Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

A Unique Copper-Catalyzed Cross-Hydrogen (H_2) Removal Coupling to Stereoselective Synthesis of 3-Phosphoindoles

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1. General information.

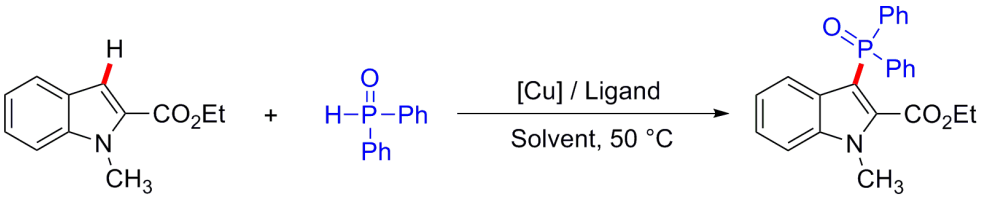
^{1}H and ^{13}C NMR spectra were recorded on a Bruker advance Ⅲ 400 spectrometer in $CDCl_3$ with TMS as internal standard. ^{31}P NMR spectra and ^{19}F NMR were recorded on the same instrument. Mass spectra were mearsured using Bruker microTOF-Q II. The starting materials were purchased from Aldrich, Acros Organics, J&K Chemicals or TCI and used without further purification. Solvents were dried and purified according to the procedure from "Purification of Laboratory Chemicals book". Column chromatography was carried out on silica gel (particle size 200-400 mesh ASTM).

2. Screening results and typical procedure.

2.1 Reaction Conditions Screening

In an initial study, we chose the *N*-methylindole-2-ethyl formate (**1a**) and $Ph_2P(O)H$ (**5a**) as model substrates. We extensively screened catalysts, solvents, and temperatures in an argon atmosphere and summarized in Table S1. Gratifyingly, we found that some copper salts such as CuCl, CuBr, CuBr·Me_2S, $CuCl_2$, $Cu(acac)_2$, and $Cu(OAc)_2$ serve to helpfully prompt the reaction (Table 1, entries 1-6). Among the CuCl gave the best results and the desired product of *N*-methyl-3-phosphoindole (**2a**) was obtained in 64% yield in CH_3CN at 50 ℃ under the Ar atmosphere (Table 1, entry 1). Encouraged by this result, we further optimized the reaction conditions. Solvents screening showed that the CH_3CN is still the best choice (Table 1, entries 7-10). If the reaction was carried out under the oxygen, the yield of *N*-methyl-3-phosphoindole (**2a**) has not obvious change (Table 1, entry 11). Subsequently, we focused our attention on a variety of nitrogen and phosphine ligands in order to understand their potential in this transformation. Extensive evaluation of ligands leads us to discover that phosphine ligands were effective in this regard (Table 1, entries 12-18). To our delight, the least expensive and most stable PPh_3 is the best choice. The yield of *N*-methyl-3-phosphoindole was further improved to 97% (Table 1, entry 18). However, when the reaction was carried out under O_2 or air, the lower yield of *N*-methyl-3-phosphoindole was observed (Table 1, entries 19-20). If the reaction time has been prolonged，the good yield would be afforded under the N_2 atmosphere. The addition of different bases to this transformation proved unhelpful. Furthermore, the control experiment demonstrated that the use of only PPh_3 failed to prompt the reaction.

Table 1. Reaction Conditions Screening.[a]



1a **1b** **2a**

Entry	Cu [mol%]	Ligand [mol%]	Solvent	Yield [%][b]
1	CuCl (5.0)		CH_3CN	64
2	CuBr (5.0)		CH_3CN	42
3	$CuBr{\cdot}Me_2S$ (5.0)		CH_3CN	46
4	$CuCl_2$ (5.0)		CH_3CN	23
5	$Cu(acac)_2$ (5.0)		CH_3CN	22
6	$Cu(OAc)_2$ (5.0)		CH_3CN	6
7	CuCl (5.0)		DMSO	45
8	CuCl (5.0)		DMF	42
9	CuCl (5.0)		NMP	38
10	CuCl (5.0)		1,4-dioxane	17
11	CuCl (5.0)		CH_3CN	62[c]
12	CuCl (5.0)	dppe (6.0)	CH_3CN	84
13	CuCl (5.0)	dppp (6.0)	CH_3CN	80
14	CuCl (5.0)	dppb (6.0)	CH_3CN	79
15	CuCl (5.0)	dpppe (6.0)	CH_3CN	82
16	CuCl (5.0)	dppf (6.0)	CH_3CN	88
17	CuCl (5.0)	binap (6.0)	CH_3CN	18
18	**CuCl (5.0)**	**PPh_3 (6.0)**	**CH_3CN**	**97**
19	CuCl (5.0)	PPh_3 (6.0)	CH_3CN	93[d]
20	CuCl (5.0)	PPh_3 (6.0)	CH_3CN	51[e]
21	-	PPh_3 (6.0)	CH_3CN	0

[a] All reactions were carried out in the presence of 0.3 mmol of $HP(O)Ph_2$ in 3.0 mL CH_3CN at 50 °C for 24 h under argon. [b] Isolated yield. [c] Reaction was carried out under the O_2. [d] Reaction was carried out under the N_2 for 36 h. [e] Reaction was carried out under the air.

2.2 General Procedures for CuCl-Catalyzed Direct Dehydrogenative Phosphorylation for Synthesis of 3-Phosphoindoles (2):

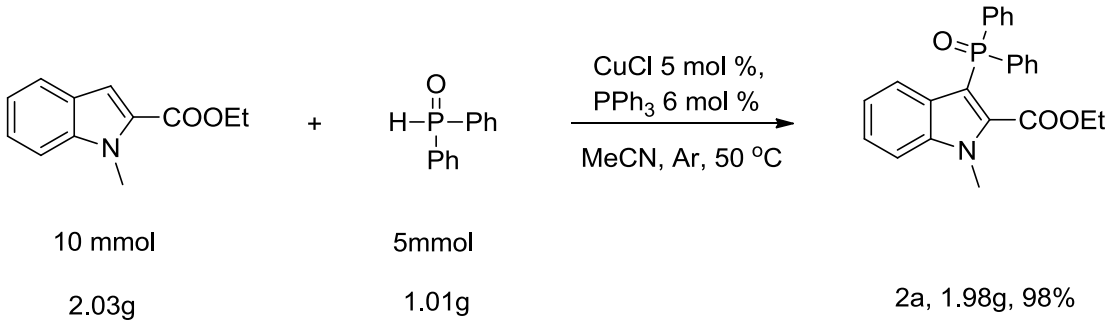
In a Schlenk tube, **1** (0.600 mmol), $HP(O)Ph_2$ (0.300 mmol), CuCl (0.015 mmol), and PPh_3 (0.018 mmol) were added and charged with Ar three times. Then, anhydrous CH_3CN (3.0 mL) were added. The mixture was allowed to stir at 50℃ for 24 hours (monitored by TLC). After substrate was consumed, the reaction was cooled to room temperature and concentrated in vacuo. the resulting residue diluted with EtOAc (5.0 ml) and washed with water (5.0 ml). The aqueous phase was extracted with EtOAc (2×10.0 mL). The combination of organic phase was dried with anhydrous Na_2SO_4

and concentrated in vacuo, and the resulting residue was purified by column chromatography using hexanes/EtOAc (10/1 to 1/1) as the eluent.

2.3 General Procedures for CuCl-Catalyzed Direct Dehydrogenative Phosphorylation for Synthesis of 3-Phosphoindoles (4):

In a Schlenk tube, **3** (0.600 mmol), optically pure H-Phosphinate (0.300 mmol), CuCl (0.015 mmol), and PPh_3 (0.018 mmol) were added and charged with Ar three times. Then, anhydrous CH_3CN (3.0 mL) were added. The mixture was allowed to stir at 80℃ for 24 hours (monitored by TLC). After substrate was consumed, the reaction was cooled to room temperature and concentrated in vacuo. the resulting residue diluted with EtOAc (5.0 ml) and washed with water (5.0 ml). The aqueous phase was extracted with EtOAc (2×10.0 mL). The combination of organic phase was dried with anhydrous Na_2SO_4 and concentrated in vacuo, and the resulting residue was purified by column chromatography using hexanes/EtOAc (10/1 to 1/1) as the eluent.

2.4 Gram-scale synthesis of 2a.



2.5 A Typical Procedure for the Preparation of Optically Pure H-Phosphinates:[S2]

The mixture of (L)-(-)-menthol (100 g, 641 mmol) and pyridine (51.3 mL, 641 mmol) in Et_2O (200 mL) was added dropwise with stirring to a $PhPCl_2$(87.2 mL, 641 mmol) solution in Et_2O (400 mL) at 0℃ and then stirred at room temperature overnight. Water (12 mL, 667 mmol) was added, and the reaction mixture was washed with water and extracted with hexane. The hexane layer was dried over magnesium sulfate, filtered, and concentrated. Recrystallization of the mixture in hexane (twice) at -30℃ gave pure H-Phosphinates a white crystal (R_P/S_P>99/1).

2.6 General Procedures for the determination of the dr values. [S1]

In a Schlenk tube, *N*-methylindole-2-ethyl formate (0.600 mmol), the racemic Menthoxyl-phenylphosphinate (0.300 mmol), CuCl (0.015 mmol), and PPh_3 (0.018 mmol) were added and charged with Ar three times. Then, anhydrous CH_3CN (3.0 mL) were added. The mixture was allowed to stir at 80℃ for 24 hours (monitored by TLC). After substrate was consumed, the reaction was cooled to room temperature and concentrated in vacuo. The resulting residue diluted with EtOAc (5.0 ml) and washed with water (5.0 ml). The aqueous phase was extracted with EtOAc (2×10.0 mL). The combination of organic phase was dried with anhydrous Na_2SO_4 and

concentrated in vacuo. Then the determination of the diatereoselectivity by ^{31}PNMR, or after by flash column chromatography and determined dr values by ^{31}PNMR (Figure S1 and Figure S2).

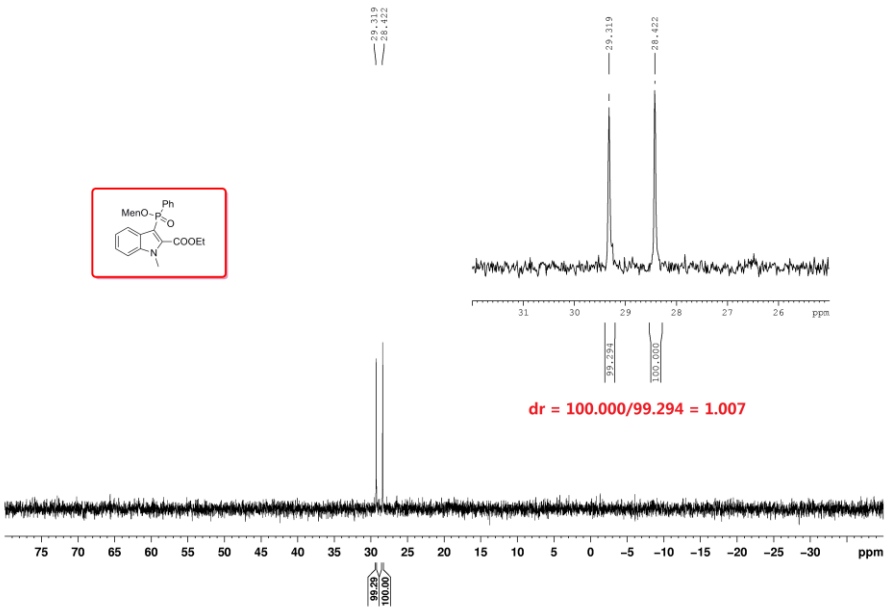


Figure S1. The dr value of racemic H-Phosphinate according to the ^{31}PNMR.

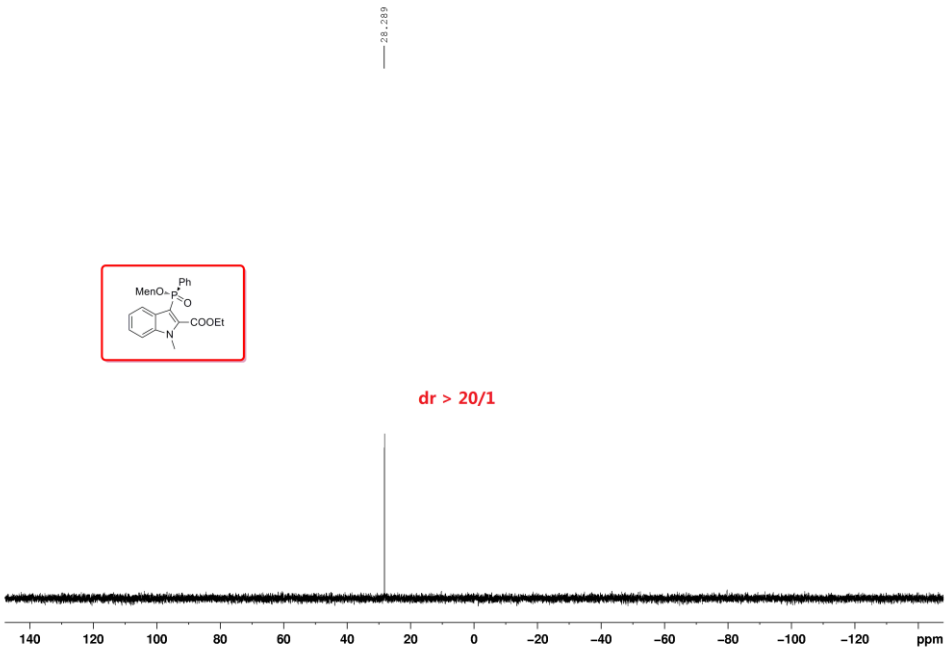


Figure S2. The dr value of the optically pure H-Phosphinate according to the ^{31}PNMR.

3. Preliminary mechanistic studies.

3.1 Radicals Trapping Experiments:

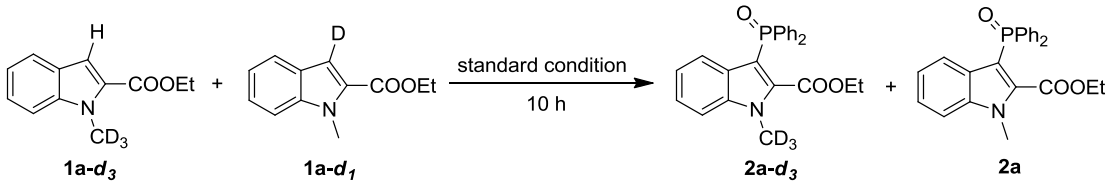
a) Procedures for using BHT: In a Schlenk tube, *N*-methylindole-2-ethyl formate (0.600 mmol), $HP(O)Ph_2$ (0.300 mmol), CuCl (0.015 mmol), BHT (0.600 mmol) and PPh_3 (0.018 mmol) were added and charged with Ar three times. Then, anhydrous CH_3CN (3.0 mL) were added. The mixture was allowed to stir at 50℃ for 24 hours (monitored by TLC). After substrate was consumed, the reaction was cooled to room temperature and concentrated in vacuo. the resulting residue diluted with EtOAc (5.0 ml) and washed with water (5.0 ml). The aqueous phase was extracted with EtOAc (2×10.0 mL). The combination of organic phase was dried with anhydrous Na_2SO_4 and concentrated in vacuo, and the resulting residue was purified by column chromatography to give **2a** in 94% yield.

b) Procedures for using cyclohexa-1, 4-diene: In a Schlenk tube, *N*-methylindole-2-ethyl formate (0.600 mmol), $HP(O)Ph_2$ (0.300 mmol), CuCl (0.015 mmol), cyclohexa-1, 4-diene (0.600 mmol) and PPh_3 (0.018 mmol) were added and charged with Ar three times. Then, anhydrous CH_3CN (3.0 mL) were added. The mixture was allowed to stir at 50℃ for 24 hours (monitored by TLC). After substrate was consumed, the reaction was cooled to room temperature and concentrated in vacuo. the resulting residue diluted with EtOAc (5.0 ml) and washed with water (5.0 ml). The aqueous phase was extracted with EtOAc (2×10.0 mL). The combination of organic phase was dried with anhydrous Na_2SO_4 and concentrated in vacuo, and the resulting residue was purified by column chromatography to give **2a** in 89% yield.

3.2 Competing kinetic isotope effect (KIE) experiment: Intermolecular KIE experiment:

1a-*d*$_1$ were synthesized deuterium substrates according the literature procedure.[S3]

In a Schlenk tube, **1a-*d*$_3$** (0.300 mmol), **1a-*d*$_1$** (0.300 mmol), $HP(O)Ph_2$ (0.300 mmol), CuCl (0.015 mmol), and PPh_3 (0.018 mmol) were added and charged with Ar three times. Then, anhydrous CH_3CN (3.0 mL) were added. The mixture was allowed to stir at 50℃ for 10 hours (monitored by TLC). After substrate was consumed, the reaction was cooled to room temperature and concentrated in vacuo. the resulting residue diluted with EtOAc (5.0 ml) and washed with water (5.0 ml). The aqueous phase was extracted with EtOAc (2×10.0 mL). The combination of organic phase was dried with anhydrous Na_2SO_4 and concentrated in vacuo, and the resulting residue was purified by column chromatography to give **2a-*d*$_3$** and **2a**. The products were under 1H-NMR analysis. (Figure S3).



1a-*d*$_3$ **1a-*d*$_1$** **2a-*d*$_3$** **2a**

1a-*d*$_3$:1a-*d*$_1$ = 1 :1

10 h, 31% yield, K_H/K_D = 1.2

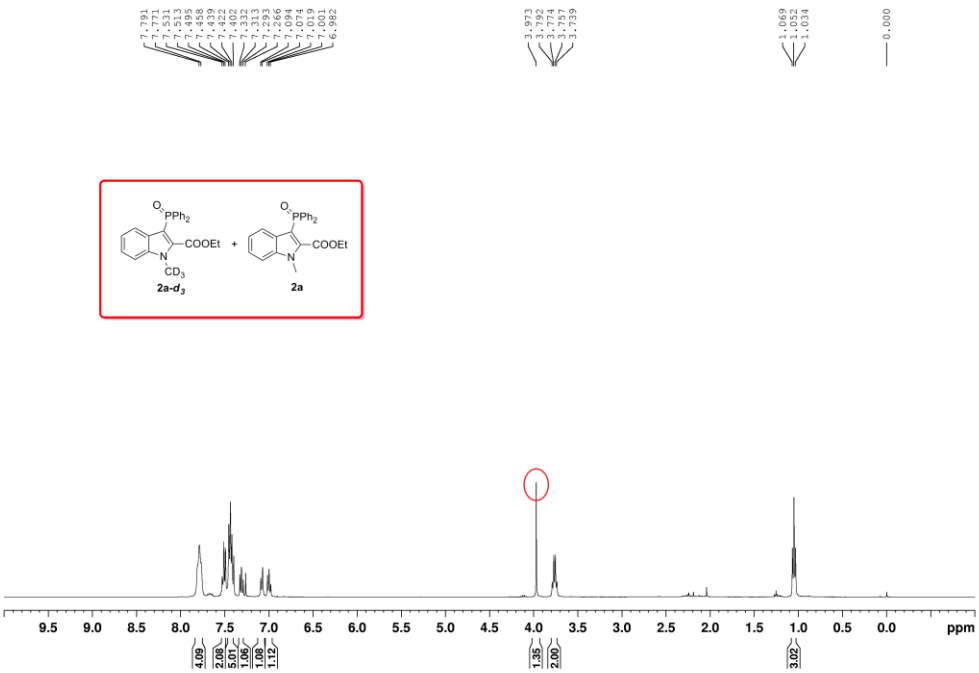


Figure S3. H NMR spectra of the mixture of compound **2a** and **2a-*d*$_3$**.

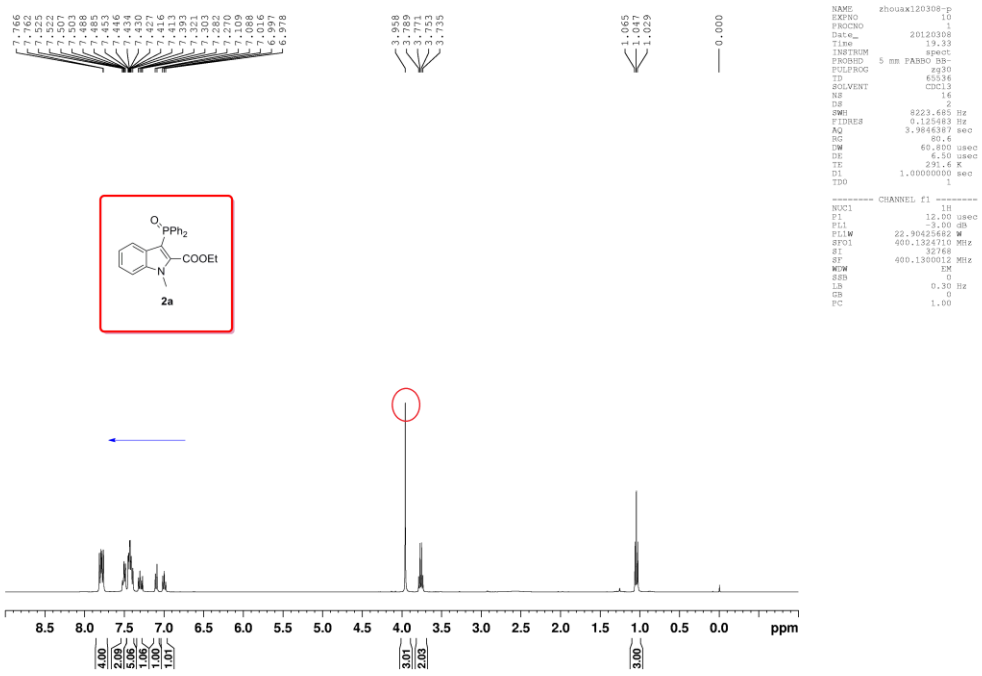


Figure S4. H NMR spectra of **2a**.

3.3 CuH and Stryker's Reagent-Catalyzed Direct Dehydrogenative Coupling Reaction:

a) Procedures for using CuH: In a Schlenk tube, *N*-methylindole-2-ethyl formate (0.600 mmol), $HP(O)Ph_2$ (0.300 mmol), CuH (0.015 mmol), BHT (0.600 mmol) and PPh_3 (0.018 mmol) were added and charged with Ar three times. Then, anhydrous CH_3CN (3.0 mL) were added. The mixture was allowed to stir at 50℃ for 24 hours (monitored by TLC). After substrate was consumed, the reaction was cooled to room temperature and concentrated in vacuo. the resulting residue diluted with EtOAc (5.0 ml) and washed with water (5.0 ml). The aqueous phase was extracted with EtOAc (2×10.0 mL). The combination of organic phase was dried with anhydrous Na_2SO_4 and concentrated in vacuo, and the resulting residue was purified by column chromatography to give **2a** in 76 % yield.

b) Procedures for using Stryker's Reagent: In a Schlenk tube, *N*-methylindole-2-ethyl formate (0.600 mmol), $HP(O)Ph_2$ (0.300 mmol), $[CuH(PPh_3)]_6$ (0.003 mmol), BHT (0.600 mmol) and PPh_3 (0.018 mmol) were added and charged with Ar three times. Then, anhydrous CH_3CN (3.0 mL) were added. The mixture was allowed to stir at 50℃ for 24 hours (monitored by TLC). After substrate was consumed, the reaction was cooled to room temperature and concentrated in vacuo. the resulting residue diluted with EtOAc (5.0 ml) and washed with water (5.0 ml). The aqueous phase was extracted with EtOAc (2×10.0 mL). The combination of organic phase was dried with anhydrous Na_2SO_4 and concentrated in vacuo, and the resulting residue was purified by column chromatography to give **2a** in 78 % yield.

Reference:

[S1] Han, L. B. and Zhao, C. Q., *J. Org. Chem.*, **2005**, *70*, 10121.

[S2] (a) Farnham, W. B.; Murray, R. K.; Mislow, K. *J. Am. Chem. Soc*.**1970**, *92*, 5809. (b) Reiff, L. P.; Aaron, H. S. *J. Am. Chem. Soc*.**1970**, *92*, 5275. (c) Benschop, H. P.; Platenburg, D. H. J. M.; Meppelder, F. H.; Boter, H. *J. Chem. Commun*. **1970**, 33. (d) Bodalski, R.; Koszuk, J. *Phosphorus, Sulfur, Silicon Relat. Elem*.**1989**, *44*, 99. (e) Han, L.-B.; Zhao, C.-Q.; Onozawa, S.; Goto, M.; Tanaka, M. *J. Am. Chem. Soc*. **2002**, *124*, 3842. (f) Han, L.-B.; Zhao, C.-Q. *J. Org. Chem*. **2005**, *70*, 10121. (g) Han, L.-B.; Zhao, C.-Q.; Xu, Q. *J. Am. Chem. Soc*. **2008**, *130*, 12648. (h) Storer, R.; Alexandre, F. R.; Dousson, C.; Moussa, A. M.; Bridges, E. *Enantiomerically pure phosphoindoles as HIV inhibitors*. WO PCT Int. Appl. 042240, 2008.

[S3] Wang, R., Shi, H. F., Zhao, J. F., He, Y. P., Zhang, H. B., Liu, J. P., *Bioorg. Med. Chem. Lett*., **2013**, ***23***, 1760.

3.4 Hydrogen detection

1. a) Hydrogen detector：

HEWLETT5890 PACKADR SERIES II GAS CHROMATOGRAPH

b) Carrier gas： Ar

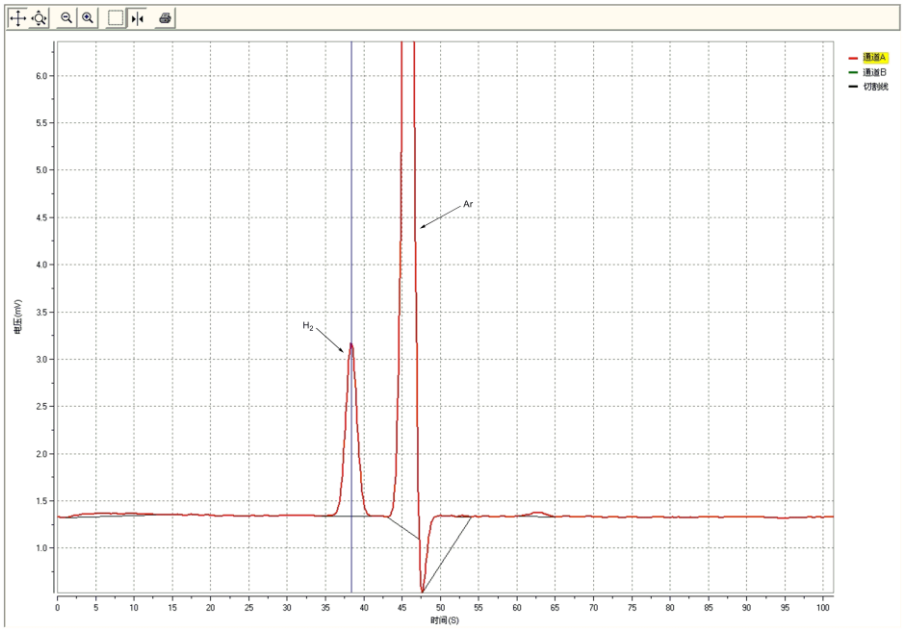


Figure S5. The standard spectrogram of H_2.

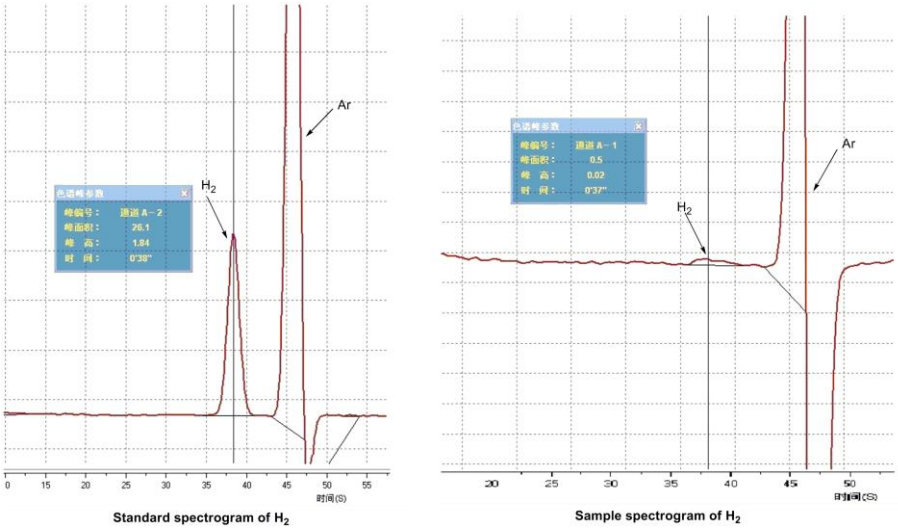


Figure S6. The detected spectrogram of H_2.

a) Procedures for the detected H_2 by Gas Chromatograph:

By using the high purity of Ar as carrier gas, we first injected the pure H_2 into the Hewlett5890 Packadr Series II Gas Chromatograph and detected standard spectrogram of H_2 (Figure S5**)**. Then 15 minutes later, we injected the sample gas of reaction system into the Gas Chromatograph and observed the peak of H_2 (Figure S6**)**. This result showed that the H_2 has been released from the reaction.

2. Hydrogen detector：**TAYASAF-MG01 portable H_2 Detector**

Figure S7. The concentration of H_2 (223 ppm).

b) Procedures for the detected H_2 by Portable H_2 Detector:

In a 50 mL round-bottom flask, *N*-methylindole-2-ethyl formate (10.0 mmol), H-Phosphinate (5.0 mmol), CuCl 5.0 mmol%, and 6.0 mmol% were added and charged with Ar three times. Then, anhydrous CH_3CN (20.0 mL) were added. The mixture was allowed to stir at 50℃ for 24 hours and then the reaction was cooled to room temperature. After we injected the syringe needle, which linked with Portable H_2 Detector, into the reaction system and H_2 Detector began to alarm and the instantaneous H_2 concentration was detected with 223 ppm. It worth to note that the highest concentration reached 476 ppm.

3. Mixing Stryker's reagent with $HP(O)Ph_2$

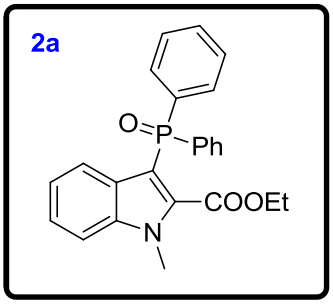
a) Hydrogen detector：TAYASAF-MG01 portable H_2 Detector

Figure S8. the observation of H_2 evolution

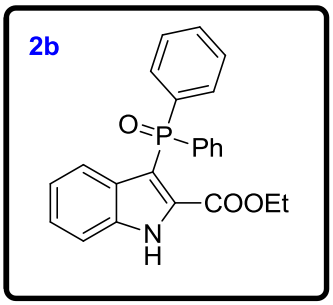
b) Procedures for the detected H_2 by Portable H_2 Detector:

In a 10 mL round-bottom flask, H-Phosphinate (0.5 mmol) , Stryker's reagent (0.5 mmol) were added and charged with Ar three times. Then, anhydrous CH_3CN (5.0 mL) were added. The mixture was allowed to stir at 50℃ for 12 hours and then the reaction was cooled to room temperature. After we injected the syringe needle, which linked with Portable H_2 Detector, into the reaction system and H_2 Detector began to alarm and the instantaneous H_2 concentration was detected with 113 ppm.

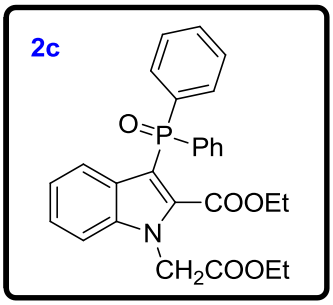
4. Characterization of new compounds.



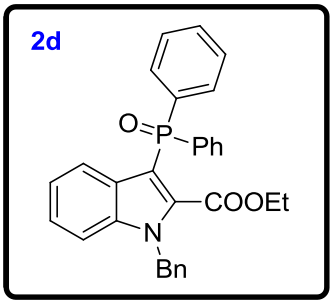
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (97% yield) as a white solid. Mp: 148-150 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.29. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.84 - 7.76 (m, 4H), 7.53 - 7.48 (m, 2H), 7.46 - 7.39 (m, 5H), 7.33 - 7.27 (m, 1H), 7.10 (d, *J* = 8.4 Hz, 1H), 7.00 (t, *J* = 7.6 Hz, 1H), 3.96 (s, 3H), 3.76 (q, *J* = 7.2 Hz, 2H), 1.05 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.6, 138.1 (d, $J_{C\text{-}P}$ = 11.0 Hz), 135.5 (d, $J_{C\text{-}P}$ = 16.0 Hz), 134.6 (d, $J_{C\text{-}P}$ = 109.0 Hz), 131.5 , 131.4 (d, $J_{C\text{-}P}$ = 3.0 Hz), 128.5 (d, $J_{C\text{-}P}$ = 9.0 Hz), 128.3 (d, $J_{C\text{-}P}$ = 13.0 Hz), 124.6, 122.7, 121.9, 110.3, 107.2 (d, $J_{C\text{-}P}$ = 120.0 Hz), 61.9, 31.8, 13.4. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 21.66. **HRMS** Calcd for $C_{24}H_{22}NaO_3P$ [M + Na]$^{+}$ 426.1230, found 426.1224.



Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (93% yield) as a white solid. Mp: 226-228 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.13. **^{1}H NMR (400 MHz, d_6-DMSO):** δ 12.85 (brs, 1H), 7.72 - 7.66 (m, 4H), 7.59 - 7.56 (m, 3H), 7.51 - 7.49 (m, 4H), 7.31 - 7.26 (m, 2H), 6.98 (t, *J* = 7.6 Hz , 1H), 3.81 (q, *J* = 6.8 Hz , 2H), 0.89 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR (100 MHz, d_6-DMSO):** δ 160.2, 136.7 (d, $J_{C\text{-}P}$ = 12.0 Hz), 135.2 (d, $J_{C\text{-}P}$ = 108.0 Hz), 132.3 (d, $J_{C\text{-}P}$ = 15.0 Hz), 131.5, 131.0 (d, $J_{C\text{-}P}$ = 10.0 Hz), 129.9 (d, $J_{C\text{-}P}$ = 8.0 Hz), 128.5 (d, $J_{C\text{-}P}$ = 12.0 Hz), 124.9, 122.5, 121.5, 113.1, 107.4 (d, $J_{C\text{-}P}$ = 118.0 Hz), 61.1, 13.6. **^{31}P NMR (162 MHz, d_6-DMSO):** δ 21.13. **HRMS** Calcd for $C_{23}H_{20}NNaO_3P$ [M + Na]$^{+}$ 412.1073, found 412.1065.

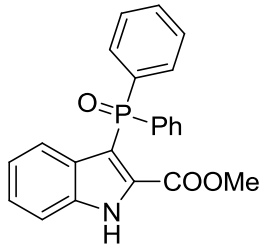


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (78% yield) as a white solid. Mp: 155-157 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.24. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.82 - 7.76 (m, 4H), 7.53 - 7.49 (m, 2H), 7.47 - 7.41 (m, 4H), 7.34 - 7.32 (m, 2H), 7.19 (d, *J* = 8.4 Hz, 1H), 7.04 - 7.00 (m, 1H), 5.24 (s, 2H), 4.21 (q, *J* = 7.2 Hz, 2H), 3.73 (q, *J* = 7.2 Hz, 2H), 1.25 (t, *J* = 7.2 Hz, 3H), 0.97 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 167.8, 161.2, 138.2 (d, $J_{C\text{-}P}$ = 10.0Hz), 134.8 (d, $J_{C\text{-}P}$ = 110.0 Hz), 134.4, 131.5 (d, $J_{C\text{-}P}$ = 10.0 Hz), 128.9 (d, $J_{C\text{-}P}$ = 8.0 Hz), 128.3 (d, $J_{C\text{-}P}$ = 12.0 Hz), 125.3, 123.4, 122.3, 109.7, 109.6 (d, $J_{C\text{-}P}$ = 118.0 Hz), 61.8, 61.6, 46.5, 14.0, 13.4. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 22.64. **HRMS** Calcd for $C_{27}H_{26}NNaO_5P$ $[M + Na]^+$ 498.1441, found 498.1450.



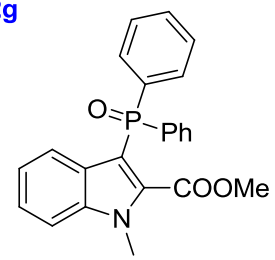
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (80% yield) as a yellow oil. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.42. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.83 - 7.76 (m, 4H), 7.55 - 7.50 (m, 2H), 7.47 - 7.42 (m, 4H), 7.36 (d, *J* = 8.4 Hz, 1H), 7.31 - 7.23 (m, 4H), 7.15 (d, *J* = 8.0 Hz, 1H), 7.08 (d, *J* = 6.8 Hz, 1H), 7.00 (t, *J* = 7.2 Hz, 1H), 5.64 (s, 2H), 3.65 (q, *J* = 7.2 Hz, 2H), 0.93 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.7, 137.9 (d, $J_{C\text{-}P}$ = 11.0 Hz), 136.6, 135.7 (d, $J_{C\text{-}P}$ = 16.0 Hz), 134.6 (d, $J_{C\text{-}P}$ = 109.0 Hz), 131.7, 131.5 (d, $J_{C\text{-}P}$ = 4.0 Hz), 128.9, 1128.7 (d, $J_{C\text{-}P}$ = 5.0 Hz), 128.4, 128.3, 127.7, 126.5, 124.9, 123.0, 122.1, 110.9, 108.0 (d, $J_{C\text{-}P}$ = 109.0 Hz), 61.8, 48.5, 13.4. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 21.44. **HRMS** Calcd for $C_{30}H_{26}NNaO_3P$ $[M + Na]^+$ 502.1546, found 502.1548.

2f

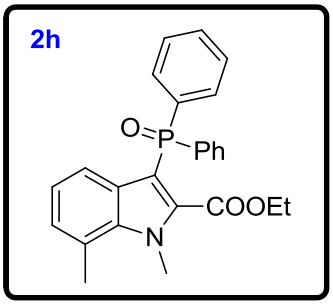


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (88% yield) as a white solid. Mp: 215-217 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.10. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.70 - 7.64 (m, 4H), 7.53 - 7.46 (m, 3H), 7.45 - 7.40 (m, 4H), 7.22 - 7.17 (m, 1H), 6.93 - 6.84 (m, 2H), 4.77 (s, 3H), 3.36 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 162.0, 138.4 (d, J_{C-P} = 12.0 Hz), 135.4 (d, J_{C-P} = 111.0 Hz), 134.4 (d, J_{C-P} = 16.0 Hz), 133.2.0 (d, J_{C-P} = 3.0 Hz), 132.7, 132.6, 131.2 (d, J_{C-P} = 9.0 Hz), 129.9, 129.8, 126.5, 123.4, 123.1, 114.0, 108.0 (d, J_{C-P} = 123.0 Hz), 52.5. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 26.10. **HRMS** Calcd for $C_{22}H_{18}NNaO_3P$ $[M + Na]^+$ 398.0917, found 398.0902.

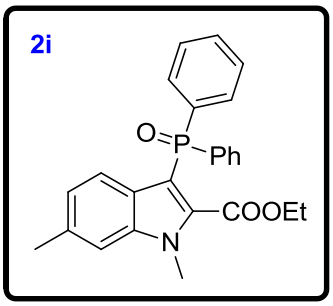
2g



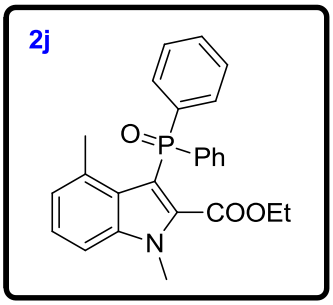
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (91% yield) as a white solid. Mp: 171-173 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.19. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.83 - 7.75 (m, 4H), 7.52 - 7.47 (m, 2H), 7.44 - 7.39 (m, 5H), 7.31 (t, J = 7.2 Hz, 1H), 7.17 (d, J = 8.4 Hz, 1H), 7.00 (t, J = 7.6 Hz, 1H), 3.96 (s, 3H), 3.27 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 162.0, 138.4 (d, J_{C-P} = 10.0 Hz), 134.9 134.8 (d, J_{C-P} = 109.0 Hz), 131.4 (d, J_{C-P} = 2.0 Hz), 131.3 (d, J_{C-P} = 10.0 Hz), 128.7 (d, J_{C-P} = 10.0 Hz), 128.5, 128.4, 124.9, 123.0, 122.0, 110.4, 108.1 (d, J_{C-P} = 119.0 Hz), 51.8, 31.9. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 21.05. **HRMS** Calcd for $C_{23}H_{20}NNaO_3P$ $[M + Na]^+$ 412.1073, found 412.1079.



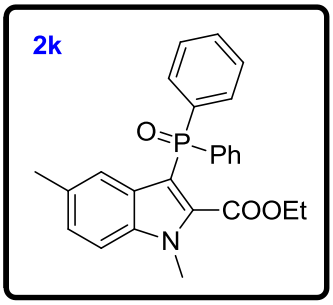
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (90% yield) as a yellow oil. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.35. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.80 - 7.74 (m, 4H), 7.52 - 7.47 (m, 2H), 7.44 - 7.40 (m, 4H), 7.12 (d, *J* = 8.4 Hz, 1H), 6.99 (d, *J* = 7.2 Hz, 1H), 6.87 (t, *J* = 7.6 Hz, 1H), 4.13 (s, 3H), 3.76 (q, *J* = 7.2 Hz, 2H), 2.77 (s, 3H), 1.09 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 162.2, 137.6 (d, $J_{C\text{-}P}$ = 16.0 Hz), 137.0 (d, $J_{C\text{-}P}$ = 11.0 Hz), 134.3 (d, $J_{C\text{-}P}$ = 109.0 Hz), 132.0, 131.9, 131.8, 131.6, 131.5, 131.4 (d, $J_{C\text{-}P}$ = 2.0 Hz), 129.3 (d, $J_{C\text{-}P}$ = 8.0 Hz), 128.5, 128.3 (d, $J_{C\text{-}P}$ = 6.0 Hz), 128.2, 127.3, 121.9, 121.8, 120.7, 106.0 (d, $J_{C\text{-}P}$ = 121.0 Hz), 61.9, 34.9, 20.5, 13.5. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 21.57. **HRMS** Calcd for $C_{25}H_{24}NNaO_3P$ $[M + Na]^+$ 440.1410, found 440.1398.



Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (90% yield) as a white solid. Mp: 163-165 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.27. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.81 - 7.76 (m, 4H), 7.51 - 7.48 (m, 2H), 7.45 - 7.40 (m, 4H), 7.19 (s, 1H), 6.90 (d, *J* = 8.4 Hz, 1H), 6.82 (t, *J* = 8.4 Hz, 1H), 3.94 (s, 3H), 3.76 (q, *J* = 7.2 Hz, 2H), 2.43 (s, 3H), 1.03 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.6, 138.7 (d, $J_{C\text{-}P}$ = 11.0 Hz), 135.1 (d, $J_{C\text{-}P}$ = 109.0 Hz), 134.9, 134.8 (d, $J_{C\text{-}P}$ = 12.0 Hz), 131.4 (d, $J_{C\text{-}P}$ = 10.0 Hz), 131.3 (d, $J_{C\text{-}P}$ = 3.0 Hz), 128.2 (d, $J_{C\text{-}P}$ = 12.0 Hz), 126.5 (d, $J_{C\text{-}P}$ = 9.0 Hz), 123.9, 122.3, 110.0, 107.9 (d, $J_{C\text{-}P}$ = 121.0 Hz), 61.5, 31.7, 21.7, 13.4. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 21.60. **HRMS** Calcd for $C_{25}H_{24}NNaO_3P$ $[M + Na]^+$ 440.1386, found 440.1382.

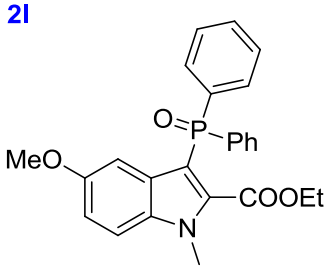


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (89% yield) as a yellow oil. $\mathbf{R_f}$ (ethyl acetate: petroleum ether, 2:1): 0.32. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.76 - 7.70 (m, 4H), 7.49 - 7.45 (m, 2H), 7.42 - 7.38 (m, 4H), 7.27 - 7.23 (m, 2H), 6.96 - 6.93 (m, 1H), 3.76 (s, 3H), 3.47 (q, J = 7.2 Hz, 2H), 2.73 (s, 3H), 1.11 (t, J = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 162.4, 138.2 (d, $J_{C\text{-}P}$ = 10.0 Hz), 138.0, 135.0 (d, $J_{C\text{-}P}$ = 108.0 Hz), 133.7, 132.0 (d, $J_{C\text{-}P}$ = 10.0 Hz), 131.3 (d, $J_{C\text{-}P}$ = 2.0 Hz), 128.1, 128.0 (d, $J_{C\text{-}P}$ = 6.0 Hz), 124.5, 124.3, 107.6, 104.9 (d, $J_{C\text{-}P}$ = 120.0 Hz), 61.9, 31.5, 22.6, 13.6. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 22.36. **HRMS** Calcd for $C_{25}H_{24}NNaO_3P$ $[M + Na]^+$ 440.1386, found 440.1398.



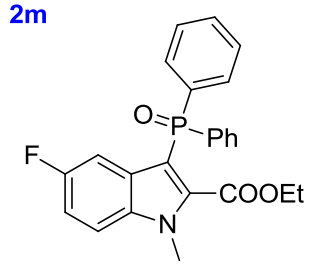
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (91% yield) as a white solid. Mp: 77-79 ^{o}C. $\mathbf{R_f}$ (ethyl acetate: petroleum ether, 2:1): 0.33. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.79 - 7.76 (m, 4H), 7.52 - 7.48 (m, 2H), 7.45 - 7.40 (m, 4H), 7.30 - 7.27 (m, 1H), 7.15 - 7.11 (m, , 1H), 6.97 (s, 1H), 3.93 (s, 3H), 3.72 (q, J = 7.2 Hz, 2H), 2.22 (s, 3H), 1.01 (t, J = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.5, 136.7 (d, $J_{C\text{-}P}$ = 11.0 Hz), 135.0 (d, $J_{C\text{-}P}$ = 17.0 Hz), 134.8 (d, $J_{C\text{-}P}$ = 109.0 Hz), 131.5 (d, $J_{C\text{-}P}$ = 10.0 Hz), 131.3 (d, $J_{C\text{-}P}$ = 3.0 Hz), 129.0 (d, $J_{C\text{-}P}$ = 9.0 Hz), 128.2 (d, $J_{C\text{-}P}$ = 12.0 Hz), 128.1, 126.5, 109.9, 106.7 (d, $J_{C\text{-}P}$ = 120.0 Hz), 61.5, 31.9, 21.3, 13.4. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 22.48. **HRMS** Calcd for $C_{25}H_{24}NNaO_3P$ $[M + Na]^+$ 440.1386, found 440.1398.

2l



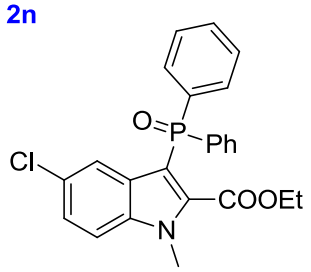
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (96% yield) as a white solid. Mp: 145-147 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.26. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.84 - 7.78 (m, 4H), 7.51 - 7.42 (m, 6H), 7.28 (d, *J* = 9.6 Hz, 1H), 6.93 (dd, *J* = 9.2 Hz, 2.4Hz, 1H), 6.38 (d, *J* = 2.0 Hz, 1H), 3.95 (s, 3H), 3.76 (q, *J* = 7.2 Hz, 2H), 3.42 (s, 3H), 1.02 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.3, 155.2, 135.1 (d, $J_{C\text{-}P}$ = 108.0 Hz), 134.9 (d, $J_{C\text{-}P}$ = 16.0 Hz), 133.5 (d, $J_{C\text{-}P}$ = 11.0 Hz), 131.5, 131.4, 131.2 (d, $J_{C\text{-}P}$ = 3.0 Hz), 129.1 (d, $J_{C\text{-}P}$ = 9.0 Hz), 128.3, 128.2, 116.4, 111.1, 106.7 (d, $J_{C\text{-}P}$ = 120.0 Hz), 102.5, 61.4, 55.1, 32.0, 13.3. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 21.25. **HRMS** Calcd for $C_{25}H_{24}NNaO_4P$ $[M + Na]^+$ 456.1335, found 456.1338.

2m



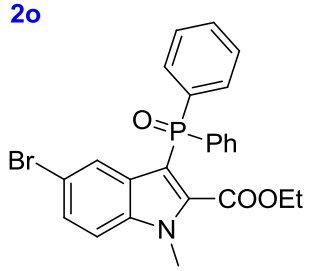
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (87% yield) as a white solid. Mp: 152-154 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.25. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.81 - 7.76 (m, 4H), 7.57 - 7.52 (m, 2H), 7.49 - 7.43 (m, 4H), 7.37 - 7.33 (m, 1H), 7.07 (td, *J* = 8.8 Hz, 2.4Hz, 1H), 6.70 (dd, *J* = 10.0 Hz, 6.4 Hz, 1H), 3.97 (s, 3H), 3.78 (q, *J* = 7.2 Hz, 2H), 1.03 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.2, 158.6 (d, $J_{C\text{-}P}$ = 237.0 Hz), 136.6 (d, $J_{C\text{-}P}$ = 16.0 Hz), 134.8 (d, $J_{C\text{-}P}$ = 11.0 Hz), 133.9 (d, $J_{C\text{-}P}$ = 109.0 Hz), 131.8 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.6, 131.5, 129.1 (d, $J_{C\text{-}P}$ = 11.0 Hz), 129.0, 128.5, 128.4, 113.8 (d, $J_{C\text{-}P}$ = 27.0 Hz), 111.4 (d, $J_{C\text{-}P}$ = 10.0 Hz), 107.6 (d, $J_{C\text{-}P}$ = 25.0 Hz), 106.8 (d, $J_{C\text{-}P}$ = 122.0 Hz), 61.9, 32.2, 13.5. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 22.78. **^{19}F NMR (376 MHz, $CDCl_3$):** δ -119.88. **HRMS** Calcd for $C_{24}H_{21}FNNaO_3P$ $[M + Na]^+$ 444.1135, found 444.1128.

2n

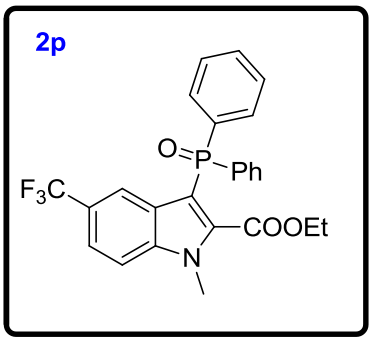


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (90% yield) as a white solid. Mp: 149-151 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.25. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.81 - 7.75 (m, 4H), 7.55 - 7.51 (m, 2H), 7.47 - 7.43 (m, 4H), 7.33 - 7.22 (m, 2H), 7.19 (s, 1H), 3.94 (s, 3H), 3.75 (q, *J* = 7.2 Hz, 2H), 1.03 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.4, 136.8 (d, $J_{C\text{-}P}$ = 11.0 Hz), 136.5 (d, $J_{C\text{-}P}$ = 16.0 Hz), 134.5 (d, $J_{C\text{-}P}$ = 110.0 Hz), 131.9 (d, $J_{C\text{-}P}$ = 2.0 Hz), 131.8 (d, $J_{C\text{-}P}$ = 10.0 Hz), 129.9 (d, $J_{C\text{-}P}$ = 9.0 Hz), 128.7, 128.6, 128.1, 125.5, 122.6, 111.7, 107.5 (d, $J_{C\text{-}P}$ = 119.0 Hz), 62.2, 32.4, 13.7. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 22.03. **HRMS** Calcd for $C_{24}H_{21}ClNNaO_3P$ $[M + Na]^+$ 460.0840, found 460.0828.

