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# Supplementary Information

## Copper-Catalyzed Oxidative Phosphonoheteroarylation of Alkenes with Phosphonates and *N*-Heteroarenes via P-H/C-H Functionalization

Chong-Hui Xu,[a] Zhi-Qiang Xiong,[a] Yang Li,[a] Yan-Ping Zhu,*[b] and Jin-Heng Li,*[abcd]

[a.] *Key Laboratory of Jiangxi Province for Persistent Pollutants Control and Resources Recycle, Nanchang Hangkong University, Nanchang 330063, China. E-mail: jhli@hnu.edu.cn*

[b.] *School of Pharmacy, Key Laboratory of Molecular Pharmacology and Drug Evaluation, Ministry of Education, Collaborative Innovation Center of Advanced Drug Delivery System and Biotech Drugs in Universities of Shandong, Yantai University, Shandong, Yantai, 264005, China. E-mail: chemzyp@foxmail.com*

[c.] *State Key Laboratory of Applied Organic Chemistry, Lanzhou University, Lanzhou 730000, China.*

[d.] *School of Chemistry and Chemical Engineering, Henan Normal University, Xinxiang, Henan 475004, China.*

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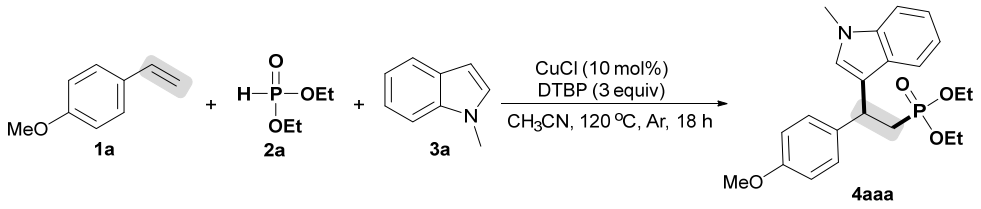
## (A) General Experimental Procedure

All $^{1}H$ and $^{13}C$ NMR spectra were recorded on a 400 MHz spectrometer or 500 MHz spectrometer at room temperature in $CDCl_3$ with tetramethylsilane as internal standard. Low-resolution mass spectra (LRMS) data were measured on GCMS-QP2010 Ultra. High-resolution mass spectra (HRMS) was recorded on an electrospray ionization (ESI) apparatus using time-of-flight (TOF) mass spectrometry. Melting Points were recorded on Hanon MP100 Apparatus. Unless otherwise noted, all reactions were carried out using standard Schlenk techniques, and all starting materials and solvents were commercially available and were used without further purification. Column chromatography was performed on silica gel (300-400 mesh) using petroleum ether (PE)/ethyl acetate (EA). Alkenes were prepared according to reported literatures[1], and other alkenes were purchased from commercial sources and used as received.

**(a) Typical Experimental Procedure for Intermolecular 1,2-Difunctionalization of Alkenes with phosphoric acid and Nucleophiles(4a):**



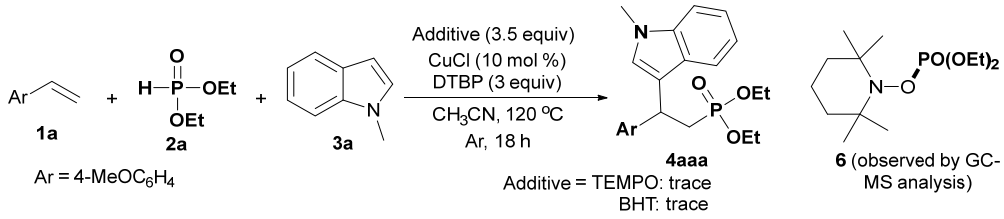

To a Schlenk tube were added Alkene **1a** (0.2 mmol), phosphoric acid **2a** (2 equiv), Nucleophiles **3a** (2 equiv), CuCl (10 mol%), DTBP (3 equiv), and $CH_3CN$ (2 mL). Then the tube was charged with argon and was then stirred at 120 °C (oil bath temperature) for the indicated time (about 18 h) until complete consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the reaction mixture was filtered by a crude column with ethyl acetate as eluent and concentrated in vacuum. The resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate) to afford the desired product **4aaa**.

**(b) Experimental Procedure for the 1 mmol Scale.**

To a Schlenk tube were added 4-methoxystyrene **1a** (1 mmol; 134.0 mg), diethyl

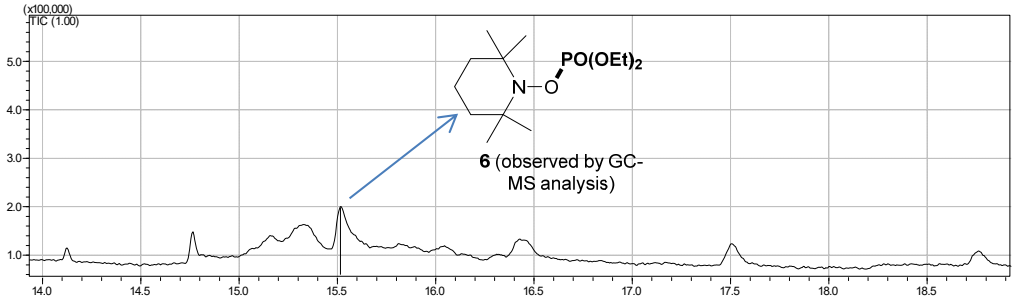
phosphonate **2a** (2 equiv; 276 mg), *N*-methyl-1*H*-indole **3a** (2 equiv; 262 mg), CuCl (10 mol%; 0.1 mmol; 9.9 mg), DTBP (3 equiv; 3 mmol; 438.7 mg), and $CH_3CN$ (10 mL). Then the tube was charged with argon and was then stirred at 120 °C (oil bath temperature) for the indicated time (about 18 h) until complete consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the reaction mixture was filtered by a crude column with ethyl acetate as eluent and concentrated in vacuum. The combined organic extracts were dried over $Na_2SO_4$ and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate = 2:1) to afford the brown oil liquid products **4aaa** in 72% yield (288.7 mg).

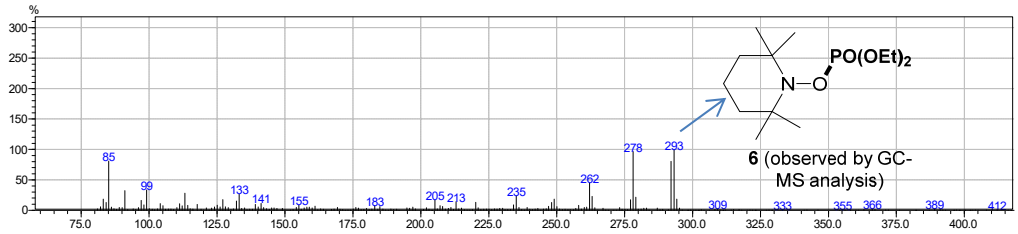
**(c) Control Experiments**




To a Schlenk tube were added Alkene **1a** (0.2 mmol), phosphoric acid **2a** (2 equiv), Nucleophiles **3a** (2 equiv), CuCl (10 mol%), DTBP (3 equiv), TEMPO (3.5 equiv), and $CH_3CN$ (2 mL). Then the tube was charged with argon and was then stirred at 120 °C (oil bath temperature) for the indicated time (about 18 h). The reaction mixture was filtered by a crude column with ethyl acetate as eluent and concentrated in vacuum. Finally, the organic phase was detected by GCMS analysis.

**GC-MS dates of 6:**






[**MS Spectrum**]

# of Peaks 519

Raw Spectrum 15.525 (scan : 2306)

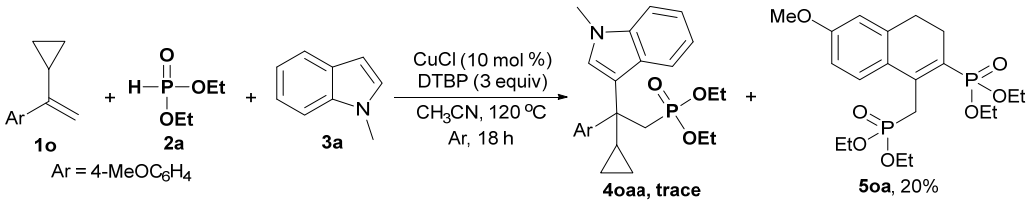
Background No Background Spectrum

Base Peak m/z 85.10 (Inten : 12,380)

Event# 1

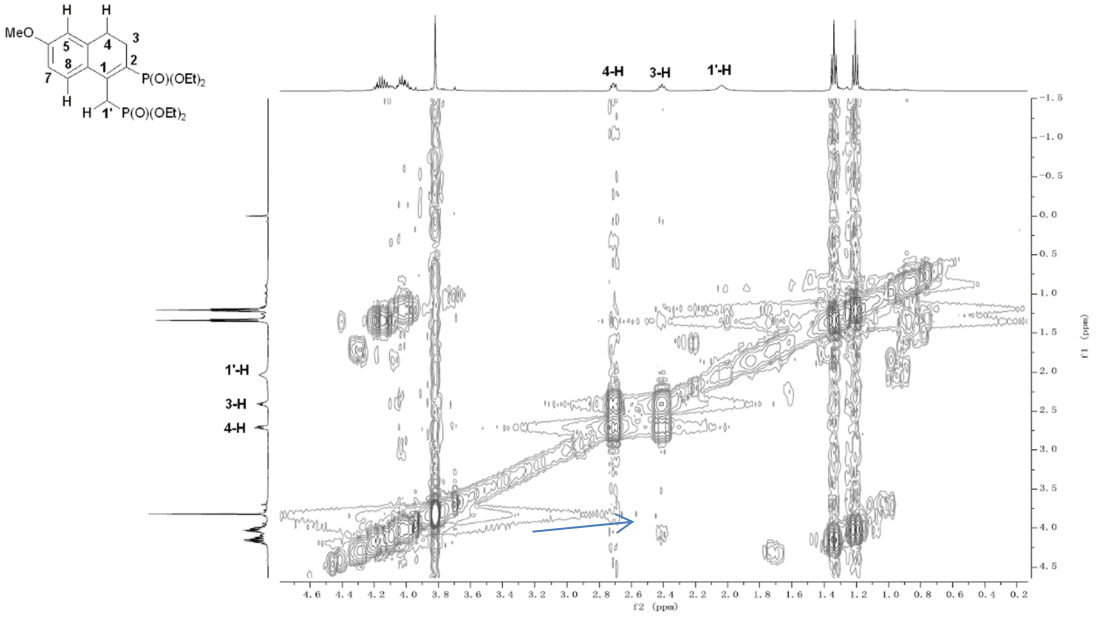
| m/z | Absolute Intensity | Relative Intensity | m/z | Absolute Intensity | Relative Intensity | m/z | Absolute Intensity | Relative Intensity |
|---|---|---|---|---|---|---|---|---|
| 80.00 | 193 | 1.56 | 87.10 | 257 | 2.08 | 290.00 | 102 | 0.82 |
| 81.05 | 899 | 7.26 | 88.00 | 286 | 2.31 | 291.10 | 172 | 1.39 |
| 82.10 | 1137 | 9.18 | 89.00 | 810 | 6.54 | 292.10 | 5431 | 43.87 |
| 83.15 | 3455 | 27.91 | 286.00 | 89 | 0.72 | **293.15** | **6178** | **49.90** |
| 84.15 | 2035 | 16.44 | 287.05 | 1382 | 11.16 | 294.15 | 1239 | 10.01 |
| **85.10** | **12380** | **100.00** | 288.00 | 482 | 3.89 | 295.25 | 490 | 3.96 |
| 86.15 | 1023 | 8.26 | 289.00 | 244 | 1.97 | | | |

**(d) Control Experiments**

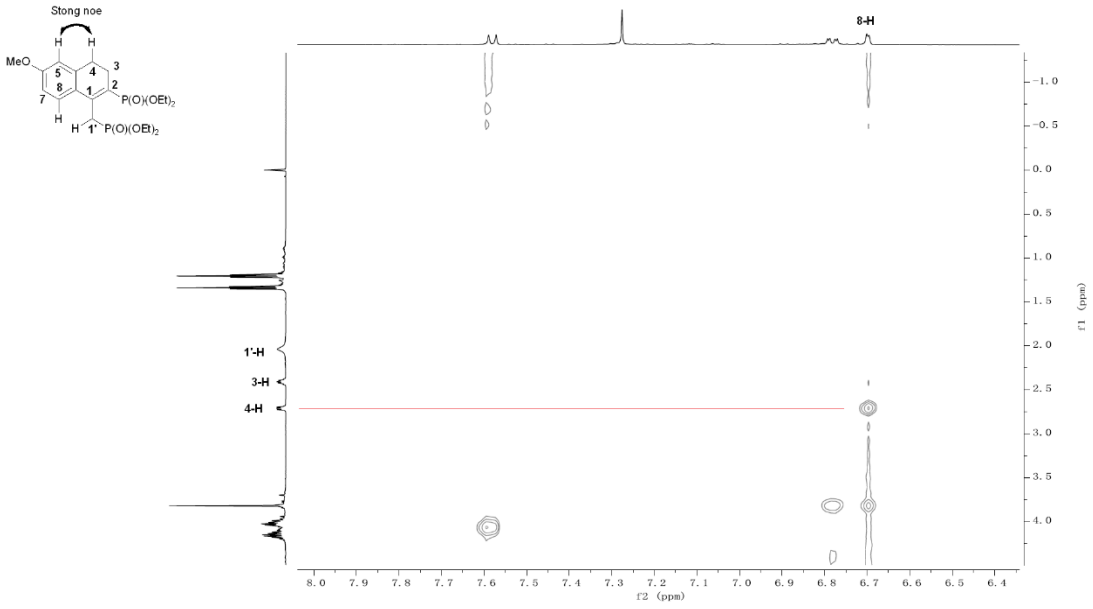



To a Schlenk tube were added 1-(1-cyclopropylvinyl)-4-methoxybenzene **1o** (0.2 mmol), phosphoric acid **2a** (2 equiv), Nucleophiles **3a** (2 equiv), CuCl (10 mol%), DTBP (3 equiv), and $CH_3CN$ (2 mL). Then the tube was charged with argon and was then stirred at 120 °C (oil bath temperature) for the indicated time (about 18 h). The reaction mixture was filtered by a crude column with ethyl acetate as eluent and concentrated in vacuum. Finally, the organic product 5**oa** was detected by COSY analysis.

## H-H COSY of 5oa:

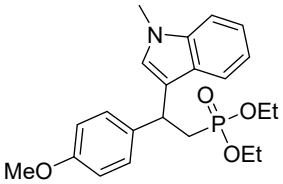



## H-H Noesy of 5oa:



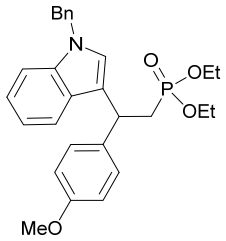
## (B) Analytical data

**Diethyl (2-(4-methoxyphenyl)-2-(1-methyl-1*H*-indol-3-yl)ethyl)phosphonate (4aaa):**



Following the typical experimental procedure on 0.2 mmol scale, compound **4aaa** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 57.7 mg; 72% yield; brown oil liquid; $^{1}$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.47 (d, *J* = 8.0 Hz, 1H), 7.30-7.23 (m, 3H), 7.17 (t, *J* = 7.2 Hz, 1H), 7.02 (t, *J* = 7.2 Hz, 1H), 6.85 (s, 1H), 6.82 (d, *J* = 8.0 Hz, 2H), 4.70-4.60 (m, 1H), 3.90-3.78 (m, 4H), 3.76 (s, 3H), 3.73 (s, 3H), 2.73-2.63 (m, 1H), 2.58-2.46 (m, 1H), 1.11 (m, 6H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 158.1, 137.3, 136.2 (d, $J_{C\text{-}P}$ = 7.6 Hz), 128.8, 126.7, 126.1, 121.7, 119.5, 118.9, 118.5 (d, $J_{C\text{-}P}$ = 14.4 Hz), 113.7, 109.1, 61.3 (d, $J_{C\text{-}P}$ = 6.6 Hz), 61.2 (d, $J_{C\text{-}P}$ = 6.6 Hz), 55.2, 36.6 (d, $J_{C\text{-}P}$ = 3.2 Hz), 32.9 (d, $J_{C\text{-}P}$ = 138.2 Hz), 32.6, 16.2 (d, $J_{C\text{-}P}$ = 6.6 Hz), 16.1 (d, $J_{C\text{-}P}$ = 6.6 Hz); $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 29.8. LRMS (EI, 70 eV) *m/z* (%): 401 ($M^+$, 20), 263 (100), 250 (30), 206 (10), 131 (2). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{22}H_{29}NO_4P$ 402.1829; found 402.1827.

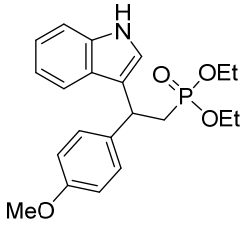
**Diethyl-(2-(1-benzyl-1H-indol-3-yl)-2-(4-methoxyphenyl)ethyl)phosphonate (4aab):**



Following the typical experimental procedure on 0.2 mmol scale, compound **4aab** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 48.7 mg; 51% yield; yellow oil liquid; $^{1}$H NMR ($CDCl_3$, 500 MHz) δ (ppm) 7.46 (m, 2H), 7.29-7.18 (m, 5H), 7.12-7.06 (m, 3H), 7.04-6.95 (m, 2H), 6.81 (d, *J* = 7.5 Hz, 2H), 5.23 (s, 2H), 4.72-4.57 (m, 1H), 4.14-4.10 (m, 1H), 3.91-3.82 (m, 2H), 3.79 (s, 1H), 3.74 (s, 3H), 2.80-2.62 (m, 1H), 2.56-2.45 (m, 1H), 1.13-1.03 (m, 6H). $^{13}$C NMR ($CDCl_3$, 125 MHz) δ (ppm) δ 158.0, 137.5, 136.9, 129.2, 128.7 (d, $J_{C\text{-}P}$ = 15.5 Hz), 127.4, 126.6, 125.3, 121.8, 119.6, 119.0, 114.1, 113.6, 109.6, 61.6-61.0 (m), 55.1, 49.8, 36.53 (d, $J_{C\text{-}P}$ = 2.0 Hz), 33.3(d, $J_{C\text{-}P}$ = 13.5 Hz), 16.4 (d, $J_{C\text{-}P}$ = 2.0

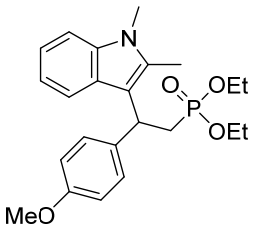
Hz), 16.3 (d, $J_{C\text{-}P}$ = 6.5 Hz). $^{31}$P NMR ($CDCl_3$, 202 MHz) δ (ppm) 29.7. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{28}H_{33}NO_4$ 478.2142, found 478.2137.

**Diethyl (2-(1*H*-indol-3-yl)-2-(4-methoxyphenyl)ethyl)phosphonate (4aae):**



Following the typical experimental procedure on 0.2 mmol scale, compound **4aae** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 49.5 mg; 64% yield; brown oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 8.74 (s, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.22-7.15 (m, 3H), 7.02 (t, *J* = 7.2 Hz, 1H), 6.96-6.86 (m, 2H), 6.70 (d, *J* = 7.6 Hz, 2H), 4.66-4.53 (m, 1H), 3.85-3.74 (m, 2H), 3.74-3.66 (m, 2H), 3.64 (s, 3H), 2.67-2.55 (m, 1H), 2.49-2.37 (m, 1H), 1.06-0.98 (m, 6H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 158.0, 136.7, 136.2 (d, $J_{C\text{-}P}$ = 8.0 Hz), 128.7, 126.2, 121.7, 121.5, 119.2, 119.1, 119.0, 113.6, 111.2, 61.4 (d, $J_{C\text{-}P}$ = 6.5 Hz), 61.3 (d, $J_{C\text{-}P}$ = 6.5 Hz), 55.1, 36.5 (d, $J_{C\text{-}P}$ = 3.0 Hz), 32.8 (d, $J_{C\text{-}P}$ = 138.2 Hz), 16.1 (t, $J_{C\text{-}P}$ = 6.3 Hz). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 30.0. LRMS (EI, 70 eV) *m/z* (%): 387 ($M^+$, 20), 341 (4), 267 (19), 193 (100), 135 (96),121 (30). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{21}H_{27}NO_4P$ 388.1672, found388.1671.

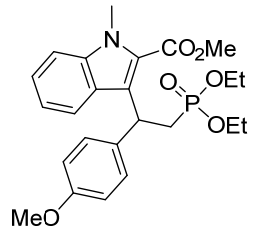
**Diethyl (2-(1,2-dimethyl-1*H*-indol-3-yl)-2-(4-methoxyphenyl)ethyl)phosphonate (4aaf):**



Following the typical experimental procedure on 0.2 mmol scale, compound **4aaf** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 64.7 mg; 78% yield; yellow oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.38 (d, *J* = 8.0 Hz, 1H), 7.23-7.12 (m, 3H), 7.02 (t, *J* = 7.4 Hz, 1H), 6.90 (t, *J* = 7.4 Hz, 1H), 6.70 (d, *J* = 8.0 Hz, 2H), 4.67-4.57 (m, 1H), 3.83-3.72 (m, 1H), 3.69-3.58 (m, 5H), 3.55 (s, 3H), 3.51-3.40 (m, 1H), 2.76-2.63 (m, 2H), 2.37 (s, 3H), 1.00 (t, *J* = 7.0 Hz, 3H), 0.85 (t, *J* = 7.0 Hz, 3H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 157.7, 136.9, 136.6 (d, $J_{C\text{-}P}$ = 14.2 Hz), 133.3, 128.2, 126.2, 120.3, 119.1, 118.7, 113.5, 112.8 (d, $J_{C\text{-}P}$ = 6.8 Hz), 108.6, 61.1 (d, $J_{C\text{-}P}$ = 6.6 Hz), 61.0 (d, $J_{C\text{-}P}$ = 6.6 Hz), 55.1, 35.4 (d, $J_{C\text{-}P}$ = 3.3 Hz), 31.0 (d, $J_{C\text{-}P}$ = 138.7 Hz), 29.4, 16.0 (d, $J_{C\text{-}P}$ = 6.5 Hz), 15.8 (d, $J_{C\text{-}P}$ = 6.5 Hz), 10.5. $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 30.7. LRMS (EI,

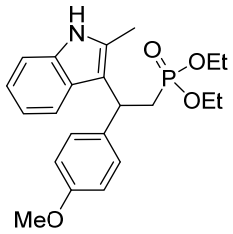
70 eV) *m/z* (%): 415 ($M^+$, 20), 277 (100), 264 (30), 220 (10), 145 (2). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{23}H_{31}NO_4P$ 416.1985; found 416.1981.

**3-(2-(diethoxyphosphoryl)-1-(4-methoxyphenyl)ethyl)-1-methyl-1*H*-indole-2-carboxylate (4aag):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4aag** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 53.2 mg; 58% yield; yellow oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.53 (d, *J* = 8.4 Hz, 1H), 7.29-7.17 (m, 4H), 6.97 (t, *J* = 7.4 Hz, 1H), 6.73 (d, *J* = 8.0 Hz, 2H), 5.48-5.40 (m, 1H), 3.89 (s, 6H), 3.80-3.56 (m, 8H), 2.77-2.67 (m, 2H), 0.99-0.92 (m, 6H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 163.1, 157.8, 139.0, 135.8 (d, $J_{C-P}$ = 14.0 Hz), 128.3, 125.6,125.2, 125.1, 124.8, 122.1, 120.0, 113.6, 110.3, 61.2 (d, $J_{C-P}$ = 2.6 Hz), 61.1 (d, $J_{C-P}$ = 2.6 Hz), 55.2, 51.7, 34.7 (d, $J_{C-P}$ = 3.1 Hz), 32.0, 30.8 (d, $J_{C-P}$ = 139.6 Hz), 16.0 (t, $J_{C-P}$ = 6.8 Hz). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 30.1. LRMS (EI, 70 eV) *m/z* (%): 459 ($M^+$, 31), 400 (100), 321 (75), 278 (37), 218 (20), 134(7). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{24}H_{31}NO_6P$ 460.1884, found 460.1887.

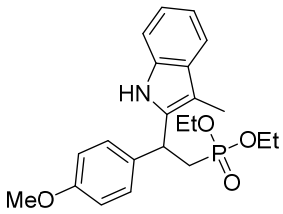
**Diethyl-(2-(4-methoxyphenyl)-2-(2-methyl-1*H*-indol-3-yl)ethyl)phosphonate (4aah):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4aah** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 52.1 mg; 65% yield; brown oil liquid; $^1$H NMR ($CDCl_3$, 500 MHz) δ (ppm) 8.07 (s, 1H), 7.44 (d, *J* = 8.0 Hz, 1H), 7.28 (d, *J* = 8.5 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 1H), 7.05 (t, *J* = 7.5 Hz, 1H), 6.98 (t, *J* = 7.5 Hz, 1H), 6.78 (d, *J* = 9.0 Hz, 2H), 4.71-4.64 (m, 1H), 3.90-3.83 (m, 1H), 3.80-3.75 (m, 1H), 3.74 (s, 3H), 3.72-3.66 (m, 1H), 3.60-3.50 (m, 1H), 2.87-2.76 (m, 1H), 2.75-2.66 (m, 1H), 2.44 (s, 3H), 1.09 (t, *J* = 7.0 Hz, 3H), 0.94 (t, *J* = 7.0 Hz, 3H). $^{13}$C NMR ($CDCl_3$, 125 MHz) δ (ppm) 157.7, 136.5 (d, $J_{C-P}$ = 14.5 Hz), 135.5, 131.6, 128.3, 127.2, 120.7, 119.1, 119.0, 113.6, 113.3 (d, $J_{C-P}$ = 6.5 Hz), 110.4, 61.2 (d, $J_{C-P}$ = 6.5 Hz), 61.1 (d, $J_{C-P}$ = 6.5 Hz), 55.2, 35.1 (d,

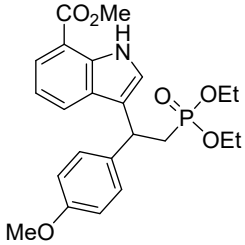
$J_{C\text{-}P}$ = 3.0 Hz), 31.0 (d, $J_{C\text{-}P}$ = 139.0 Hz), 16.1 (d, $J_{C\text{-}P}$ = 6.5 Hz), 16.0 (d, $J_{C\text{-}P}$ = 6.5 Hz), 12.1. $^{31}$P NMR ($CDCl_3$, 202 MHz) δ (ppm) 30.6. LRMS (EI, 70 eV) *m/z* (%): 401 ($M^+$, 60), 272 (11), 263 (100), 232 (39), 134 (18). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{22}H_{29}NO_4P$ 402.1829, found 402.1828.

**Diethyl (2-(4-methoxyphenyl)-2-(3-methyl-1*H*-indol-2-yl)ethyl)phosphonate (4aai):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4aai** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 43.3 mg; 54% yield; brown oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 8.91 (s, 1H), 7.40 (d, *J* = 7.6 Hz, 1H), 7.20 (t, *J* = 8.8 Hz, 1H), 7.12 (d, *J* = 8.0 Hz, 2H), 7.05-6.95 (m, 2H), 6.74 (d, *J* = 7.6 Hz, 2H), 4.71-4.62 (m, 1H), 3.91-3.70 (m, 3H), 3.68 (s, 3H), 3.61-3.50 (m, 1H), 2.59-2.44 (m, 2H), 2.15 (s, 3H), 1.08 (t, *J* = 7.0 Hz, 3H), 0.95 (t, *J* = 7.0 Hz, 3H). $^{13}$C NMR ($CDCl_3$, 125 MHz) δ (ppm) 158.4, 135.6, 135.5, 134.0 (d, $J_{C\text{-}P}$ = 10.0 Hz), 129.2, 128.5, 121.2, 118.9, 118.2, 113.9, 110.7, 107.3, 61.6 (t, $J_{C\text{-}P}$ = 7.0 Hz), 55.2, 36.3 (d, $J_{C\text{-}P}$ = 3.5 Hz), 31.5 (d, $J_{C\text{-}P}$ = 139.0 Hz), 16.2 (d, $J_{C\text{-}P}$ = 6.5 Hz), 16.1 (d, $J_{C\text{-}P}$ = 6.5 Hz), 8.6. $^{31}$P NMR (162 MHz, $CDCl_3$) δ 29.5. LRMS (EI, 70 eV) *m/z* (%): 401 ($M^+$, 55), 263 (100), 248 (69), 217 (28), 130 (35). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{22}H_{29}NO_4P$ 402.1829, found 402.1827.

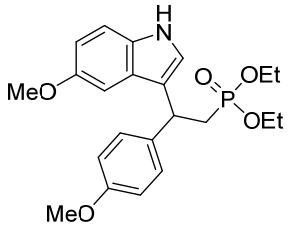
**Methyl-3-(2-(diethoxyphosphoryl)-1-(4-methoxyphenyl)ethyl)-1*H*-indole-7-carboxylate (4aaj):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4aaj** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 42.7 mg; 48% yield; brown oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 9.62 (s, 1H), 7.75 (d, *J* = 7.6 Hz, 1H), 7.58 (d, *J* = 8.0 Hz, 1H), 7.18 (d, *J* = 7.2 Hz, 2H), 7.10 (s, 1H), 6.97 (t, *J* = 7.6 Hz, 1H), 6.74 (d, *J* = 8.0 Hz, 2H), 4.64-4.57 (m, 1H), 3.88 (s, 3H), 3.85-3.78 (m, 2H), 3.77-3.70 (m, 2H), 3.68 (s, 3H), 2.65-2.54 (m, 1H), 2.51-2.39 (m, 1H), 1.06-1.02 (m, 6H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm)

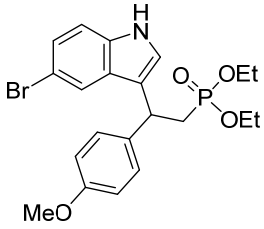
167.8, 158.2, 136.5, 136.1 (d, $J_{C-P}$ = 2.0 Hz), 128.8, 127.6, 125.2, 124.4, 122.3, 119.9 (d, $J_{C-P}$ = 13.0 Hz), 118.7, 113.8, 112.4, 61.4 (d, $J_{C-P}$ = 6.5 Hz), 61.3 (d, $J_{C-P}$ = 6.5 Hz), 55.2, 51.8, 36.4, 33.0 (d, $J_{C-P}$ = 139.0 Hz), 16.2 (d, $J_{C-P}$ = 3.0 Hz),16.23 (d, $J_{C-P}$ = 3.0 Hz). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 29.5. LRMS (EI, 70 eV) *m/z* (%): 445 ($M^+$, 5), 307 (20), 207 (100), 191 (5), 133 (12). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{23}H_{29}NO_6P$ 446.1727, found 446.1727.

**Diethyl (2-(5-methoxy-1*H*-indol-3-yl)-2-(4-methoxyphenyl)ethyl)phosphonate (4aak):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4aak** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 60.8 mg; 73% yield; yellow oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 8.53 (s, 1H), 7.26 (d, *J* = 7.2 Hz, 2H), 7.18 (d, *J* = 8.4 Hz, 1H), 6.96 (s, 1H), 6.87 (s, 1H), 6.79 (t, *J* = 7.6 Hz, 3H), 4.66-4.55 (m, 1H), 3.93-3.85 (m, 2H),3.82-3.76 (m, 2), 3.74 (s, 6H), 2.73-2.61 (m, 1H), 2.56-2.44 (m, 1H), 1.15-1.08 (m, 6H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 158.0, 153.6, 136.1 (d, $J_{C-P}$ = 7.8 Hz), 131.8, 128.7, 126.6, 122.2, 119.1 (d, $J_{C-P}$ = 13.8 Hz), 113.6, 111.8, 101.3, 61.4 (d, $J_{C-P}$ = 6.4 Hz), 61.3 (d, $J_{C-P}$ = 6.4 Hz), 55.7, 55.2, 36.5 (d, $J_{C-P}$ = 3.0 Hz), 32.6 (d, $J_{C-P}$ = 138.0 Hz), 16.15 (t, $J_{C-P}$ = 6.2 Hz). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 30.0. LRMS (EI, 70 eV) *m/z* (%): 417 ($M^+$, 18), 279 (100), 248 (10), 207 (47), 133 (9). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{22}H_{29}NO_5P$ 418.1778, found418.1772.

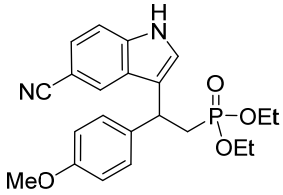
**Diethyl (2-(5-bromo-1H-indol-3-yl)-2-(4-methoxyphenyl)ethyl)phosphonate (4aal):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4aal** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 65.2 mg; 70% yield; brown oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 8.99 (s, 1H), 7.55 (s, 1H), 7.22-7.14 (m, 4H), 7.00 (s, 1H), 6.79 (d, *J* = 8.0 Hz, 2H), 4.61-4.53 (m, 1H), 3.95-3.77 (m, 4H), 3.74 (s, 3H), 2.70-2.58 (m, 1H), 2.55-2.43 (m, 1H), 1.18-1.08 (m, 6H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 158.2,

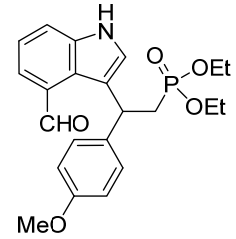
135.9 (d, $J_{C\text{-}P}$ = 9.2 Hz), 135.3, 128.5, 128.0, 124.7, 122.9, 121.7, 118.7 (d, $J_{C\text{-}P}$ = 12.4 Hz), 113.8, 112.7, 112.4, 61.5 (d, $J_{C\text{-}P}$ = 6.6 Hz), 61.4 (d, $J_{C\text{-}P}$ = 6.6 Hz), 55.2, 36.4 (d, $J_{C\text{-}P}$ = 3.0 Hz), 32.8 (d, $J_{C\text{-}P}$ = 138.4 Hz), 16.2 (d, $J_{C\text{-}P}$ = 6.2 Hz), 16.1 (d, $J_{C\text{-}P}$ = 6.2 Hz). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 29.4. LRMS (EI, 70 eV) *m/z* (%): 466 ($M^+$, 3), 327 (60), 207 (100), 191 (17), 133(19). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{21}H_{26}BrNO_4P$ 466.0777, found 466.0773.

