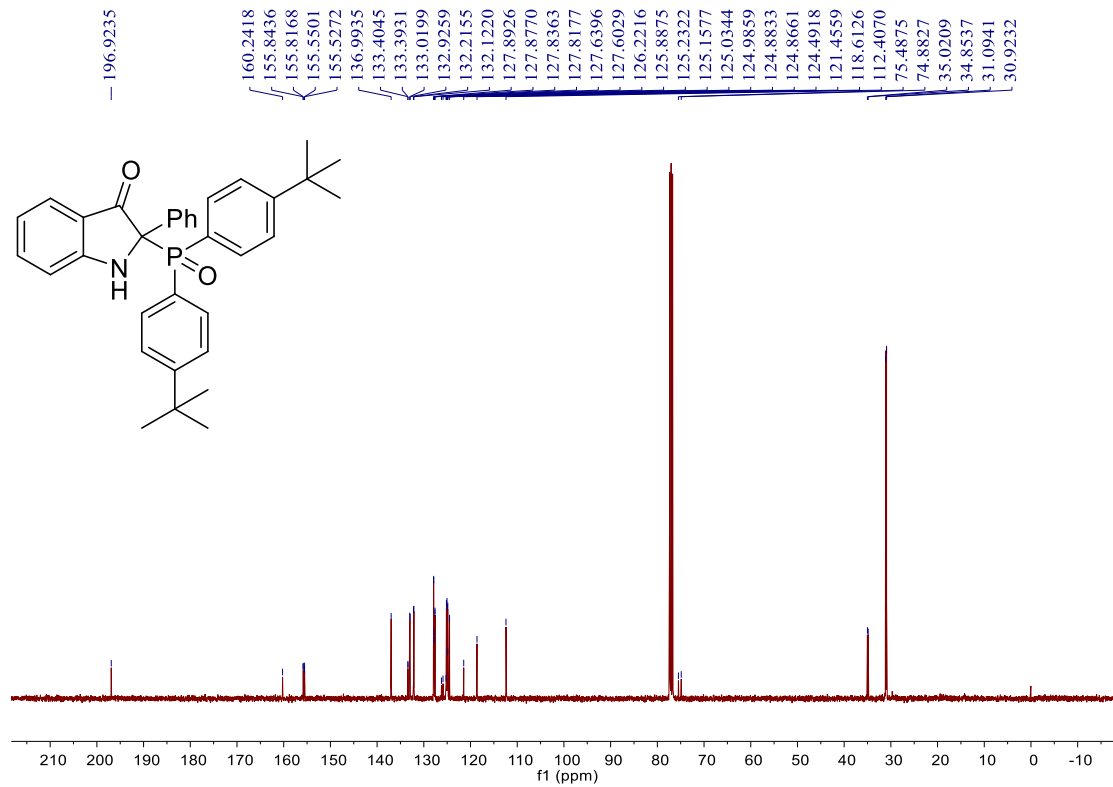
2-(bis(benzo[d][1,3]dioxol-4-yl)phosphoryl)-2-phenylindolin-3-one (3n)



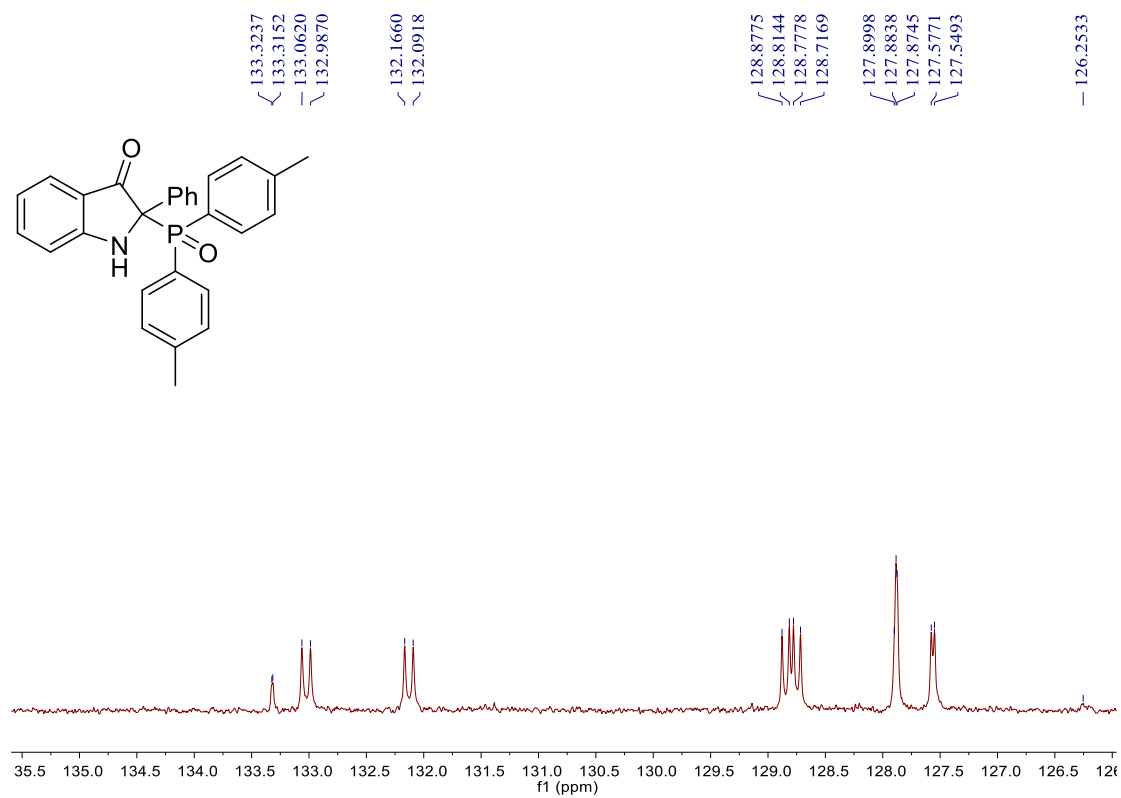
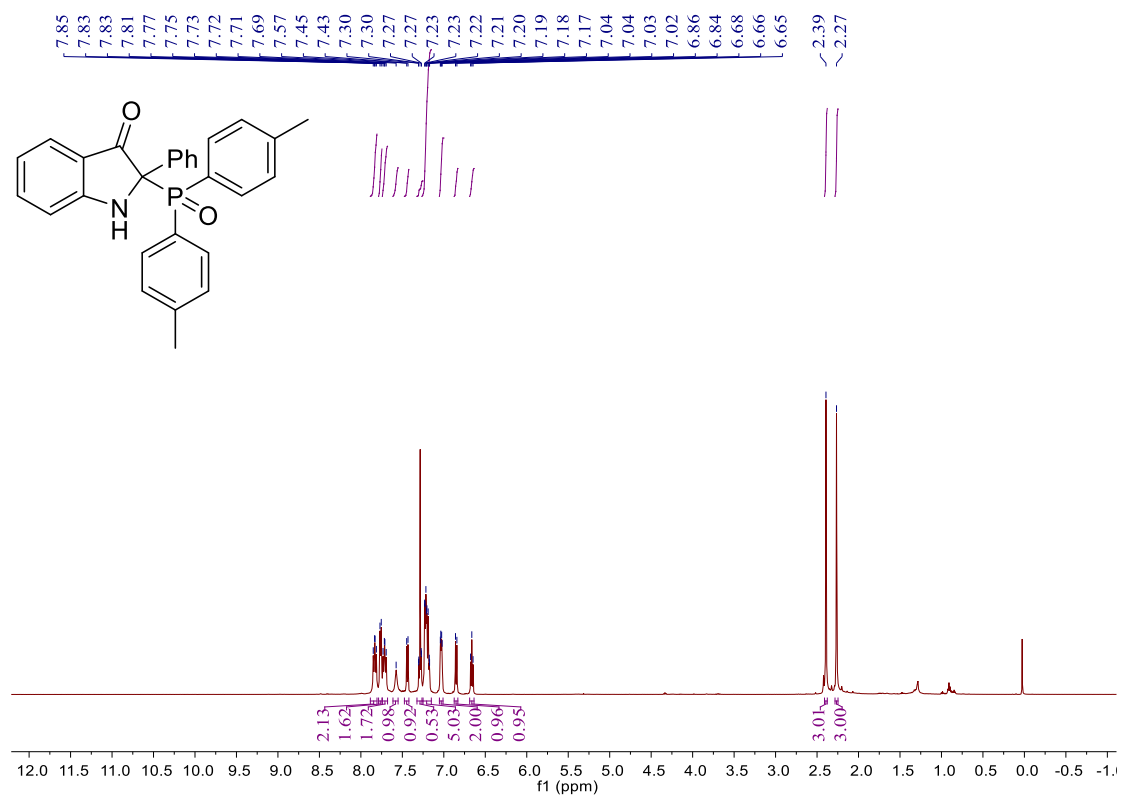


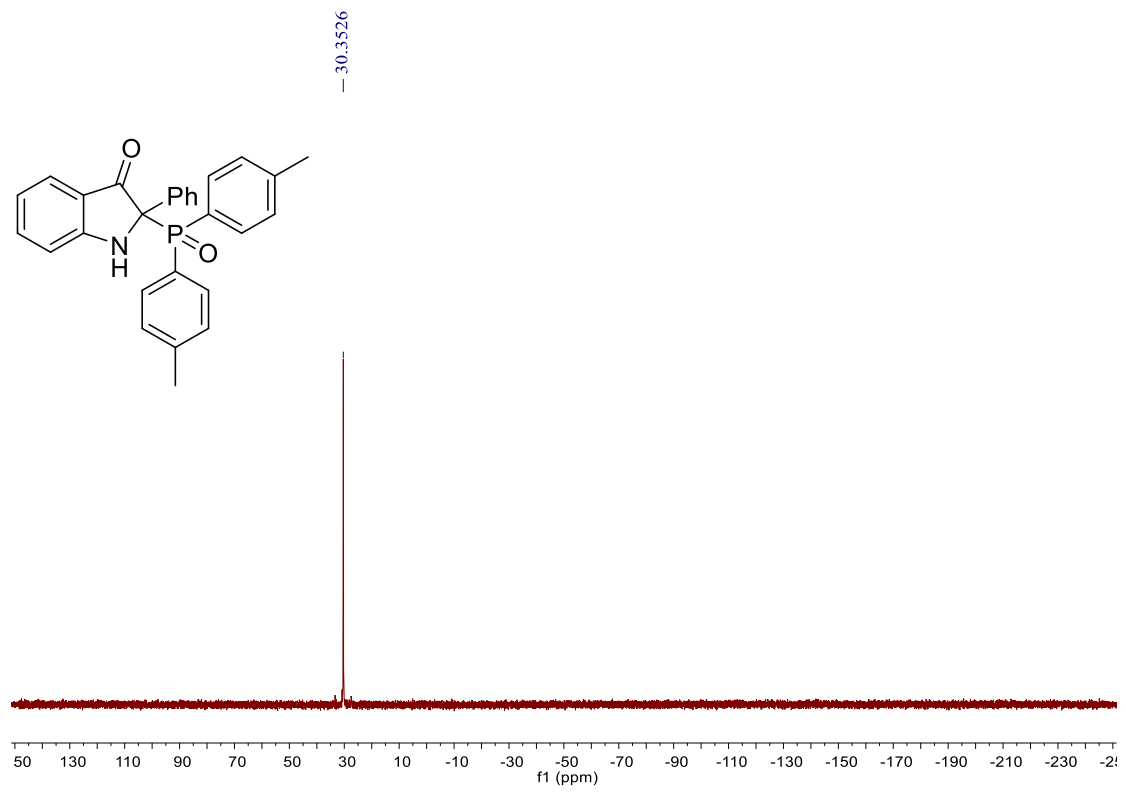
2-(bis(4-(tert-butyl)phenyl)phosphoryl)-2-phenylindolin-3-one (3o)