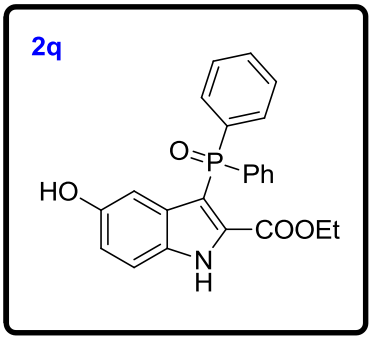
2o



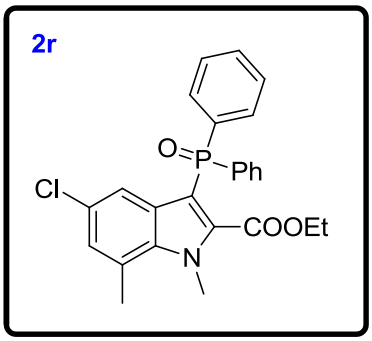
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (81% yield) as a white solid. Mp: 162-164 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.22. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.78 - 7.74 (m, 4H), 7.56 - 7.52 (m, 2H), 7.48 - 7.43 (m, 4H), 7.40 - 7.36 (m, 1H), 7.31 - 7.25 (m, 2H), 3.94 (s, 3H), 3.76 (q, *J* = 7.2 Hz, 2H), 1.03 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.1, 136.8 (d, $J_{C\text{-}P}$ = 10.0 Hz), 136.1 (d, $J_{C\text{-}P}$ = 6.0 Hz), 134.2 (d, $J_{C\text{-}P}$ = 109.0 Hz), 131.7 (d, $J_{C\text{-}P}$ = 2.0 Hz), 131.6, 131.5, 130.2 (d, $J_{C\text{-}P}$ = 8.0 Hz), 128.4, 128.3, 127.8, 125.5, 115.5, 111.7, 107.3 (d, $J_{C\text{-}P}$ = 119.0 Hz), 61.9, 32.1, 13.4. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 21.99. **HRMS** Calcd for $C_{24}H_{21}BrNNaO_3P$ $[M + Na]^+$ 504.0335, found 504.0325.



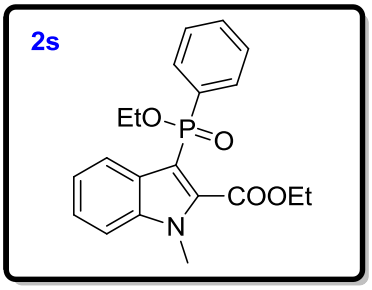
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (81% yield) as a white solid. Mp: 158-160 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.35. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.81 - 7.75 (m, 4H), 7.57 - 7.53 (m, 2H), 7.51 - 7.44 (m, 7H), 7.31 - 7.27 (m, 1H), 4.01 (s, 3H), 3.82 (q, *J* = 7.2 Hz, 2H), 1.04 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.1, 139.4 (d, J_{C-P} = 10.0 Hz), 137.1 (d, J_{C-P} = 15.0 Hz), 134.2 (d, J_{C-P} = 110.0 Hz), 132.0, 131.8 (d, J_{C-P} = 3.0 Hz), 131.6, 131.5, 131.4, 131.3, 128.6, 128.5 (d, J_{C-P} = 5.0 Hz), 128.4, 127.9 (d, J_{C-P} = 9.0 Hz), 124.4 (d, J_{C-P} = 270.0 Hz), 124.3 (q, J_{C-P} = 32.0 Hz), 121.2 (d, J_{C-P} = 4.0 Hz), 110.9, 109.2 (d, J_{C-P} = 118.0 Hz), 62.1, 32.2, 13.4. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 21.35. **HRMS** Calcd for $C_{25}H_{21}F_3NNaO_3P$ $[M + Na]^+$ 494.1103, found 494.1104.



Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (61% yield) as a yellow oil. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.18. **^{1}H NMR (400 MHz, DMSO):** δ 12.55 (brs, 1H), 9.03 (brs, 1H), 7.70 - 7.64 (m, 4H), 7.61 - 7.47 (m, 6H), 7.38 (d, *J* = 7.2 Hz, 1H), 7.03 (d, *J* = 2.0 Hz, 1H), 6.85 (dd, *J* = 8.8 Hz, 2.4 Hz, 1H), 3.77 (q, *J* = 7.2 Hz, 2H), 0.88 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, DMSO):** δ 160.1, 152.2, 135.9, 134.8, 134.8, 131.5 131.3, 131.2, 131.1, 130.9, 130.8, 128.3, 128.2, 116.4, 113.4, 105.8, 60.7, 13.5. **^{31}P NMR (162 MHz, DMSO):** δ 22.06. **HRMS** Calcd for $C_{23}H_{20}NNaO_4P$ $[M + Na]^+$ 428.1022, found 428.1020.

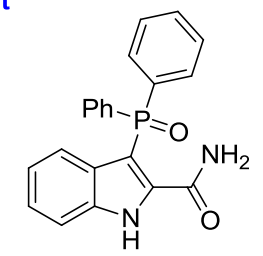


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (89% yield) as a white solid. Mp: 187-189 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.34. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.78 - 7.73 (m, 4H), 7.54 - 7.49 (m, 2H), 7.46 - 7.43 (m, 4H), 7.23 (s, 1H) , 6.94 (s, 1H), 4.08 (s, 3H), 3.72 (q, J = 7.2 Hz, 2H), 2.71 (s, 3H), 1.06 (t, J = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.7, 138.1 (d, $J_{C\text{-}P}$ = 16.0 Hz), 135.4 (d, $J_{C\text{-}P}$ = 10.0 Hz), 133.8 (d, $J_{C\text{-}P}$ = 109.0 Hz), 131.6, 131.5, 130.4 (d, $J_{C\text{-}P}$ = 8.0 Hz), 128.3, 128.2, 127.4, 127.2, 123.5, 119.9, 107.5 (d, $J_{C\text{-}P}$ = 119.0 Hz), 62.0, 34.9, 20.1, 13.7. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 21.98. **HRMS** Calcd for $C_{25}H_{23}ClNNaO_3P$ $[M + Na]^+$ 474.0996, found 474.0988.



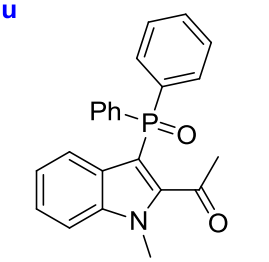
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (69% yield) as a yellow oil. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.33. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.25 (d, J = 8.0 Hz, 1H), 7.88 - 7.82 (m, 2H), 7.48 - 7.36 (m, 5H), 7.30 - 7.25 (m, 1H), 4.30 - 4.22 (m, 1H), 4.19 - 4.05 (m, 1H), 3.94 (s, 3H), 1.37 (t, J = 7.2 Hz, 3H), 1.21 (t, J = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.7, 138.0 (d, $J_{C\text{-}P}$ = 12.0 Hz), 135.6 (d, $J_{C\text{-}P}$ = 20.0 Hz), 133.9 (d, $J_{C\text{-}P}$ = 146.0 Hz), 131.4 (d, $J_{C\text{-}P}$ = 2.0 Hz), 130.7, 130.6, 128.3, 128.2, 127.9, 124.7, 123.1, 122.3, 110.3, 106.6 (d, $J_{C\text{-}P}$ = 157.0 Hz), 61.8, 60.5 (d, $J_{C\text{-}P}$ = 6.0 Hz), 16.4 (d, $J_{C\text{-}P}$ = 6.0 Hz), 13.6. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 29.06. **HRMS** Calcd for $C_{20}H_{22}NNaO_4P$ $[M + Na]^+$ 394.1179, found 394.1180.

2t

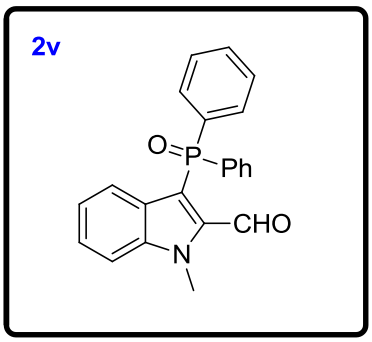


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (22% yield) as a yellow oil. $\mathbf{R_f}$ (ethyl acetate: petroleum ether, 4:1): 0.35. **^{1}H NMR (400 MHz, DMSO):** δ12.62 (brs, 1H), 10.56 (brs, 1H), 7.82 (brs, 1H), 7.69 - 7.64 (m, 2H), 7.61 - 7.53 (m, 9H), 7.21 - 7.17 (m, 1H), 6.87 - 6.82 (m, 1H), 6.37 (d, J = 8.4 Hz, 1H). **^{13}C NMR (100 MHz, DMSO):** δ 161.6, 139.6 (d, $J_{C\text{-}P}$ = 16.0 Hz), 135.9 (d, $J_{C\text{-}P}$ = 12.0 Hz),133.9, 133.0 (d, $J_{C\text{-}P}$ = 2.0 Hz), 132.8, 131.8, 131.7, 129.5, 129.4, 129.3 129.2, 124.4, 121.9, 121.3, 113.8, 101.7 (d, $J_{C\text{-}P}$ = 119.0 Hz). **^{31}P NMR (162 MHz, DMSO):** δ 26.10. **HRMS** Calcd for $C_{21}H_{18}N_2O_2P$ $[M + H]^+$ 361.1100, found 361.1105.

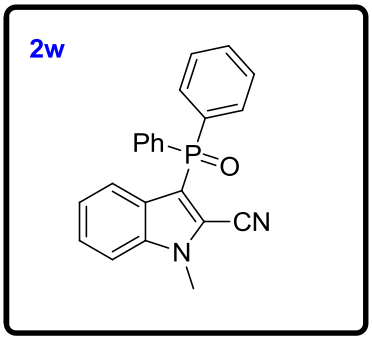
2u



Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (95% yield) as a white solid. Mp: 191-193 ^{o}C. $\mathbf{R_f}$ (ethyl acetate: petroleum ether, 2:1): 0.35. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.78 - 7.72 (m, 4H), 7.51 - 7.48 (m, 2H), 7.43 - 7.36 (m, 5H), 7.28 - 7.24 (m, 1H), 6.96 (t, J = 7.6 Hz, 1H), 6.79 (t, J = 8.4 Hz, 1H), 3.75 (s, 3H), 2.61 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 198.1, 145.3 (d, $J_{C\text{-}P}$ = 17.0 Hz), 137.5 (d, $J_{C\text{-}P}$ = 11.0 Hz), 133.9 (d, $J_{C\text{-}P}$ = 109.0 Hz), 131.7 (d, $J_{C\text{-}P}$ = 2.0 Hz), 131.6 (d, $J_{C\text{-}P}$ = 11.0 Hz), 128.4 (d, $J_{C\text{-}P}$ = 13.0 Hz), 127.6 (d, $J_{C\text{-}P}$ = 10.0 Hz), 123.9, 121.8, 110.4, 104.2 (d, $J_{C\text{-}P}$ = 120.0 Hz), 33.5, 31.3. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 21.36. **HRMS** Calcd for $C_{23}H_{20}NNaO_2P$ $[M + Na]^+$ 396.1124, found 396.1123.

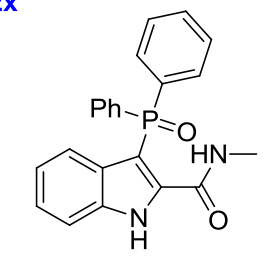


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (96% yield) as a yellow oil. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.28. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 10.83 (brs, 1H), 7.77 - 7.71 (m, 4H), 7.60 - 7.55 (m, 2H), 7.50 - 7.45 (m, 5H), 7.42 - 7.37 (m, 1H), 7.03 - 6.98 (m, 1H), 6.80 (d, J = 8.4 Hz, 1H), 4.19 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 186.8, 140.0 (d, $J_{C\text{-}P}$ = 15.0 Hz), 139.4 (d, $J_{C\text{-}P}$ = 12.0 Hz), 133.8 (d, $J_{C\text{-}P}$ = 108.0 Hz), 132.2 (d, $J_{C\text{-}P}$ = 3.0 Hz), 132.1, 132.0, 131.8, 131.7, 129.0, 128.8, 128.7, 127.9, 127.8, 126.6, 122.8, 122.4, 114.8 (d, $J_{C\text{-}P}$ = 115.0 Hz), 111.0, 32.8. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 23.19. **HRMS** Calcd for $C_{22}H_{18}NNaO_2P$ $[M + Na]^+$ 382.0967, found 382.0957.



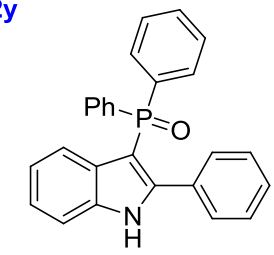
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (93% yield) as a white solid. Mp: 201-203 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.35. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.85 - 7.74 (m, 4H), 7.68 - 7.61 (m, 1H), 7.58 - 7.54 (m, 2H), 7.50 - 7.44 (m, 4H), 7.41 - 7.38 (m, 2H), 7.18 - 7.12 (m, 1H), 3.88 (d, J = 6.0 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 138.2 (d, $J_{C\text{-}P}$ = 11.0 Hz), 132.6, 132.3 (d, $J_{C\text{-}P}$ = 3.0 Hz), 128.5 (d, $J_{C\text{-}P}$ = 12.0 Hz), 126.4, 122.9, 122.8, 110.9 (d, $J_{C\text{-}P}$ = 1.0 Hz), 110.4, 76.7 (d, $J_{C\text{-}P}$ = 3.0 Hz), 31.8. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 19.71. **HRMS** Calcd for $C_{22}H_{17}N_2NaOP$ $[M + Na]^+$ 379.0971, found 379.0958.

2x



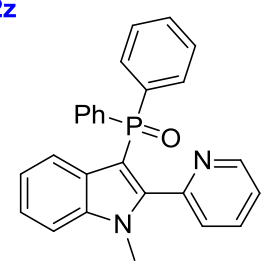
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (86% yield) as a yeollow oil. **R_f** (ethyl acetate: petroleum ether, 4:1): 0.38. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 11.43 (brs, 1H), 11.29 (brs, 1H), 7.73 - 7.67 (m, 4H), 7.62 - 7.55 (m, 3H), 7.48 - 7.44 (m, 4H), 7.26 - 7.21 (m, 1H), 6.92 - 6.87 (m, 1H), 6.57 (d, *J* = 8.0 Hz, 1H), 3.07 (d, *J* = 4.0 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 160.0, 139.2 (d, $J_{C\text{-}P}$ = 15.0 Hz), 133.3, 132.3 (d, $J_{C\text{-}P}$ = 3.0 Hz), 132.1, 132.0, 131.9, 131.8, 129.5 (d, $J_{C\text{-}P}$ = 12.0 Hz), 128.8, 128.7, 124.3, 121.7, 121.6, 112.8, 101.1 (d, $J_{C\text{-}P}$ = 121.0 Hz), 26.9. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 28.39. **HRMS** Calcd for $C_{22}H_{20}N_2O_2P$ $[M + H]^+$ 375.1257, found 375.1263.

2y



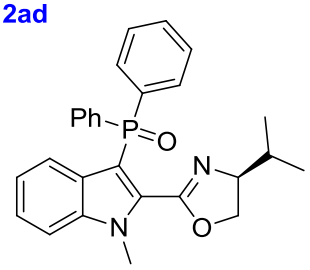
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (78% yield) as a white solid. Mp: 173-175 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.50. **^{1}H NMR (400 MHz, d_6-DMSO):** δ 12.31 (brs, 1H), 8.33 (s, 1H), 7.65 - 7.59 (m, 4H), 7.54 - 7.48 (m, 3H), 7.43 - 7.38 (m, 2H), 7.34 - 7.30 (m, 4H), 7.20 - 7.10 (m, 4H), 6.82 (t, *J* = 7.6 Hz, 1H), 6.52 (d, *J* = 8.4 Hz, 1H). **^{13}C NMR (100 MHz, d_6-DMSO):** δ 147.1 (d, $J_{C\text{-}P}$ = 18.0 Hz), 137.0 (d, $J_{C\text{-}P}$ = 12.0 Hz), 135.5 (d, $J_{C\text{-}P}$ = 106.0 Hz), 132.0, 131.9, 131, 131.5, 130.2, 130.0 (d, $J_{C\text{-}P}$ = 5.0 Hz), 128.8, 128.6 (d, $J_{C\text{-}P}$ = 12.0 Hz), 128.0, 122.5, 120.7 (d, $J_{C\text{-}P}$ = 3.0 Hz), 101.0 (d, $J_{C\text{-}P}$ = 124.0 Hz), 79.6. **^{31}P NMR (162 MHz, d_6-DMSO):** δ 16.87. **HRMS** Calcd for $C_{26}H_{21}NOP$ $[M + H]^+$ 394.1355, found 394.1362.

2z

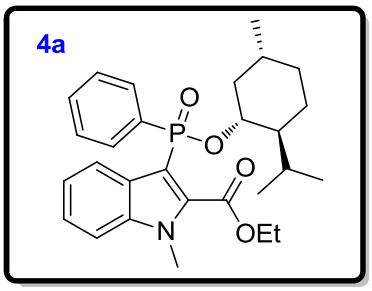


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (73% yield) as a yellow oil. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.39. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.50 (d, *J* = 4.4 Hz, 1H), 7.71 - 7.65 (m, 4H), 7.52 (d, *J* = 7.6 Hz, 1H), 7.42 (d, *J* = 8.4 Hz, 1H), 7.38 - 7.31 (m, 3H), 7.30 - 7.22 (m, 5H), 7.05 - 7.01 (m, 1H), 6.99 - 6.97 (m, 2H), 3.70 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 149.8, 148.8, 145.8 (d, $J_{C\text{-}P}$ = 18.0 Hz), 137.9 (d, $J_{C\text{-}P}$ = 11.0 Hz), 135.7, 134.5 (d, $J_{C\text{-}P}$ = 107.0 Hz), 131.7, 131.6, 131.0 (d, $J_{C\text{-}P}$ = 3.0 Hz), 129.0 (d, $J_{C\text{-}P}$ = 9.0 Hz), 128.2 (d, $J_{C\text{-}P}$ = 2.0 Hz), 128.1, 123.1, 121.9, 110.0, 103.4 (d, $J_{C\text{-}P}$ = 125.0 Hz), 31.3. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 18.72. **HRMS** Calcd for $C_{26}H_{22}N_2OP$ $[M + H]^+$ 409.1464, found 409.1471.

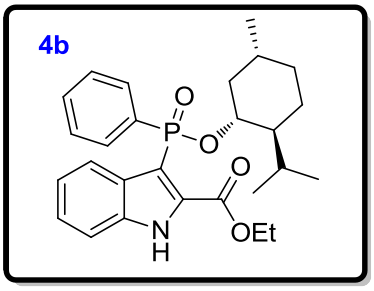
2ad



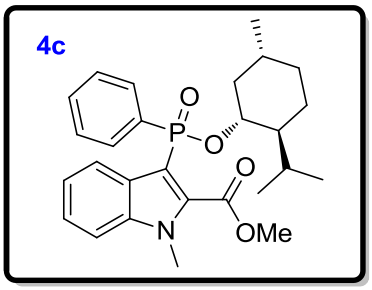
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 2: 1) give the product (77% yield) as a yellow oil. **R_f** (ethyl acetate: petroleum ether, 2:1): 0.45. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.88 - 7.82 (m, 4H), 7.51 - 7.39 (m, 7H), 7.36 - 7.26 (m, 2H), 7.03 (t, *J* = 8.0 Hz, 1H), 3.97 (s, 3H), 3.58 - 3.42 (m, 3H), 1.61 - 1.48 (m, 1H), 0.95 (d, *J* = 4.0 Hz, 1H), 0.82 (t, *J* = 8.0 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 156.5, 138.3 (d, $J_{C\text{-}P}$ = 11.0 Hz), 134.7 (d, $J_{C\text{-}P}$ = 108.0 Hz), 132.8 (d, $J_{C\text{-}P}$ = 16.0 Hz),131.7, 131.6 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.5, 131.2 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.2 (d, $J_{C\text{-}P}$ = 2.0 Hz), 129.1 (d, $J_{C\text{-}P}$ = 9.0 Hz), 128.3 (d, $J_{C\text{-}P}$ = 5.0 Hz), 128.2 (d, $J_{C\text{-}P}$ = 5.0 Hz), 124.2, 122.8, 121.7, 110.2, 106.7 (d, $J_{C\text{-}P}$ = 122.0 Hz), 73.3, 69.9, 32.7, 31.7, 19.1, 18.7. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 20.32.



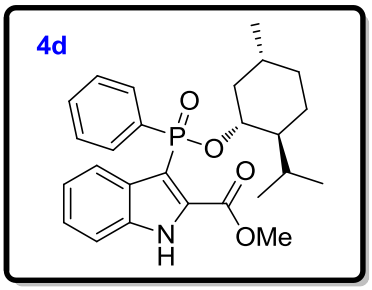
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (88% yield) as a white solid. Mp: 65-67 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 1:2): 0.37. **$[\alpha]_D^{16}$** = +13.0 (*c* 1.00, $CHCl_3$). **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.35 (d, *J* = 8.4 Hz, 1H), 7.84 - 7.78 (m, 2H), 7.46 - 7.35 (m, 5H), 7.28 - 7.23 (m, 1H), 4.44 - 4.40 (m, 1H), 4.25 - 4.09(m, 2H), 3.96 (s, 3H), 2.13 - 2.08 (m, 1H), 1.92 (d, *J* = 12 Hz, 1H), 1.66 - 1.61 (m, 2H), 1.49 - 1.42 (t, *J* = 11.2 Hz, 1H), 1.37 - 1.31 (m, 1H), 1.24 - 1.14 (m, 4H), 1.00 - 0.83 (m, 2H), 0.81 (d, *J* = 6.8 Hz, 3H), 0.79 (d, *J* = 6.8 Hz, 3H), 0.51 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.6, 138.1(d, $J_{C\text{-}P}$ = 12.0 Hz), 135.8 (d, $J_{C\text{-}P}$ = 143.0 Hz), 134.6 (d, $J_{C\text{-}P}$ = 20.0 Hz), 131.1 (d, $J_{C\text{-}P}$ = 3.0 Hz), 130.6 (d, $J_{C\text{-}P}$ = 11.0 Hz), 128.6 (d, $J_{C\text{-}P}$ = 8.0 Hz), 127.9 (d, $J_{C\text{-}P}$ = 13.0 Hz), 124.7, 123.8, 122.0, 110.1, 108.0 (d, $J_{C\text{-}P}$ = 159.0 Hz), 76.3 (d, $J_{C\text{-}P}$ = 7.0 Hz), 61,5, 49.0 (d, $J_{C\text{-}P}$ = 5.0 Hz), 43.4, 34.1, 31.8, 31.5, 25.2, 22.7, 22.0, 21.1, 15.3, 13.7. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 28.14. **HRMS** Calcd for $C_{28}H_{37}NO_4P$ $[M + H]^+$ 482.2455, found 482.2458.



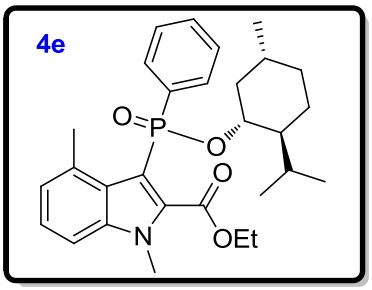
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (65% yield) as a white solid. Mp: 232-234 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 1:2): 0.25. **$[\alpha]_D^{16}$** = +2.0 (*c* 1.00, $CHCl_3$). **^{1}H NMR (400 MHz, $CDCl_3$):** δ 10.61 (brs, 1H), 8.58 (d, *J* = 8.0 Hz, 1H), 7.83 - 7.77 (m, 2H), 7.44 - 7.40 (m, 1H), 7.35 - 7.30 (m, 3H), 7.27 - 7.23 (m, 1H), 4.49 - 4.40 (m, 1H), 4.07 - 4.03 (m, 2H), 2.18 - 2.12 (m, 1H), 1.92 (d, *J* = 10.4 Hz, 1H), 1.63 - 1.61 (m, 2H), 1.51 (t, *J* = 11.2 Hz, 1H), 1.31 - 1.16 (m, 2H), 1.04 - 0.77 (m, 11H), 0.51 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 160.5, 136.7 (d, $J_{C\text{-}P}$ = 145.0 Hz), 133.6 (d, $J_{C\text{-}P}$ = 12.0 Hz), 134.6, 131.4, 131.2(d, $J_{C\text{-}P}$ = 3.0 Hz), 130.8, 130.7, 130.6, 130.5, 127.9, 127.8, 125.7, 124.6 122.3, 111.8, 109.6 (d, $J_{C\text{-}P}$ = 156.0 Hz), 76.4 (d, $J_{C\text{-}P}$ = 7.0 Hz), 61,6, 49.0 (d, $J_{C\text{-}P}$ = 5.0 Hz), 43.6, 34.1, 31.5, 25.4, 22.7, 22.0, 21.1, 15.3, 13.9. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 29.26. **HRMS** Calcd for $C_{27}H_{35}NO_4P$ $[M + H]^+$ 468.2298, found 468.2291.



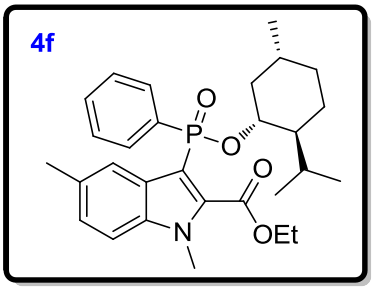
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (86% yield) as a white solid. Mp: 60-62 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 1:2): 0.22. **$[\alpha]_D^{16}$** = +85.0 (*c* 1.00, $CHCl_3$). **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.89 - 7.78 (m, 3H), 7.49 - 7.44 (m, 1H), 7.48 - 7.37 (m, 3H), 7.34 - 7.26 (m, 1H), 4.34 - 4.25 (m, 1H), 3.73 (s, 3H), 2.80 (s, 3H), 2.06 - 1.98 (m, 2H), 1.63 - 1.60 (m, 2H), 1.42 (t, *J* = 11.2 Hz, 1H), 1.35 - 1.30 (m, 1H), 1.26 - 1.20 (m, 1H), 0.99 - 0.82 (m, 2H), 0.81 - 0.77 (m, 3H), 0.73 (d, *J* = 7.2 Hz, 3H), 0.46 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.8, 138.1 (d, $J_{C\text{-}P}$ = 13.0 Hz), 135.6 (d, $J_{C\text{-}P}$ = 143.0 Hz), 134.0 (d, $J_{C\text{-}P}$ = 21.0 Hz), 132.3, 131.1 (d, $J_{C\text{-}P}$ = 3.0 Hz), 130.8, 130.5, 130.4, 128.7, 128.3, 127.9, 127.8, 124.8, 123.7, 122.1, 110.2, 108.8 (d, $J_{C\text{-}P}$ = 159.0 Hz), 76.2 (d, $J_{C\text{-}P}$ = 7.0 Hz), 68.0, 49.0 (d, $J_{C\text{-}P}$ = 6.0 Hz), 43.3, 34.0, 31.7, 31.4, 25.2, 22.9, 21.9, 21.0, 15.3. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 28.01. **HRMS** Calcd for $C_{27}H_{35}NO_4P$ $[M + H]^+$ 468.2298, found 468.2292.



Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (67% yield) as a white solid. Mp: 227-229 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 1:2): 0.19. **$[\alpha]_D^{16}$** = -110.0 (*c* 1.00, $CHCl_3$). **^{1}H NMR (400 MHz, $CDCl_3$):** δ 10.49 (brs, 1H), 8.56 - 8.53 (m, 1H), 7.85 - 7.79 (m, 2H), 7.51 - 7.40 (m, 1H), 7.36 - 7.31 (m, 3H), 7.28 - 7.23 (m, 1H), 4.44 - 4.41 (m, 1H), 3.54 (s, 3H), 2.23 - 2.10 (m, 1H), 1.87 - 1.84 (m, 1H), 1.67 - 1.51 (m, 3H), 1.26 - 1.17 (m, 2H), 1.02 - 0.87 (m, 2H), 0.82 (d, *J* = 6.8 Hz, 3H), 0.76 (d, *J* = 6.4 Hz, 3H), 0.57 (d, *J* = 6.4 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 160.8, 136.2, 136.0, 134.7, 131.6, 131.4, 130.8, 130.6, 130.4, 130.3, 127.8, 127.7, 125.4, 124.2 122.1, 112.3, 109.1 (d, $J_{C\text{-}P}$ = 156.0 Hz), 76.4 (d, $J_{C\text{-}P}$ = 6.0 Hz), 61,5, 48.9 (d, $J_{C\text{-}P}$ = 5.0 Hz), 43.5, 34.0, 31.4, 25.3, 22.6, 21.9, 21.0, 15.2. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 28.54. **HRMS** Calcd for $C_{27}H_{35}NO_4P$ $[M + H]^+$ 468.2298, found 468.2291. **HRMS** Calcd for $C_{26}H_{33}NO_4P$ $[M + H]^+$ 454.2142, found 454.2147.

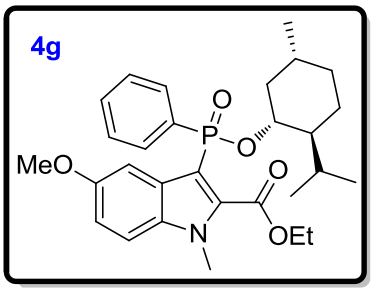


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (80% yield) as a yellow oil. **R_f** (ethyl acetate: petroleum ether, 1:2): 0.39. **$[\alpha]_D^{16}$** = -8.0 (*c* 1.00, $CHCl_3$). **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.92 - 7.86 (m, 2H), 7.47 - 7.35 (m, 3H), 7.19 - 7.14 (m, 2H), 7.40 - 7.37 (m, 2H), 7.34 (s, 2H), 4.49 - 4.39 (m, 1H), 4.24 - 4.07 (m, 2H), 3.96 (s, 3H), 2.14 - 2.07 (m, 1H), 1.85 (d, *J* = 12 Hz, 1H), 1.68 - 1.61 (m, 2H), 1.51 - 1.45 (t, *J* = 11.2 Hz, 1H), 1.37 - 1.32 (m, 1H), 1.21 - 1.11 (m, 4H), 1.01 - 0.84 (m, 2H), 0.80 (d, *J* = 6.4 Hz, 3H), 0.71 (d, *J* = 7.2 Hz, 3H), 0.51 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 163.9, 140.2 (d, $J_{C\text{-}P}$ = 16.0 Hz), 137.8 (d, $J_{C\text{-}P}$ = 12.0 Hz), 135.3 (d, $J_{C\text{-}P}$ = 138.0 Hz), 132.9, 131.5 (d, $J_{C\text{-}P}$ = 11.0 Hz), 131.4 (d, $J_{C\text{-}P}$ = 3.0 Hz), 128.1, 128.0, 126.8 (d, $J_{C\text{-}P}$ = 11.0 Hz), 123.8, 123.6, 107.5, 103.5 (d, $J_{C\text{-}P}$ = 158.0 Hz), 76.1 (d, $J_{C\text{-}P}$ = 7.0 Hz), 62.3, 48.8 (d, $J_{C\text{-}P}$ = 5.0 Hz), 43.2, 34.2, 31.5, 31.2, 25.0, 22.6, 22.0, 21.7, 21.0, 15.0, 14.0. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 27.48. **HRMS** Calcd for $C_{29}H_{39}NO_4P$ $[M + H]^+$ 496.2611, found 496.2615.

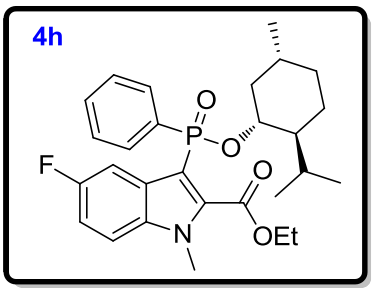


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (85% yield) as a yellow oil. Rf (ethyl acetate: petroleum ether, 1:2): 0.45. [α]D16 = +10.0 (c 1.00, CHCl3). 1H NMR (400 MHz, CDCl3): δ 8.20 (s, 1H), 7.83 - 7.77 (m, 2H), 7.45 - 7.37 (m, 3H), 7.30 - 7.27 (m, 1H), 7.20 (d, J = 8.4 Hz, 1H), 4.48 - 4.39 (m, 1H), 4.22 - 4.03 (m, 2H), 3.93 (s, 3H), 2.48 (s, 3H), 2.17 - 2.10 (m, 1H), 1.95 (d, J = 12.0 Hz, 1H), 1.64 (d, J = 10.8 Hz, 1H), 1.49 - 1.36 (m, 2H), 1.26 - 1.16 (m, 1H), 1.21 - 1.11 (m, 4H), 1.05 - 0.85 (m, 2H), 0.83 (d, J = 7.2 Hz, 3H), 0.81 (d, J = 6.4 Hz, 3H), 0.55 (d, J = 7.2 Hz, 3H). 13C NMR (100 MHz, CDCl3): δ 161.5, 136.6, 136.6 (d, JC-P = 12.0 Hz), 136.0 (d, JC-P = 144.0 Hz), 134.1(d, JC-P = 21.0 Hz), 131.5, 131.0 (d, JC-P = 3.0 Hz), 130.4 (d, JC-P = 11.0 Hz), 128.9 (d, JC-P = 9.0 Hz), 127.9, 127.7, 126.7, 123.1 109.7, 107.4 (d, JC-P = 159.0 Hz), 76.2 (d, JC-P = 6.0 Hz), 61,3, 48.9 (d, JC-P = 6.0 Hz), 43.4, 34.0, 31.8, 31.4, 25.2, 22.6, 22.0, 21.6, 21.0, 15.2, 13.7. 31P NMR

(162 MHz, CDCl3): δ 28.88. HRMS Calcd for C29H39NO4P [M + H]+ 496.2611, found 496.2615.

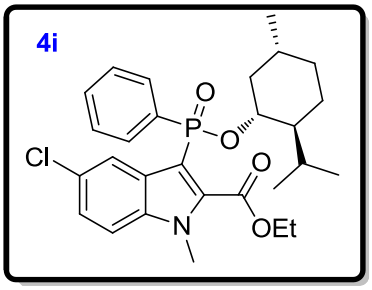


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (84% yield) as a white solid. Mp: 58-60 °C. **R_f** (ethyl acetate: petroleum ether, 1:2): 0.34. **$[\alpha]_D^{16}$** = +24.0 (*c* 1.00, $CHCl_3$). **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.89 (d, *J* = 2.4 Hz, 1H), 7.82 - 7.76 (m, 2H), 7.45 - 7.42 (m, 1H), 7.41 - 7.36 (m, 2H), 7.33 - 7.27 (m, 1H), 7.07 - 7.04 (m, 1H), 4.44 - 4.40 (m, 1H), 4.20 - 4.14 (m, 1H), 4.07 - 4.01 (m, 1H), 3.96 (s, 3H), 3.87 (s, 3H), 2.14 - 2.12 (m, 1H), 1.92 (d, *J* = 12.0 Hz, 1H), 1.64 (d, *J* = 11.6 Hz, 2H), 1.44 - 1.34 (m, 3H), 1.23 - 1.16 (m, 1H), 1.12 (t, *J* = 7.2 Hz, 3H), 1.00 - 0.85 (m, 2H), 0.83 - 0.79 (m, 3H), 0.55 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.4, 155.6, 136.0 (d, $J_{C\text{-}P}$ = 144.0 Hz), 134.0 (d, $J_{C\text{-}P}$ = 20.0 Hz), 133.5 (d, $J_{C\text{-}P}$ = 13.0 Hz), 131.3 (d, $J_{C\text{-}P}$ = 3.0 Hz), 130.4 (d, $J_{C\text{-}P}$ = 11.0 Hz), 129.4 (d, $J_{C\text{-}P}$ = 9.0 Hz), 128.0, 127.8, 116.4, 111.0 107.4 (d, $J_{C\text{-}P}$ = 159.0 Hz), 103.9, 76.1 (d, $J_{C\text{-}P}$ = 7.0 Hz), 61,3, 55.6, 49.2 (d, $J_{C\text{-}P}$ = 5 .0 Hz), 43.7, 34.1, 32.1, 31.5, 25.3, 22.6, 22.0, 21.1, 15.2, 13.7. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 29.11. **HRMS** Calcd for $C_{29}H_{39}NO_5P$ $[M + H]^+$ 512.1560, found 512.2555.

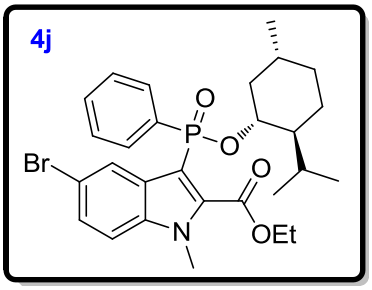


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (73% yield) as a yellow solid. Mp: 94-96 °C. **R_f** (ethyl acetate: petroleum ether, 1:2): 0.33. **$[\alpha]_D^{16}$** = +26.0 (*c* 1.00, $CHCl_3$). **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.05 (dd, *J* = 10.0 Hz, 2.4 Hz, 1H), 7.82 - 7.76 (m, 2H), 7.47 - 7.33 (m, 3H), 7.15 (td, *J* = 8.8 Hz, 2.4 Hz, 1H), 4.44 - 4.40 (m, 1H), 4.23 - 4.08 (m, 2H), 3.96 (s, 3H), 2.12 - 2.07 (m, 1H), 1.88 - 1.84 (m, 1H), 1.68 - 1.62 (m, 2H), 1.48 - 1.44 (m, 1H), 1.35 - 1.33 (m, 1H), 1.22 - 1.13 (m, 4H), 1.01 - 0.84 (m, 2H), 0.83 - 0.79 (m, 6H), 0.55 (d, *J* = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.2, 159.0 (d, $J_{C\text{-}P}$ = 236.0 Hz), 135.6 (d, $J_{C\text{-}P}$ = 20.0 Hz), 135.5 (d,

$J_{C\text{-}P}$ = 143.0 Hz), 134.8 (d, $J_{C\text{-}P}$ = 11.0 Hz), 131.3 (d, $J_{C\text{-}P}$ = 3.0 Hz), 130.6 (d, $J_{C\text{-}P}$ = 11.0 Hz), 129.1, 128.9 (d, $J_{C\text{-}P}$ = 8.0 Hz), 128.1, 127.9, 114.4 (d, $J_{C\text{-}P}$ = 27.0 Hz), 111.1 (d, $J_{C\text{-}P}$ = 10.0 Hz), 108.2 (d, $J_{C\text{-}P}$ = 164.0 Hz), 108.5 (d, $J_{C\text{-}P}$ = 26.0 Hz), 76.5 (d, $J_{C\text{-}P}$ = 7.0 Hz), 61,7, 49.0 (d, $J_{C\text{-}P}$ = 5.0 Hz), 43.4, 34.1, 32.2, 31.5, 25.3, 22.7, 22.0, 21.0, 15.3, 13.7. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 27.98. **^{19}F NMR (376 MHz, $CDCl_3$):** δ -120.53. **HRMS** Calcd for $C_{28}H_{36}FNO_4P$ $[M + H]^+$ 500.2360, found 500.2367.

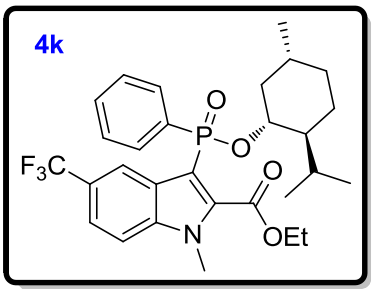


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (78% yield) as a yellow solid. Mp: 89-91 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 1:2): 0.33. **$[\alpha]_D^{16}$** = +17.0 (*c* 1.00, $CHCl_3$). **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.39 (s, 1H), 7.82 - 7.76 (m, 2H), 7.48 - 7.40 (m, 1H), 7.40 - 7.37 (m, 2H), 7.34 (s, 2H), 4.49 - 4.39 (m, 1H), 4.24 - 4.07 (m, 2H), 3.96 (s, 3H), 2.14 - 2.07 (m, 1H), 1.85 (d, *J* = 12 Hz, 1H), 1.68 - 1.61 (m, 2H), 1.51 - 1.45 (t, *J* = 11.2 Hz, 1H), 1.37 - 1.32 (m, 1H), 1.21 - 1.11 (m, 4H), 1.05 - 0.85 (m, 2H), 0.83 (d, *J* = 7.2 Hz, 3H), 0.81 (d, *J* = 6.4 Hz, 3H), 0.55 (d, *J* = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.2, 136.6, 136.5, 135.6 (d, $J_{C\text{-}P}$ = 144.0 Hz), 135.4 (d, $J_{C\text{-}P}$ = 21.0 Hz), 134.9, 131.3 (d, $J_{C\text{-}P}$ = 2.0 Hz), 130.6 (d, $J_{C\text{-}P}$ = 11.0 Hz), 129.4 (d, $J_{C\text{-}P}$ = 8.0 Hz), 128.1 (d, $J_{C\text{-}P}$ = 14.0 Hz), 128.0, 125.4, 123.2 111.3, 108.1 (d, $J_{C\text{-}P}$ = 159.0 Hz), 76.6 (d, $J_{C\text{-}P}$ = 7.0 Hz), 61,7, 49.0 (d, $J_{C\text{-}P}$ = 5.0 Hz), 43.4, 34.1, 33.2, 31.5, 25.4, 22.7, 22.0, 21.1, 15.4, 13.8. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 27.75. **HRMS** Calcd for $C_{28}H_{36}ClNO_4P$ $[M + H]^+$ 516.2065, found 516.2072.

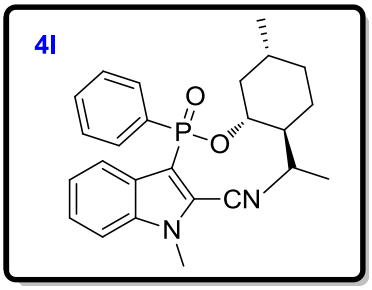


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (83% yield) as a white solid. Mp: 62-64 ^{o}C. **R_f** (ethyl acetate: petroleum ether, 1:2): 0.31. **$[\alpha]_D^{16}$** = +37.0 (*c* 1.00, $CHCl_3$). **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.57 (d, *J* = 1.6 Hz, 1H), 7.82 - 7.76 (m, 2H), 7.48 - 7.37 (m, 4H), 7.29 - 7.26 (m, 1H), 4.46 - 4.42 (m, 1H), 4.22 - 4.09 (m,

2H), 3.94 (s, 3H), 2.14 - 2.09 (m, 1H), 1.86 (d, *J* = 12.0 Hz, 1H), 1.69 - 1.61 (m, 2H), 1.52 - 1.32 (m, 2H), 1.21 - 1.12 (m, 4H), 1.01 - 0.88 (m, 2H), 0.85 (d, *J* = 7.2 Hz, 3H), 0.81 (d, *J* = 6.8 Hz, 3H), 0.56 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.1, 136.7 (d, J_{C-P} = 12.0 Hz), 135.5 (d, J_{C-P} = 144.0 Hz), 135.2 (d, J_{C-P} = 11.0 Hz), 131.3 (d, J_{C-P} = 2.0 Hz), 130.5 (d, J_{C-P} = 11.0 Hz), 129.9 (d, J_{C-P} = 8.0 Hz), 128.0, 127.9, 127.8, 126.2 115.6, 108.0 (d, J_{C-P} = 158.0 Hz), 76.5 (d, J_{C-P} = 6.0 Hz), 61,7, 48.9 (d, J_{C-P} = 6.0 Hz), 43.4, 34.0, 32.1, 31.5, 25.4, 22.7, 21.9, 21.0, 15.3, 13.7. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 27.75. **HRMS** Calcd for $C_{28}H_{36}BrNO_4P$ $[M + H]^+$ 560.1560, found 560.1567.

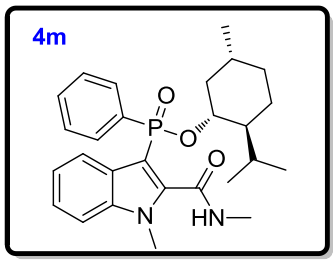


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (79% yield) as a yellow solid. Mp: 101-103 °C. **R_f** (ethyl acetate: petroleum ether, 1:2): 0.43. **$[\alpha]_D^{16}$** = +34.0 (*c* 1.00, $CHCl_3$). **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.73 (s, 1H), 7.83 - 7.77 (m, 2H), 7.60 (d, *J* = 8.8 Hz, 1H), 7.52 - 7.45 (m, 2H), 7.43 - 7.38 (m, 2H), 4.48 - 4.44 (m, 1H), 4.25 - 4.16 (m, 2H), 4.00 (s, 3H), 2.13-2.08 (m, 1H), 1.80 (d, *J* = 12.0 Hz, 1H), 1.69 - 1.61 (m, 2H), 1.50 - 1.46 (m, 1H), 1.35 - 1.27 (m, 2H), 1.18 (t, (d, *J* = 7.2 Hz, 4H), 1.01 - 0.85 (m, 2H), 0.81 (d, *J* = 7.2 Hz, 3H), 0.80 (d, *J* = 6.4 Hz, 3H), 0.58 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.0, 139.2 (d, J_{C-P} = 11.0 Hz), 136.2 (d, J_{C-P} = 20.0 Hz), 135.2 (d, J_{C-P} = 144.0 Hz), 131.4 (d, J_{C-P} = 3.0 Hz), 130.5 (d, J_{C-P} = 11.0 Hz), 128.1, 127.9, 127.5 (d, J_{C-P} = 8.0 Hz), 124.8 (d, J_{C-P} = 270.0 Hz), 124.4 (q, J_{C-P} = 32.0 Hz) 121.7 (d, J_{C-P} = 5.0 Hz), 121.3 (d, J_{C-P} = 3.0 Hz), 110.8, 109.6 (d, J_{C-P} = 158.0 Hz), 76.6 (d, J_{C-P} = 7.0 Hz), 61,8, 48.9 (d, J_{C-P} = 5.0 Hz), 43.3, 34.0, 32.2, 31.5, 25.3, 22.6, 21.9, 20.8, 15.3, 13.7. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 27.52. **^{19}F NMR (376 MHz, $CDCl_3$):** δ -60.76. **HRMS** Calcd for $C_{29}H_{36}F_3NO_4P$ $[M + H]^+$ 550.2329, found 550.2336..

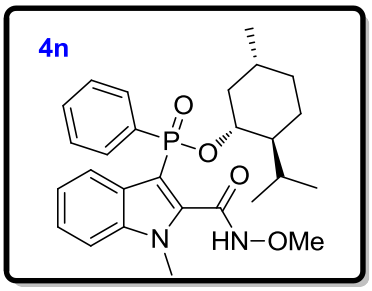


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (79% yield) as a white

solid. Mp: 119-121 °C. **R_f** (ethyl acetate: petroleum ether, 1:2): 0.45. **$[\alpha]_D^{16}$** = +22.0 (*c* 1.00, $CHCl_3$). **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.03 (d, *J* = 8 Hz, 1H), 7.97 - 7.91 (m, 2H), 7.53 - 7.48 (m, 1H), 7.46 - 7.40 (m, 3H), 7.37 - 7.34 (m, 1H), 7.25 - 7.22 (m, 1H), 4.46 - 4.37 (m, 1H), 3.95 (s, 3H), 2.30 - 2.20 (m, 1H), 1.97 - 1.93 (m, 1H), 1.70 - 1.53 (m, 3H), 1.35 - 1.27 (m, 2H), 1.04 - 0.94 (m, 2H), 0.88 (d, *J* = 7.2 Hz, 3H), 0.82 (d, *J* = 5.6 Hz, 3H), 0.64 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 139.0 (d, $J_{C\text{-}P}$ = 1.0 Hz), 133.2 (d, $J_{C\text{-}P}$ = 142.0 Hz), 132.1, 132.0, 131.4 (d, $J_{C\text{-}P}$ = 10.0 Hz), 128.0 (d, $J_{C\text{-}P}$ = 13 .0 Hz), 127.4 (d, $J_{C\text{-}P}$ = 10.0 Hz), 126.3, 122.9 (d, $J_{C\text{-}P}$ = 13.0 Hz), 115.5 (d, $J_{C\text{-}P}$ = 154.0 Hz), 114.6, 114.5, 112.2, 110.2, 77.7 (d, $J_{C\text{-}P}$ = 7.0 Hz), 48.5 (d, $J_{C\text{-}P}$ = 5.0 Hz), 43.2, 33.9, 31.9, 31.6, 25.5, 22.7, 21.9, 21.0, 15.4. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 21.75. **HRMS** Calcd for $C_{26}H_{32}N_2O_2P$ $[M + H]^+$ 435.2196, found 435.2190.

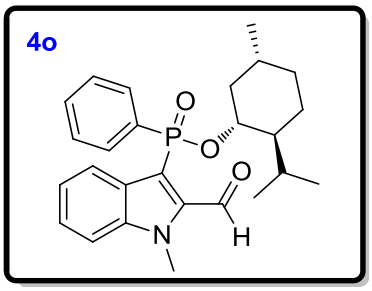


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (52% yield) as a yellow oil. **R_f** (ethyl acetate: petroleum ether, 1:2): 0.28. **$[\alpha]_D^{16}$** = +13.0 (*c* 1.00, $CHCl_3$). **^{1}H NMR (400 MHz, $CDCl_3$):** δ 11.55 (br, 1H), 11.36 (br, 1H), 7.81 - 7.75 (m, 2H), 7.62 - 7.59 (m, 1H), 7.50 - 7.37 (m, 4H), 7.29 - 7.24 (m, 1H), 7.05 (t, *J* = 7.6 Hz, 1H), 4.29 - 4.24 (m, 1H), 3.15 (d, *J* = 4.0Hz, 3H), 2.28 (s, 1H), 1.90 - 1.86 (m, 1H), 1.65 - 1.59 (m, 3H), 1.50 - 1.40 (m, 2H), 0.91 (d, *J* = 5.6 Hz, 3H), 0.68 (d, *J* = 6.8 Hz, 3H), 0.24 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.2, 137.6 (d, $J_{C\text{-}P}$ = 21.0 Hz), 135.1(d, $J_{C\text{-}P}$ = 10.0 Hz), 133.9 (d, $J_{C\text{-}P}$ = 148.0 Hz), 131.9 (d, $J_{C\text{-}P}$ = 3.0 Hz), 130.6, 130.5, 129.4, 129.2, 128.5, 128.4, 124.4, 122.1, 121.6, 112.5, 101.6 (d, $J_{C\text{-}P}$ = 153.0 Hz), 78.3 (d, $J_{C\text{-}P}$ = 8.0 Hz), 48.7 (d, $J_{C\text{-}P}$ = 6.0 Hz), 43.8, 33.9, 31.6, 29.7, 26.8, 25.3, 22.6, 22.0, 20.9, 14.9. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 31.16. **HRMS** Calcd for $C_{27}H_{36}N_2O_3P$ $[M + H]^+$ 453.2302, found 453.2297.