**Diethyl (2-(5-cyano-1*H*-indol-3-yl)-2-(4-methoxyphenyl)ethyl)phosphonate (4aam):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4aam** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 47.4 mg; 58% yield; brown oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 9.67 (s, 1H), 7.72 (s, 1H), 7.29 (s, 2H), 7.20 (d, *J* = 6.4 Hz, 3H), 6.81 (d, *J* = 8.0 Hz, 2H), 4.71-4.54 (m, 1H), 3.97-3.83 (m, 4H), 3.76 (s, 3H), 2.72-2.60 (m, 1H), 2.57-2.45 (m, 1H), 1.21-1.13 (m, 6H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 158.3, 138.4, 135.7 (d, $J_{C\text{-}P}$ = 10.7 Hz), 128.4, 126.1, 124.9, 124.6, 124.0, 120.9, 119.5 (d, $J_{C\text{-}P}$ = 10.8 Hz), 114.0, 112.2, 102.0, 61.7 (d, $J_{C\text{-}P}$ = 6.7 Hz), 61.5 (d, $J_{C\text{-}P}$ = 6.7 Hz), 55.2, 36.4, 32.7 (d, $J_{C\text{-}P}$ = 138.8 Hz), 16.2 (d, *J* = 6.2 Hz) ,16.1 (d, *J* = 6.2 Hz). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 29.0. LRMS (EI, 70 eV) *m/z* (%): 412 ($M^+$, 22), 274 (100), 243 (12), 217 (9), 140 (3). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{22}H_{26}N_2O_4P$ 413.1625, found 413.1627.

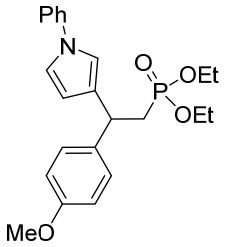
**Diethyl (2-(4-formyl-1*H*-indol-3-yl)-2-(4-methoxyphenyl)ethyl)phosphonate (4aan):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4aan** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 33.5 mg; 40% yield; yellow oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 10.28 (s, 1H), 9.73 (s, 1H), 7.60 (d, *J* = 7.2 Hz, 1H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.40 (s, 1H), 7.19-7.12 (m, 3H), 6.74 (d, *J* = 8.4 Hz, 2H), 5.37-5.28 (m, 1H), 4.05-3.88 (m, 4H), 3.72 (s, 3H), 2.72-2.60 (m, 1H), 2.54-2.42 (m, 1H), 1.25-1.17 (m,

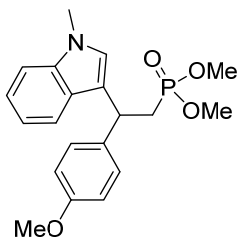
6H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 192.5, 158.0, 138.1, 136.8 (d, $J_{C-P}$ = 12.3 Hz), 129.9, 128.8, 126.4, 125.1, 124.6, 121.0, 118.7 (d, $J_{C-P}$ = 9.0 Hz) , 117.7, 113.8, 62.0 (d, $J_{C-P}$ = 6.6 Hz), 61.40 (d, $J_{C-P}$ = 6.6 Hz), 55.2, 37.8, 34.4 (d, $J_{C-P}$ = 136.8 Hz), 16.3 (d, $J_{C-P}$ = 6.2 Hz). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 28.9. LRMS (EI, 70 eV) *m/z* (%): 415 ($M^+$, 37), 277 (70), 264 (100), 220 (21), 144 (21). HRMS (ESI) m/z: [M + H]$^+$ Calcd for $C_{22}H_{27}NO_5P$ [M+H]$^+$ 416.1621, found 416.1619.

**Diethyl (2-(4-methoxyphenyl)-2-(1-phenyl-1*H*-pyrrol-2-yl)ethyl)phosphonate (4aao):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4aao** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 58.6 mg; 71% yield; brown oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.31 (s, 3H), 7.06 (s, 2H), 6.83 (d, *J* = 7.6 Hz, 2H), 6.66 (d, *J* = 7.6 Hz, 3H), 6.25 (d, *J* = 10.0 Hz, 2H), 4.35-4.26 (m, 1H), 3.95-3.83 (m, 1H), 3.82-3.65 (m, 6H), 2.60-2.49 (m, 1H), 2.40-2.27 (m, 1H), 1.15 (t, *J* = 7.0 Hz, 3H), 1.07 (t, *J* = 7.0 Hz, 3H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 158.0, 139.8, 135.6 (d, $J_{C-P}$ = 14.3 Hz), 135.4 (d, $J_{C-P}$ = 9.0 Hz), 128.7, 128.6, 127.4, 127.0, 122.4, 113.5, 107.7, 106.5, 61.3 (d, $J_{C-P}$ = 6.6 Hz), 61.2 (d, $J_{C-P}$ = 6.6 Hz), 55.2, 36.7 (d, $J_{C-P}$ = 2.6 Hz), 33.3 (d, $J_{C-P}$ = 139.2 Hz), 16.2 (d, $J_{C-P}$ = 6.4 Hz) ,16.1 (d, $J_{C-P}$ = 6.4 Hz). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 29.2. LRMS (EI, 70 eV) *m/z* (%): 413 ($M^+$, 24), 275 (52), 262 (24), 184 (100), 154(13). HRMS (ESI) m/z: [M + H]$^+$ Calcd for $C_{23}H_{29}NO_4P$ 414.1829, found 414.1825.

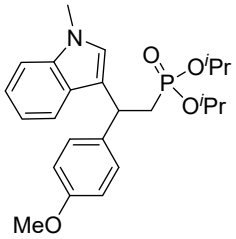
**Dimethyl (2-(4-methoxyphenyl)-2-(1-methyl-1*H*-indol-3-yl)ethyl)phosphonate (4aba):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4aba** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 50.7 mg; 68% yield; brown oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.46 (d, *J* = 8.0 Hz, 1H), 7.30-7.23 (m, 3H), 7.17 (t, *J* = 6.8 Hz, 1H), 7.02 (t, *J* = 7.6 Hz, 1H), 6.83 (t, *J* = 4.8 Hz, 3H), 4.68-4.60 (m, 1H), 3.75 (s, 3H), 3.71 (s, 3H),

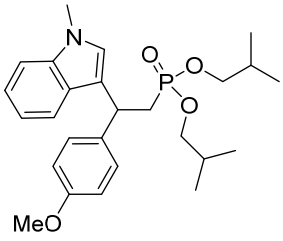
3.48-3.42 (m, 6H), 2.75-2.64 (m, 1H), 2.59-2.46 (m, 1H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 158.1, 137.3, 135.9 (d, $J_{C-P}$ = 7.1 Hz), 128.7, 126.5, 126.0, 121.7, 119.4, 118.9, 118.3 (d, $J_{C-P}$ = 14.7 Hz), 113.7, 109.1, 55.1, 52.1 (d, $J_{C-P}$ = 6.5 Hz), 52.0 (d, $J_{C-P}$ = 6.5 Hz), 36.4 (d, $J_{C-P}$ = 2.9 Hz), 32.6, 32.1 (d, $J_{C-P}$ = 138.0 Hz). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 32.6. LRMS (EI, 70 eV) *m/z* (%): 373 ($M^+$, 20), 263 (100), 250 (52), 207 (29), 109 (7). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{20}H_{25}NO_4P$ 374.1516, found 374.1511.

**Diisopropyl (2-(4-methoxyphenyl)-2-(1-methyl-1H-indol-3-yl)ethyl)phosphonate (4aca):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4aca** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 48.4 mg; 57% yield; yellow oil liquid;$^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.39 (d, *J* = 7.6 Hz, 1H), 7.21-7.14 (m, 3H), 7.08 (t, *J* = 7.2 Hz, 1H), 6.93 (t, *J* = 7.2 Hz, 1H), 6.77 (s, 1H), 6.72 (d, *J* = 7.6 Hz, 2H), 4.64-4.51 (m, 1H), 4.50-4.41 (m, 2H), 3.66 (s, 3H), 3.62 (s, 3H), 2.60-2.50 (m, 1H), 2.45-2.33 (m, 1H), 1.15-1.10 (m, 6H), 0.97 (t, *J* = 6.8 Hz, 6H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 158.0, 137.2, 136.5 (d, $J_{C-P}$ = 7.5 Hz), 128.8, 126.7, 126.1, 121.5, 119.5, 118.7, 118.6, 113.6, 109.0, 69.9 (d, $J_{C-P}$ = 1.7 Hz), 69.8 (d, $J_{C-P}$ = 1.7 Hz), 55.2, 36.7 (d, $J_{C-P}$ = 3.4 Hz), 34.3 (d, $J_{C-P}$ = 139.7 Hz), 32.6 , 24.0 (t, $J_{C-P}$ = 3.0 Hz), 23.8-23.5 (m). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 27.7. LRMS (EI, 70 eV) *m/z* (%): 429 ($M^+$, 2), 355 (4), 281 (19), 207 (100), 149 (96),135 (30). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{24}H_{33}NO_4P$ 430.2142, found 430.2144.

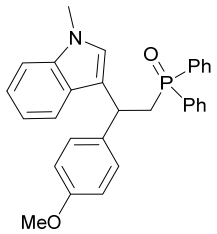
**Diisobutyl (2-(4-methoxyphenyl)-2-(1-methyl-1*H*-indol-3-yl)ethyl)phosphonate (4ada):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4ada** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 59.4 mg; 65% yield; brown oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.40 (d, *J* = 8.0 Hz, 1H), 7.23-7.15 (m, 3H), 7.10 (t, *J* = 7.2 Hz, 1H),

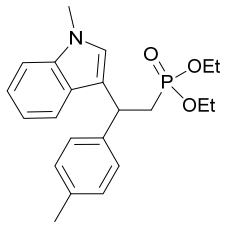
6.95 (t, $J$ = 7.6 Hz, 1H), 6.78 (s, 1H), 6.74 (d, $J$ = 7.6 Hz, 2H), 4.63-4.55 (m, 1H), 3.68 (s, 3H), 3.64 (s, 3H), 3.55-3.47 (m, 2H), 3.43-3.34 (m, 2H), 2.69-2.57 (m, 1H), 2.51-2.40 (m, 1H), 1.69-1.54 (m, 2H), 0.77-0.67 (m, 12H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 158.1, 137.3, 136.3 (d, $J_{C-P}$ = 7.5 Hz), 128.7, 126.6, 126.1, 121.6, 119.5, 118.8, 118.5 (d, $J_{C-P}$ = 13.7 Hz), 113.7, 109.1, 71.3 (d, $J_{C-P}$ = 5.0 Hz), 71.2 (d, $J_{C-P}$ = 5.0 Hz), 55.2, 36.6, 32.6, 32.1 (d, $J_{C-P}$ = 138.5 Hz), 29.1-28.9 (m), 18.7-18.5 (m). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 29.6. LRMS (EI, 70 eV) *m/z* (%): 457 ($M^+$, 15), 344 (3), 263 (100), 250 (24), 206 (8). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{26}H_{37}NO_4P$ 458.2455, found 458.2462.

**(2-(4-methoxyphenyl)-2-(1-methyl-1*H*-indol-3-yl)ethyl)diphenylphosphine oxide (4aea):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4aea** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 1:1, v/v). 38.1 mg; 41% yield; yellow oil liquid; $^1$H NMR ($CDCl_3$, 500 MHz) δ (ppm) 7.83-7.72 (m, 1H), 7.57 (t, $J$ = 9.5 Hz, 2H), 7.51-7.42 (m, 4H), 7.37 (d, $J$ = 8.5 Hz, 2H), 7.30 (d, $J$ = 8.0 Hz, 2H), 7.20 (d, $J$ = 8.1 Hz, 1H), 7.16 (d, $J$ = 7.5 Hz, 2H), 7.10 (d, $J$ = 9.0 Hz, 2H), 6.75 (s, 1H), 6.65 (d, $J$ = 8.0 Hz, 2H), 3.80 (d, $J$ = 8.5 Hz, 1H), 3.69 (s, 3H), 3.52 (s, 3H), 3.30-3.20 (m, 1H), 3.10-2.97 (m, 1H). $^{13}$C NMR ($CDCl_3$, 125 MHz) δ (ppm) 157.8, 137.2, 131.3 (d, $J_{C-P}$ = 10.0 Hz), 131.1, 130.7, 130.5 (d, $J_{C-P}$ = 9.0 Hz), 130.2 (d, $J_{C-P}$ = 9.0 Hz), 128.7, 128.3, 128.2, 127.7, 127.6, 127.0, 126.4, 121.3, 119.2 (d, $J_{C-P}$ = 136.0Hz), 113.5, 109.0, 55.1, 36.5 (d, $J_{C-P}$ = 71.0 Hz), 35.4, 32.3. $^{31}$P NMR ($CDCl_3$, 202 MHz) δ (ppm) 29.7. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{30}H_{29}NO_2P$ $[M+H]^+$ 466.1930, found 466.1933.

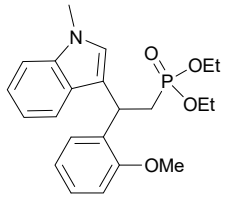
**Diethyl (2-(1-methyl-1*H*-indol-3-yl)-2-(p-tolyl)ethyl)phosphonatete (4baa):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4baa** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 40.0 mg; 52% yield; brown oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.42 (d, $J$ = 8.0 Hz, 1H), 7.20-7.15 (m, 3H), 7.10 (t, $J$ = 7.2 Hz, 1H), 7.00 (d, $J$

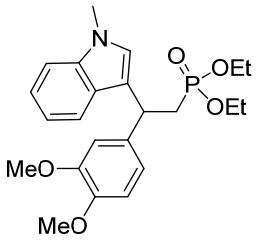
= 7.2 Hz, 2H), 6.95 (t, *J* = 7.2 Hz, 1H), 6.79 (s, 1H), 4.62-4.54 (m, 1H), 3.88-3.67 (m, 4H), 3.65 (s, 3H), 2.67-2.56 (m, 1H), 2.52-2.41 (m, 1H), 2.21 (s, 3H), 1.02 (q, *J* = 10.8 Hz, 6H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 141.0 (d, *J* = 7.7 Hz), 137.3, 135.8, 129.0, 127.7, 126.7, 126.1, 121.6, 119.5, 118.8, 118.3(d, $J_{C\text{-}P}$ = 13.9 Hz), 109.1, 61.3 (d, $J_{C\text{-}P}$ = 6.4 Hz), 61.2 (d, $J_{C\text{-}P}$ = 6.4 Hz), 36.9 (d, $J_{C\text{-}P}$ = 3.2 Hz), 33.5(d, $J_{C\text{-}P}$ = 138.2 Hz), 32.6, 21.0, 16.2 (d, $J_{C\text{-}P}$ = 6.8 Hz), 16.1 (d, $J_{C\text{-}P}$ = 6.8 Hz). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 29.8. LRMS (EI, 70 eV) *m/z* (%): 385 ($M^+$, 14), 247 (100), 234 (30), 218 (8), 115 (7). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{22}H_{29}NO_3P$ 386.1880, found 386.2009.

**Diethyl (2-(2-methoxyphenyl)-2-(1-methyl-1*H*-indol-3-yl)ethyl)phosphonate (4caa):**



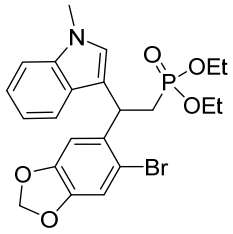

Following the typical experimental procedure on 0.2 mmol scale, compound **4caa** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 42.5 mg; 53% yield; brown oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.47 (d, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.12-7.04 (m, 2H), 6.95 (t, *J* = 7.2 Hz, 1H), 6.85 (s, 1H), 6.77 (t, *J* = 8.0 Hz, 2H), 5.07-5.00 (m, 1H), 3.82-3.65 (m, 8H), 3.64 (s, 3H), 2.59 (d, *J* = 7.2 Hz, 1H), 2.55 (d, *J* = 7.2 Hz, 1H), 1.03 (q, *J* = 7.6 Hz, 6H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 156.7, 137.0, 132.3 (d, $J_{C\text{-}P}$ = 9.1 Hz), 128.5, 127.3, 127.1, 126.7, 121.4, 120.4, 119.5, 118.6, 117.3 (d, $J_{C\text{-}P}$ = 11.5 Hz), 110.7, 109.0, 99.9, 61.2 (d, $J_{C\text{-}P}$ = 5.7 Hz), 61.1 (d, $J_{C\text{-}P}$ = 5.7 Hz), 55.4, 32.4 (d, $J_{C\text{-}P}$ = 42.9 Hz), 30.8, 30.4 (d, $J_{C\text{-}P}$ = 3.1 Hz), 16.11 (d, $J_{C\text{-}P}$ = 6.0 Hz), 16.0 (d, $J_{C\text{-}P}$ = 6.0 Hz). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 30.2. LRMS (EI, 70 eV) *m/z* (%): 401 ($M^+$, 20), 263 (100), 250 (30), 206 (10), 131 (2). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{22}H_{29}NO_4P$ 402.1829, found 402.1830.

**Diethyl (2-(3,4-dimethoxyphenyl)-2-(1-methyl-1*H*-indol-3-yl)ethyl)phosphonate (4daa):**



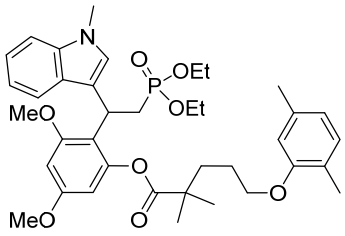
Following the typical experimental procedure on 0.2 mmol scale, compound **4daa** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 62.9 mg; 73% yield; brown oil liquid; $^{1}$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.43 (d, *J* = 8.0 Hz, 1H), 7.18 (d, *J* = 8.0 Hz, 1H), 7.11 (t, *J* = 7.2 Hz, 1H), 6.96 (t, *J* = 7.6 Hz, 1H), 6.85 (d, *J* = 9.6 Hz, 2H), 6.78 (s, 1H), 6.71 (d, *J* = 8.0 Hz, 1H), 4.64-4.53 (m, 1H), 3.87-3.78 (m, 2H), 3.76 (s, 6H), 3.74-3.67 (m, 2H), 3.65 (s, 3H), 2.67-2.55 (m, 1H), 2.51-2.40 (m, 1H), 1.08-1.00 (m, 6H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 148.7, 147.5, 137.3, 136.7 (d, $J_{C-P}$ = 8.2 Hz), 126.6, 126.1, 121.6, 119.6, 119.4, 118.8, 118.2 (d, $J_{C-P}$ = 14.0 Hz), 111.4, 111.0, 109.1, 61.3 (d, $J_{C-P}$ = 6.8 Hz), 61.2 (d, $J_{C-P}$ = 6.8 Hz), 55.8, 36.9 (d, $J_{C-P}$ = 3.6 Hz), 32.9 (d, $J_{C-P}$ = 138.2 Hz), 32.6, 16.1 (t, $J_{C-P}$ = 7.0 Hz). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 29.8. LRMS (EI, 70 eV) *m/z* (%): 431 ($M^+$, 16), 293 (100), 280 (24), 207 (9), 131 (6). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{23}H_{31}NO_5P$ 432.1934, found 432.1937.

**Diethyl-(2-(6-bromobenzo[*d*][1,3]dioxol-5-yl)-2-(1-methyl-1*H*-indol-3-yl)ethyl)phosphonate (4eaa):**



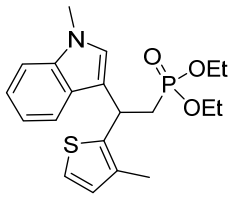
Following the typical experimental procedure on 0.2 mmol scale, compound **4eaa** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 49.5 mg; 50% yield; yellow oil liquid; $^{1}$H NMR ($CDCl_3$, 500 MHz) δ (ppm) 7.49 (d, *J* = 8.0 Hz, 1H), 7.27(t, *J* = 4.0 Hz, 2H), 7.19 (t, *J* = 7.5 Hz, 1H), 7.06-7.02 (m, 2H), 7.01 (s, 1H), 6.71 (s, 1H), 5.89 (s, 1H), 5.84 (s, 1H), 5.17-5.10 (m, 1H), 4.00-3.87 (m, 4H), 3.76 (s, 3H), 2.64-2.54 (m, 1H), 2.46-2.35 (m, 1H), 1.20-1.15 (m, 6H). $^{13}$C NMR ($CDCl_3$, 125 MHz) δ (ppm) 147.4, 146.7, 137.2, 136.9 (d, $J_{C-P}$ = 10.5 Hz), 126.8, 126.7, 121.8, 119.5, 119.0, 116.6 (d, $J_{C-P}$ = 10.0 Hz), 114.0, 112.4, 109.1, 108.8, 101.5, 61.5 (d, $J_{C-P}$ = 6.5 Hz), 61.4 (d, *J* = 6.5 Hz), 36.2 (d, $J_{C-P}$ = 3.0 Hz), 32.8, 31.7, 16.26 (t, $J_{C-P}$ = 6.0 Hz). $^{31}$P NMR ($CDCl_3$, 202 MHz) δ (ppm) 28.3. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{22}H_{26}BrNO_5P$ 494.0726, found 494.0726.

**2-(2-(diethoxyphosphoryl)-1-(1-methyl-1*H*-indol-3-yl)ethyl)-3,5-dimethoxyphenyl 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate (4faa) (Gemfibrozi derivative):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4faa** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 51.9 mg; 39% yield; yellow oil liquid; $^{1}$H NMR ($CDCl_3$, 500 MHz) δ (ppm) 7.64 (d, *J* = 8.0 Hz, 1H), 7.20 (d, *J* = 8.0 Hz, 1H), 7.15 (t, *J* = 7.5 Hz, 1H), 7.04 (t, *J* = 7.5 Hz, 1H), 6.98 (d, *J* = 7.5 Hz, 1H), 6.78 (s, 1H), 6.64 (d, *J* = 7.5 Hz, 1H), 6.52 (s, 1H), 6.34 (d, *J* = 2.0 Hz, 1H), 6.22 (d, *J* = 2.5 Hz, 1H), 3.98-3.78 (m, 5H), 3.75 (s, 3H), 3.74 (s, 3H), 3.66 (s, 3H), 2.95-2.85 (m, 1H), 2.80-2.70 (m, 1H), 2.30 (s, 3H), 2.13 (s, 3H), 1.83-1.68 (m, 6H), 1.36 (s, 3H), 1.33 (s, 3H), 1.18-1.11 (m, 6H). $^{13}$C NMR ($CDCl_3$, 125 MHz) δ (ppm) 175.9, 159.7, 159.0, 156.9, 136.9, 136.4, 130.2, 127.0 (d, $J_{C-P}$ = 12.0 Hz), 123.4, 121.2, 120.6, 119.2, 118.6, 116.4 (d, $J_{C-P}$ = 6.0 Hz), 116.0, 115.9, 111.9, 109.0, 99.5, 96.9, 67.7, 61.2 (d, $J_{C-P}$ = 6.5 Hz), 61.1 (d, $J_{C-P}$ = 6.5 Hz), 55.5, 55.4, 42.6, 37.0, 32.6, 25.2, 25.1, 25.0, 21.4, 16.3 (d, $J_{C-P}$ = 6.0 Hz), 16.2 (d, $J_{C-P}$ = 6.0 Hz), 15.7. $^{31}$P NMR ($CDCl_3$, 202 MHz) δ (ppm) 31.1. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{38}H_{51}NO_8P$ 680.3347, found 680.3351.

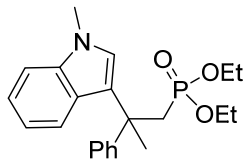
**Diethyl (2-(1-methyl-1*H*-indol-3-yl)-2-(3-methylthiophen-2-yl)ethyl)phosphonate (4gaa):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4gaa** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 32.8 mg; 42% yield; yellow oil liquid; $^{1}$H NMR ($CDCl_3$, 500 MHz) δ (ppm) 7.57 (d, *J* = 8.0 Hz, 1H), 7.26 (m, *J* = 8.0 Hz, 1H), 7.20 (t, *J* = 7.5 Hz, 1H), 7.10-7.02 (m, 2H), 6.92 (s, 1H), 6.75 (d, *J* = 5.5 Hz, 1H),5.05-4.98(m,1H) 3.95-3.86 (m, 2H), 3.84-3.74 (m, 2H), 3.73(s, 3H), 2.78-2.67 (m, 1H), 2.60-2.50 (m, 1H), 2.30 (s, 3H), 1.16 (t, *J* = 7.0 Hz, 3H), 1.10 (t, *J* = 7.0 Hz, 3H). $^{13}$C NMR ($CDCl_3$, 125 MHz) δ (ppm) 142.0 (d, $J_{C-P}$ = 8.0 Hz), 137.1, 132.8, 129.8, 126.5, 126.2, 121.7, 119.2, 119.0, 118.0 (d, $J_{C-P}$ = 13.5 Hz), 109.2 , 61.4 (d, $J_{C-P}$ = 3.0 Hz), 61.3 (d, $J_{C-P}$ = 3.0 Hz),

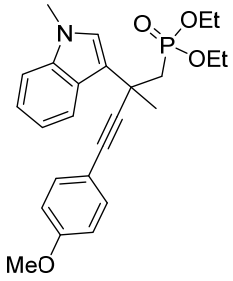
34.2 (d, $J_{C\text{-}P}$ = 138.0 Hz), 32.7, 30.6 (d, $J_{C\text{-}P}$ = 3.0 Hz), 16.2 (d, $J_{C\text{-}P}$ = 6.5 Hz), 16.1 (d, $J_{C\text{-}P}$ = 6.5 Hz), 14.0. $^{31}$P NMR ($CDCl_3$, 202 MHz) δ (ppm) 28.9. HRMS (ESI) m/z: [M + H]$^+$ Calcd for $C_{20}H_{27}NO_3PS$ 392.1444, found 392.1448.

**Diethyl (2-(1-methyl-1*H*-indol-3-yl)-2-phenylpropyl)phosphonate (4haa):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4haa** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 40.0 mg; 52% yield; brown oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.28 (d, *J* = 7.6 Hz, 2H), 7.16 (t, *J* = 7.6 Hz, 3H), 7.10-7.01 (m, 2H), 6.99 (s, 1H), 6.81 (d, *J* = 8.0 Hz, 1H), 6.75 (t, *J* = 7.6 Hz, 1H), 3.80-3.68 (m, 4H), 3.65-3.50 (m, 2H), 3.35-3.25 (h, 1H), 2.86-2.61 (m, 2H), 1.94 (s, 3H), 1.03 (t, *J* = 6.8 Hz, 3H), 0.81 (t, *J* = 6.8 Hz, 3H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 148.3 (d, $J_{C\text{-}P}$ = 12.2 Hz), 137.7, 128.0, 126.6, 126.4, 126.2, 125.9, 122.4 (d, $J_{C\text{-}P}$ = 11.2 Hz), 121.2, 120.9, 118.5, 109.0, 60.9 (d, $J_{C\text{-}P}$ = 7.2 Hz), 60.8 (d, $J_{C\text{-}P}$ = 7.2 Hz), 40.0 (d, $J_{C\text{-}P}$ = 2.1 Hz), 37.8 (d, *J* = 141.1 Hz), 32.7, 28.9, 16.2 (d, $J_{C\text{-}P}$ = 6.5 Hz,), 15.8 (d, $J_{C\text{-}P}$ = 6.5 Hz,). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 28.7. LRMS (EI, 70 eV) *m/z* (%): 385 (M$^+$, 11), 370 (4), 234 (100), 218 (7), 103 (11). HRMS (ESI) m/z: [M + H]$^+$ Calcd for $C_{22}H_{29}NO_3P$ 386.1880, found 386.1879.

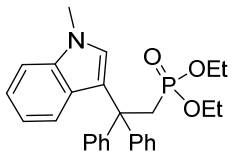
**Diethyl (4-(4-methoxyphenyl)-2-methyl-2-(1-methyl-1*H*-indol-3-yl)but-3-yn-1-yl) phosphonate (4iaa):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4iaa** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 41.7 mg; 51% yield; brown oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.87 (d, *J* = 8.0 Hz, 1H), 7.30 (d, *J* = 7.6 Hz, 2H), 7.25-7.12 (m, 3H), 7.05 (t, *J* = 7.6 Hz, 1H), 7.01 (s, 1H), 6.73 (d, *J* = 7.6 Hz, 2H), 4.00-3.86 (m, 2H), 3.82-3.73 (m, 2H), 3.72 (s, 3H), 3.68 (s, 3H), 2.68-2.51 (m, 2H), 1.96 (s, 3H), 1.12 (t, *J* = 6.8 Hz, 3H), 0.98 (t, *J* = 6.8 Hz, 3H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 159.2, 137.8, 132.9, 125.7, 125.5, 121.5, 120.6, 119.5 (d, $J_{C\text{-}P}$ = 12.0 Hz), 118.9, 115.9, 113.7, 109.4, 93.2, 81.7, 61.4 (d, $J_{C\text{-}P}$ = 6.6 Hz), 61.2 (d, $J_{C\text{-}P}$ =

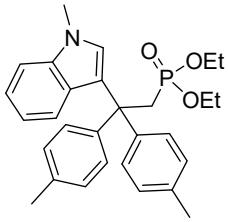
6.6 Hz), 55.2, 39.0 (d, $J$ = 137.6 Hz), 32.7, 32.2 (d, $J_{C\text{-}P}$ = 3.0 Hz), 28.7, 16.3 (d, $J_{C\text{-}P}$ = 6.2 Hz), 16.1 (d, $J_{C\text{-}P}$ = 6.2 Hz). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 26.7; LRMS (EI, 70 eV) *m/z* (%): 439 ($M^+$, 7), 288 (93), 207 (100), 133 (13), 96 (3). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{25}H_{31}NO_4P$ 440.1985, found 440.1882.

**Diethyl (2-(1-methyl-1*H*-indol-3-yl)-2,2-diphenylethyl)phosphonate (4jaa):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4jaa** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 49.2 mg; 55% yield; White solid, mp 141.2-143.1 ºC (uncorrected); $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.44 (d, $J$ =8.0 Hz, 4H), 7.23 (t, $J$ = 8.0 Hz, 5H), 7.17-7.08 (m, 3H), 6.97 (s, 1H), 6.91 (d, $J$ = 8.0 Hz, 1H), 6.85 (t, $J$ = 7.2 Hz, 1H), 3.71 (s, 3H), 3.69-3.60 (m, 2H), 3.50-3.38 (m, 2H), 3.36 (s, 1H), 3.31 (s, 1H), 3.31 (s, 1H), 0.96 (t, $J$ = 1.8 Hz, 6H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 146.1 (d, $J_{C\text{-}P}$ = 8.3 Hz), 137.5, 129.6, 128.6, 127.6, 127.0, 126.0, 121.9, 121.0, 119.9 (d, $J_{C\text{-}P}$ = 6.9 Hz), 118.5, 109.0, 60.8 (d, $J_{C\text{-}P}$ = 6.6 Hz), 50.0 (d, $J_{C\text{-}P}$ = 2.8 Hz), 38.8 (d, $J_{C\text{-}P}$ = 140.8 Hz) , 32.7, 16.0 (d, $J_{C\text{-}P}$ = 6.4 Hz). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 27.5. LRMS (EI, 70 eV) *m/z* (%): 447 ($M^+$, 7), 296 (100), 218 (8), 165 (6), 131 (4). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{27}H_{31}NO_3P$ 448.2036, found 448.2032.

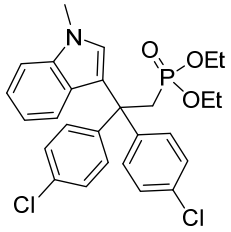
**Diethyl (2-(1-methyl-1*H*-indol-3-yl)-2,2-di-p-tolylethyl)phosphonate (4kaa):**




Following the typical experimental procedure on 0.2 mmol scale, compound **4kaa** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 50.3 mg; 53% yield; yellow oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.31 (d, $J$ = 7.6 Hz, 4H), 7.24 (d, $J$ = 8.4 Hz, 1H), 7.12 (t, $J$ = 7.2 Hz, 1H), 7.03 (d, $J$ = 7.6 Hz, 4H), 6.95 (t, $J$ = 4.8 Hz, 2H), 6.86 (t, $J$ = 7.2 Hz, 1H), 3.72 (s, 3H), 3.70-3.53 (m, 2H), 3.50-3.35 (m, 2H), 3.32 (s, 1H), 3.27 (s, 1H), 2.28 (s, 6H), 0.96 (t, $J$ = 6.8 Hz, 6H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 143.5 (d, $J_{C\text{-}P}$ = 8.4 Hz), 137.6, 135.3, 129.7, 128.4, 128.3, 127.1, 122.1, 121.0, 120.1(d, $J_{C\text{-}P}$ = 6.8 Hz), 118.4, 109.0 , 60.8 (d, $J_{C\text{-}P}$ = 6.6 Hz), 49.4, 38.7 (d, $J_{C\text{-}P}$ = 140.7 Hz), 32.7, 20.8, 16.0 (d, $J_{C\text{-}P}$ = 6.5 Hz). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 27.7. LRMS (EI, 70 eV) *m/z* (%): 475 ($M^+$,

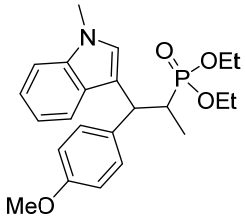
6), 324 (100), 308 (3), 207 (7), 131 (3). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{29}H_{35}NO_3P$ 476.2349, found 476.2346.