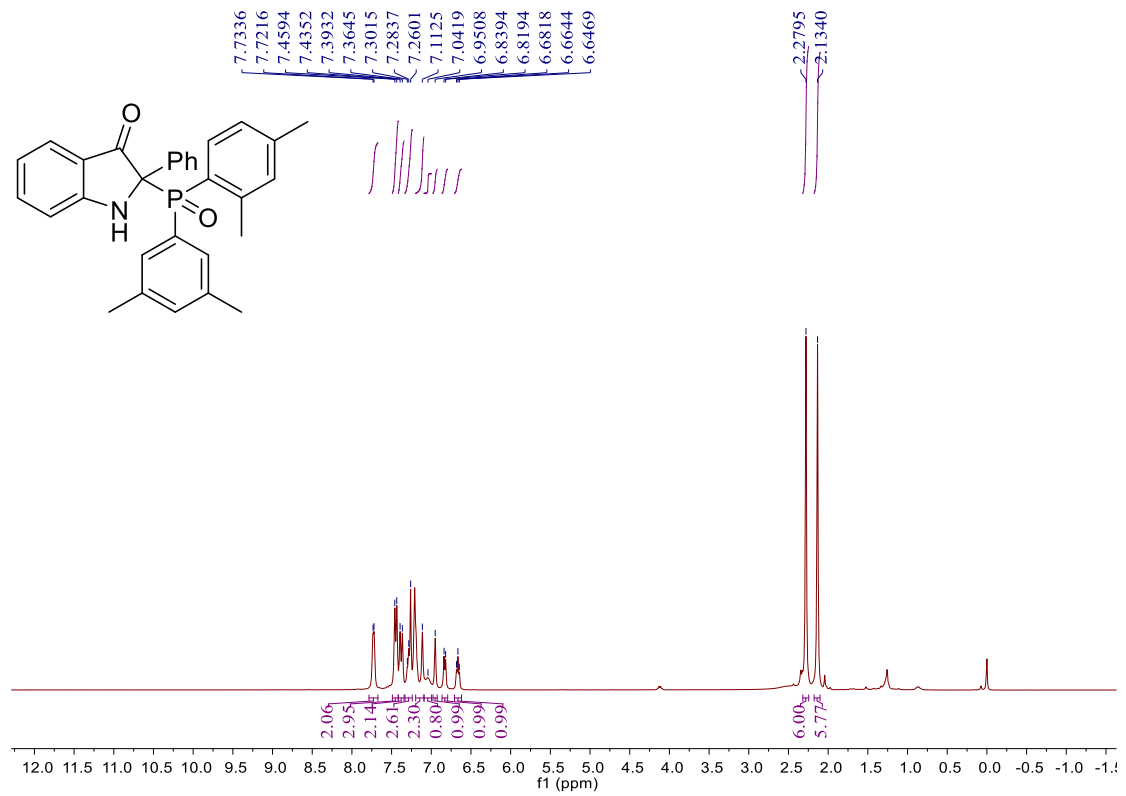


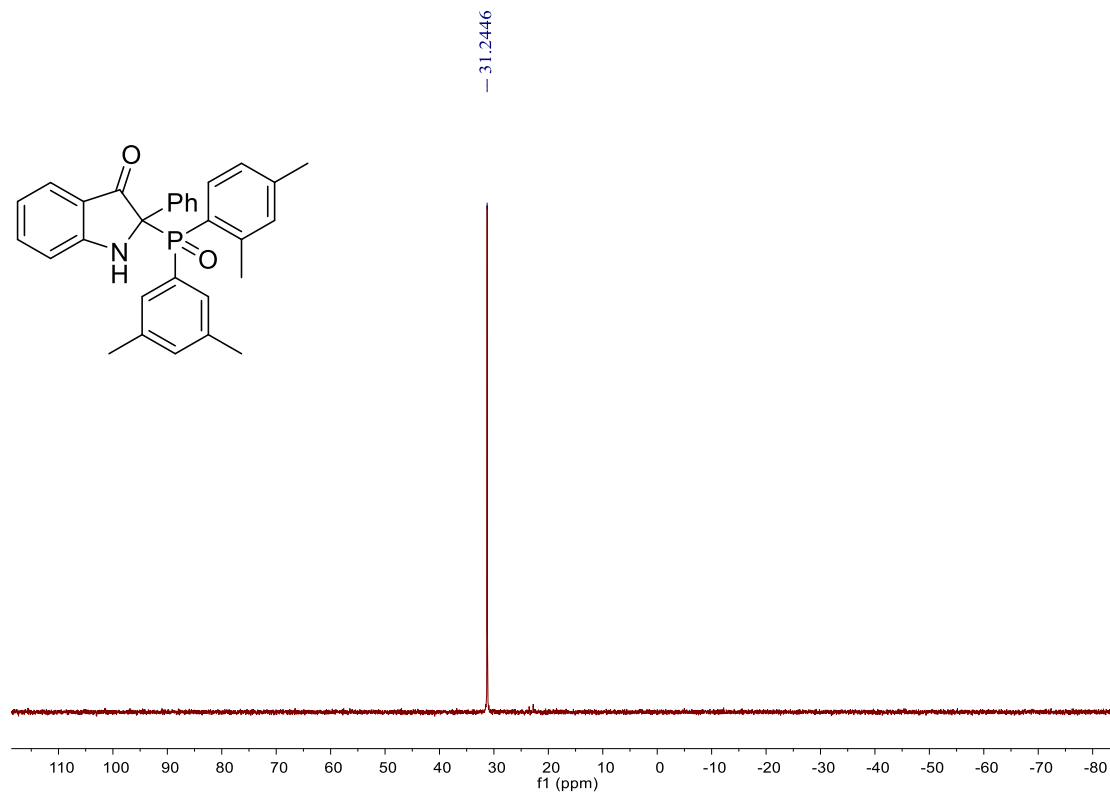
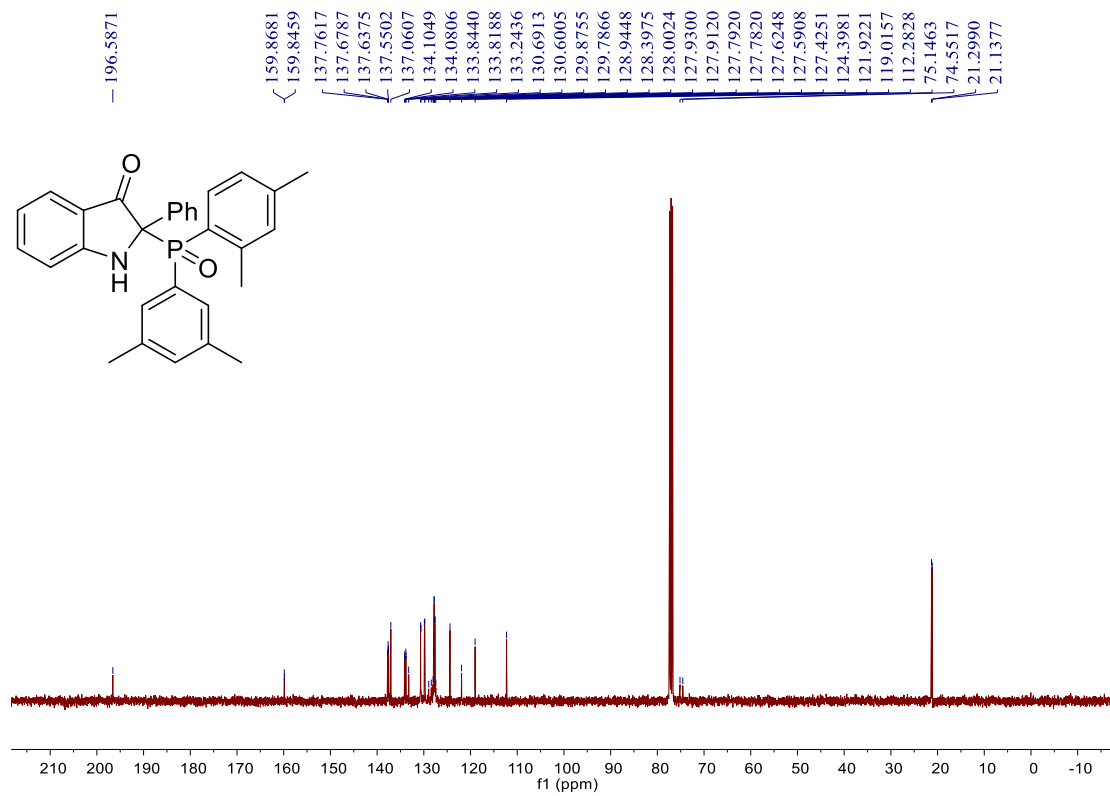
2-(di-p-tolylphosphoryl)-2-phenylindolin-3-one (3p)



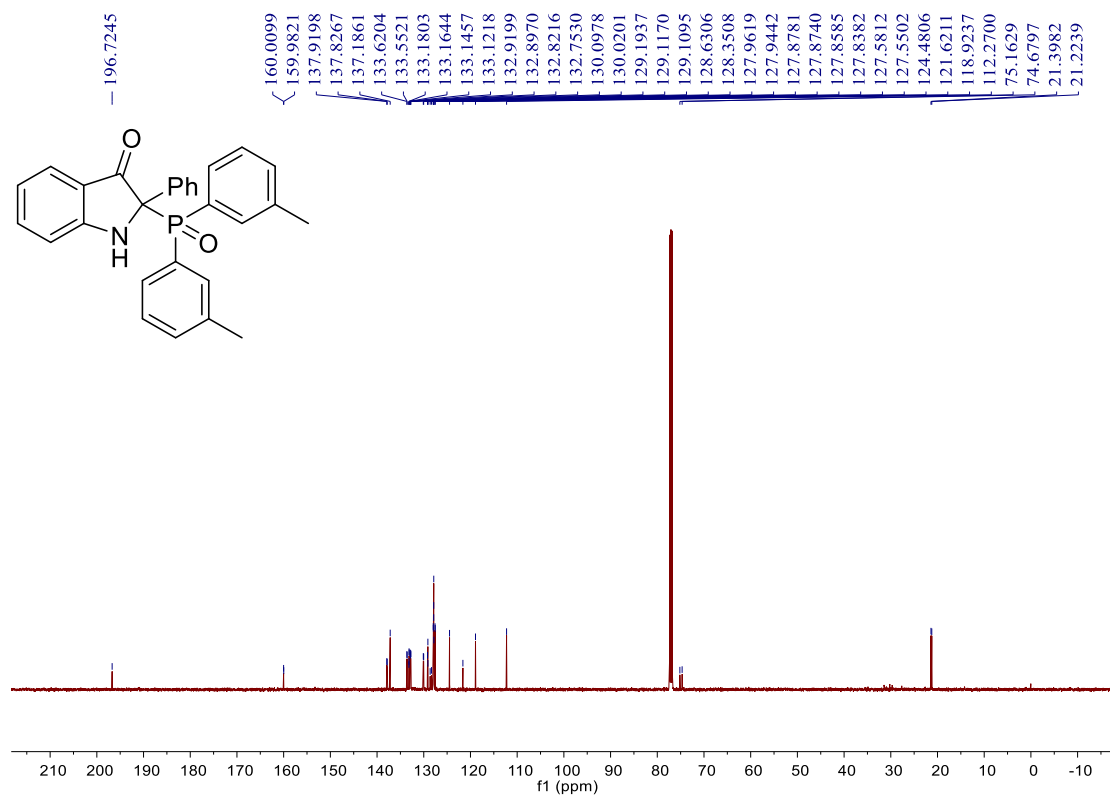
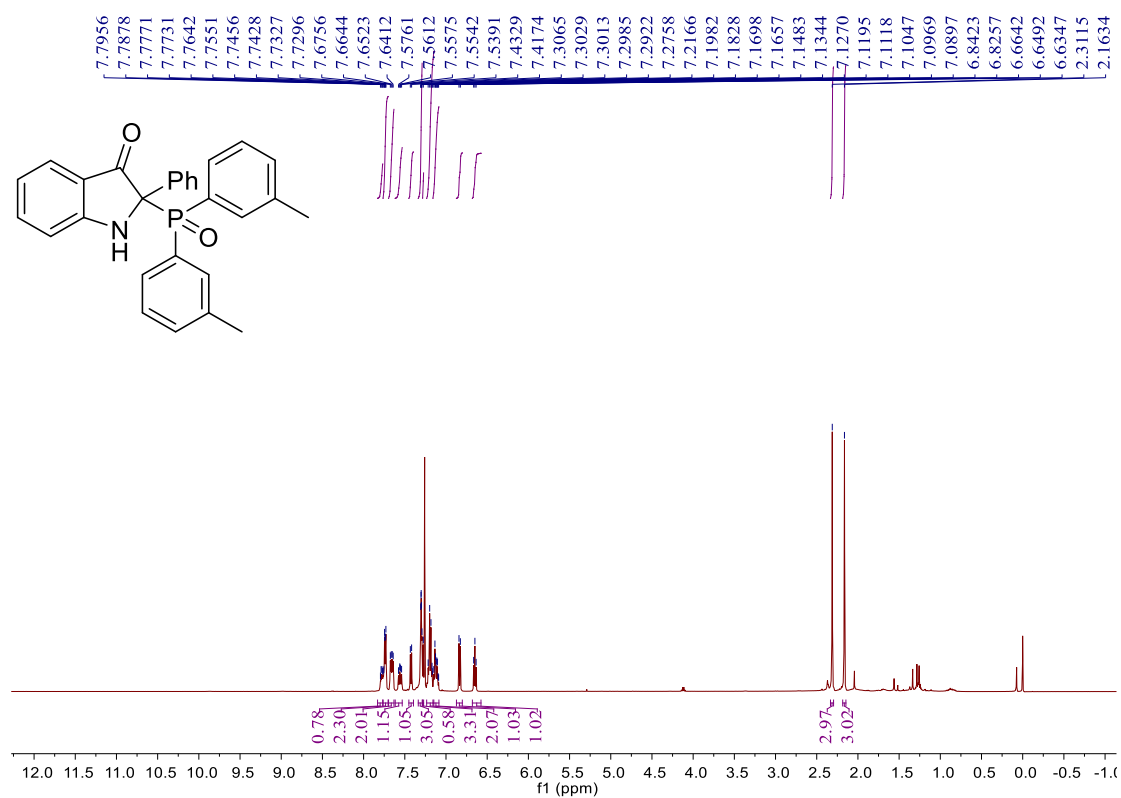


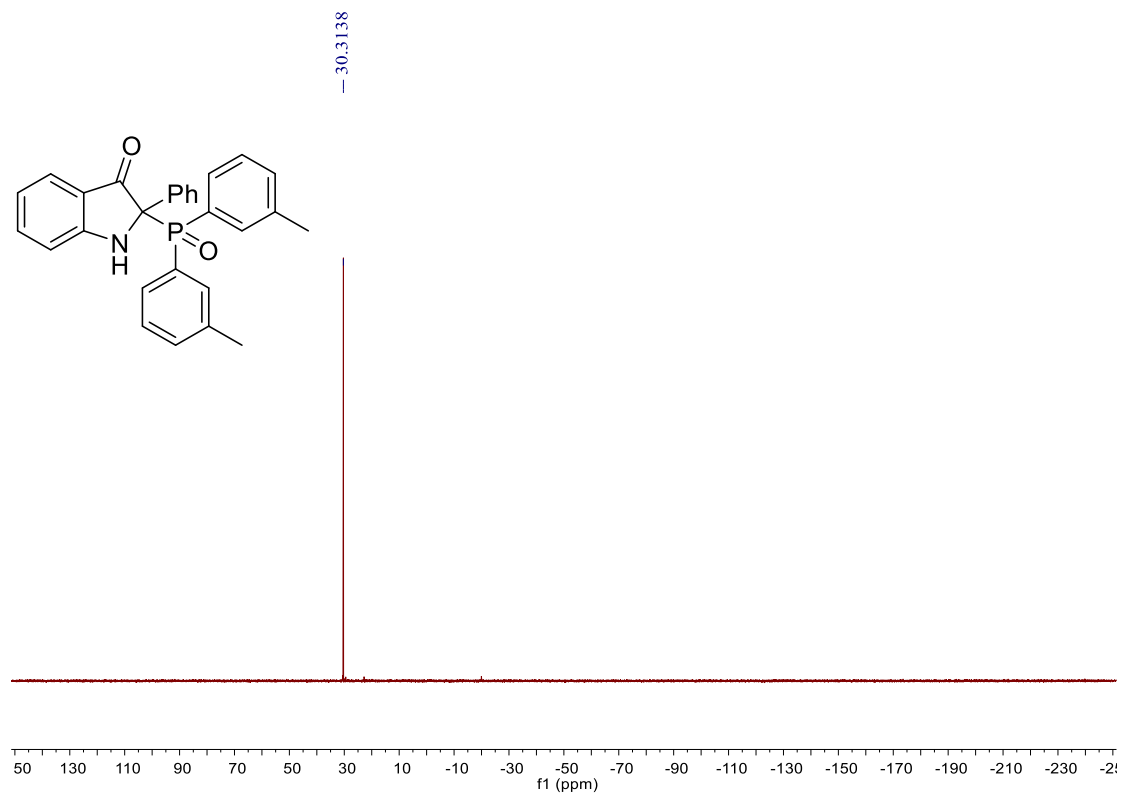
2-((2,4-dimethylphenyl)(3,5-dimethylphenyl)phosphoryl)-2-phenylindolin-3-one (3q)