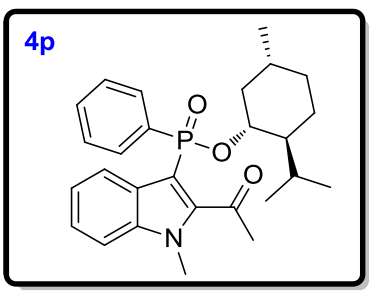


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (73% yield) as a

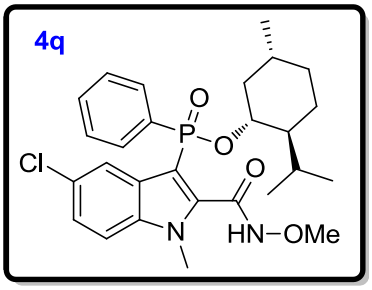
yellow oil. $\mathbf{R_f}$ (ethyl acetate: petroleum ether, 1:1): 0.25. $\mathbf{[\alpha]_D^{16}}$ = -61.0 (*c* 1.00, $CHCl_3$). **^{1}H NMR (400 MHz, $CDCl_3$):** δ 14.32 (br, 1H), 11.23 (br, 1H), 7.80 (dd, *J* = 13.6 Hz, 7.2 Hz, 2H), 7.62 (d, *J* = 8.4 Hz, 1H), 7.52 - 7.47 (m, 1H), 7.44 - 7.37 (m, 3H), 7.30 - 7.25 (m, 1H), 7.05 (t, *J* = 7.6 Hz, 1H), 4.30 - 4.21 (m, 1H), 3.98 (s, 3H), 2.35-2.31 (m, 1H), 1.88 - 1.80 (m, 1H), 1.65 - 1.59 (m, 2H), 1.50 - 1.41 (m, 3H), 0.92 - 0.86 (m, 5H), 0.68 (d, *J* = 6.8 Hz, 3H), 0.19 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 158.3, 135.5 (d, $J_{C\text{-}P}$ = 12.0 Hz), 135.1, 134.9, 133.2 (d, $J_{C\text{-}P}$ = 150.0 Hz), 132.2 (d, $J_{C\text{-}P}$ = 2.0 Hz), 130.7 (d, $J_{C\text{-}P}$ = 11.0 Hz), 129.0 (d, $J_{C\text{-}P}$ = 10.0 Hz), 128.5 (d, $J_{C\text{-}P}$ = 13.0 Hz), 124.8, 121.9 (d, $J_{C\text{-}P}$ = 10.0 Hz), 112.6, 101.7 (d, $J_{C\text{-}P}$ = 151.0 Hz), 78.7 (d, $J_{C\text{-}P}$ = 6.0 Hz), 64.1 (d, $J_{C\text{-}P}$ = 5.0 Hz), 48.7 (d, $J_{C\text{-}P}$ = 7.0 Hz), 43.8, 33.9, 31.7, 25.2, 22.6, 22.0, 20.9, 14.8. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 30.67. **HRMS** Calcd for $C_{27}H_{36}N_2O_3P$ $[M + H]^+$ 453.2302, found 453.2297.



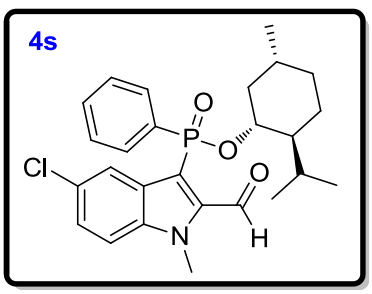
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (82% yield) as a yellow solid. Mp: 173-175 oC. Rf (ethyl acetate: petroleum ether, 1:2): 0.59. [α]D16 = -58.0 (c 1.00, CHCl3). 1H NMR (400 MHz, CDCl3): δ 10.86 (s, 1H), 7.68 (d, J = 8.4 Hz, 1H), 7.88 - 7.82 (m, 2H), 7.49 - 7.38 (m, 5H), 7.24 - 7.20 (m, 1H), 4.47 - 4.37 (m, 1H), 4.14 (s, 3H), 2.10 - 1.97 (m, 2H), 1.64 (d, J = 10.8 Hz, 2H), 1.53 - 1.47 (m, 1H), 1.36 - 1.25 (m, 2H), 1.02 - 0.92 (m, 2H), 0.90 (d, J = 6.4 Hz, 3H), 0.85 (d, J = 6.0 Hz, 3H), 0.76 (d, J = 7.2 Hz, 3H). 13C NMR (100 MHz, CDCl3): δ 186.6(d, JC-P = 18.0 Hz), 139.4 (d, JC-P = 12.0 Hz), 138.2 (d, JC-P = 22.0 Hz), 134.9 (d, JC-P = 142.0 Hz), 131.9, 130.7 (d, JC-P = 11.0 Hz), 128.4 (d, JC-P = 13.0 Hz), 127.6 (d, JC-P = 9.0 Hz), 126.9, 123.6, 122.4, 115.6 (d, JC-P = 153.0 Hz), 110.6, 77.5, 49.0 (d, JC-P = 5.0 Hz), 43.6, 34.0, 32.5, 31.6, 25.5, 22.7, 22.0, 21.0, 15.2. 31P NMR (162 MHz, CDCl3): δ 25.95. HRMS Calcd for C26H33NO3P [M + H]+ 438.2193, found 438.2199.



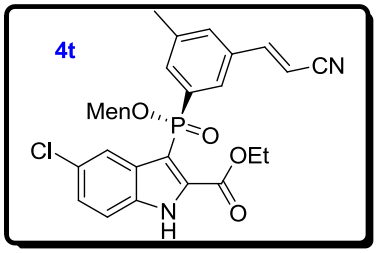
Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (82% yield) as a yellow oil. Rf (ethyl acetate: petroleum ether, 1:2): 0.55. [α]D16 = +20.0 (c 1.00, CHCl3). 1H NMR (400 MHz, CDCl3): δ 7.89 - 7.83 (m, 2H), 7.80 (d, J = 8 Hz, 1H), 7.49 - 7.44 (m, 1H), 7.43 - 7.29 (m, 4H), 7.20 - 7.15 (m, 1H), 4.34 - 4.24 (m, 1H), 3.74 (s, 3H), 2.80 (s, 3H), 2.05 - 1.97 (m, 2H), 1.61 (d, J = 10 Hz, 2H), 1.45 - 1.39 (m, 1H), 1.35 - 1.20 (m, 2H), 0.99 - 0.82 (m, 2H), 0.80 (d, J = 2.8 Hz, 3H), 0.74 (d, J = 7.2 Hz, 3H), 0.45 (d, J = 6.8 Hz, 3H). 13C NMR (100 MHz, CDCl3): δ 198.4, 144.5 (d, JC-P = 22.0 Hz), 137.5 (d, JC-P = 11.0 Hz), 134.6 (d, JC-P = 141.0 Hz), 131.6 (d, JC-P = 3.0 Hz), 131.0 (d, JC-P = 11.0 Hz), 128.2 (d, JC-P = 13.0 Hz), 127.5 (d, JC-P = 10.0 Hz), 124.1, 122.4, 121.9, 110.2, 104.3 (d, JC-P = 159.0 Hz), 77.0, 49.0 (d, JC-P = 6.0 Hz), 43.4, 34.0, 33.4, 31.5, 31.3, 25.5, 22.7, 22.0, 21.0, 15.1. 31P NMR (162 MHz, CDCl3): δ 26.08. HRMS Calcd for C27H35NO3P [M + H]+ 452.2349, found 452.2354.



Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (68% yield) as a yellow oil. **R_f** (ethyl acetate: petroleum ether, 1:1): 0.27. **$[\alpha]_D^{16}$** = -42.0 (*c* 1.00, $CHCl_3$). **^{1}H NMR (400 MHz, $CDCl_3$):** δ 14.24 (br, 1H), 11.15(br, 1H), 7.81 - 7.74 (m, 2H), 7.56 - 7.51 (m, 2H), 7.48 - 7.41 (m, 3H), 7.25 - 7.24 (m, 1H), 4.31 - 4.22 (m, 1H), 3.97 (s, 3H), 2.30 - 2.26 (m, 1H), 1.92 - 1.83 (m, 1H), 1.66 - 1.62 (m, 2H), 1.54 - 1.51 (m, 1H), 1.49 - 1.37 (m, 2H), 0.98-0.85 (m, 5H), 0.75 (d, *J* = 6.8 Hz, 3H), 0.28 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 157.9, 136.0 (d, J_{C-P} = 20.0 Hz), 133.8 (d, J_{C-P} = 12.0 Hz), 132.9 (d, J_{C-P} = 150.0 Hz), 132.5 (d, J_{C-P} = 3.0 Hz), 130.6 (d, J_{C-P} = 12.0 Hz), 129.9 (d, J_{C-P} = 11.0 Hz), 128.7 (d, J_{C-P} = 13.0 Hz), 127.8, 125.5, 121.3, 113.8, 102.0 (d, J_{C-P} = 152.0 Hz), 79.2 (d, J_{C-P} = 7.0 Hz), 64.2, 48.7 (d, J_{C-P} = 7.0 Hz), 43.7, 33.9, 31.7, 25.5, 22.7, 22.0, 20.9, 14.9. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 29.95. **HRMS** Calcd for $C_{27}H_{35}ClN_2O_4P$ $[M + H]^+$ 503.1861, found 503.1855.

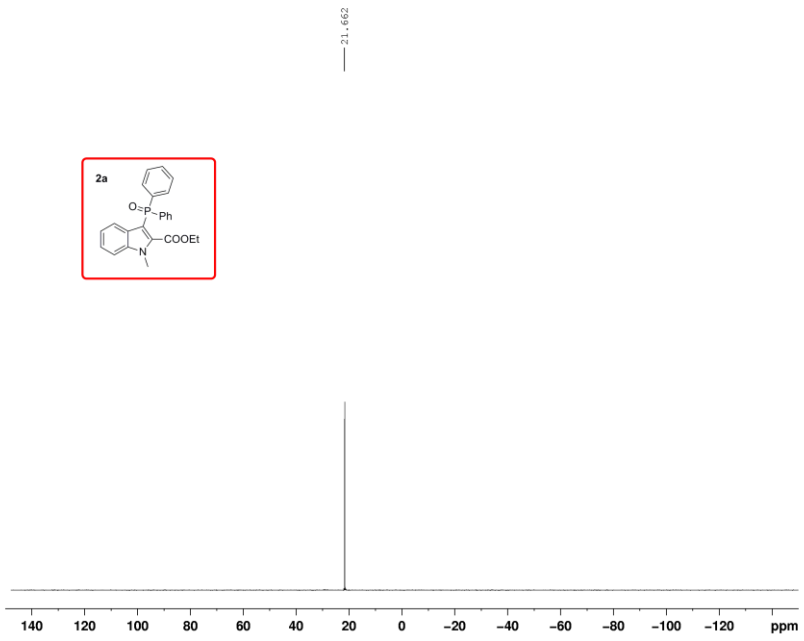


Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (76% yield) as a yellow solid. Mp: 177-179 ^{o}C. $\mathbf{R_f}$ (ethyl acetate: petroleum ether, 1:2): 0.51. $\mathbf{[\alpha]_D^{16}}$ = +17.0 (*c* 1.00, $CHCl_3$). **^{1}H NMR (400 MHz, $CDCl_3$):** δ 10.78 (s, 1H), 8.03 (d, *J* = 1.2 Hz, 1H), 7.88 - 7.80 (m, 2H), 7.52 - 7.47 (m, 1H), 7.45 - 7.34 (m, 4H), 4.50 - 4.41 (m, 1H), 4.11 (s, 3H), 2.10 - 2.00 (m, 2H), 1.70 - 1.65 (m, 2H), 1.57 - 1.50 (m, 1H), 1.42 - 1.23 (m, 2H), 1.05 - 0.87 (m, 2H), 0.86 - 0.80 (m, 6H), 0.85 (d, *J* = 6.0 Hz, 3H), 0.76 (d, *J* = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 186.2, 138.7 (d, $J_{C\text{-}P}$ = 21.0 Hz), 137.8 (d, $J_{C\text{-}P}$ = 12.0 Hz), 134.6 (d, $J_{C\text{-}P}$ = 142.0 Hz), 132.1 (d, $J_{C\text{-}P}$ = 2.0 Hz), 130.7 (d, $J_{C\text{-}P}$ = 11.0 Hz), 128.7, 128.5, 128.4, 127.5, 122.8, 115.2 (d, $J_{C\text{-}P}$ = 153.0 Hz), 111.9, 78.0 (d, $J_{C\text{-}P}$ = 7.0 Hz), 49.0 (d, $J_{C\text{-}P}$ = 6.0 Hz), 43.6, 34.0, 32.8, 31.6, 25.7, 22.8, 21.9, 21.0, 15.3. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 25.44. **HRMS** Calcd for $C_{26}H_{32}ClNO_3P$ $[M + H]^+$ 472.1803, found 472.1796.



Prepared according to the general procedure. Flash column chromatography on a silica gel (ethyl acetate: petroleum ether, 1: 2) give the product (56% yield) as a yellow oil. $\mathbf{R_f}$ (ethyl acetate: petroleum ether, 1:2): 0.20. $\mathbf{[\alpha]_D^{16}}$= +25.0 (*c* 1.00, $CHCl_3$). **^{1}H NMR (400 MHz, $CDCl_3$):** δ 9.78, (s, 1H), 8.58 (d, *J* = 1.6 Hz, 1H), 7.72 (d, *J* = 13.6 Hz, 1H), 7.63 (d, *J* = 13.6 Hz, 1H), 7.45 - 7.34 (m, 4H),, 5.91 (d, *J* = 16.8 Hz, 1H), 4.52 – 4.42 (m, 1H), 4.21 - 4.11(m, 2H), 2.37 (s, 3H), 2.37 (s, 3H), 2.14 - 2.07 (m, 1H), 1.85 (d, *J* = 11.6 Hz, 1H), 1.70 - 1.52 (m, 3H), 1.41 – 1.17 (m, 2H), 1.14 (t, *J* = 11.6 Hz, 3H), 1.05 - 0.81 (m, 8H), 0.53 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$):** δ 159.9, 149.7, 138.8 (d, $J_{C\text{-}P}$ = 14.0 Hz), 135.9, 134.5, 134.2 (d, $J_{C\text{-}P}$ = 11.0 Hz), 133.3, 133.1 (d, $J_{C\text{-}P}$ = 10.0 Hz), 132.9, 130.7 (d, $J_{C\text{-}P}$ = 7.0 Hz), 129.7, 128.4, 127.5 (d, $J_{C\text{-}P}$ = 11.0 Hz), 126.4, 123.5, 117.8, 113.3, 97.3, 77.6 (d, $J_{C\text{-}P}$ = 6.0 Hz), 61,9, 49.1 (d, $J_{C\text{-}P}$ = 7.0 Hz), 43.7, 33.9, 31.9, 31.5, 25.6, 22.7, 21.9, 21.2, 21.1, 15.1, 13.8. **^{31}P NMR (162 MHz, $CDCl_3$):** δ 27.02. **HRMS** Calcd for $C_{27}H_{35}NO_3P$ $[M + H]^+$ 567.2174, found 567.2179.

5. Charts of products.

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PROCNO                       1
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DS                           4
SWH                  64102.563 Hz
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RG                        2050
DW                       7.800 usec
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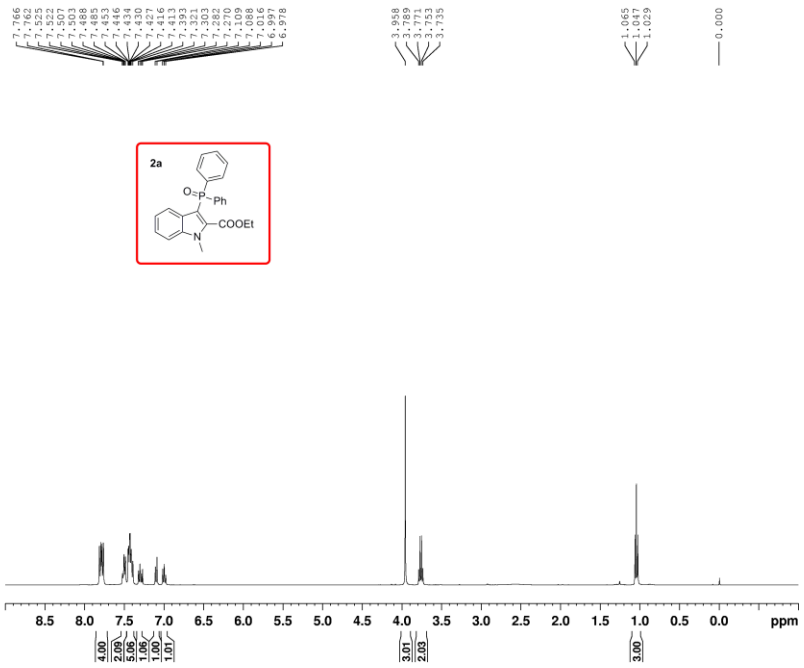
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          1

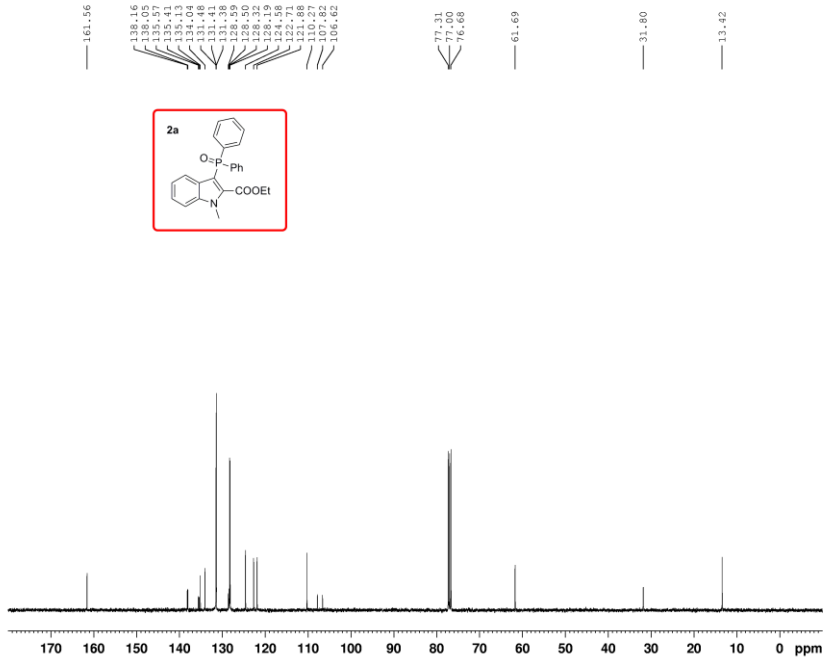
======== CHANNEL f1 ========
NUC1                        31P
P1                        14.85 usec
PL1                        2.80 dB
PL1W                13.31439304 W
SFO1                161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     90.00 usec
PL2                       -2.00 dB
PL12                      15.50 dB
PL13                      15.50 dB
PL2W                18.19349861 W
PL12W               0.32353121 W
PL13W               0.32353121 W
SFO2                400.1316005 MHz
SI                       32768
SF                  161.9755930 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```




```
NAME            zhouax120308-p
EXPNO                       10
PROCNO                       1
Date_             20120308
Time                     19.33
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                  zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                  8223.685 Hz
FIDRES               0.125483 Hz
AQ                   3.9846387 sec
RG                        80.6
DW                       60.800 usec
DE                       6.50 usec
TE                      291.6 K
D1                   1.00000000 sec
TD0                          1

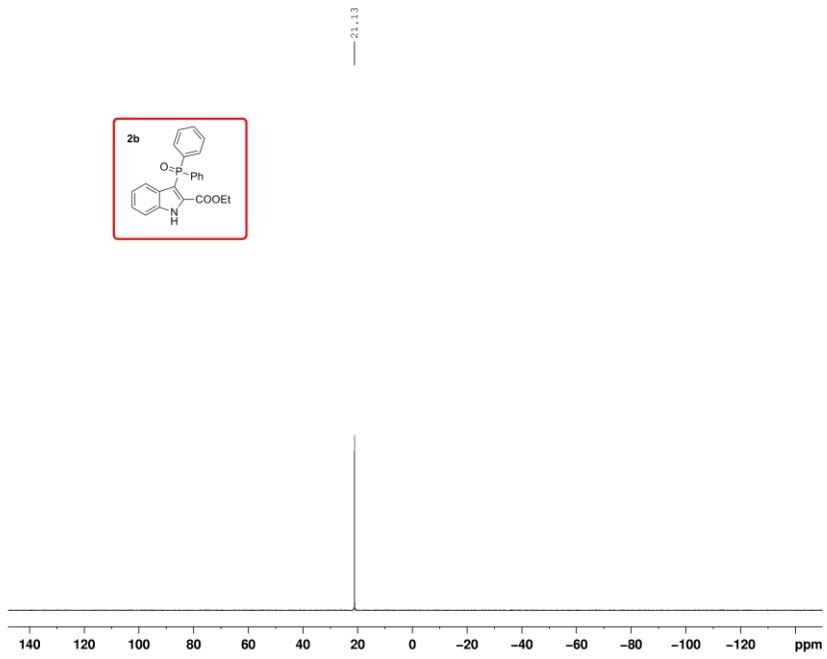
======== CHANNEL f1 ========
NUC1                        1H
P1                        12.00 usec
PL1                       -3.00 dB
PL1W                22.90425682 W
SFO1                400.1324710 MHz
SI                       32768
SF                  400.1300012 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```




```
NAME          zhouax120308-p
EXPNO                    12
PROCNO                    1
Date_              20120308
Time                  19.47
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      120
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                     2050
DW                   20.800 usec
DE                    6.50 usec
TE                   292.4 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                     9.60 usec
PL1                   -2.00 dB
PL1W           57.32743073 W
SFO1          100.6228298 MHz

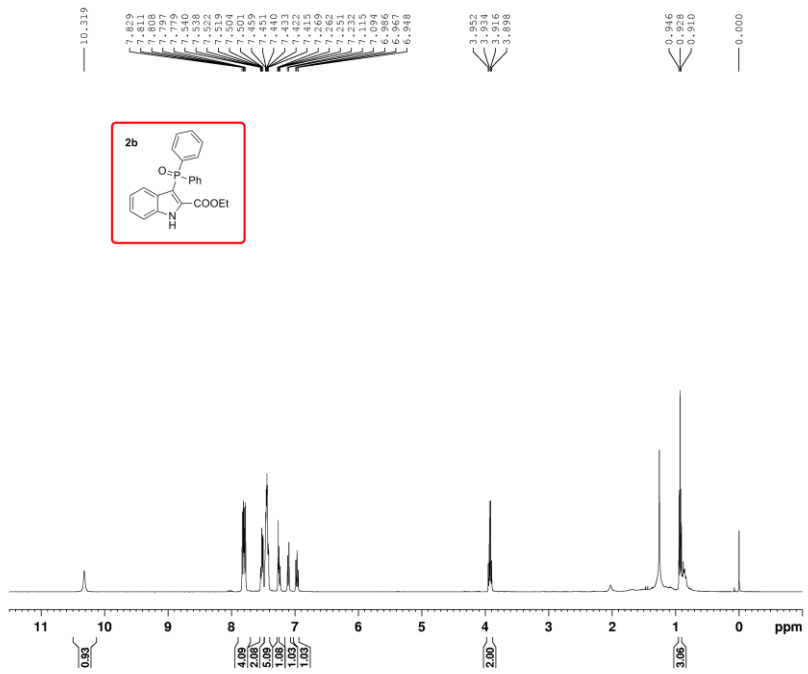
======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                   -2.00 dB
PL12                  15.50 dB
PL13                  15.50 dB
PL2W           18.19369861 W
PL12W           0.32353121 W
PL13W           0.32353121 W
SFO2          400.1316005 MHz
SI                    32768
SF            100.6127817 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```




```
NAME          zhouax120405-P-Et-H
EXPNO                    11
PROCNO                    1
Date_              20120405
Time                  15.18
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT              DMSO
NS                      16
DS                        4
SWH               64102.563 Hz
FIDRES             0.978127 Hz
AQ                 0.5112308 sec
RG                     2050
DW                    7.800 usec
DE                    6.50 usec
TE                   295.5 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

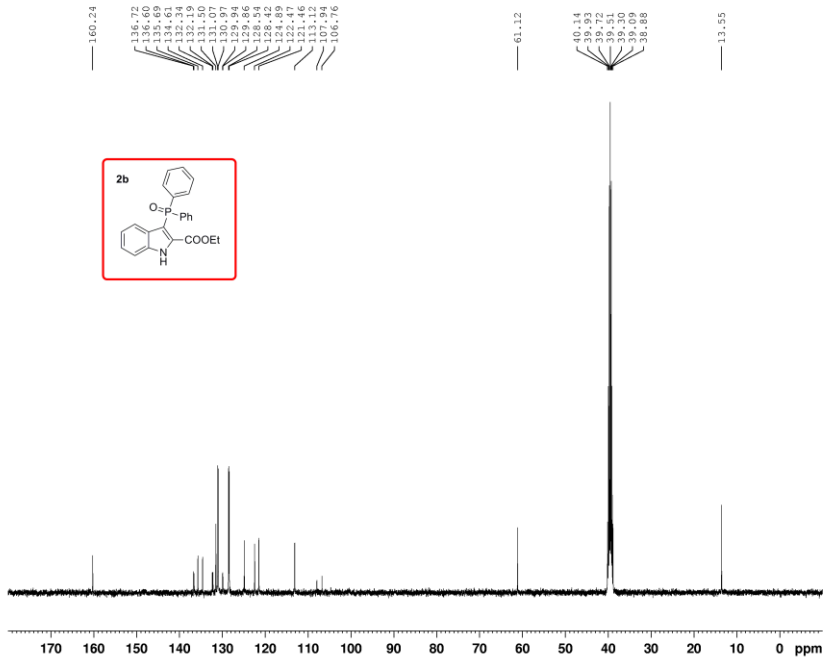
======== CHANNEL f1 ========
NUC1                    31P
P1                    10.80 usec
PL1                    0.00 dB
PL1W           26.12361908 W
SFO1          161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   -2.10 dB
PL12                  13.90 dB
PL13                  13.90 dB
PL2W           17.72106263 W
PL12W           0.44513249 W
PL13W           0.44513249 W
SFO2          400.1316005 MHz
SI                    32768
SF            161.9755930 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```




```
NAME          zhouax120304-2
EXPNO                 10
PROCNO                 1
Date_           20120304
Time               20.32
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9846387 sec
RG                   181
DW               60.800 usec
DE                6.50 usec
TE                292.8 K
D1             1.00000000 sec
TD0                    1

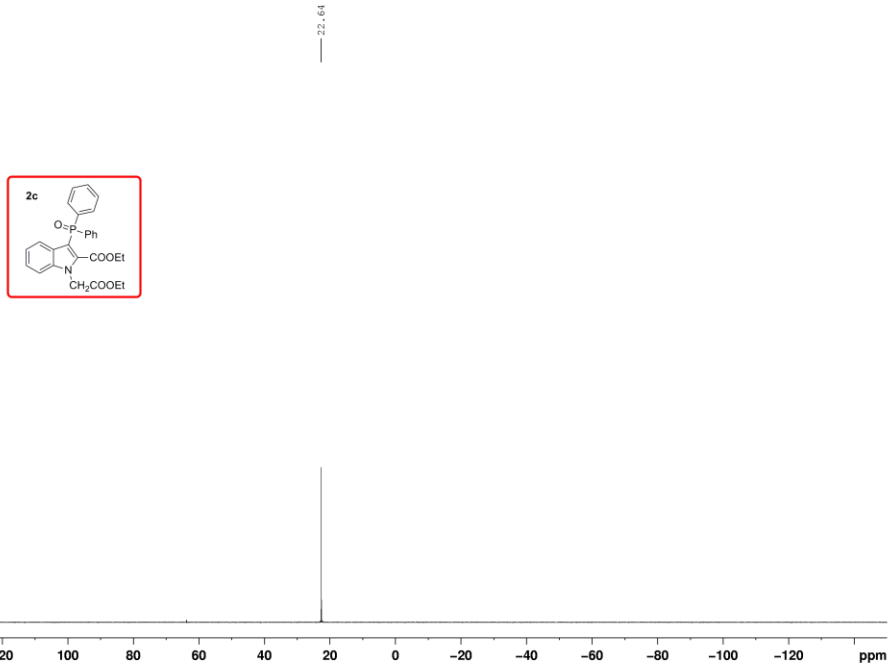
======== CHANNEL f1 ========
NUC1                  1H
P1                12.00 usec
PL1               -3.00 dB
PL1W        22.90425682 W
SFO1        400.1324710 MHz
SI                 32768
SF          400.1300048 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```



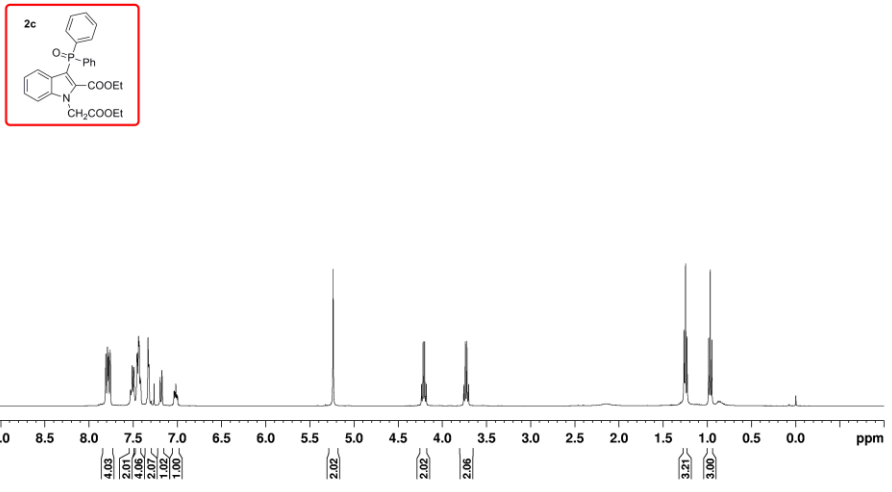

```
NAME      zhouax120405-P-Et-H
EXPNO                 12
PROCNO                 1
Date_           20120405
Time               15.34
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT              DMSO
NS                    240
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                    64
DW               20.800 usec
DE                6.50 usec
TE                296.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

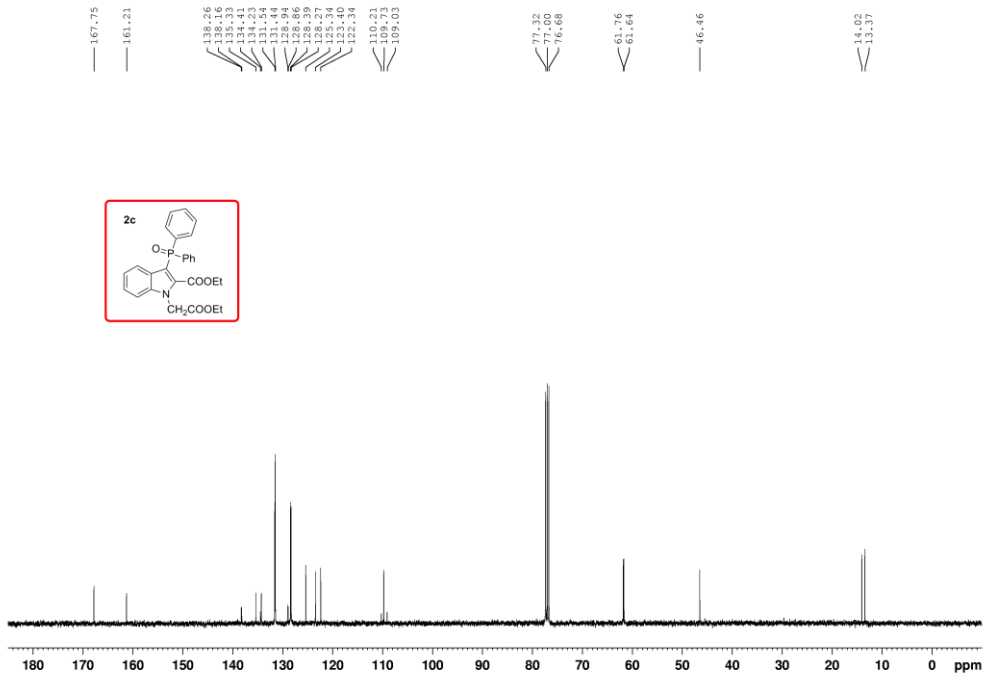
======== CHANNEL f1 ========
NUC1                  13C
P1                 9.70 usec
PL1               -2.00 dB
PL1W        56.13311005 W
SFO1        100.6228298 MHz

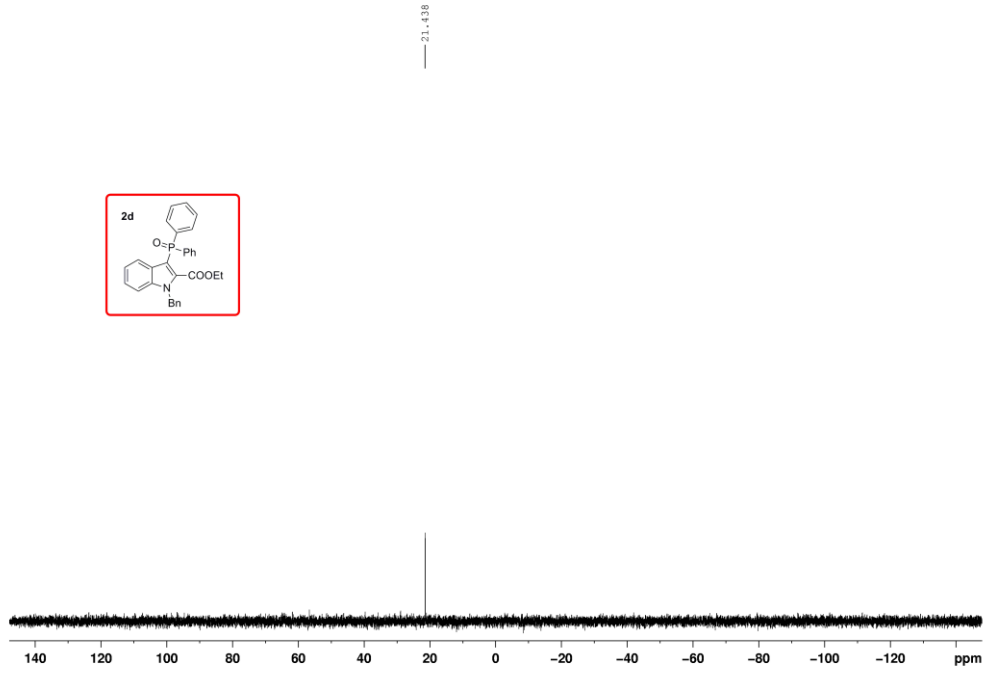
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -2.10 dB
PL12              13.90 dB
PL13              13.90 dB
PL2W        17.72106263 W
PL12W        0.44513249 W
PL13W        0.44513249 W
SFO2        400.1316005 MHz
SI                 32768
SF          100.6128050 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```

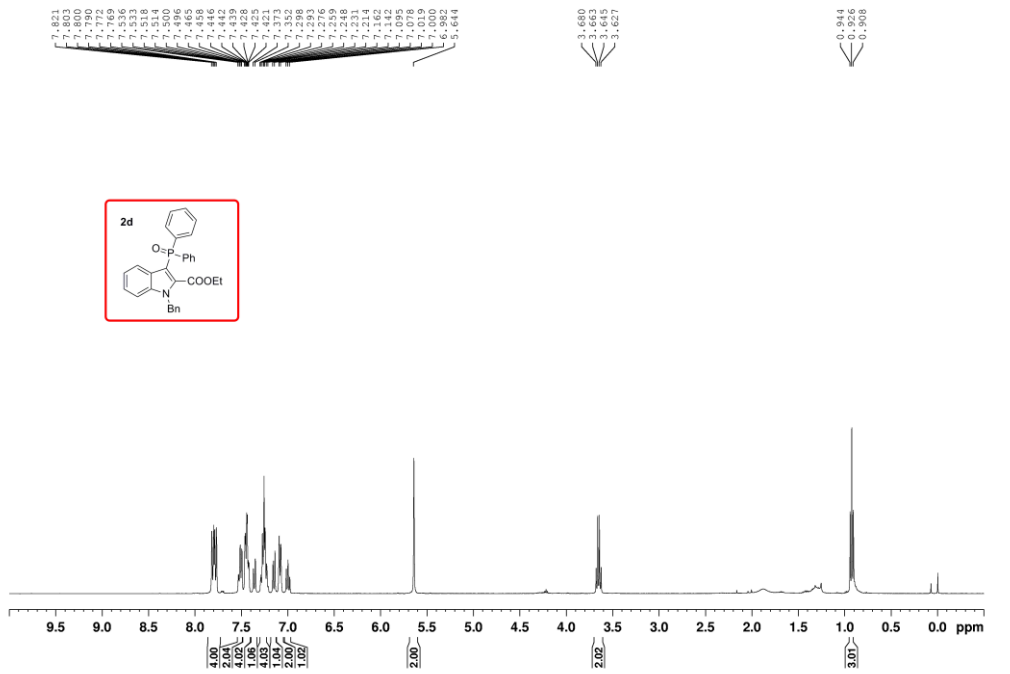

22.64
2c
O
P
Ph
COOEt
N
CH2COOEt
140
120
100
80
60
40
20
0
−20
−40
−60
−80
−100
−120
ppm

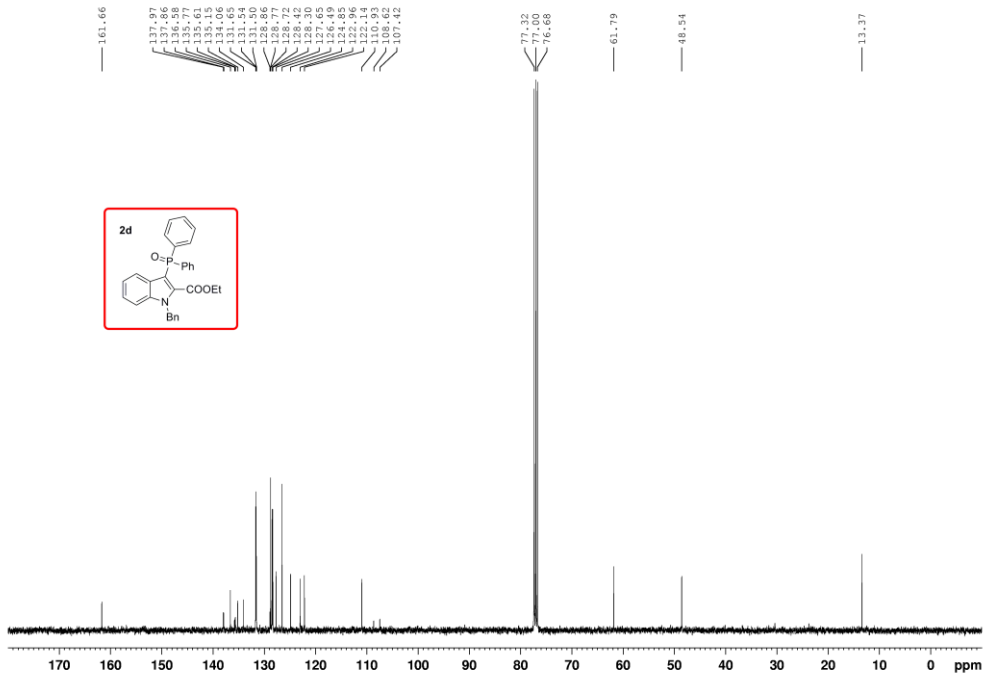

7.812
7.794
7.791
7.781
7.763
7.533
7.531
7.515
7.512
7.497
7.494
7.461
7.454
7.432
7.435
7.424
7.421
7.417
7.414
7.327
7.323
7.267
7.197
7.176
7.040
7.033
7.028
7.020
7.012
7.007
7.000
5.239
4.238
4.220
4.203
4.185
3.760
3.742
3.724
3.706
1.267
1.249
1.231
0.987
0.969
0.951
0.000

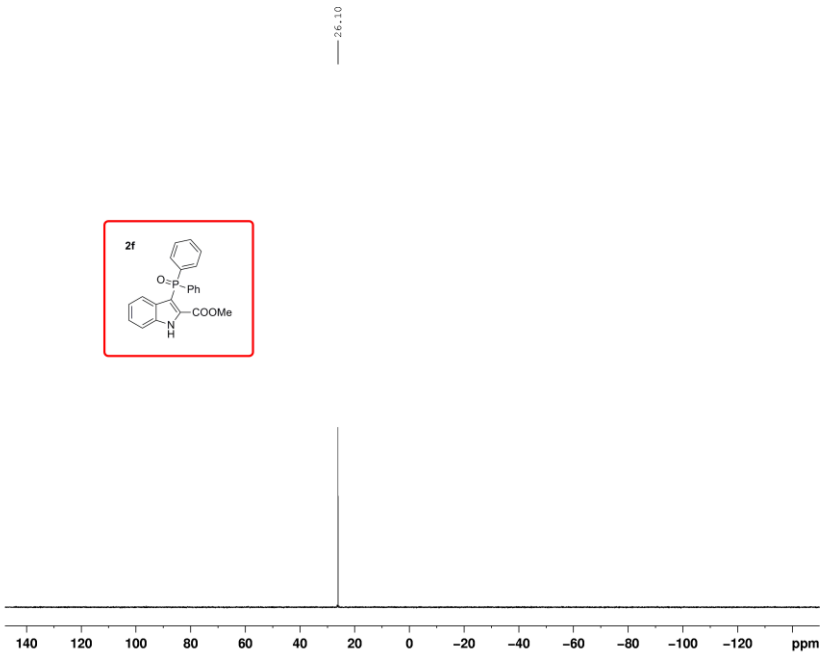

2c
O
P
Ph
COOEt
N
CH2COOEt
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
4.03
2.01
4.06
2.07
1.02
1.00
2.02
2.02
2.06
3.21
3.00


167.75
161.21
138.26
138.16
135.33
134.41
134.23
131.54
131.44
128.94
128.86
128.39
128.27
125.78
123.40
122.34
110.21
109.73
109.03
77.32
77.00
76.68
61.76
61.64
46.46
14.02
13.37
2c
O
P
Ph
COOEt
N
CH2COOEt
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


21.438
2d
O
P
Ph
COOEt
N
Bn
140
120
100
80
60
40
20
0
−20
−40
−60
−80
−100
−120
ppm


7.821
7.803
7.800
7.790
7.772
7.769
7.536
7.533
7.518
7.514
7.500
7.496
7.465
7.458
7.446
7.442
7.439
7.428
7.425
7.421
7.373
7.353
7.298
7.293
7.276
7.259
7.248
7.241
7.231
7.214
7.162
7.142
7.095
7.078
7.019
7.000
6.982
5.644
3.680
3.663
3.645
3.627
0.944
0.926
0.908
2d
Ph
COOEt
Bn
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
4.00
2.04
4.02
1.06
4.03
1.04
2.00
1.09
2.00
2.02
3.01

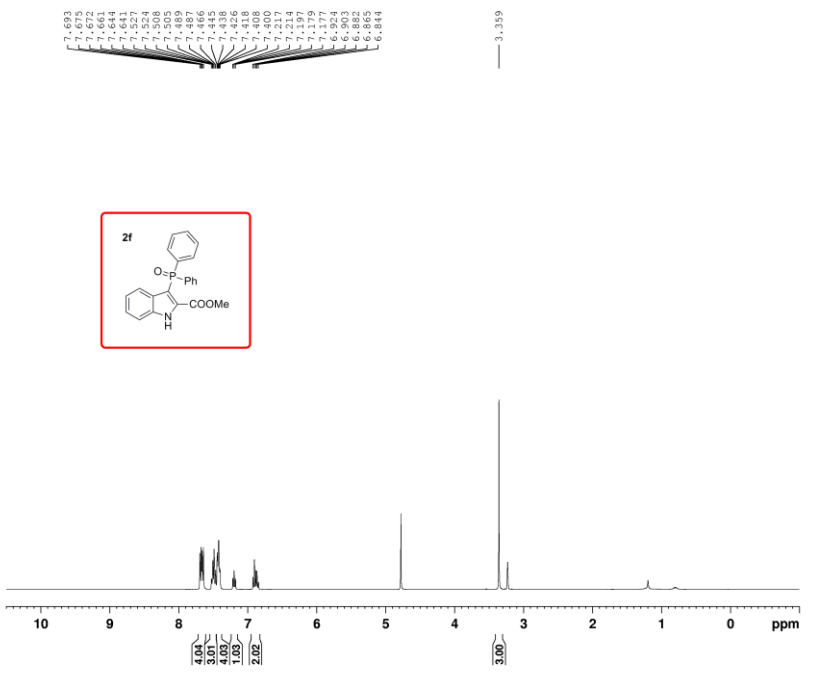

161.66
137.97
137.86
136.88
135.77
135.61
135.15
134.06
131.65
131.54
131.50
128.86
128.77
128.72
128.42
128.30
127.65
126.49
124.85
122.96
122.14
110.93
109.42
107.42
77.32
77.00
76.68
61.79
48.54
13.37
2d
Ph
COOEt
Bn
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm




```
NAME            zhouax120715-P-Me-H
EXPNO                        11
PROCNO                        1
Date_                  20120715
Time                      10.07
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                zgpg30
TD                        65536
SOLVENT                   MeOD
NS                           16
DS                            4
SWH                   64102.563 Hz
FIDRES                0.978127 Hz
AQ                    0.5112308 sec
RG                         2050
DW                        7.800 usec
DE                        6.50 usec
TE                       296.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

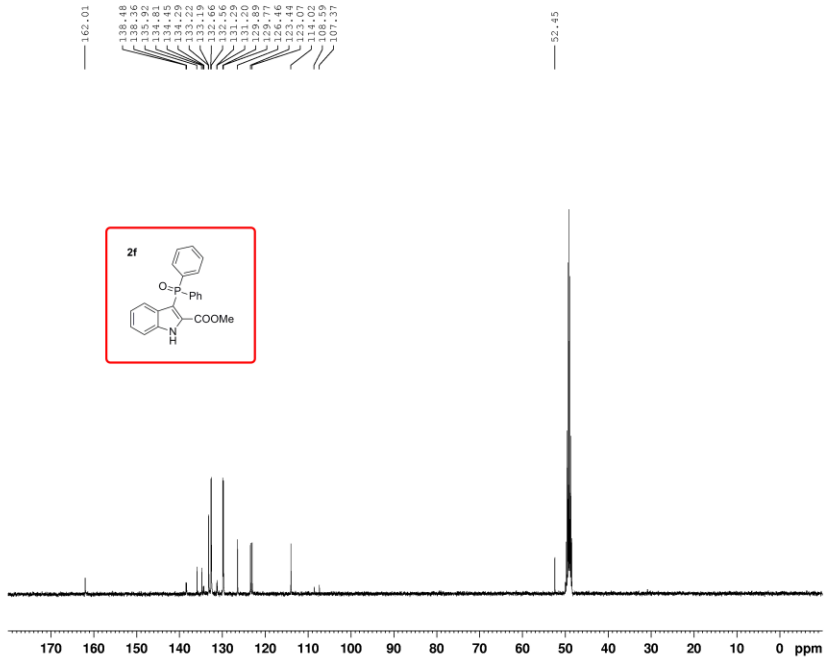
======== CHANNEL f1 ========
NUC1                        31P
P1                        14.85 usec
PL1                        2.80 dB
PL1W                 13.31493304 W
SFO1                161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     90.00 usec
PL2                       -2.00 dB
PL12                      15.50 dB
PL13                      15.50 dB
PL2W                 18.19369861 W
PL12W                 0.32353121 W
PL13W                 0.32353121 W
SFO2                400.1316005 MHz
SI                         32768
SF                  161.9755930 MHz
WDW                           EM
SSB                            0
LB                         1.00 Hz
GB                             0
PC                          1.40
```




```
NAME            zhouax120715-2
EXPNO                        10
PROCNO                        1
Date_                  20120715
Time                      10.49
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                  zg30
TD                        65536
SOLVENT                   MeOD
NS                            8
DS                            2
SWH                    8223.685 Hz
FIDRES                0.125483 Hz
AQ                    3.9846387 sec
RG                          161
DW                       60.800 usec
DE                        6.50 usec
TE                       296.0 K
D1                    1.00000000 sec
TD0                           1

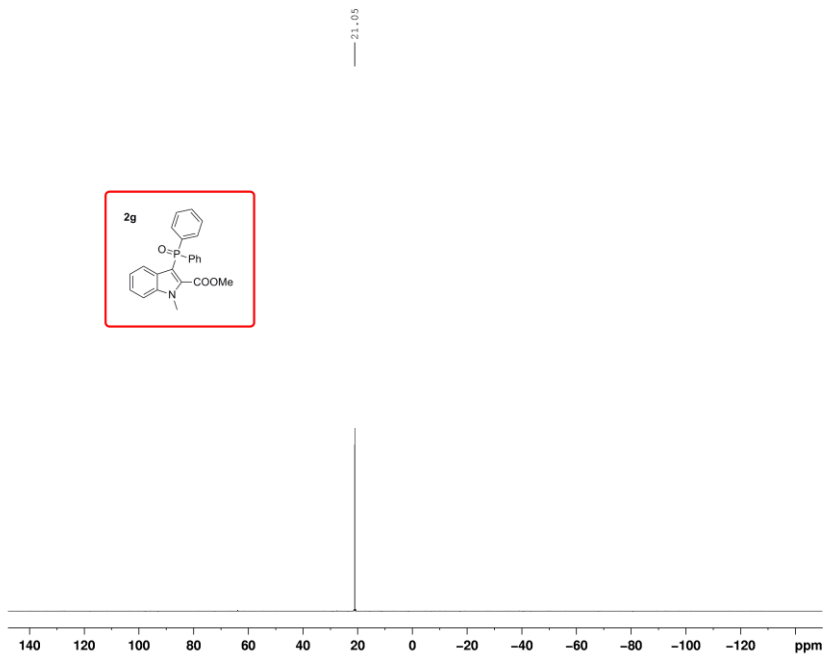
======== CHANNEL f1 ========
NUC1                        1H
P1                        12.00 usec
PL1                       -3.00 dB
PL1W                 22.90425682 W
SFO1                400.1324710 MHz
SI                         32768
SF                  400.1300337 MHz
WDW                           EM
SSB                            0
LB                         0.30 Hz
GB                             0
PC                          1.00
```




```
NAME            zhouax120721-P-Me-H-2
EXPNO                              10
PROCNO                              1
Date_                        20120721
Time                            23.40
INSTRUM                         spect
PROBHD     5 mm PABBO BB-
PULPROG                      zgpg30
TD                            65536
SOLVENT                         CDCl3
NS                                620
DS                                  4
SWH                         24038.461 Hz
FIDRES                       0.366798 Hz
AQ                           1.3631988 sec
RG                                 57
DW                           20.800 usec
DE                            6.50 usec
TE                           297.4 K
D1                        2.00000000 sec
D11                       0.03000000 sec
TD0                                 1

======== CHANNEL f1 ========
NUC1                              13C
P1                               9.70 usec
PL1                             -2.00 dB
PL1W                    56.13311005 W
SFO1                    100.6228298 MHz