**Diethyl (2,2-bis(4-chlorophenyl)-2-(1-methyl-1*H*-indol-3-yl)ethyl)phosphonate (4laa):**



Following the typical experimental procedure on 0.2 mmol scale, compound **4laa** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 58.8 mg; 57% yield; yellow oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.35 (d, *J* = 8.0 Hz, 4H), 7.26 (d, *J* = 8.4 Hz, 1H), 7.21 (d, *J* = 7.6 Hz, 4H), 7.18-7.12 (m, 1H), 6.93-6.86 (m, 3H), 3.73 (s, 3H), 3.72-3.64 (m, 2H), 3.53-3.44 (m, 2H), 3.28 (s, 1H), 3.23 (s, 1H), 0.99 (t, *J* = 6.8 Hz, 6H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 144.2 (d, $J_{C\text{-}P}$ = 8.2 Hz), 137.6, 132.1, 130.1, 129.4, 127.8, 126.5, 121.6, 121.4, 119.2 (d, $J_{C\text{-}P}$ = 7.4 Hz), 118.8, 109.3, 61.1 (d, $J_{C\text{-}P}$ = 6.8 Hz), 49.5 (d, $J_{C\text{-}P}$ = 2.6 Hz), 38.6 (d, $J_{C\text{-}P}$ = 141.4 Hz), 32.7, 16.0 (d, $J_{C\text{-}P}$ = 6.3 Hz). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 26.5. LRMS (EI, 70 eV) *m/z* (%): 517 ($M^+$, 5), 364 (100), 328 (6), 252 (9), 146 (8). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{27}H_{29}Cl_2NO_3P$ 516.1257, found 516.1258.

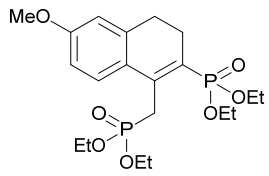
**Diethyl (1-(4-methoxyphenyl)-1-(1-methyl-1*H*-indol-3-yl)propan-2-yl)-phosphonate (4maa):**



Following the typical experimental procedure on 0.2 mmol scale, compound **4maa** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 57.3 mg; 69% yield; yellow oil liquid; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.48 (d, *J* = 8.0 Hz, 1H), 7.26-7.14 (m, 3H), 7.09 (t, *J* = 7.2 Hz, 1H), 6.99-6.92 (m, 2H), 6.70 (d, *J* = 7.6 Hz, 2H), 4.47 (t, *J* = 9.6 Hz, 1H), 3.88-3.73 (m, 4H), 3.65 (s, 6H), 3.59-3.50 (m, 1H), 1.22-1.52 (m, 3H), 1.09-0.98 (m, 6H). $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 157.8, 136.8, 136.2 (d, $J_{C\text{-}P}$ = 6.5 Hz), 129.2, 127.6, 126.7, 121.4, 119.5, 118.8, 116.0 (d, $J_{C\text{-}P}$ = 14.0 Hz), 113.4, 109.0, 61.2 (d, $J_{C\text{-}P}$ = 7.3 Hz), 61.1 (d, $J_{C\text{-}P}$ = 7.3 Hz), 55.2, 42.8, 36.9 (d, $J_{C\text{-}P}$ = 138.8 Hz), 32.7, 16.2 (d, $J_{C\text{-}P}$ = 5.9 Hz), 13.2 (d, $J_{C\text{-}P}$ = 5.4 Hz). $^{31}$P NMR ($CDCl_3$, 162 MHz) δ (ppm) 33.3. LRMS (EI, 70 eV) *m/z*

(%): 415 ($M^+$, 8), 277 (37), 250 (100), 206 (19), 131 (4). HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{24}H_{33}NO_4P$ 416.1985, found 416.1984.

**Diethyl (1-((diethoxyphosphoryl)methyl)-6-methoxy-3,4-dihydronaphthalen-2-yl)phosphonate (5oa):**

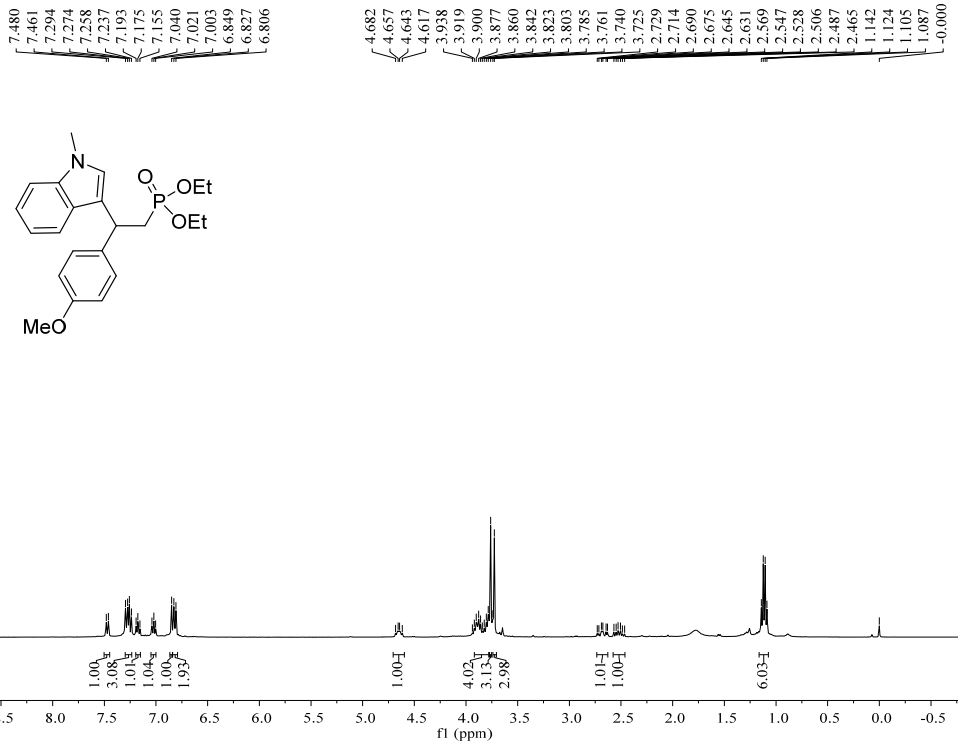



Following the typical experimental procedure on 0.2 mmol scale, compound **5oa** was obtained by silica gel column chromatography (eluent: petroleum ether/EtOAc = 2:1, v/v). 17.8 mg; 20% yield; yellow oil liquid; $^1$H NMR ($CDCl_3$, 500 MHz) δ (ppm) 7.58 (d, $J$ = 8.5 Hz, 1H), 6.80-6.76 (m, 1H), 6.70 (d, $J$ = 2.5 Hz, 1H), 4.21-4.10 (m, 4H), 4.06-3.99 (m, 4H), 3.82 (s, 3H), 2.70 (t, $J$ = 7.0 Hz, 2H), 2.51-2.26 (m, 2H), 2.07-2.01 (m, 2H), 1.34 (t, $J$ = 7.0 Hz, 6H), 1.21 (t, $J$ = 7.0 Hz, 6H). $^{13}$C NMR ($CDCl_3$, 125 MHz) δ (ppm) 160.1, 142.0, 139.6, 127.8, 126.8 (d, $J_{C\text{-}P}$ = 17.5 Hz), 122.7 (d, $J_{C\text{-}P}$ = 12.0 Hz), 121.2 (d, $J_{C\text{-}P}$ = 12.0 Hz), 113.1, 111.1, 61.8 (d, $J_{C\text{-}P}$ = 6.5 Hz), 61.5 (d, $J_{C\text{-}P}$ = 6.5 Hz), 55.2 , 28.4 (d, $J_{C\text{-}P}$ = 7.0 Hz), 28.0 (d, $J_{C\text{-}P}$ = 6.0 Hz), 26.9 (d, $J_{C\text{-}P}$ = 6.0 Hz), 25.4 (d, $J_{C\text{-}P}$ = 3.0 Hz), 25.3 (d, $J_{C\text{-}P}$ = 3.0 Hz), 16.4 (d, $J_{C\text{-}P}$ = 6.5 Hz), 16.3 (d, $J_{C\text{-}P}$ = 6.5 Hz). $^{31}$P NMR ($CDCl_3$, 202 MHz) δ (ppm) 26.4, 20.1. HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{20}H_{33}O_7P_2$ 447.1696, found 447.1699.

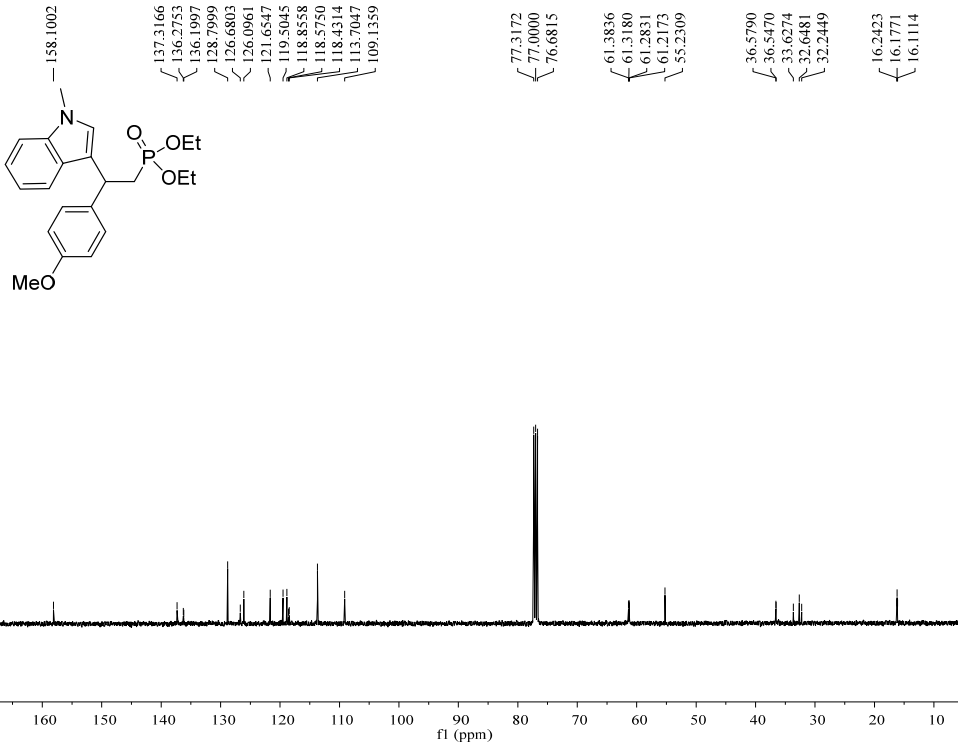
# (C) Spectra

## Diethyl (2-(4-methoxyphenyl)-2-(1-methyl-1*H*-indol-3-yl)ethyl)phosphonate (4aaa):

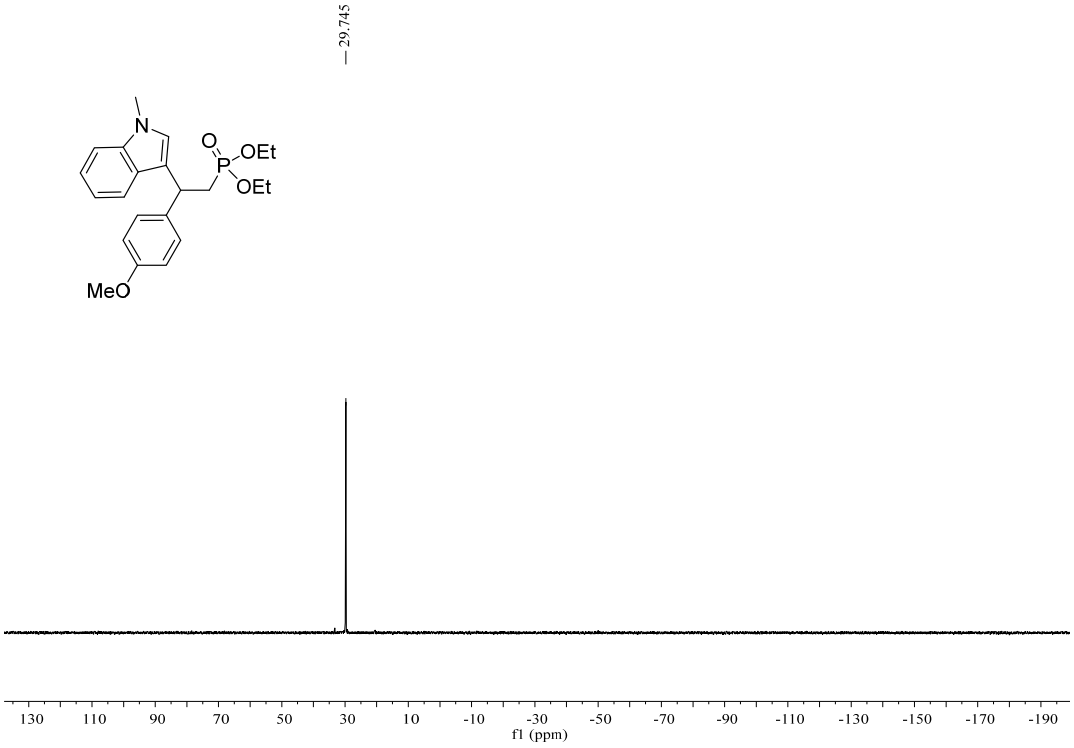
$^{1}$H NMR ($CDCl_3$, 400 MHz)




$^{13}$C NMR ($CDCl_3$, 100 MHz)

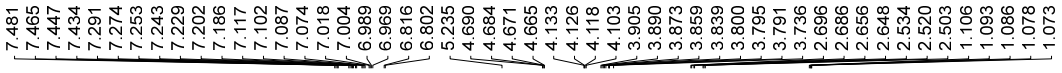


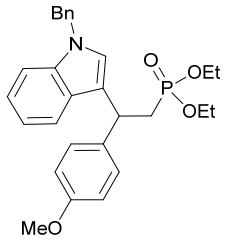
$^{31}P$ NMR ($CDCl_3$, 162 MHz)

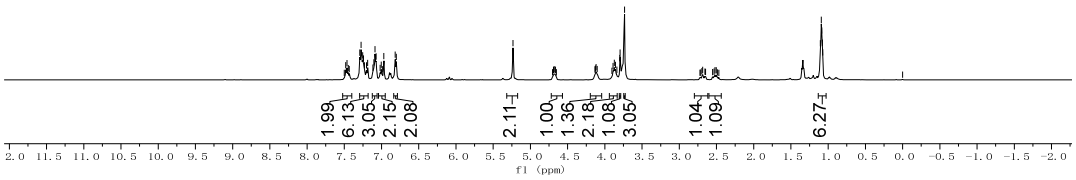


**Diethyl-(2-(1-benzyl-1H-indol-3-yl)-2-(4-methoxyphenyl)ethyl)phosphonate (4aab):**

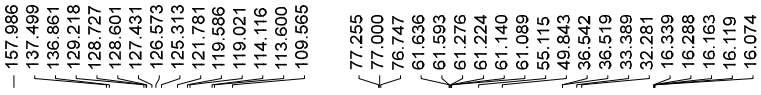
$^{1}$H NMR ($CDCl_3$, 500 MHz)

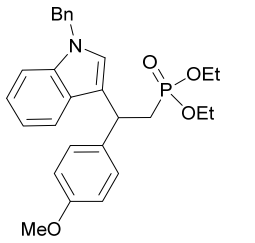


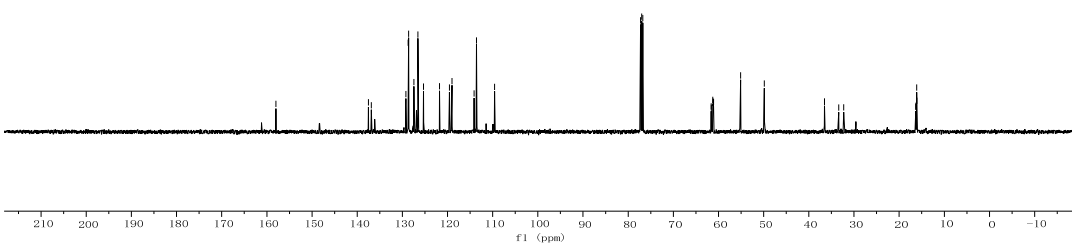



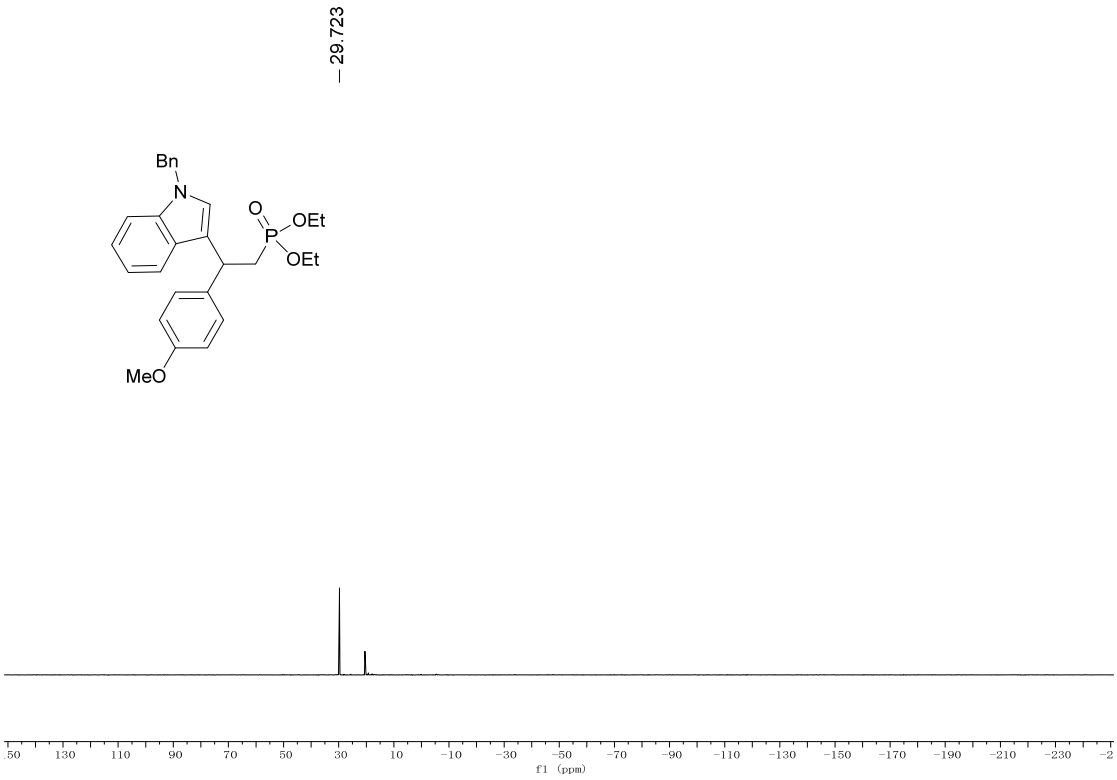

$^{13}$C NMR ($CDCl_3$, 125 MHz)



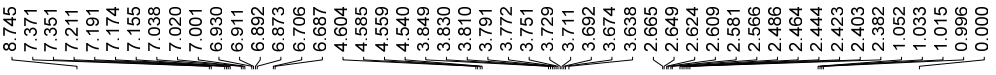



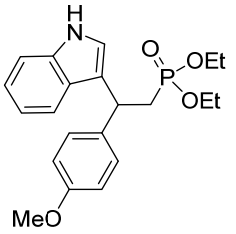
$^{31}P$ NMR ($CDCl_3$, 202 MHz)

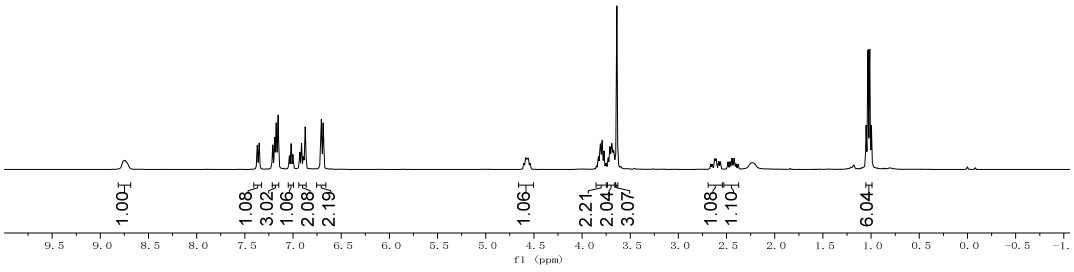


**Diethyl (2-(1*H*-indol-3-yl)-2-(4-methoxyphenyl)ethyl)phosphonate (4aae):**

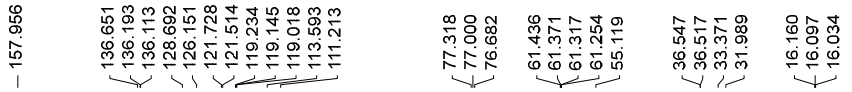
$^{1}$H NMR ($CDCl_3$, 400 MHz)

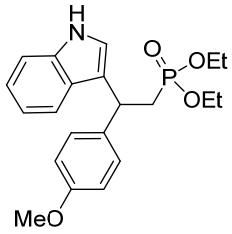


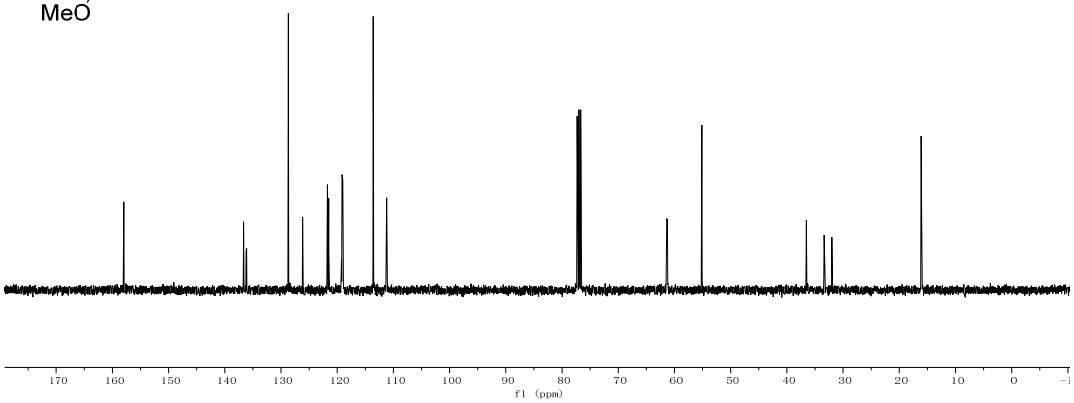



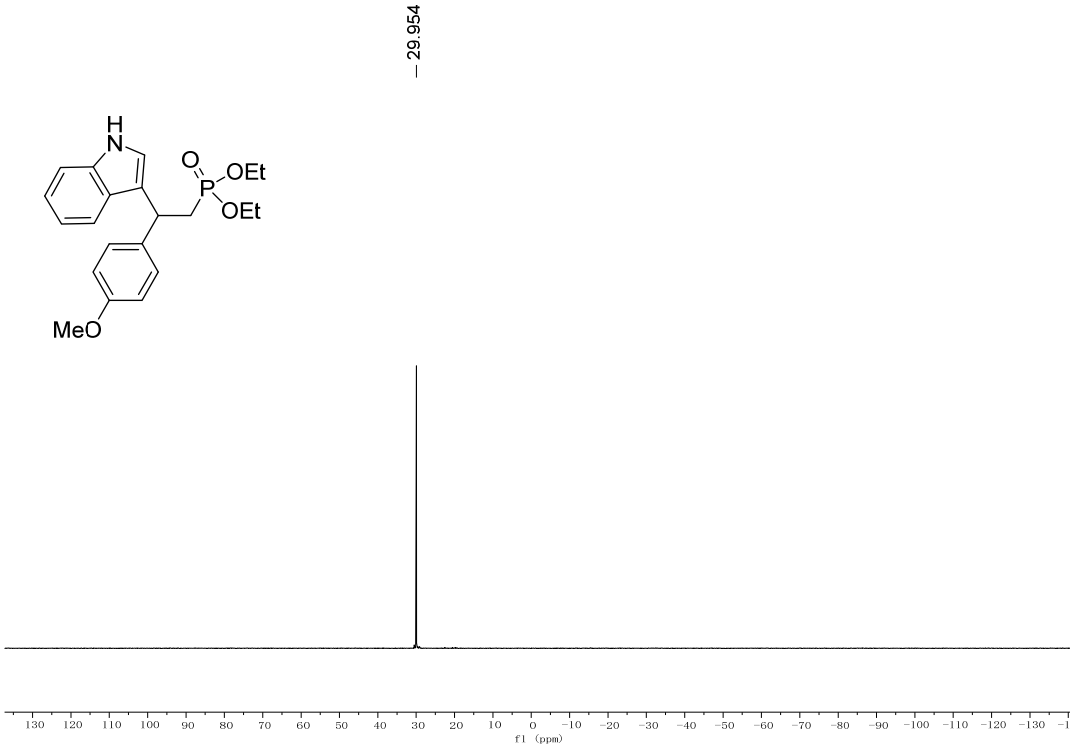

$^{13}$C NMR ($CDCl_3$, 100 MHz)



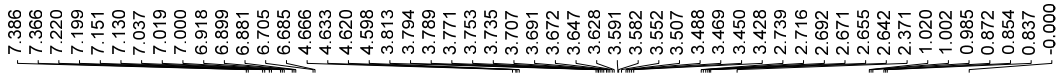



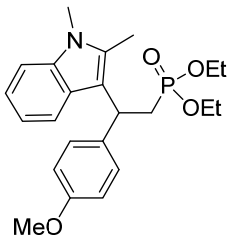
$^{31}P$ NMR ($CDCl_3$, 162 MHz)

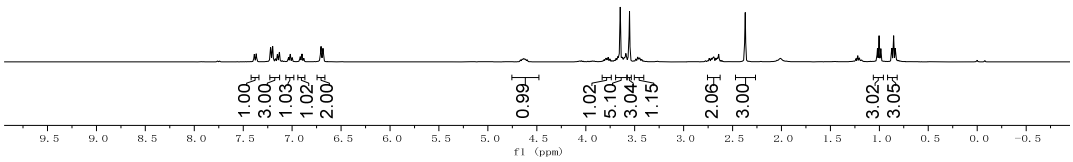


**Diethyl (2-(1,2-dimethyl-1H-indol-3-yl)-2-(4-methoxyphenyl)ethyl)phosphonate (4aaf):**

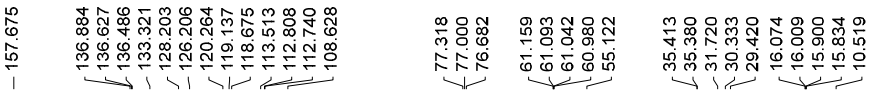
$^{1}$H NMR ($CDCl_3$, 400 MHz)

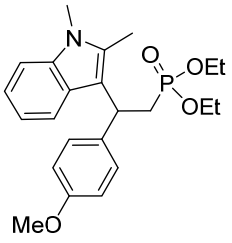


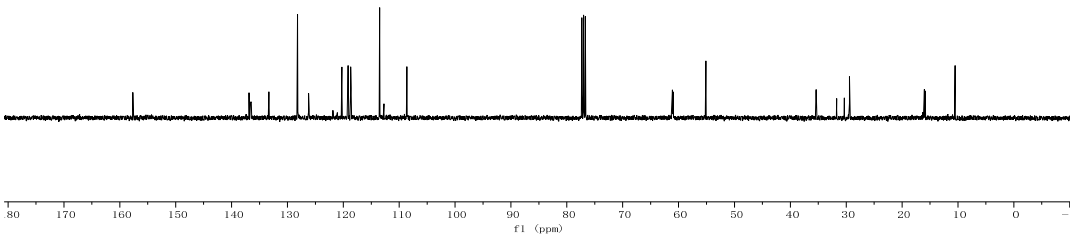



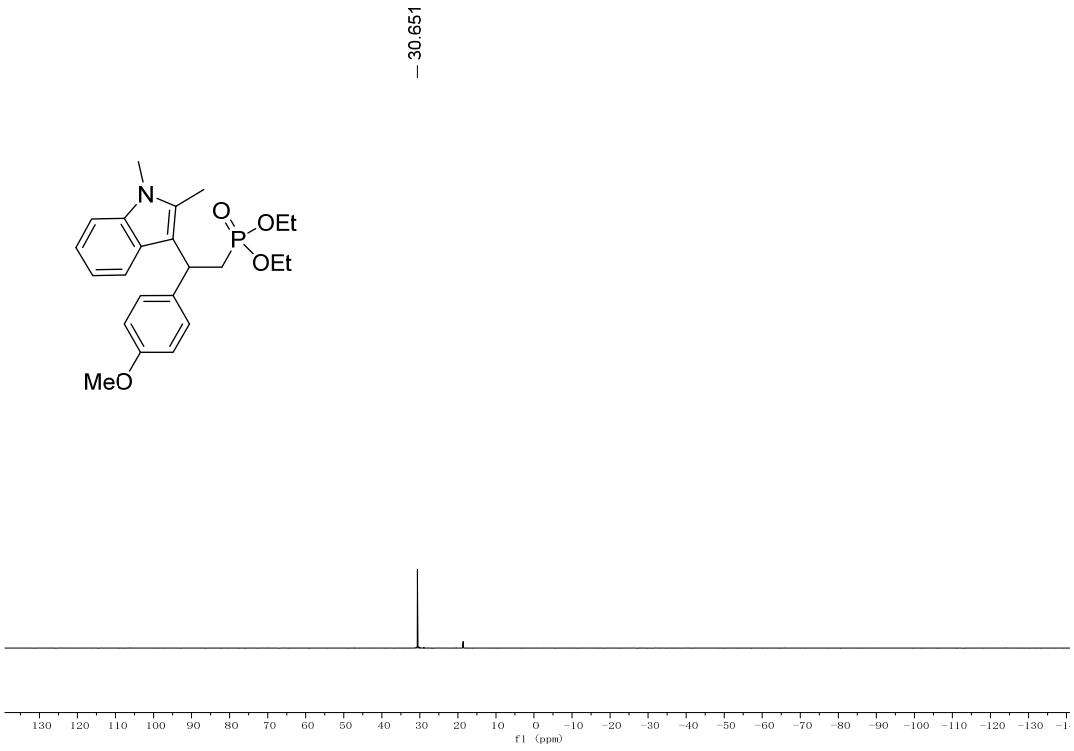

$^{13}$C NMR ($CDCl_3$, 100 MHz)



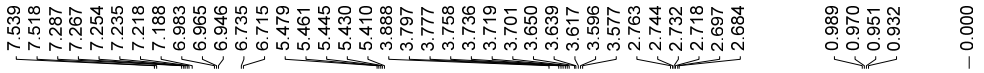



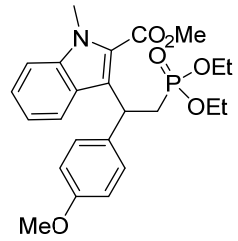
$^{31}P$ NMR ($CDCl_3$, 162 MHz)

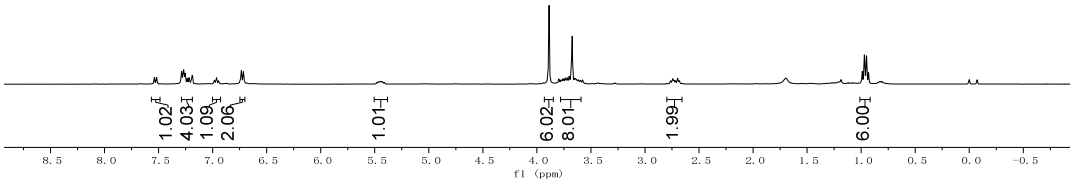


**Methyl 3-(2-(diethoxyphosphoryl)-1-(4-methoxyphenyl)ethyl)-1-methyl-1*H*-indole-2-carboxylate (4aag):**

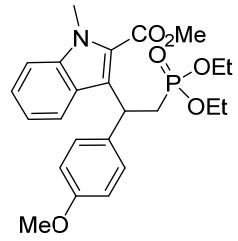
$^{1}$H NMR ($CDCl_3$, 400 MHz)



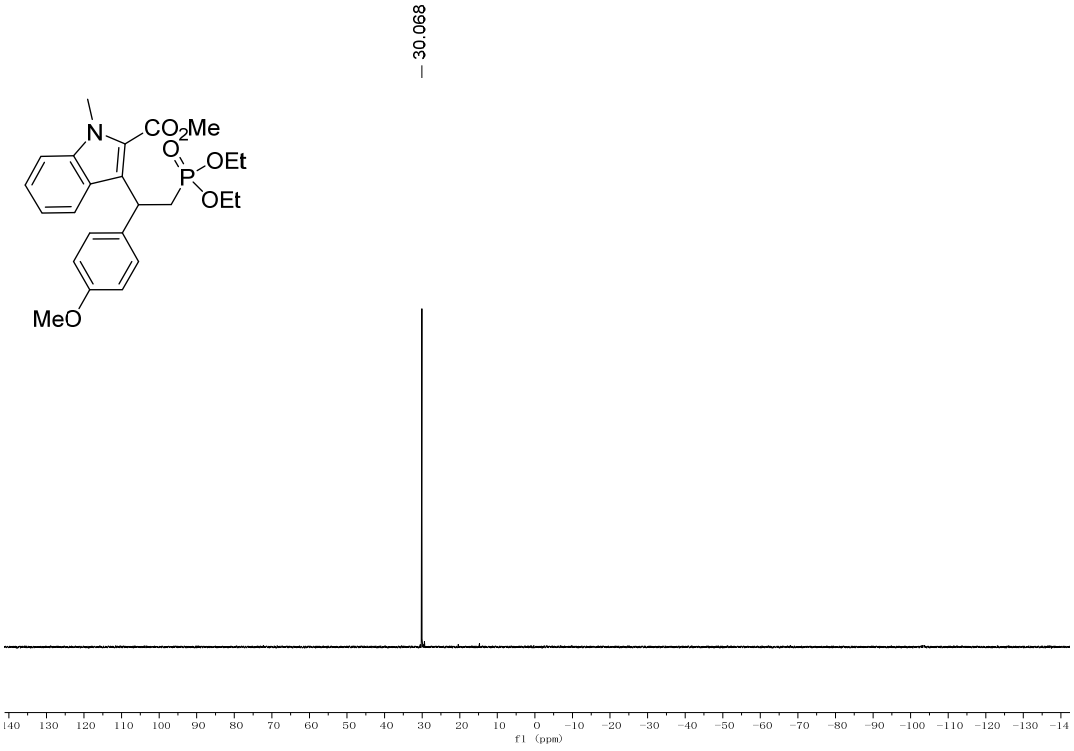




$^{13}$C NMR ($CDCl_3$, 100 MHz)