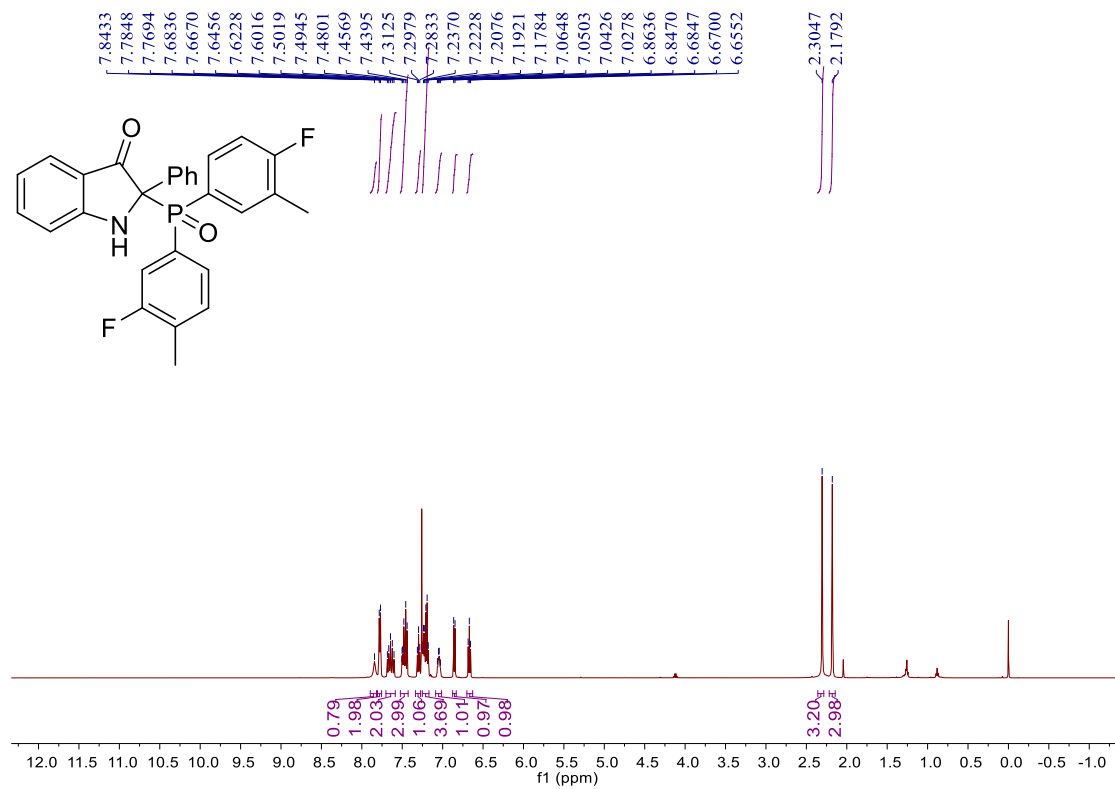


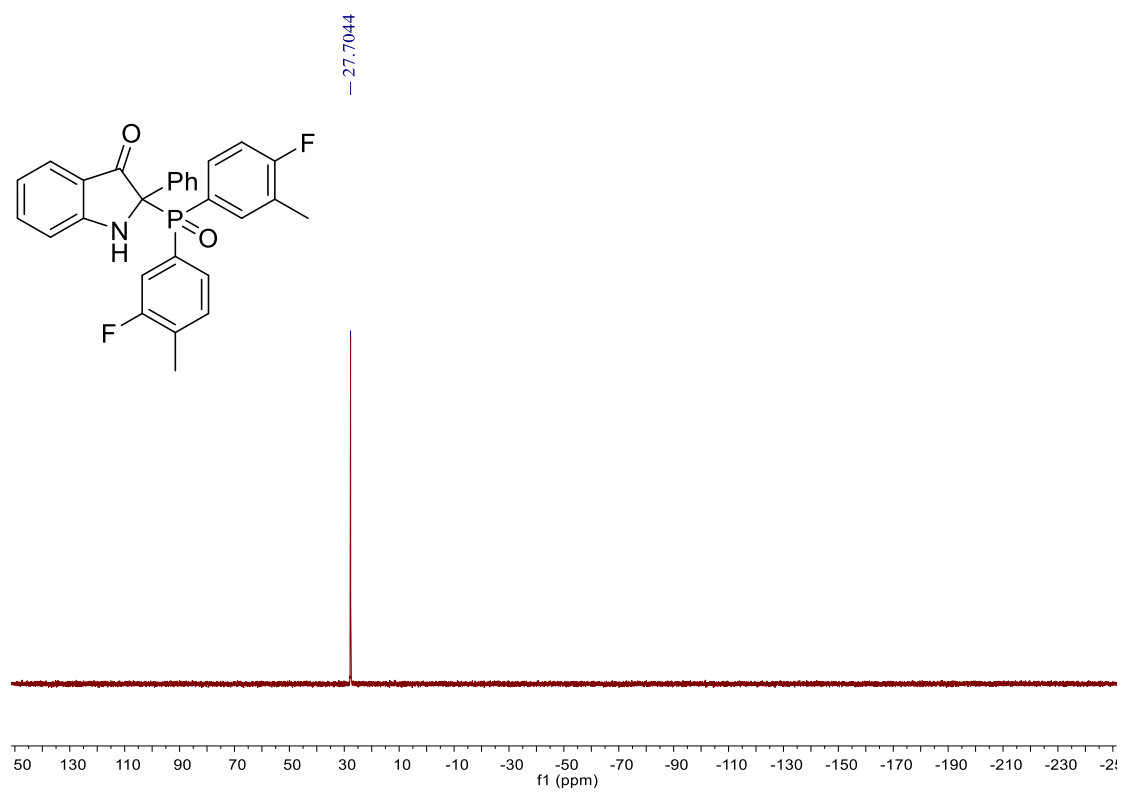
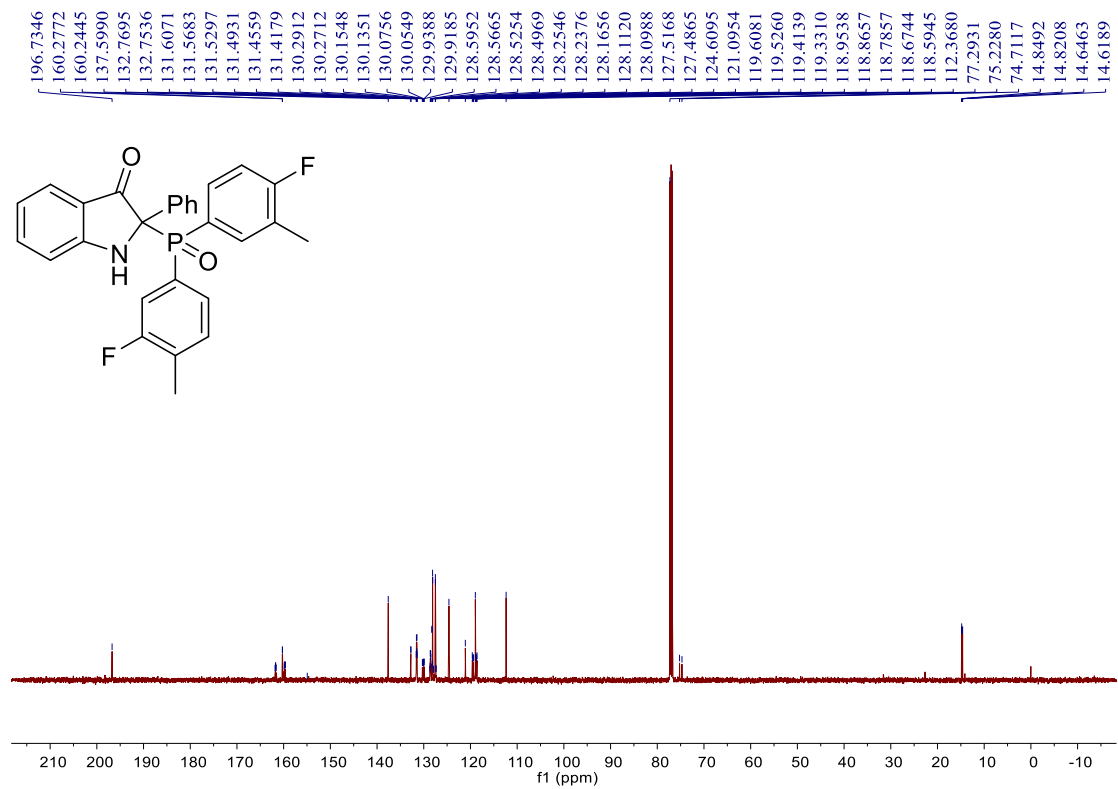
2-(di-m-tolylphosphoryl)-2-phenylindolin-3-one (3r)

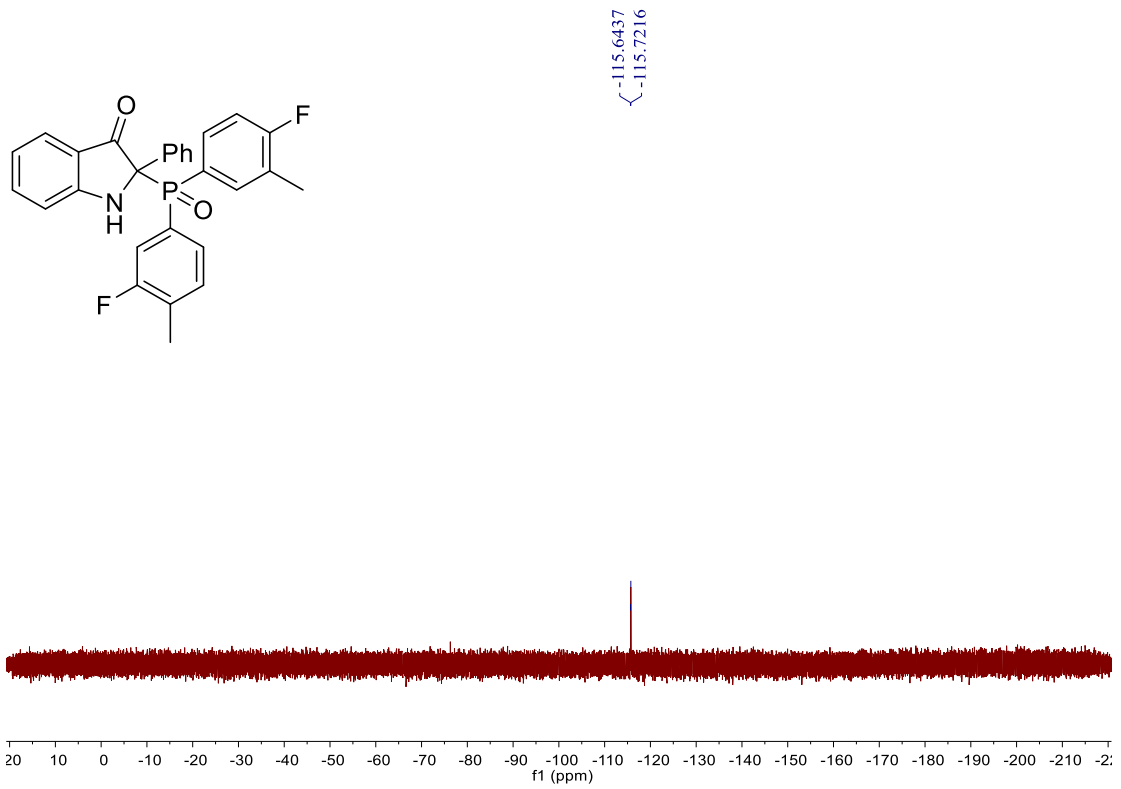




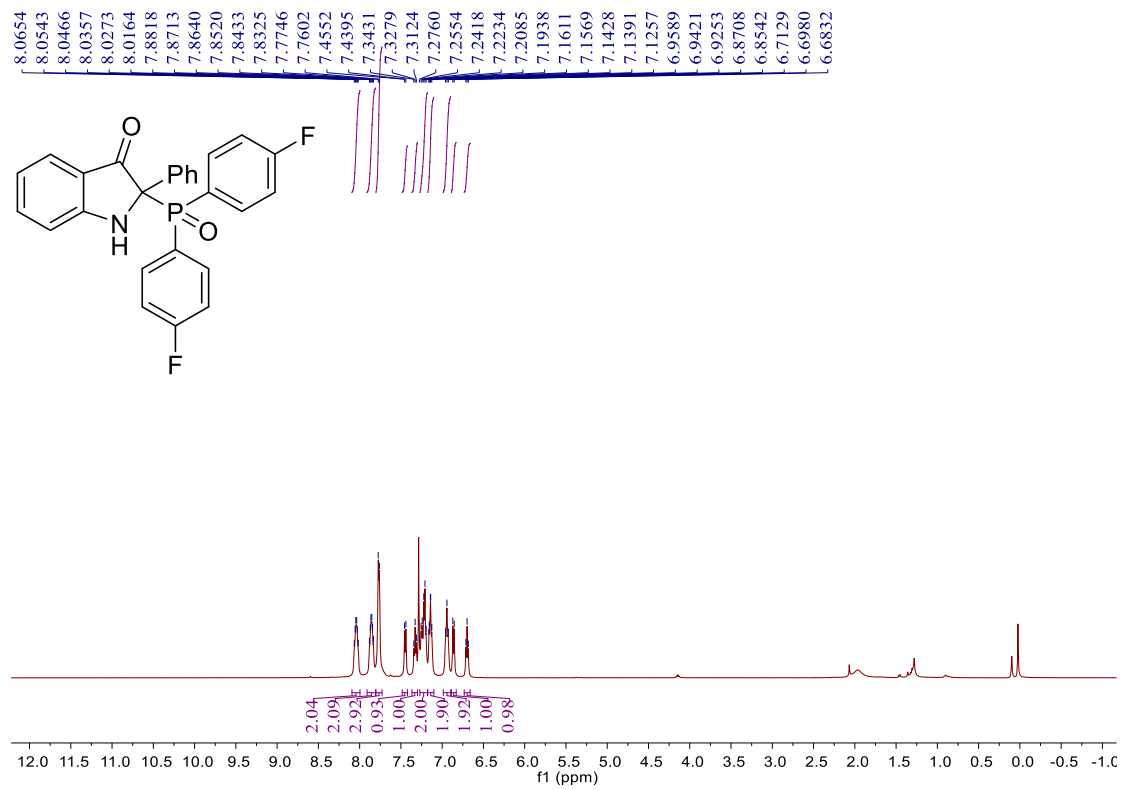
2-((4-fluoro-3-methylphenyl)(3-fluoro-4-methylphenyl)phosphoryl)-2-phenylindolin-3-one (3s)

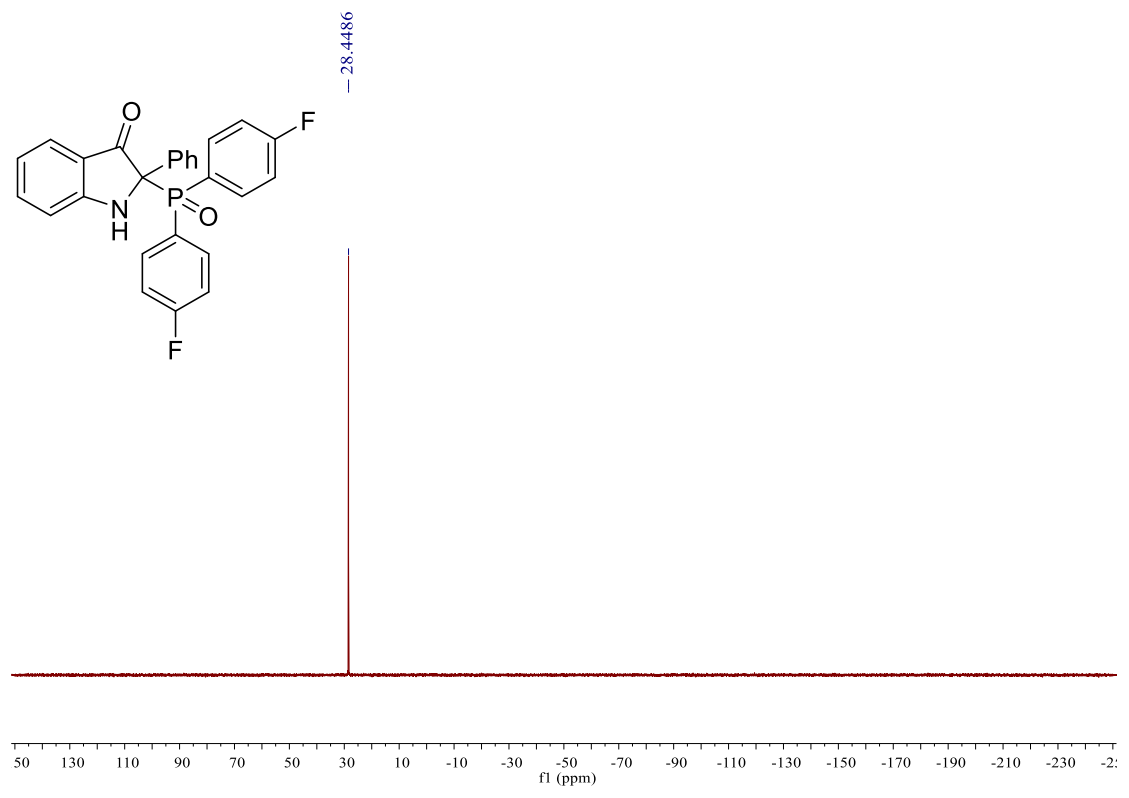
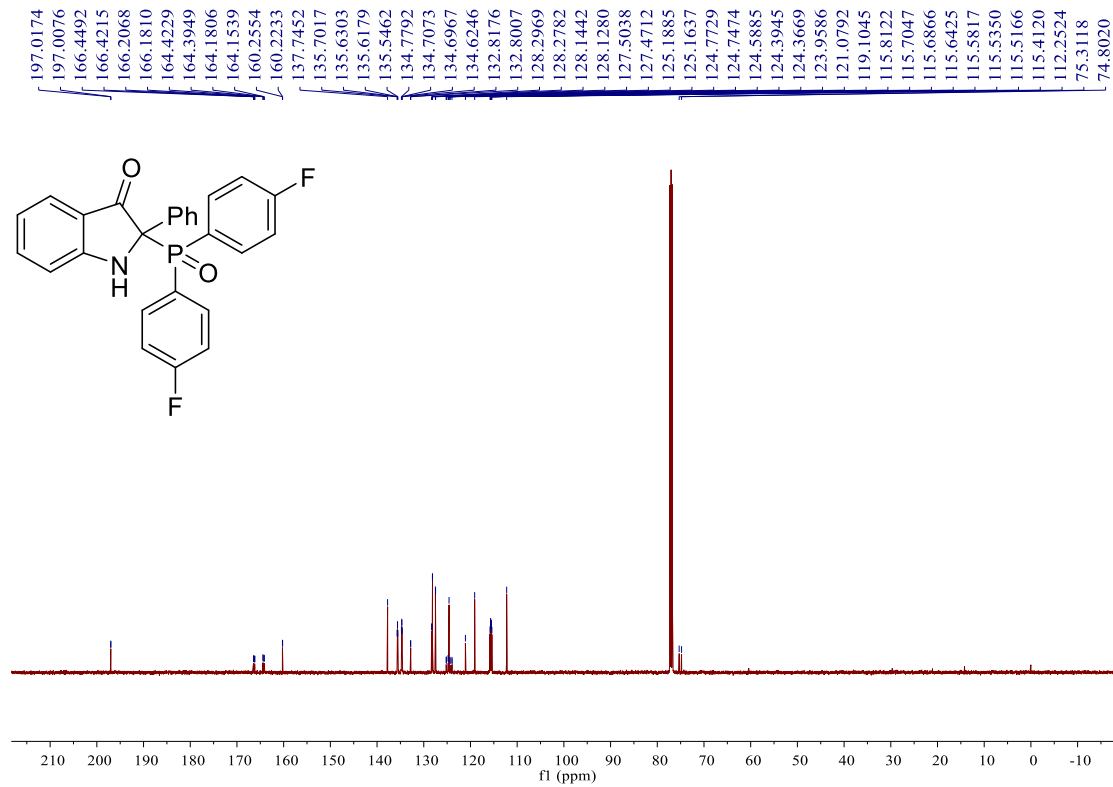