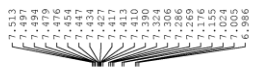
======== CHANNEL f2 ========
CPDPRG2                     waltz16
NUC2                              1H
PCPD2                           80.00 usec
PL2                             -2.10 dB
PL12                            13.90 dB
PL13                            13.90 dB
PL2W                    17.72104263 W
PL12W                    0.44513249 W
PL13W                    0.44513249 W
SFO2                    400.1316005 MHz
SI                             32768
SF                      100.6130118 MHz
WDW                            EM
SSB                             0
LB                           1.00 Hz
GB                              0
PC                           1.40
```




```
NAME            zhouax120405-P-Me-Me
EXPNO                              11
PROCNO                              1
Date_                        20120405
Time                            15.44
INSTRUM                         spect
PROBHD     5 mm PABBO BB-
PULPROG                      zgpg30
TD                            65536
SOLVENT                         CDCl3
NS                                 16
DS                                  4
SWH                         64102.563 Hz
FIDRES                       0.978127 Hz
AQ                           0.5112308 sec
RG                              2050
DW                            7.800 usec
DE                            6.50 usec
TE                           295.5 K
D1                        2.00000000 sec
D11                       0.03000000 sec
TD0                                 1

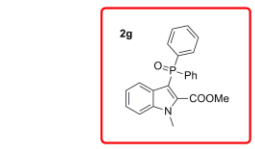
======== CHANNEL f1 ========
NUC1                              31P
P1                              10.80 usec
PL1                              9.00 dB
PL1W                    26.12361908 W
SFO1                    161.9674942 MHz

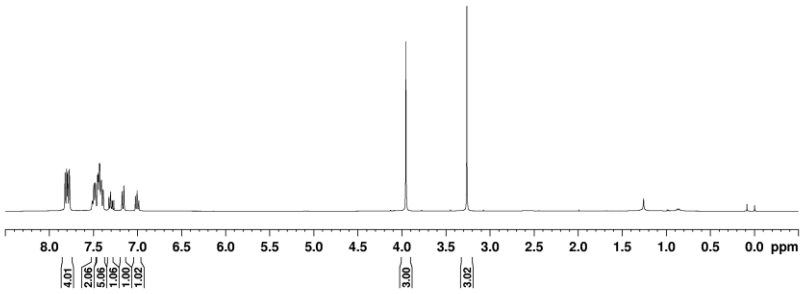
======== CHANNEL f2 ========
CPDPRG2                     waltz16
NUC2                              1H
PCPD2                           80.00 usec
PL2                             -2.10 dB
PL12                            13.90 dB
PL13                            13.90 dB
PL2W                    17.72104263 W
PL12W                    0.44513249 W
PL13W                    0.44513249 W
SFO2                    400.1316005 MHz
SI                             32768
SF                      161.9755930 MHz
WDW                            EM
SSB                             0
LB                           1.00 Hz
GB                              0
PC                           1.40
```

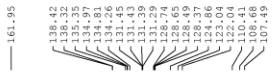



```
NAME            zhouax120405-P-Me-Me
EXPNO                 10
PROCNO                 1
Date_           20120405
Time                15.42
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT             CDCl3
NS                    16
DS                     2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                    64
DW                60.800 usec
DE                 6.50 usec
TE                294.8 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.70 usec
PL1                -1.00 dB
PL1W          13.75590801 W
SFO1          400.1324710 MHz
SI                 32768
SF            400.1300016 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



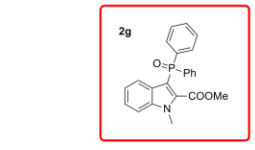


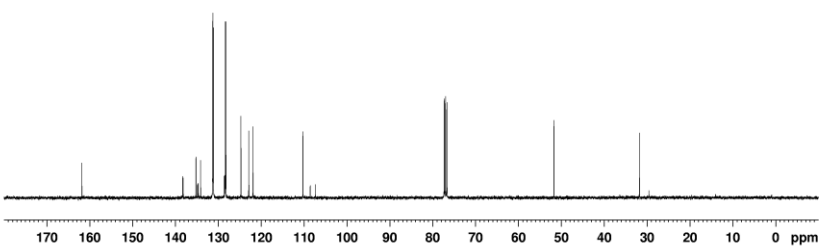


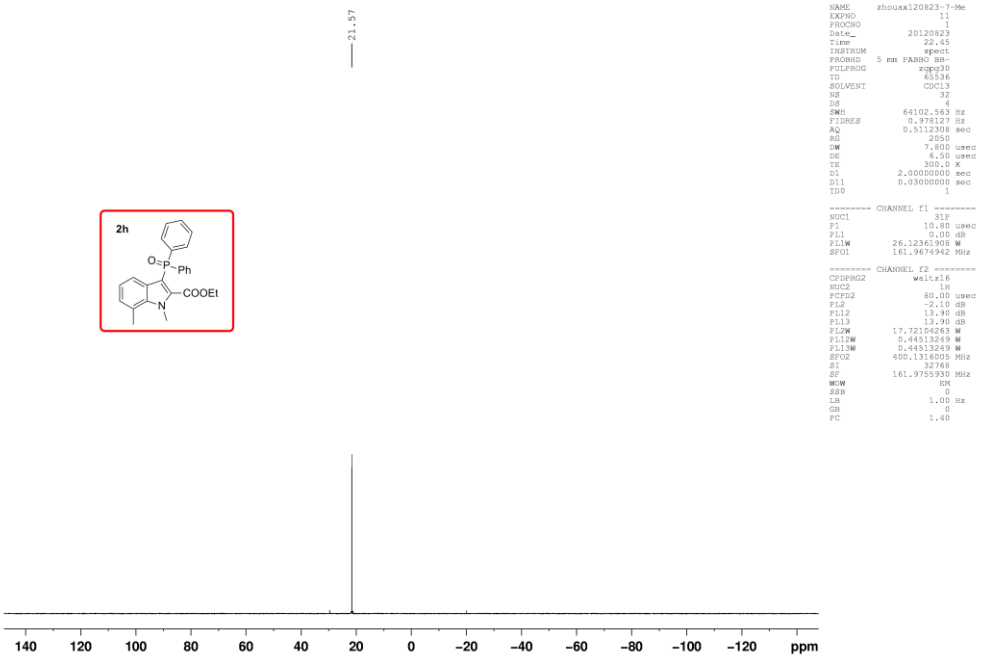

```
NAME            zhouax120405-P-Me-Me
EXPNO                 12
PROCNO                 1
Date_           20120405
Time                15.51
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT             CDCl3
NS                   120
DS                     4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                  1150
DW                20.800 usec
DE                 6.50 usec
TE                296.4 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

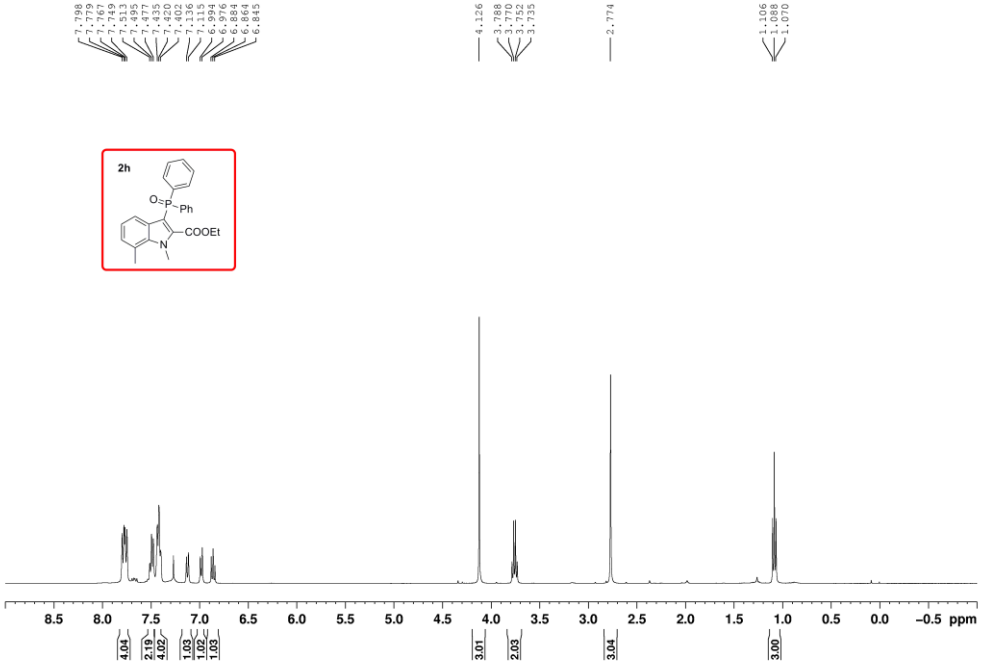
======== CHANNEL f1 ========
NUC1                  13C
P1                  9.70 usec
PL1                -2.00 dB
PL1W          56.13311805 W
SFO1          100.6228298 MHz

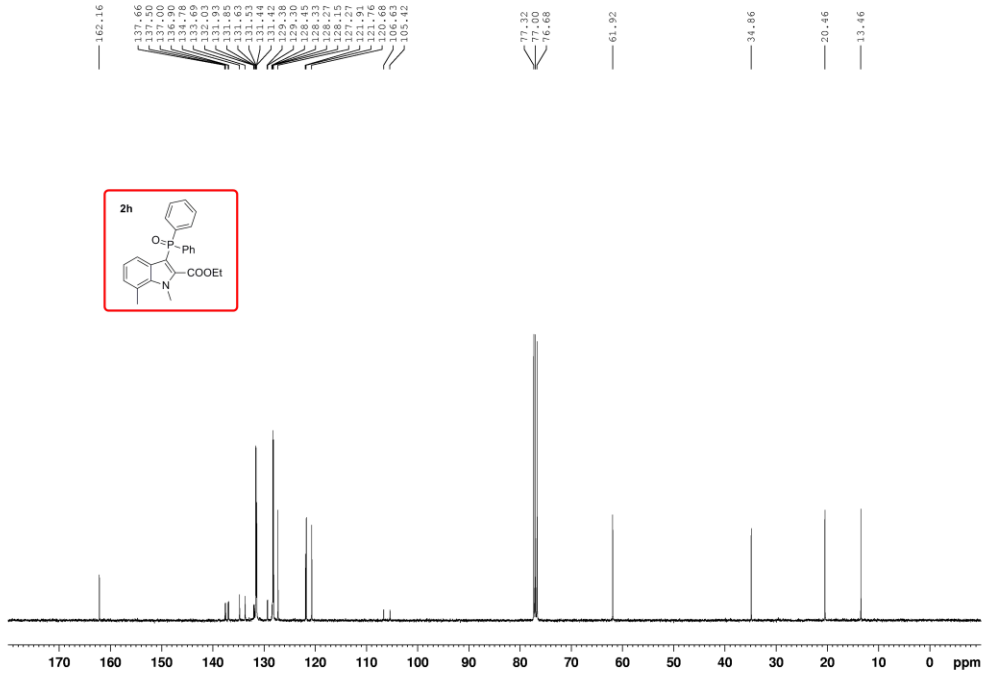
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.10 dB
PL12               13.90 dB
PL13               13.90 dB
PL2W          17.72104263 W
PL12W          0.44513249 W
PL13W          0.44513249 W
SFO2          400.1316005 MHz
SI                 32768
SF            100.6127810 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

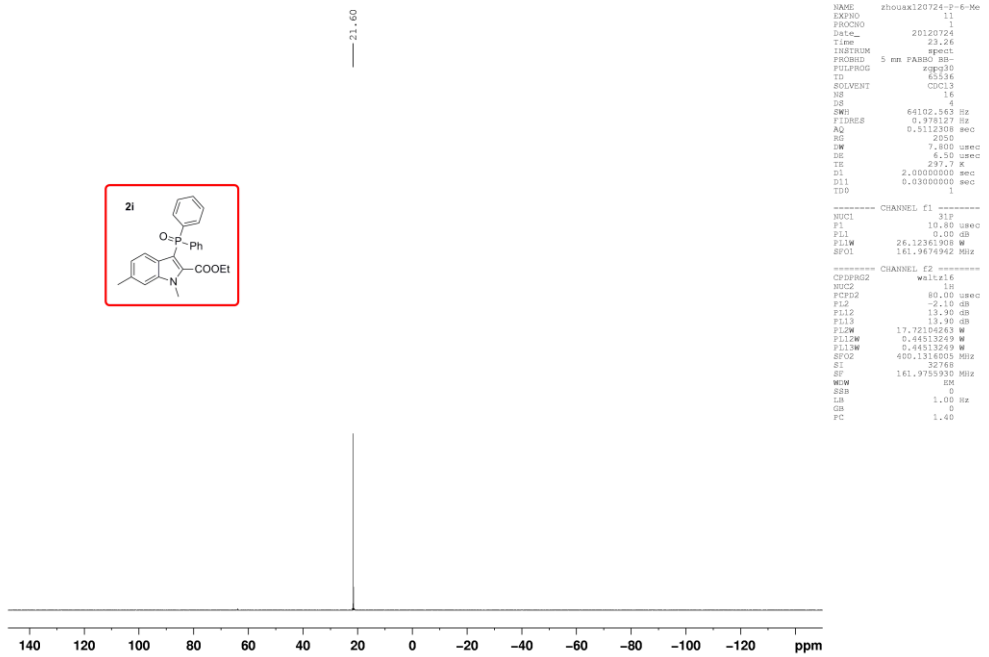





21.57
2h
O
P
Ph
COOEt
N
NAME zhouax120823-7-Me
EXPNO 11
PROCNO 1
Date_ 20120823
Time 22.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 64102.563 Hz
FIDRES 0.978127 Hz
AQ 0.5112308 sec
RG 2050
DW 7.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 10.80 usec
PL1 0.00 dB
PL1W 26.12361908 W
SFO1 161.9674942 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.10 dB
PL12 13.90 dB
PL13 13.90 dB
PL2W 17.72104263 W
PL12W 0.44513249 W
PL13W 0.44513249 W
SFO2 400.1316005 MHz
SI 32768
SF 161.9755930 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 ppm


7.798
7.779
7.767
7.749
7.513
7.495
7.437
7.435
7.420
7.402
7.136
7.115
6.994
6.976
6.884
6.864
6.845
4.126
3.788
3.770
3.752
3.735
2.774
1.106
1.088
1.070
2h
O
P
Ph
COOEt
N
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 ppm
4.04
2.19
4.02
1.03
1.02
1.03
3.01
2.03
3.04
9.00

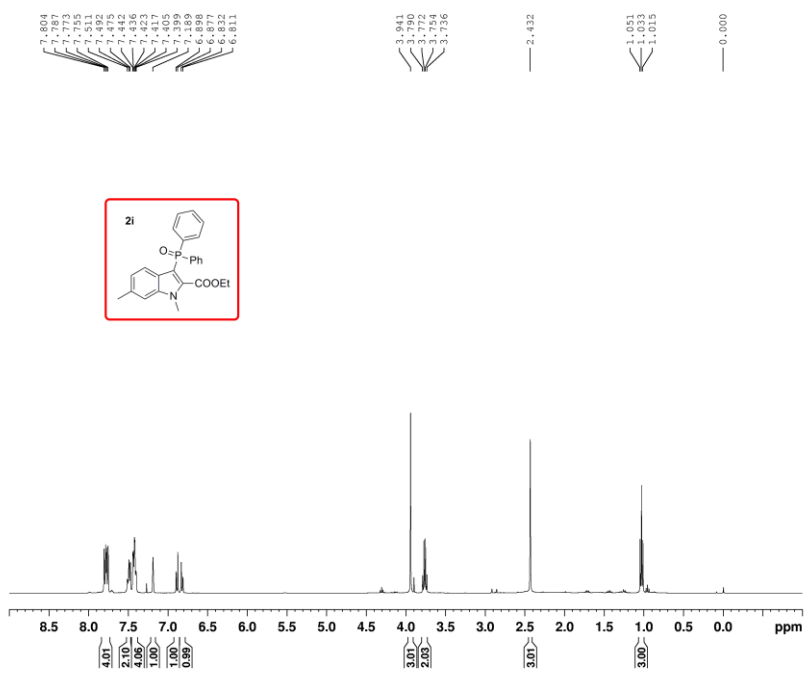





```
NAME        zhouax120724-P-6-Me
EXPNO                        11
PROCNO                        1
Date_                  20120724
Time                      23.26
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                zgpg30
TD                         65536
SOLVENT                  CDCl3
NS                            16
DS                             4
SWH                  64102.563 Hz
FIDRES               0.978127 Hz
AQ                   0.5112308 sec
RG                          2050
DW                       7.800 usec
DE                       6.50 usec
TE                       297.7 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                           1

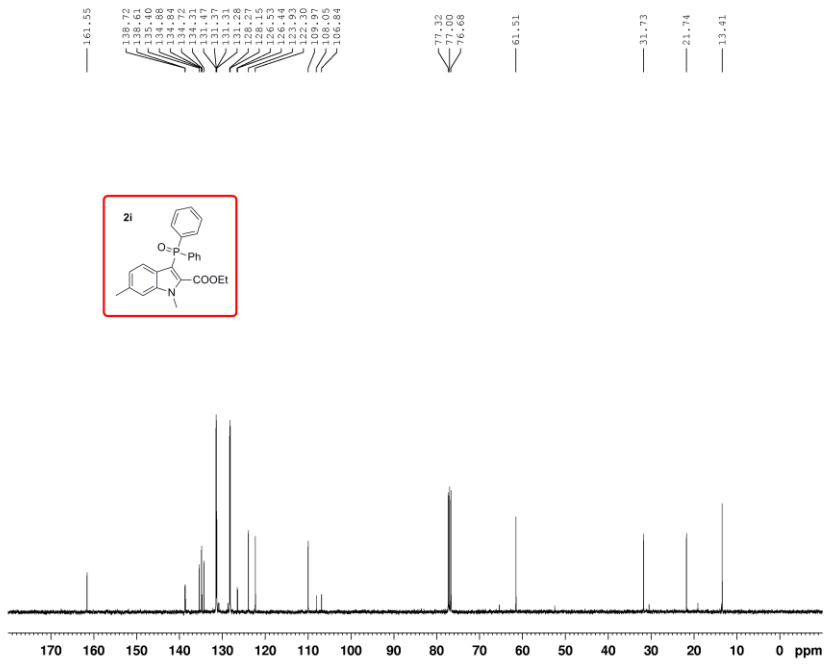
======== CHANNEL f1 ========
NUC1                        31P
P1                        10.80 usec
PL1                        0.00 dB
PL1W                26.12361908 W
SFO1               161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                     80.00 usec
PL2                       -2.10 dB
PL12                      13.90 dB
PL13                      13.90 dB
PL2W                17.72106263 W
PL12W                0.44513249 W
PL13W                0.44513249 W
SFO2               400.1316005 MHz
SI                         32768
SF                 161.9755930 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```




```
NAME            zhouax120724-P-6-Me
EXPNO                            10
PROCNO                            1
Date_                      20120724
Time                          23.24
INSTRUM                       spect
PROBHD     5 mm PABBO BB-
PULPROG                        zg30
TD                            65536
SOLVENT                       CDCl3
NS                               16
DS                                2
SWH                      8223.685 Hz
FIDRES                   0.125483 Hz
AQ                       3.9846387 sec
RG                               64
DW                          60.800 usec
DE                           6.50 usec
TE                          297.1 K
D1                     1.00000000 sec
TD0                               1

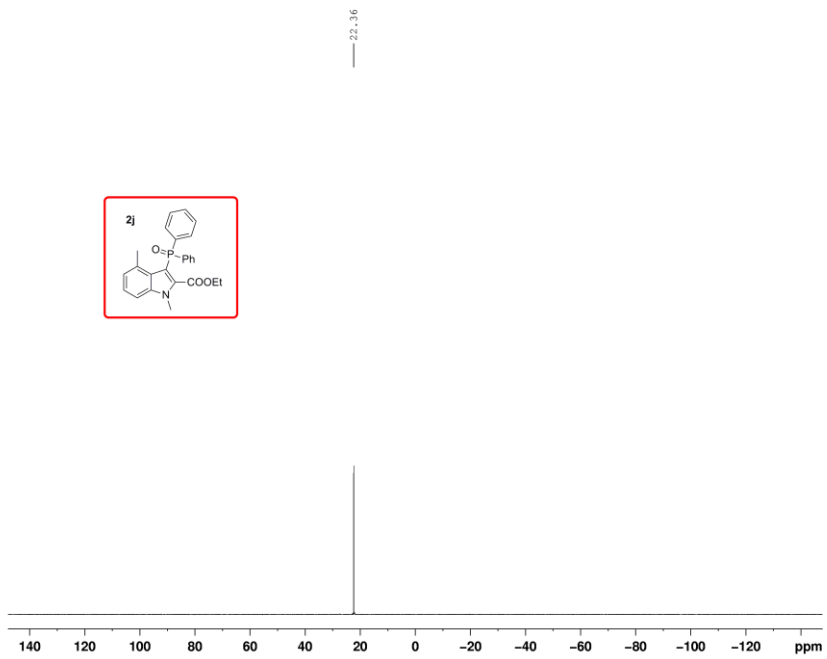
======== CHANNEL f1 ========
NUC1                             1H
P1                           14.70 usec
PL1                          -1.00 dB
PL1W                  13.75590801 W
SFO1                  400.1324710 MHz
SI                            32768
SF                    400.1300011 MHz
WDW                              EM
SSB                               0
LB                            0.30 Hz
GB                                0
PC                            1.00
```




```
NAME            zhouax120724-P-6-Me
EXPNO                            12
PROCNO                            1
Date_                      20120724
Time                          23.35
INSTRUM                       spect
PROBHD     5 mm PABBO BB-
PULPROG                    zgpg30
TD                            65536
SOLVENT                       CDCl3
NS                              120
DS                                4
SWH                     24038.461 Hz
FIDRES                   0.366798 Hz
AQ                       1.3631988 sec
RG                              912
DW                          20.800 usec
DE                           6.50 usec
TE                          298.3 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                               1

======== CHANNEL f1 ========
NUC1                             13C
P1                            9.70 usec
PL1                          -2.00 dB
PL1W                  56.13311005 W
SFO1                  100.6228298 MHz

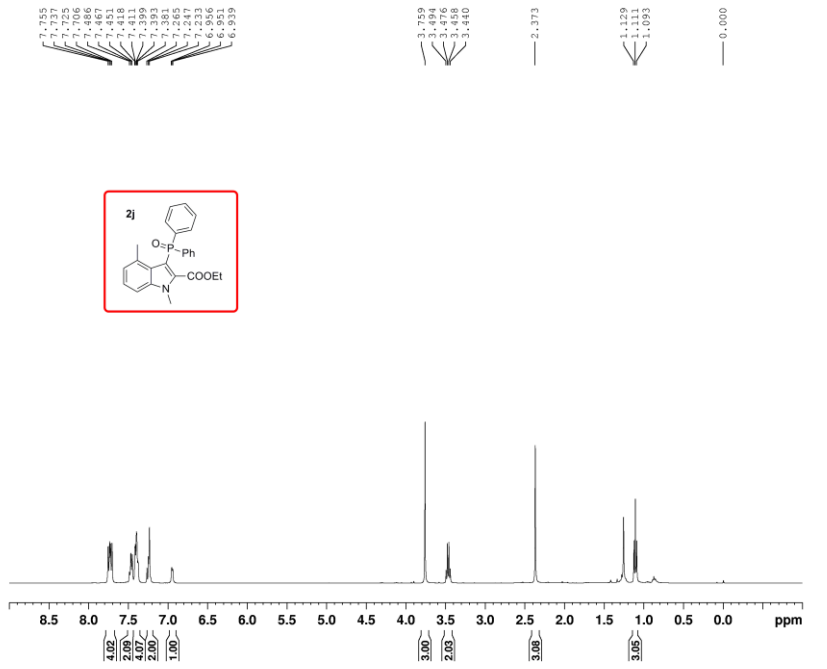
======== CHANNEL f2 ========
CPDPRG2                     waltz16
NUC2                             1H
PCPD2                        80.00 usec
PL2                          -2.10 dB
PL12                         13.90 dB
PL13                         13.90 dB
PL2W                  17.72106263 W
PL12W                  0.44513249 W
PL13W                  0.44513249 W
SFO2                  400.1316005 MHz
SI                            32768
SF                    100.6127802 MHz
WDW                              EM
SSB                               0
LB                            1.00 Hz
GB                                0
PC                            1.40
```




```
NAME          zhouax120823-P-4-Me
EXPNO                          12
PROCNO                          1
Date_                    20120823
Time                        16.56
INSTRUM                      spect
PROBHD    5 mm PABBO BB-
PULPROG                   zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                            16
DS                             4
SWH                    64102.563 Hz
FIDRES                 0.978127 Hz
AQ                     0.5112308 sec
RG                          2050
DW                         7.800 usec
DE                         6.50 usec
TE                        297.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                            1

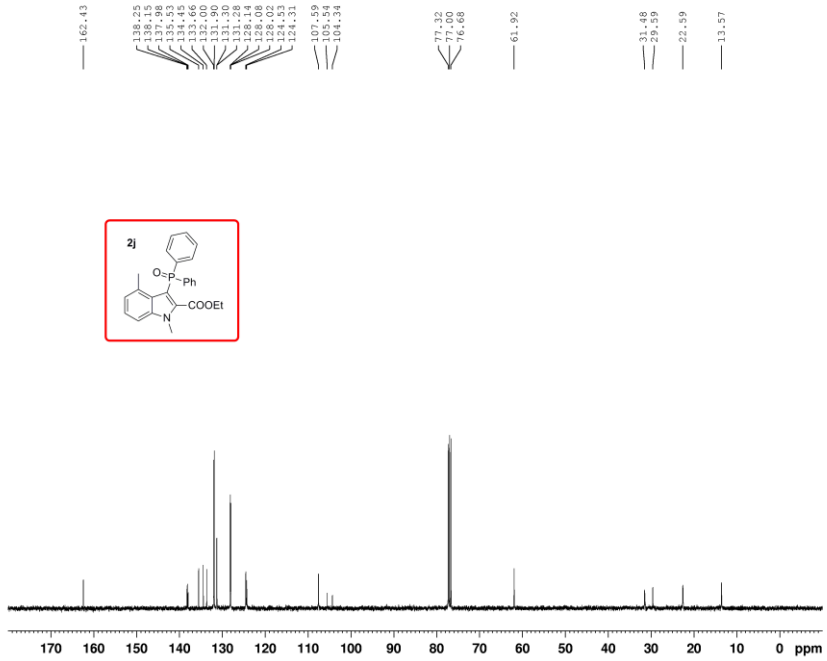
======== CHANNEL f1 ========
NUC1                         31P
P1                         14.85 usec
PL1                         2.80 dB
PL1W                13.31493304 W
SFO1                161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                     90.00 usec
PL2                       -2.00 dB
PL12                      15.50 dB
PL13                      15.50 dB
PL2W                18.19369861 W
PL12W                0.32353121 W
PL13W                0.32353121 W
SFO2                400.1316005 MHz
SI                         32768
SF                  161.9755930 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```




```
NAME          zhouax120823-P-4-Me
EXPNO                          10
PROCNO                          1
Date_                    20120823
Time                        16.47
INSTRUM                      spect
PROBHD    5 mm PABBO BB-
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                            16
DS                             2
SWH                     8223.685 Hz
FIDRES                 0.125483 Hz
AQ                     3.9846387 sec
RG                           101
DW                        60.800 usec
DE                         6.50 usec
TE                        296.4 K
D1                   1.00000000 sec
TD0                            1

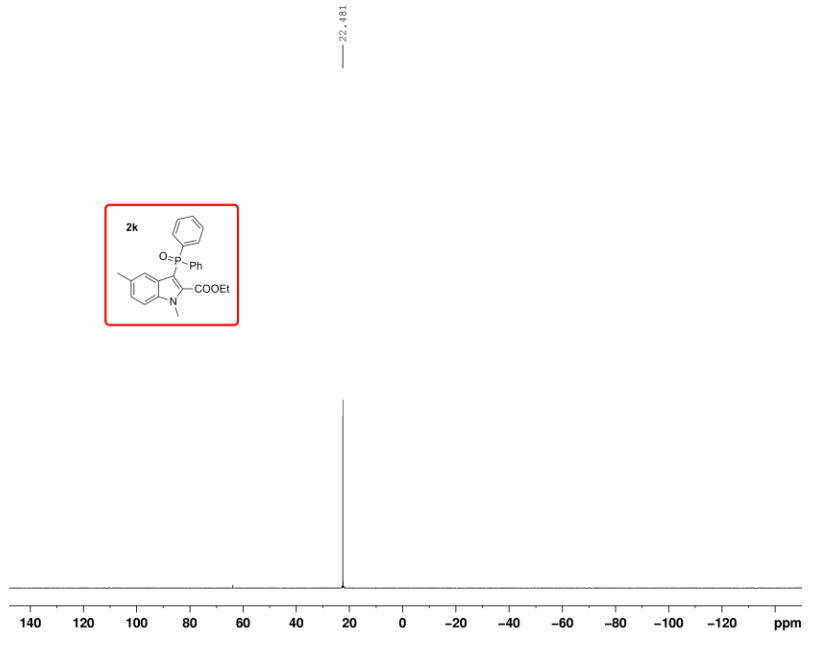
======== CHANNEL f1 ========
NUC1                         1H
P1                         12.00 usec
PL1                        -3.00 dB
PL1W                22.90425682 W
SFO1                400.1324710 MHz
SI                         32768
SF                  400.1300035 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```




```
NAME          zhouax120823-P-4-Me
EXPNO                          11
PROCNO                          1
Date_                    20120823
Time                        16.55
INSTRUM                     spect
PROBHD   5 mm PABBO BB-
PULPROG                  zgpg30
TD                            65536
SOLVENT                     CDCl3
NS                              120
DS                                4
SWH                     24038.461 Hz
FIDRES                   0.366798 Hz
AQ                       1.3631988 sec
RG                             2050
DW                          20.800 usec
DE                           6.50 usec
TE                          297.0 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                          13C
P1                           9.40 usec
PL1                         -2.00 dB
PL1W                57.32743073 W
SFO1               100.6228298 MHz

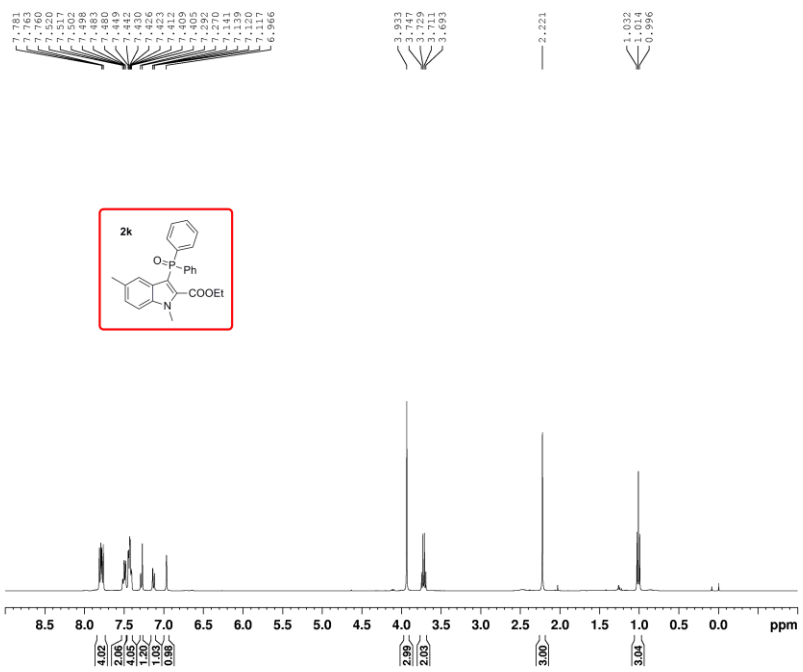
======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                           1H
PCPD2                       90.00 usec
PL2                         -2.00 dB
PL12                        15.50 dB
PL13                        15.50 dB
PL2W                18.19369861 W
PL12W                0.32353121 W
PL13W                0.32353121 W
SFO2               400.1316005 MHz
SI                          32768
SF                 100.6127780 MHz
WDW                            EM
SSB                             0
LB                           1.00 Hz
GB                              0
PC                           1.40
```




```
NAME          zhouax120331-P-Me
EXPNO                          11
PROCNO                          1
Date_                    20120331
Time                        18.07
INSTRUM                     spect
PROBHD   5 mm PABBO BB-
PULPROG                  zgpg30
TD                            65536
SOLVENT                     CDCl3
NS                              16
DS                               4
SWH                     64102.563 Hz
FIDRES                   0.978127 Hz
AQ                       0.5112308 sec
RG                             2050
DW                           7.800 usec
DE                           6.50 usec
TE                          296.6 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                             1

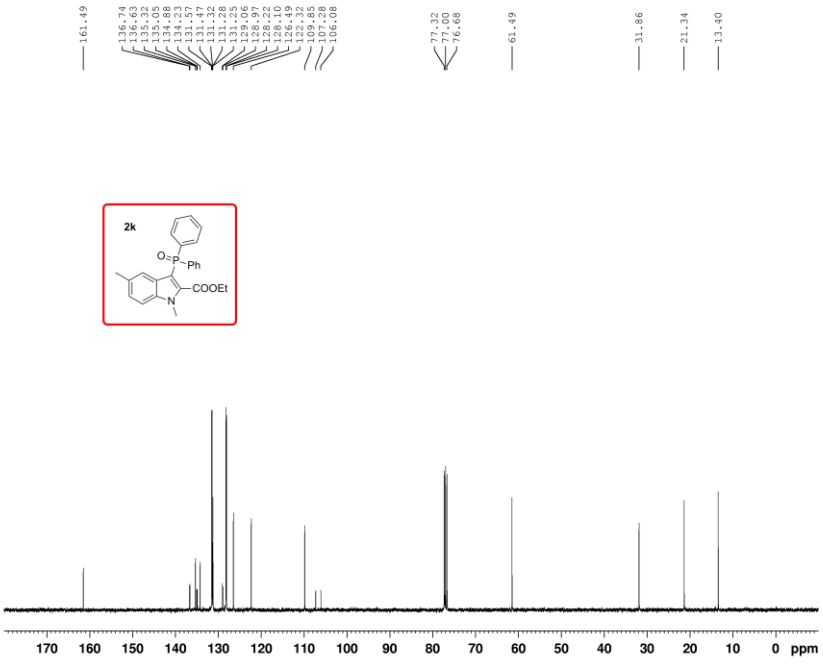
======== CHANNEL f1 ========
NUC1                          31P
P1                          10.80 usec
PL1                          0.00 dB
PL1W                26.12361908 W
SFO1               161.9676942 MHz

======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                           1H
PCPD2                       80.00 usec
PL2                         -2.10 dB
PL12                        13.90 dB
PL13                        13.90 dB
PL2W                17.72104263 W
PL12W                0.44513249 W
PL13W                0.44513249 W
SFO2               400.1316005 MHz
SI                          32768
SF                 161.9755930 MHz
WDW                            EM
SSB                             0
LB                           1.00 Hz
GB                              0
PC                           1.40
```




```
NAME            zhouax120331-P-Me
EXPNO                           10
PROCNO                           1
Date_                     20120331
Time                         18.05
INSTRUM                      spect
PROBHD     5 mm PABBO BB-
PULPROG                      zg30
TD                          65536
SOLVENT                     CDCl3
NS                             16
DS                              2
SWH                     8223.685 Hz
FIDRES                  0.125483 Hz
AQ                      3.9846387 sec
RG                             57
DW                         60.800 usec
DE                          6.50 usec
TE                         295.9 K
D1                    1.00000000 sec
TD0                             1

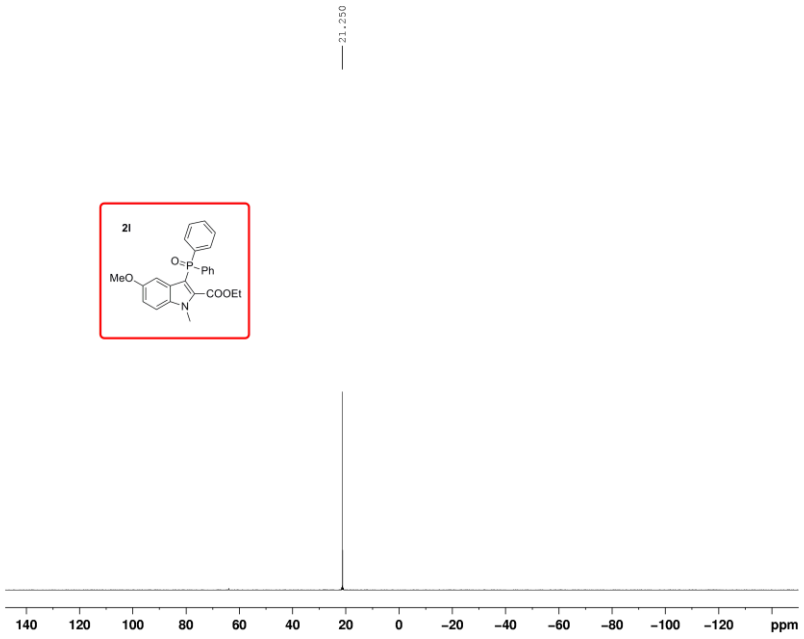
======== CHANNEL f1 ========
NUC1                           1H
P1                         14.70 usec
PL1                        -1.00 dB
PL1W                13.75590801 W
SFO1                400.1324710 MHz
SI                         32768
SF                  400.1300008 MHz
WDW                            EM
SSB                             0
LB                           0.30 Hz
GB                              0
PC                           1.00
```




```
NAME            zhouax120331-P-Me
EXPNO                           12
PROCNO                           1
Date_                     20120331
Time                         18.17
INSTRUM                      spect
PROBHD     5 mm PABBO BB-
PULPROG                  zgpg30
TD                          65536
SOLVENT                     CDCl3
NS                            120
DS                              4
SWH                    24038.461 Hz
FIDRES                  0.366798 Hz
AQ                      1.3631988 sec
RG                             57
DW                         20.800 usec
DE                          6.50 usec
TE                         297.2 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                           13C
P1                          9.70 usec
PL1                        -2.00 dB
PL1W                56.13311005 W
SFO1                100.6228298 MHz

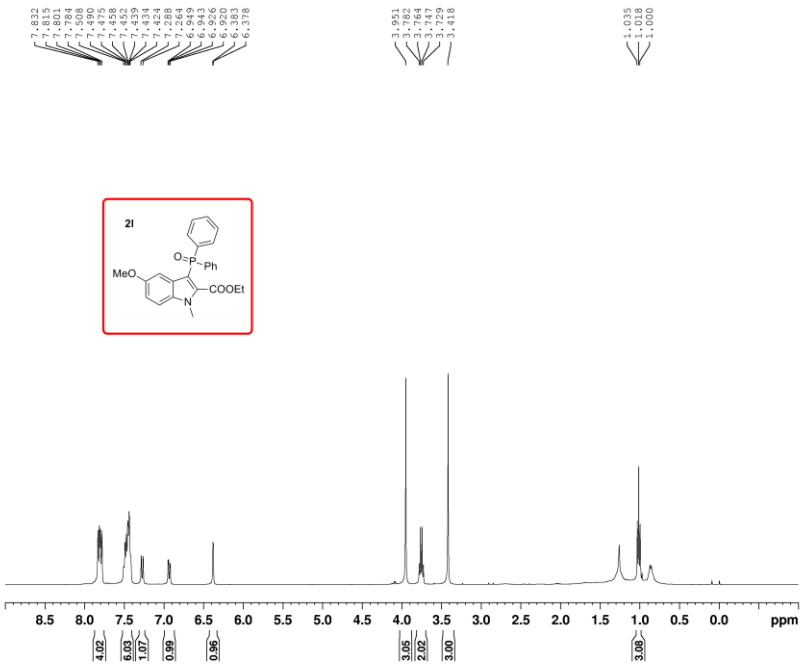
======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                           1H
PCPD2                      80.00 usec
PL2                        -2.10 dB
PL12                       13.90 dB
PL13                       13.90 dB
PL2W                17.72106263 W
PL12W                0.44513249 W
PL13W                0.44513249 W
SFO2                400.1316005 MHz
SI                         32768
SF                  100.6127810 MHz
WDW                            EM
SSB                             0
LB                          1.00 Hz
GB                              0
PC                           1.40
```




```
NAME            zhouax120331-P-OMe
EXPNO                           11
PROCNO                           1
Date_                     20120331
Time                         18.25
INSTRUM                      spect
PROBHD     5 mm PABBO BB-
PULPROG                    zgpg30
TD                          65536
SOLVENT                     CDCl3
NS                             16
DS                              4
SWH                     64102.563 Hz
FIDRES                  0.978127 Hz
AQ                      0.5112308 sec
RG                           2050
DW                         7.800 usec
DE                         6.50 usec
TE                         296.4 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                             1

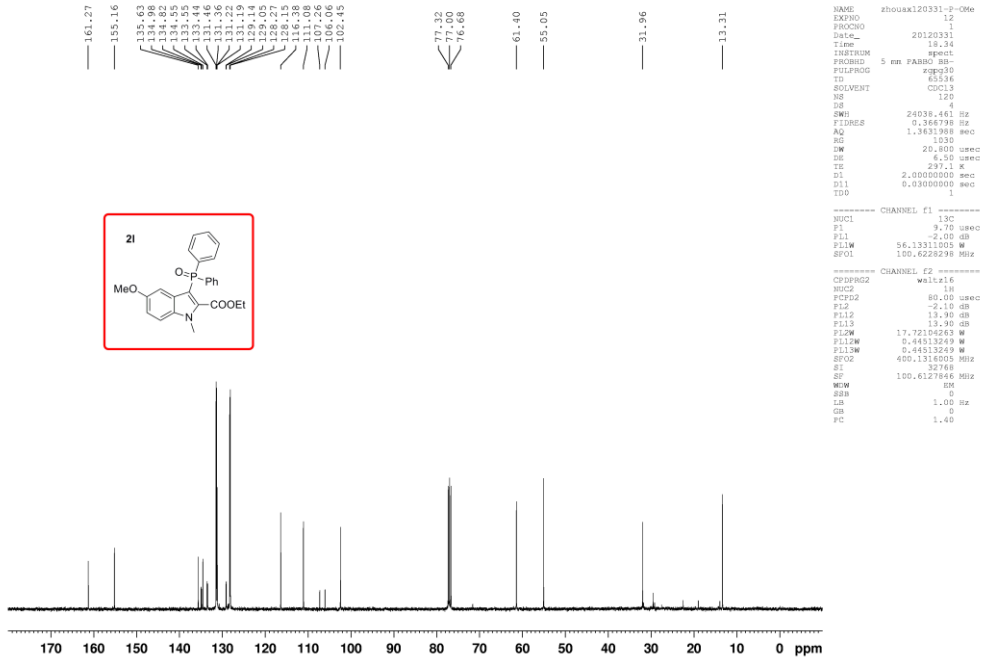
======== CHANNEL f1 ========
NUC1                          31P
P1                          10.80 usec
PL1                          0.00 dB
PL1W                26.12361908 W
SFO1                161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                          1H
PCPD2                       80.00 usec
PL2                         -2.10 dB
PL12                        13.90 dB
PL13                        13.90 dB
PL2W                17.72104263 W
PL12W                0.44513249 W
PL13W                0.44513249 W
SFO2                400.1316005 MHz
SI                           32768
SF                  161.9755930 MHz
WDW                             EM
SSB                              0
LB                            1.00 Hz
GB                               0
PC                            1.40
```




```
NAME            zhouax120331-P-OMe
EXPNO                           10
PROCNO                           1
Date_                     20120331
Time                         18.23
INSTRUM                      spect
PROBHD     5 mm PABBO BB-
PULPROG                    zg30
TD                          65536
SOLVENT                     CDCl3
NS                             16
DS                              2
SWH                      8223.685 Hz
FIDRES                  0.125483 Hz
AQ                      3.9846387 sec
RG                            60.3
DW                         60.800 usec
DE                         6.50 usec
TE                         295.8 K
D1                    1.00000000 sec
TD0                             1

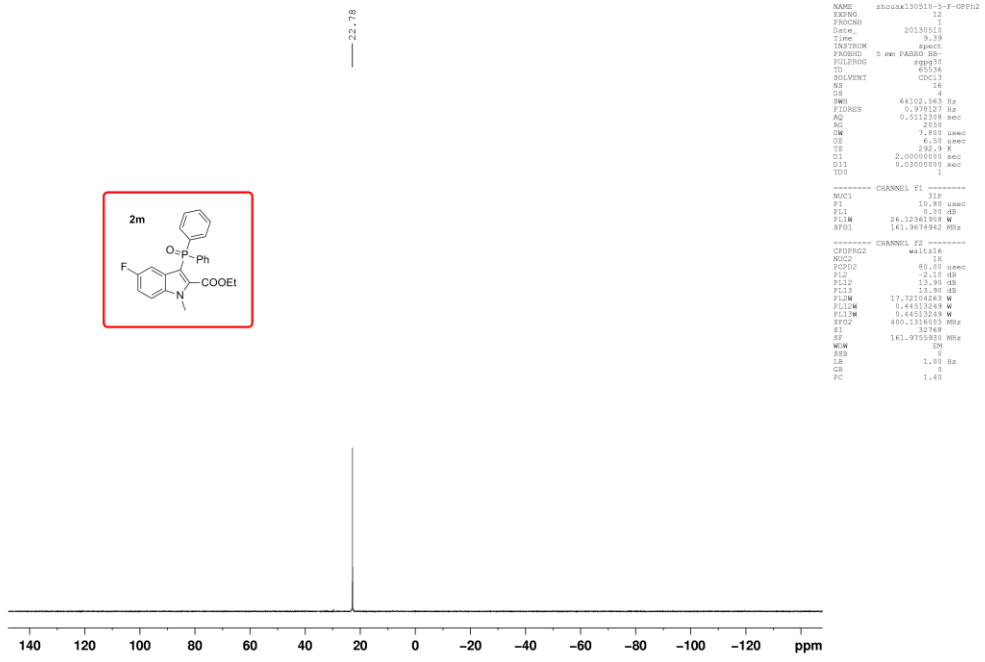
======== CHANNEL f1 ========
NUC1                          1H
P1                          14.70 usec
PL1                         -1.00 dB
PL1W                13.75590801 W
SFO1                400.1324710 MHz
SI                           32768
SF                  400.1299932 MHz
WDW                             EM
SSB                              0
LB                            0.30 Hz
GB                               0
PC                            1.00
```




```
NAME          zhousx120331-P-OMe
EXPNO                         12
PROCNO                         1
Date_                   20120331
Time                       18.34
INSTRUM                    spect
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                           65536
SOLVENT                   CDCl3
NS                             120
DS                               4
SWH                    24038.461 Hz
FIDRES                  0.366798 Hz
AQ                      1.3631988 sec
RG                            1030
DW                        20.800 usec
DE                         6.50 usec
TE                          297.1 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                          9.70 usec
PL1                        -2.00 dB
PL1W                56.13311005 W
SFO1                100.6228298 MHz

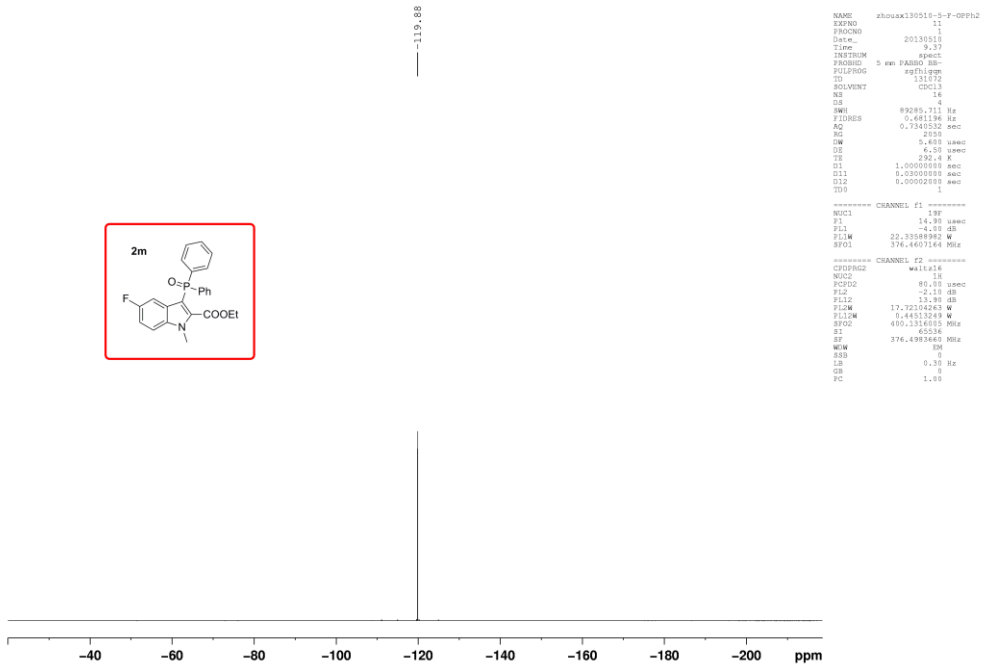
======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                          1H
PCPD2                      80.00 usec
PL2                        -2.10 dB
PL12                       13.90 dB
PL13                       13.90 dB
PL2W                17.72104263 W
PL12W                0.44513249 W
PL13W                0.44513249 W
SFO2                400.1316005 MHz
SI                         32768
SF                  100.6127846 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```




```
NAME          zhousx130510-5-F-OPPh2
EXPNO                         12
PROCNO                         1
Date_                   20130510
Time                        9.39
INSTRUM                    spect
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                           65536
SOLVENT                   CDCl3
NS                             16
DS                              4
SWH                    64102.563 Hz
FIDRES                  0.978127 Hz
AQ                      0.5112308 sec
RG                           2050
DW                         7.800 usec
DE                         6.50 usec
TE                         292.9 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         31P
P1                         10.80 usec
PL1                         0.00 dB
PL1W                26.12361908 W
SFO1                161.9674942 MHz