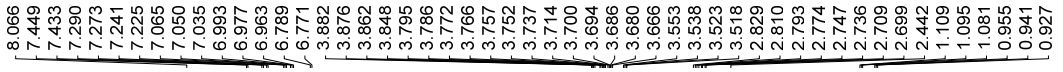



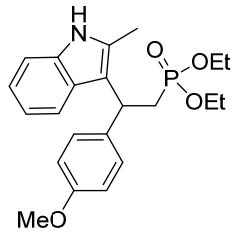
$^{31}P$ NMR ($CDCl_3$, 162 MHz)

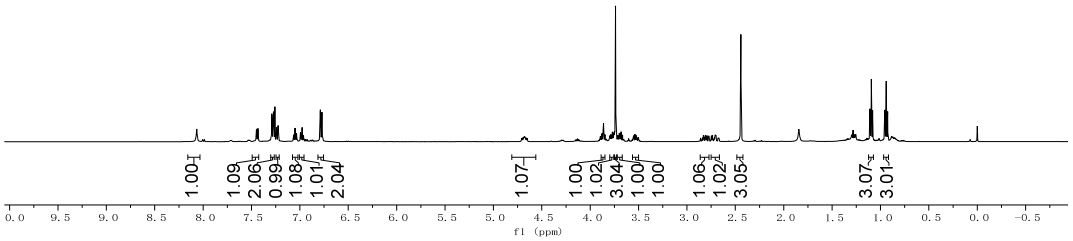


**Diethyl-(2-(4-methoxyphenyl)-2-(2-methyl-1*H*-indol-3-yl)ethyl)phosphonate (4aah):**

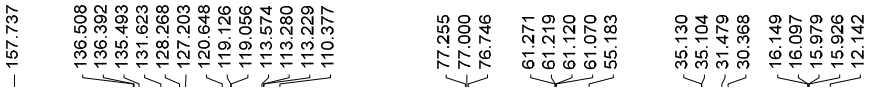
$^{1}$H NMR ($CDCl_3$, 500 MHz)

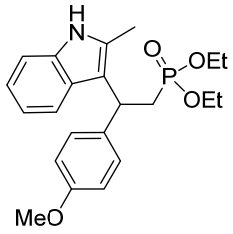


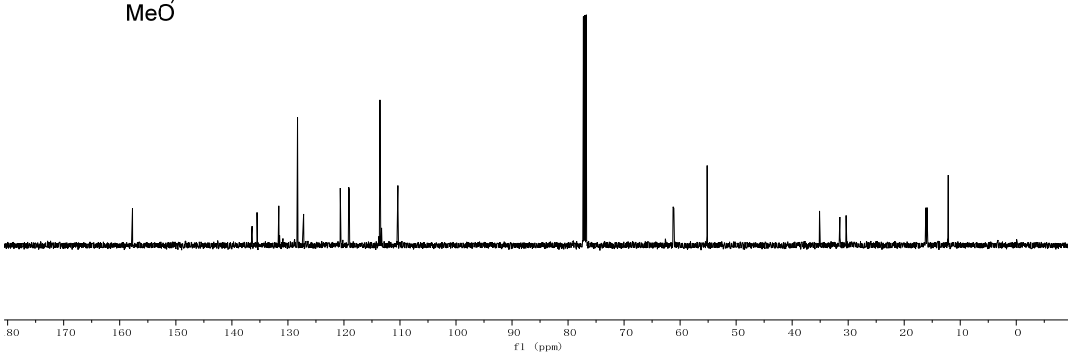



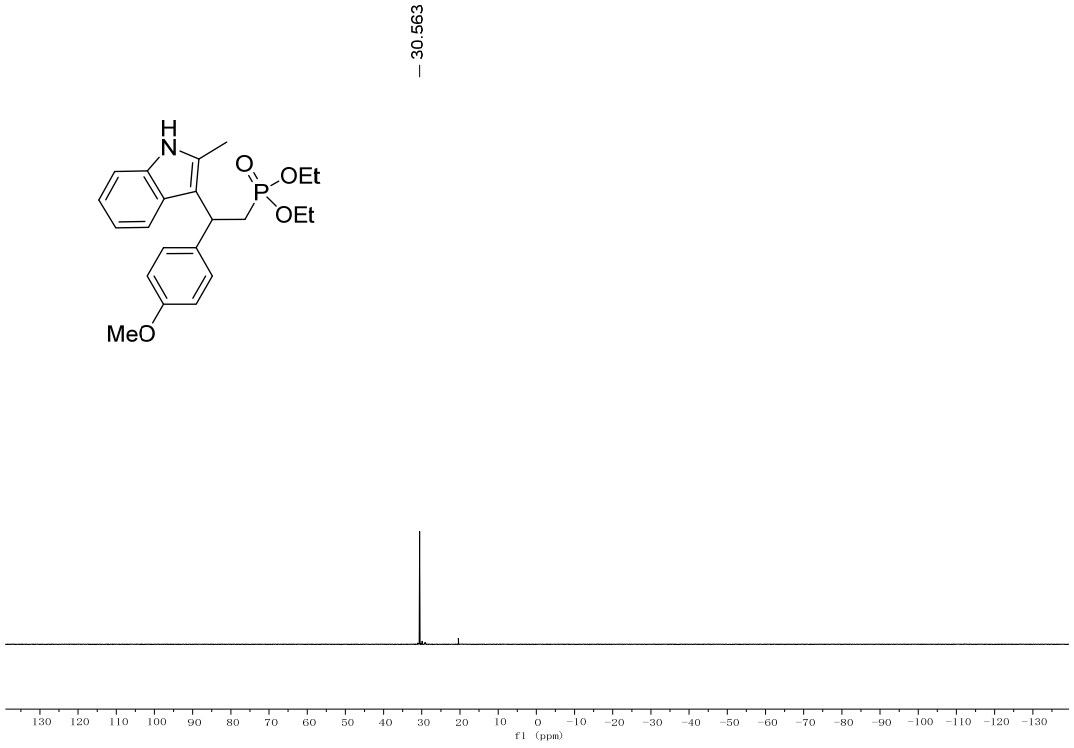

$^{13}$C NMR ($CDCl_3$, 125 MHz)



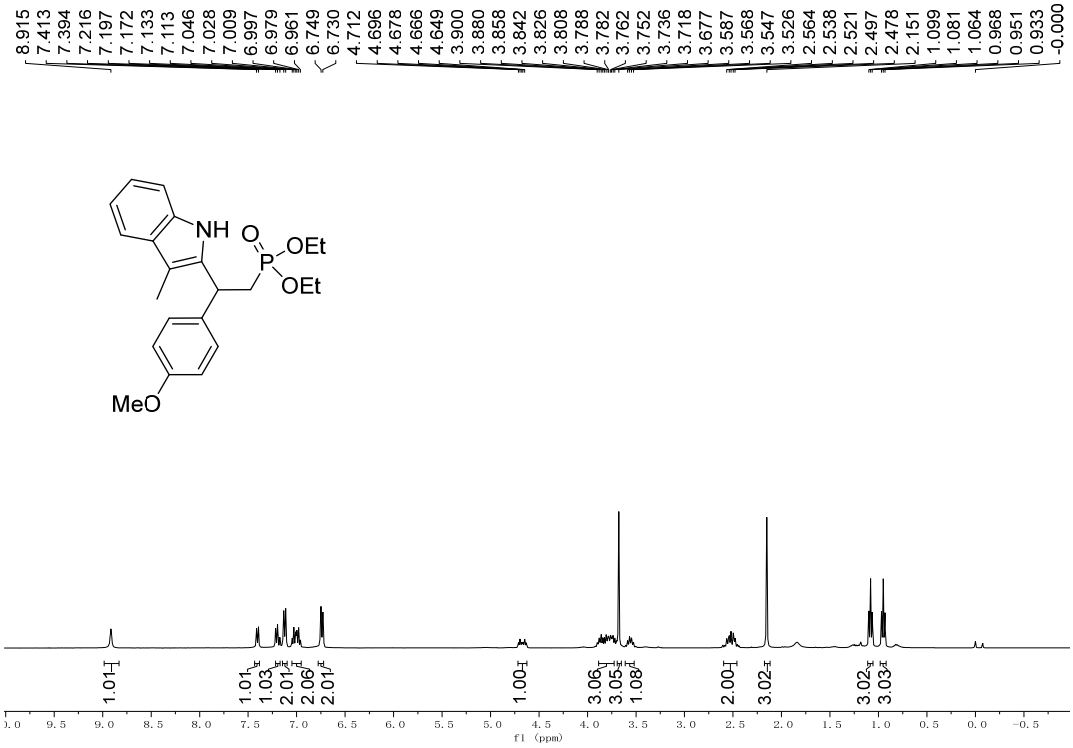



$^{31}P$ NMR ($CDCl_3$, 202 MHz)

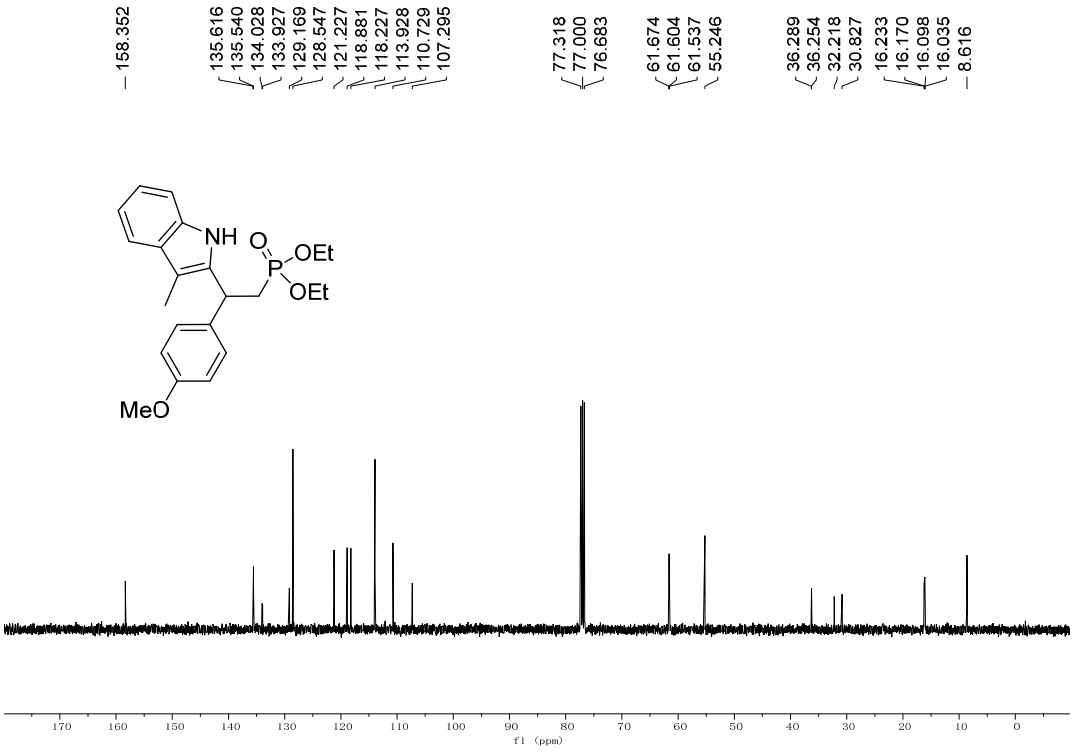


**Diethyl (2-(4-methoxyphenyl)-2-(3-methyl-1*H*-indol-2-yl)ethyl)phosphonate (4aai):**

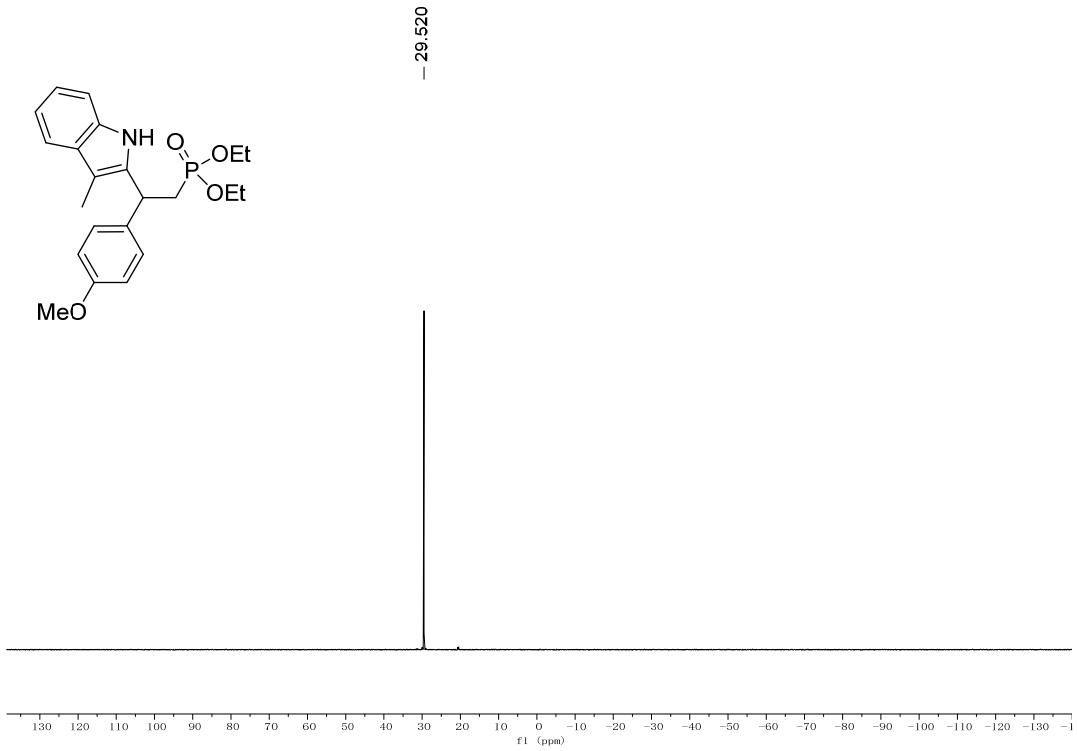
$^{1}$H NMR ($CDCl_3$, 400 MHz)




$^{13}$C NMR ($CDCl_3$, 100 MHz)

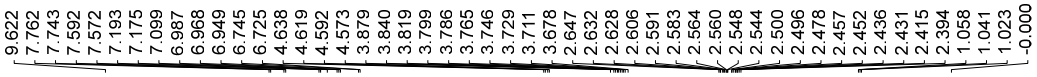


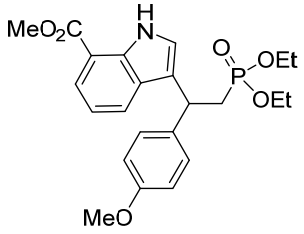
$^{31}P$ NMR ($CDCl_3$, 162 MHz)

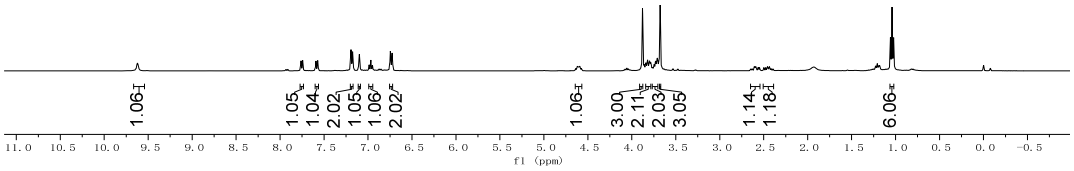


**Methyl 3-(2-(diethoxyphosphoryl)-1-(4-methoxyphenyl)ethyl)-1*H*-indole-7-carboxylate (4aaj):**

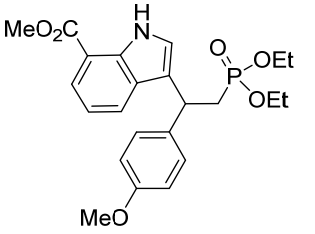
$^{1}$H NMR ($CDCl_3$, 400 MHz)

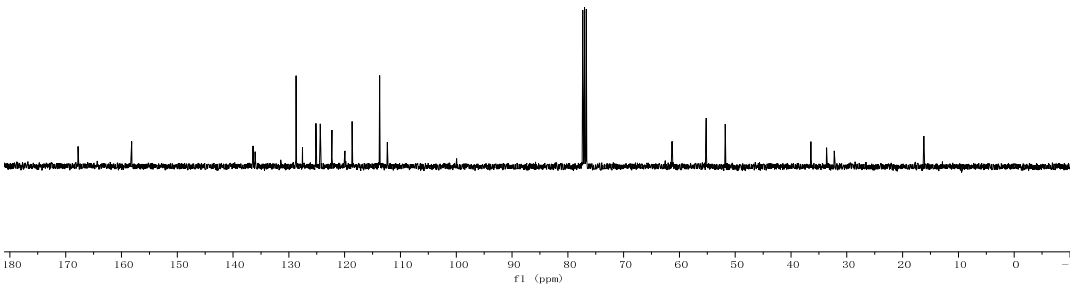


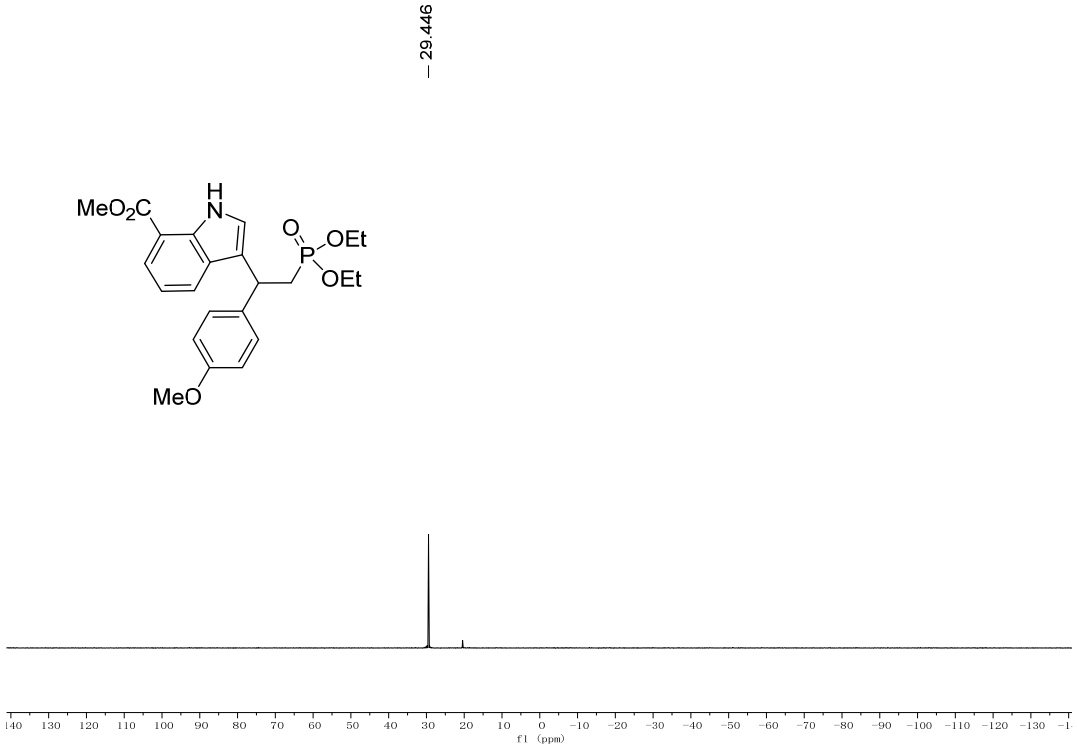




$^{13}$C NMR ($CDCl_3$, 100 MHz)

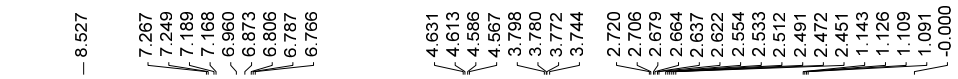



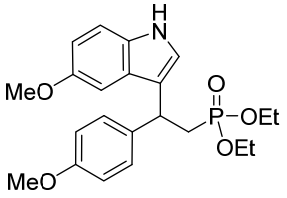
$^{31}P$ NMR ($CDCl_3$, 162 MHz)

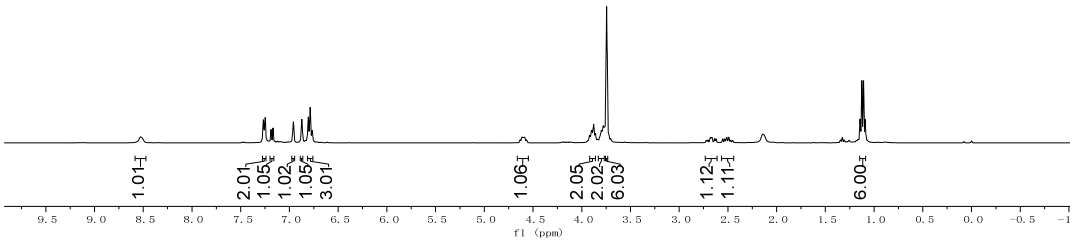


**Diethyl (2-(5-methoxy-1*H*-indol-3-yl)-2-(4-methoxyphenyl)ethyl)phosphonate (4aak):**

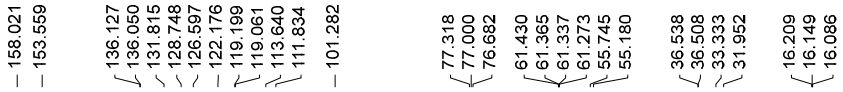
$^{1}$H NMR ($CDCl_3$, 400 MHz)

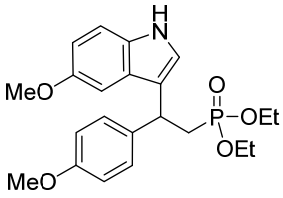


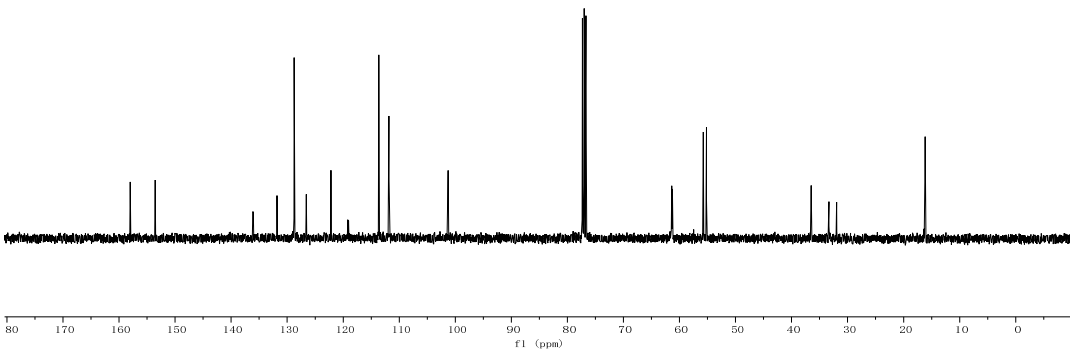



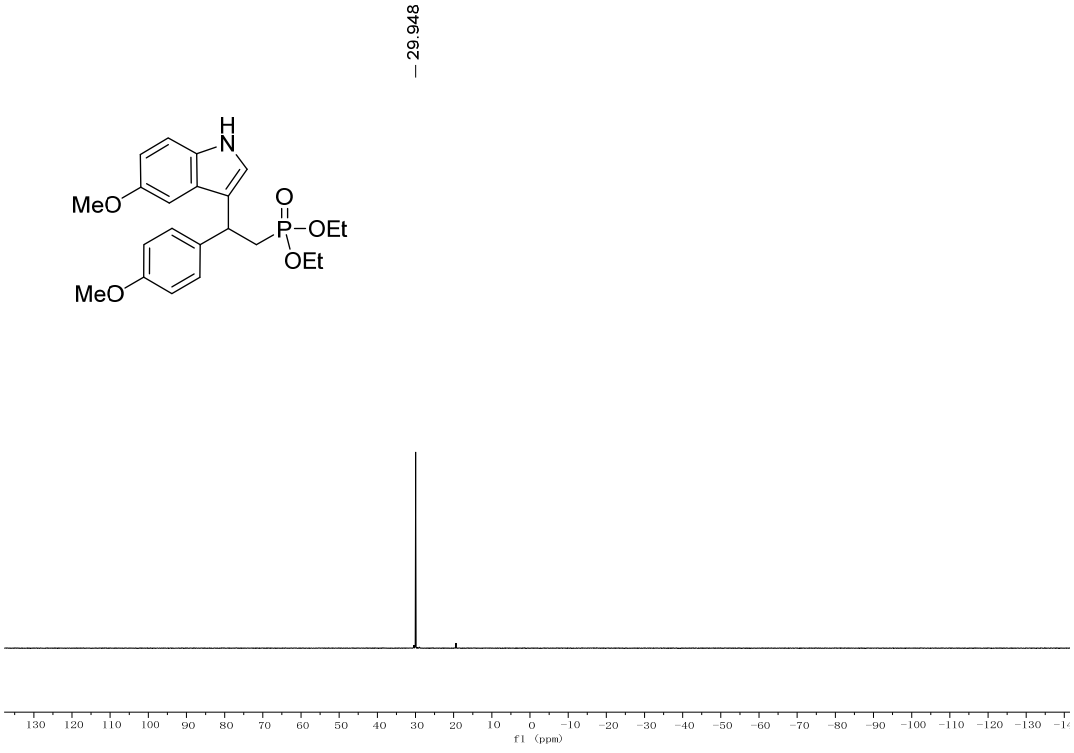

$^{13}$C NMR ($CDCl_3$, 100 MHz)



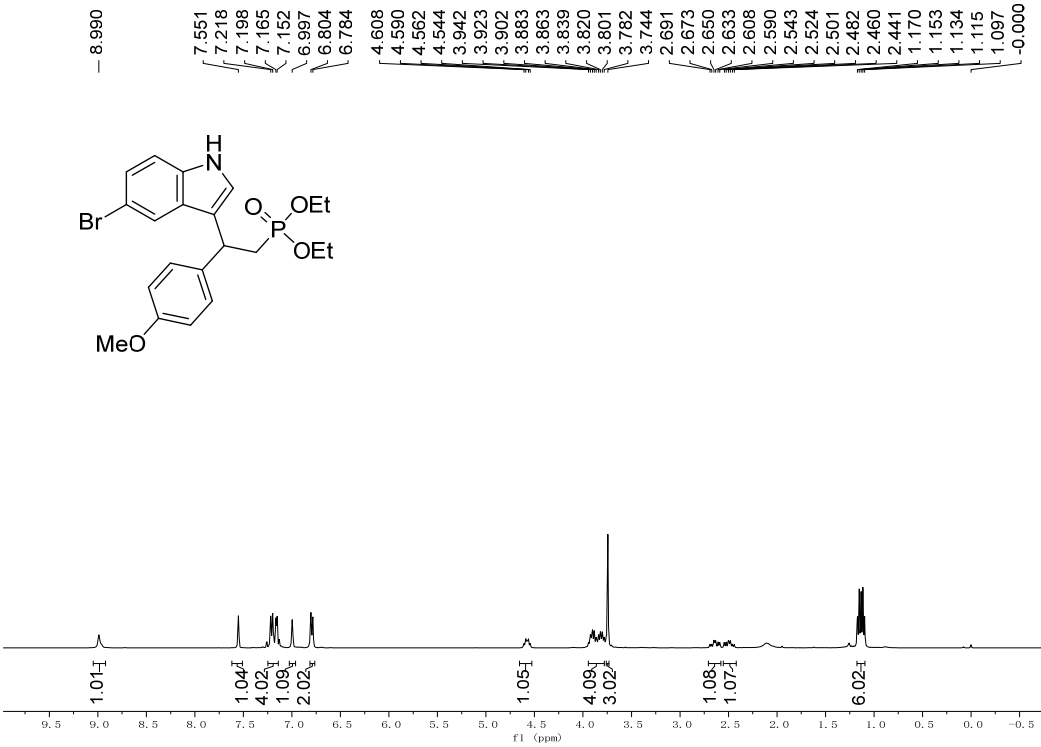



$^{31}P$ NMR ($CDCl_3$, 162 MHz)

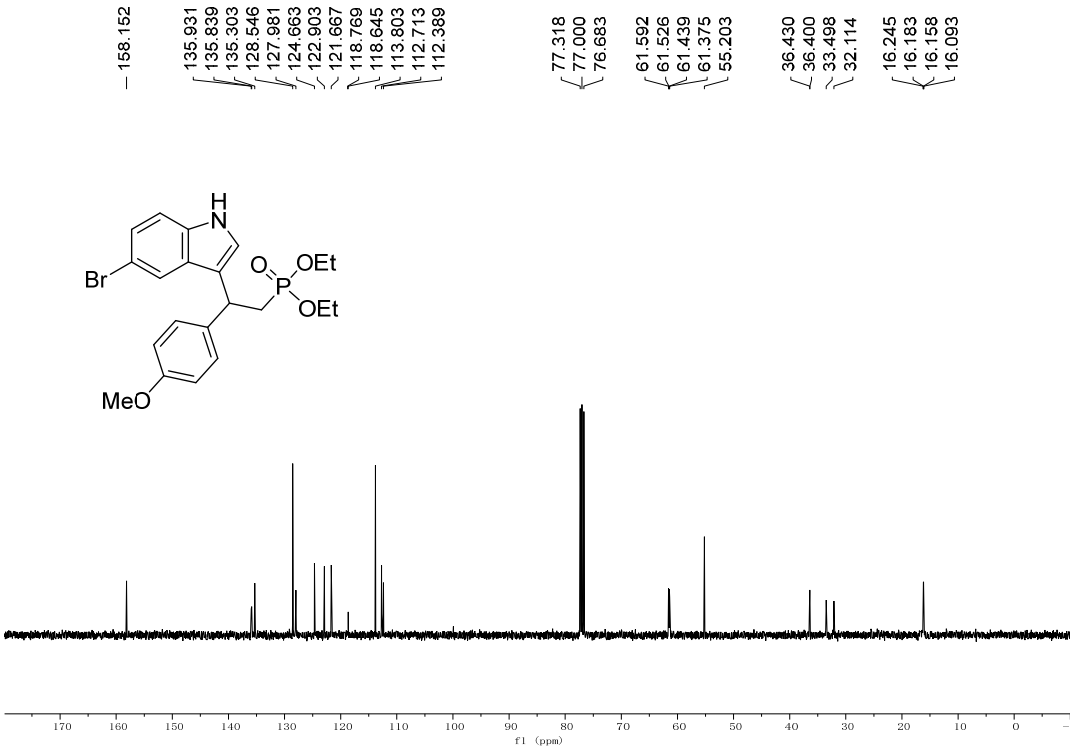


**Diethyl (2-(5-bromo-1*H*-indol-3-yl)-2-(4-methoxyphenyl)ethyl)phosphonate (4aal):**

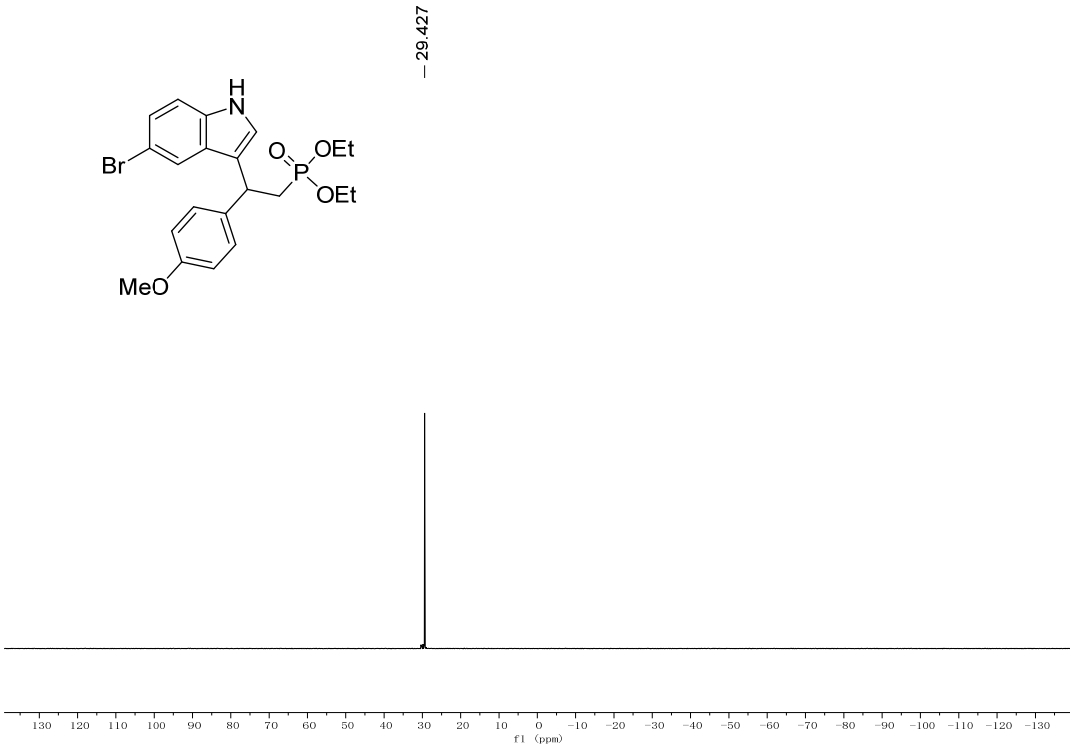
$^{1}$H NMR ($CDCl_3$, 400 MHz)