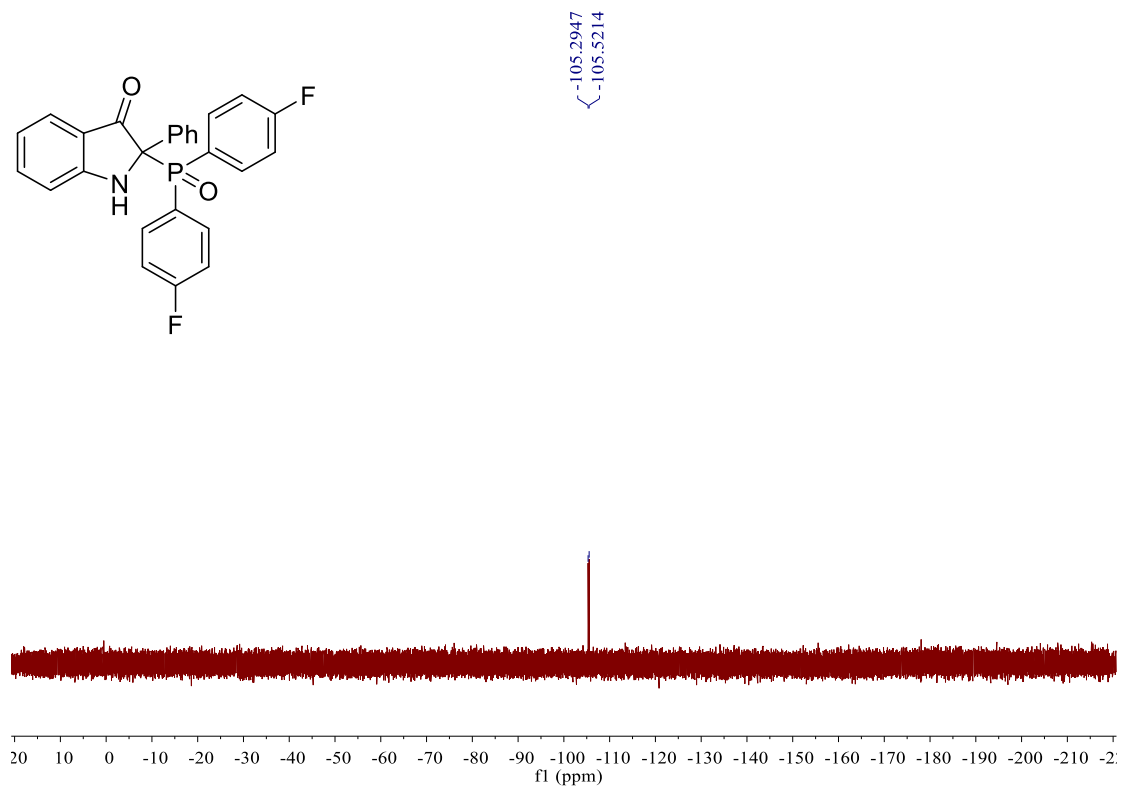




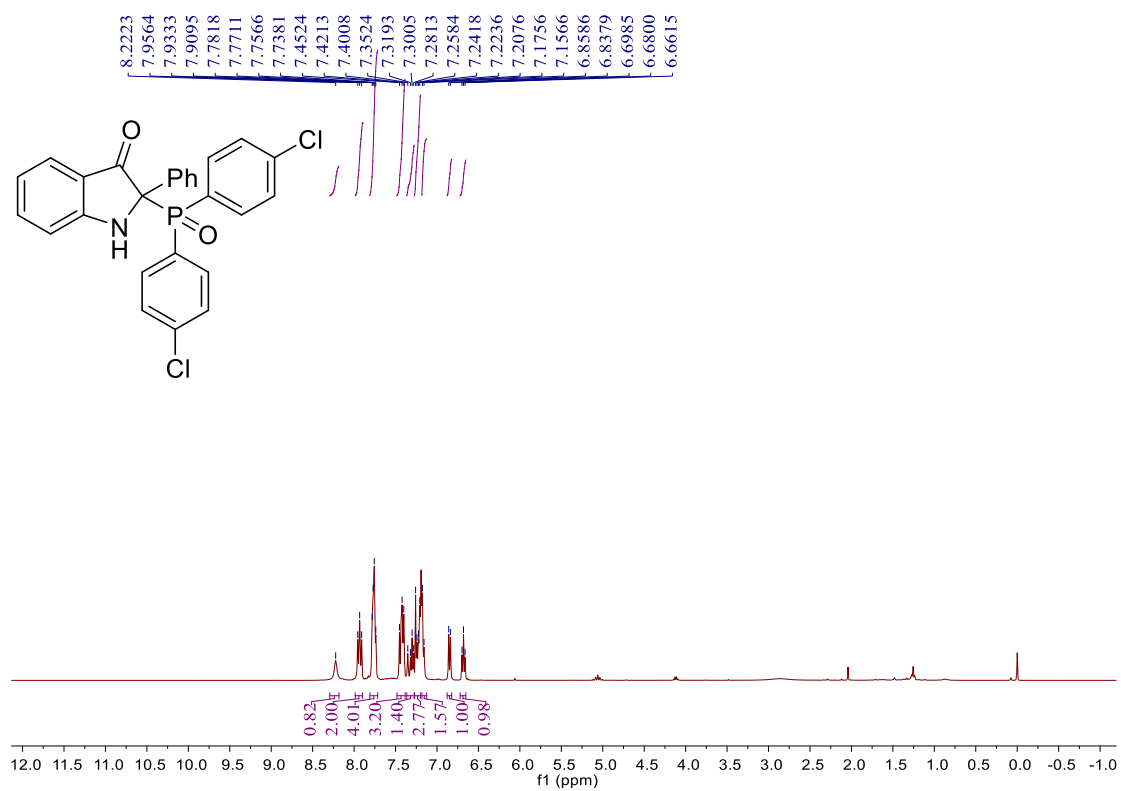
2-(bis(4-fluorophenyl)phosphoryl)-2-phenylindolin-3-one (3t)

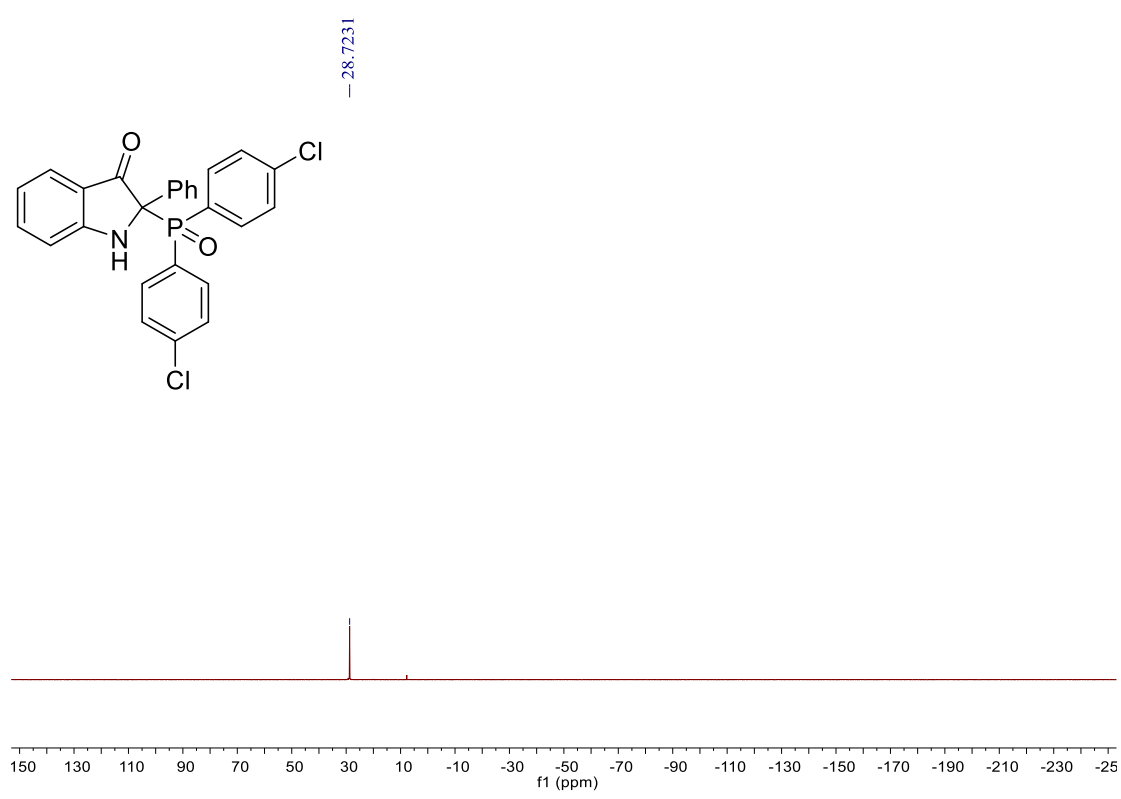
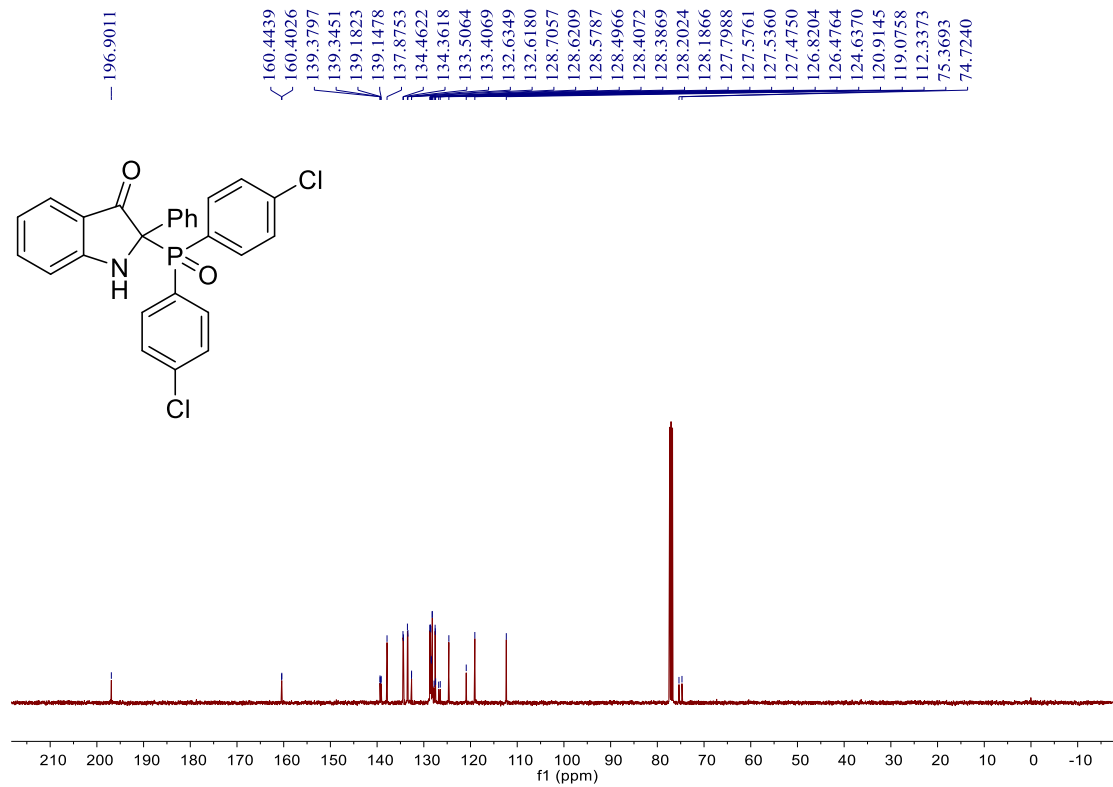




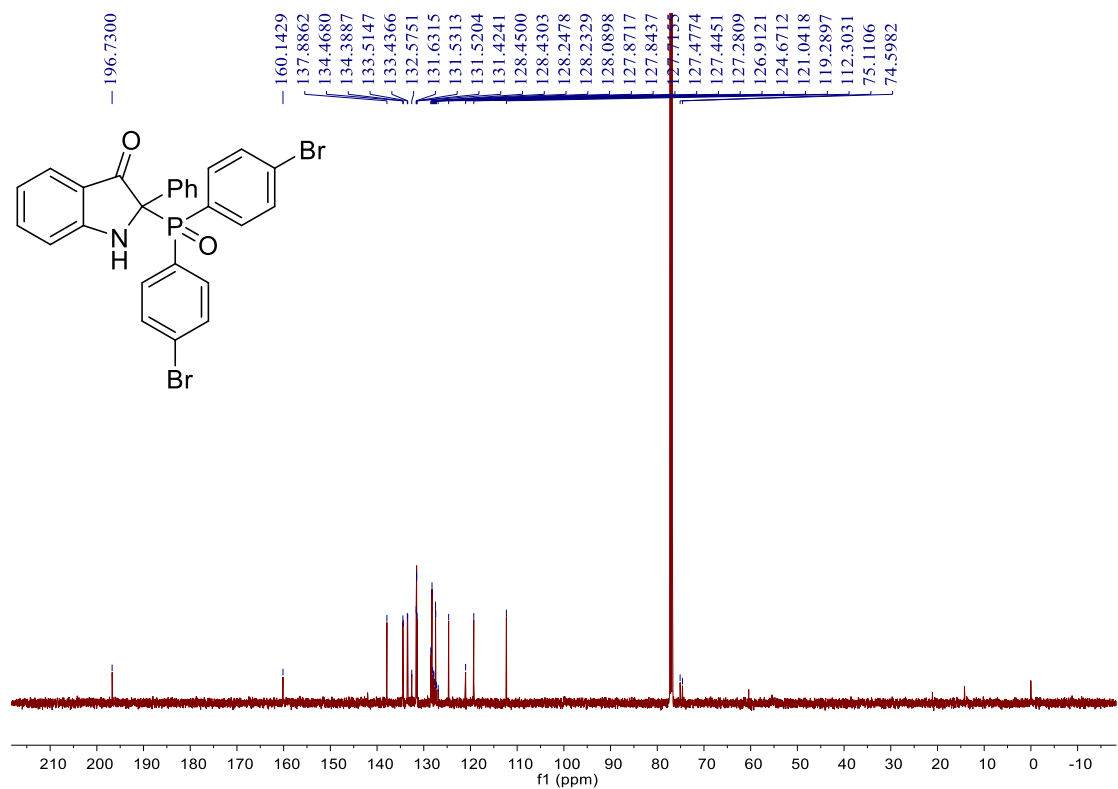
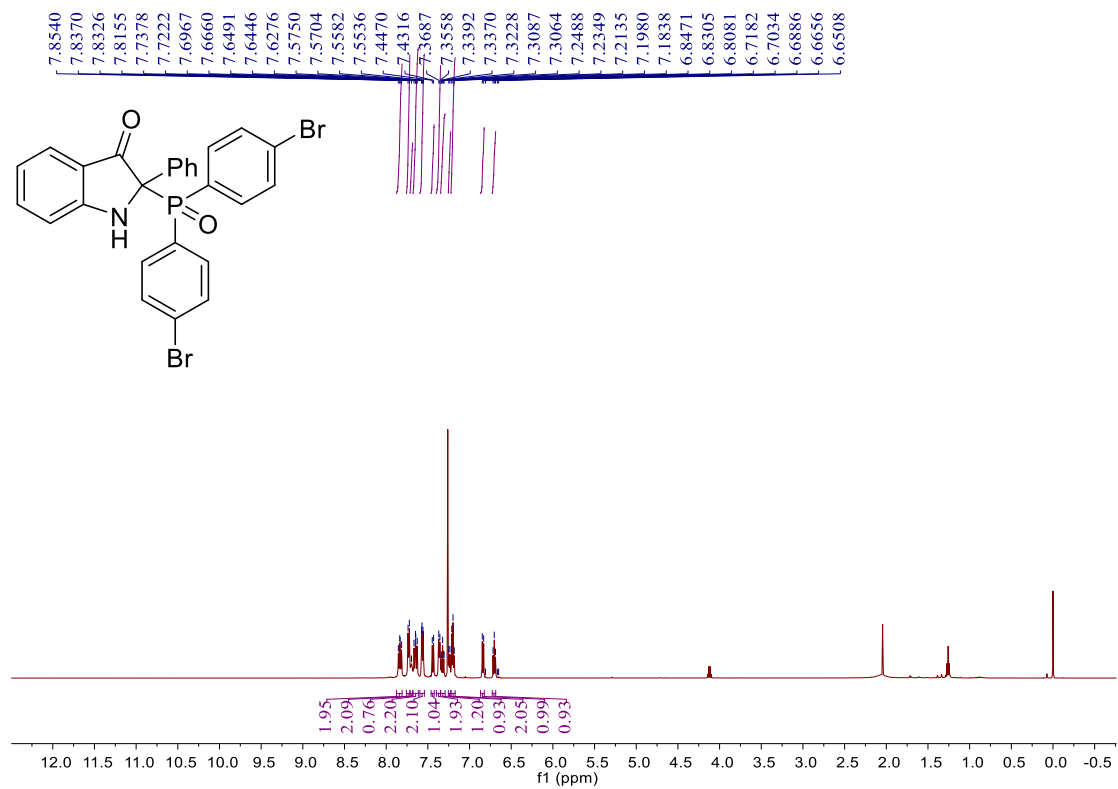


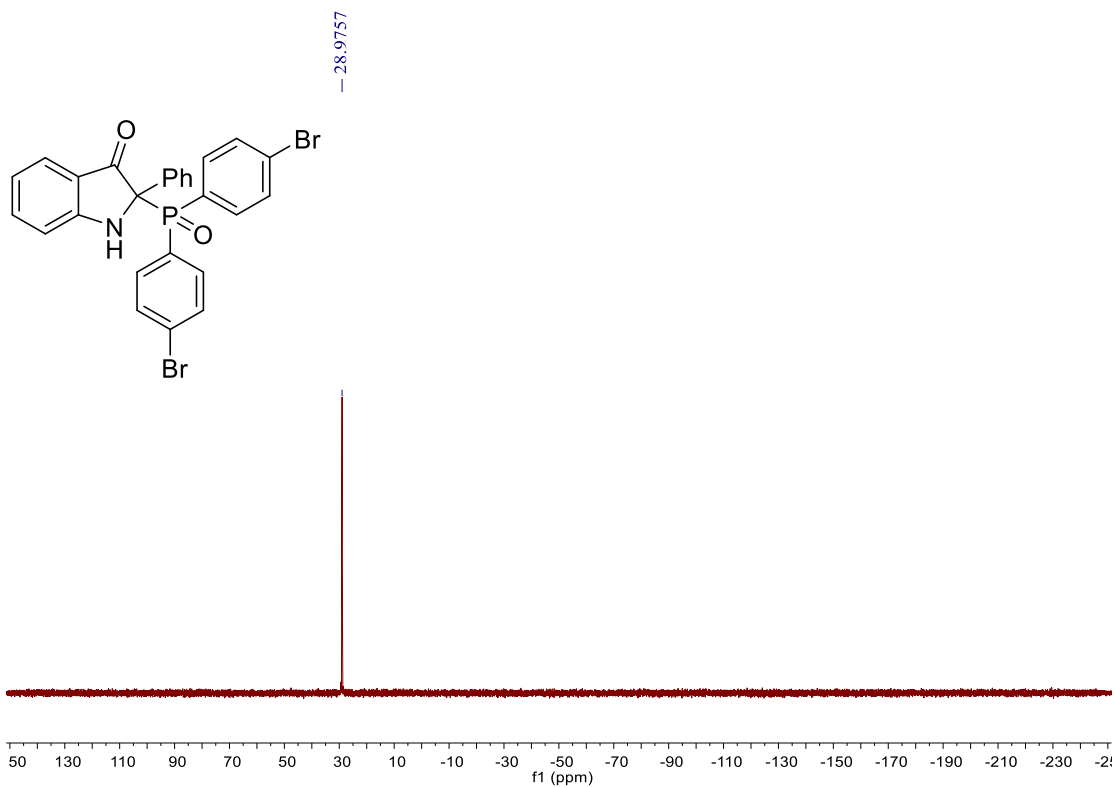
2-(bis(4-chlorophenyl)phosphoryl)-2-phenylindolin-3-one (3u)