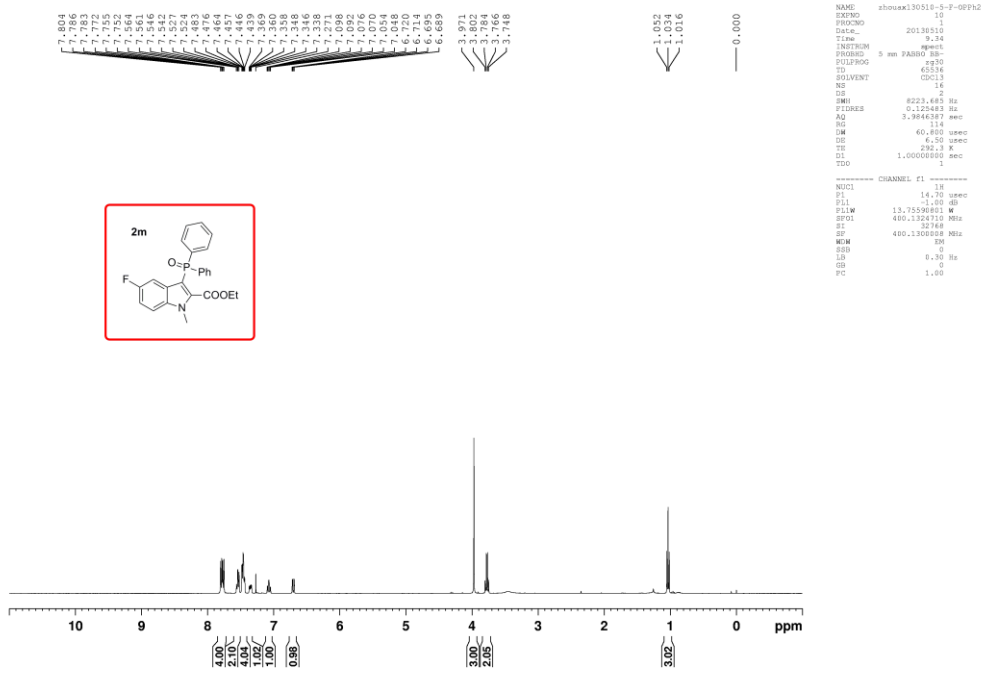
======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                          1H
PCPD2                      80.00 usec
PL2                        -2.10 dB
PL12                       13.90 dB
PL13                       13.90 dB
PL2W                17.72104263 W
PL12W                0.44513249 W
PL13W                0.44513249 W
SFO2                400.1316005 MHz
SI                         32768
SF                  161.9755930 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```




```
NAME            zhouax130510-5-F-OPPh2
EXPNO                   11
PROCNO                   1
Date_         20130510
Time                  9.37
INSTRUM               spect
PROBHD     5 mm PABBO BB-
PULPROG         zgfhigqn
TD                 131072
SOLVENT               CDCl3
NS                       16
DS                        4
SWH              89285.711 Hz
FIDRES               0.681196 Hz
AQ                0.7340532 sec
RG                     2050
DW                   5.600 usec
DE                   6.50 usec
TE                  292.4 K
D1                1.00000000 sec
D11               0.03000000 sec
D12               0.00002000 sec
TD0                       1

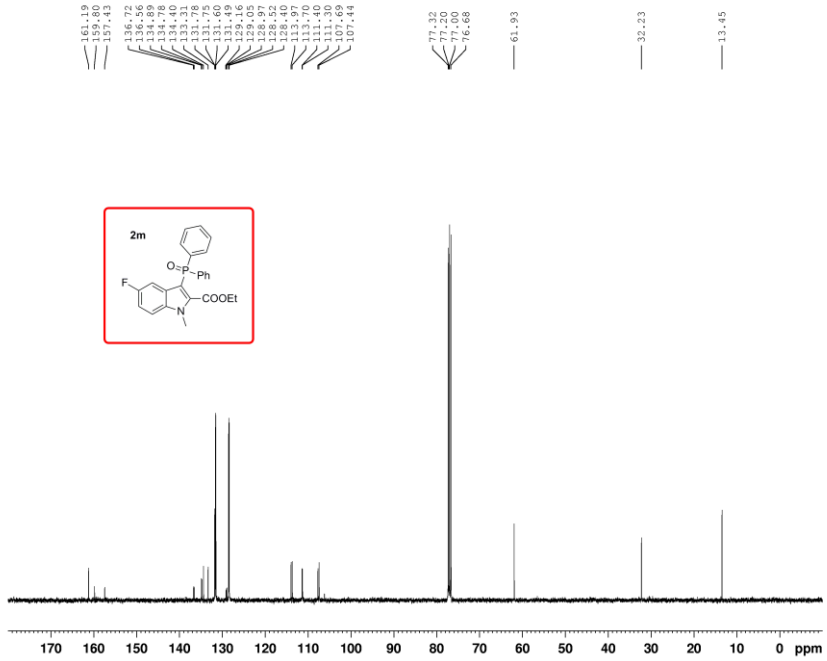
======== CHANNEL f1 ========
NUC1                    19F
P1                    14.90 usec
PL1                   -4.00 dB
PL1W            22.33588982 W
SFO1           376.4607164 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                 80.00 usec
PL2                   -2.10 dB
PL12                  13.90 dB
PL2W            17.72304263 W
PL12W            0.44513249 W
SFO2           400.1316005 MHz
SI                      65536
SF             376.4983668 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.88
```




```
NAME            zhouax130510-5-F-OPPh2
EXPNO                   10
PROCNO                   1
Date_         20130510
Time                  9.34
INSTRUM               spect
PROBHD     5 mm PABBO BB-
PULPROG             zg30
TD                  65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH              8223.685 Hz
FIDRES               0.125483 Hz
AQ                3.9846387 sec
RG                      114
DW                   60.800 usec
DE                   6.50 usec
TE                  298.3 K
D1                1.00000000 sec
TD0                       1

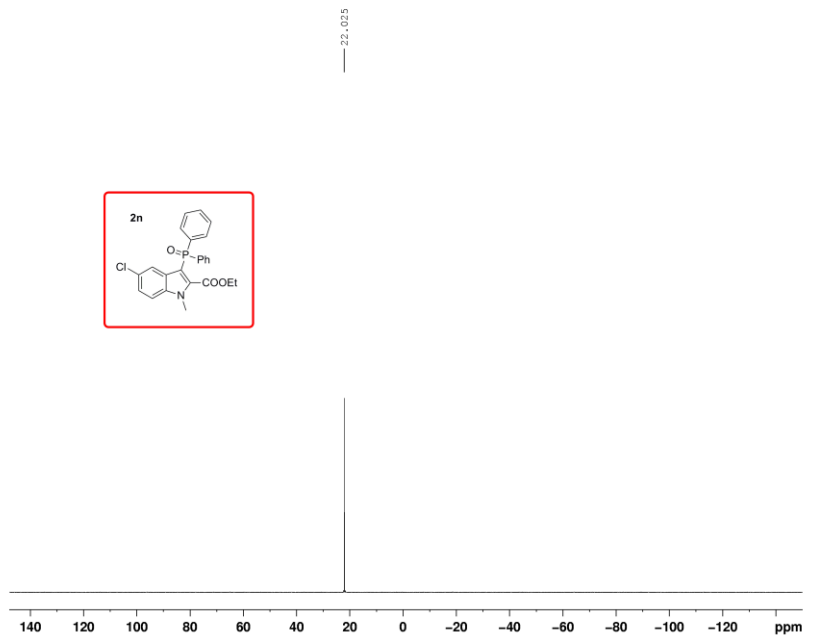
======== CHANNEL f1 ========
NUC1                    1H
P1                    14.70 usec
PL1                   -1.00 dB
PL1W            13.75590801 W
SFO1           400.1324710 MHz
SI                      32768
SF             400.1300000 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```




```
NAME          zhouax130518-5-F-OPPh2
EXPNO                 13
PROCNO                 1
Date_           20130518
Time                9.55
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  250
DS                    4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                 1150
DW               20.800 usec
DE                6.50 usec
TE               293.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.70 usec
PL1               -2.00 dB
PL1W        56.13311885 W
SFO1       100.6228298 MHz

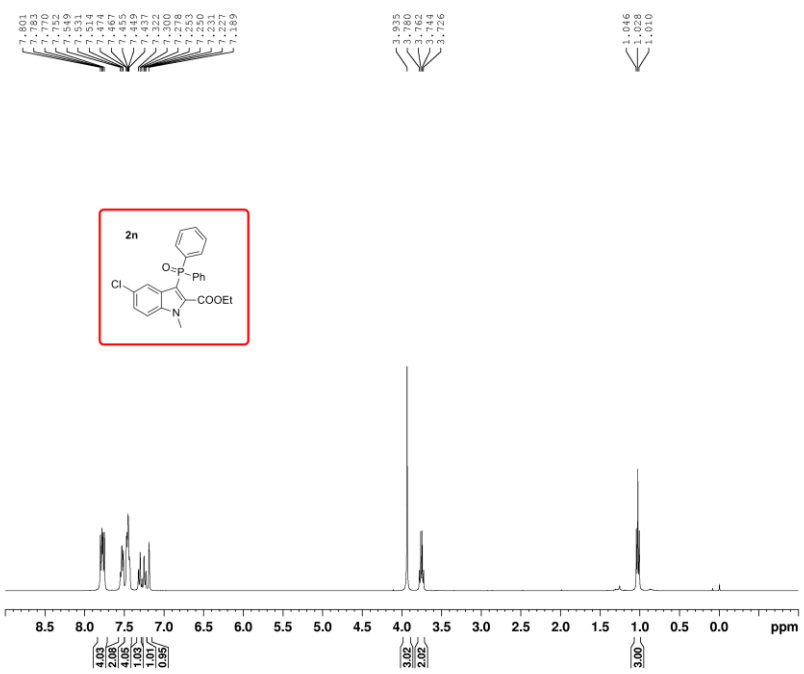
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             80.00 usec
PL2               -2.10 dB
PL12              13.90 dB
PL13              13.90 dB
PL2W        17.72104263 W
PL12W        0.44513249 W
PL13W        0.44513249 W
SFO2       400.1316005 MHz
SI                32768
SF         100.6127744 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```




```
NAME          zhouax120331-P-Cl
EXPNO                 11
PROCNO                 1
Date_           20120331
Time                15.10
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES        0.978127 Hz
AQ            0.5112308 sec
RG                 2050
DW               7.800 usec
DE                6.50 usec
TE               296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

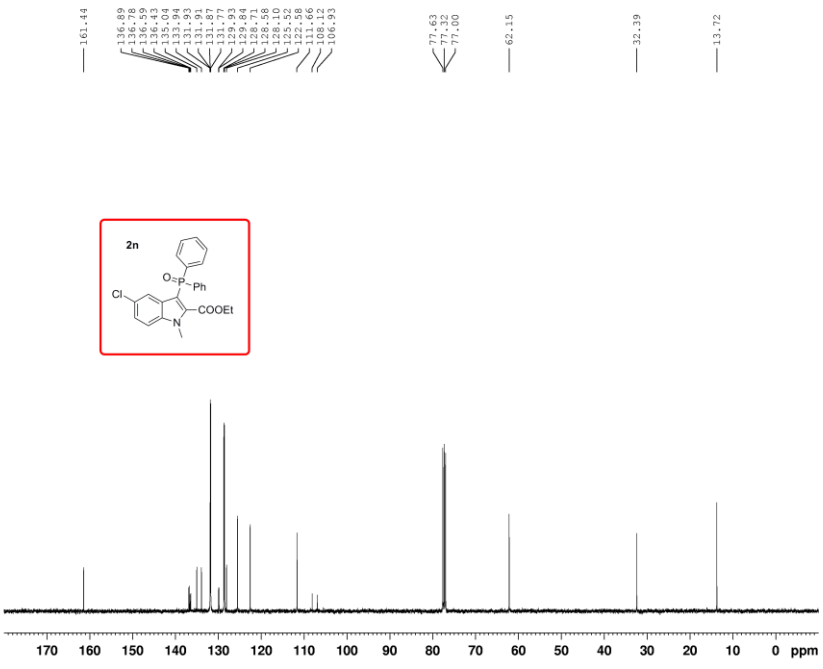
======== CHANNEL f1 ========
NUC1                31P
P1                10.80 usec
PL1                0.00 dB
PL1W        26.12361908 W
SFO1       161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             80.00 usec
PL2               -2.10 dB
PL12              13.90 dB
PL13              13.90 dB
PL2W        17.72104263 W
PL12W        0.44513249 W
PL13W        0.44513249 W
SFO2       400.1316005 MHz
SI                32768
SF         161.9755930 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```




```
NAME          zhouax120331-P-Cl
EXPNO                        10
PROCNO                        1
Date_                  20120331
Time                      15.08
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           16
DS                            2
SWH                   8223.685 Hz
FIDRES                0.125483 Hz
AQ                    3.9846387 sec
RG                         71.8
DW                       60.800 usec
DE                        6.50 usec
TE                       295.4 K
D1                  1.00000000 sec
TD0                           1

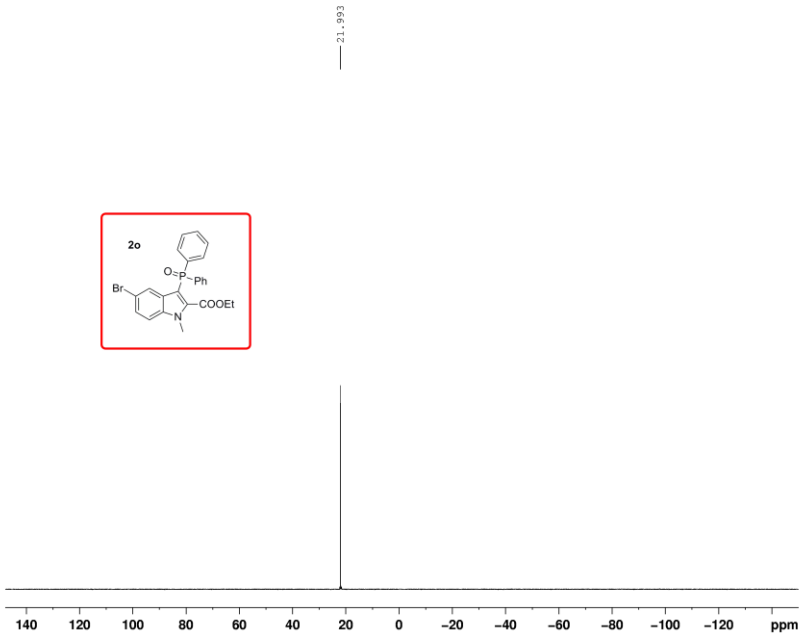
======== CHANNEL f1 ========
NUC1                         1H
P1                        14.70 usec
PL1                       -1.00 dB
PL1W                13.75590801 W
SFO1                400.1324710 MHz
SI                        32768
SF                  400.1299977 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```




```
NAME          zhouax120331-P-Cl
EXPNO                        12
PROCNO                        1
Date_                  20120331
Time                      15.19
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                          120
DS                            4
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631988 sec
RG                          812
DW                       20.800 usec
DE                        6.50 usec
TE                       296.9 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         13C
P1                         9.70 usec
PL1                       -2.00 dB
PL1W                56.13311005 W
SFO1                100.6228298 MHz

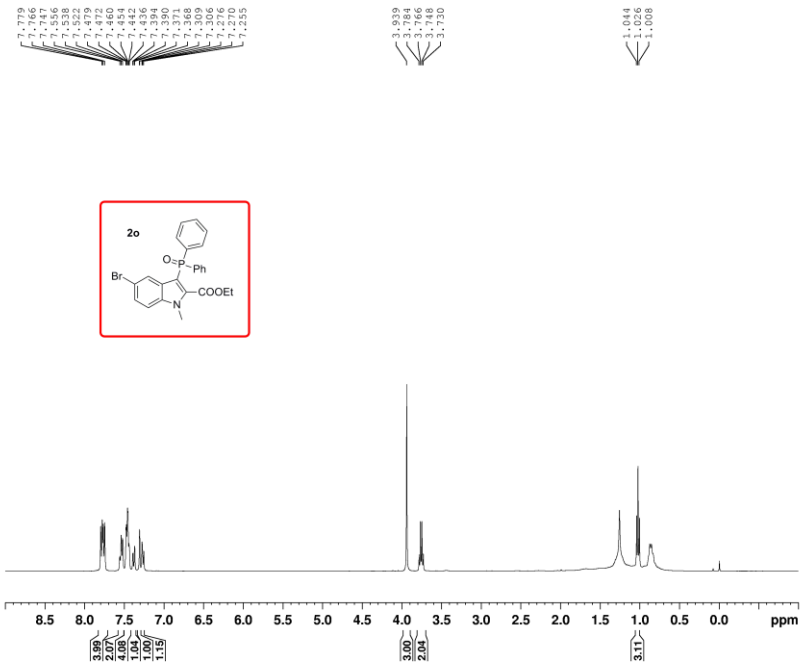
======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                     80.00 usec
PL2                       -2.10 dB
PL12                      13.90 dB
PL13                      13.90 dB
PL2W                17.72106263 W
PL12W               0.44513249 W
PL13W               0.44513249 W
SFO2                400.1316005 MHz
SI                        32768
SF                  100.6127465 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```




```
NAME          zhouax120331-P-Br
EXPNO                       11
PROCNO                       1
Date_              20120331
Time                    14.53
INSTRUM                 spect
PROBHD   5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                 CDCl3
NS                          16
DS                           4
SWH                64102.563 Hz
FIDRES              0.978127 Hz
AQ                  0.5112308 sec
RG                        2050
DW                     7.800 usec
DE                     6.50 usec
TE                     295.9 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                          1

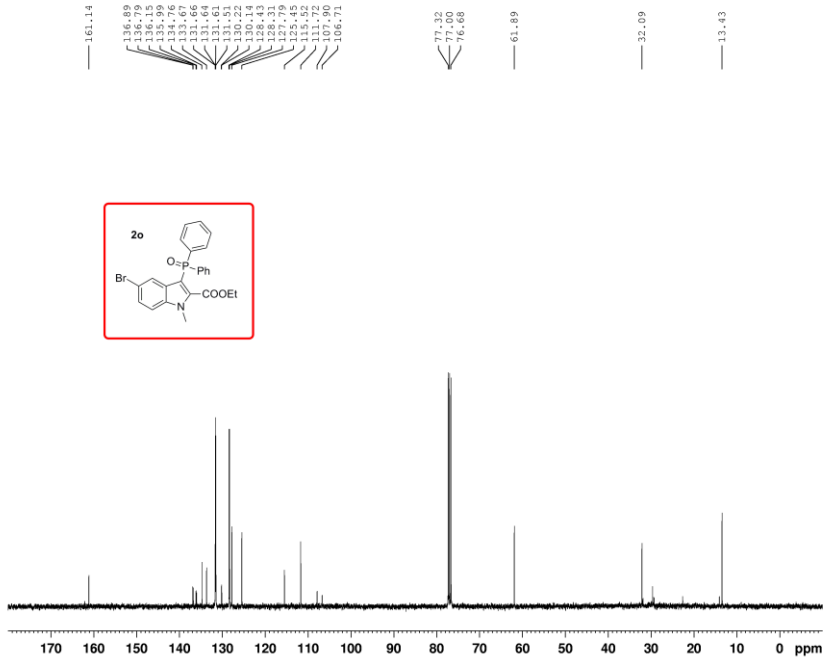
======== CHANNEL f1 ========
NUC1                       31P
P1                       10.80 usec
PL1                       0.00 dB
PL1W               26.12361908 W
SFO1              161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    80.00 usec
PL2                      -2.10 dB
PL12                     13.90 dB
PL13                     13.90 dB
PL2W               17.72104263 W
PL12W               0.44513249 W
PL13W               0.44513249 W
SFO2              400.1316005 MHz
SI                        32768
SF                161.9755930 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```




```
NAME          zhouax120331-P-Br
EXPNO                       10
PROCNO                       1
Date_              20120331
Time                    14.51
INSTRUM                 spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg30
TD                       65536
SOLVENT                 CDCl3
NS                          16
DS                           2
SWH                8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9846387 sec
RG                        71.8
DW                    60.800 usec
DE                     6.50 usec
TE                     295.3 K
D1                1.00000000 sec
TD0                          1

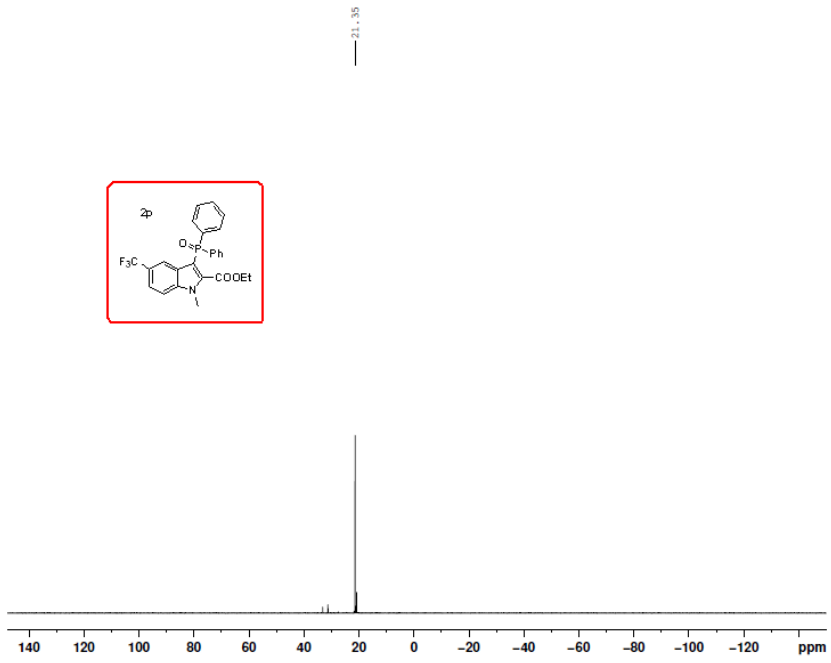
======== CHANNEL f1 ========
NUC1                       1H
P1                       14.70 usec
PL1                      -1.00 dB
PL1W               13.75590801 W
SFO1              400.1324710 MHz
SI                        32768
SF                400.1300011 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```




```
NAME            zhouax120331-P-Br
EXPNO                 12
PROCNO                 1
Date_           20120331
Time                15.03
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   120
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                    64
DW                20.800 usec
DE                 6.50 usec
TE                296.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.70 usec
PL1               -2.00 dB
PL1W        56.13311005 W
SFO1       100.6228298 MHz

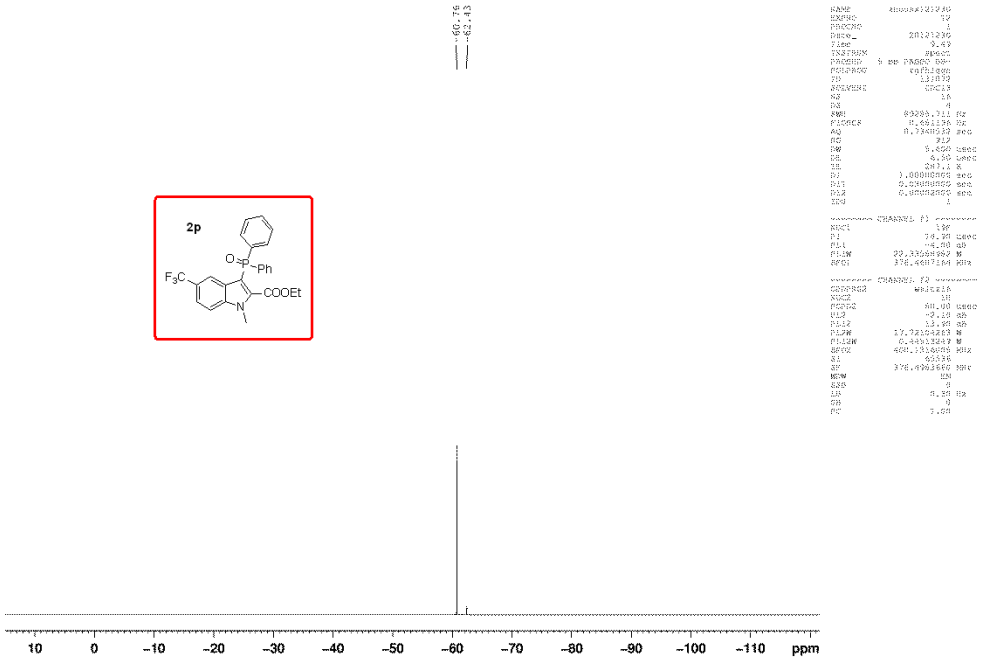
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             80.00 usec
PL2               -2.10 dB
PL12              13.90 dB
PL13              13.90 dB
PL2W        17.72104263 W
PL12W        0.44513249 W
PL13W        0.44513249 W
SFO2       400.1316005 MHz
SI                 32768
SF         100.6127744 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

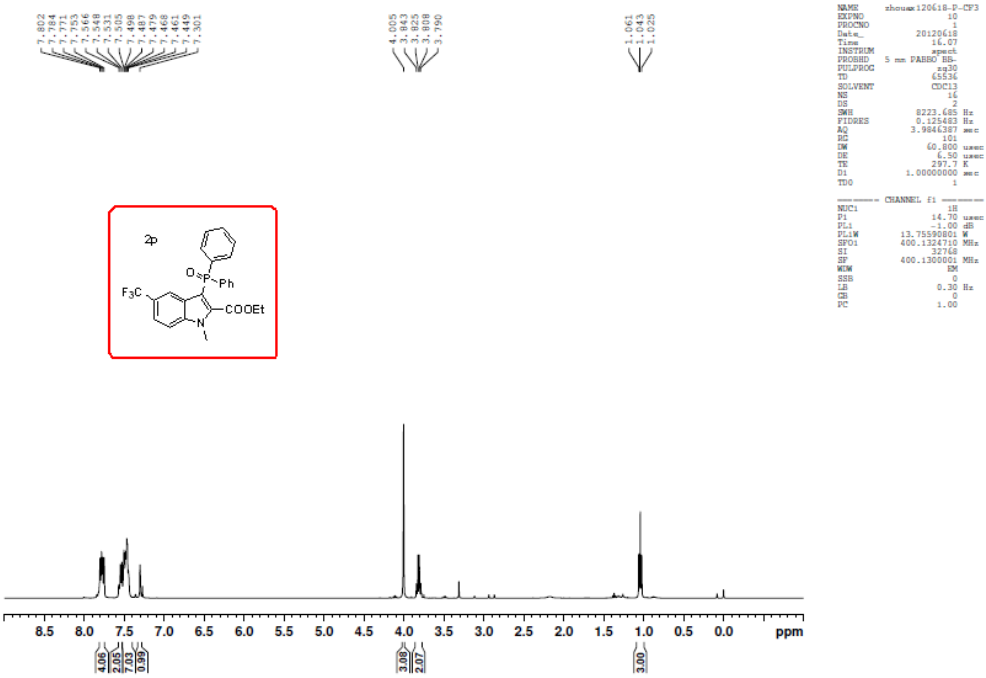


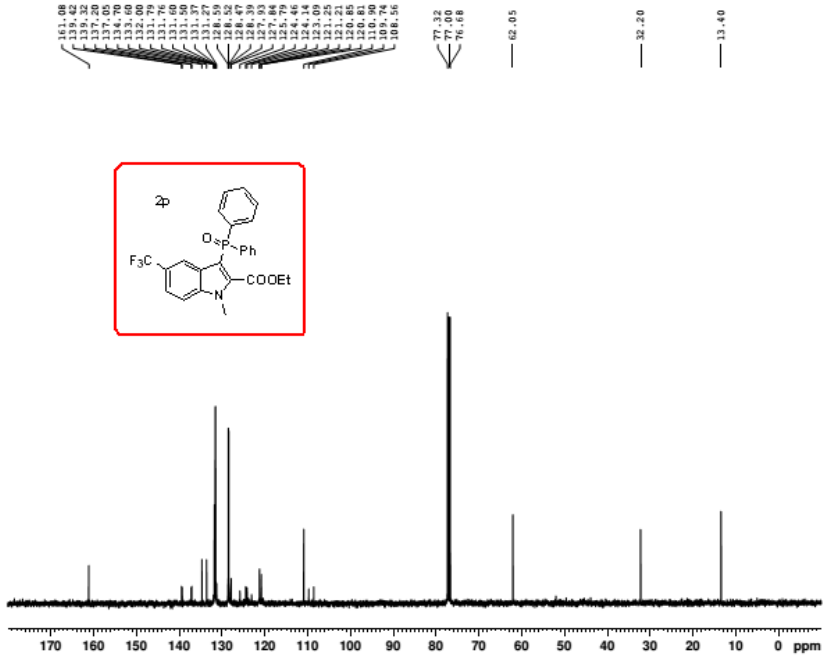

```
NAME            zhouax120618-P-CF3
EXPNO                 11
PROCNO                 1
Date_           20120618
Time                16.10
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5112308 sec
RG                  2050
DW                 7.800 usec
DE                 6.50 usec
TE                298.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                31P
P1                10.80 usec
PL1                0.00 dB
PL1W        26.12361908 W
SFO1       161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             80.00 usec
PL2               -2.10 dB
PL12              13.90 dB
PL13              13.90 dB
PL2W        17.72104263 W
PL12W        0.44513249 W
PL13W        0.44513249 W
SFO2       400.1316005 MHz
SI                 32768
SF         161.9755930 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```


-60.76
-62.43
2p
F3C
O
P
Ph
COOEt
N
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
ppm

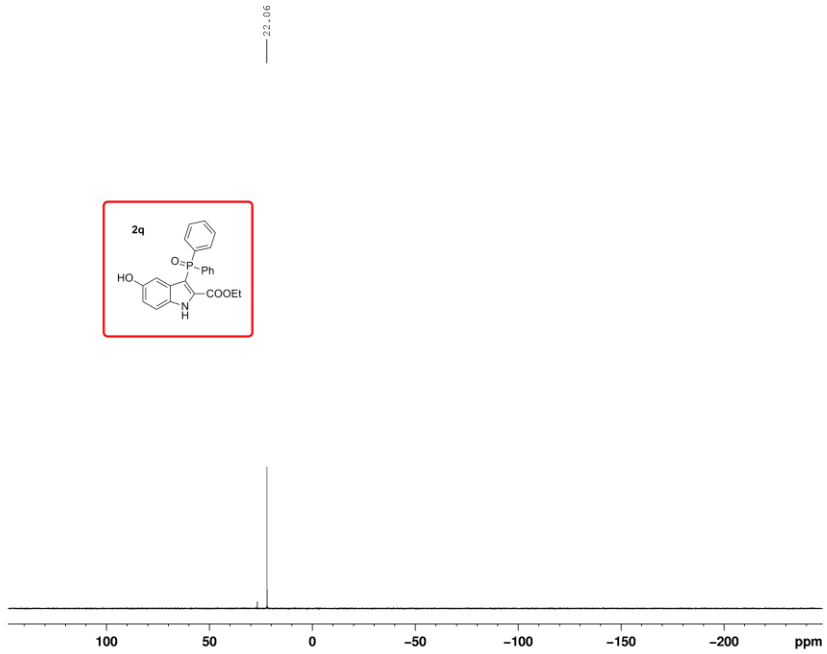

7.802
7.784
7.771
7.755
7.566
7.548
7.531
7.505
7.498
7.479
7.468
7.461
7.445
7.301
4.005
3.843
3.825
3.808
3.790
1.061
1.043
1.025
2p
F3C
O
P
Ph
COOEt
N
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
4.06
2.05
2.03
0.99
3.08
2.07
3.00




```
NAME            zhouax120618-P-CF3
EXPNO                   12
PROCNO                   1
Date_           20120618
Time                 16.21
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     160
DS                       4
SWH              24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                      57
DW                  20.800 usec
DE                   6.50 usec
TE                  299.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    13C
P1                    9.70 usec
PL1                  -2.00 dB
PL1W            56.13311005 W
SFO1           100.6228298 MHz

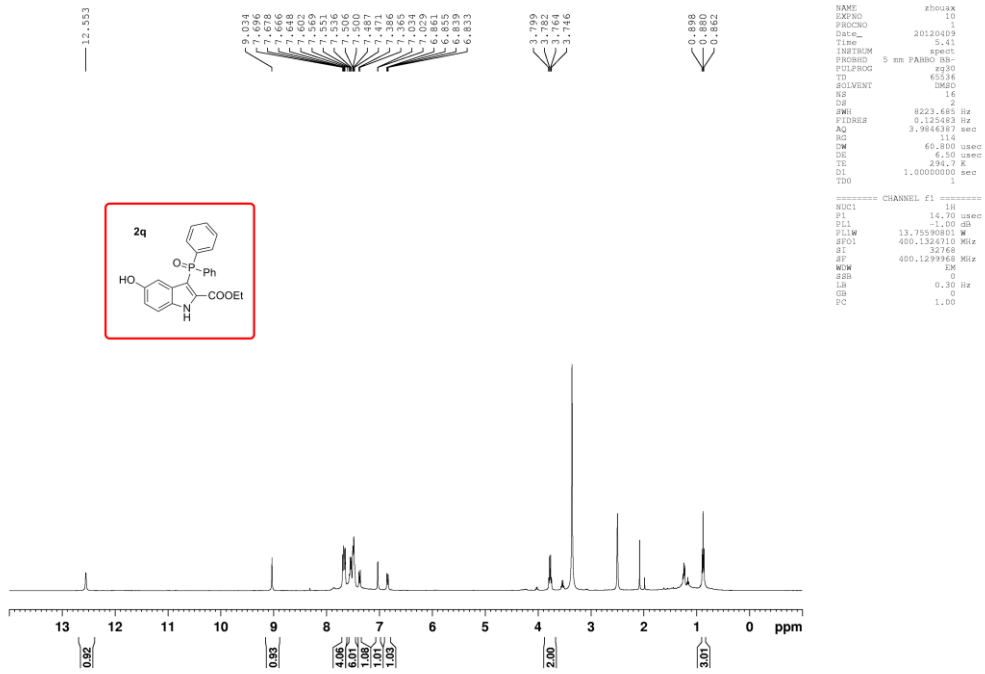
======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -2.10 dB
PL12                 13.90 dB
PL13                 13.90 dB
PL2W            17.72104263 W
PL12W            0.44513249 W
PL13W            0.44513249 W
SFO2           400.1316005 MHz
SI                    32768
SF             100.6127722 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```




```
NAME                 zhouax
EXPNO                    12
PROCNO                    1
Date_            20120409
Time                  6.00
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT               DMSO
NS                       16
DS                        4
SWH              64102.563 Hz
FIDRES           0.978127 Hz
AQ               0.5112308 sec
RG                     2050
DW                  7.800 usec
DE                   6.50 usec
TE                  295.8 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

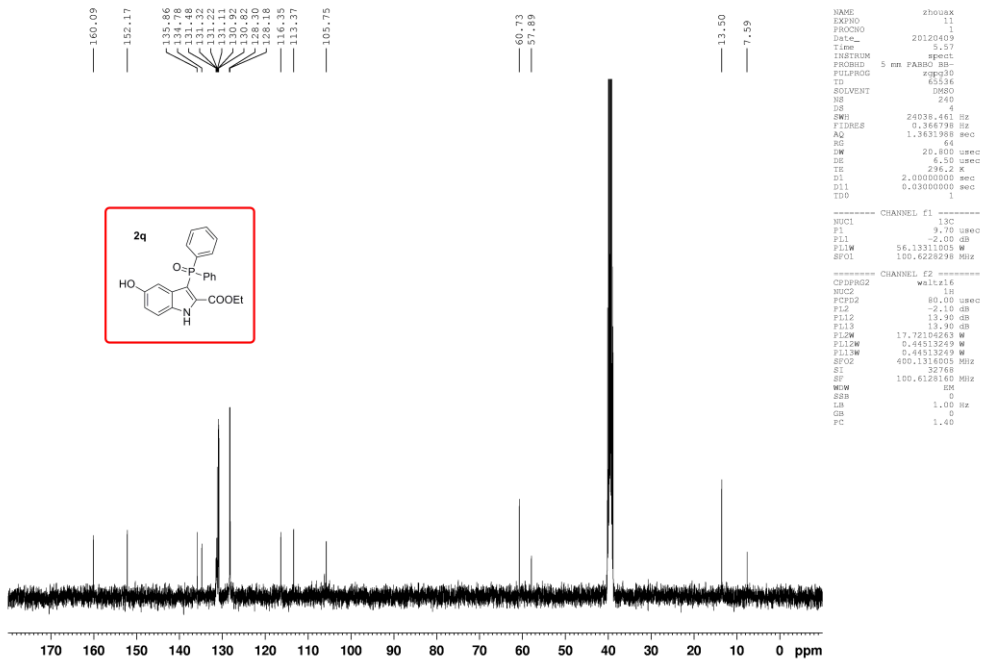
======== CHANNEL f1 ========
NUC1                    31P
P1                   10.80 usec
PL1                   0.00 dB
PL1W            26.12361908 W
SFO1           161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -2.10 dB
PL12                 13.90 dB
PL13                 13.90 dB
PL2W            17.72104263 W
PL12W            0.44513249 W
PL13W            0.44513249 W
SFO2           400.1316005 MHz
SI                    32768
SF             161.9755930 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```




```
NAME                zhouax
EXPNO                   10
PROCNO                   1
Date_             20120409
Time                  5.41
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                       16
DS                        2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                      114
DW                 60.800 usec
DE                  6.50 usec
TE                  294.7 K
D1                1.00000000 sec
TD0                      1

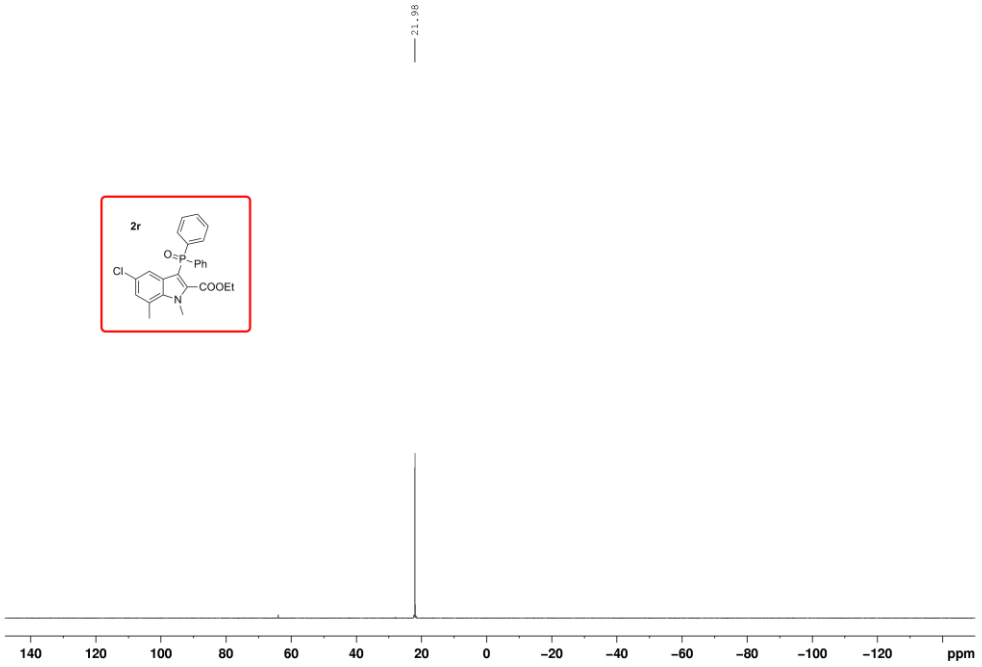
======== CHANNEL f1 ========
NUC1                    1H
P1                   14.70 usec
PL1                  -1.00 dB
PL1W           13.75590801 W
SFO1           400.1324710 MHz
SI                    32768
SF             400.1299968 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

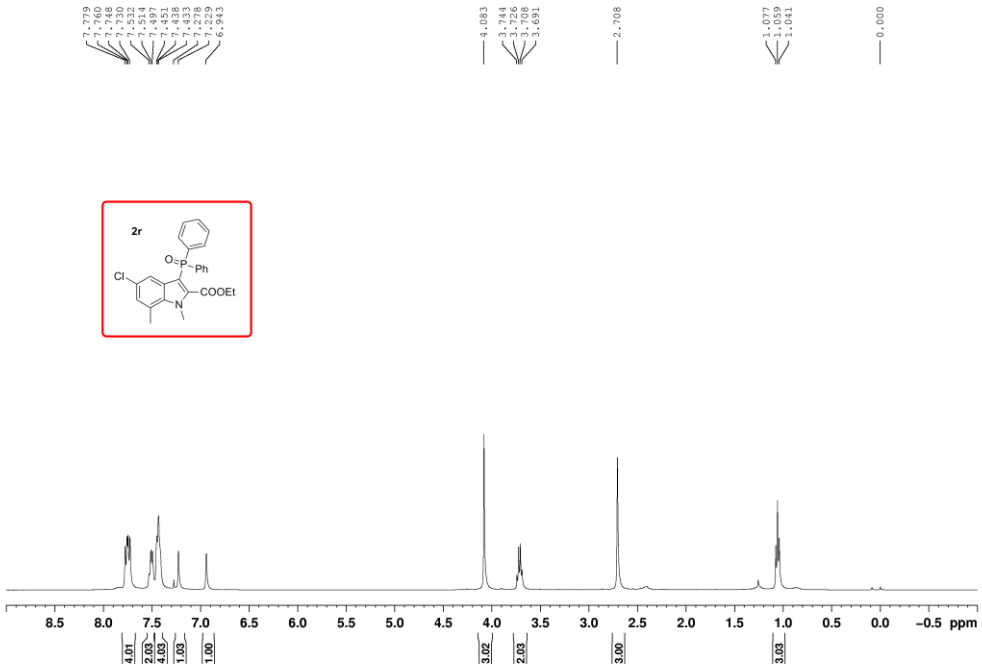


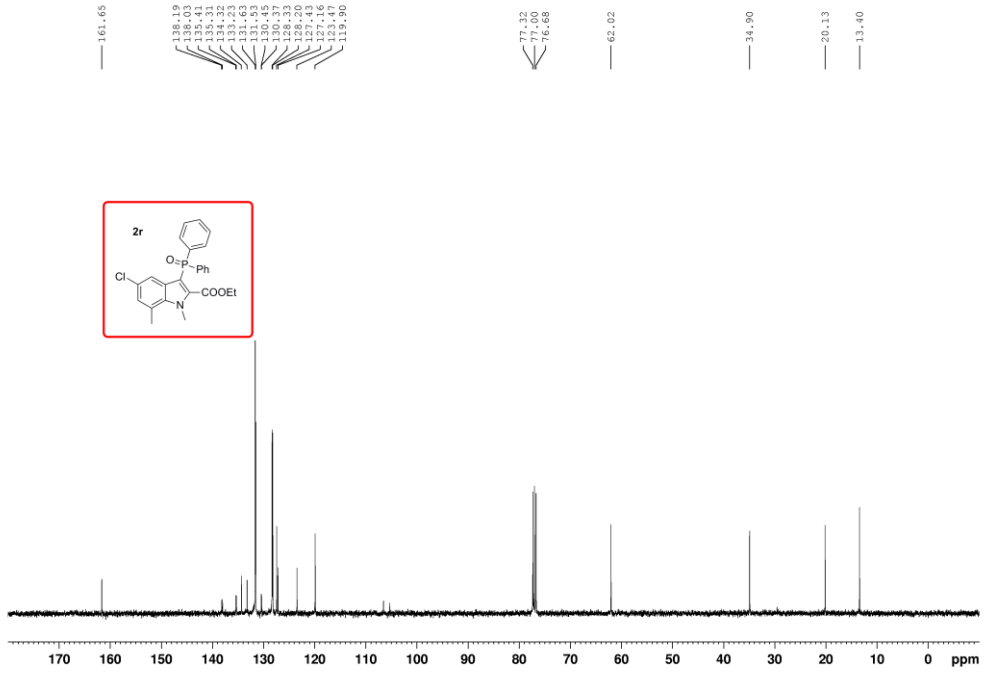

```
NAME                zhouax
EXPNO                   11
PROCNO                   1
Date_             20120409
Time                  5.57
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT                DMSO
NS                      240
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                       64
DW                 20.800 usec
DE                  6.50 usec
TE                  296.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      1

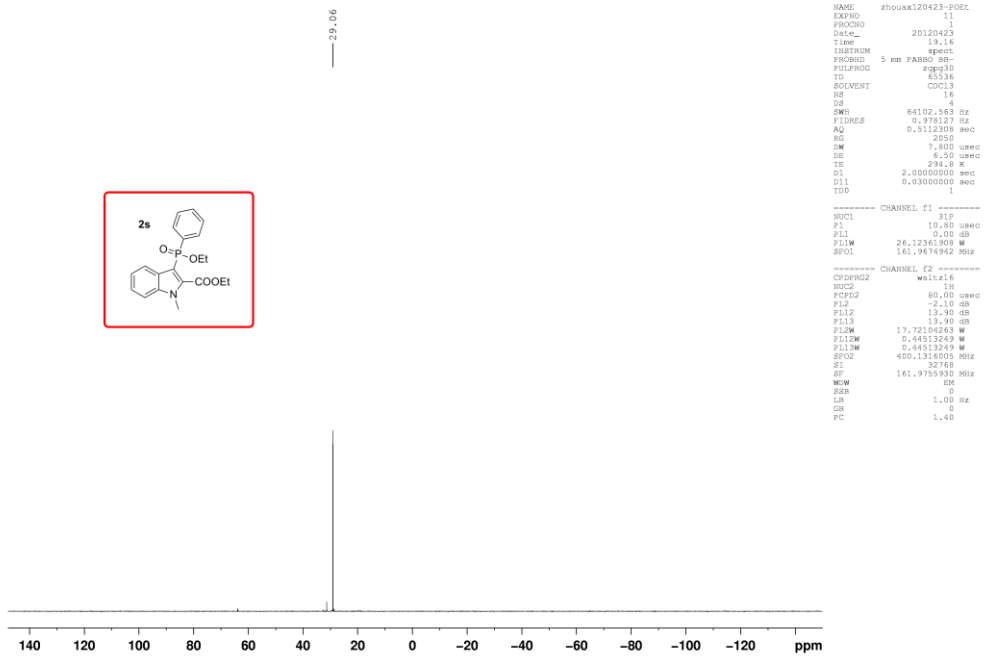
======== CHANNEL f1 ========
NUC1                    13C
P1                    9.70 usec
PL1                  -2.00 dB
PL1W           56.13311005 W
SFO1           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -2.10 dB
PL12                 13.90 dB
PL13                 13.90 dB
PL2W           17.72106263 W
PL12W           0.44513249 W
PL13W           0.44513249 W
SFO2           400.1316005 MHz
SI                    32768
SF             100.6128160 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```


21.98
2r
Cl
O
P
Ph
COOEt
N
140
120
100
80
60
40
20
0
−20
−40
−60
−80
−100
−120
ppm


7.779
7.760
7.748
7.730
7.532
7.514
7.497
7.451
7.438
7.433
7.278
7.229
6.943
4.083
3.744
3.726
3.708
3.691
2.708
1.077
1.059
1.041
0.000
2r
Cl
O
P
Ph
COOEt
N
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
ppm
4.01
2.03
4.03
1.03
1.00
3.02
2.03
3.00
3.03

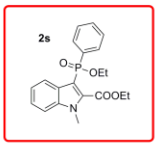


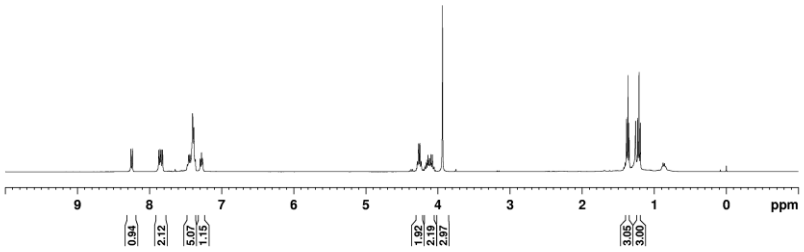



```
NAME          zhouax120423-POEt
EXPNO                        11
PROCNO                        1
Date_                  20120423
Time                      19.16
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          4
SWH                 64102.563 Hz
FIDRES               0.978127 Hz
AQ                   0.5112308 sec
RG                        2050
DW                     7.800 usec
DE                     6.50 usec
TE                     294.8 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       31P
P1                       10.80 usec
PL1                       0.00 dB
PL1W               26.12361908 W
SFO1               161.9674942 MHz

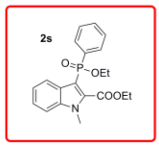
======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    80.00 usec
PL2                      -2.10 dB
PL12                     13.90 dB
PL13                     13.90 dB
PL2W               17.72106263 W
PL12W               0.44513249 W
PL13W               0.44513249 W
SFO2               400.1316005 MHz
SI                        32768
SF                 161.9755930 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```

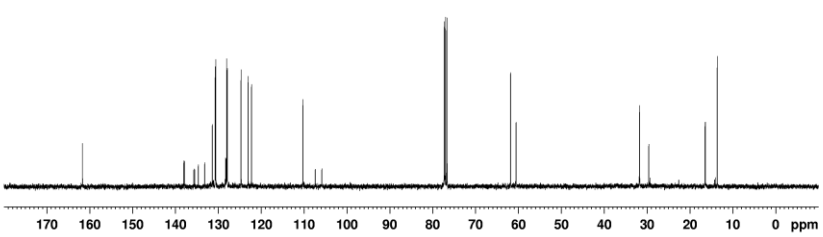





```
NAME          zhouax120423-POEt
EXPNO                        10
PROCNO                        1
Date_               20120423
Time                     19.13
INSTRUM                  spect
PROBHD   5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9846387 sec
RG                          64
DW                     60.800 usec
DE                      6.50 usec
TE                     293.9 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                     14.70 usec
PL1                    -1.00 dB
PL1W             13.75590801 W
SFO1             400.1324710 MHz
SI                       32768
SF               400.1300023 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```

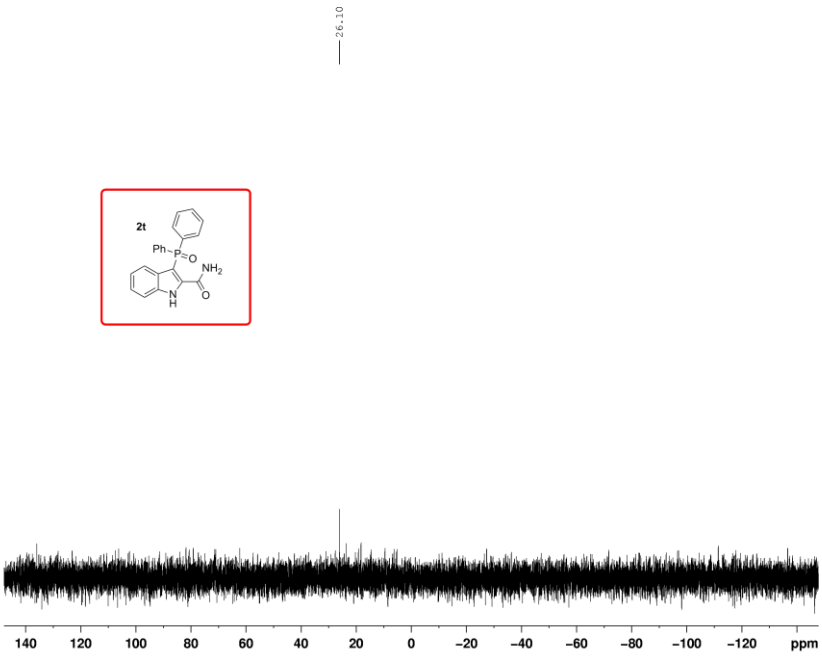


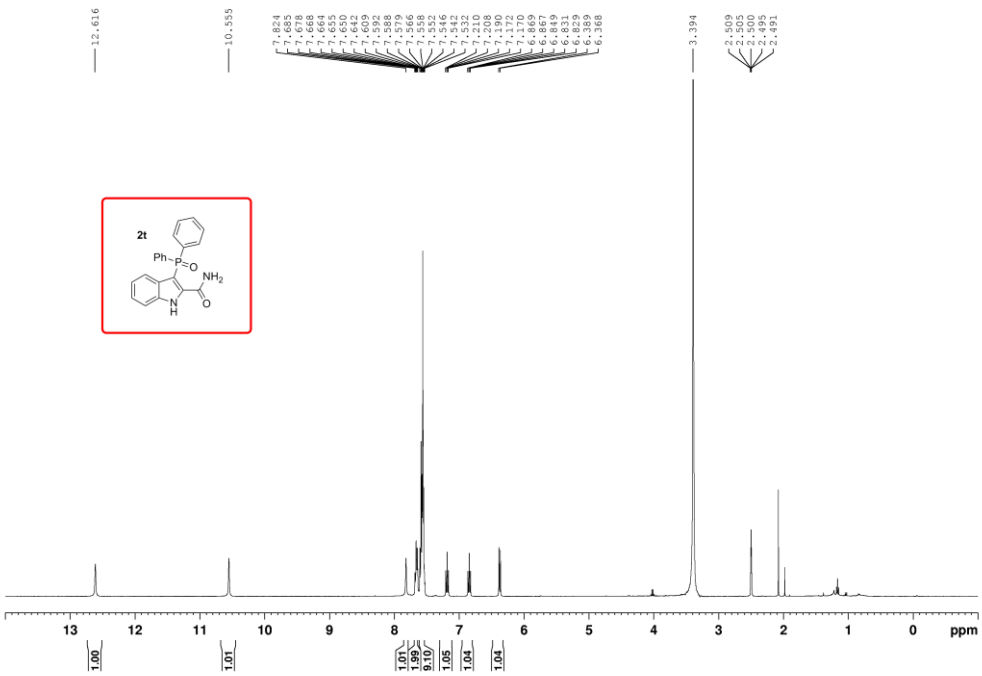


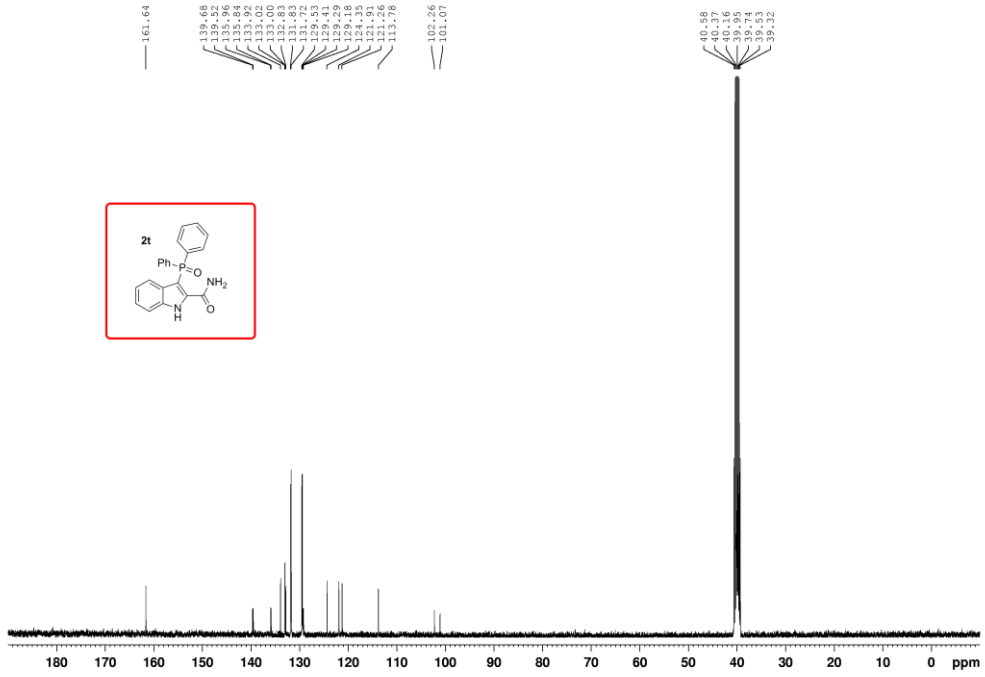