$^{13}$C NMR ($CDCl_3$, 100 MHz)

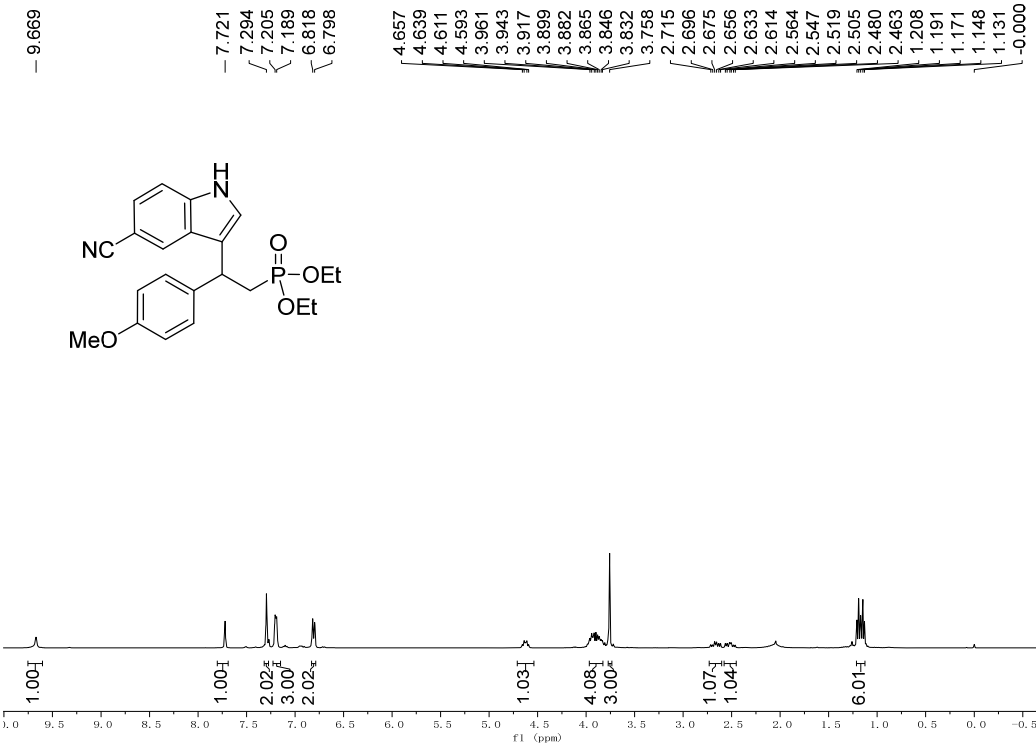


$^{31}P$ NMR ($CDCl_3$, 162 MHz)

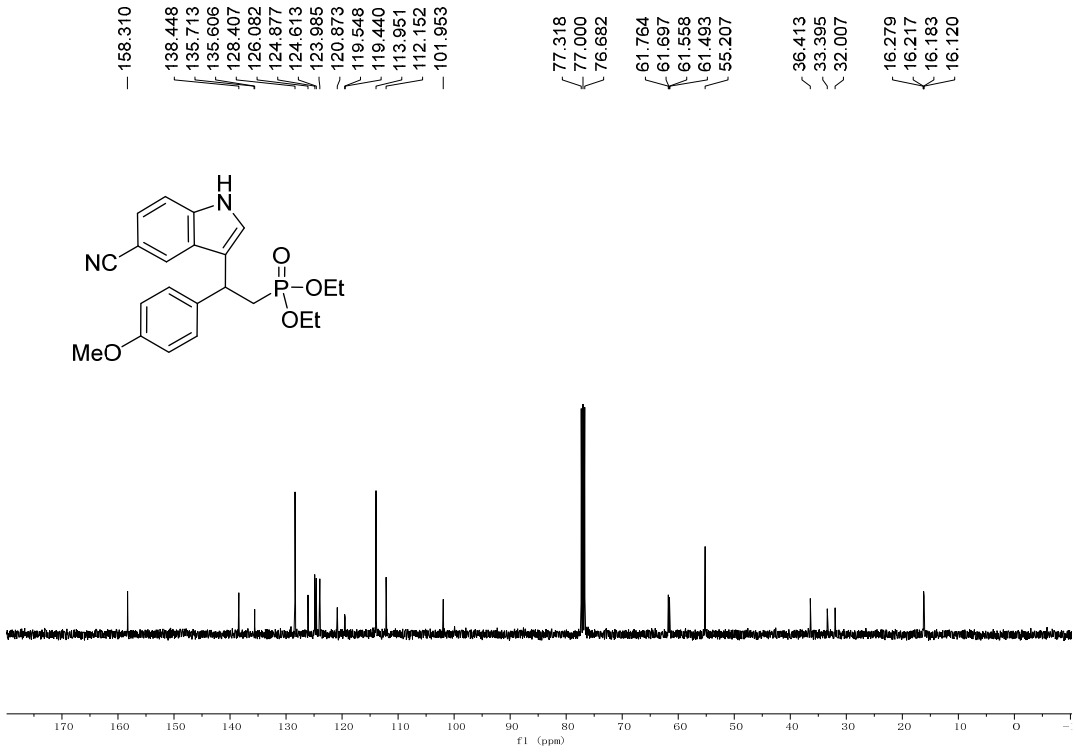


**Diethyl (2-(5-cyano-1*H*-indol-3-yl)-2-(4-methoxyphenyl)ethyl)phosphonate (4aam):**

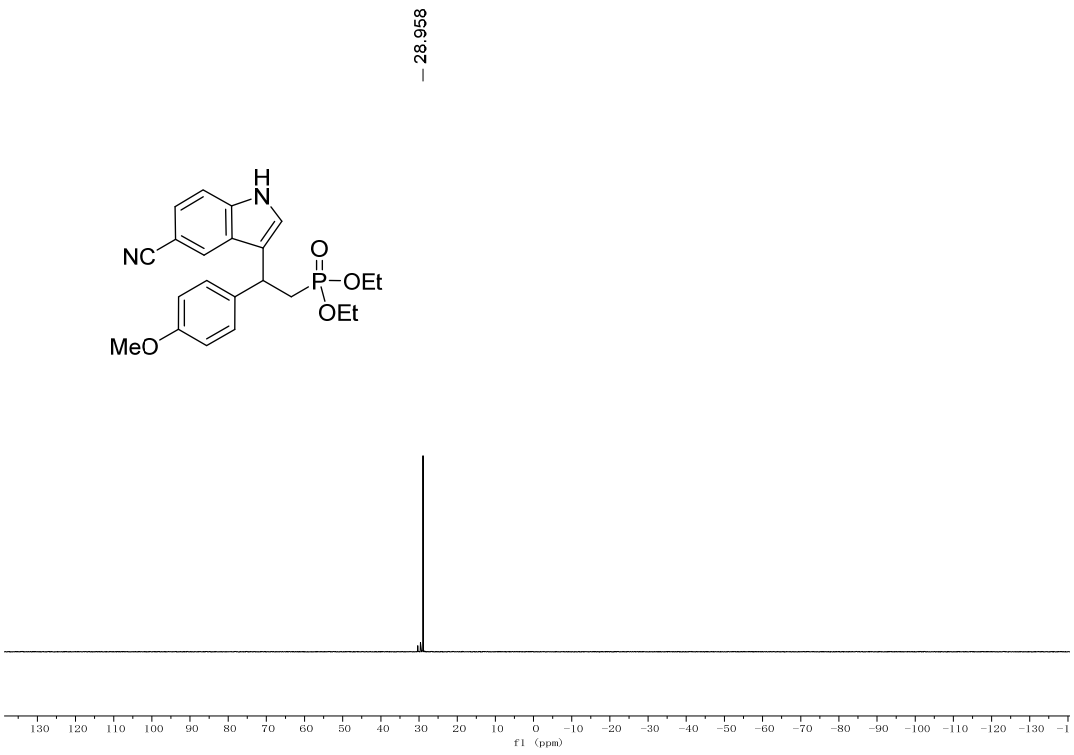
$^{1}$H NMR ($CDCl_3$, 400 MHz)




$^{13}$C NMR ($CDCl_3$, 100 MHz)

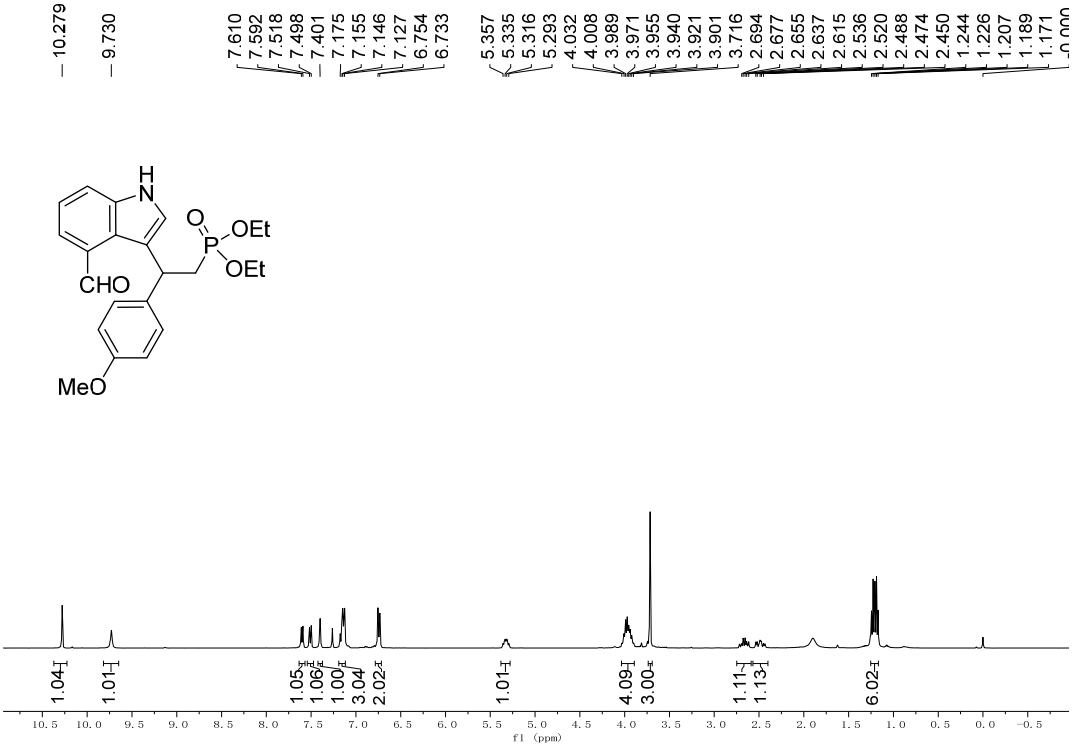


$^{31}P$ NMR ($CDCl_3$, 162 MHz)

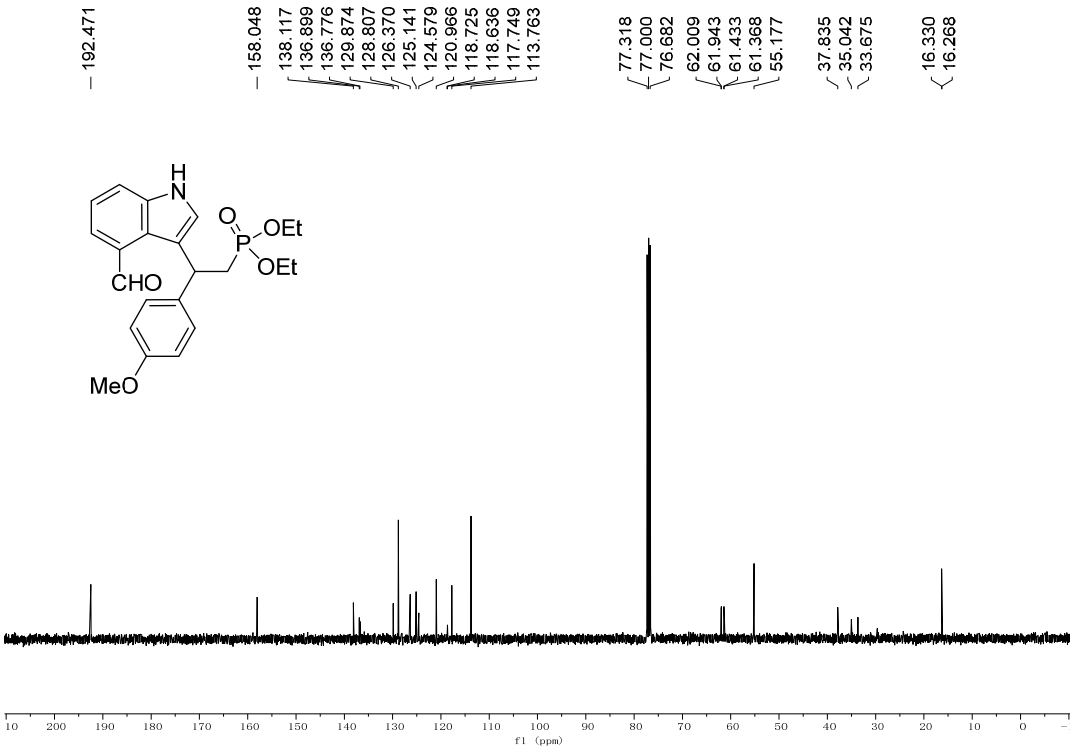


**Diethyl (2-(4-formyl-1*H*-indol-3-yl)-2-(4-methoxyphenyl)ethyl)phosphonate (4aan):**

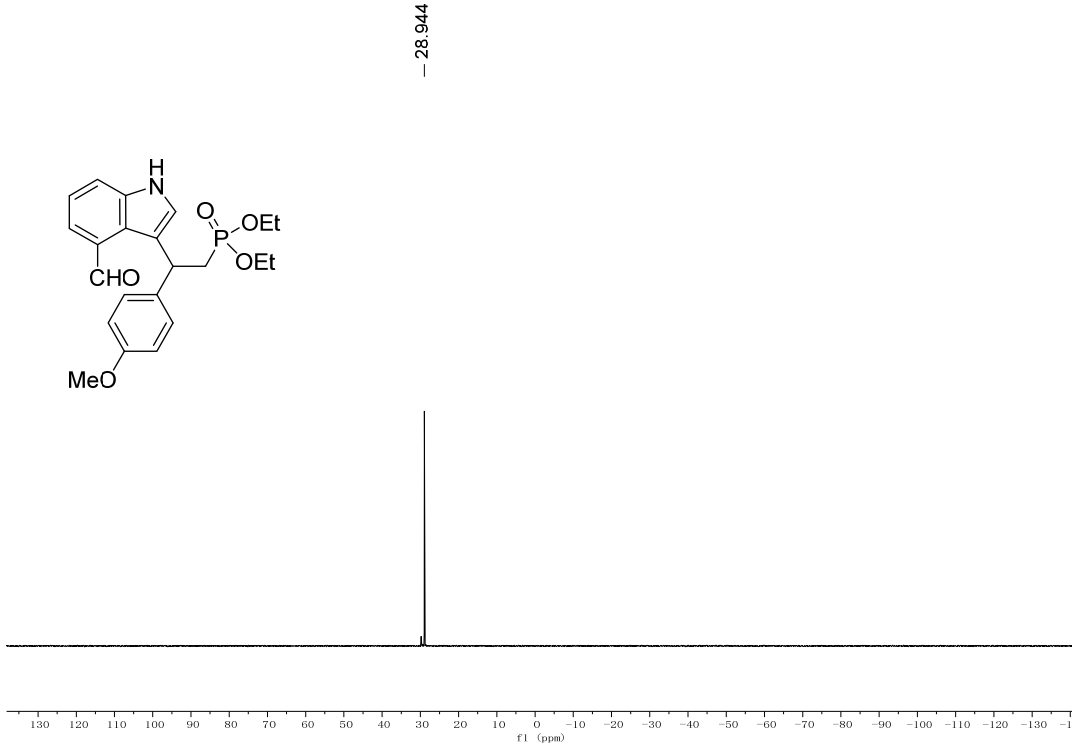
$^{1}$H NMR ($CDCl_3$, 400 MHz)




$^{13}$C NMR ($CDCl_3$, 100 MHz)

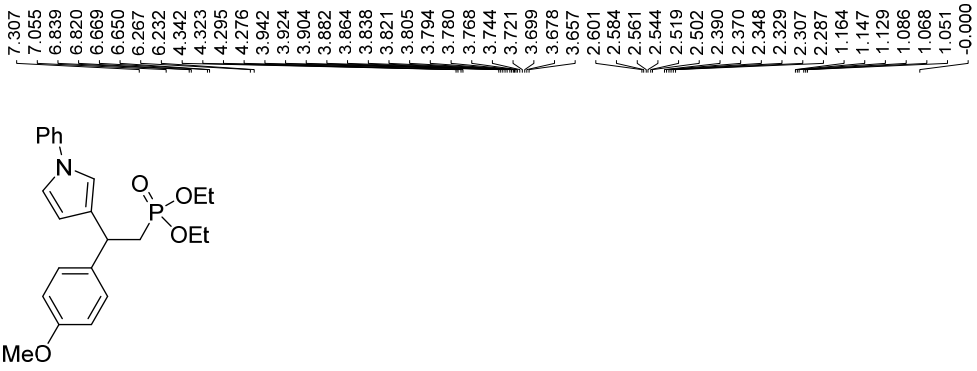


$^{31}P$ NMR ($CDCl_3$, 162 MHz)



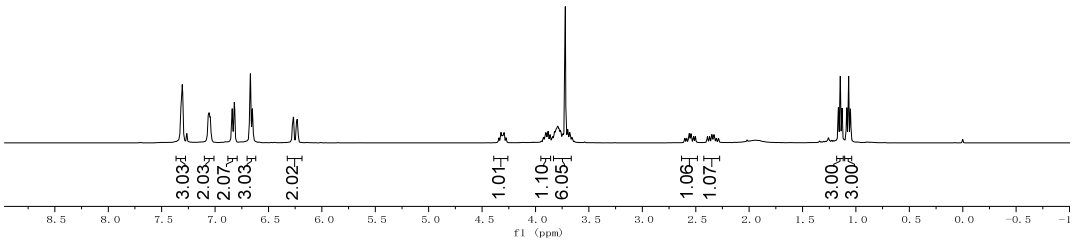
**Diethyl (2-(4-methoxyphenyl)-2-(1-phenyl-1*H*-pyrrol-2-yl)ethyl)phosphonate (4aao):**

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

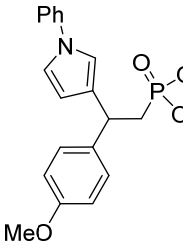


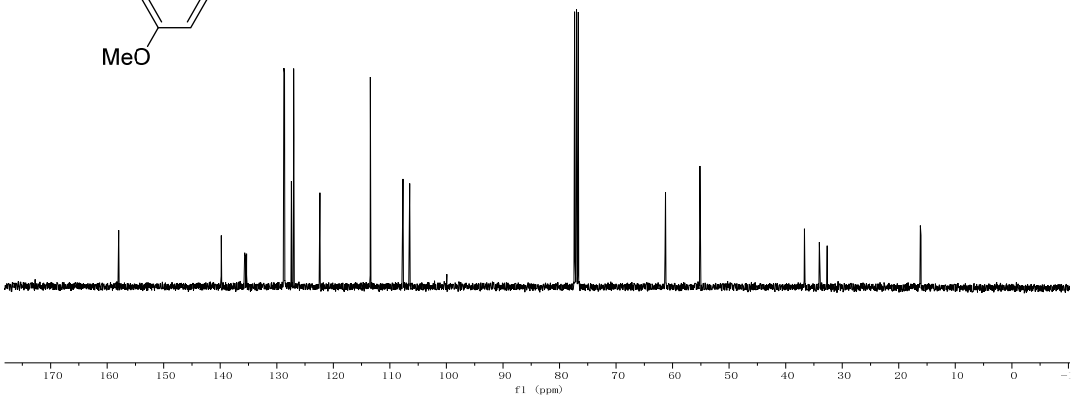




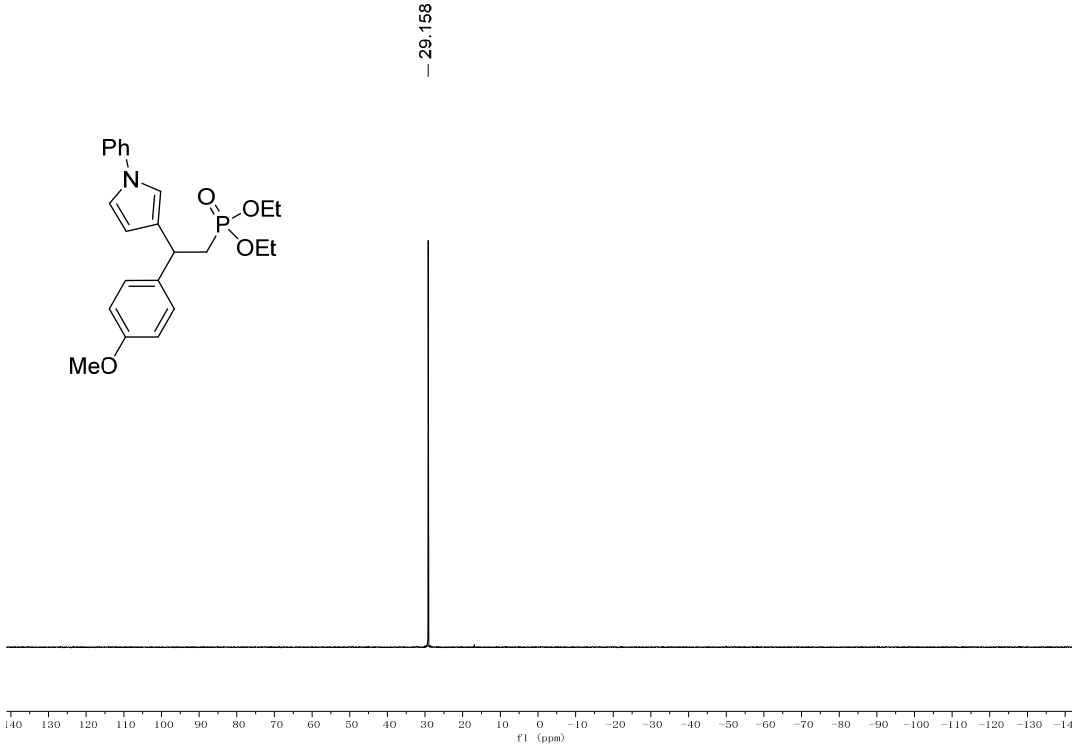



$^{13}$C NMR ($CDCl_3$, 100 MHz)

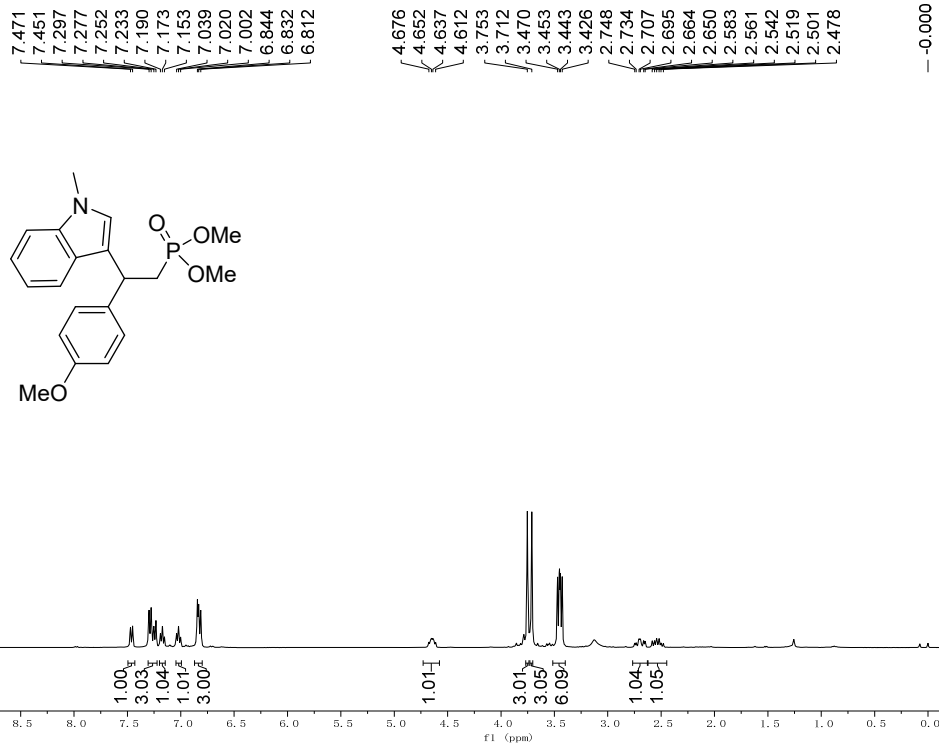



$^{31}P$ NMR ($CDCl_3$, 162 MHz)

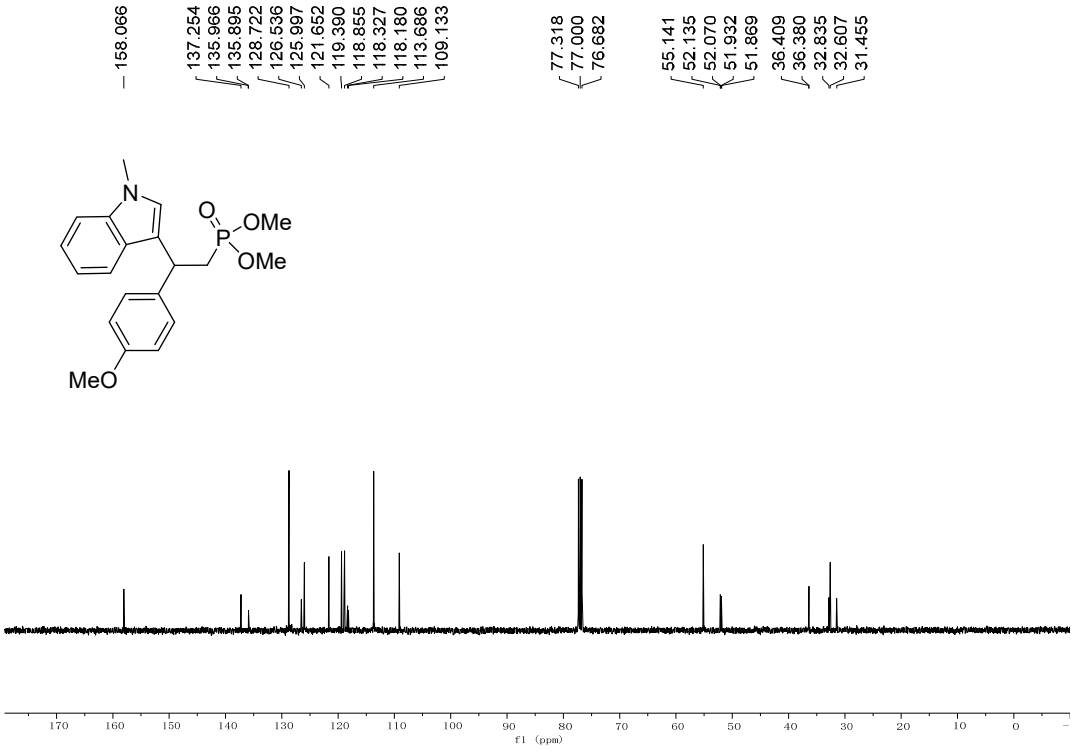


**Dimethyl (2-(4-methoxyphenyl)-2-(1-methyl-1*H*-indol-3-yl)ethyl)phosphonate (4aba):**

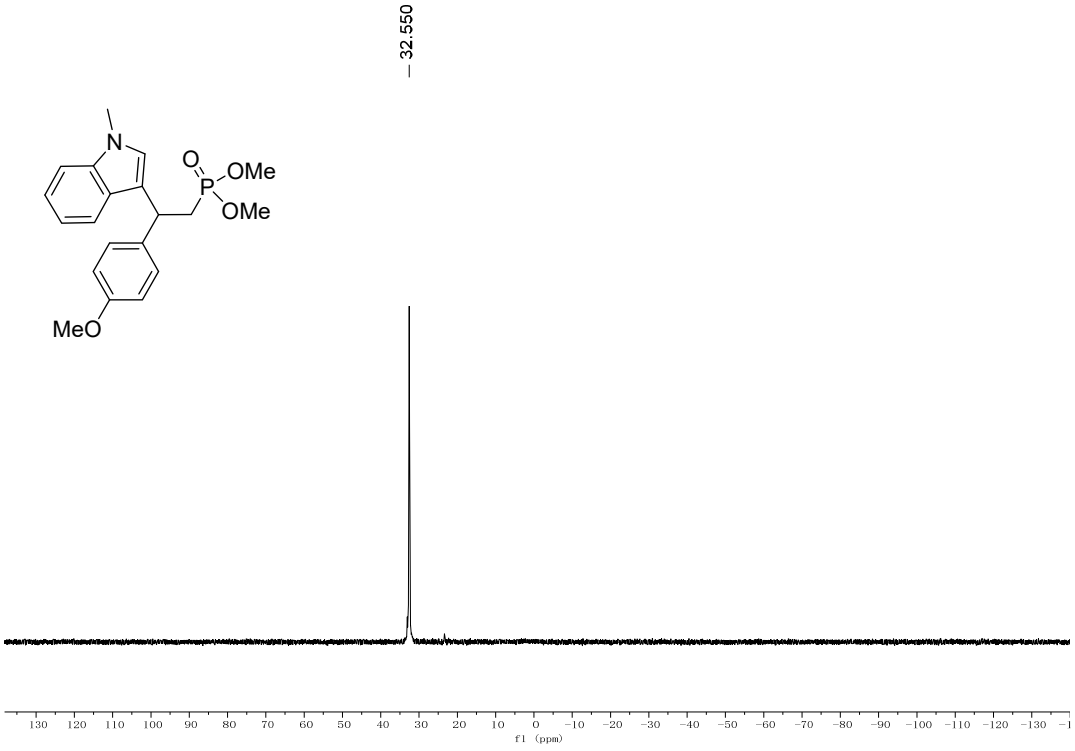
$^{1}$H NMR ($CDCl_3$, 400 MHz)




$^{13}$C NMR ($CDCl_3$, 100 MHz)

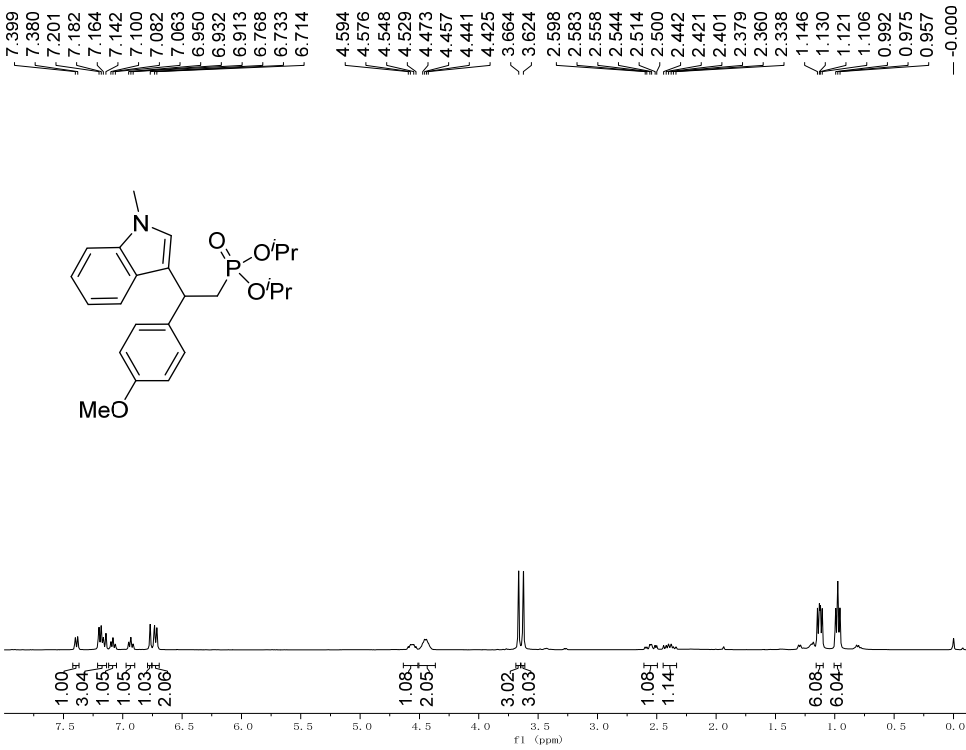


$^{31}P$ NMR ($CDCl_3$, 162 MHz)