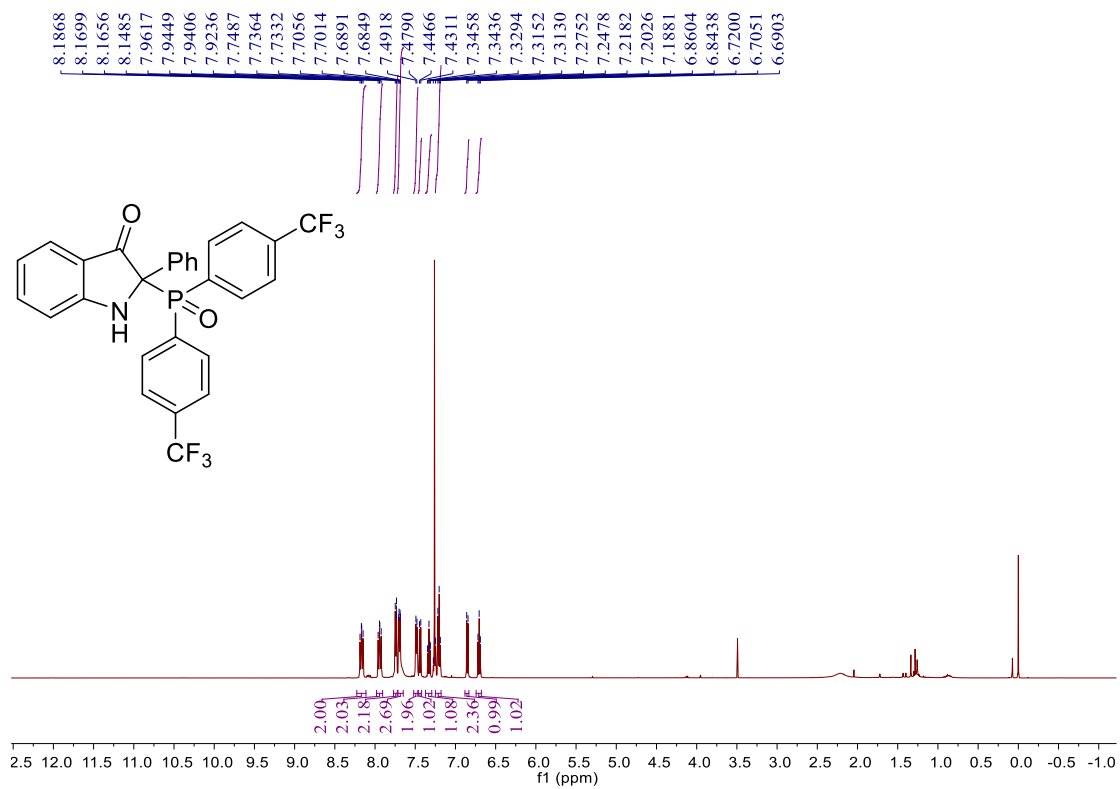


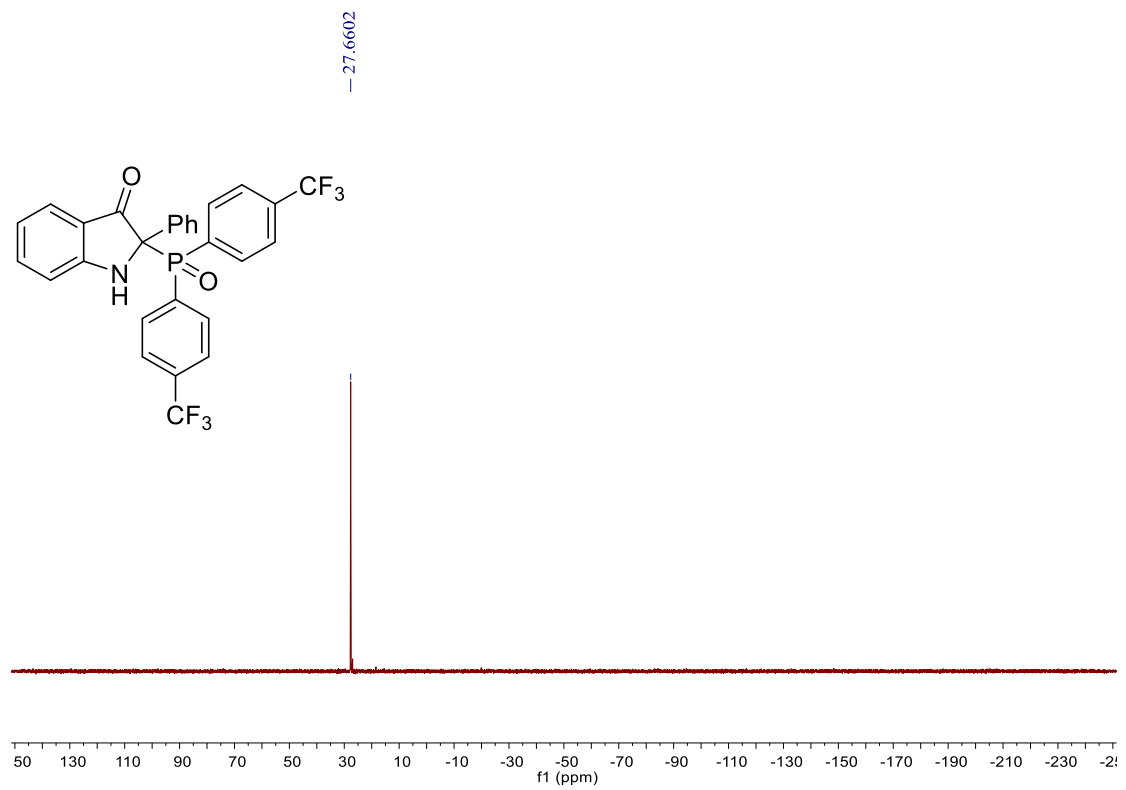
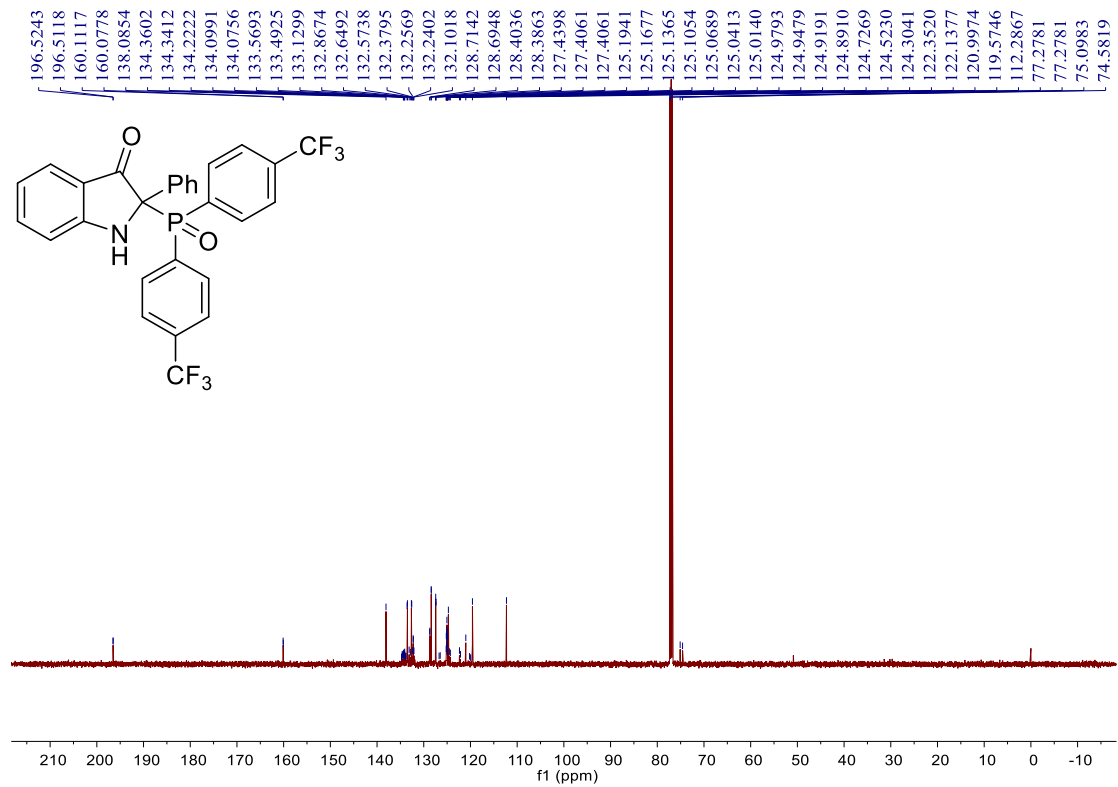
2-(bis(4-bromophenyl)phosphoryl)-2-phenylindolin-3-one (3v)

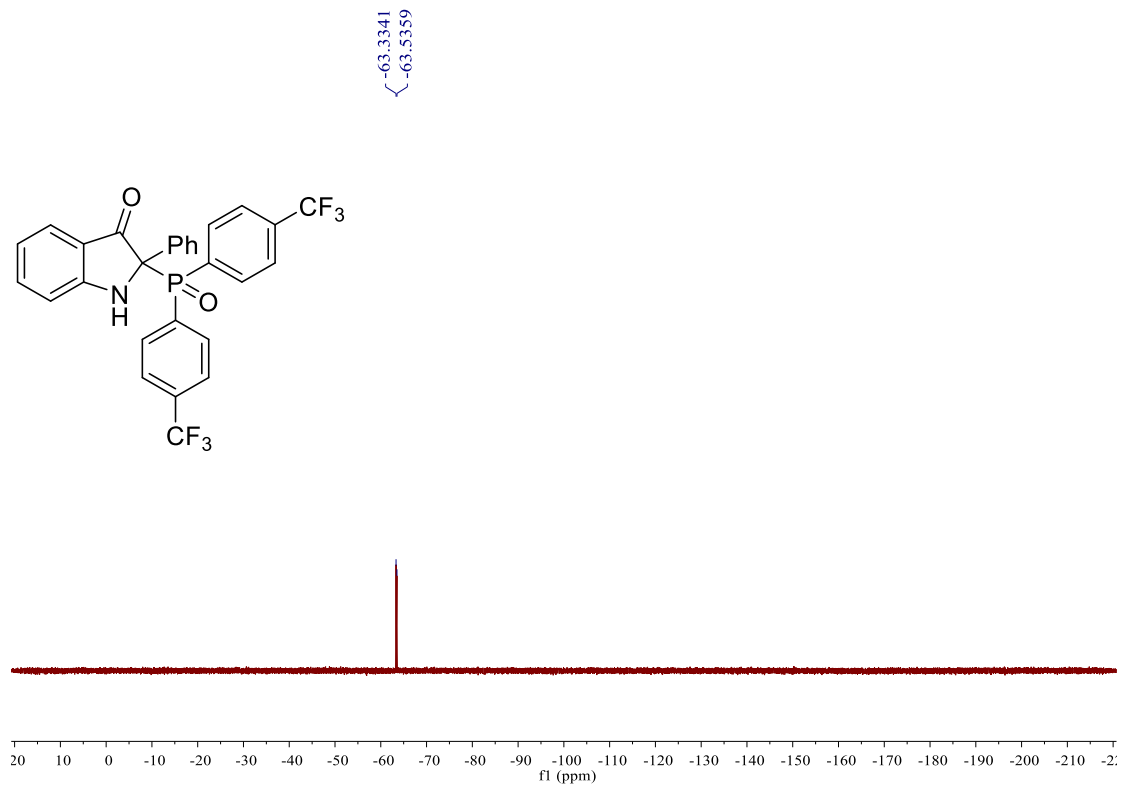




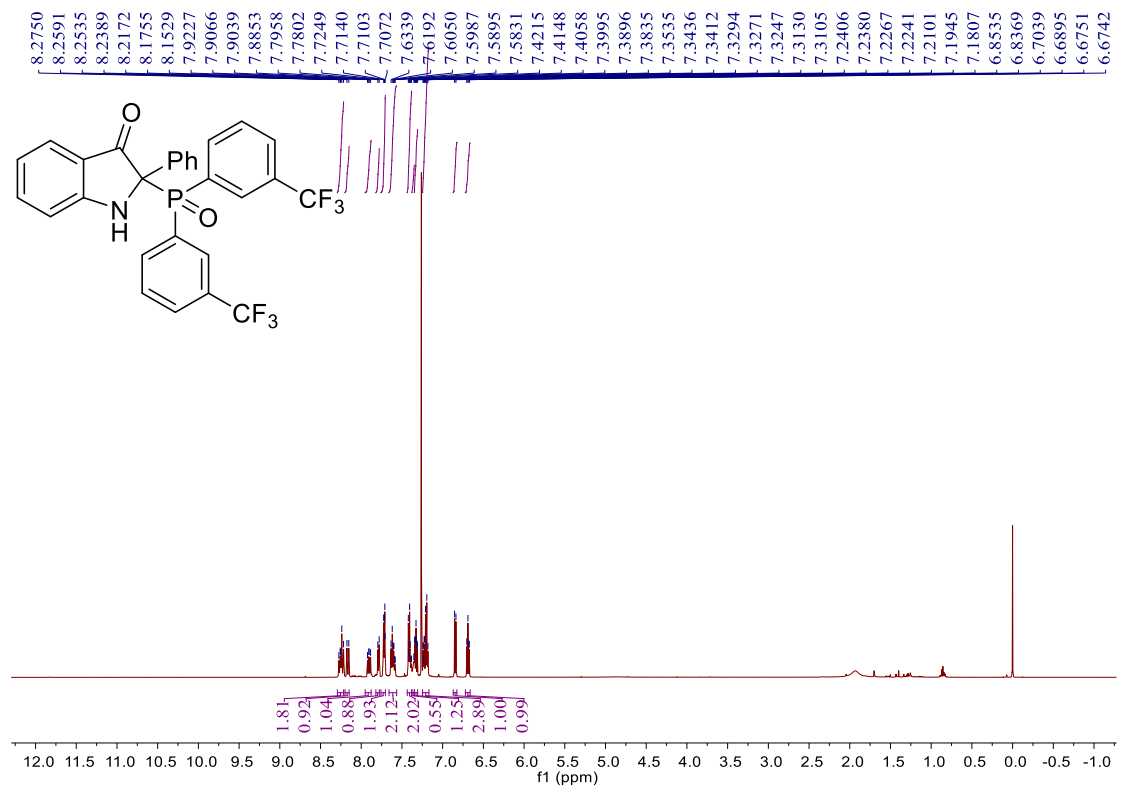
2-(bis(4-(trifluoromethyl)phenyl)phosphoryl)-2-phenylindolin-3-one (3w)