```
NAME          zhouax120423-POEt
EXPNO                        12
PROCNO                        1
Date_               20120423
Time                     19.24
INSTRUM                  spect
PROBHD   5 mm PABBO BB-
PULPROG                zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         120
DS                           4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631988 sec
RG                         912
DW                     20.800 usec
DE                      6.50 usec
TE                     295.3 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

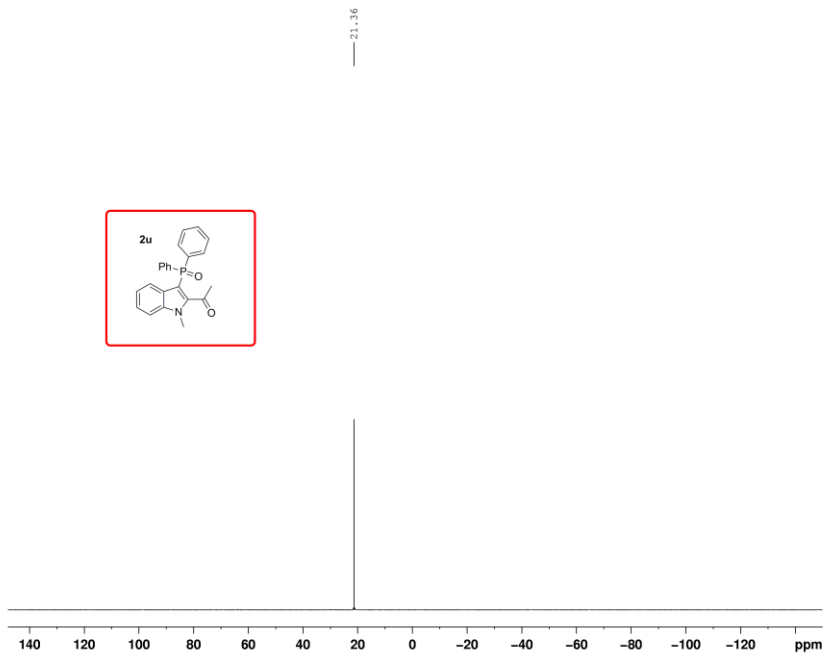
======== CHANNEL f1 ========
NUC1                        13C
P1                      9.70 usec
PL1                    -2.00 dB
PL1W             56.13311005 W
SFO1             100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                  80.00 usec
PL2                    -2.10 dB
PL12                   13.90 dB
PL13                   13.90 dB
PL2W             17.72106263 W
PL12W             0.44513249 W
PL13W             0.44513249 W
SFO2             400.1316005 MHz
SI                       32768
SF               100.6127780 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```


26.10
2t
Ph
P
O
NH2
N
H
O
140
120
100
80
60
40
20
0
−20
−40
−60
−80
−100
−120
ppm


12.616
10.555
7.824
7.685
7.678
7.668
7.664
7.655
7.650
7.643
7.609
7.592
7.588
7.579
7.566
7.558
7.552
7.546
7.542
7.532
7.210
7.208
7.190
7.172
7.170
6.869
6.867
6.849
6.831
6.389
6.368
3.394
2.509
2.505
2.500
2.495
2.491
2t
Ph
P
O
NH2
N
H
O
13
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.01
1.01
1.99
9.10
1.05
1.04
1.04

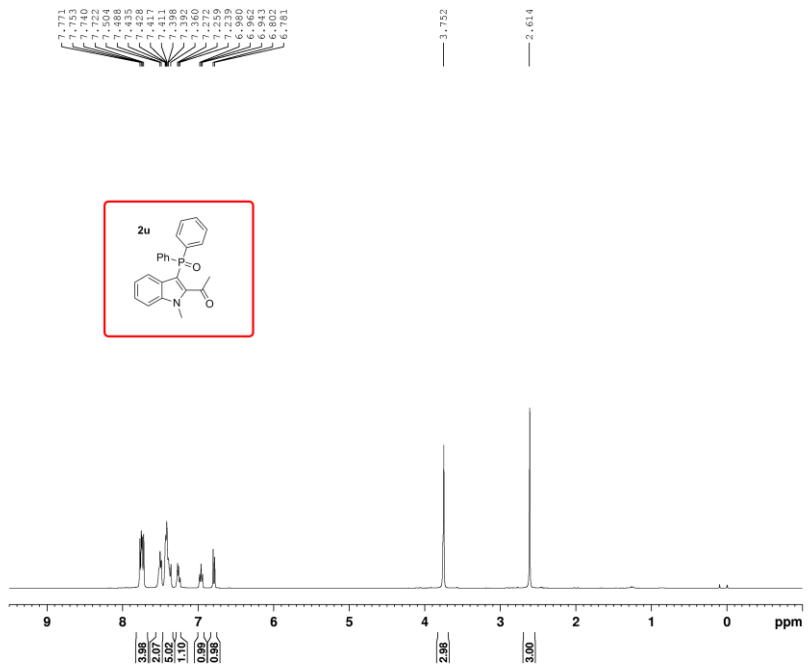





```
NAME          zhousx120423-P-Ac
EXPNO                        11
PROCNO                        1
Date_                  20120423
Time                      19.33
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           4
SWH                  64102.563 Hz
FIDRES               0.978127 Hz
AQ                   0.5112308 sec
RG                        2050
DW                       7.800 usec
DE                       6.50 usec
TE                      294.6 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          1

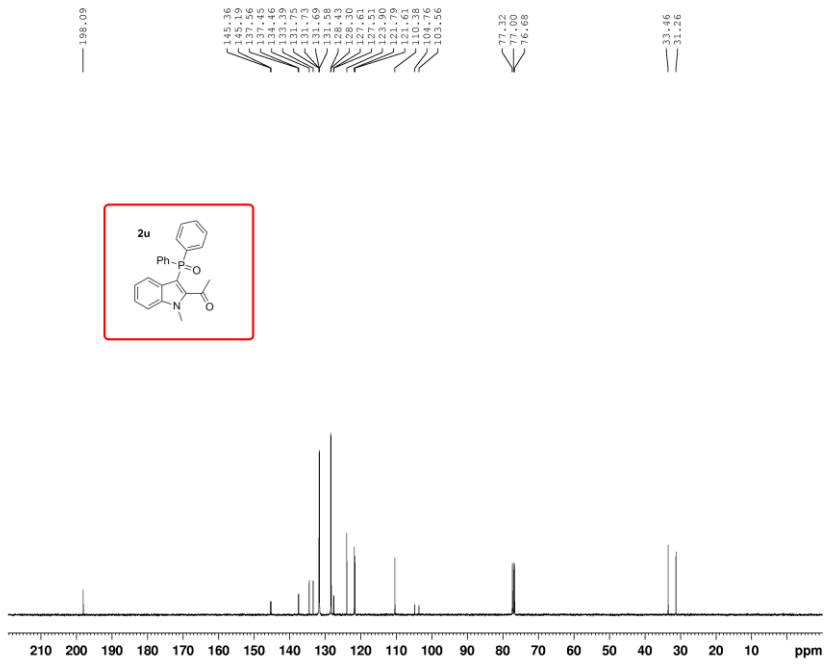
======== CHANNEL f1 ========
NUC1                       31P
P1                       10.80 usec
PL1                       0.00 dB
PL1W                26.12361908 W
SFO1               161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                      -2.10 dB
PL12                     13.90 dB
PL13                     13.90 dB
PL2W                17.72104263 W
PL12W                0.44513249 W
PL13W                0.44513249 W
SFO2               400.1316005 MHz
SI                        32768
SF                 161.9755930 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```




```
NAME        zhouax120423-P-Ac
EXPNO               10
PROCNO               1
Date_         20120423
Time             19.31
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG          zg30
TD              65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                50.8
DW               60.800 usec
DE                6.50 usec
TE              293.8 K
D1            1.00000000 sec
TD0                  1

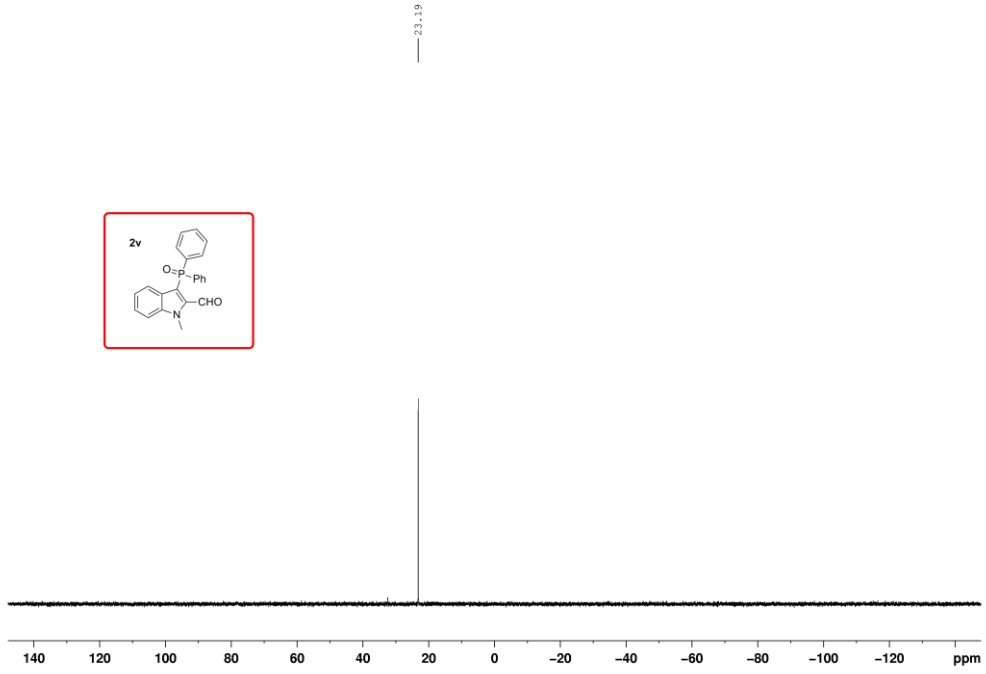
======== CHANNEL f1 ========
NUC1                1H
P1               14.70 usec
PL1              -1.00 dB
PL1W        13.75590801 W
SFO1        400.1324710 MHz
SI                32768
SF          400.1300001 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

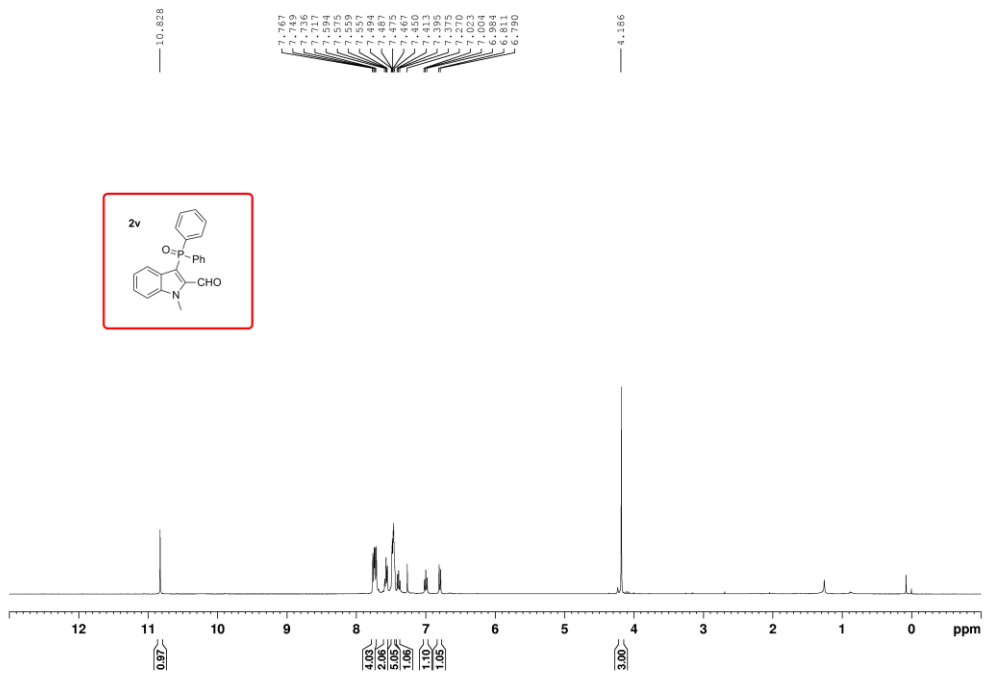


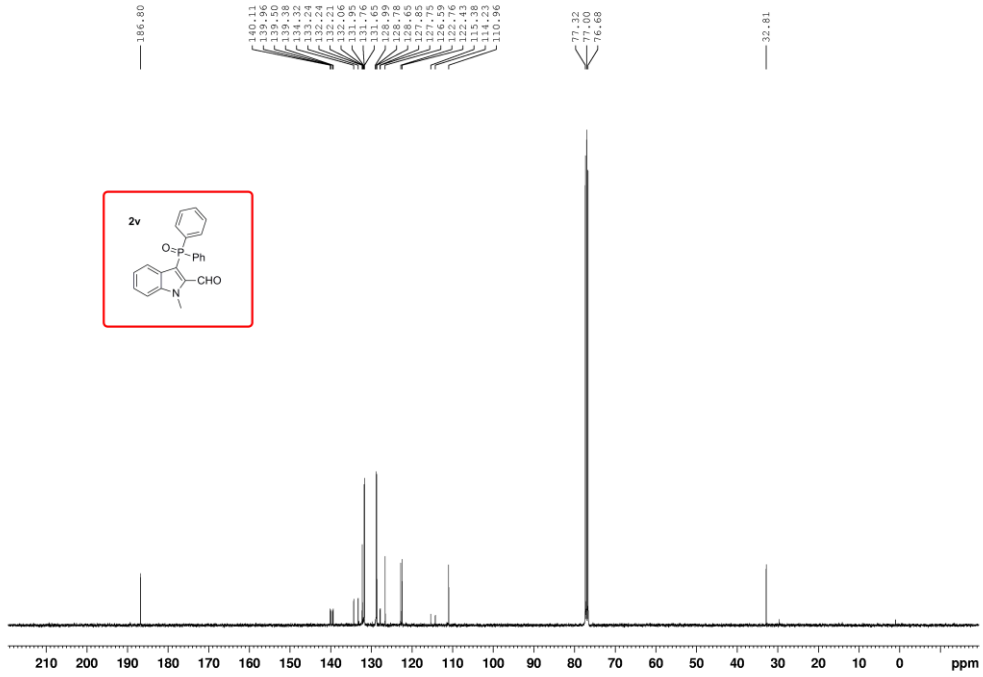

```
NAME        zhouax120423-P-Ac
EXPNO               12
PROCNO               1
Date_         20120423
Time             19.42
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG        zgpg30
TD              65536
SOLVENT          CDCl3
NS                 120
DS                   4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                1030
DW               20.800 usec
DE                6.50 usec
TE              295.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1

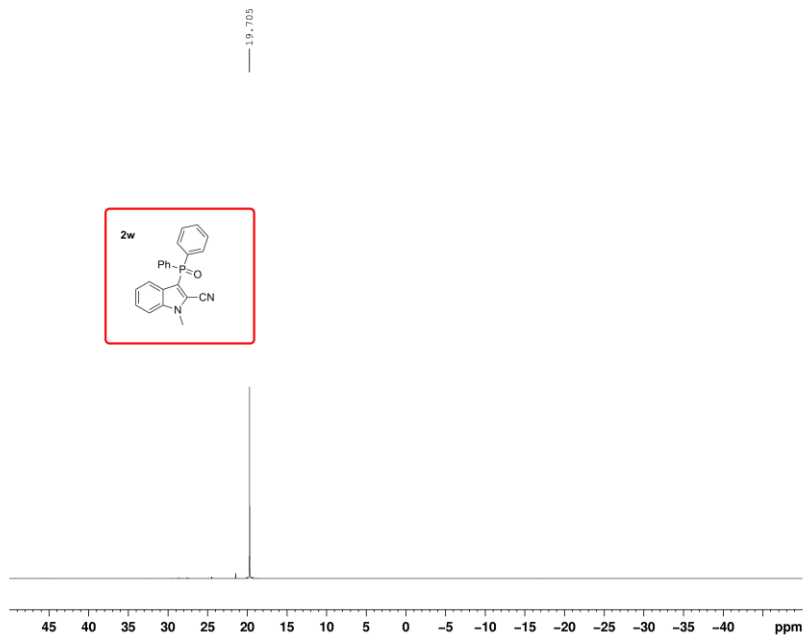
======== CHANNEL f1 ========
NUC1                13C
P1                9.70 usec
PL1              -2.00 dB
PL1W        56.13311005 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             80.00 usec
PL2              -2.10 dB
PL12             13.90 dB
PL13             13.90 dB
PL2W        17.72106263 W
PL12W        0.44513249 W
PL13W        0.44513249 W
SFO2        400.1316005 MHz
SI                32768
SF          100.6127876 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```


23.19
2v
O
P
Ph
CHO
N
140
120
100
80
60
40
20
0
−20
−40
−60
−80
−100
−120
ppm


10.828
7.767
7.749
7.736
7.717
7.594
7.575
7.559
7.537
7.488
7.487
7.475
7.467
7.450
7.431
7.395
7.375
7.270
7.053
7.004
6.984
6.811
6.790
4.186
2v
O
P
Ph
CHO
N
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.97
4.03
2.06
5.05
1.06
1.10
1.05
3.00

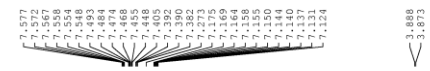


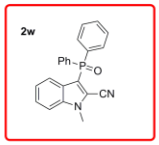


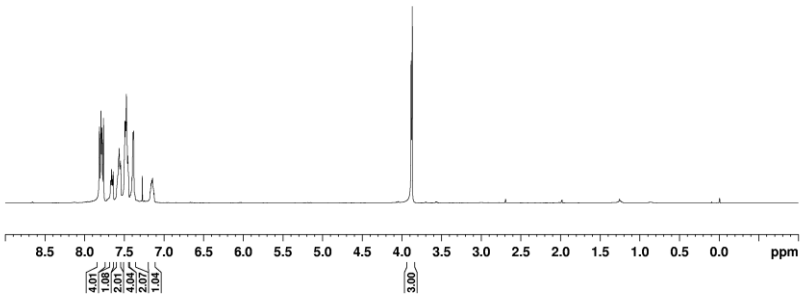

```
NAME            zhouax120302-CN-H
EXPNO                         12
PROCNO                         1
Date_                   20120302
Time                       20.17
INSTRUM                    spect
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                            16
DS                             4
SWH                    64102.563 Hz
FIDRES                 0.978127 Hz
AQ                     0.5112308 sec
RG                          2050
DW                        7.800 usec
DE                        6.50 usec
TE                        293.7 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         31P
P1                         10.80 usec
PL1                         0.00 dB
PL1W               26.12361908 W
SFO1               161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                     80.00 usec
PL2                       -2.10 dB
PL12                      13.90 dB
PL13                      13.90 dB
PL2W               17.72104263 W
PL12W               0.44513249 W
PL13W               0.44513249 W
SFO2               400.1316005 MHz
SI                         32768
SF                 161.9755930 MHz
WDW                           EM
SSB                            0
LB                         1.00 Hz
GB                             0
PC                          1.40
```

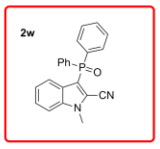


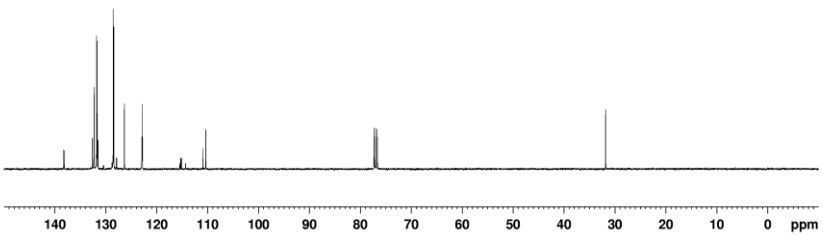





```
NAME          zhouax120302-CN-H
EXPNO                       10
PROCNO                       1
Date_               20120302
Time                    20.06
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                          16
DS                           2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9846387 sec
RG                          57
DW                     60.800 usec
DE                      6.50 usec
TE                     292.7 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                      14.70 usec
PL1                     -1.00 dB
PL1W               13.75590801 W
SFO1               400.1324710 MHz
SI                       32768
SF                 400.1300000 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```

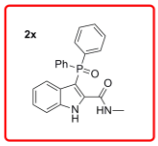




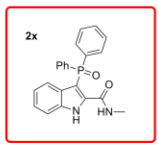

```
NAME          zhouax120302-CN-H
EXPNO                       11
PROCNO                       1
Date_               20120302
Time                    20.14
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                         120
DS                           4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631988 sec
RG                         812
DW                     20.800 usec
DE                      6.50 usec
TE                     294.2 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                       9.70 usec
PL1                     -2.00 dB
PL1W               56.13311005 W
SFO1               100.6228298 MHz

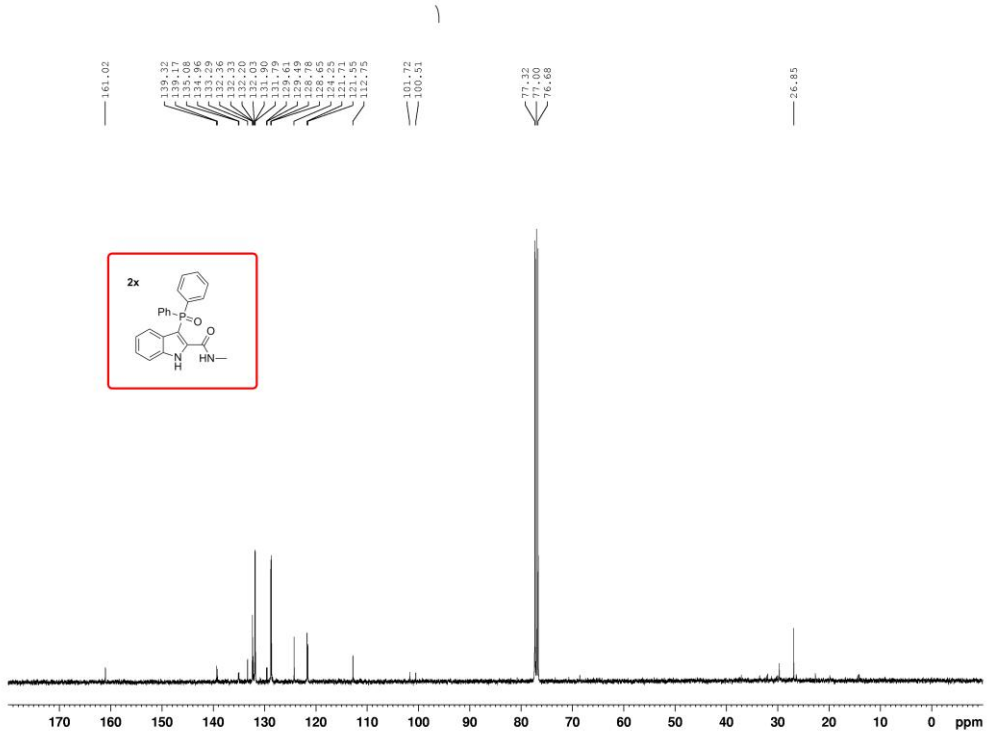
======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                   80.00 usec
PL2                     -2.10 dB
PL12                    13.90 dB
PL13                    13.90 dB
PL2W               17.72106263 W
PL12W               0.44513249 W
PL13W               0.44513249 W
SFO2               400.1316005 MHz
SI                       32768
SF                 100.6127913 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

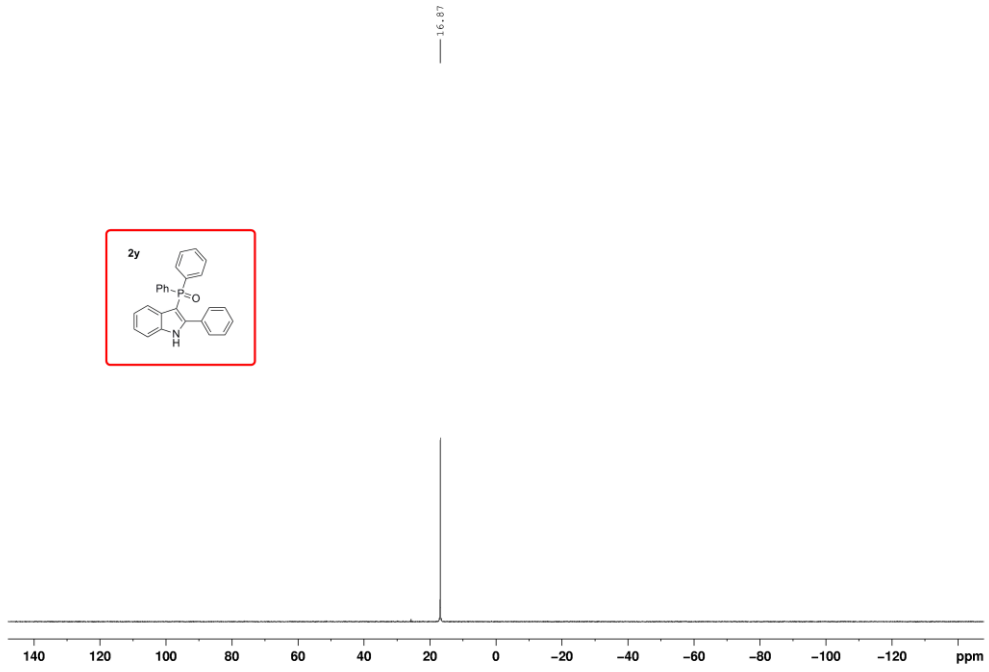

2x
Ph
P
O
O
N
H
HN—

28.39
140
120
100
80
60
40
20
0
−20
−40
−60
−80
−100
−120
ppm

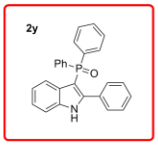

2x
Ph
P
O
O
N
H
HN—

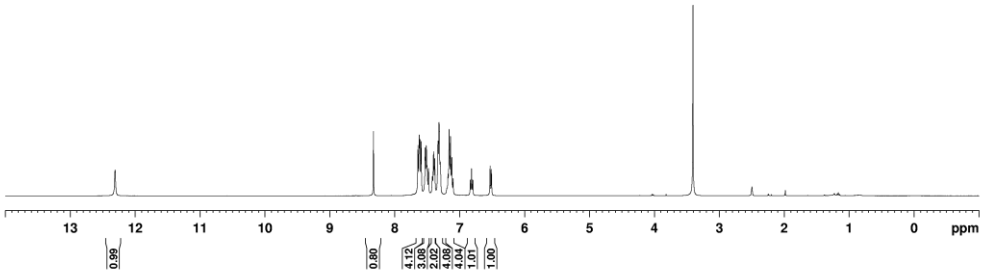
11.434
11.285
7.725
7.706
7.693
7.674
7.614
7.592
7.571
7.554
7.481
7.474
7.462
7.456
7.449
7.259
7.237
7.218
6.922
6.892
6.873
6.580
6.560
3.074
3.064
0.000
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.91
4.06
3.04
4.04
1.28
1.01
1.00
3.04


161.02
139.32
138.17
135.08
134.96
133.29
132.36
132.33
132.20
132.03
131.90
131.79
131.64
129.49
128.78
128.65
124.95
121.71
121.55
112.75
101.72
100.51
77.32
77.00
76.68
26.85
2x
Ph
P
O
O
N
H
HN
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


16.87
2y
Ph
P
O
N
H
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
ppm


12.310
8.332
7.646
7.627
7.617
7.598
7.535
7.518
7.506
7.484
7.424
7.406
7.389
7.356
7.323
7.305
7.198
7.183
7.164
7.145
7.125
7.106
6.839
6.820
6.801
6.535
6.514
3.409
2.500


2y
Ph
P
O
H
N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10
9
8
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6
5
4
3
2
1
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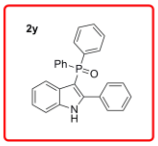
0.80
4.12
3.08
2.02
4.06
4.04
1.01
1.00


147.22
147.04
137.07
136.95
136.02
134.96
134.00
131.85
131.61
131.52
130.17
130.06
130.01
128.82
128.70
128.58
128.37
122.54
120.69
120.66
112.33
101.66
100.42
79.61
40.58
40.37
40.16
39.95
39.74
39.53
39.32


2y
Ph
P
O
H
N

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120
110
100
90
80
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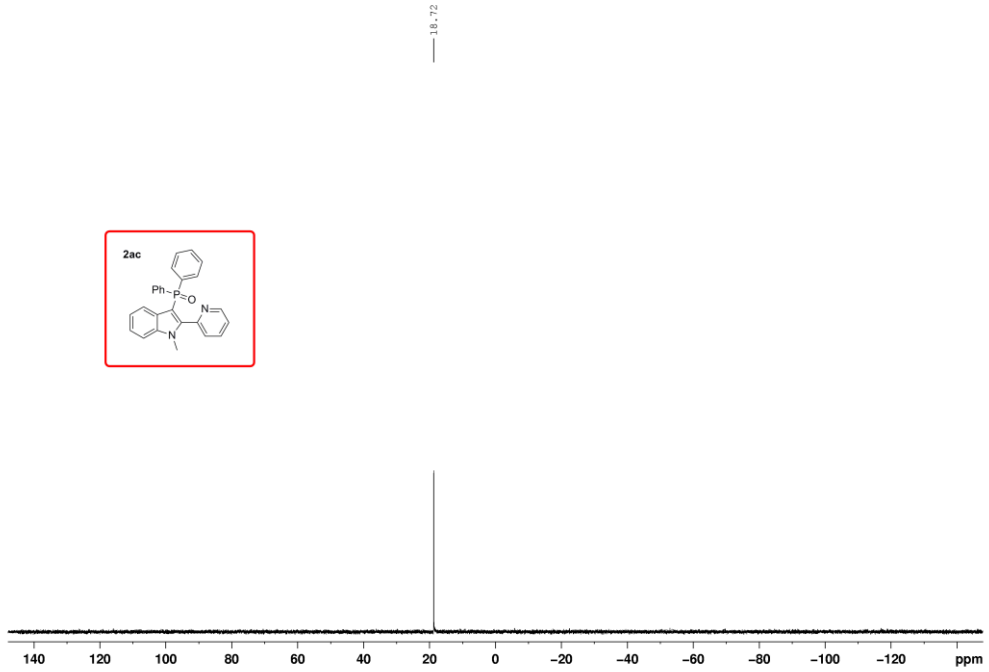
60
50
40
30
20
10
0
ppm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2ac
Ph
P
O
N
N
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120
100
80
60
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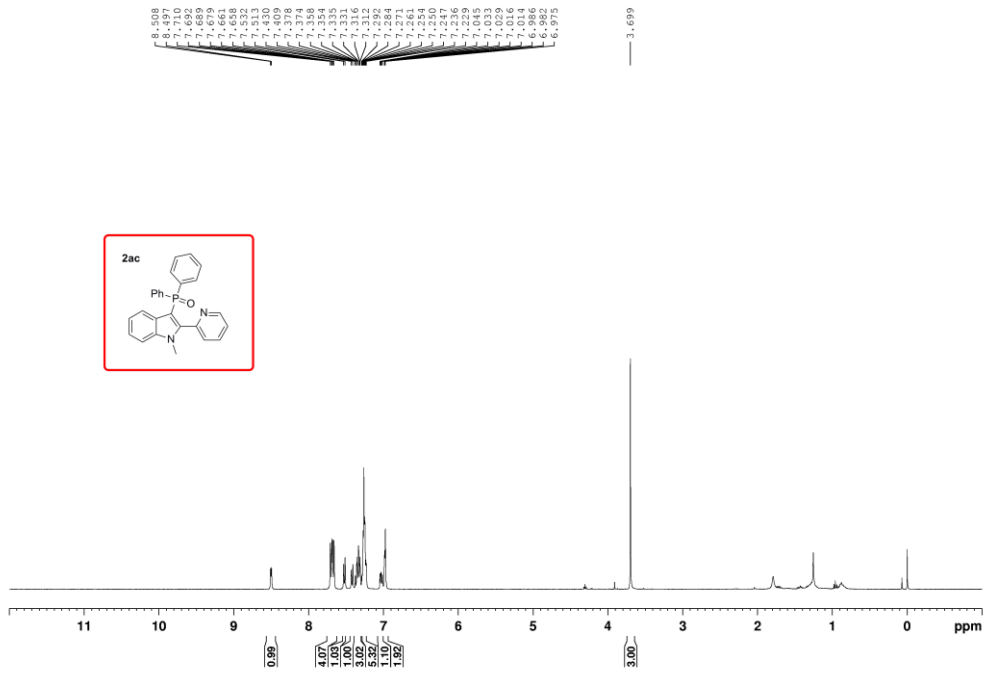
−80
−100
−120
ppm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7.513
7.430
7.409
7.378
7.374
7.358
7.354
7.335
7.331
7.316
7.312
7.292
7.284
7.271
7.261
7.254
7.250
7.247
7.236
7.229
7.045
7.033
7.029
7.016
7.014
6.986
6.982
6.975
3.699
2ac
Ph
P
O
N
N
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.98
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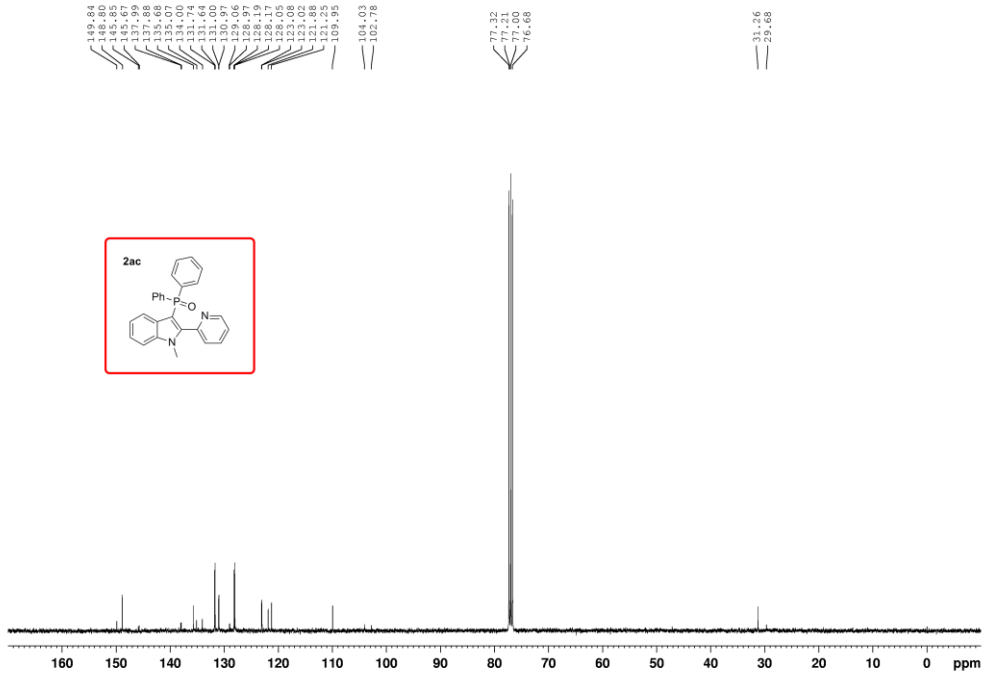
1.03
1.00
3.02
5.92
1.10
1.92
3.00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148.80
145.85
145.67
137.99
137.88
135.68
135.07
134.00
131.74
131.66
131.00
130.97
129.06
128.97
128.19
128.17
128.05
123.08
123.02
121.88
121.25
109.95
104.03
102.78
77.32
77.21
77.00
76.68
31.26
29.68
2ac
Ph
P
O
N
N
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150
140
130
120
110
100
90
80
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60
50
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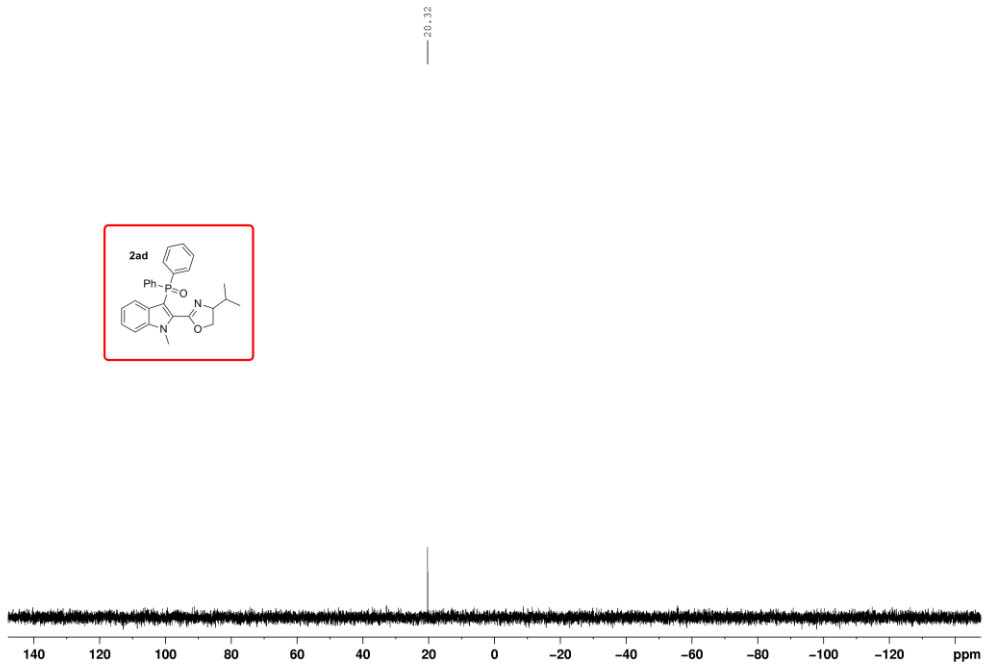
30
20
10
0
ppm


20.32
2ad
Ph
P
O
N
N
O
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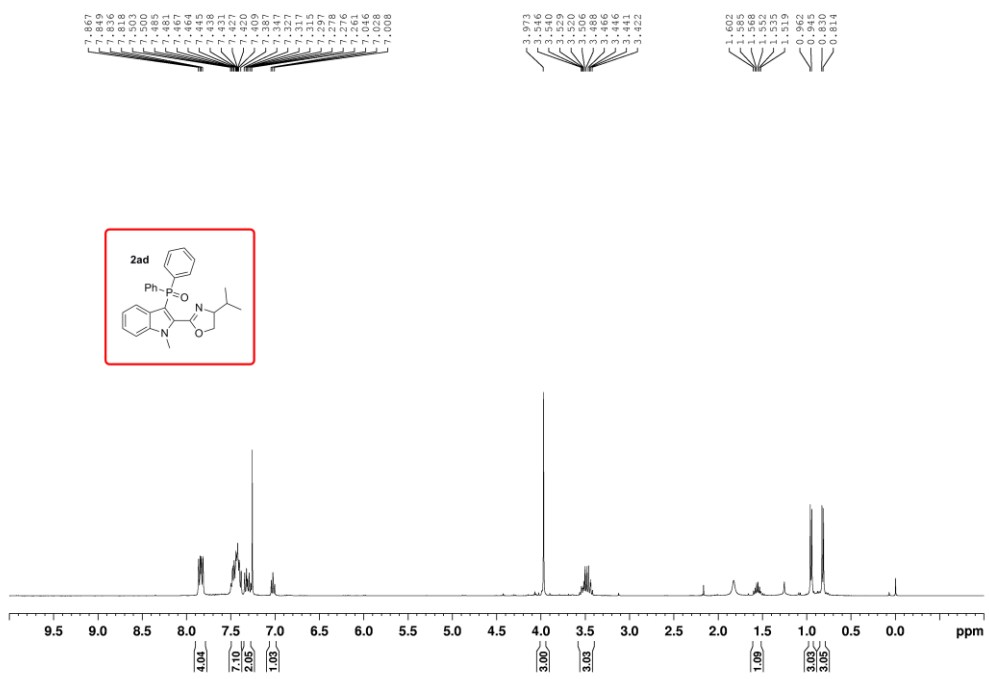
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
ppm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7.464
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7.438
7.431
7.427
7.420
7.409
7.387
7.347
7.327
7.317
7.315
7.297
7.278
7.276
7.261
7.046
7.028
7.008
3.973
3.546
3.540
3.529
3.520
3.506
3.488
3.466
3.446
3.441
3.422
1.602
1.585
1.568
1.552
1.535
1.519
0.962
0.945
0.830
0.814
2ad
Ph
P
O
N
O
N
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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ppm
4.04
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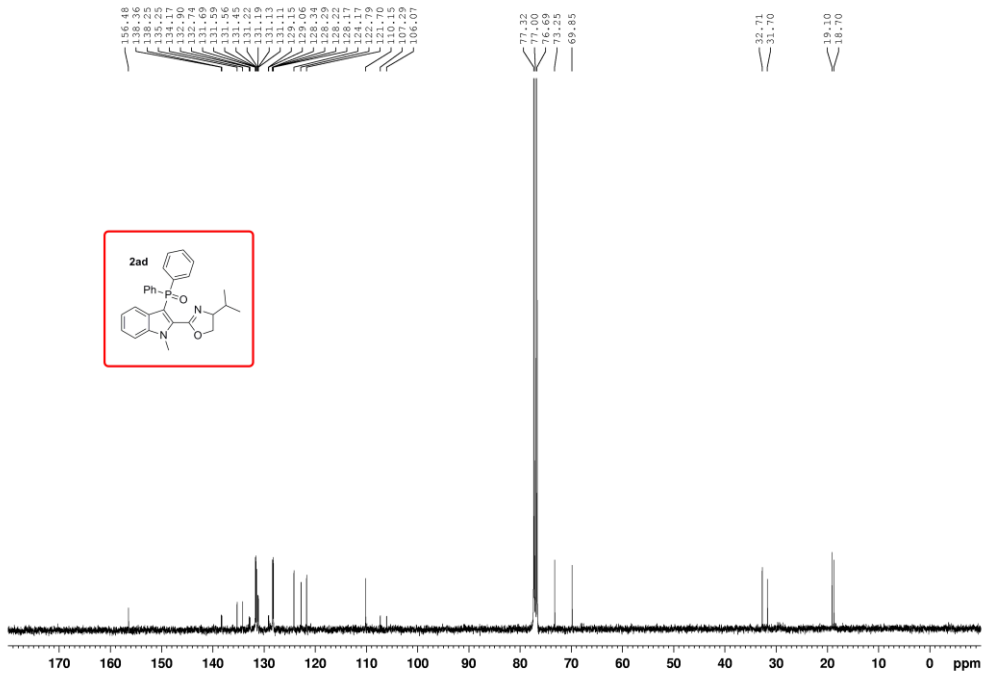
2.05
1.03
3.00
3.03
1.09
3.03
3.05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134.17
132.90
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131.68
131.59
131.56
131.45
131.22
131.19
131.13
131.11
129.15
129.06
128.38
128.29
128.22
128.17
123.77
122.79
121.70
110.15
107.29
106.07
77.32
77.00
76.69
73.25
69.85
32.71
31.70
19.10
18.70
2ad
Ph
P
O
N
O
N
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140
130
120
110
100
90
80
70
60
50
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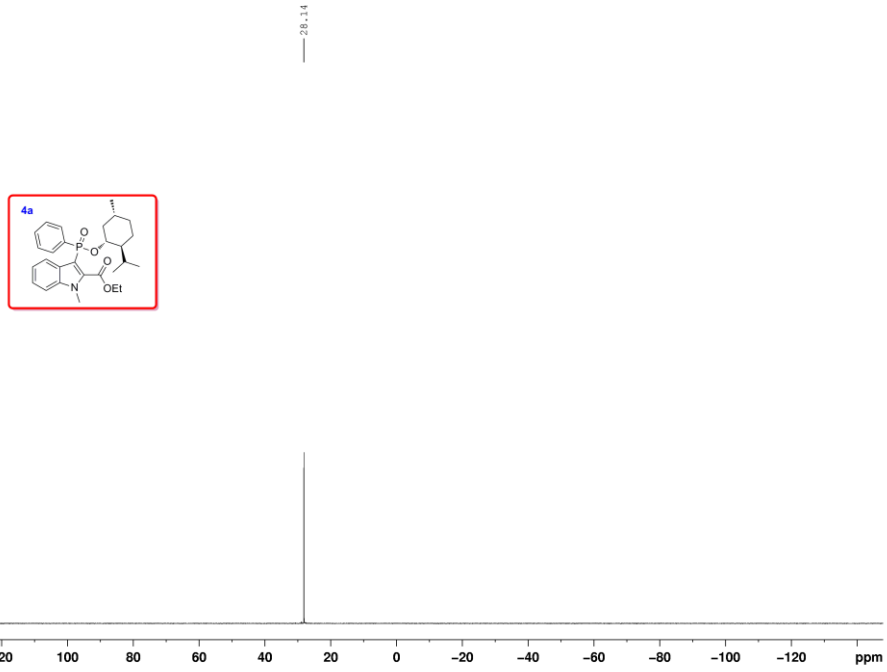
30
20
10
0
ppm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4a
OEt
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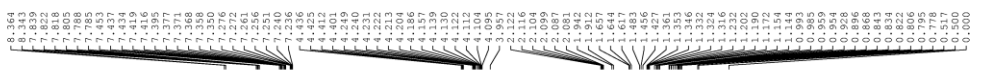
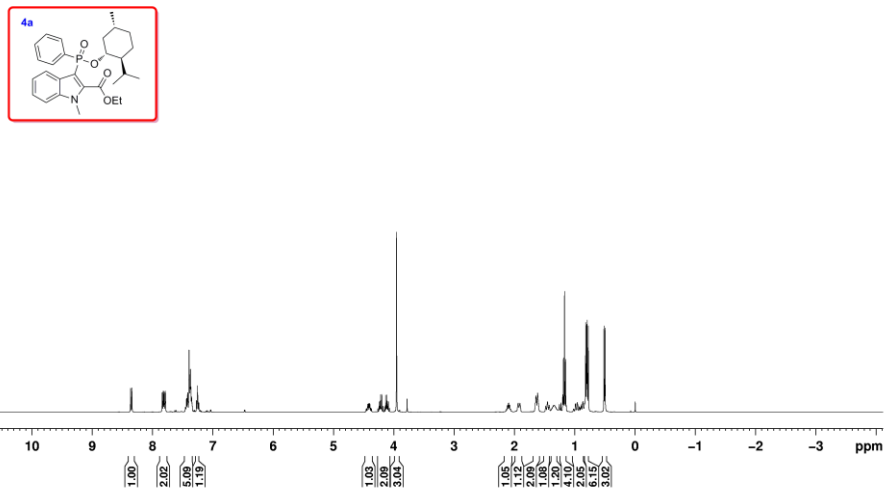
120
100
80
60
40
20
0
−20
−40
−60
−80
−100
−120
ppm




4a
OEt
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
ppm
1.00
2.02
5.08
1.19
1.03
2.09
3.04
1.05
1.12
2.05
1.08
1.20
4.10
2.05
6.15
3.02


161.56
138.06
137.93
136.48
135.05
134.70
134.50
131.05
131.02
130.45
130.34
128.53
128.44
127.89
127.76
124.63
123.69
121.97
121.30
120.68
119.08
110.06
109.01
108.80
107.21
100.73
77.32
77.20
77.00
76.68
76.29
76.22
61.45
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34.04
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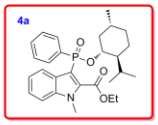
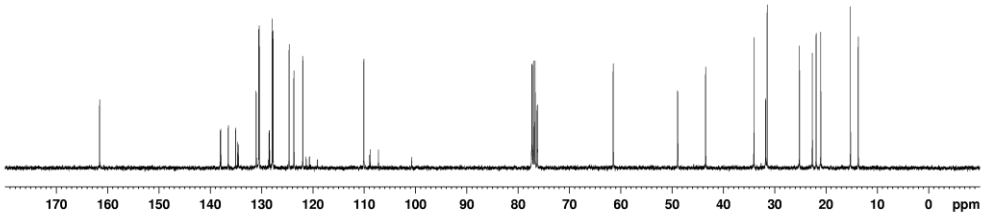
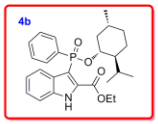
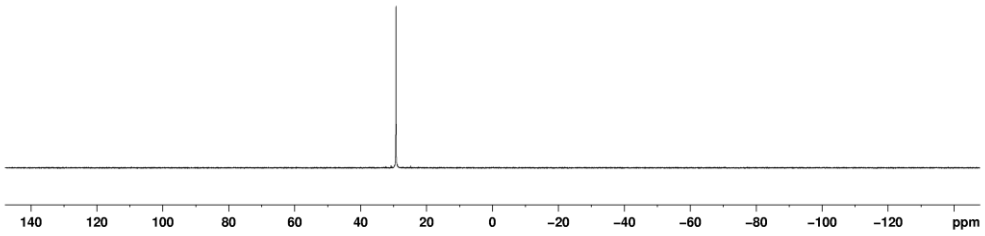
31.45
25.15
22.66
21.91
21.00
15.21
13.68

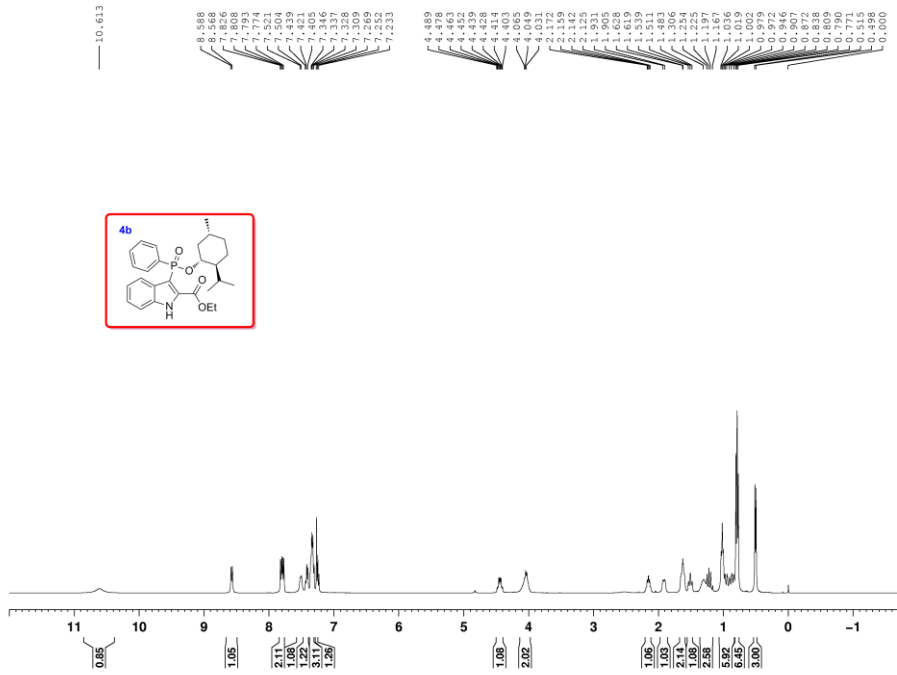

4a
O
P
O
O
N
OEt


170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

29.26


4b
O
P
O
O
N
H
OEt


140
120
100
80
60
40
20
0
−20
−40
−60
−80
−100
−120
ppm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7.826
7.808
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7.521
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7.439
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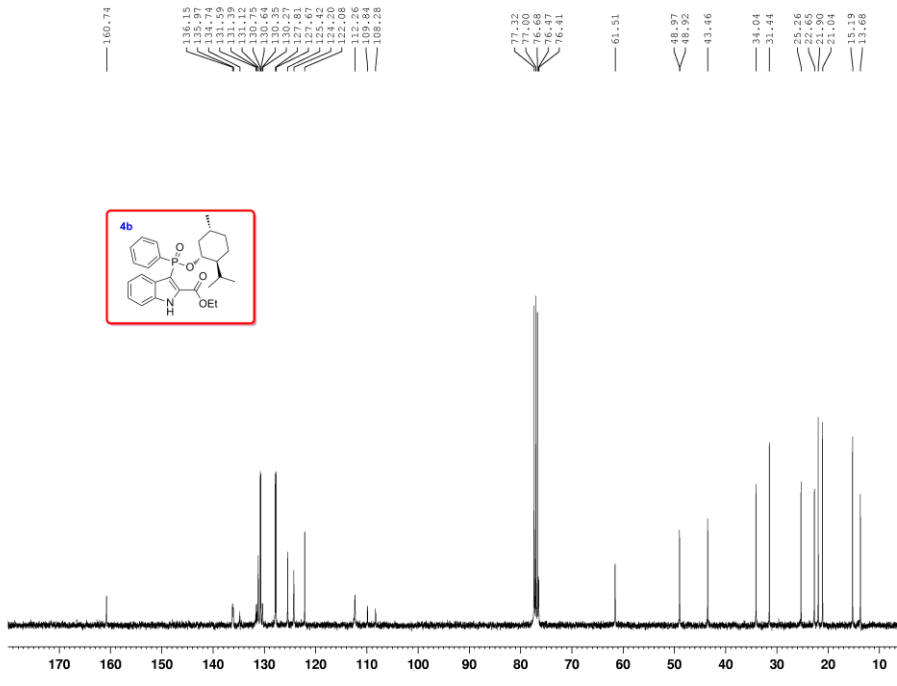
7.405
7.346
7.337
7.328
7.308
7.269
7.252
7.233
4.489
4.478
4.463
4.452
4.439
4.428
4.414
4.403
4.065
4.049
4.031
2.172
2.159
2.142
2.125
2.031
1.905
1.628
1.619
1.589
1.511
1.483
1.306
1.254
1.225
1.197
1.167
1.036
1.019
1.002
0.979
0.972
0.946
0.907
0.872
0.838
0.809
0.790
0.771
0.515
0.498
0.000
4b
O
P
O
O
N
H
OEt
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
ppm
0.85
1.05
2.11
1.08
1.22
3.15
1.29
1.08
2.02
1.05
1.03
2.14
1.08
2.59
5.92
6.45
3.00


160.74
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134.74
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131.38
131.12
130.75
130.64
130.35
130.27
127.81
127.67
125.42
123.08
122.08
112.26
109.84
108.28
77.32
77.00
76.68
76.47
76.41
61.51
48.97
48.92
43.46
34.04
31.44
25.26
22.65
21.90
21.04
15.19
13.68
4b
O
P
O
O
N
H
OEt
170
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120
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100
90
80
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60
50
40
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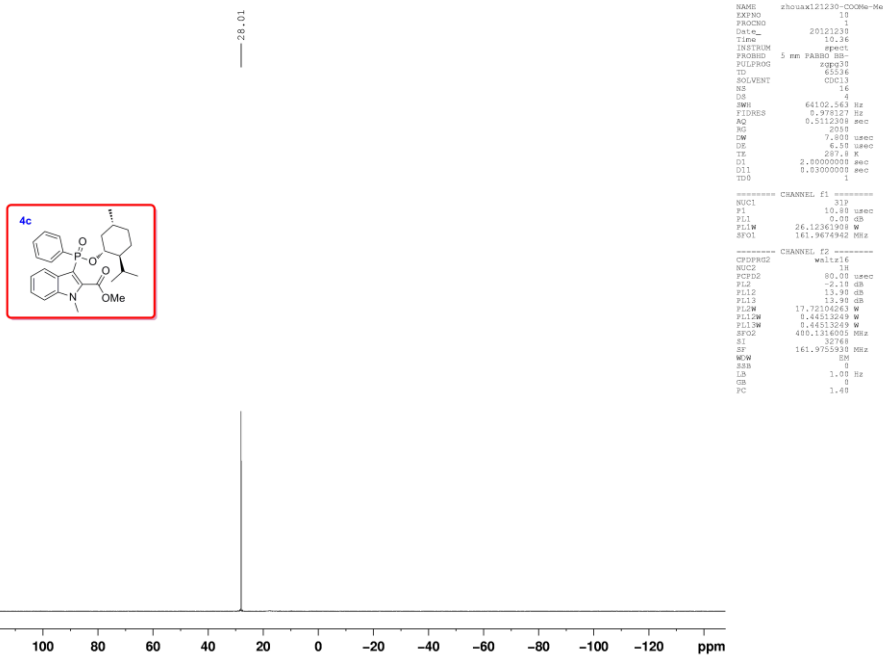
20
10
0
ppm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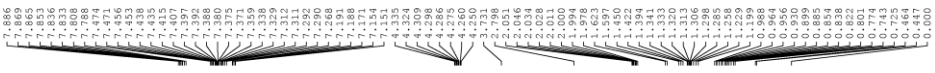
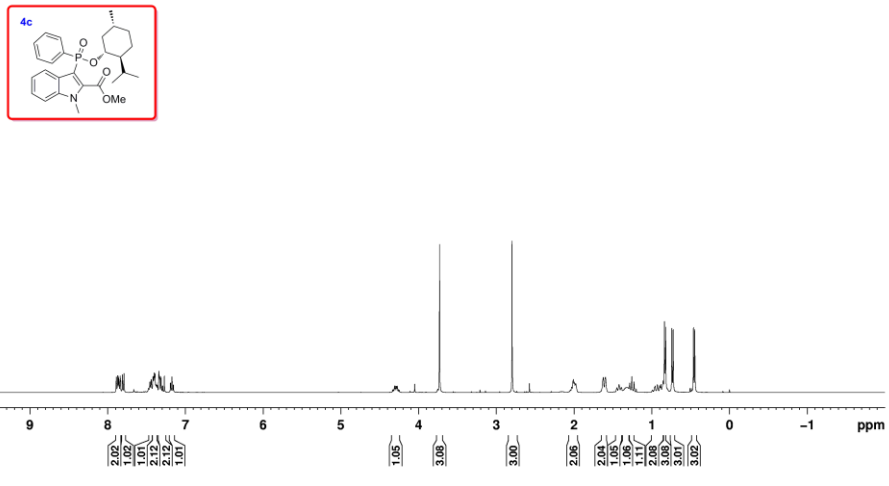
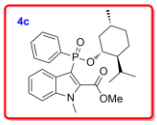
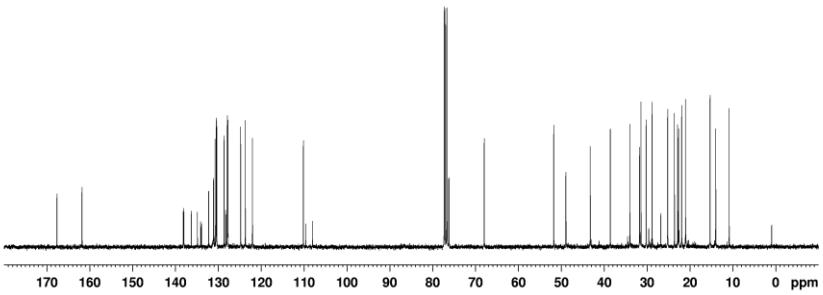
4c
OMe
140 120 100 80 60 40 20 0 −20 −40 −60 −80 −100 −120 ppm




4c
OMe
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
2.02 1.02 1.01 2.12 1.07 1.05 3.08 3.00 2.06 2.04 1.05 1.11 1.06 2.08 3.01 3.02