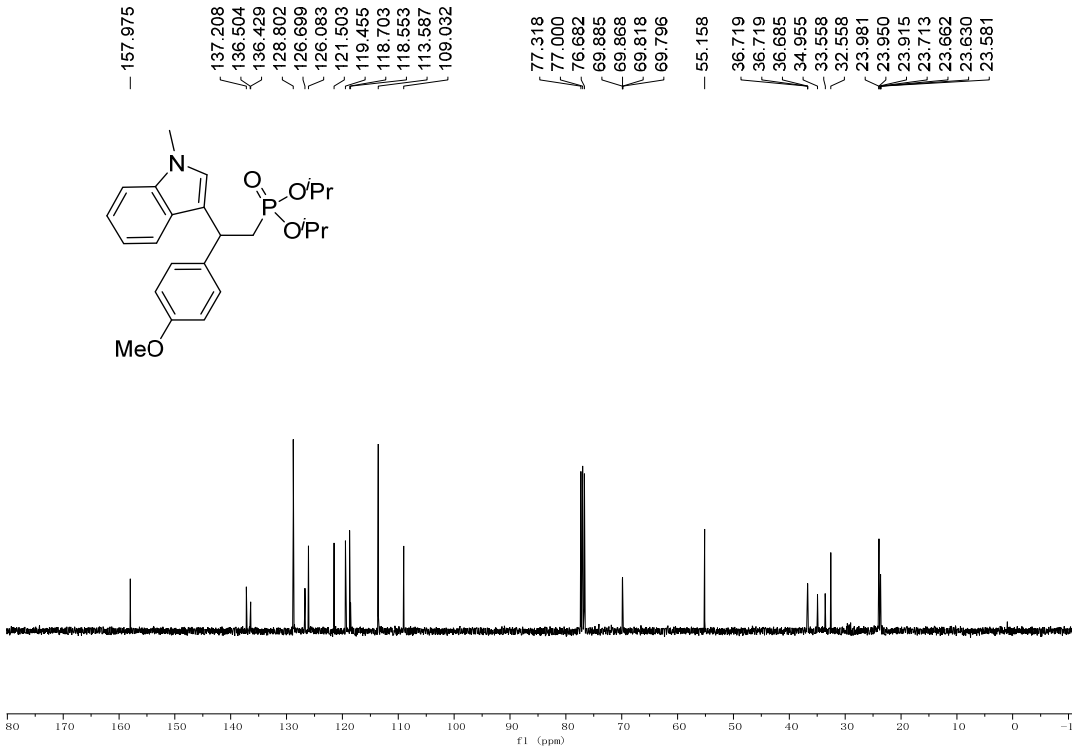


**Diisopropyl (2-(4-methoxyphenyl)-2-(1-methyl-1*H*-indol-3-yl)ethyl)phosphonate (4aca):**

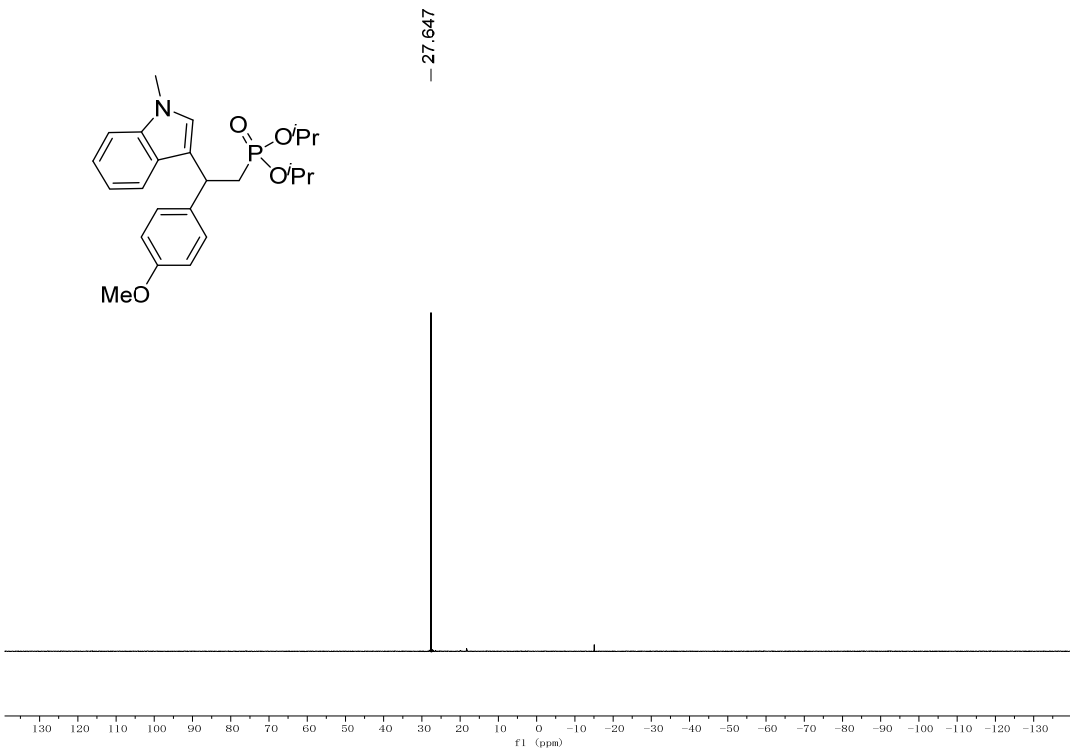
$^{1}$H NMR ($CDCl_3$, 400 MHz)




$^{13}$C NMR ($CDCl_3$, 100 MHz)

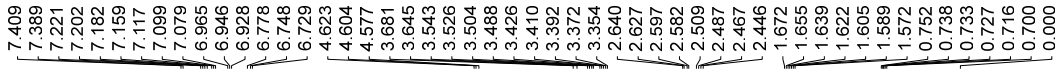


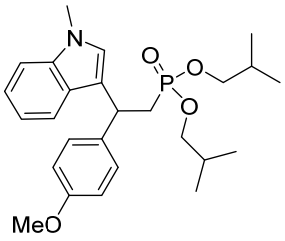
$^{31}P$ NMR ($CDCl_3$, 162 MHz)

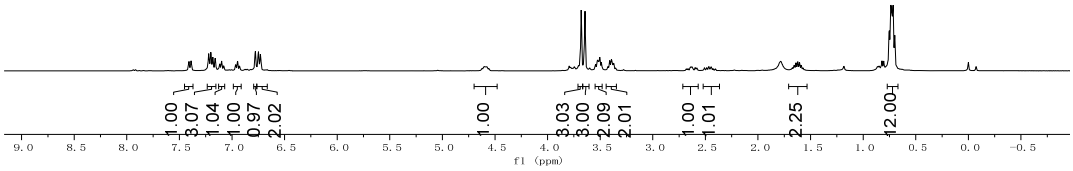


**Diisobutyl (2-(4-methoxyphenyl)-2-(1-methyl-1*H*-indol-3-yl)ethyl)phosphonate (4ada):**

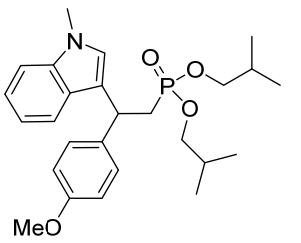
$^{1}$H NMR ($CDCl_3$, 400 MHz)

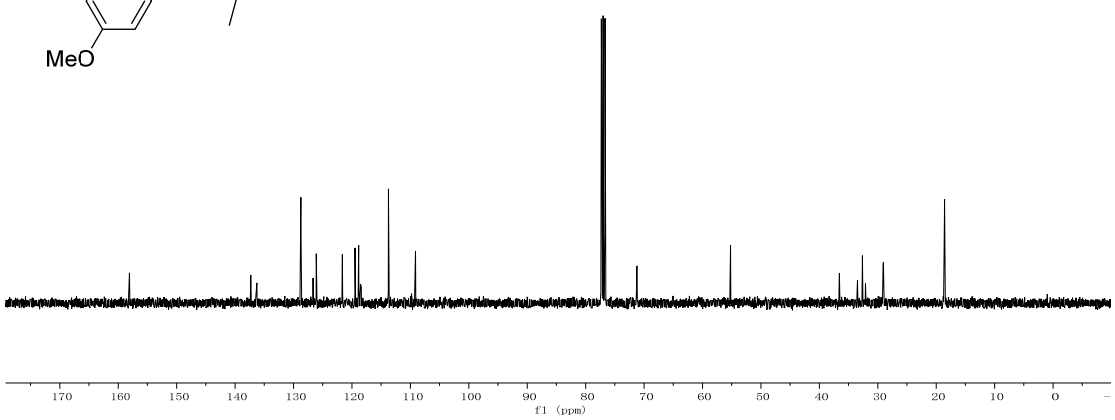


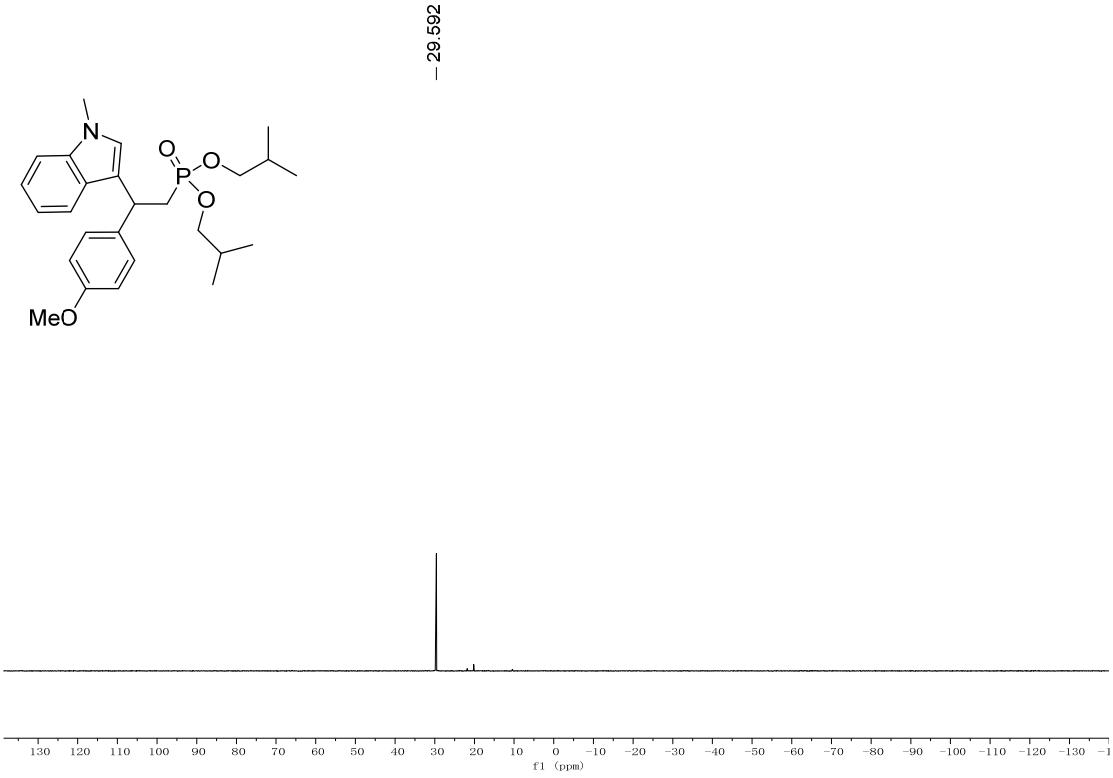




$^{13}$C NMR ($CDCl_3$, 100 MHz)

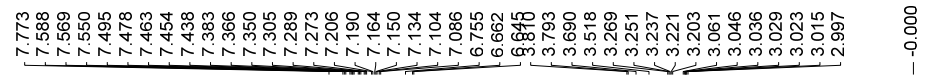



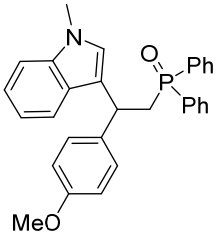
$^{31}$P NMR ($CDCl_3$, 162 MHz)

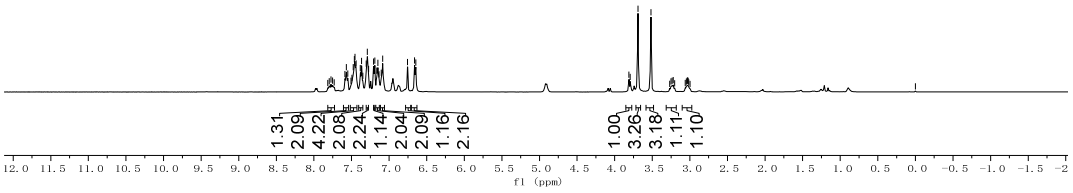


**(2-(4-methoxyphenyl)-2-(1-methyl-1*H*-indol-3-yl)ethyl)diphenylphosphine oxide (4aea):**

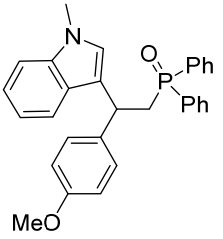
$^{1}$H NMR ($CDCl_3$, 400 MHz)

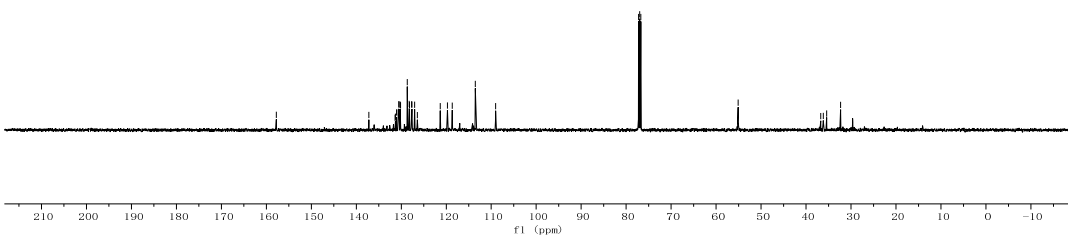


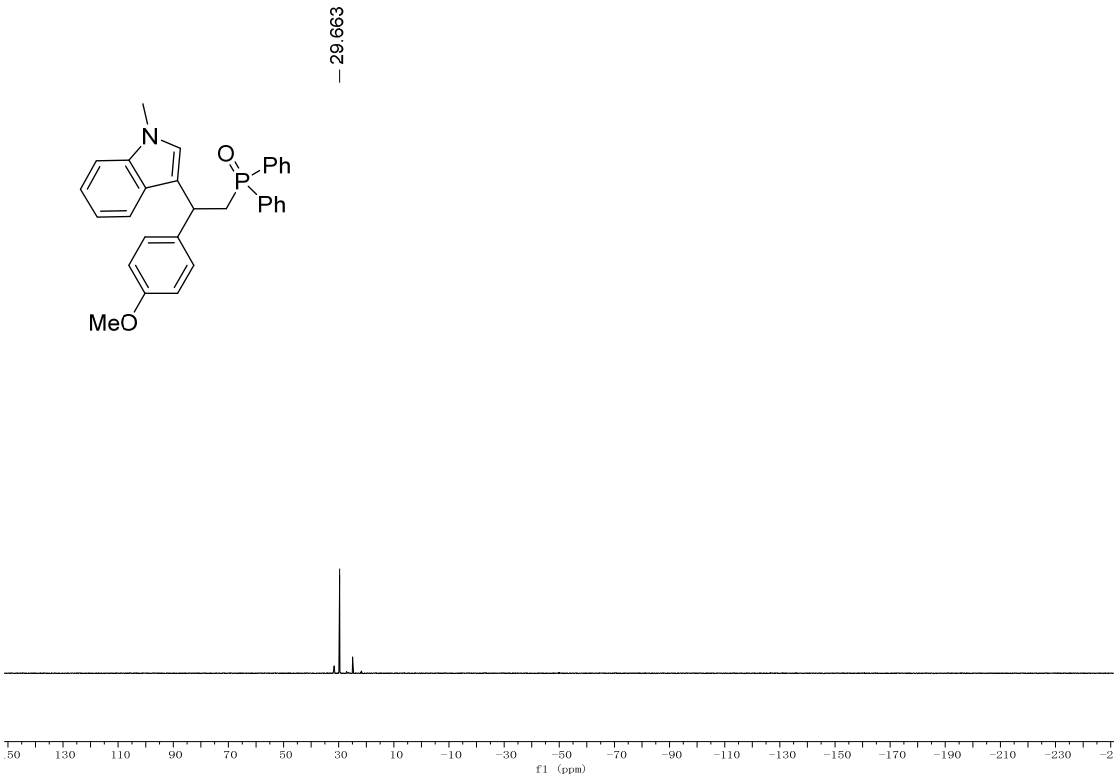




$^{13}$C NMR ($CDCl_3$, 100 MHz)

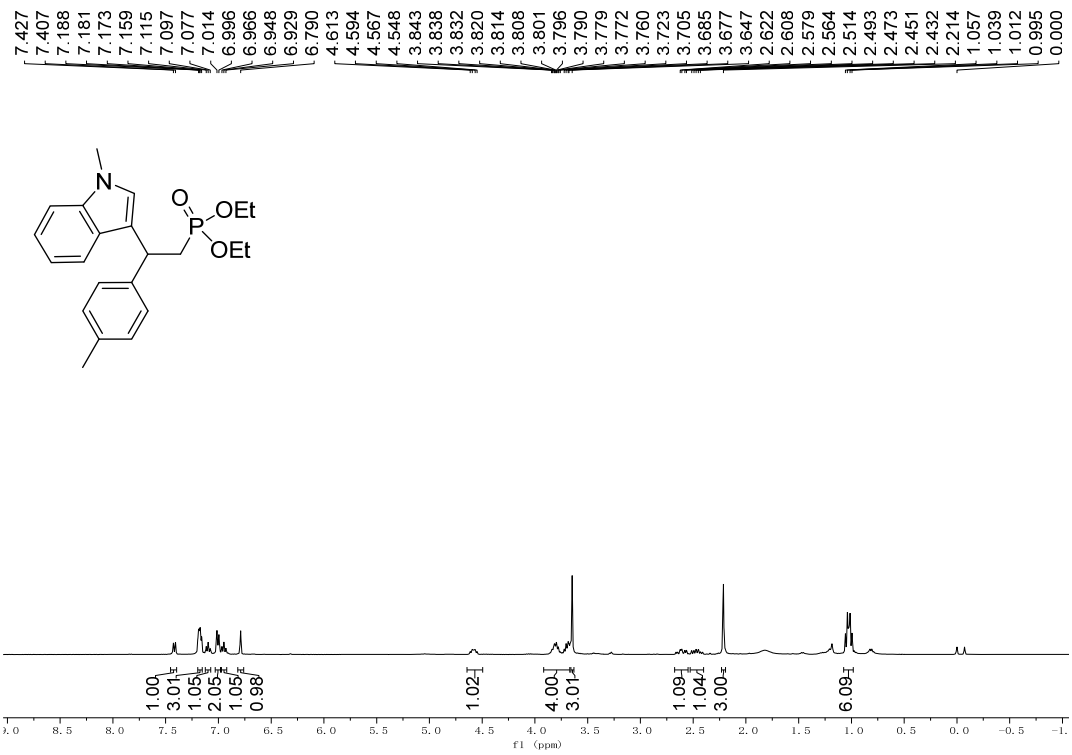



$^{31}$P NMR ($CDCl_3$, 162 MHz)

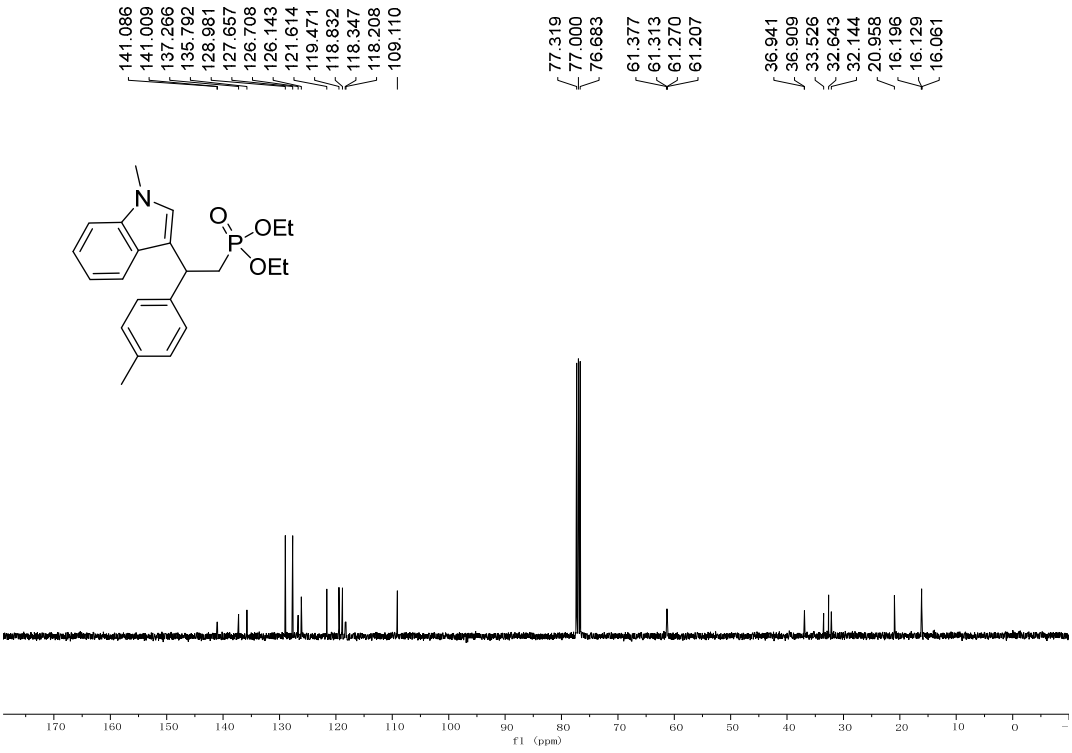


**Diethyl (2-(1-methyl-1*H*-indol-3-yl)-2-(p-tolyl)ethyl)phosphonatete (4baa):**

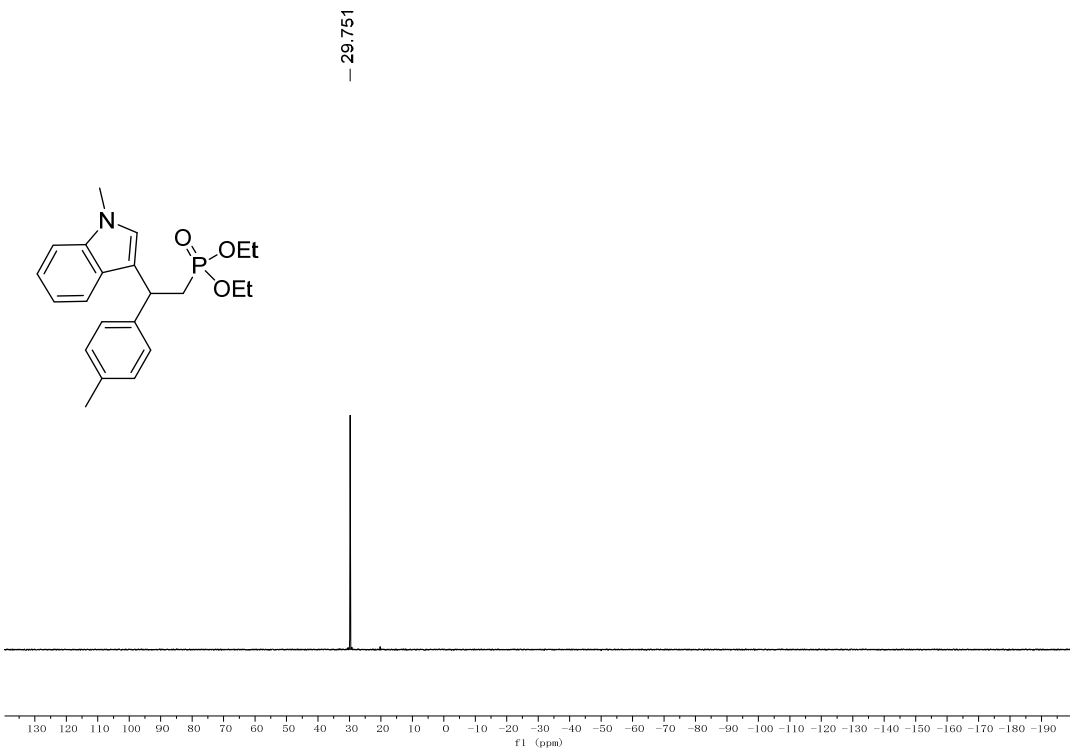
$^{1}$H NMR ($CDCl_3$, 400 MHz)




$^{13}$C NMR ($CDCl_3$, 100 MHz)

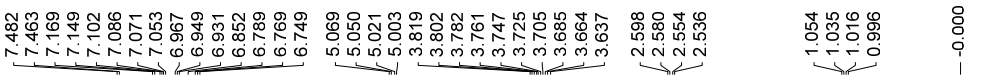


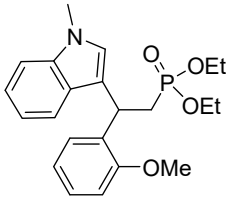
$^{31}$P NMR ($CDCl_3$, 162 MHz)

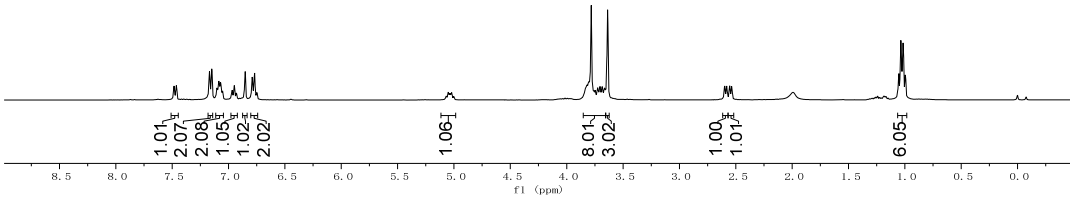


**Diethyl (2-(2-methoxyphenyl)-2-(1-methyl-1*H*-indol-3-yl)ethyl)phosphonate (4caa):**

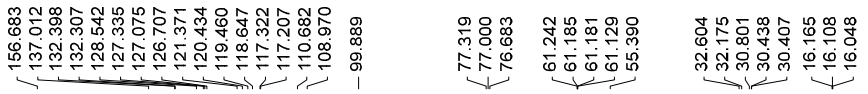
$^{1}$H NMR ($CDCl_3$, 400 MHz)

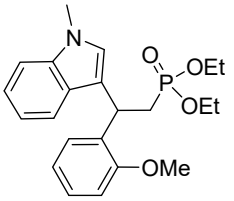


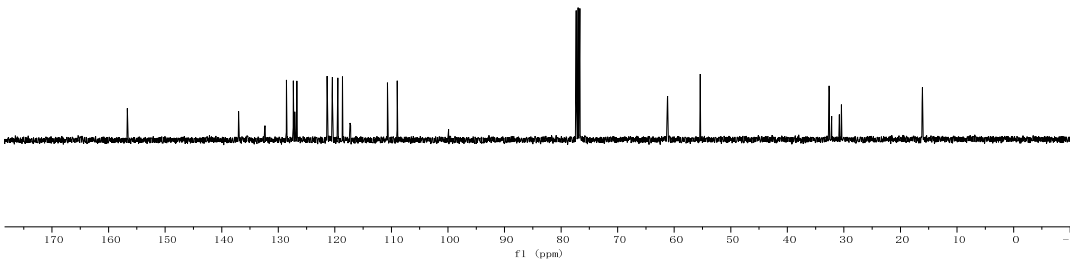



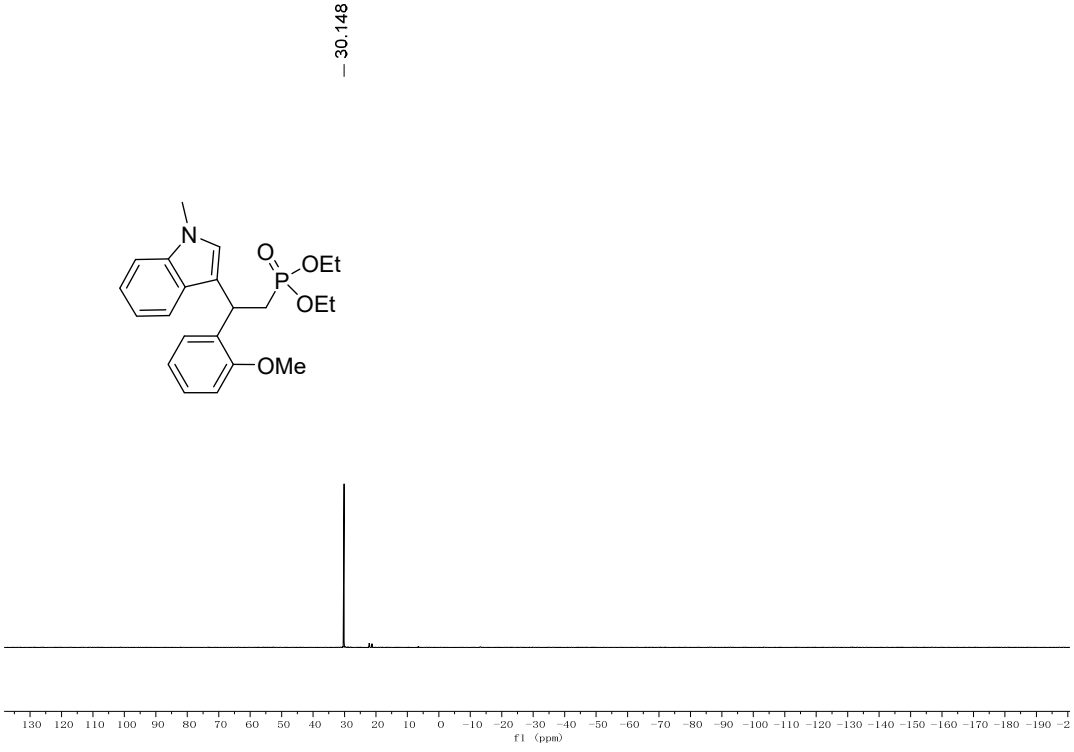

$^{13}$C NMR ($CDCl_3$, 100 MHz)



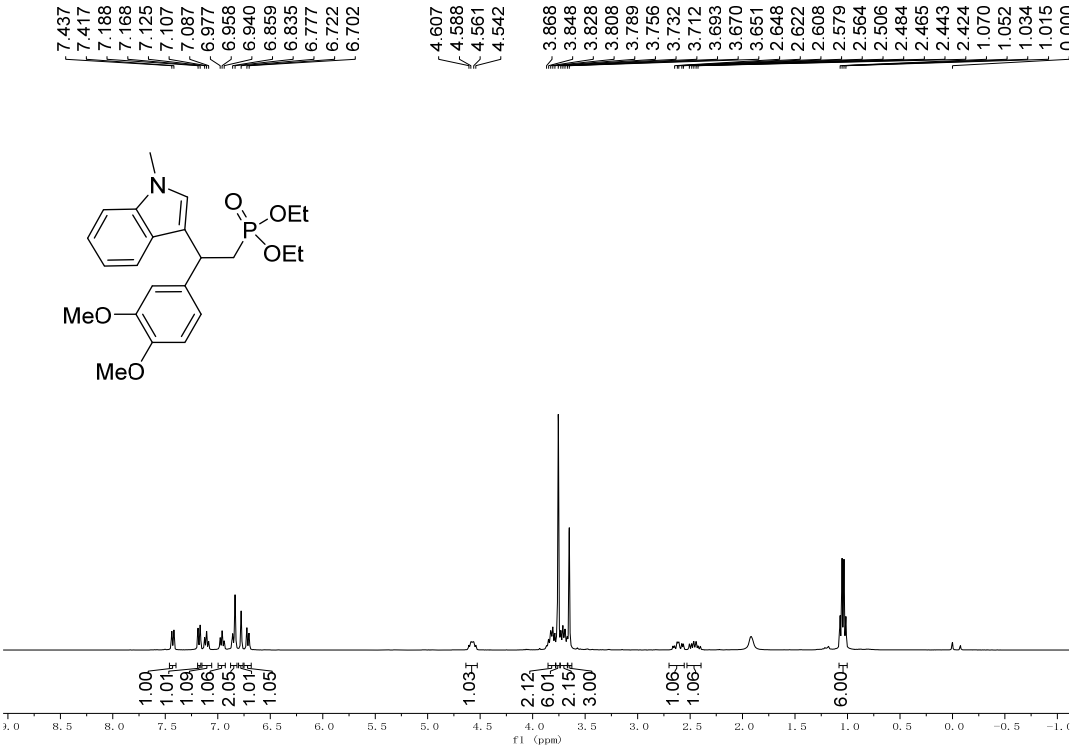



$^{31}$P NMR ($CDCl_3$, 162 MHz)