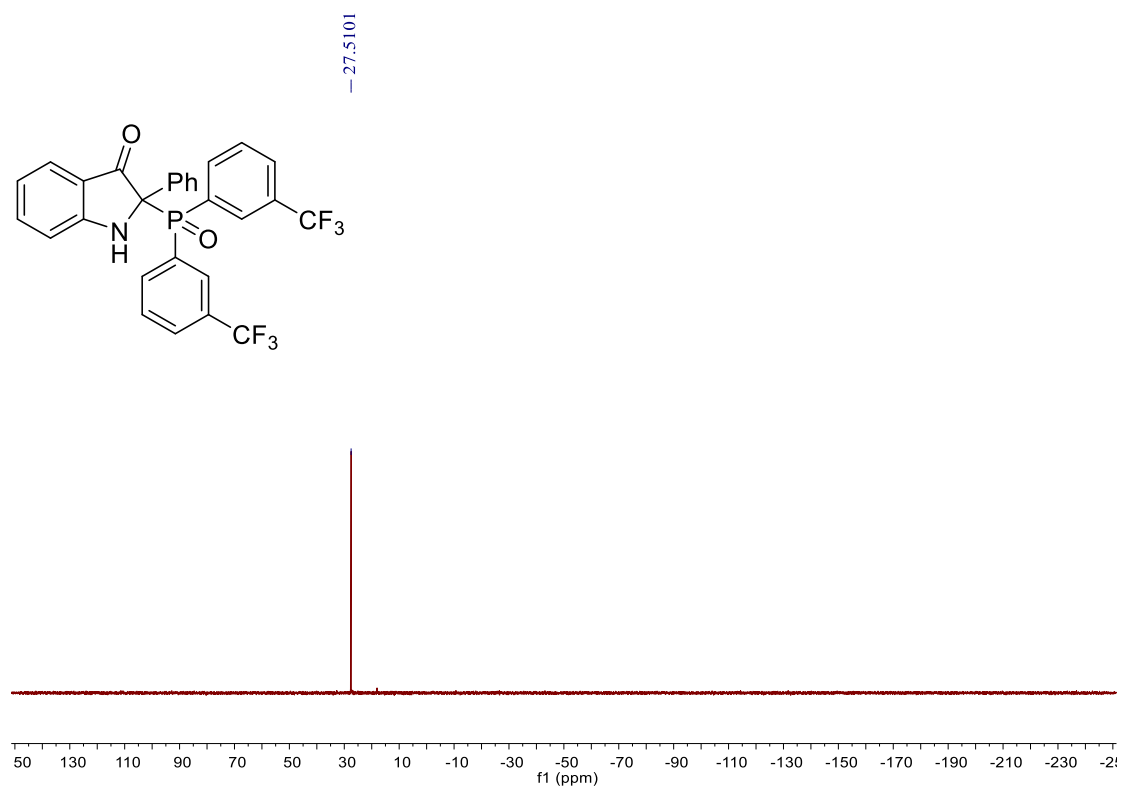
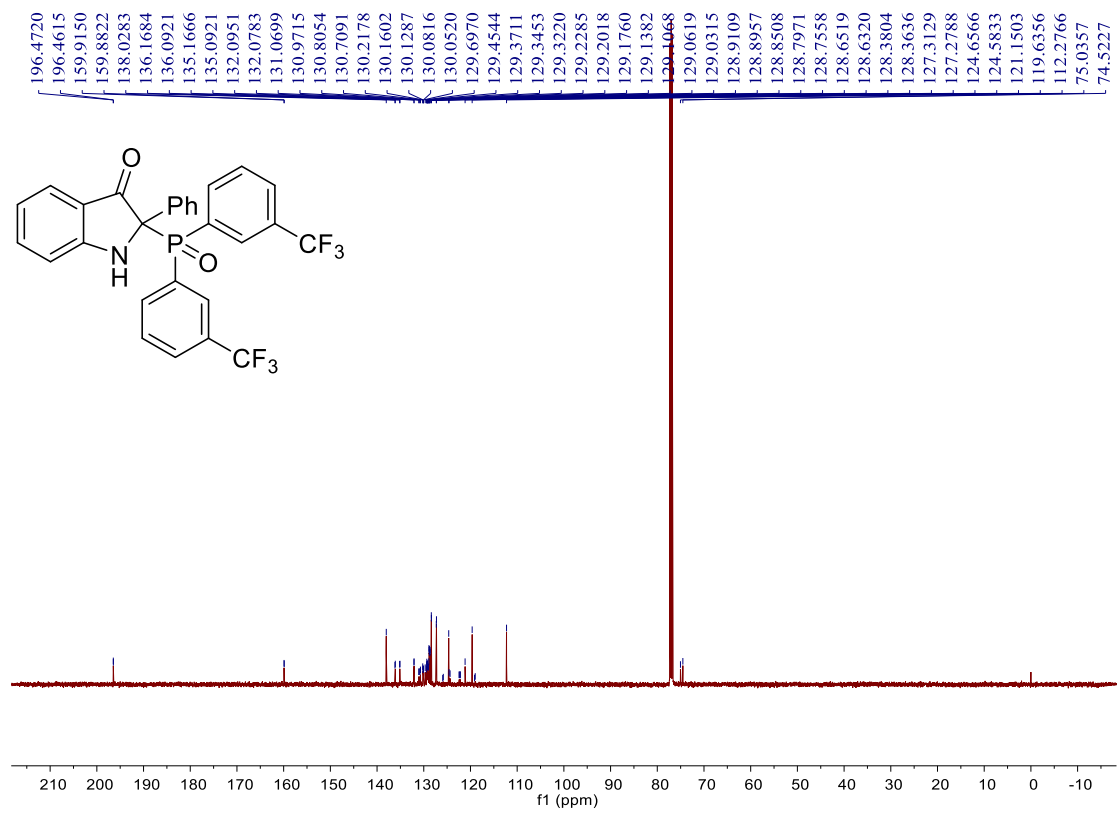


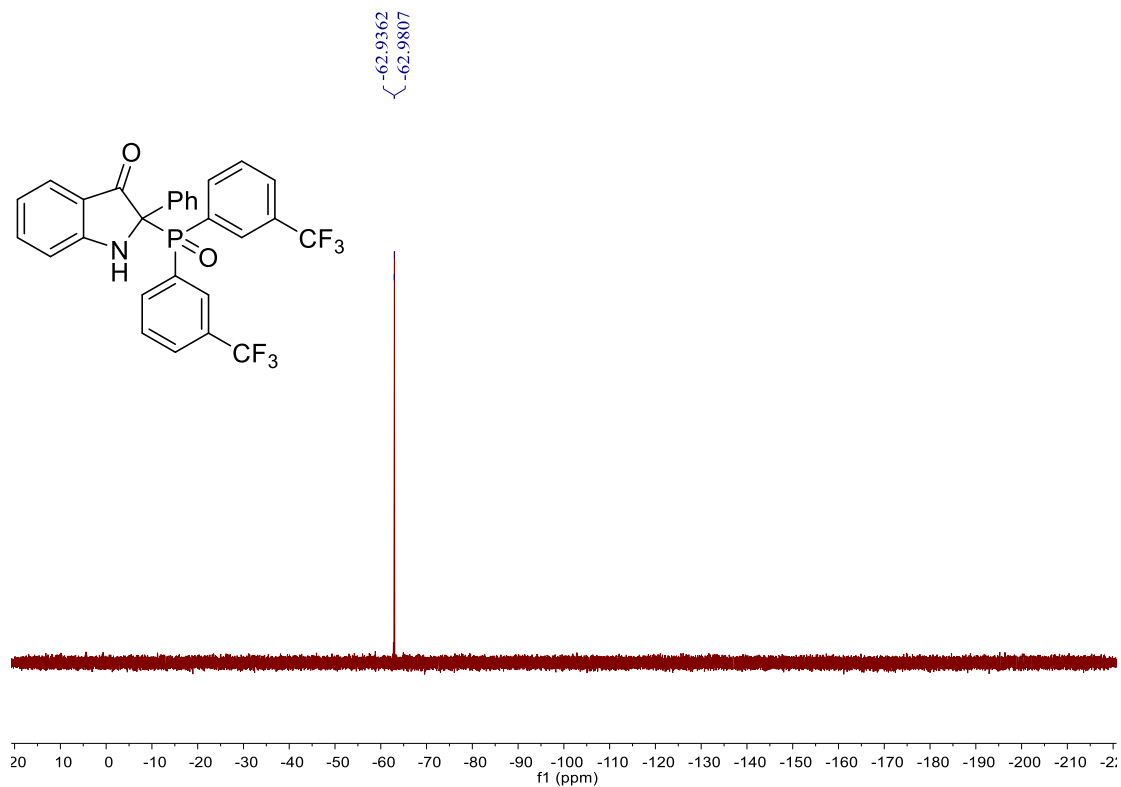




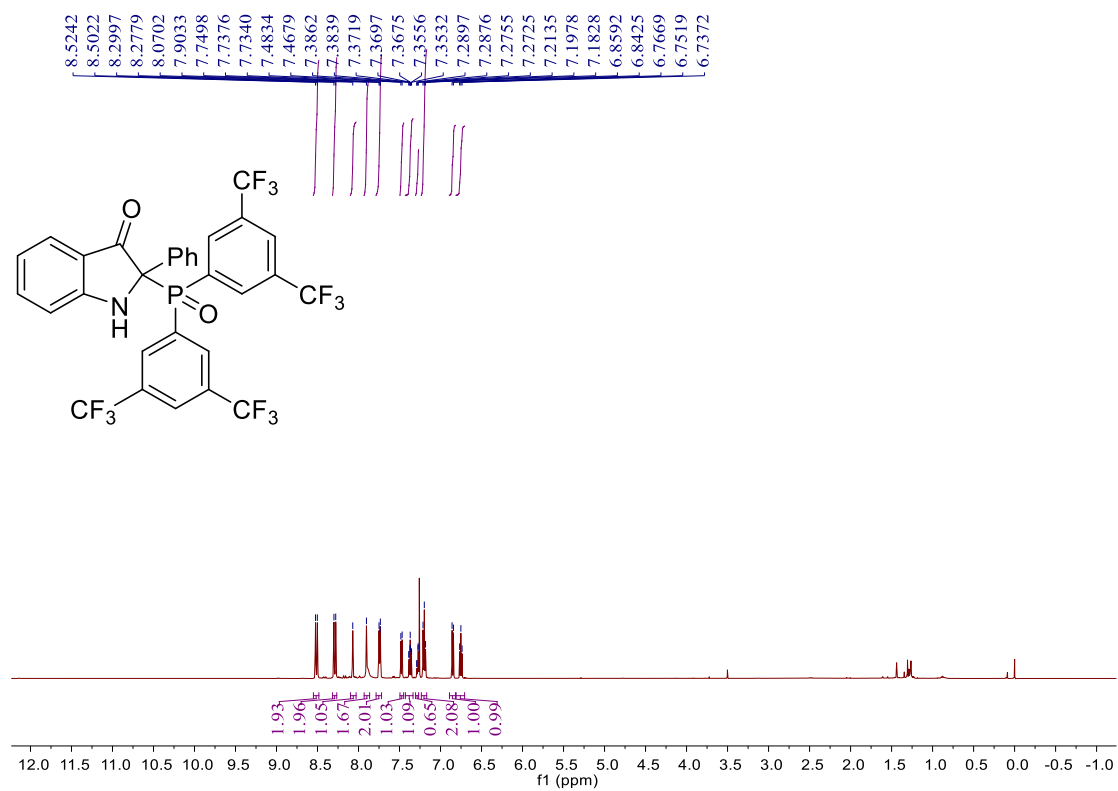
2-(bis(3-(trifluoromethyl)phenyl)phosphoryl)-2-phenylindolin-3-one (3x)



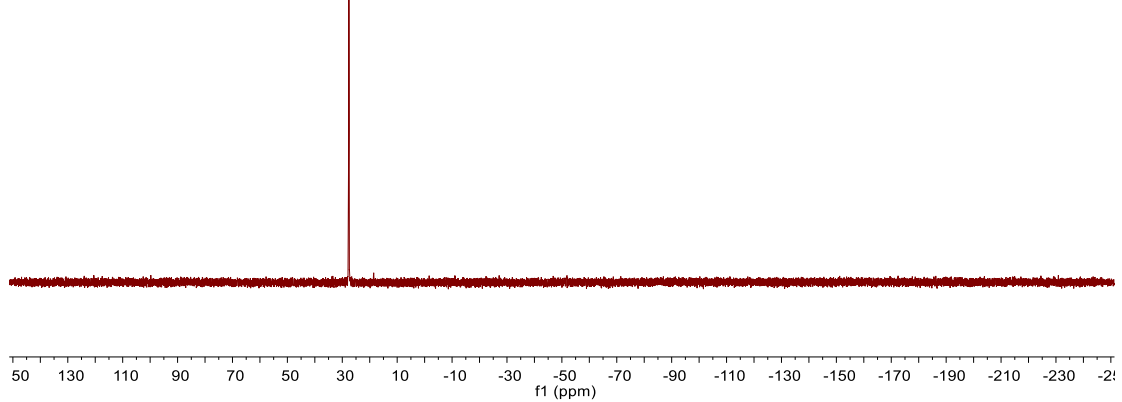
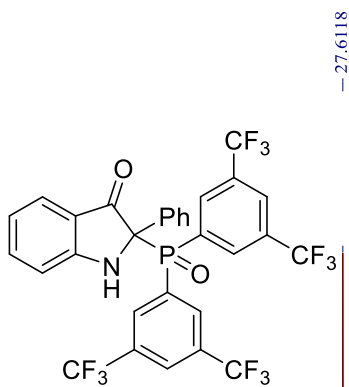
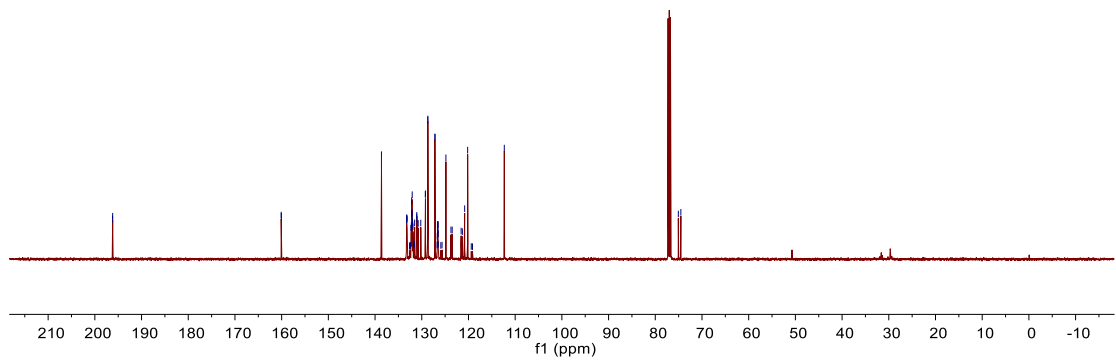
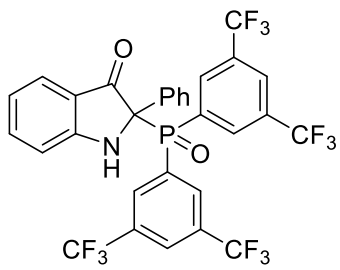


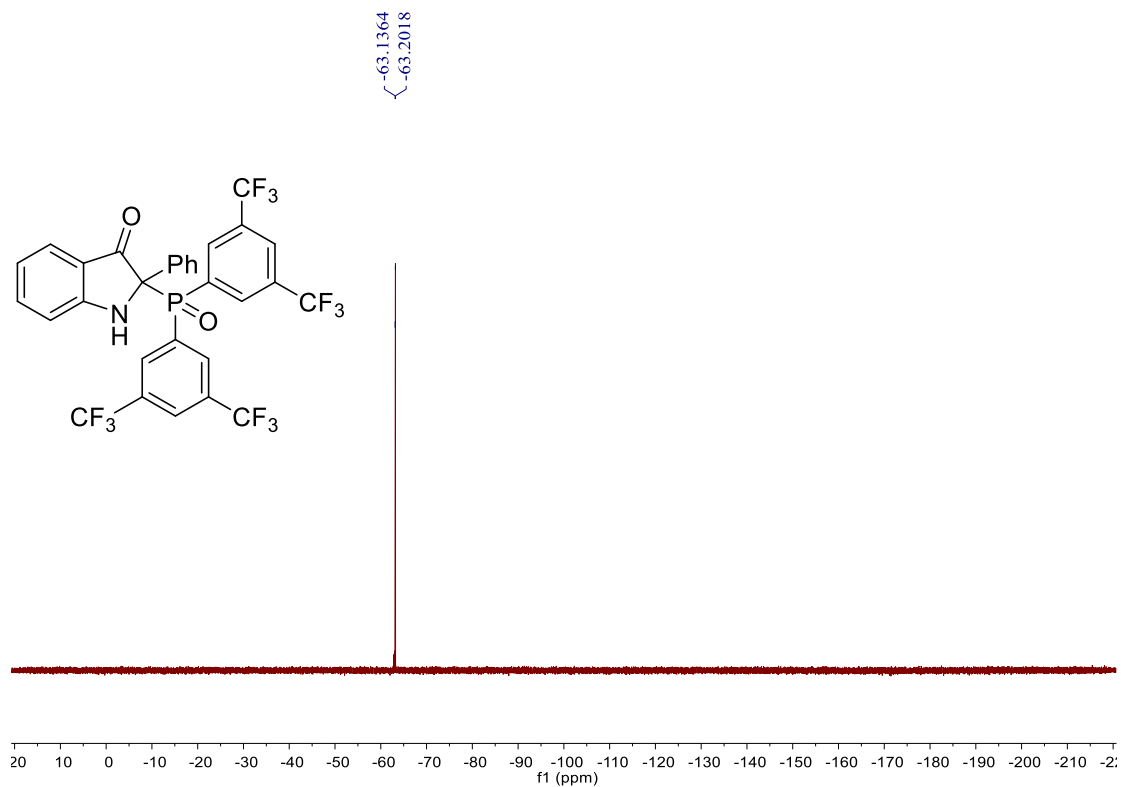


2-(bis(3,5-bis(trifluoromethyl)phenyl)phosphoryl)-2-phenylindolin-3-one (3y)

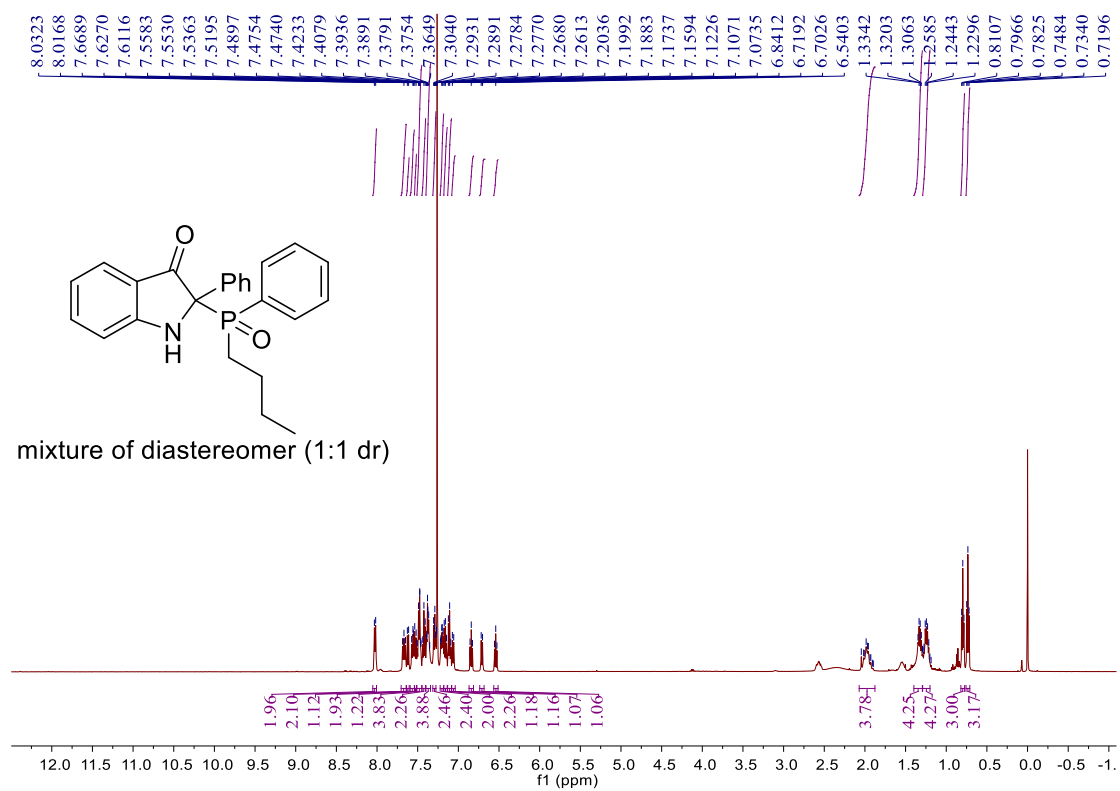


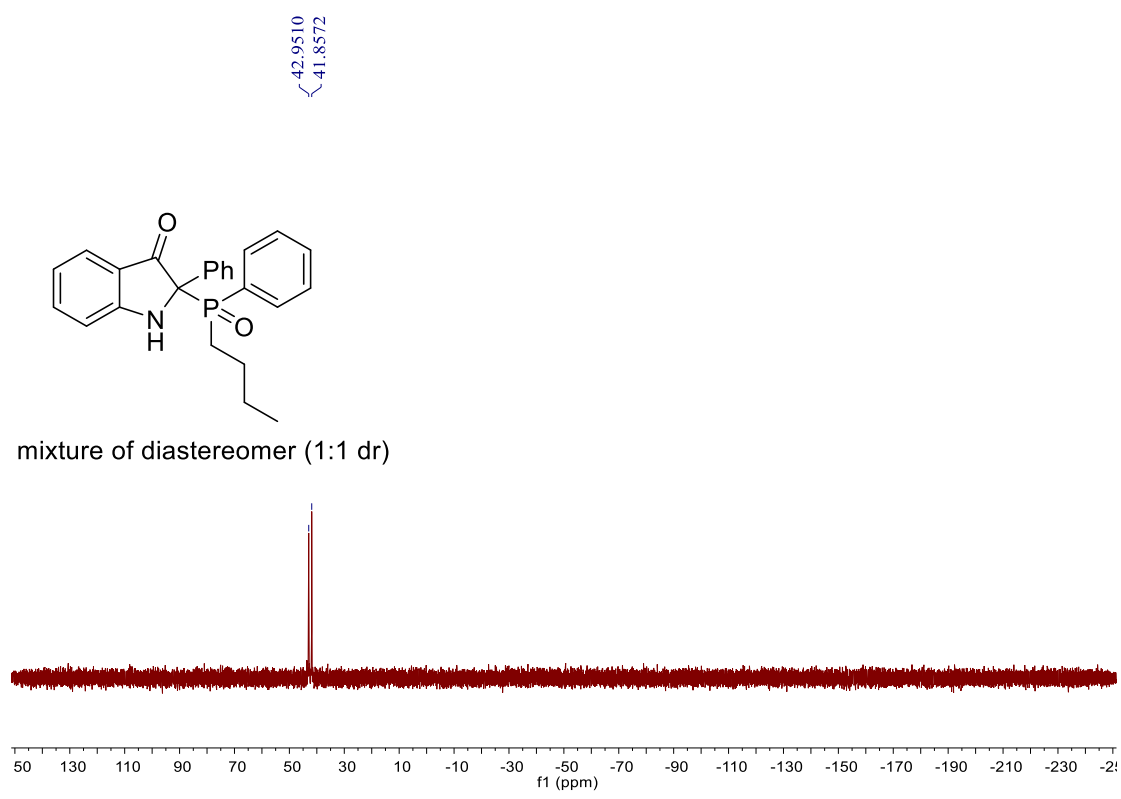
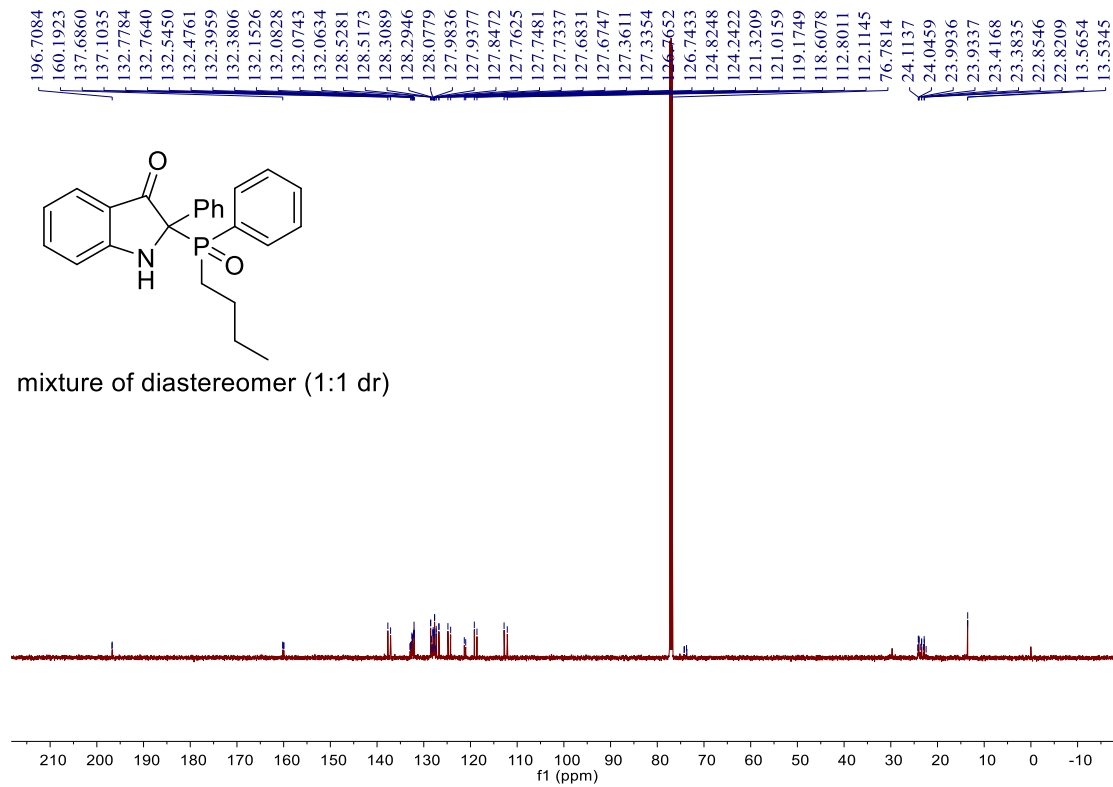
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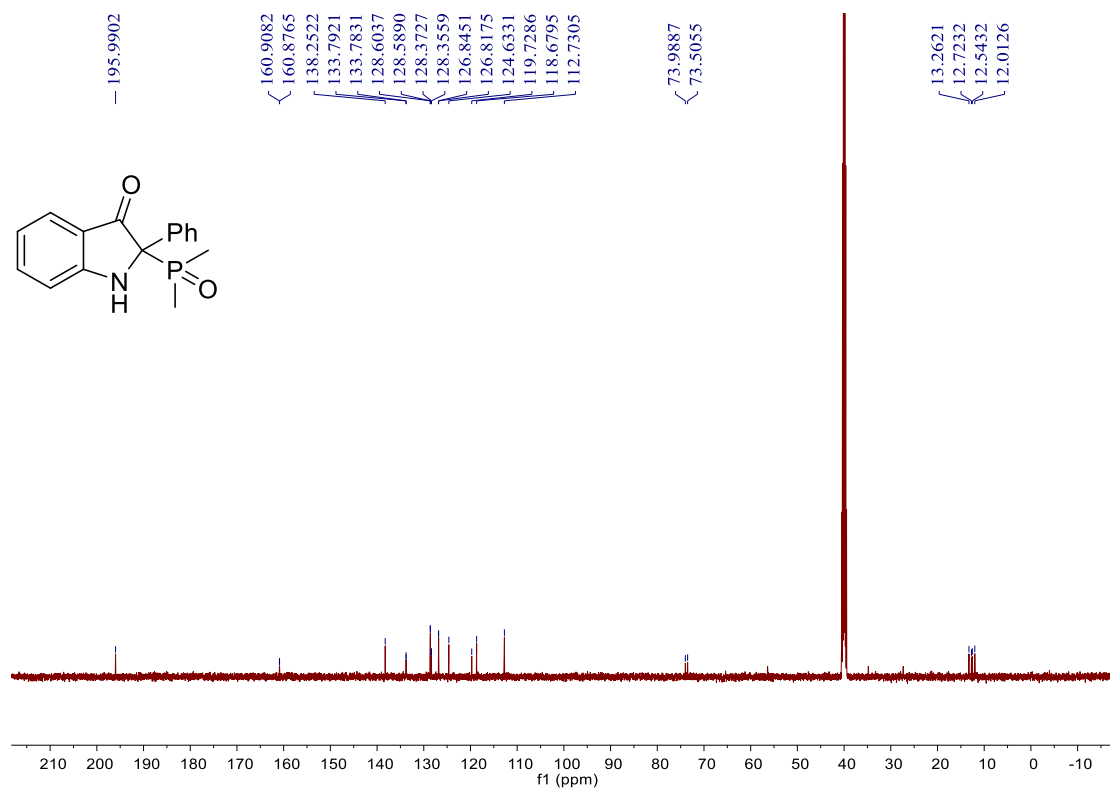
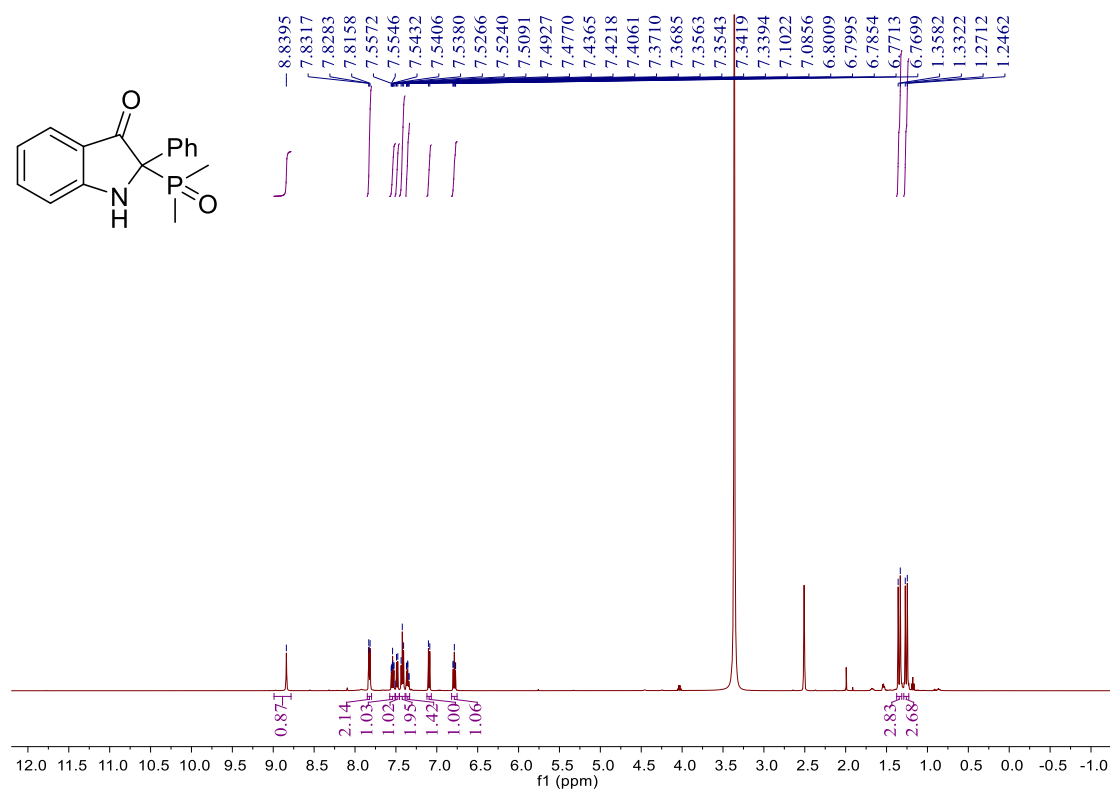


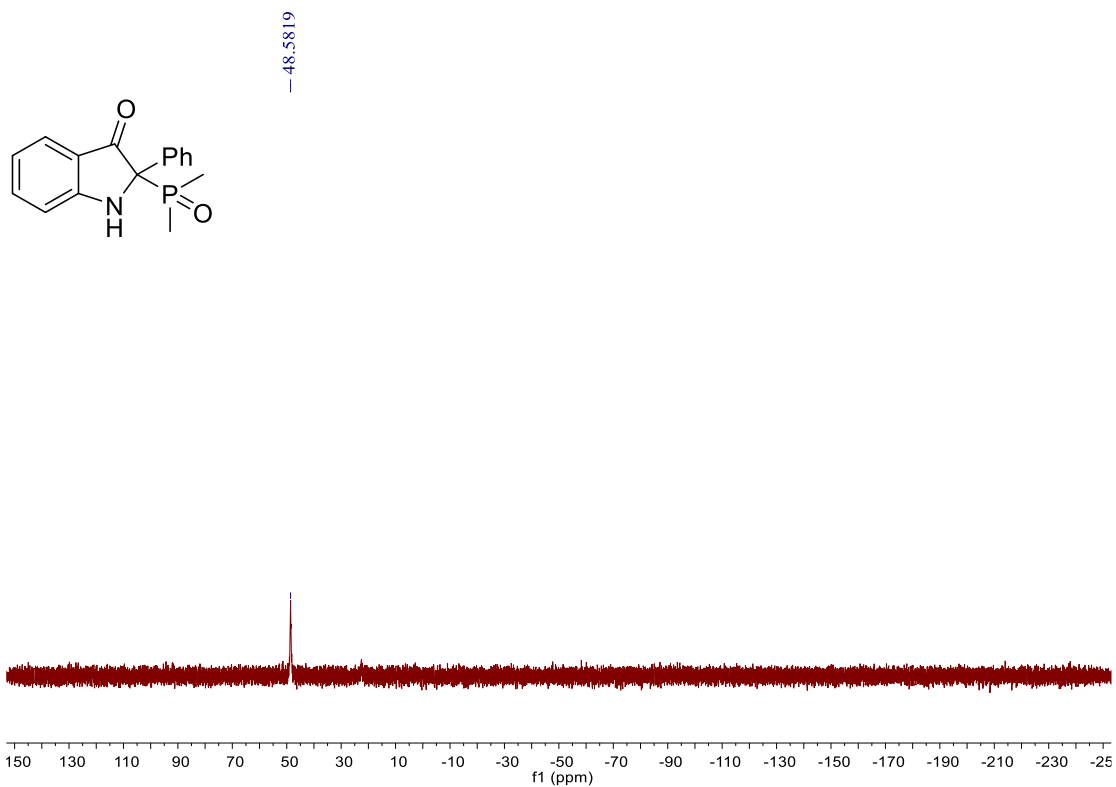
2-phenyl-2-(phenyl(propyl)phosphoryl)indolin-3-one (3z)



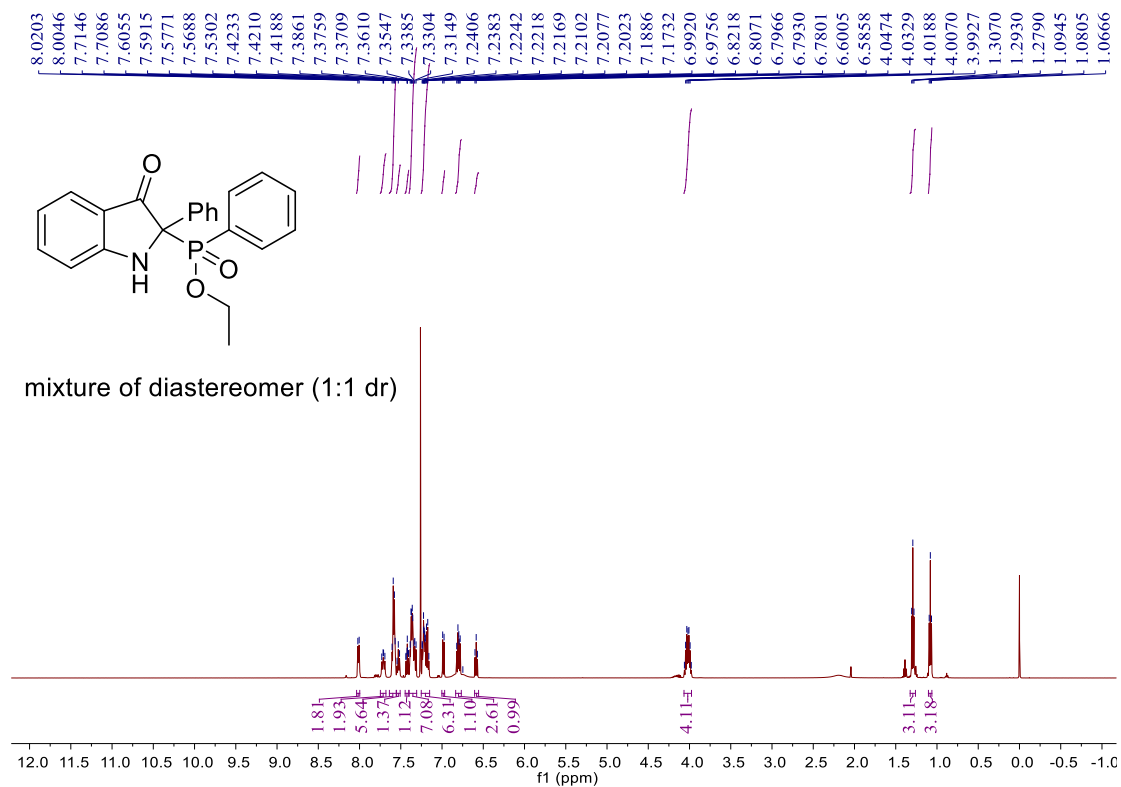


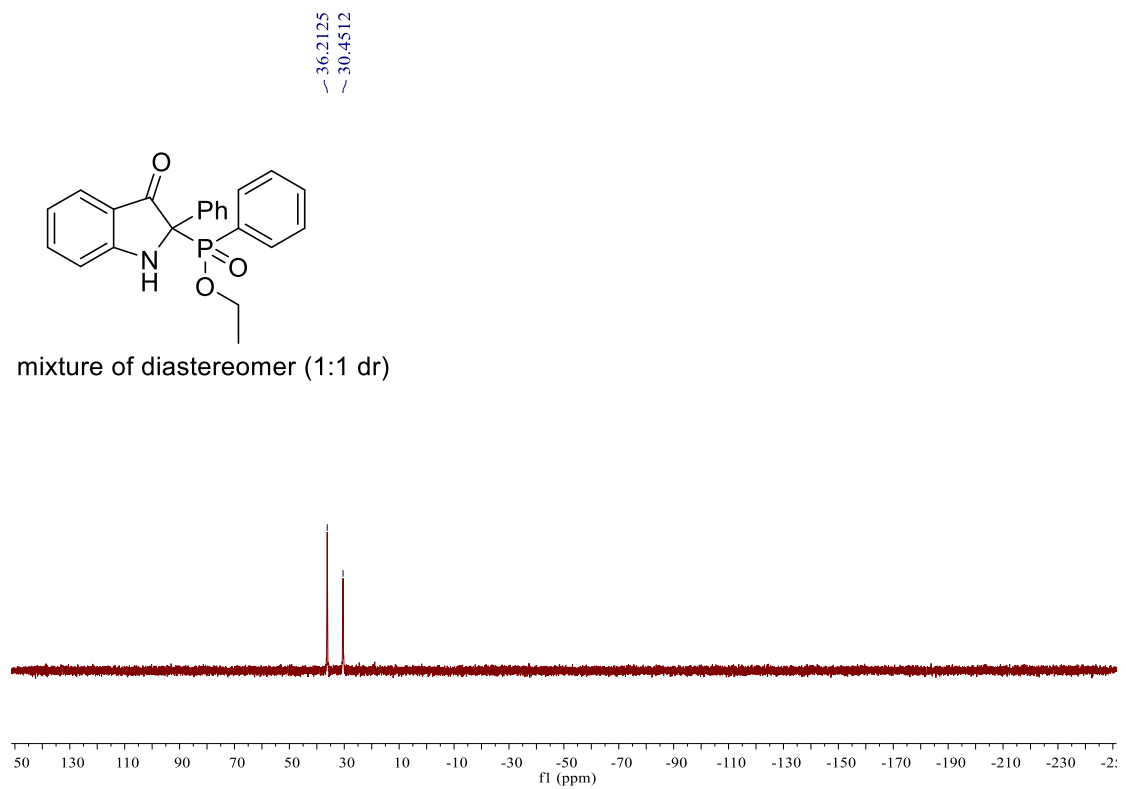
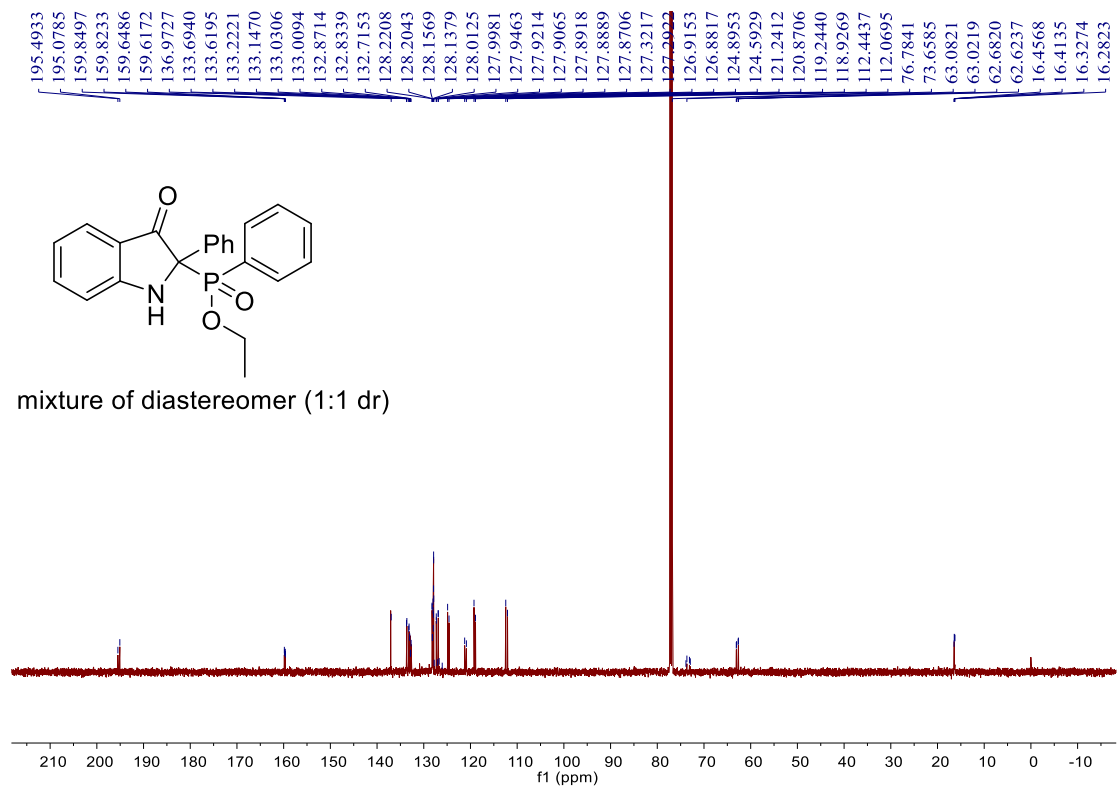
2-(dimethylphosphoryl)-2-phenylindolin-3-one (3aa).



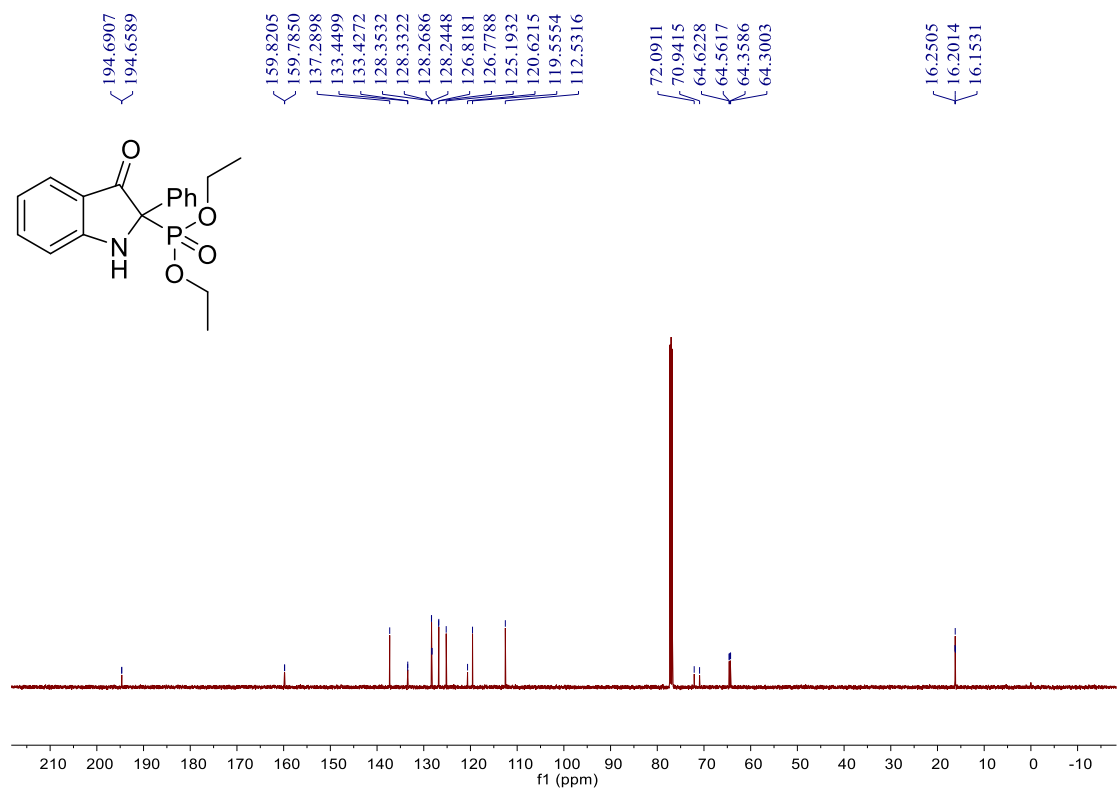
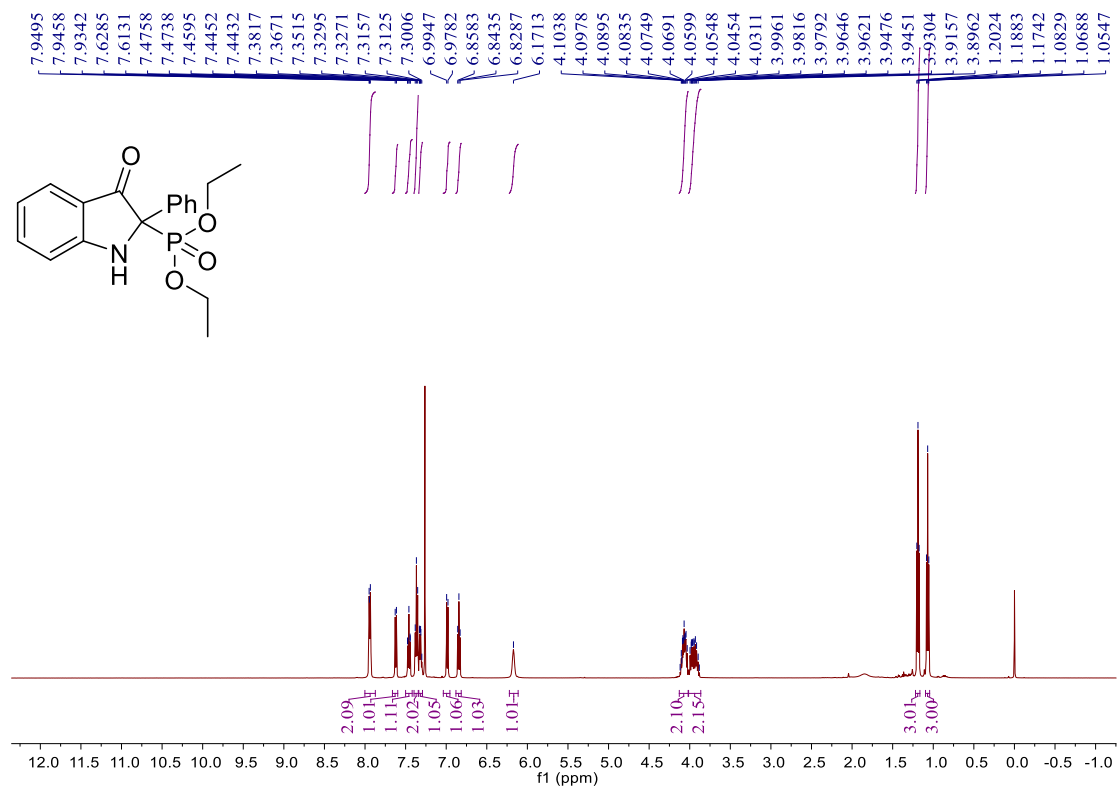


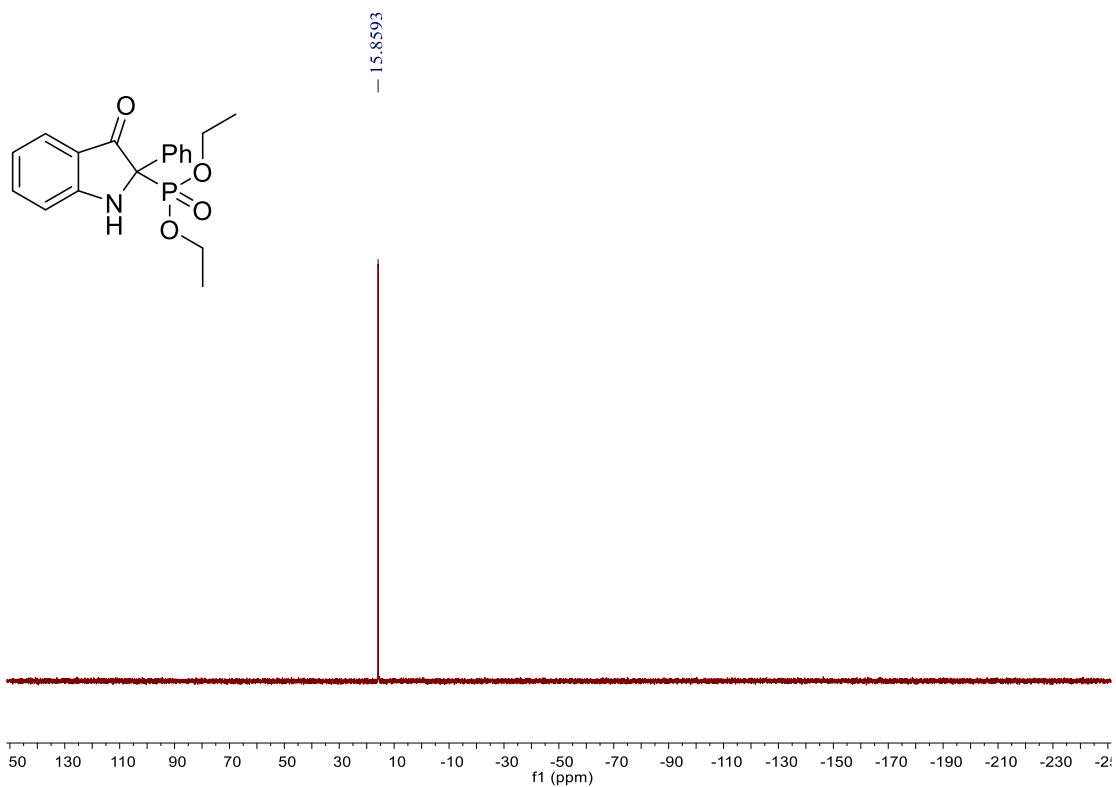
ethyl (3-oxo-2-phenylindolin-2-yl)(phenyl)phosphate(3ab)





diethyl (3-oxo-2-phenylindolin-2-yl)phosphonate(3ac)





dimethyl (3-oxo-2-phenylindolin-2-yl)phosphonate(3ad).