```
NAME          zhouax121230-COOMe-Me
EXPNO                        12
PROCNO                        1
Date_                  20121230
Time                      10.52
INSTRUM                   spect
PROBHD     5 mm PABBO BB-
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          200
DS                            4
SWH                   24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631968 sec
RG                          575
DW                       20.800 usec
DE                        6.50 usec
TE                       288.4 K
D1                    2.00000008 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        13C
P1                         9.70 usec
PL1                       -2.00 dB
PL1W             56.13311005 W
SFO1             100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     80.00 usec
PL2                       -2.10 dB
PL12                      13.90 dB
PL13                      13.90 dB
PL2W             17.72104263 W
PL12W             0.44513249 W
PL13W             0.44513249 W
SFO2             400.1316005 MHz
SI                        32768
SF               100.6127817 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
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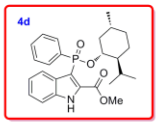

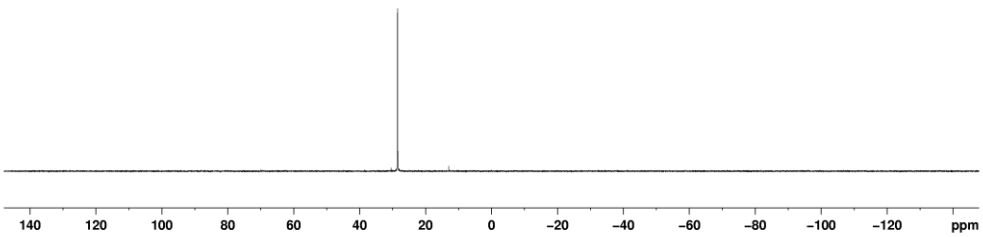


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8.556
8.537
7.848
7.829
7.815
7.796
7.701
7.483
7.443
7.426
7.408
7.352
7.335
7.315
7.276
7.263
7.239
4.437
4.427
4.414
3.541
2.221
2.206
2.190
2.169
1.869
1.845
1.664
1.651
1.600
1.566
1.538
1.512
1.358
1.228
1.199
1.170
1.102
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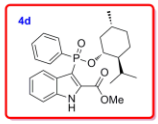
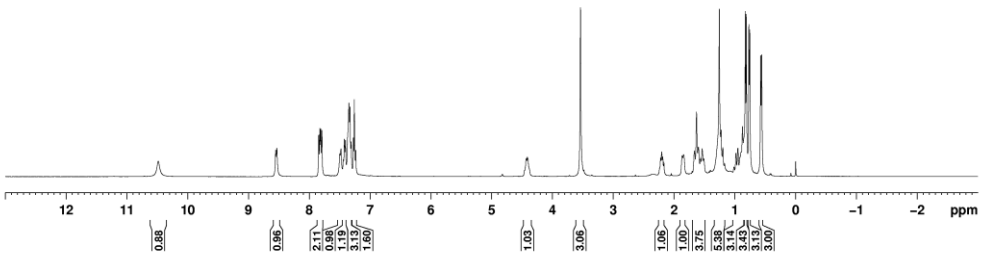
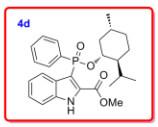
0.922
0.877
0.829
0.812
0.770
0.756
0.576
0.566
0.000

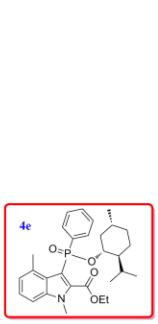

4d
O
P
O
O
N
H
OMe

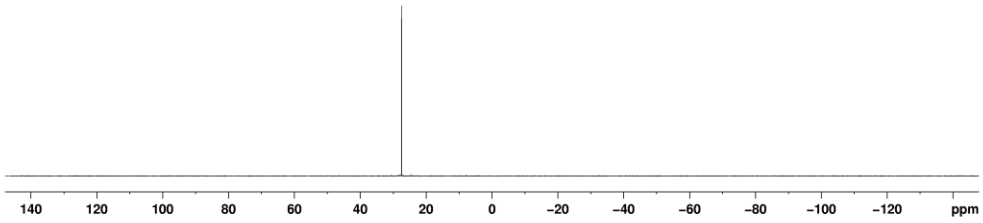

12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
ppm
0.88
0.96
2.11
0.98
1.19
3.15
1.00
1.03
3.06
1.06
1.00
3.75
5.38
3.14
3.43
3.13
3.00

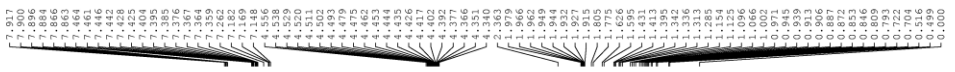

160.74
136.15
135.97
134.74
131.59
131.39
131.12
130.75
130.64
130.35
130.27
127.81
127.67
125.42
123.08
122.08
112.26
109.84
108.28
77.32
77.00
76.68
76.47
76.41
61.51
48.97
48.92
43.46
34.04
31.44
25.26
22.65
21.90
21.04
15.19
13.68

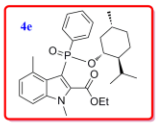

4d
O
P
O
O
N
H
OMe

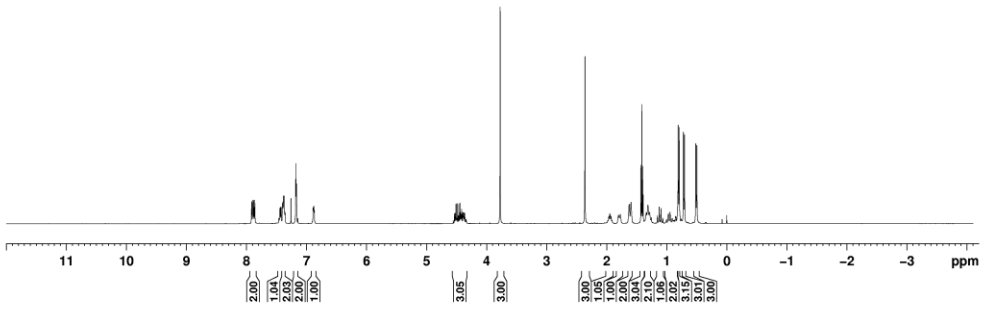
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

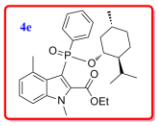

4e
OEt

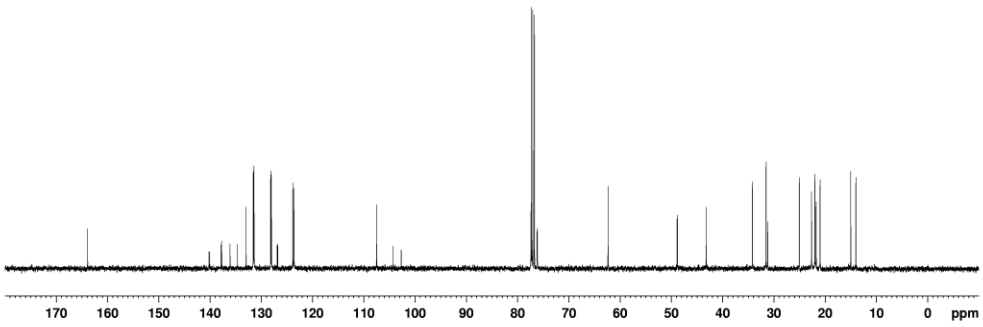

27.48
140
120
100
80
60
40
20
0
−20
−40
−60
−80
−100
−120
ppm

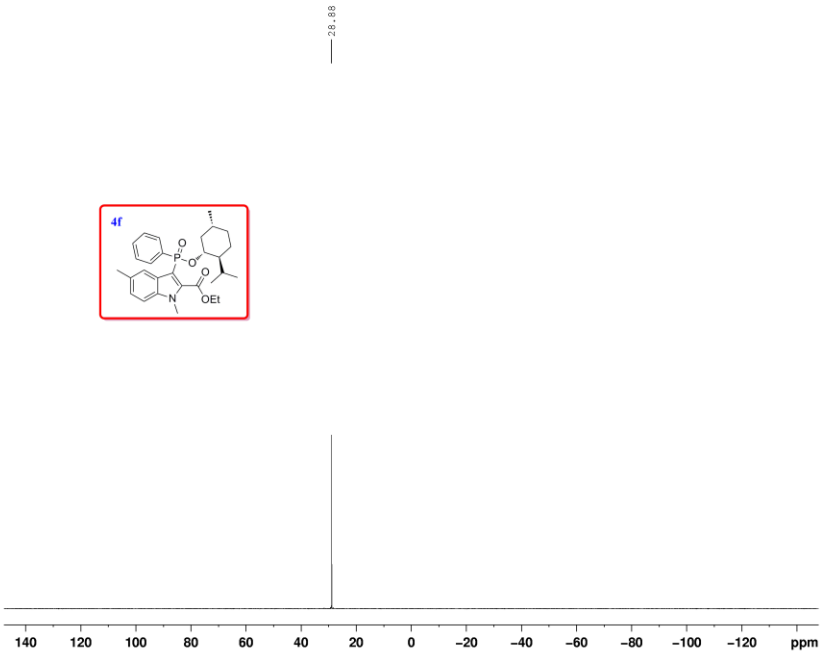



4e
OEt

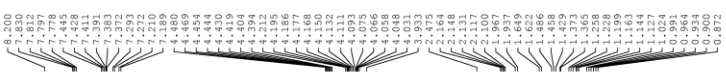

11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
ppm
2.00
1.64
2.03
2.00
1.00
3.05
3.00
3.00
1.05
1.00
2.00
2.04
2.10
1.06
2.02
3.15
3.01
3.00

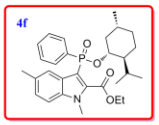


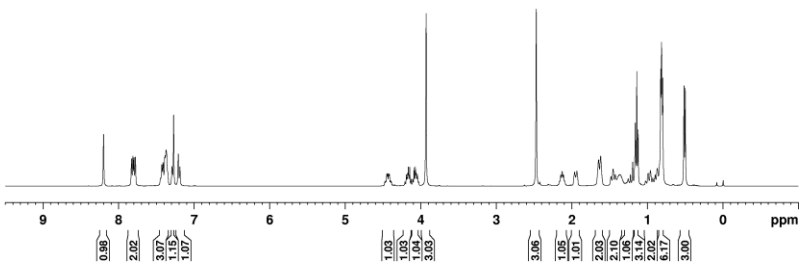


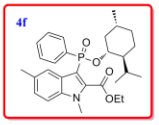


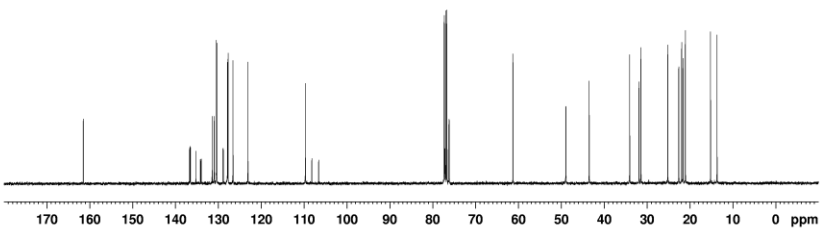

```
NAME          zhouax20130105
EXPNO                     11
PROCNO                     1
Date_               20130105
Time                    9.49
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         4
SWH                64102.563 Hz
FIDRES              0.978127 Hz
AQ                  0.5112308 sec
RG                       2050
DW                    7.800 usec
DE                    6.50 usec
TE                    298.3 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      31P
P1                      10.80 usec
PL1                      0.00 dB
PL1W               26.12361908 W
SFO1               161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   80.00 usec
PL2                     -2.10 dB
PL12                    13.90 dB
PL13                    13.90 dB
PL2W               17.72104263 W
PL12W               0.44513249 W
PL13W               0.44513249 W
SFO2               400.1316005 MHz
SI                        32768
SF                 161.9755930 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```

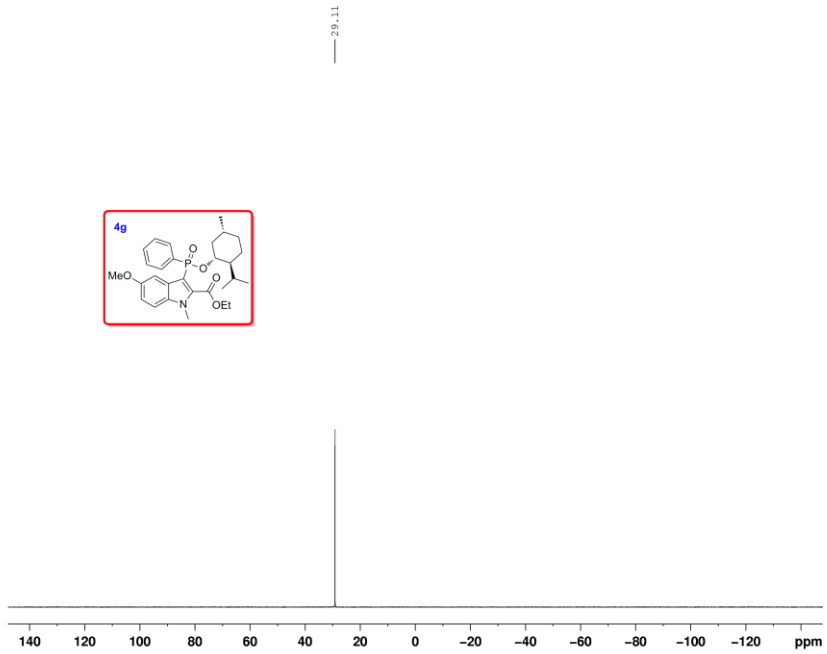



4f
OEt

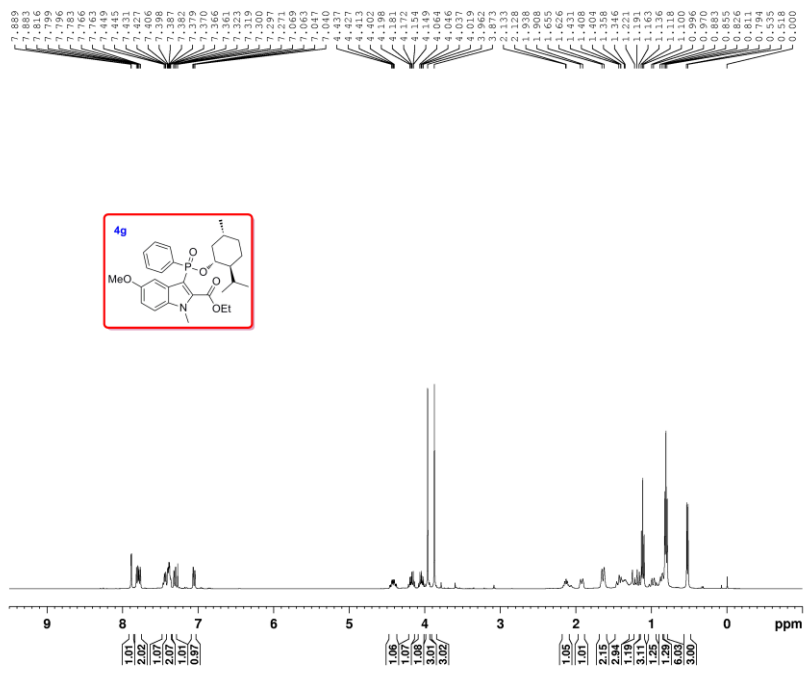

9
8
7
6
5
4
3
2
1
0
ppm
0.98
2.02
3.07
1.15
1.07
1.03
1.03
1.04
3.03
3.06
1.05
1.01
2.03
2.10
1.06
3.14
2.02
6.17
3.00


4f
OEt

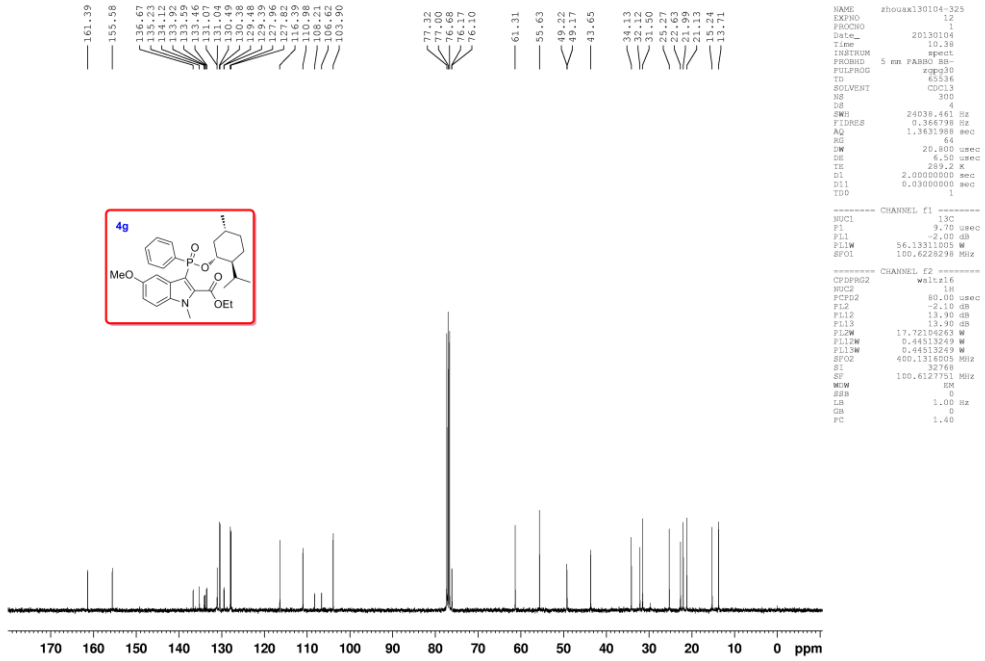

170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



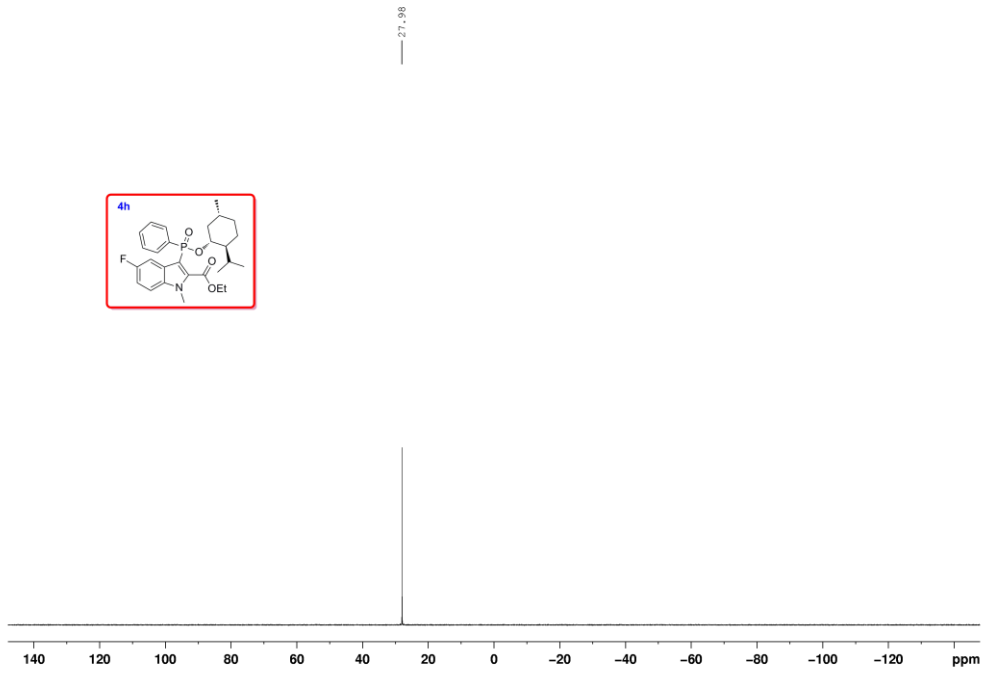

```
NAME            zhouax130104-325
EXPNO                        11
PROCNO                        1
Date_                  20130104
Time                      10.18
INSTRUM                   spect
PROBHD   5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           4
SWH                  64102.563 Hz
FIDRES               0.978127 Hz
AQ                   0.5112308 sec
RG                        2050
DW                       7.800 usec
DE                       6.50 usec
TE                      288.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        31P
P1                        10.80 usec
PL1                        0.00 dB
PL1W                26.12361908 W
SFO1                161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     80.00 usec
PL2                       -2.10 dB
PL12                      13.90 dB
PL13                      13.90 dB
PL2W                17.72104263 W
PL12W                0.44513249 W
PL13W                0.44513249 W
SFO2                400.1316005 MHz
SI                         32768
SF                  161.9755930 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```

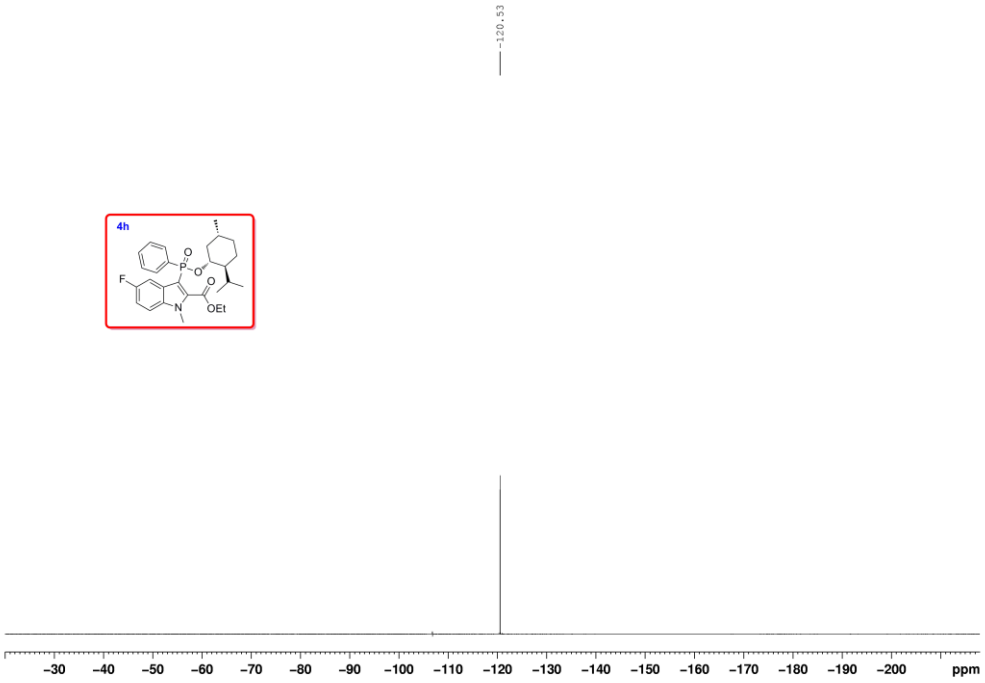


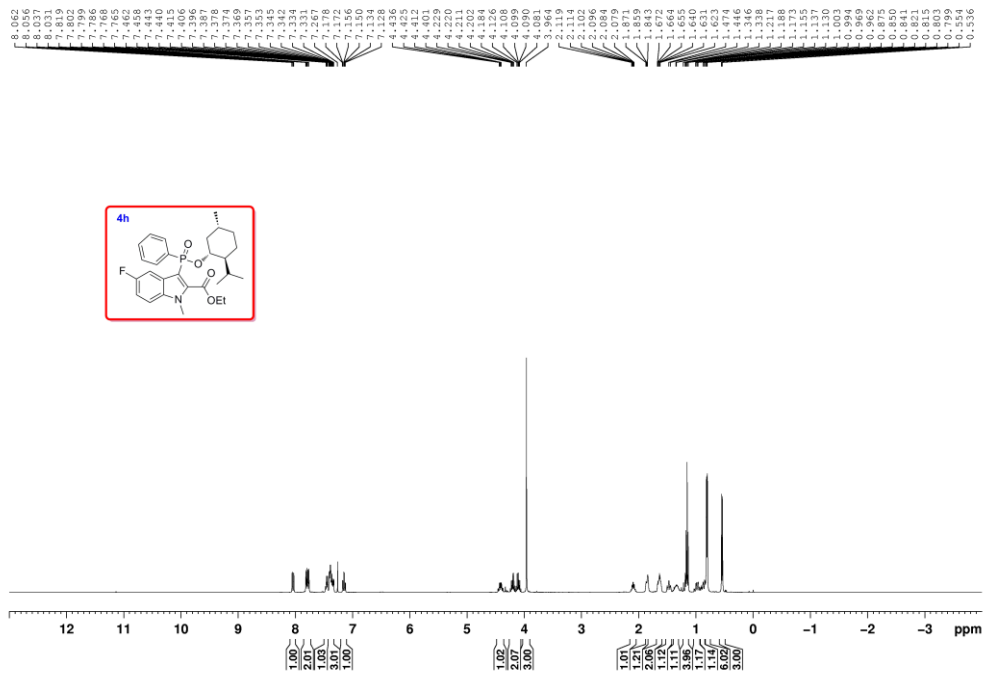

```
NAME            zhouax130104-325
EXPNO                        10
PROCNO                        1
Date_                  20130104
Time                      10.16
INSTRUM                   spect
PROBHD   5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                            8
DS                            2
SWH                  8223.685 Hz
FIDRES               0.125483 Hz
AQ                   3.9846367 sec
RG                           64
DW                       60.800 usec
DE                        6.50 usec
TE                      287.3 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        1H
P1                        14.70 usec
PL1                       -1.00 dB
PL1W                13.75590801 W
SFO1                400.1324710 MHz
SI                         32768
SF                  400.1300006 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```

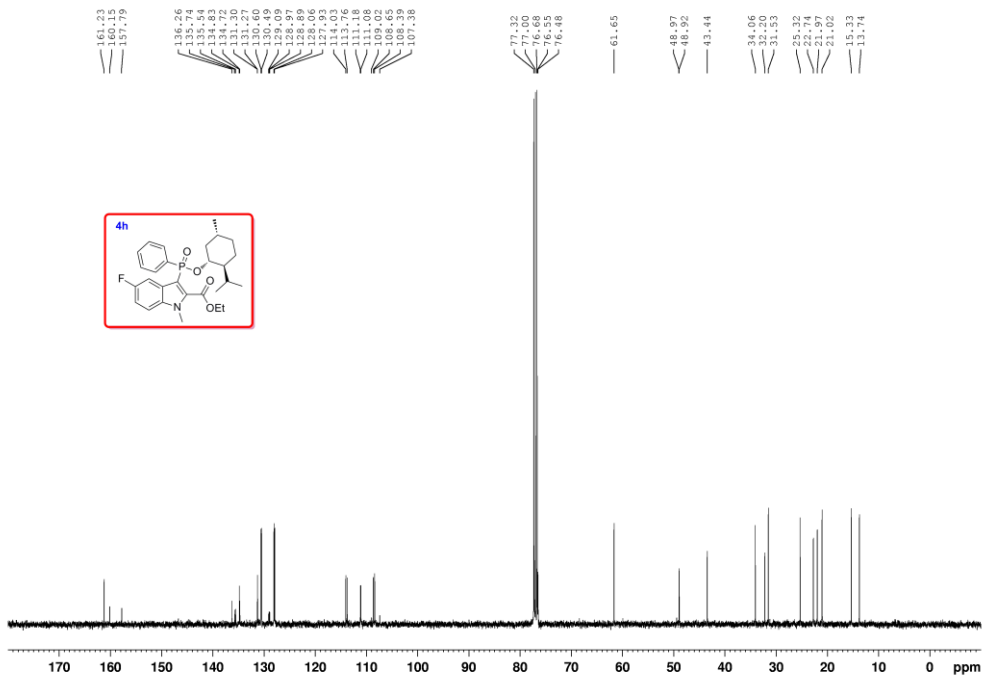


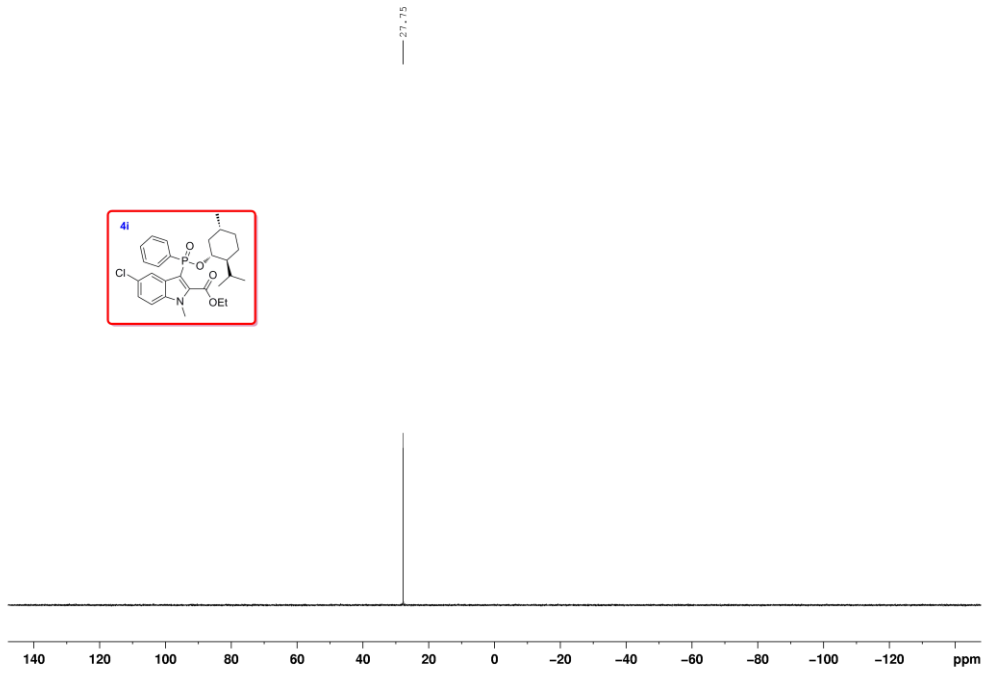

```
NAME          zhouaxl30104-325
EXPNO                      12
PROCNO                      1
Date_              20130104
Time                   10.38
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      300
DS                        4
SWH               24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                       64
DW                   20.800 usec
DE                    6.50 usec
TE                   289.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    9.70 usec
PL1                  -2.00 dB
PL1W           56.13311005 W
SFO1           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -2.10 dB
PL12                 13.90 dB
PL13                 13.90 dB
PL2W           17.72104263 W
PL12W           0.44513249 W
PL13W           0.44513249 W
SFO2           400.1316005 MHz
SI                    32768
SF             100.6127751 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

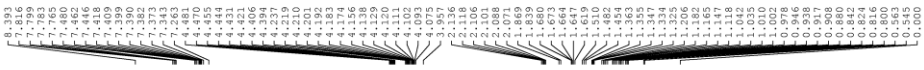


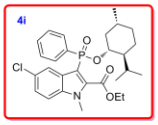

-120.53
4h
F
O
P
O
N
O
OEt
−30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 ppm

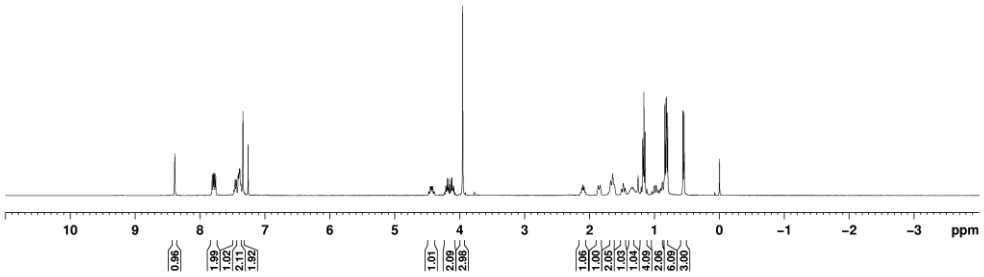

4h
F
O
P
O
N
O
OEt
12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3 ppm
1.00 2.01 1.03 3.01 1.00
1.02 2.07 3.00
1.01 1.21 2.08 1.12 1.11 3.96 1.17 1.14 6.02 3.00

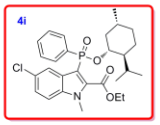

161.23
160.15
157.79
136.26
135.74
135.54
134.83
134.72
131.30
131.27
130.60
130.49
129.09
128.97
128.89
128.06
127.93
114.03
113.76
111.18
111.08
109.02
108.65
108.39
107.38
77.32
77.00
76.68
76.55
76.48
61.65
48.97
48.92
43.44
34.06
32.20
31.53
25.32
22.74
21.97
21.02
15.33
13.74
4h
F
N
O
OEt
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

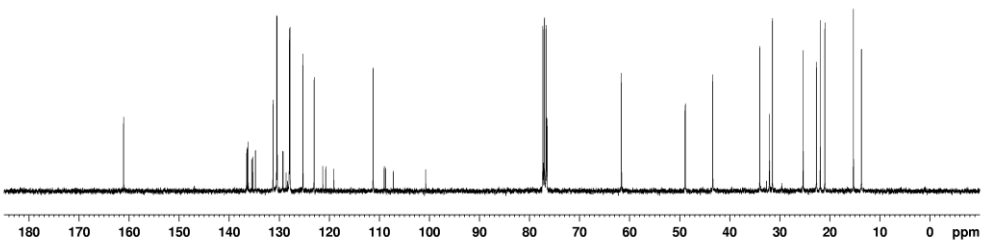

27.75
4i
Cl
N
O
OEt
140
120
100
80
60
40
20
0
−20
−40
−60
−80
−100
−120
ppm

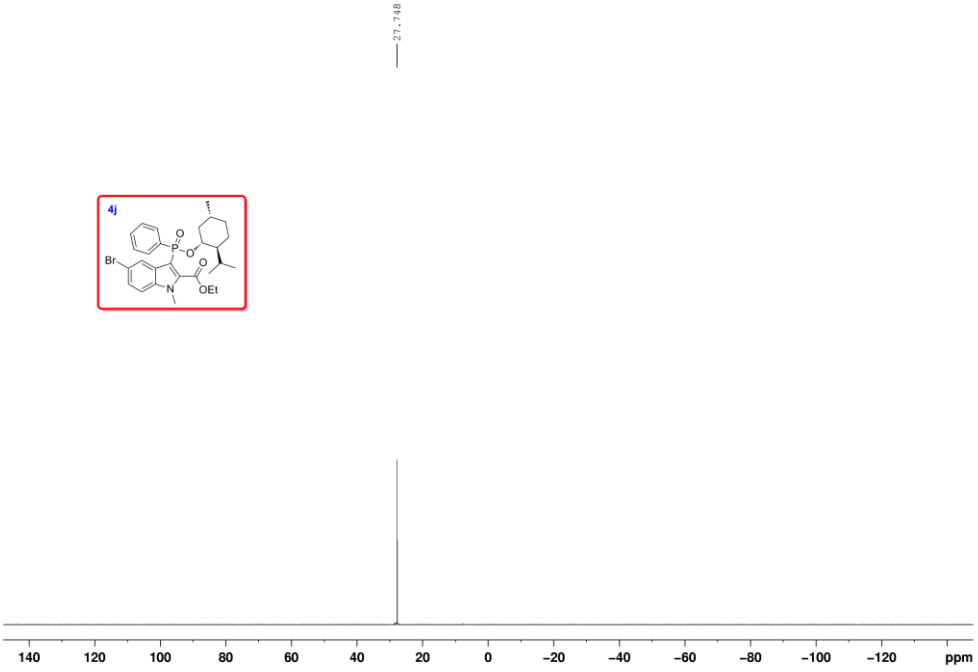



4i
Cl
N
OEt

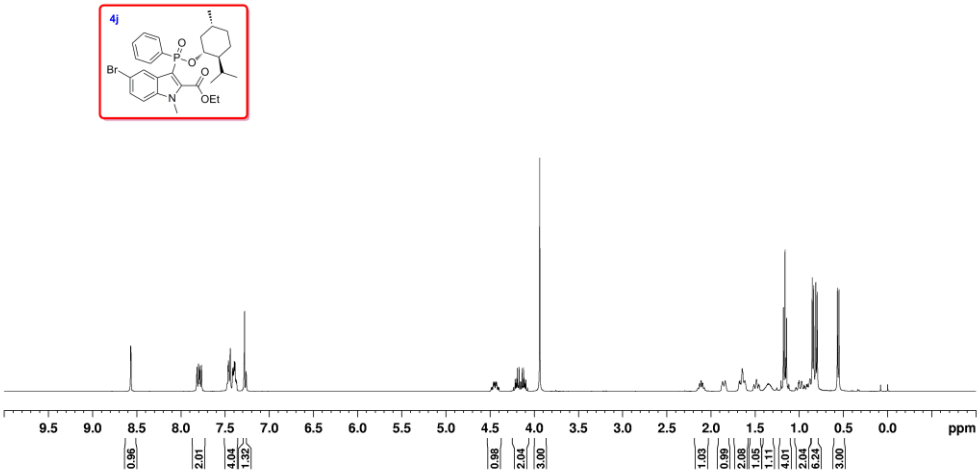

10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
ppm
0.96
1.99
1.00
2.11
1.92
1.01
2.09
2.98
1.06
1.00
2.05
1.03
1.14
4.09
2.06
6.09
3.00

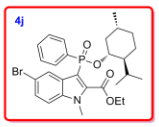

4i
Cl
N
OEt

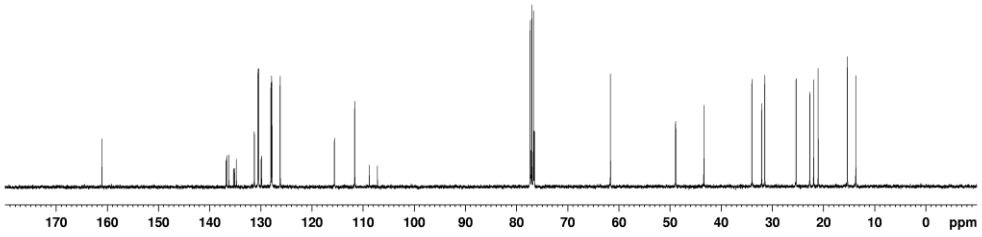

180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

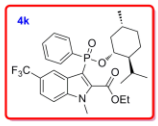

27.748
4j
Br
OEt
140
120
100
80
60
40
20
0
−20
−40
−60
−80
−100
−120
ppm


8.569
8.565
7.818
7.801
7.797
7.784
7.767
7.763
7.478
7.474
7.467
7.462
7.456
7.445
7.441
7.415
7.406
7.396
7.391
7.387
7.379
7.375
7.330
7.285
7.279
7.263
7.260
4.460
4.449
4.437
4.426
4.216
4.207
4.198
4.189
4.180
4.171
4.156
4.153
4.139
4.121
4.112
4.103
4.094
3.937
2.133
2.128
2.115
2.110
2.098
2.093
1.870
1.840
1.682
1.674
1.666
1.648
1.642
1.617
1.514
1.486
1.465
1.448
1.363
1.355
1.347
1.266
1.208
1.181
1.163
1.146
1.110
1.010
1.002
0.977
0.972
0.946
0.918
0.909
0.880
0.855
0.837
0.815
0.798
0.569
0.552
0.000

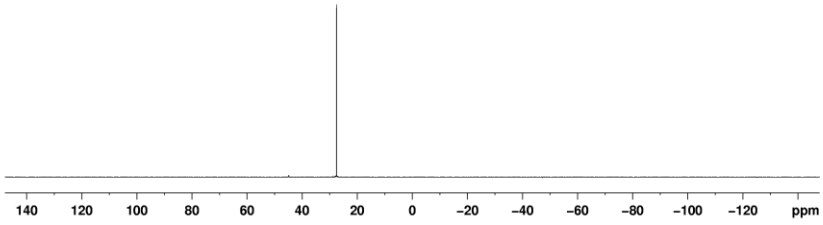

4j
Br
OEt
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.96
2.01
4.04
1.32
0.98
2.04
3.00
1.03
0.99
2.08
1.05
1.11
4.01
2.04
6.24
3.00