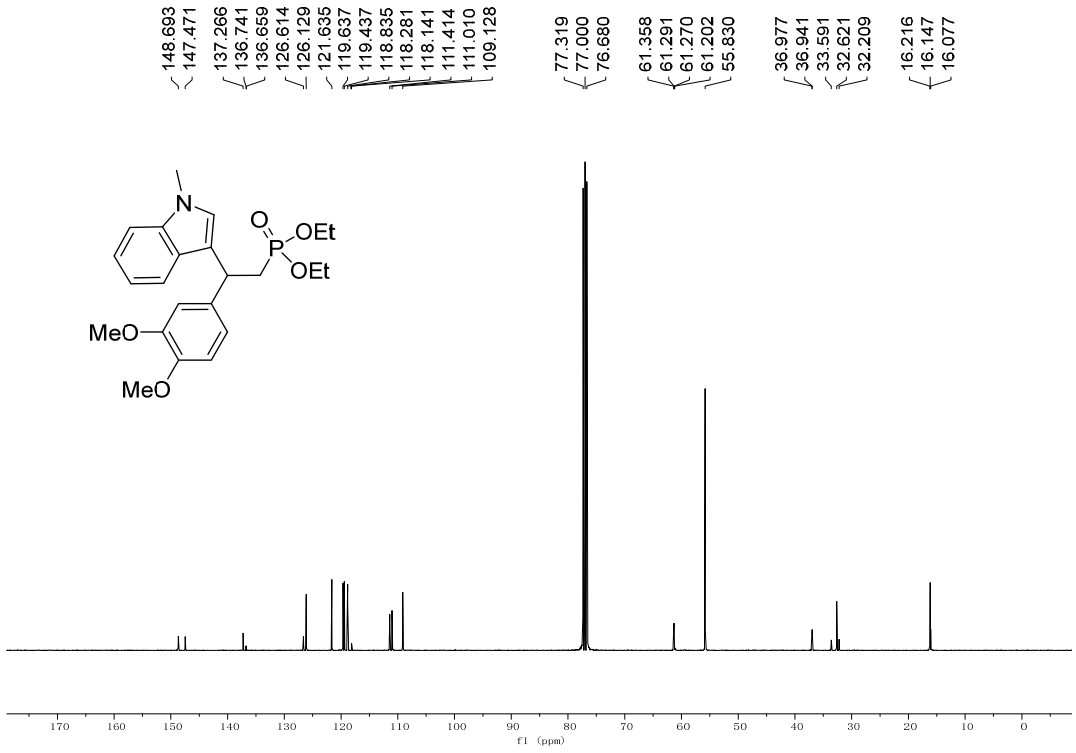


**Diethyl (2-(3,4-dimethoxyphenyl)-2-(1-methyl-1*H*-indol-3-yl)ethyl)phosphonate (4daa):**

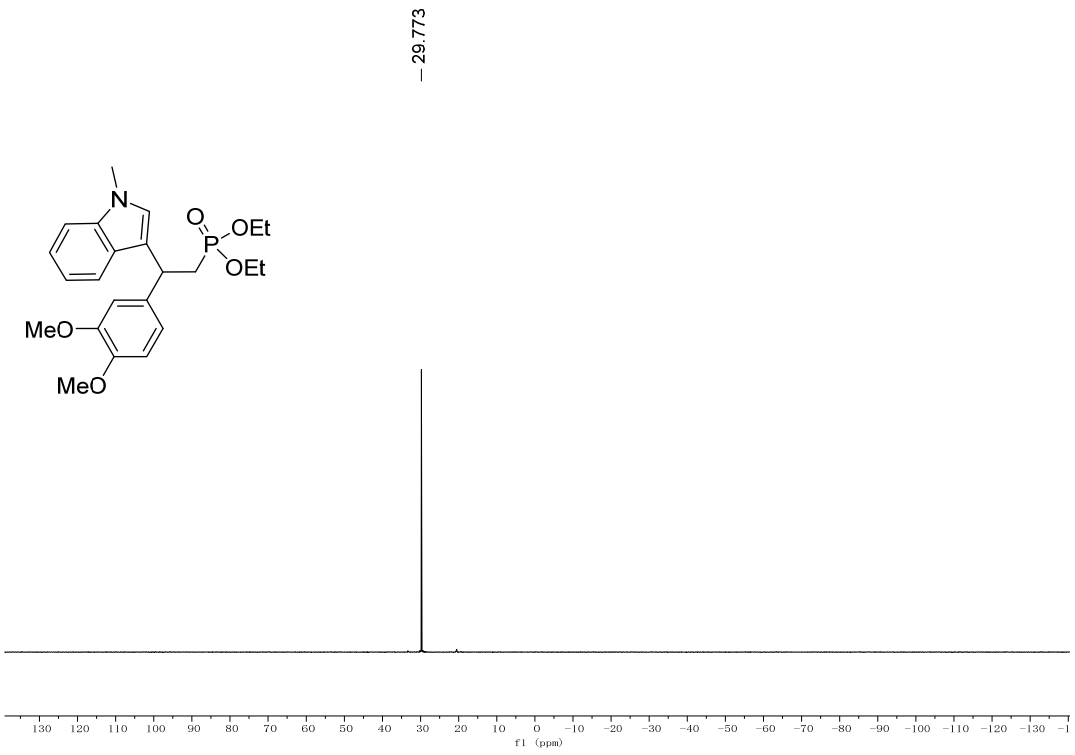
$^{1}$H NMR ($CDCl_3$, 400 MHz)




$^{13}$C NMR ($CDCl_3$, 100 MHz)

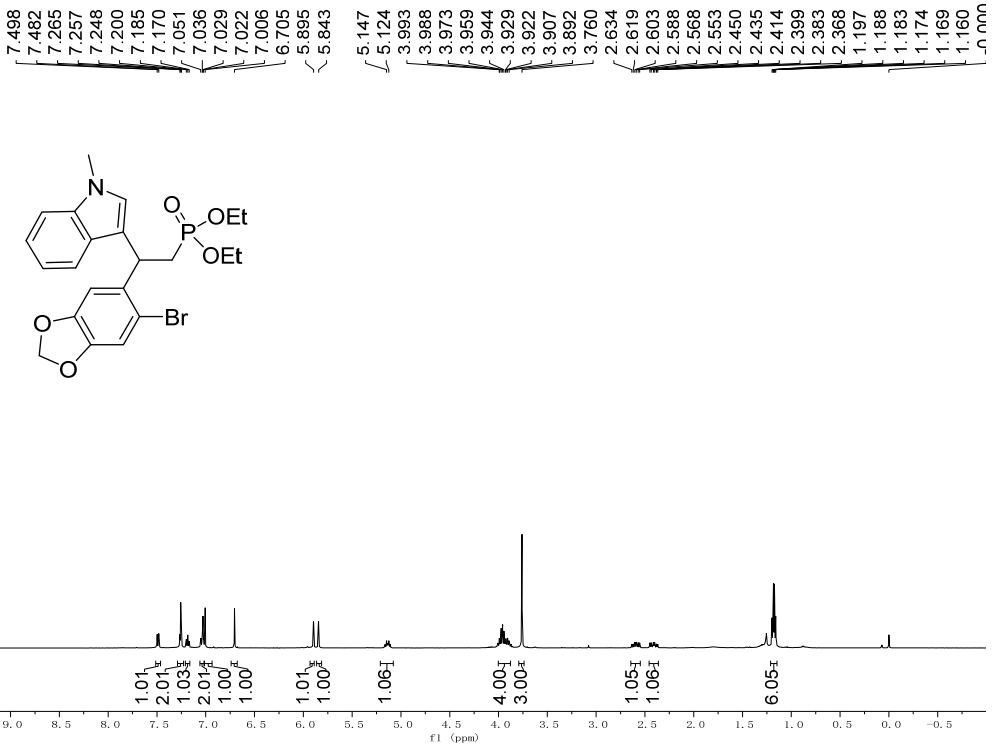


$^{31}$P NMR ($CDCl_3$, 162 MHz)

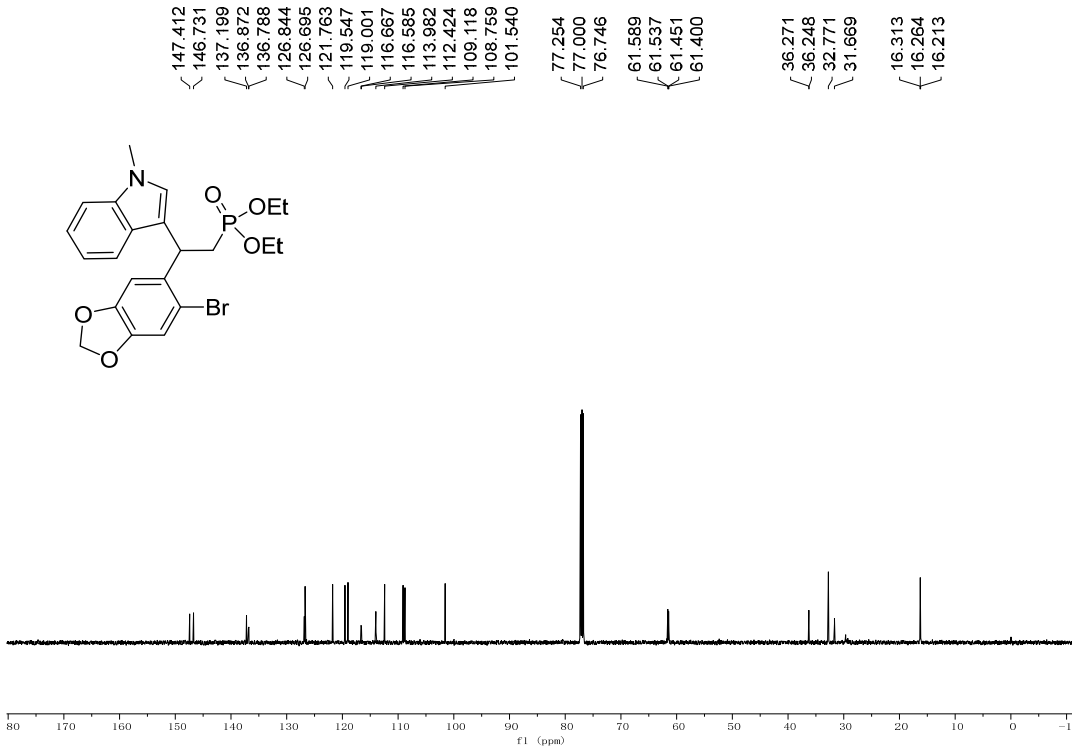


**Diethyl (2-(6-bromobenzo[*d*][1,3]dioxol-5-yl)-2-(1-methyl-1*H*-indol-3-yl)ethyl) phosphonate (4eaa):**

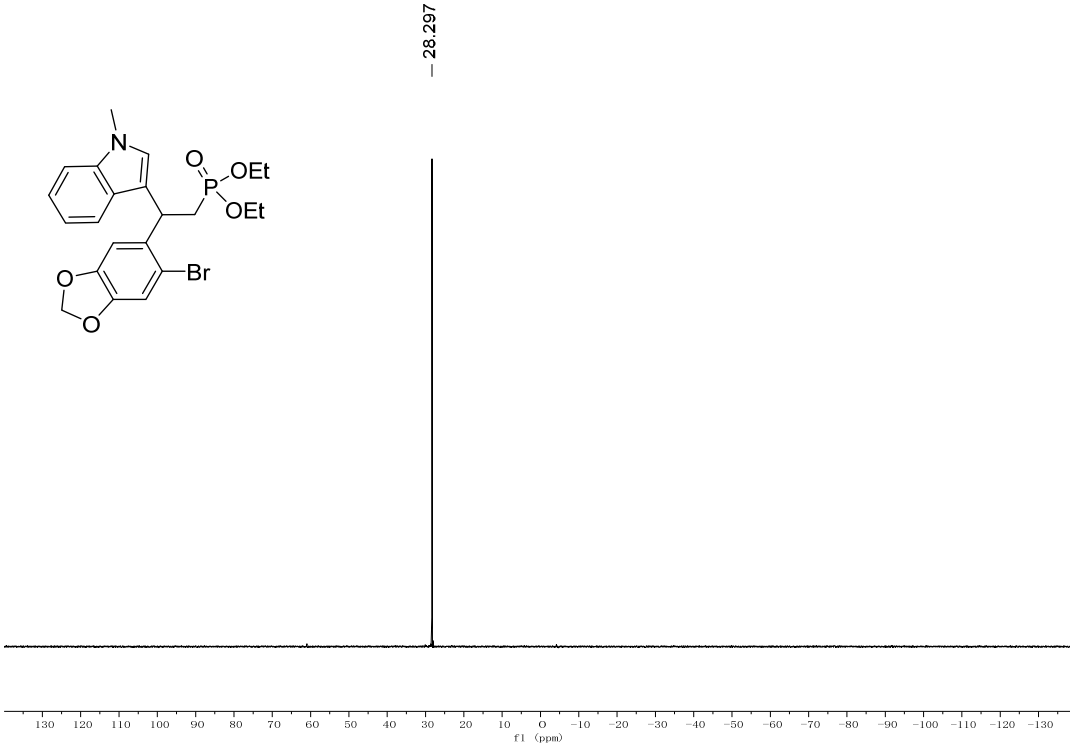
$^{1}$H NMR ($CDCl_3$, 500 MHz)




$^{13}$C NMR ($CDCl_3$, 125 MHz)

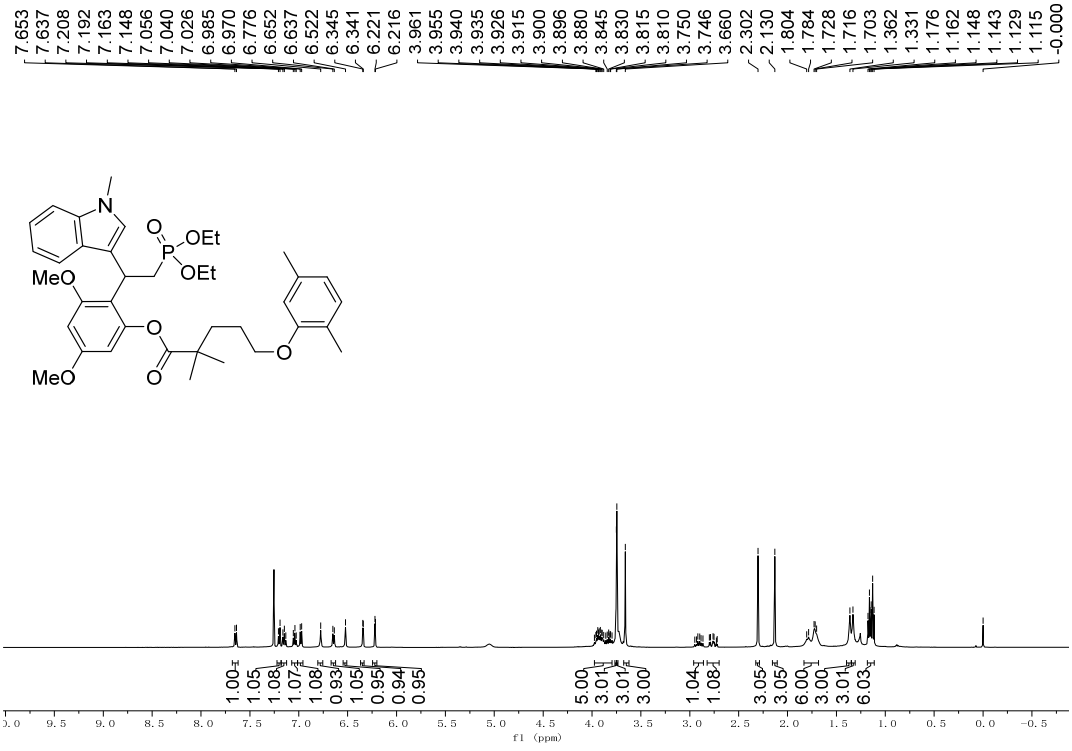


$^{31}P$ NMR ($CDCl_3$, 202 MHz)

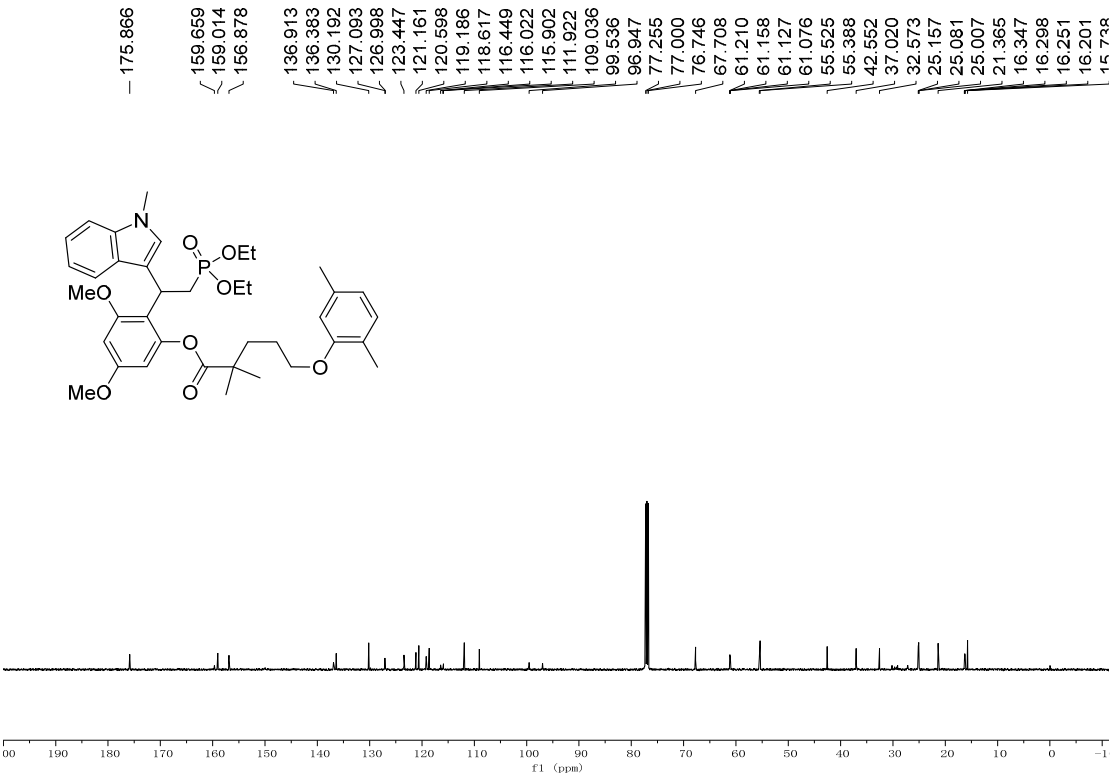


**2-(2-(diethoxyphosphoryl)-1-(1-methyl-1*H*-indol-3-yl)ethyl)-3,5-dimethoxyphenyl 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate (4faa) (Gemfibrozi dereivatives):**

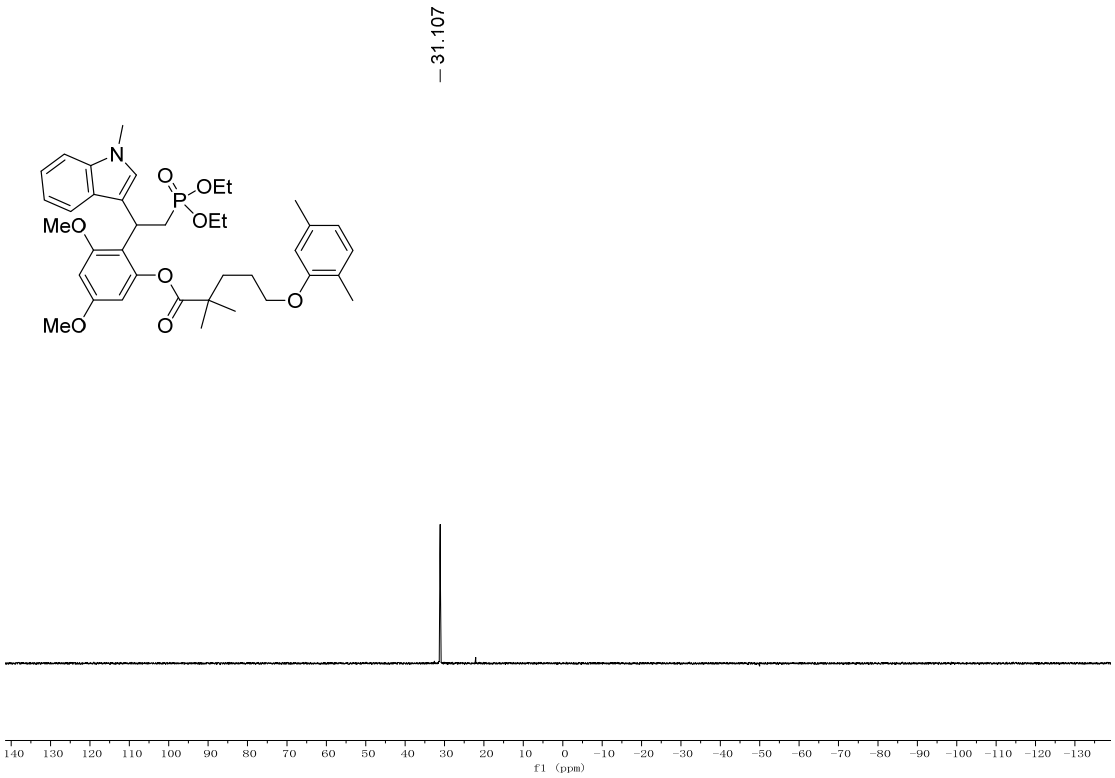
$^{1}$H NMR ($CDCl_3$, 500 MHz)




$^{13}$C NMR ($CDCl_3$, 125 MHz)

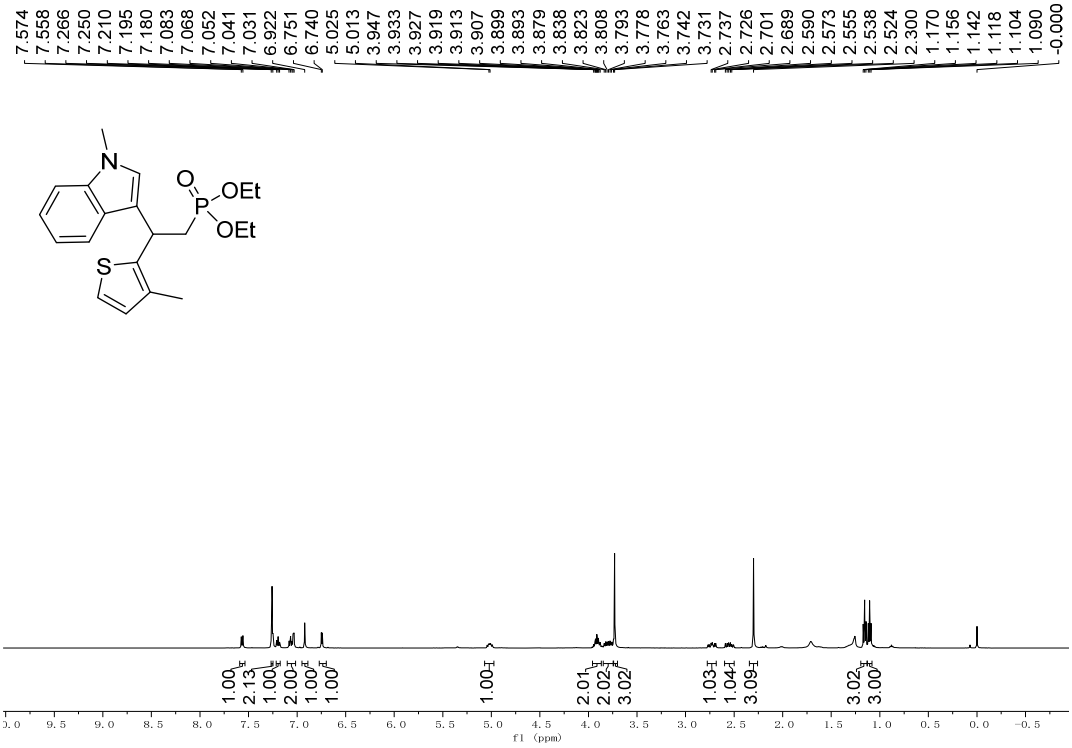


$^{31}$P NMR ($CDCl_3$, 202 MHz)

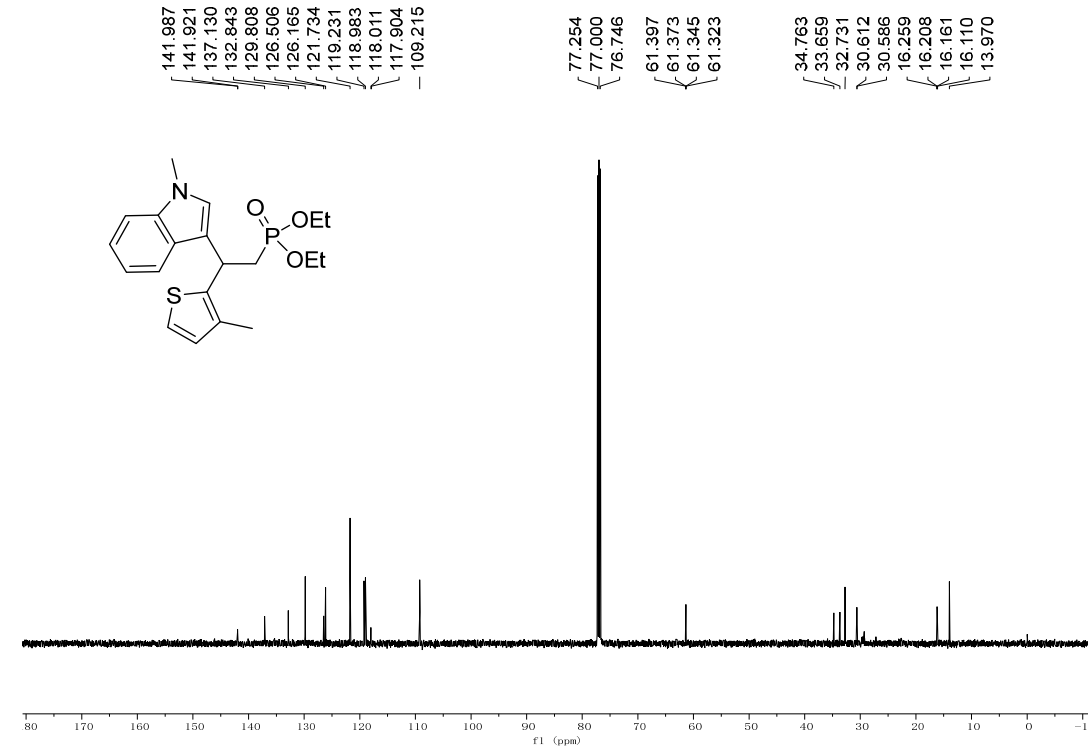


**Diethyl (2-(1-methyl-1*H*-indol-3-yl)-2-(3-methylthiophen-2-yl)ethyl)phosphonate (4naa):**

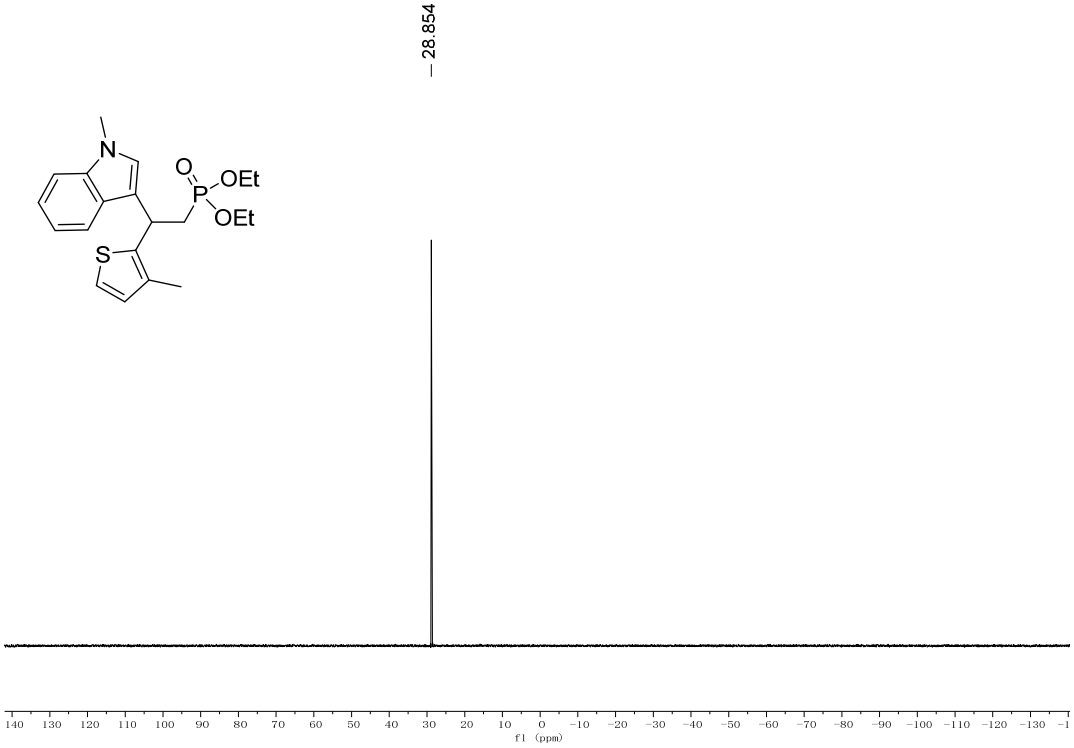
$^{1}$H NMR ($CDCl_3$, 500 MHz)




$^{13}$C NMR ($CDCl_3$, 125 MHz)

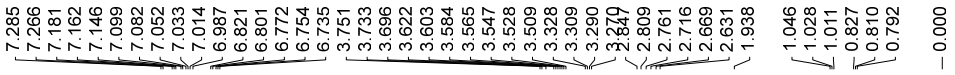


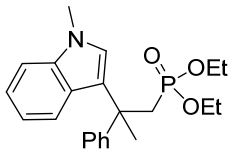
$^{31}$P NMR ($CDCl_3$, 202 MHz)

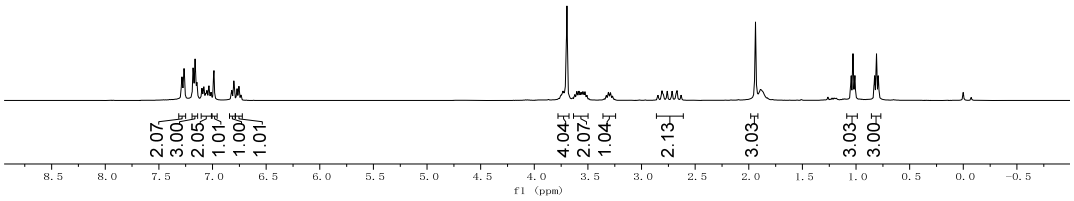


**Diethyl (2-(1-methyl-1*H*-indol-3-yl)-2-phenylpropyl)phosphonate (4haa):**

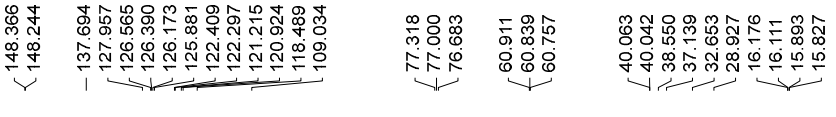
$^{1}$H NMR ($CDCl_3$, 400 MHz)

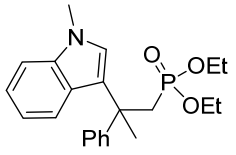


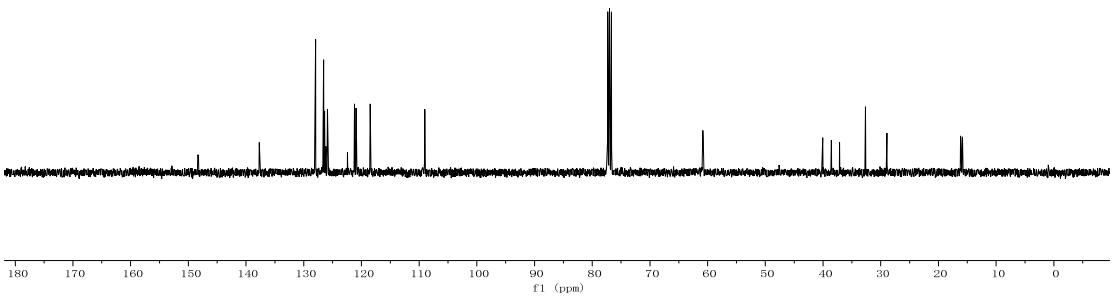



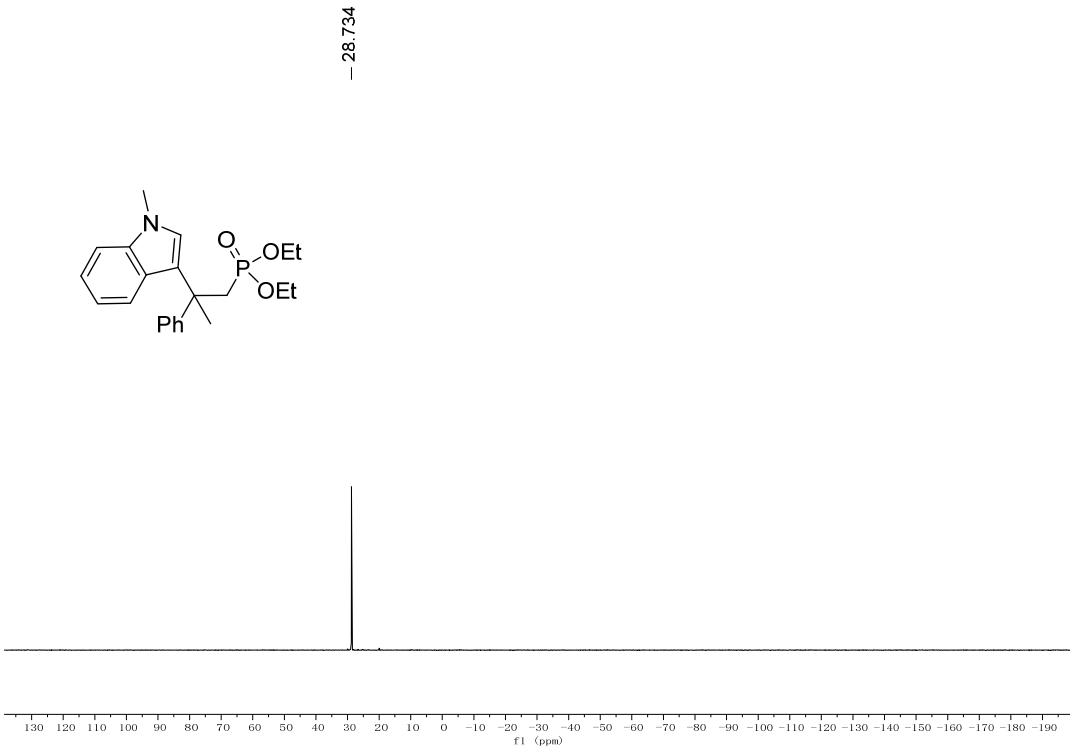

$^{13}$C NMR ($CDCl_3$, 100 MHz)



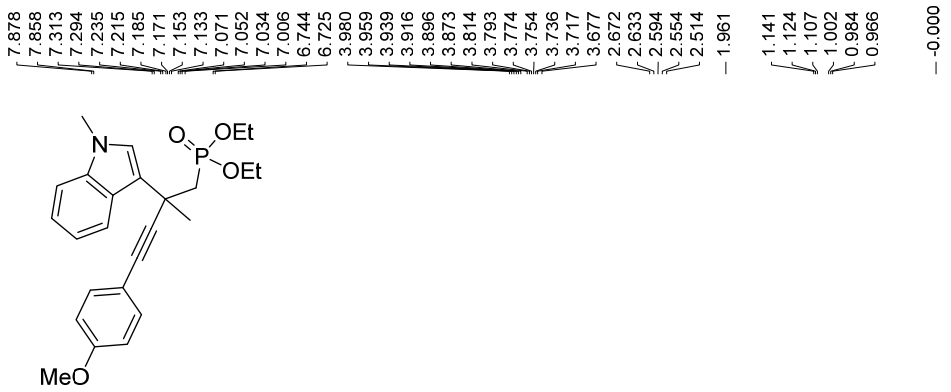



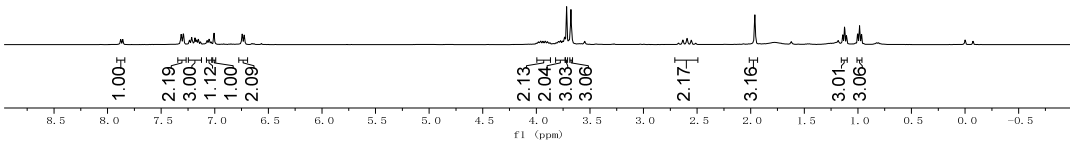
$^{31}P$ NMR ($CDCl_3$, 162 MHz)



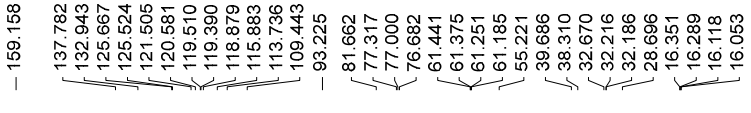
**Diethyl (4-(4-methoxyphenyl)-2-methyl-2-(1-methyl-1*H*-indol-3-yl)but-3-yn-1-yl) phosphonate (4iaa):**

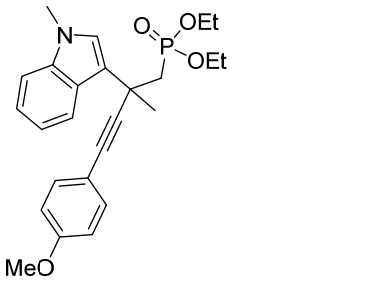
$^{1}$H NMR ($CDCl_3$, 400 MHz)



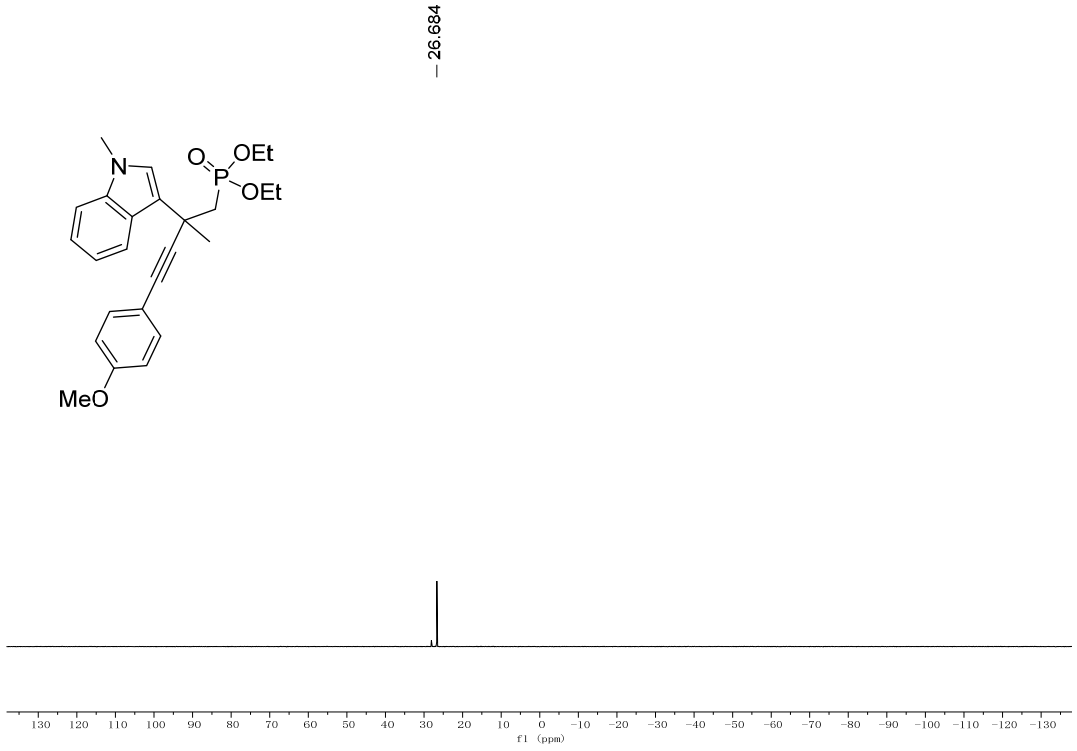




$^{13}$C NMR ($CDCl_3$, 100 MHz)



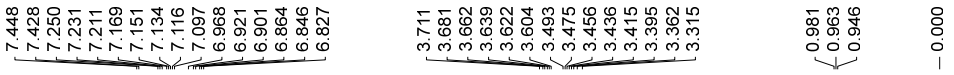



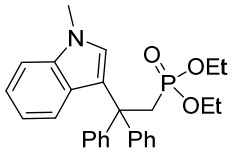
$^{31}$P NMR ($CDCl_3$, 162 MHz)

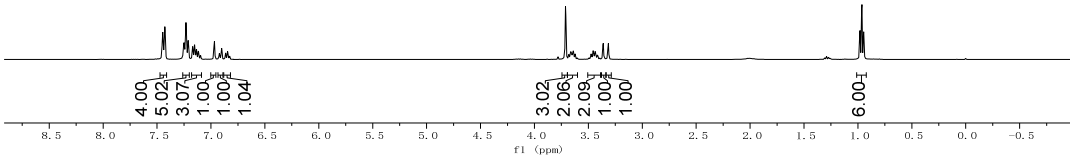


**Diethyl (2-(1-methyl-1*H*-indol-3-yl)-2,2-diphenylethyl)phosphonate (4jaa):**

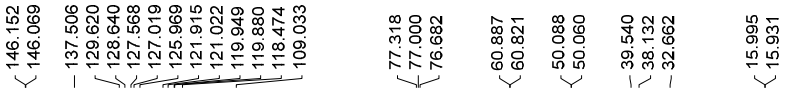
$^{1}$H NMR ($CDCl_3$, 400 MHz)

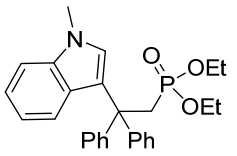


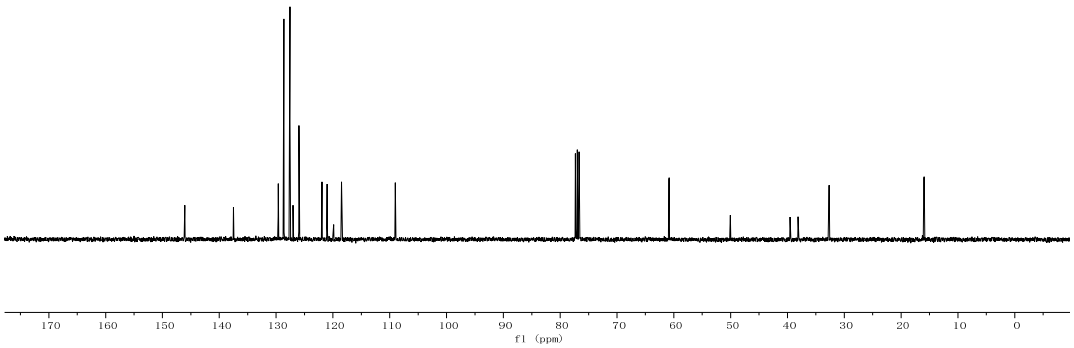



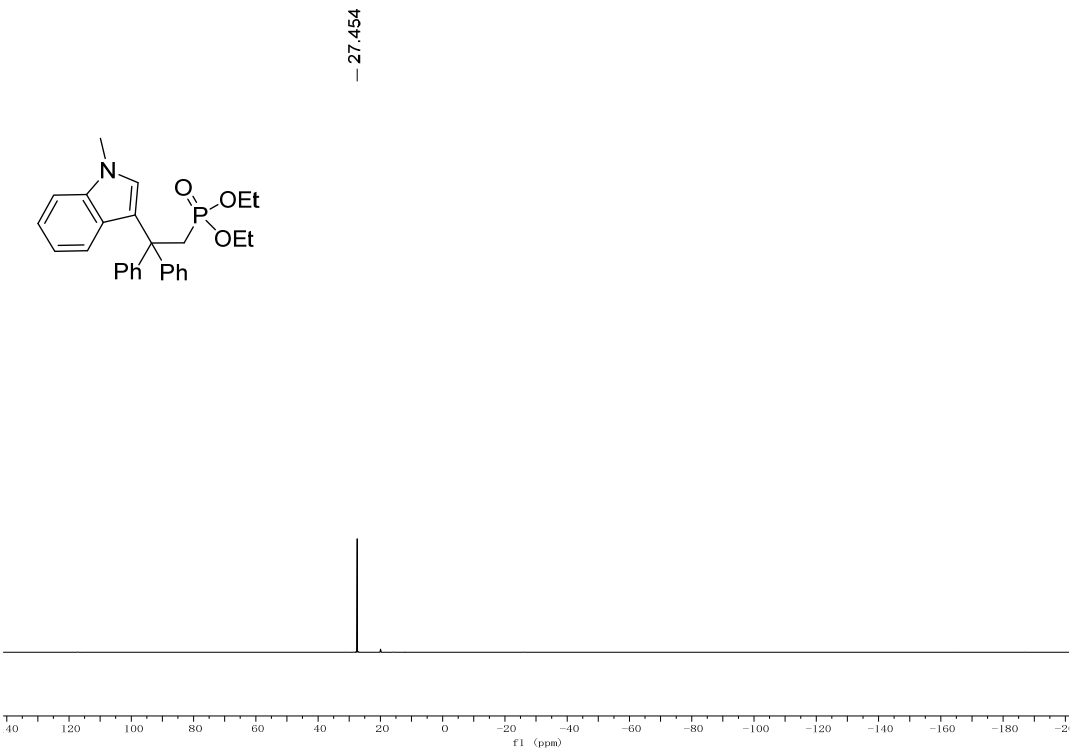

$^{13}$C NMR ($CDCl_3$, 100 MHz)



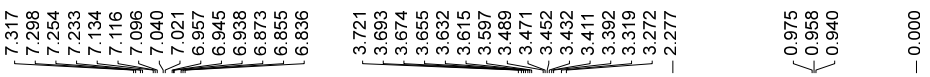



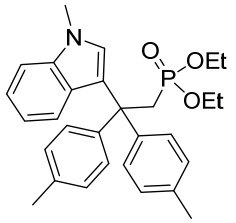
$^{31}$P NMR ($CDCl_3$, 162 MHz)

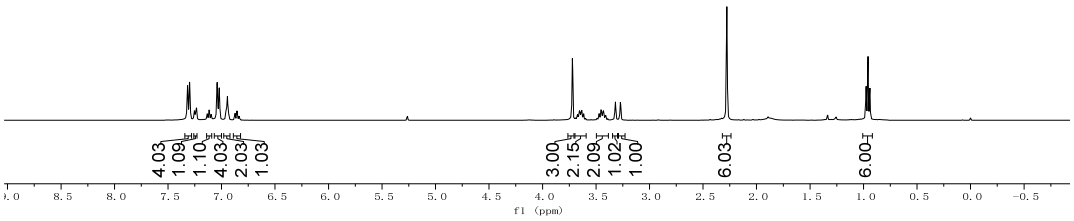


**Diethyl (2-(1-methyl-1*H*-indol-3-yl)-2,2-di-p-tolylethyl)phosphonate (4kaa):**

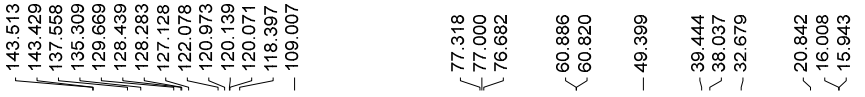
$^{1}$H NMR ($CDCl_3$, 400 MHz)

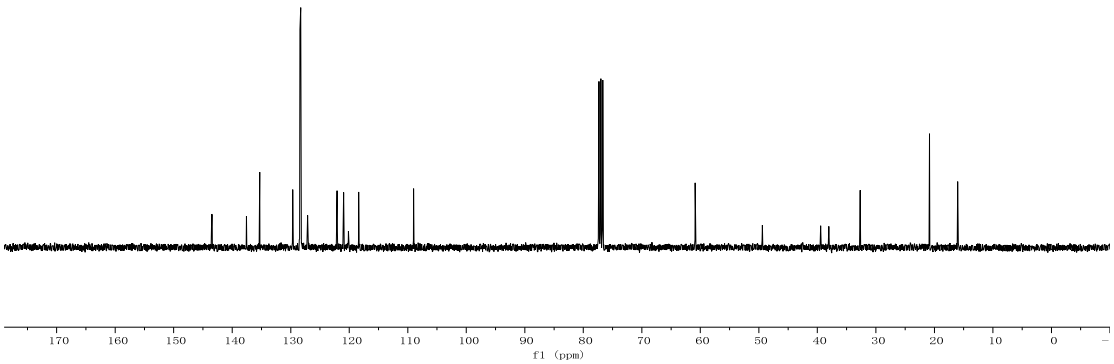


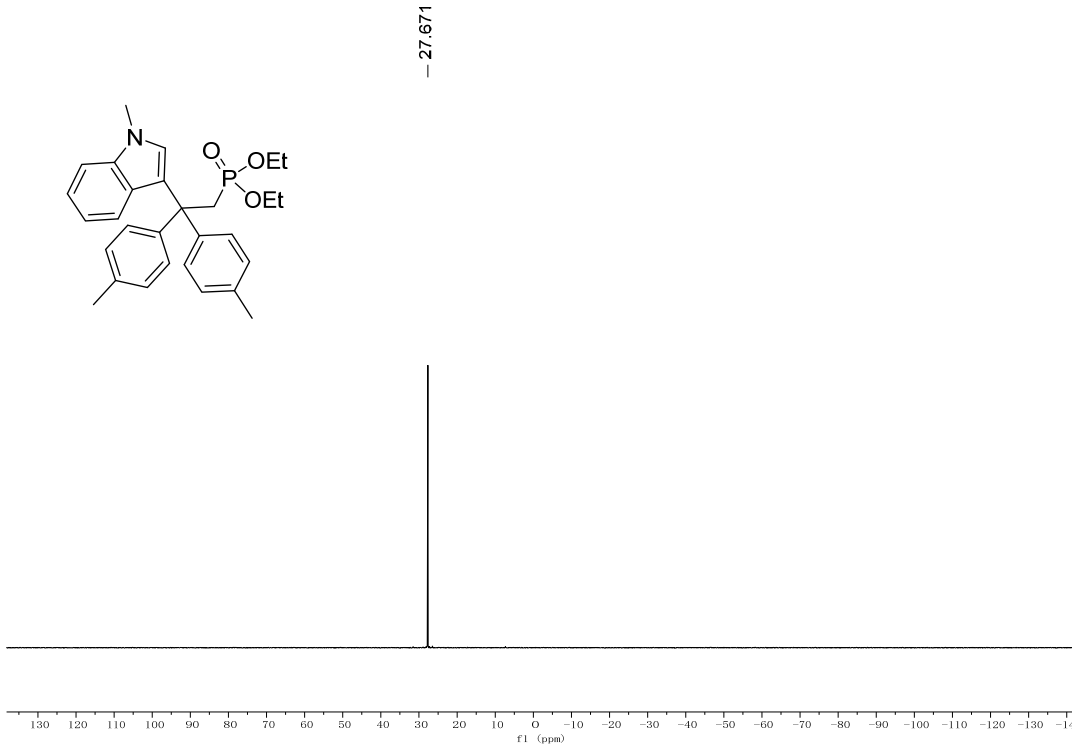




$^{13}$C NMR ($CDCl_3$, 100 MHz)



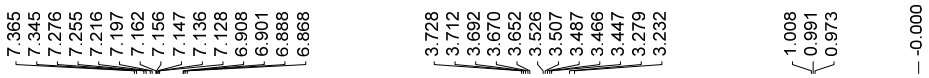



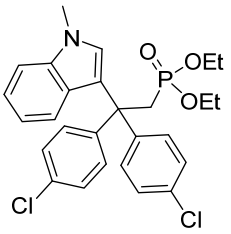
$^{31}$P NMR ($CDCl_3$, 162 MHz)

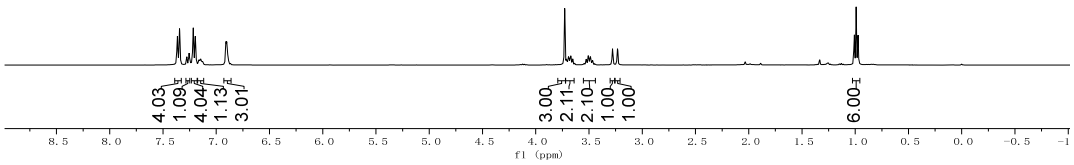


**Diethyl (2,2-bis(4-chlorophenyl)-2-(1-methyl-1*H*-indol-3-yl)ethyl)phosphonate (4laa):**

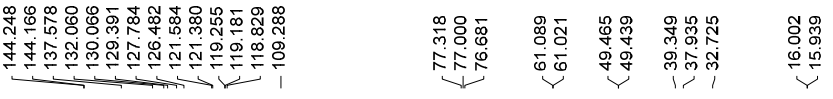
$^{1}$H NMR ($CDCl_3$, 400 MHz)



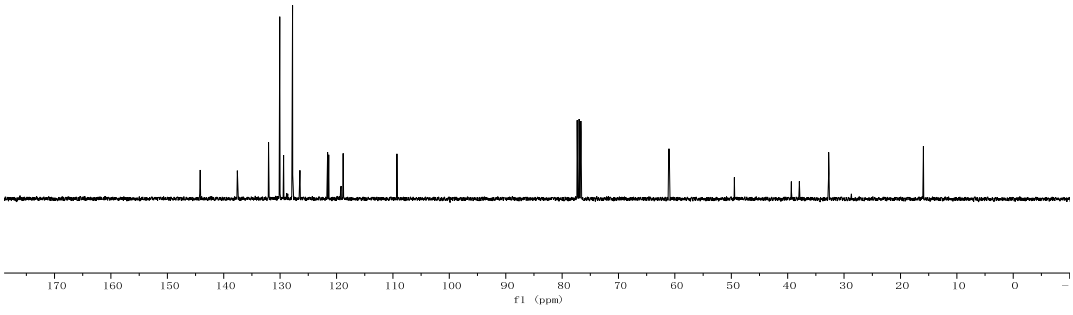



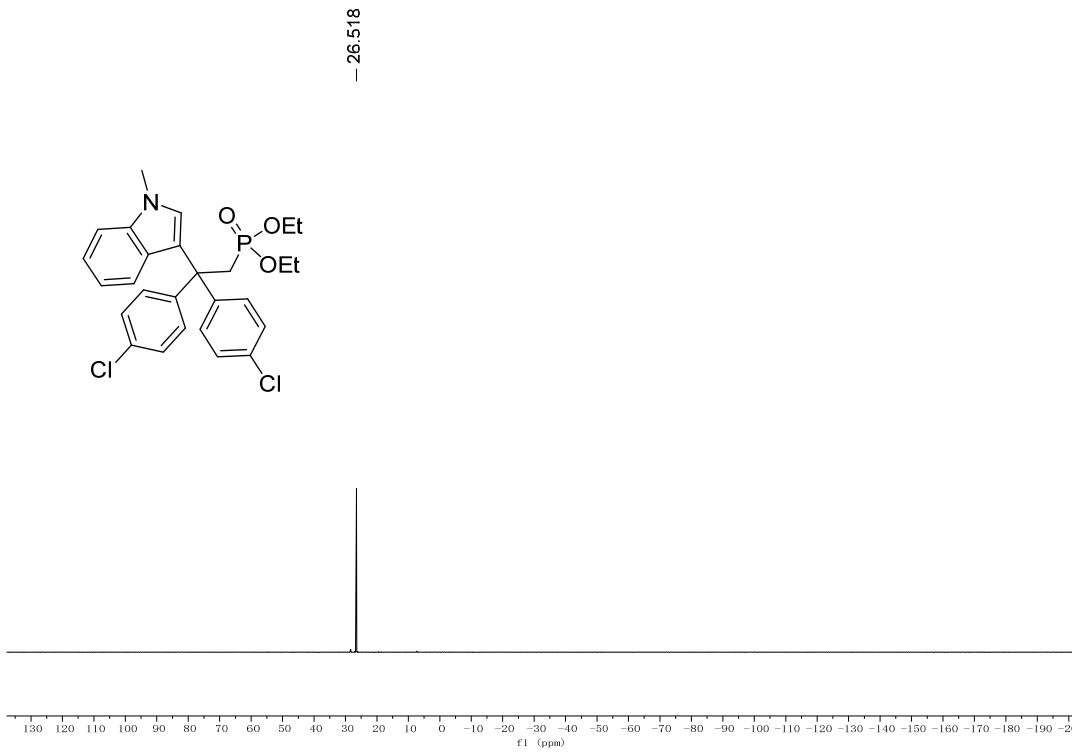

$^{13}$C NMR ($CDCl_3$, 100 MHz)





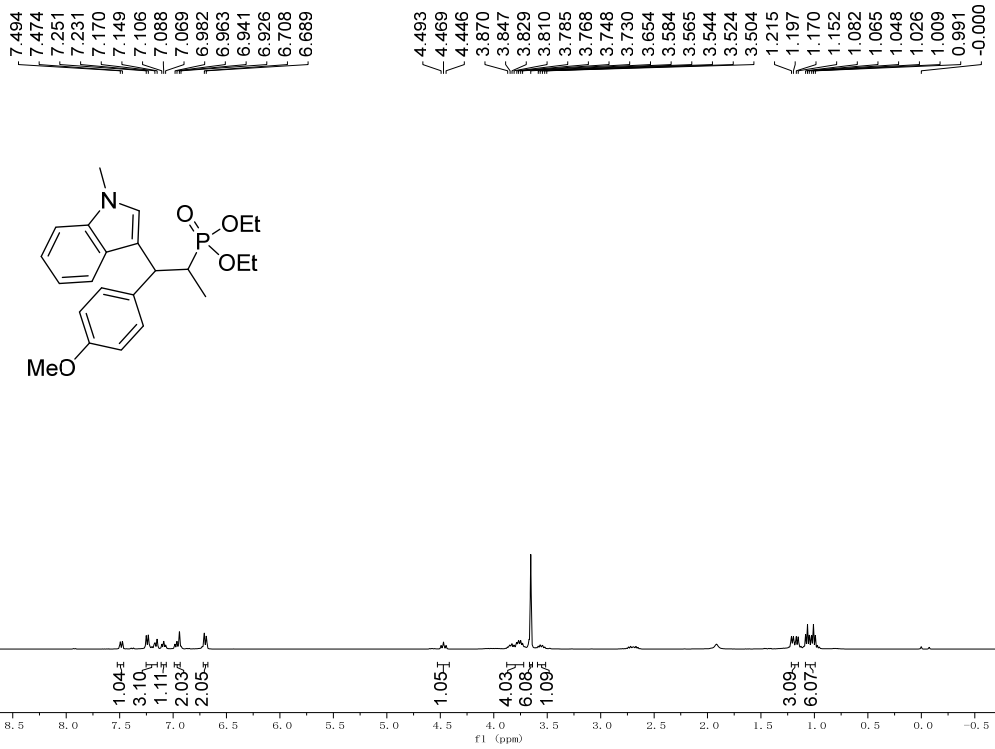


$^{31}$P NMR ($CDCl_3$, 162 MHz)

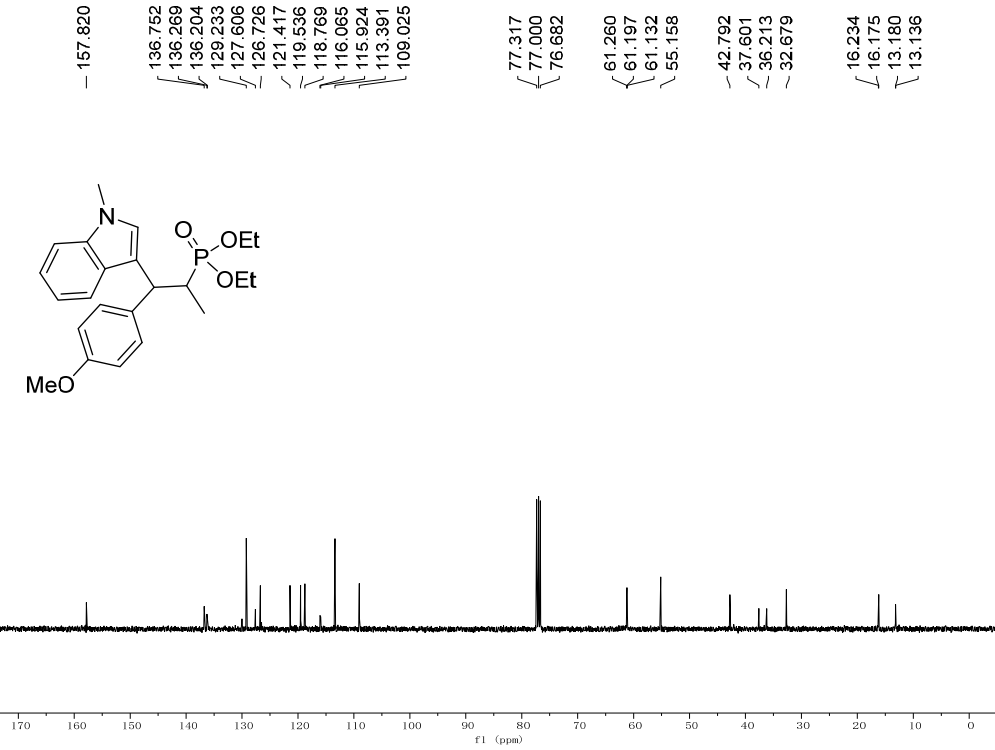


**Diethyl (1-(4-methoxyphenyl)-1-(1-methyl-1*H*-indol-3-yl)propan-2-yl) phosphonate (4maa):**

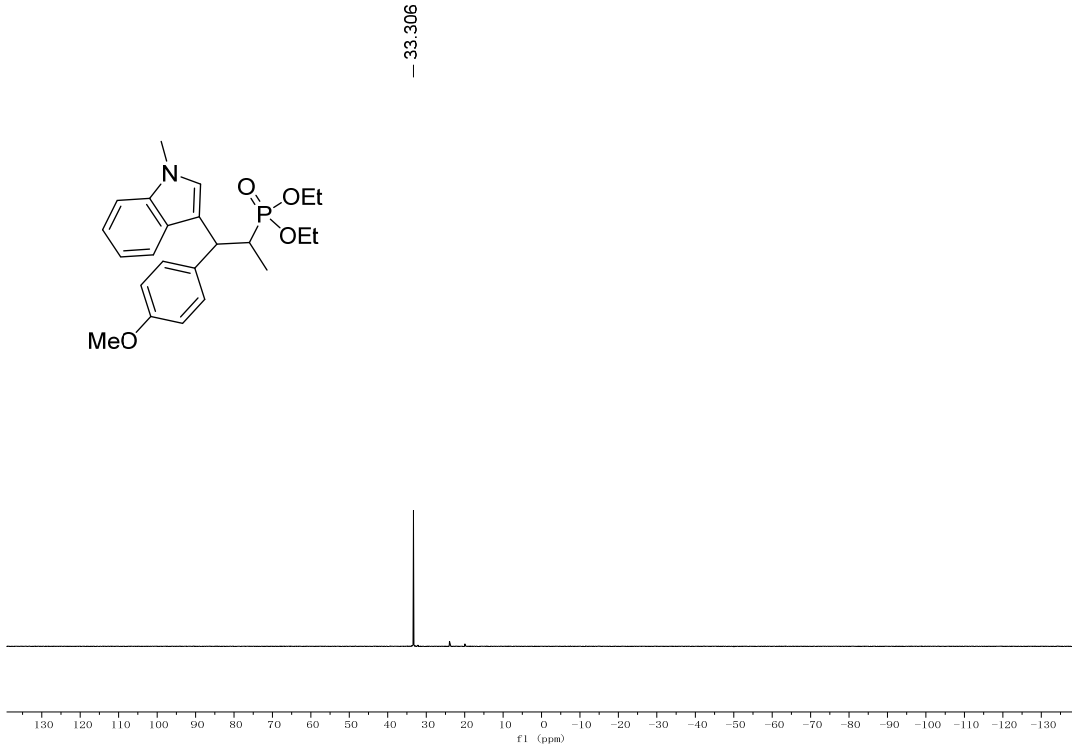
$^{1}$H NMR ($CDCl_3$, 400 MHz)




$^{13}$C NMR ($CDCl_3$, 100 MHz)

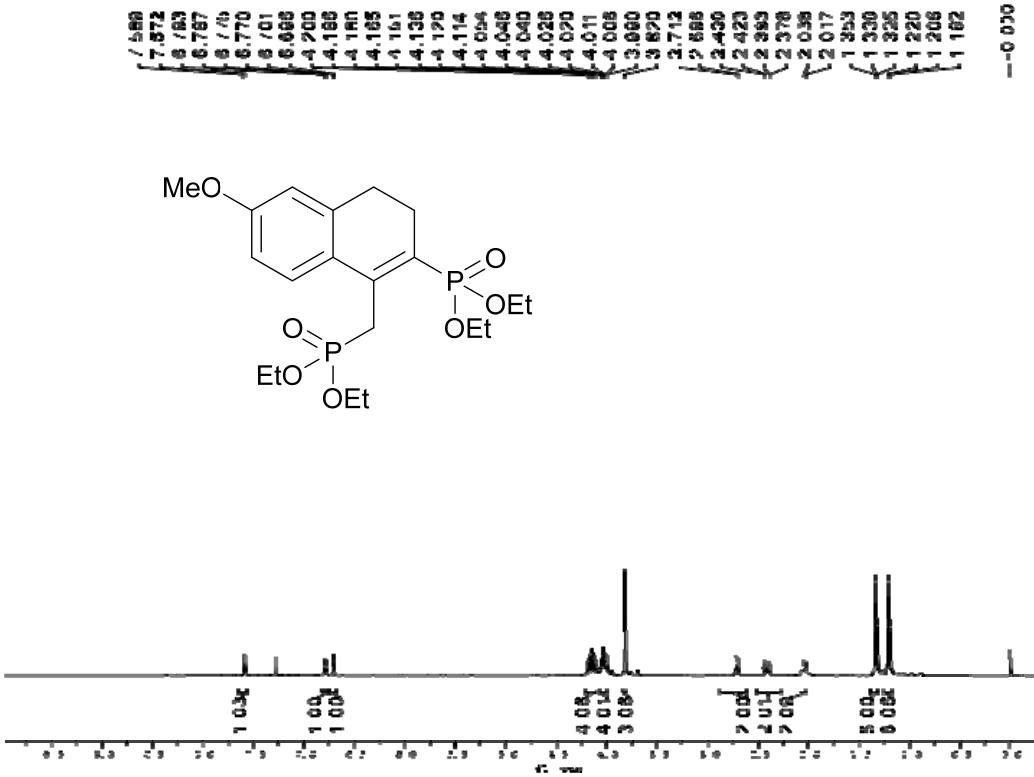


$^{31}$P NMR ($CDCl_3$, 162 MHz)

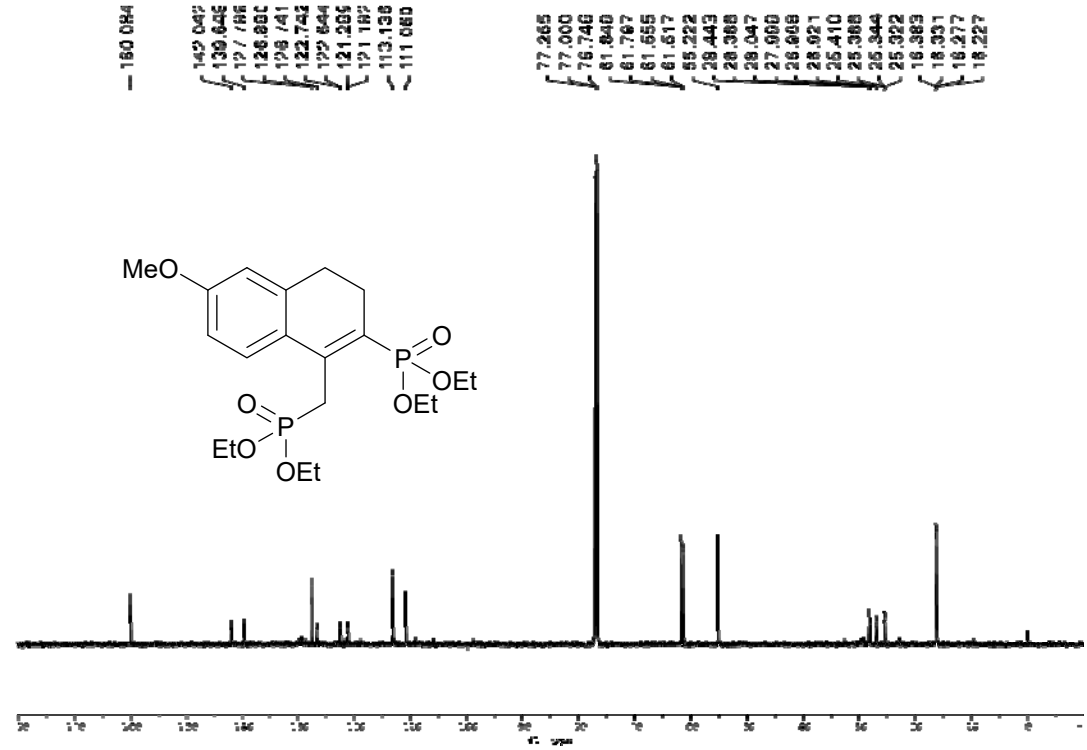


**Diethyl (1-((diethoxyphosphoryl)methyl)-6-methoxy-3,4-dihydronaphthalen-2-yl)phosphonate (5oa):**

$^{1}$H NMR ($CDCl_3$, 500 MHz)




$^{13}$C NMR ($CDCl_3$, 125 MHz)



$^{31}$P NMR ($CDCl_3$, 202 MHz)