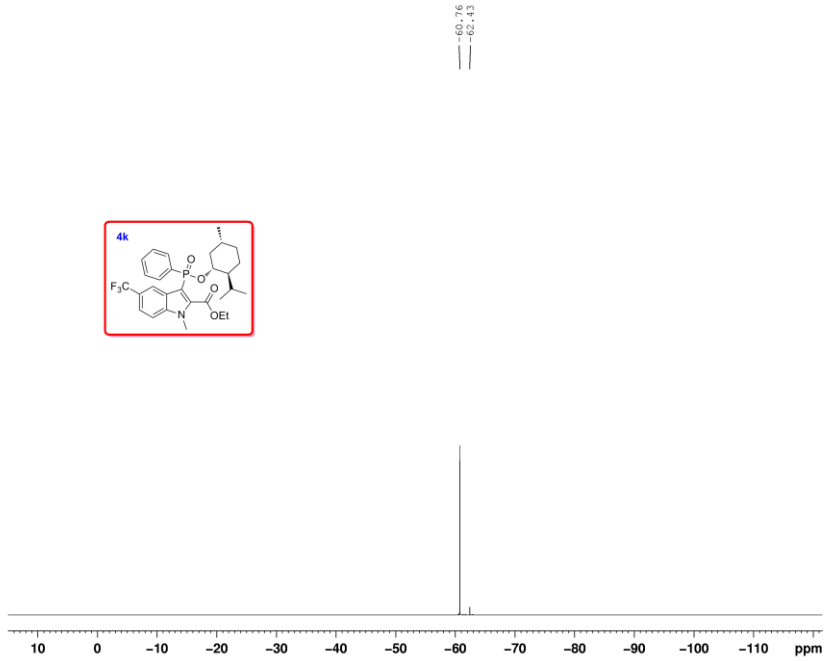




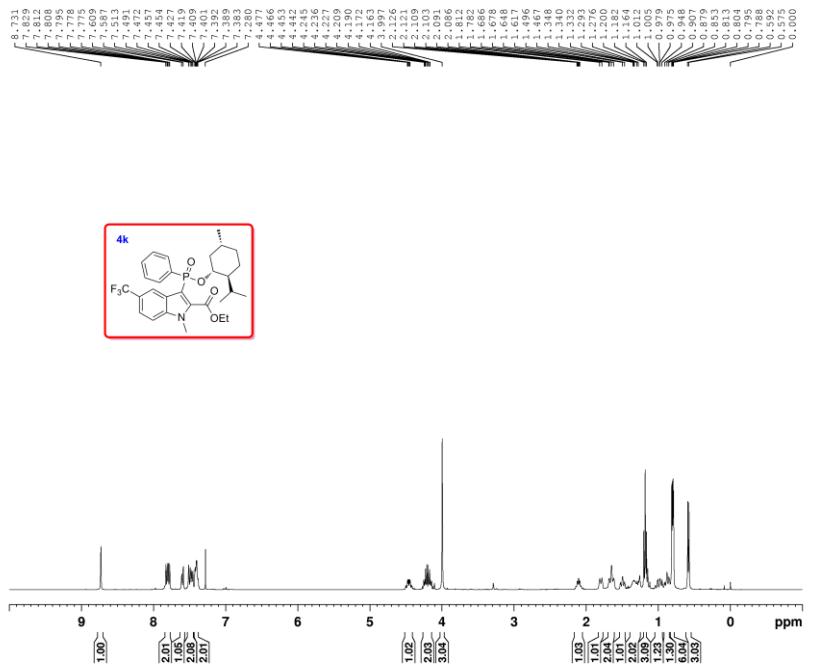



```
NAME          zhouax121230
EXPNO                   11
PROCNO                   1
Date_          20121230
Time                  9.45
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     4
SWH           64102.563 Hz
FIDRES        0.978127 Hz
AQ            0.5112308 sec
RG                  2050
DW                7.800 usec
DE                6.50 usec
TE                297.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  31P
P1                 10.80 usec
PL1                0.00 dB
PL1W         26.12361908 W
SFO1         161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2               -2.10 dB
PL12               13.90 dB
PL13               13.90 dB
PL2W         17.72106263 W
PL12W         0.44513249 W
PL13W         0.44513249 W
SFO2         400.1316005 MHz
SI                 32768
SF           161.9755930 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

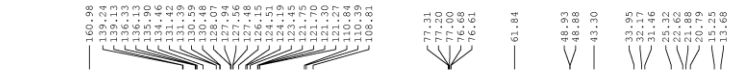


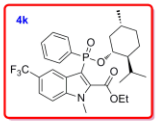



```
NAME          zhouax121230
EXPNO                   12
PROCNO                   1
Date_             20121230
Time                 9.49
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG          zgfhigqn
TD                 131072
SOLVENT              CDCl3
NS                      16
DS                       4
SWH              89285.711 Hz
FIDRES           0.681196 Hz
AQ               0.7340532 sec
RG                     912
DW                  5.600 usec
DE                  6.50 usec
TE                 287.1 K
D1              1.00000000 sec
D11             0.03000000 sec
D12             0.00002000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   19F
P1                   14.90 usec
PL1                  -4.00 dB
PL1W           22.33589982 W
SFO1           376.4607164 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -2.10 dB
PL12                 13.90 dB
PL2W           17.72104263 W
PL12W           0.44513249 W
SFO2           400.1316005 MHz
SI                   65536
SF             376.4983660 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

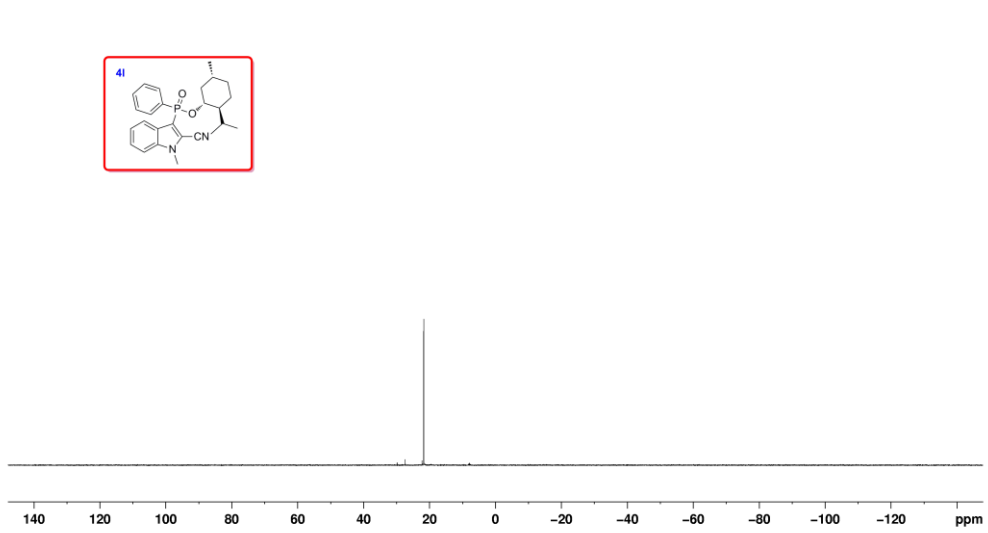


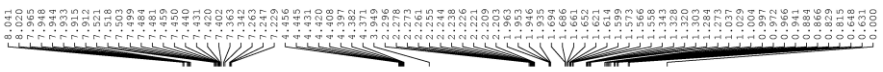

```
NAME      zhouax121231-5-CF3-OMen
EXPNO                   10
PROCNO                   1
Date_             20121231
Time                22.01
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                  65536
SOLVENT              CDCl3
NS                       8
DS                       2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                    40.3
DW                 60.800 usec
DE                  6.50 usec
TE                 288.5 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                   14.70 usec
PL1                  -1.00 dB
PL1W           13.75590801 W
SFO1           400.1324710 MHz
SI                   32768
SF             400.1299971 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

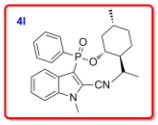


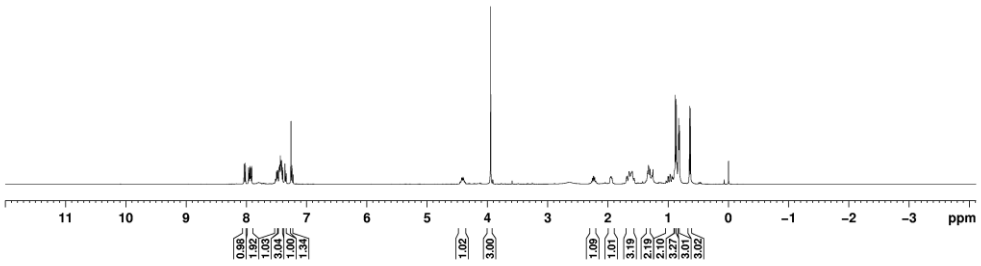


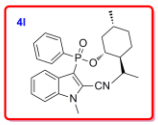

```
NAME            zhouax121230
EXPNO                     13
PROCNO                     1
Date_           20121230
Time                   10.03
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                CDCl3
NS                       200
DS                         4
SWH             24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                      71.8
DW                   20.800 usec
DE                    6.50 usec
TE                   298.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                    9.70 usec
PL1                  -2.00 dB
PL1W            56.13311005 W
SFO1            100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 80.00 usec
PL2                  -2.10 dB
PL12                 13.90 dB
PL13                 13.90 dB
PL2W            17.72104263 W
PL12W            0.44513249 W
PL13W            0.44513249 W
SFO2            400.1316005 MHz
SI                      32768
SF              100.6127773 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```



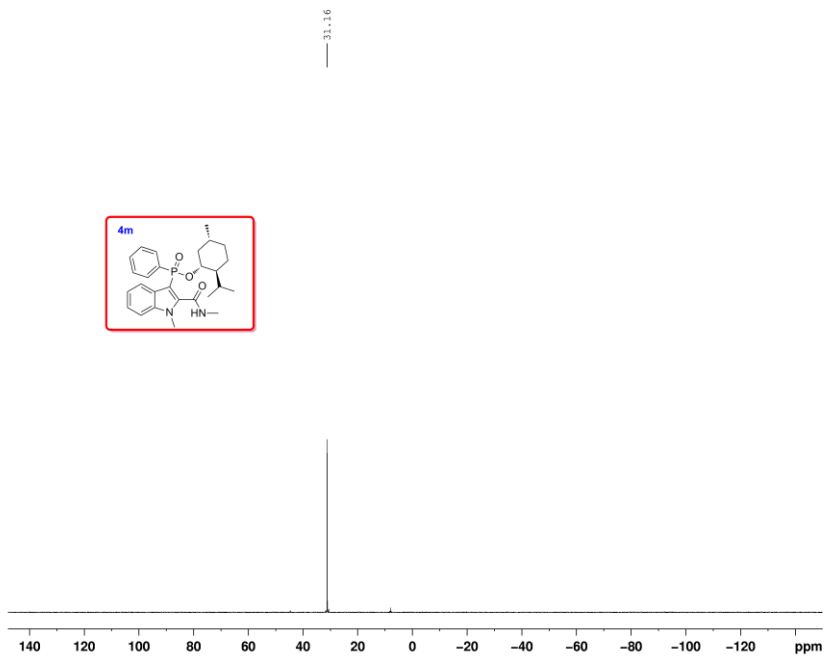



4l

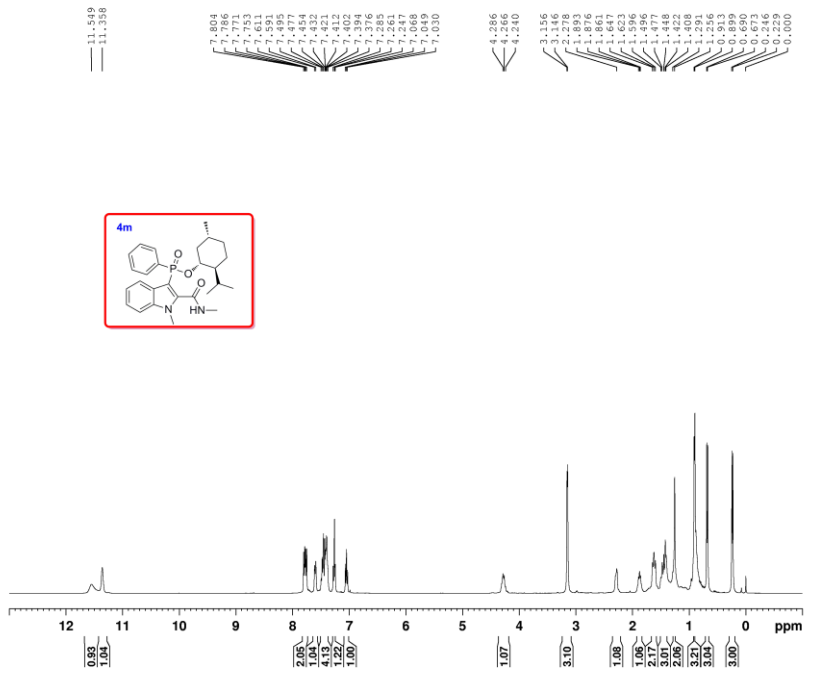

11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
ppm
0.98
1.92
1.03
3.04
1.00
1.34
1.02
3.00
1.09
1.01
3.19
2.19
2.10
3.27
3.01
3.02


4l

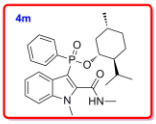
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

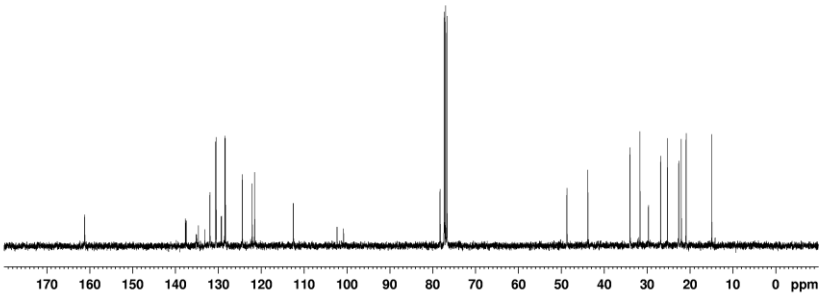



```
NAME            zhouax121228-CONHMe
EXPNO                            11
PROCNO                            1
Date_                      20121228
Time                          10.22
INSTRUM                       spect
PROBHD     5 mm PABBO BB-
PULPROG                       zgpg30
TD                             65536
SOLVENT                      CDCl3
NS                                16
DS                                 4
SWH                       64102.563 Hz
FIDRES                     0.978127 Hz
AQ                         0.5112308 sec
RG                              2050
DW                             7.800 usec
DE                             6.50 usec
TE                            289.2 K
D1                        2.00000000 sec
D11                       0.03000000 sec
TD0                               1

======== CHANNEL f1 ========
NUC1                            31P
P1                            10.80 usec
PL1                            0.00 dB
PL1W                     26.12361908 W
SFO1                    161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2                     waltz16
NUC2                            1H
PCPD2                         80.00 usec
PL2                           -2.10 dB
PL12                          13.90 dB
PL13                          13.90 dB
PL2W                     17.72104263 W
PL12W                     0.44513249 W
PL13W                     0.44513249 W
SFO2                    400.1316005 MHz
SI                              32768
SF                      161.9755930 MHz
WDW                                EM
SSB                                0
LB                              1.00 Hz
GB                                 0
PC                              1.40
```

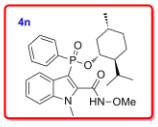


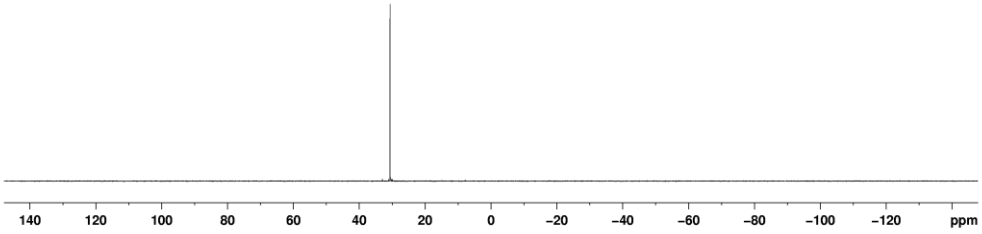

```
NAME            zhouax121228-CONHMe
EXPNO                            10
PROCNO                            1
Date_                      20121228
Time                          10.20
INSTRUM                       spect
PROBHD     5 mm PABBO BB-
PULPROG                         zg30
TD                             65536
SOLVENT                      CDCl3
NS                                14
DS                                 2
SWH                        8223.685 Hz
FIDRES                     0.125483 Hz
AQ                         3.9846367 sec
RG                                57
DW                            60.800 usec
DE                             6.50 usec
TE                            288.5 K
D1                        1.00000000 sec
TD0                               1

======== CHANNEL f1 ========
NUC1                            1H
P1                            14.70 usec
PL1                           -1.00 dB
PL1W                     13.75590801 W
SFO1                    400.1324710 MHz
SI                              32768
SF                      400.1300048 MHz
WDW                                EM
SSB                                0
LB                              0.30 Hz
GB                                 0
PC                              1.00
```

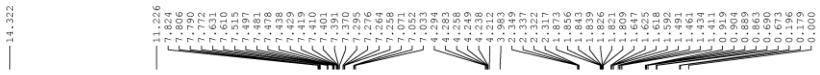


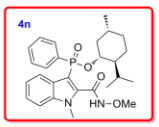


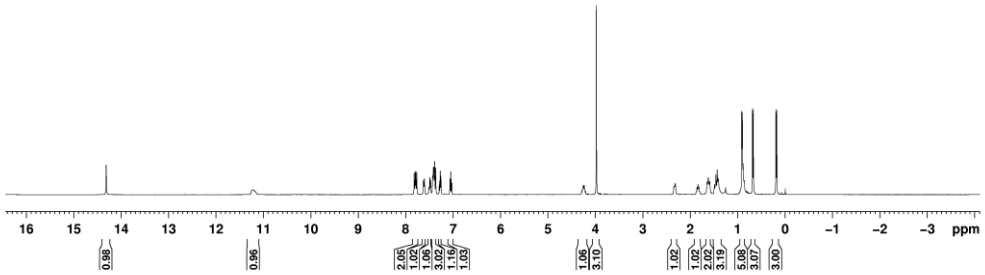

```
NAME            zhouaxl21228-CONHMe
EXPNO                            12
PROCNO                            1
Date_                      20121228
Time                          10.32
INSTRUM                       spect
PROBHD     5 mm PABBO BB-
PULPROG                   zgpg30
TD                             65536
SOLVENT                     CDCl3
NS                               120
DS                                 4
SWH                    24038.461 Hz
FIDRES                  0.366798 Hz
AQ                      1.3631988 sec
RG                              64
DW                         20.800 usec
DE                          6.50 usec
TE                           290.1 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                               1

======== CHANNEL f1 ========
NUC1                           13C
P1                            9.70 usec
PL1                          -2.00 dB
PL1W                  56.13311005 W
SFO1                 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                    waltz16
NUC2                            1H
PCPD2                      80.00 usec
PL2                          -2.10 dB
PL12                         13.90 dB
PL13                         13.90 dB
PL2W                  17.72104263 W
PL12W                  0.44513249 W
PL13W                  0.44513249 W
SFO2                400.1316005 MHz
SI                           32768
SF                  100.6127751 MHz
WDW                             EM
SSB                              0
LB                            1.00 Hz
GB                               0
PC                            1.40
```

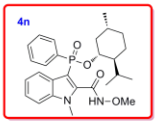


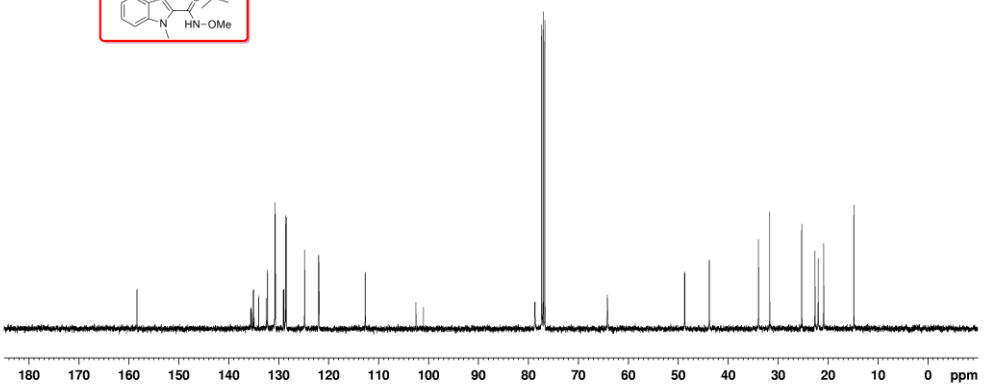

14.322
11.226
7.824
7.806
7.790
7.772
7.631
7.610
7.515
7.497
7.481
7.478
7.438
7.429
7.419
7.410
7.401
7.391
7.370
7.295
7.276
7.264
7.258
7.071
7.053
7.033
4.294
4.283
4.258
4.249
4.238
4.212
3.983
2.349
2.337
2.322
2.317
1.873
1.856
1.843
1.839
1.826
1.821
1.609
1.447
1.625
1.618
1.592
1.491
1.461
1.434
1.411
0.919
0.904
0.889
0.863
0.690
0.673
0.196
0.179
0.000

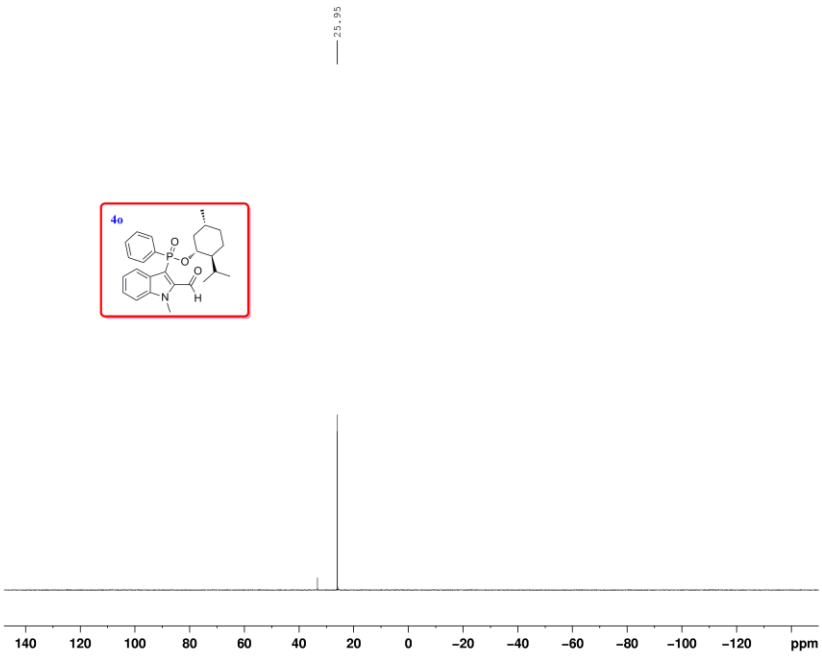

4n
HN–OMe


16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
ppm
0.98
0.96
2.05
1.02
1.06
3.02
1.16
1.09
1.06
3.10
1.02
1.02
2.02
3.19
5.08
3.07
3.00

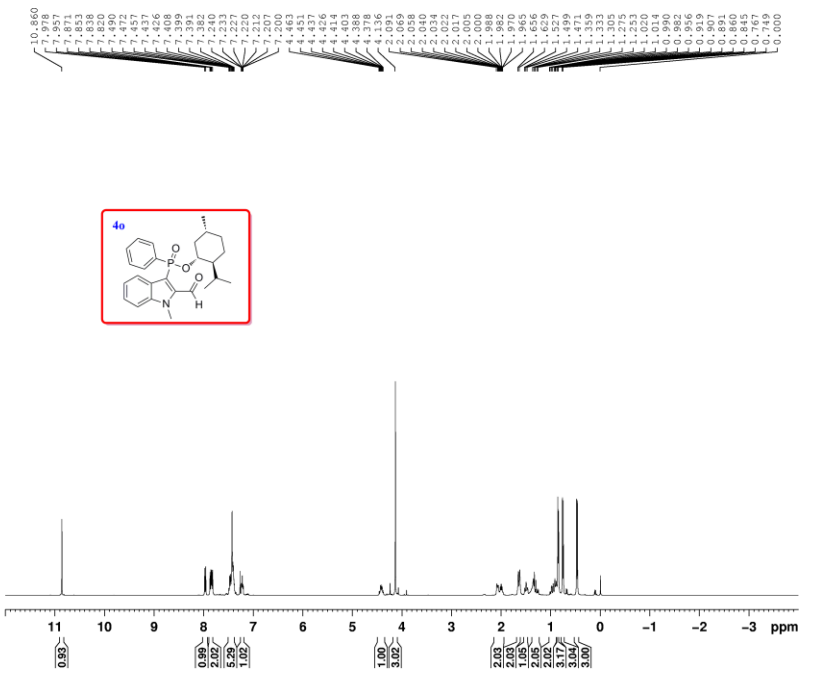

158.32
135.55
135.43
135.10
134.90
133.97
132.19
132.17
130.73
130.62
129.03
128.92
128.58
128.44
124.77
121.78
121.87
112.62
102.47
100.96
78.69
78.63
77.32
77.00
76.68
64.16
64.11
48.72
48.65
43.76
33.90
31.66
25.24
22.58
21.96
20.85
14.80


4n
HN–OMe

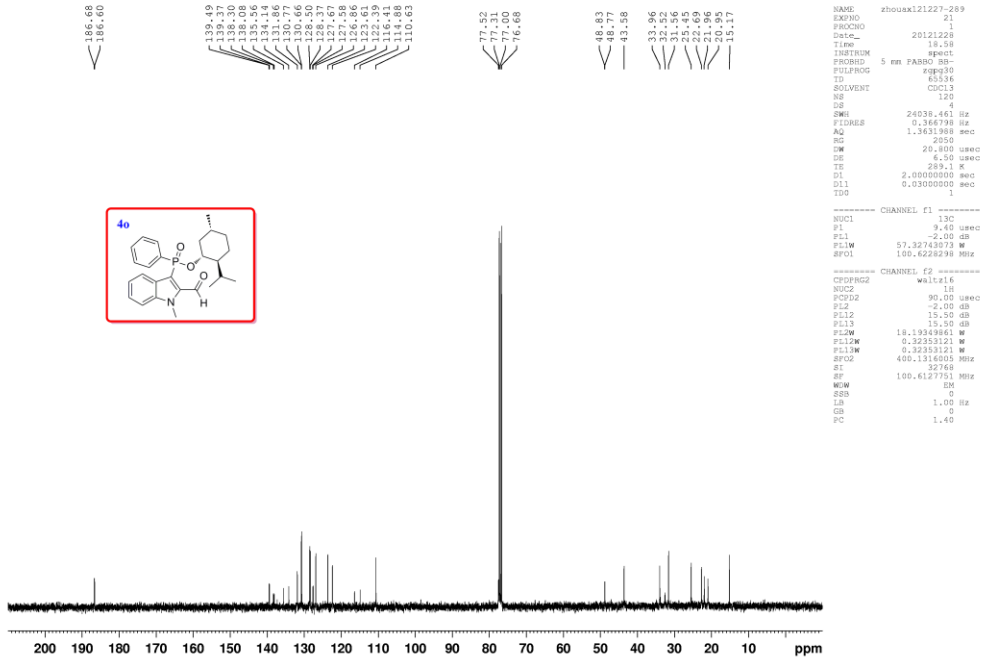

180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

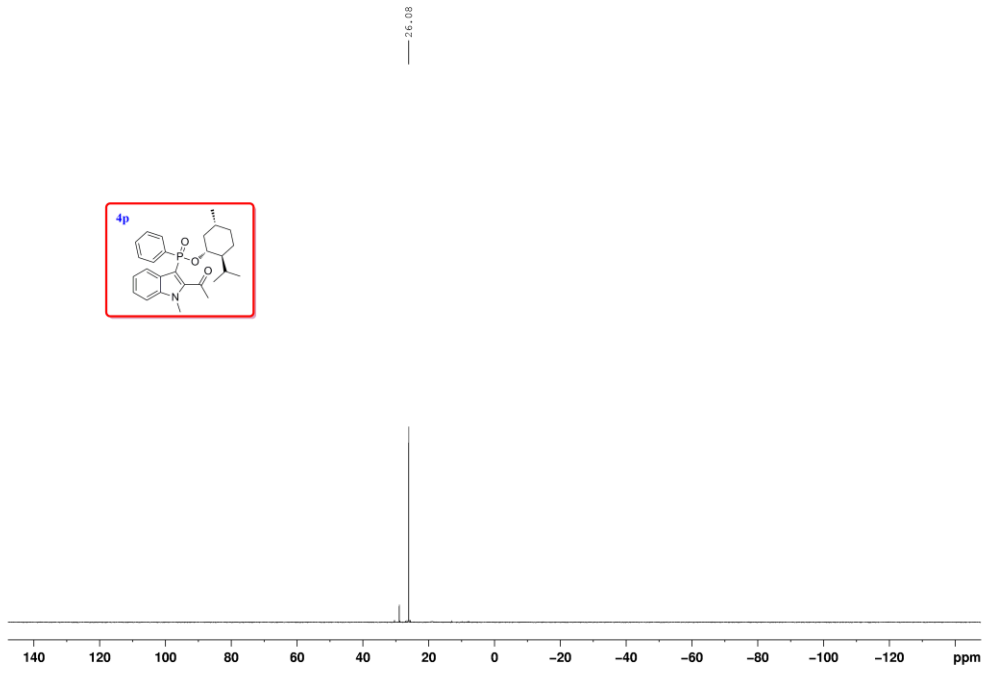


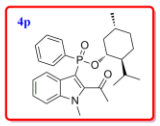

```
NAME            zhouax121227-289
EXPNO                        10
PROCNO                        1
Date_                  20121228
Time                      10.15
INSTRUM                   spect
PROBHD   5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           4
SWH                  64102.563 Hz
FIDRES               0.978127 Hz
AQ                   0.5112308 sec
RG                        2050
DW                       7.800 usec
DE                       6.50 usec
TE                      288.9 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       31P
P1                       10.80 usec
PL1                       0.00 dB
PL1W               26.12361908 W
SFO1              161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   80.00 usec
PL2                      -2.10 dB
PL12                     13.90 dB
PL13                     13.90 dB
PL2W               17.72104263 W
PL12W               0.44513249 W
PL13W               0.44513249 W
SFO2              400.1316005 MHz
SI                        32768
SF                161.9755930 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```

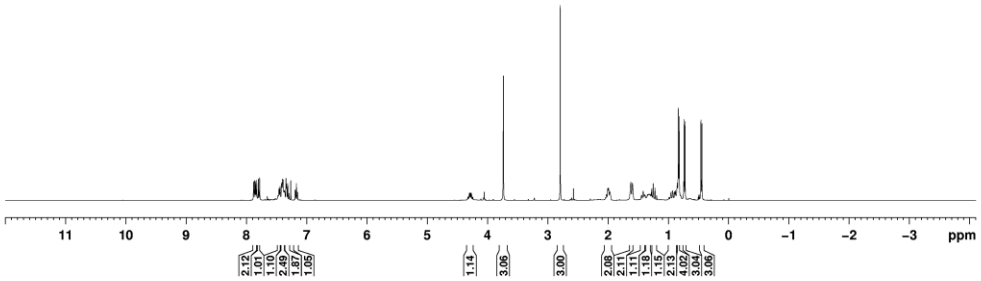


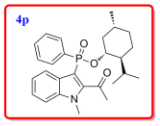

```
NAME            zhouax121227-289
EXPNO                        20
PROCNO                        1
Date_                  20121228
Time                      18.51
INSTRUM                   spect
PROBHD   5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                   8223.685 Hz
FIDRES               0.125483 Hz
AQ                   3.9846387 sec
RG                          90.5
DW                       60.800 usec
DE                       6.50 usec
TE                      288.6 K
D1                 1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       1H
P1                       12.00 usec
PL1                      -3.00 dB
PL1W               22.90425682 W
SFO1              400.1324710 MHz
SI                        32768
SF                400.1300033 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```

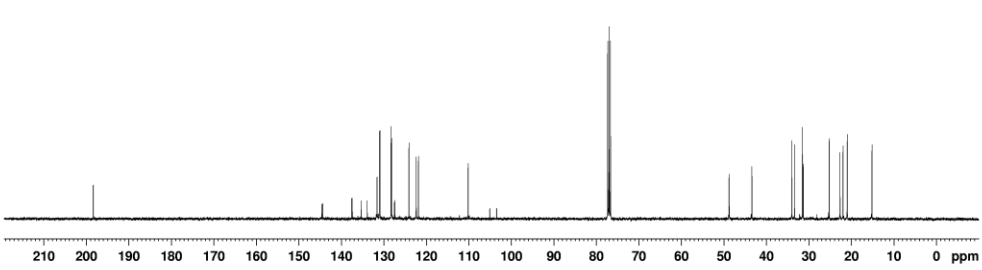

186.68
186.60
139.49
139.37
138.30
138.08
135.56
134.14
131.86
130.97
130.66
128.50
128.37
127.67
127.58
126.86
123.61
122.39
116.41
114.48
110.63
77.52
77.31
77.00
76.68
48.83
40.77
43.58
33.96
32.52
31.56
25.45
22.69
21.96
20.95
15.17
4o
NAME zhouax121227-289
EXPNO 21
PROCNO 1
Date_ 20121228
Time 18.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 120
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 289.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.40 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.50 dB
PL13 15.50 dB
PL2W 18.19349861 W
PL12W 0.32353121 W
PL13W 0.32353121 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127751 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

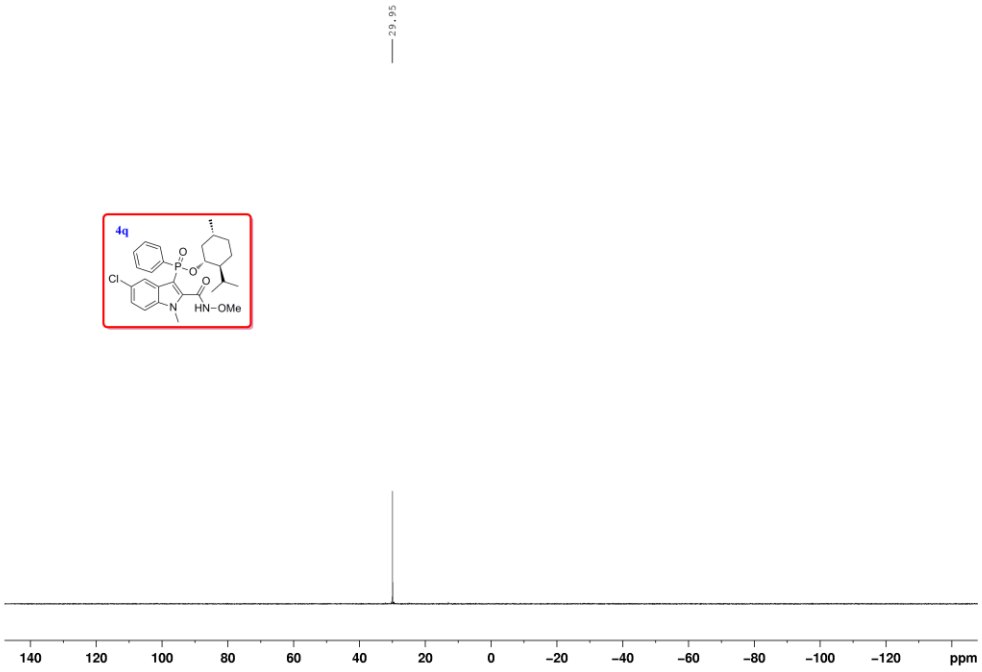

26.08
4p
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 ppm

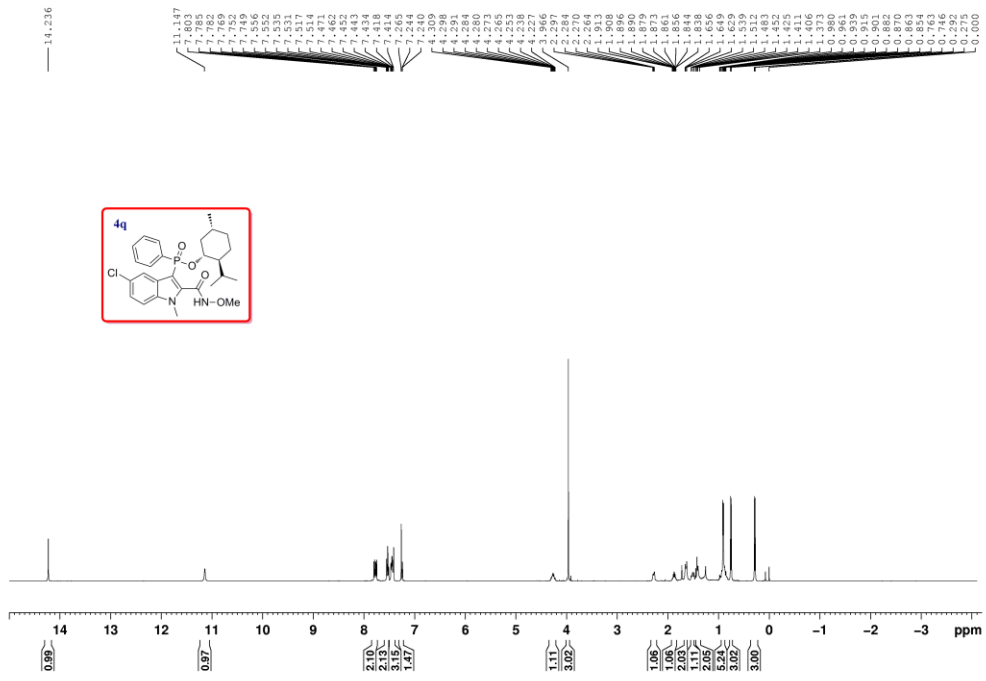

4p

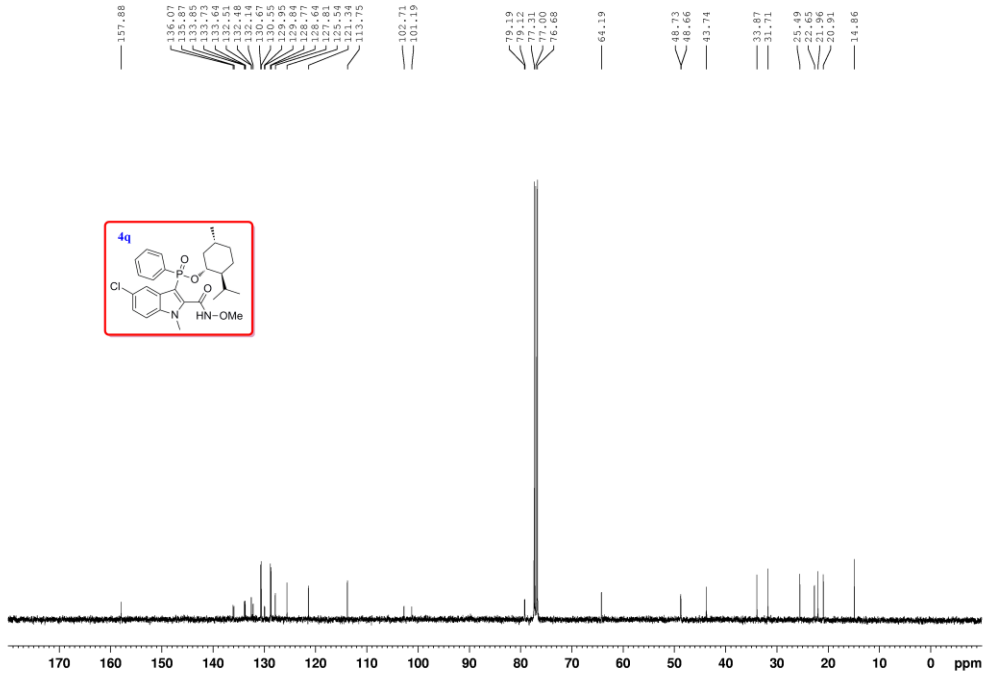

11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3 ppm
2.12 1.01 1.10 2.40 1.87 1.05 1.14 3.06 3.00 2.08 2.11 1.11 1.18 1.15 2.13 4.02 3.04 3.06


4p


210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


29.95
4q
Cl
HN–OMe
140
120
100
80
60
40
20
0
–20
–40
–60
–80
–100
–120
ppm


14.236
11.147
7.803
7.785
7.782
7.769
7.753
7.749
7.556
7.552
7.535
7.531
7.517
7.514
7.471
7.462
7.452
7.443
7.434
7.418
7.414
7.246
7.244
7.240
4.309
4.298
4.291
4.284
4.280
4.273
4.265
4.253
4.238
4.227
3.966
3.957
2.284
2.270
2.264
1.913
1.908
1.896
1.890
1.879
1.873
1.866
1.856
1.844
1.838
1.636
1.629
1.539
1.512
1.483
1.452
1.425
1.411
1.406
1.393
1.380
0.961
0.939
0.915
0.901
0.882
0.870
0.863
0.854
0.763
0.746
0.292
0.275
0.000
4q
Cl
HN–OMe
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
–1
–2
–3
ppm
0.98
0.97
2.10
2.13
3.15
1.47
1.11
9.02
1.06
1.06
2.03
1.11
2.05
5.24
3.02
3.00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136.07
135.87
133.85
133.73
133.64
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132.48
132.14
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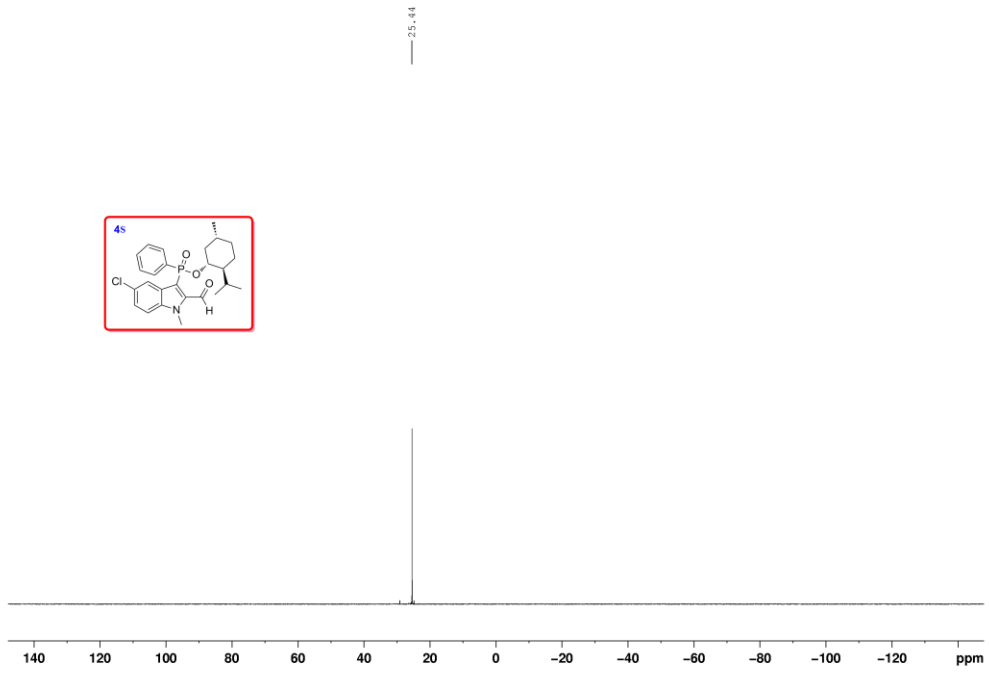
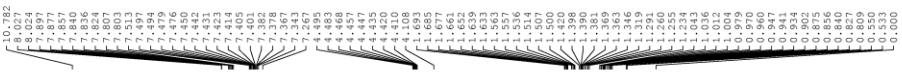
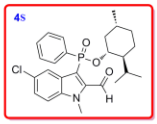
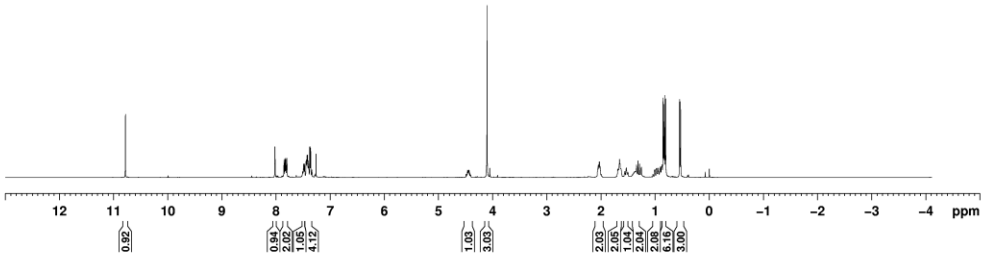
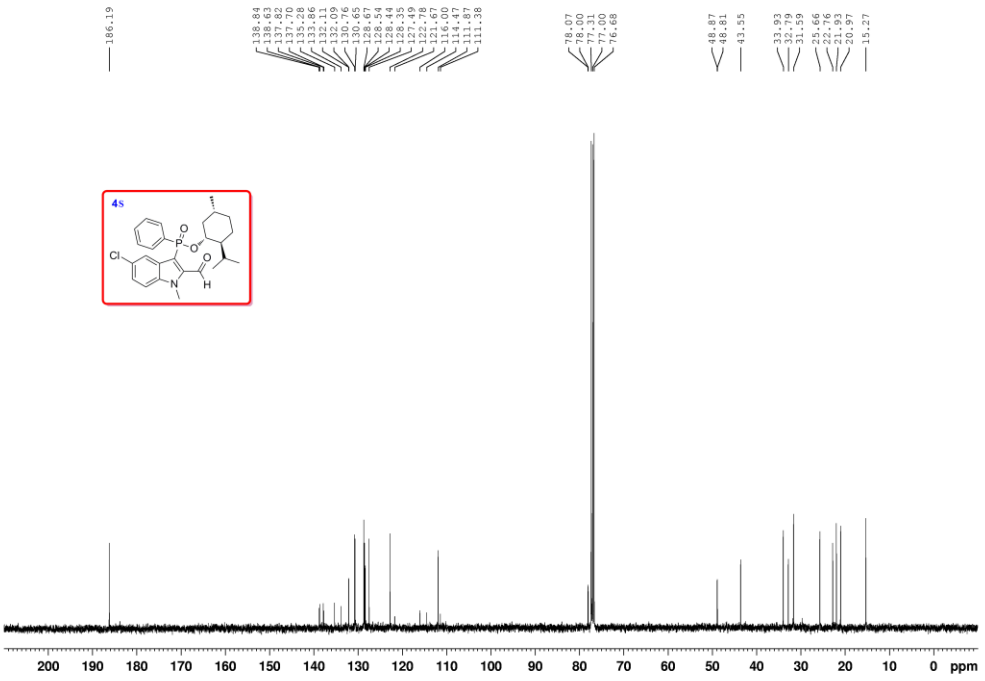
129.95
129.84
128.77
128.64
127.81
125.54
121.34
113.75
102.71
101.19
79.19
79.12
77.31
77.00
76.68
64.19
48.73
48.66
43.74
33.87
31.71
25.49
22.65
21.96
20.91
14.86
4q
Cl
N
HN−OMe
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


25.44
4s
Cl
N
H
140
120
100
80
60
40
20
0
−20
−40
−60
−80
−100
−120
ppm

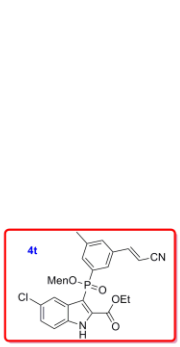


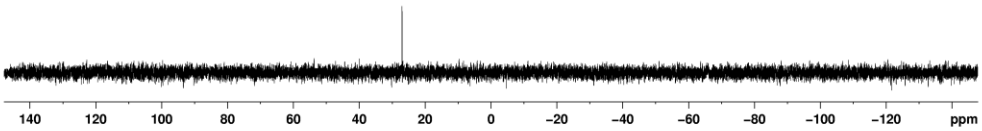

4S
Cl
O
P
O
O
N
H

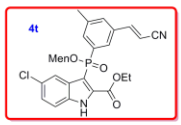

12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
−4
ppm
0.92
0.94
2.02
1.05
4.12
1.03
3.03
2.03
2.05
1.04
2.04
2.08
6.16
3.00

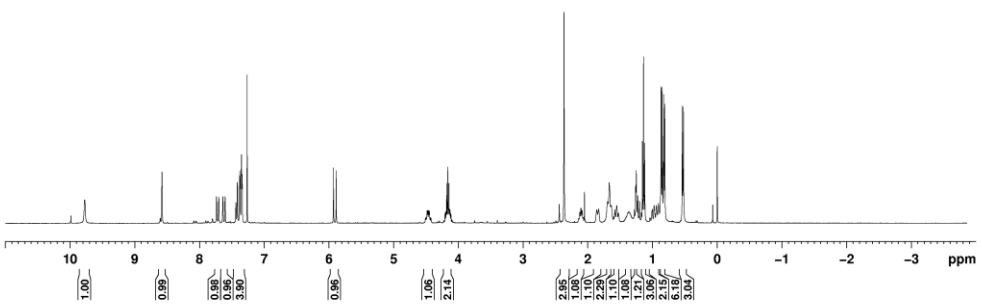

186.19
138.84
138.63
137.82
137.70
135.28
133.86
132.11
132.09
130.76
130.65
128.67
128.54
128.44
128.35
127.49
127.38
121.67
116.00
114.47
111.87
111.38
78.07
78.00
77.31
77.00
76.68
48.87
48.81
43.55
33.93
32.79
31.59
25.66
22.76
21.93
20.97
15.27
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

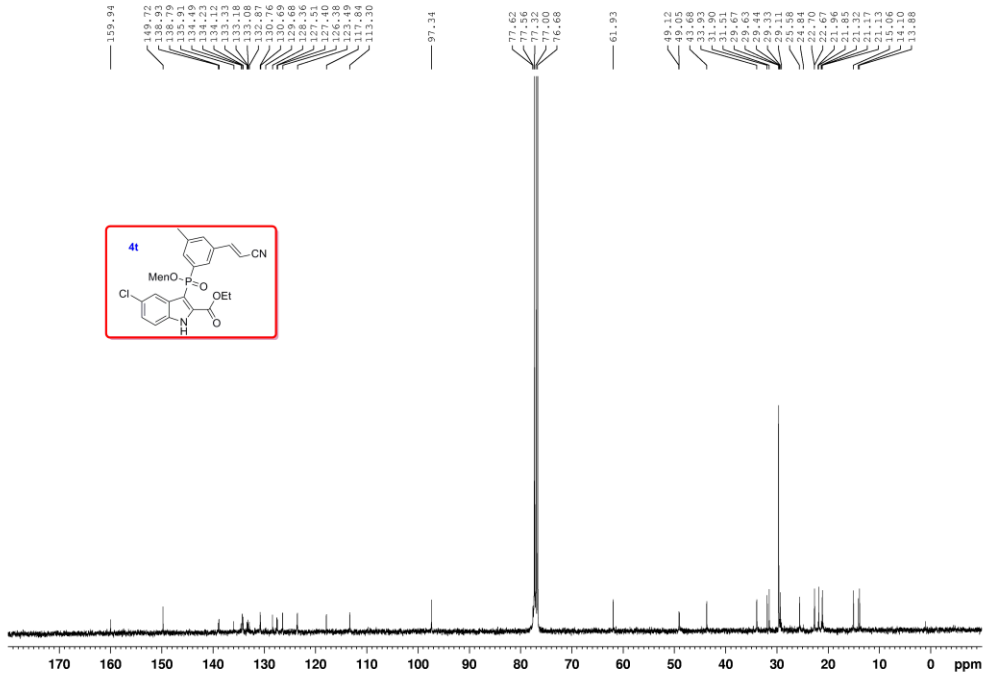
4S
Cl
O
P
O
O
N
H


4t
CN
MenO
P
O
Cl
OEt
N
H
O


140
120
100
80
60
40
20
0
−20
−40
−60
−80
−100
−120
ppm


4t
CN
MenO
P
O
Cl
OEt
N
H
O


10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
ppm
1.00
0.99
0.98
0.96
3.99
0.96
1.06
2.14
2.95
1.08
1.10
2.29
1.10
1.08
1.21
3.06
2.19
6.18
3.04


159.94
149.72
138.93
138.79
135.91
134.49
134.23
134.12
133.33
133.18
133.08
132.87
130.76
130.69
129.68
129.56
128.31
127.40
126.38
123.49
113.84
113.30
97.34
77.62
77.56
77.32
77.00
76.68
61.93
49.12
49.05
43.68
33.93
31.90
31.51
29.67
29.63
29.44
29.33
29.11
24.58
24.84
22.70
22.67
21.96
21.85
21.32
21.17
21.13
14.06
14.10
13.88
4t
CN
MenO
P
O
OEt
Cl
N
H
O
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm