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Supporting Information

Synthesis and Antitumor Activities of α-Hydroxyamino Phosphine Oxides by Catalyst-free Hydrophosphinylation of Nitrones

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General Information

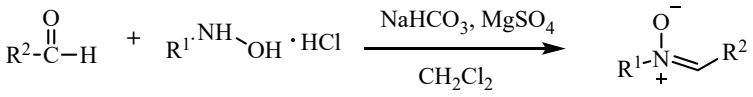
All the solvents were purified according to the solvents handbook. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. Nitrone 1a-1at needed in the experimen were prepared by condensation of commercially available N-alkyl hydroxylamine hydrochlorides with the corresponding aldehydes according to reported procedure, and the circular nitrone 4a-4g is self-made by our laboratory. Thin layer chromatography (TLC) was performed on glass 0.25mm silica gel plates. Column chromatography was carried out employing silica gel (300-400 mesh). Precoated silica gel plates F-254 were used for thin-layer analytical chromatography visualizing with UV and/or acidic aq. $KMnO_4$ solution.

The model of rotary evaporator is SB-1300 (Shanghai, China). All cancer cell lines were provided by the Stem Cell Bank of the Chinese Academy of Sciences (Shanghai, China). All new compounds were characterized by ^{1}H NMR, ^{13}C NMR and HRMS. The ^{1}H and ^{13}C NMR spectra of different products were tested with the BRUKER-AVANCE III (400 or 500 MHz) instrument and TMS (tetramethylsilane) was chosed as the internal standard. High resolution mass spectra (HRMS) were measured using electrospray ionization (ESI) and a time-of-flight (TOF) mass analyzer. The measurements were done in the positive ion mode (interface capillary voltage −4500 V) or in the negative ion mode (3200 V); mass range from m/z 50 to m/z 3000. HPLC spectra were recorded on a SHIMADZU LC-2030.

Experimental procedures, bioassay and spectral analysis

1. Experimental procedures

1) General procedure for preparation of nitrones(1a-1at).

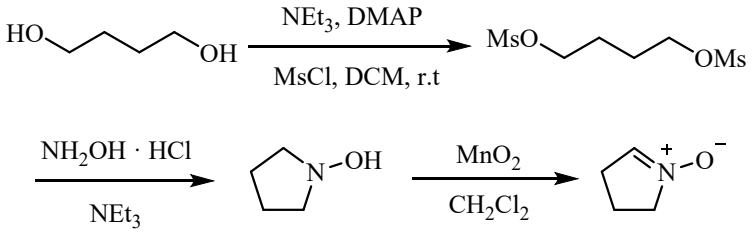


R^1, R^2 = aryl, alkyl or alcyl

The synthesis method of nitrone was improved according to the literature.[1-2]

Aryl or aliphatic aldehydes (1.0 mmol) were added to a solution containing hydroxylamine hydrochloride (1.0 mmol), $NaHCO_3$ (4.0 mmol) $MgSO_4$ (4.0 mmol) and dichloromethane (5 mL) and stirred at room temperature for 4-6 h. After completion of the reaction monitored by thin layer chromatography (TLC), the mixture was washed with dichloromethane (3 × 10 mL). The organic combination was dried with Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by recrystallization (ethyl acetate/hexane) or column chromatography (ethyl acetate/petroleum ether).

2) Preparation of cyclic nitrone(4a-4g).

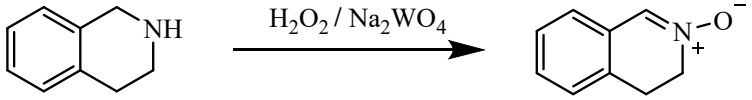


3,4-dihydropyrroline-1-oxide (**4a**) was synthesized using literature methods with some modifications.[3-4]

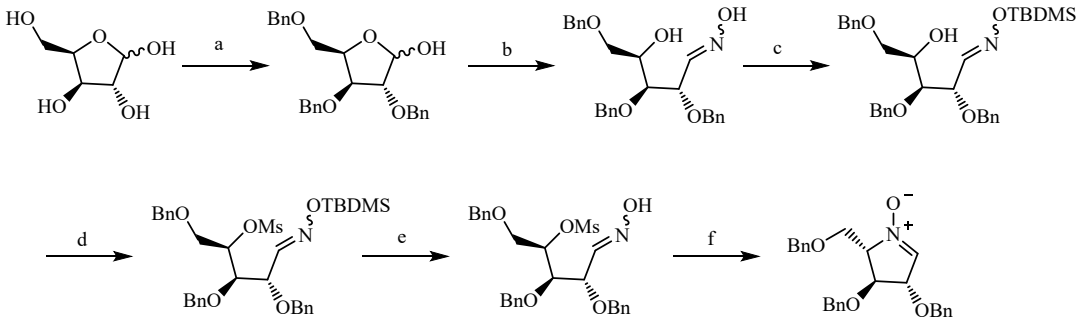
1,4-Butanediol (10 mmol) was dissolved in dry DCM (40 mL), THF (30 mmol), DMAP (0.1 mmol) were added sequentially, followed by the addition of MsCl (12 mmol) dropwise at 0 °C. The reaction was monitored by TLC for 2 h. The reaction was neutralized with 1N HCl and the mixture was washed with DCM (3×30 mL). The organic combination was dried and filtered with Na_2SO_4, the solvent was evaporated, and the crude product was directly subjected to the next reaction step.

A 250 mL round bottom flask equipped with a reflux condenser was filled with the product of the previous step, hydroxylamine hydrochloride (4 equiv), and dry Et_3N (8 mL/mmol) under nitrogen and heated at reflux for 2-3 h. The Et_3N was then evaporated off and the resulting white solid was washed thoroughly with ether. The extract was then concentrated to give crude N-hydroxypyrrolidine. Activated manganese dioxide (3 equiv) was added to the solution in DCM of N - hydroxypyrrolidine in one portion (10 mL/ mmol). The suspension was then stirred at room temperature for 2-3 h. The reaction mixture was filtered and concentrated to

give the crude product, which was purified by column chromatography to give a sufficiently pure pale yellow oily product. Other cyclic nitrone (**4b**, **4c**) were also synthesized by this method.

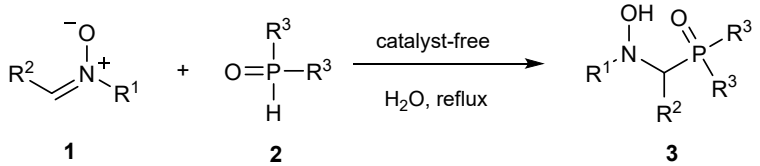


3,4-dihydroisoquinoline 2-oxide (**4d**) was synthesized using a literature procedure.[5]
Under nitrogen, a 50 mL Schlenk flask equipped with the magnetic stir bar was charged with 1,2,3,4-tetrahydroisoquinoline (1g, 7.5 mmol), Na_2WO_4 (0.1 g, 0.3 mmol) and MeOH (2 mL). After the mixture was cooled to 0°C, 30% H_2O_2 (2.6 mL, 22.5 mmol) was added dropwise via syringe. The reaction was stirred for another 30 minutes and then concentrated on rotary evaporator, diluted with brine and extracted with DCM (3×30 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered and the solvent was removed under vacuum (rotary evaporator) to obtain crude product, which was purified by column chromatography (ethyl acetate/petroleum ether).
(2S,3S,4S)-3,4-bis(benzyloxy)-2-((benzyloxy)methyl)-3,4-dihydro-2H-pyrrole 1-oxide (**4f**) was synthesized using a literature procedure.[6]



Reagents and conditions: (a) i) conc. H_2SO_4, methanol; ii) NaH, DMF, 0℃ Bu_4NI, BnBr, rt; iii) MeCN: H_2O: TFA=5:3:1, reflux; (b) $NH_2OH.HCl$, MeOH, NaOMe, reflux; (c) TBDMSCl, pyridine; (d) MsCl, pyridine, DCM; (e) TBAF, THF, 0°C (f) methanol: H_2O=4:1; $NaHCO_3$, $NH_2OH.HCl$, reflux.
Other circular nitrone **4e**, **4g** were also synthesized by this method.

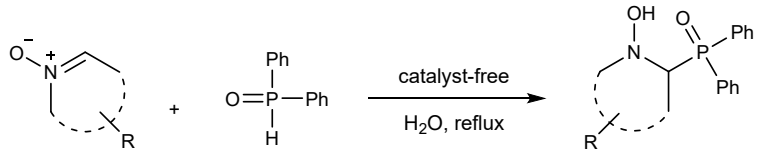
3) General procedure for preparation of α-(hydroxyamino) diaryl phosphine oxides.



1 **2** **3**

R^1, R^2 = aryl, alkyl or alcyl; R^3 = aryl
In a dry round bottom flask with a volume of 25 mL, add nitrone **1** (1.0 mmol),

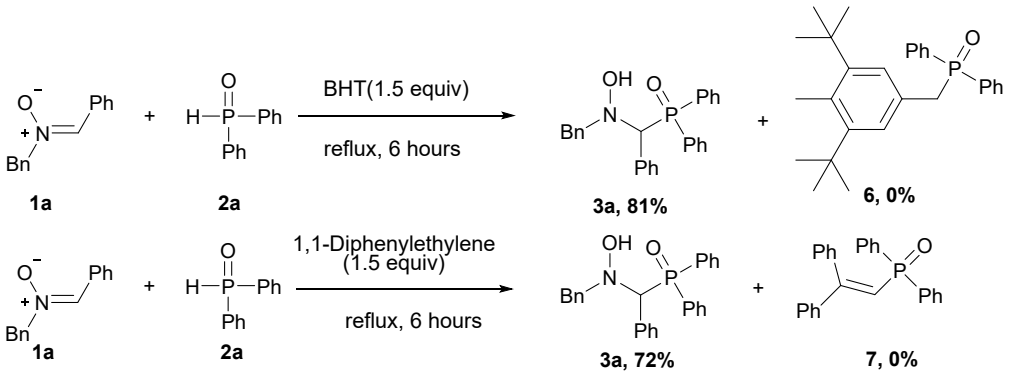
diarylphosphine oxide **2** (1.2 mmol), water (2 mL) in sequence. Put into a magnetic stirrer, placed on a magnetic stirrer stirring at 500 rpm, reflux reaction for 3-6 h. When the reaction is monitored by TLC and no nitrone compounds remain, the mixed solution is distilled under reduced pressure to remove the solvent to obtain the primary product. Then purified by recrystallization, beating or column chromatography to obtain pure product **3**.



4 **2a** **5**

In a dry round bottom flask with a volume of 25 mL, add nitrone **4** (1.0 mmol), diarylphosphine oxide **2a** (1.2 mmol), water (2 mL) in sequence. Put into a magnetic stirrer, placed on a magnetic stirrer stirring at 500 rpm, reflux reaction for 3-6 h. When the reaction is monitored by TLC and no nitrone compounds remain, the mixed solution is distilled under reduced pressure to remove the solvent to obtain the primary product. Then purified by recrystallization, beating or column chromatography to obtain pure product **5**.

4) Radical capture experiment.

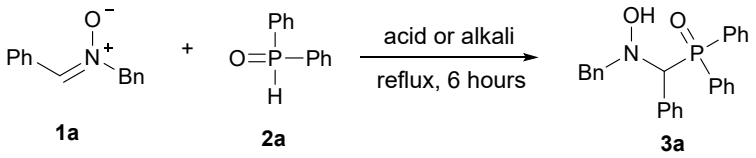


1a **2a** **3a, 81%** **6, 0%**

1a **2a** **3a, 72%** **7, 0%**

In a dry round bottom flask with a volume of 25 mL, nitrone **1a** (1.0 mmol), diarylphosphine oxide **2a** (1.2 mmol), and water (2 mL) were added sequentially. After 5 min of reaction, BHT or 1,1-Diphenylethylene was added and the reaction was refluxed for 6 h. When TLC analysis determined that the reaction was complete, the mixed solution was distilled under reduced pressure to remove the solvent to obtain the primary product. Then the primary product was purified by column chromatography (solvent: ethyl acetate/petroleum ether) to obtain the pure product.

5) Mechanistic Studies.



1a 2a 3a

Entry	Catalyst	Yield(%)
1	HCl	68
2	AcOH	56
3	NaOH	68
4	TEA	70

Reaction conditions: nitrone 1a (1.0 mmol), diaryl phosphine oxide 2a (1.2 mmol), 2mL H2O, acid: HCl(10 mol %) AcOH(10 mol %), alkali: NaOH(10 mol %) TEA(10 mol %).

In a dry round bottom flask of volume 10 mL, nitrone **1a** (1.0 mmol), diarylphosphine oxide **2a** (1.2 mmol) and water (2 mL) were added sequentially. The corresponding acid or base was then added and the reaction was refluxed for 6 h. When TLC analysis determined that the reaction was complete, the mixed solution was distilled under reduced pressure and the solvent was removed to obtain the primary product. The primary product was then purified by column chromatography (solvent: ethyl acetate/petroleum ether) to obtain the pure product.

2. Biological assays

1) Cell lines and culture conditions.

The cytotoxic activity of the synthesized compounds against A-549 (lung cancer), MCF-7 (breast cancer), Hep G2 (liver cancer) BEAS-2B (human normal lung epithelial cells) was investigated. All cells were grown in RPMI-1640 supplemented with 10% heat inactivated fetal bovine serum (FBS), 100 IU/mL penicillin, 100 mg/mL streptomycin and 2 mm-glutamine. Cultures were maintained in a humidifified atmosphere with 5% CO_2 at 37 ℃. The cells were subcultured twice each week, seeding at a density of about 1×10^5 cells/mL. Before the analysis of the compounds, cells were washed with PBS and fresh medium was added. For fifinal analysis, exponentially growing cells were collected and resuspended in fresh culture medium with 10% FBS. Cells were maintained in a humidified incubator with 5% CO_2 at 37 ℃.

2) MTT assay.

Cytotoxic activity was determined by the 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide (MTT) assay as described. Camptothecin was selected as a positive control. Cell suspensions were prepared at a concentration of 1×10^5μg/mL, added to 96-well plates at 100μL per well, and incubated at 37 °C in a 5% CO_2 incubator for 24h. Different concentrations of subjects were added to the 96-well plates of cultured tumor cells and normal cells, and incubation was continued for 24h and observed under an inverted microscope. The culture medium was discarded, and 100 μL of 0. 05% MTT application solution was added to each well and incubated for 4 h. The culture medium was discarded, 100 μL of DMSO was added to each well and shaken for 5 min to dissolve the methylzan crystals, and the absorbance (OD) of the cells was measured at 490 nm.

The concentration and growth inhibition curves were obtained by plotting the growth inhibition rates at different doses and plotted using GraphPad Prism 9.0 software to calculate the semi-inhibitory concentration IC_{50} of the target compounds on three tumor cells and one normal cell.

Effects of synthesized α-(hydroxyamino) phosphine oxides against human cancer cell lines of different tissues ($IC5_0$, μM).

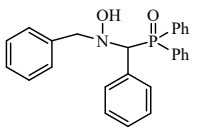
Compound	IC50 (μM)		
	A549	MCF-7	Hep G2
3a	NA	NA	NA
3b	NA	NA	NA
3c	NA	NA	NA
3d	208.1(±0.8)	247.1(±0.2)	NA
3e	160.7 (±2.4)	NA	237.4(±0.5)
3f	NA	NA	NA
3g	NA	NA	NA
3h	29.9(±1.2)	24.3(±0.1)	29.8(±0.4)
3i	62.4(±0.5)	79.0(±1.0)	112.5(±0.9)
3j	NA	NA	NA
3k	21.7(±0.8)	36.5(±0.5)	28.4(0.1)
3l	NA	NA	NA
3m	23.5(±1.6)	17.6(±0.4)	35.0(±0.9)
3n	46.3(±0.5)	22.81(±1.4)	49.8(±0.4)
3o	62.4(±0.5)	79.0(±1.2)	112.5(±0.9)
3p	NA	NA	NA
3q	NA	NA	NA
3r	NA	NA	NA
3s	90.3(0.7)	166.5(±1.2)	143.2(±0.1)
3t	NA	NA	NA
3u	NA	NA	NA
3v	NA	NA	NA

3w	NA	NA	NA
3x	210.2(±0.3)	132.8(±0.1)	165.8(±0.5)
3y	NA	NA	NA
3z	NA	NA	NA
3aa	NA	NA	NA
3ab	NA	NA	NA
3ac	NA	NA	NA
3ad	NA	NA	NA
3ae	NA	NA	NA
3af	NA	NA	NA
3ag	218.1(±0.9)	NA	NA
3ah	60.7(±0.2)	66.8(±0.1)	91.6(±1.0)
3ai	59.4(±0.3)	94.7(±0.2)	57(±0.7)
3aj	NA	NA	NA
3ak	247.8(±0.3)	347.0(±0.9)	211.5(±0.5)
3al	138.6(±0.1)	NA	96.8(±1.8)
3am	NA	NA	NA
3an	NA	NA	NA
3ao	NA	NA	NA
3ap	NA	NA	NA
3aq	NA	NA	NA
3ar	NA	NA	NA
3as	53.5(±0.3)	36.8(±0.1)	45.8(±0.6)
3at	26.4(±0.7)	28.9(±0.1)	27.1(±0.5)
5a	35.6(±0.4)	68.7(±0.5)	31.9(±0.8)
5b	210.4(±0.1)	185.9(±0.8)	NA
5c	NA	254.0(±1.3)	344.9(±0.5)
5d	143.9(±0.7)	119.4(±0.8)	195.8(±2.0)
5e	6.8(±1.5)	4.7(±2.2)	9.5(±0.7)
5f	9.7(±0.2)	2.1(±1.0)	8.7(±0.1)
5g	55.2(±1.1)	47.2(±1.0)	56.8(±1.1)
Camptothecin	4.0(±0.1)	3.1 (±0.1)	3.6(±0.1)

(±) Standard deviation; n=3.

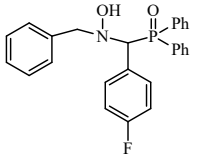
NA: Not active up to signifificant concentration.

3. spectral analysis

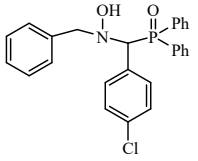


((benzyl(hydroxy)amino)(phenyl)methyl)diphenylphosphine oxide (3a): Following the **general procedure**, 3h, 96% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (t, *J* = 9.2 Hz, 2H),

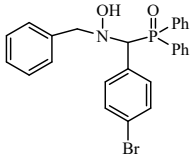
7.50 (m, 7H), 7.38 – 6.98 (m, 11H), 6.72 (s, 1H), 4.64 (d, *J* = 7.2 Hz, 1H), 4.10 (d, *J* = 13.1 Hz, 1H), 3.64 (d, *J* = 13.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.9, 135.5, 133.2, 131.8 (d, *J* = 8.8 Hz), 131.6 (d, *J* = 9.0 Hz), 131.3 (d, *J* = 97.2 Hz), 129.4, 128.5, 128.4 (d, *J* = 11.5 Hz), 128.1 (d, *J* = 11.4 Hz), 127.5, 68.9 (d, *J* = 88.2 Hz), 61.5 (d, *J* = 11.4 Hz). HRMS (ESI) m/z Calculated for $C_{26}H_{24}NO_2P$ $[M\text{-}Na]^+$ 436.1442, found 436.1440.



((benzyl(hydroxy)amino)(4-fluorophenyl)methyl)diphenylphosphine oxide (3b): Following the **general procedure**, 3h, 85% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 – 7.78 (m, 2H), 7.56 (td, *J* = 7.3, 1.5 Hz, 1H), 7.50 – 7.38 (m, 6H), 7.33 – 7.29 (m, 1H), 7.27 – 7.19 (m, 5H), 7.12 – 7.05 (m, 2H), 6.93 (t, *J* = 8.6 Hz, 2H), 6.72 (s, 1H), 4.59 (d, *J* = 7.6 Hz, 1H), 4.06 (d, *J* = 13.1 Hz, 1H), 3.58 (d, *J* = 13.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.8 (d, J_{CF}= 247.4 Hz), 136.6, 133.2 (d, *J* = 7.3 Hz), 133.1 (d, *J* = 5.3 Hz), 131.5 (d, *J* = 2.9 Hz), 131.4 (d, *J* = 97.9 Hz), 131.3 (d, J_{CF} = 8.9 Hz), 130.9 (d, *J* = 8.7 Hz), 129.1, 128.1 (d, *J* = 24.4 Hz), 128.1 (d, *J* = 3.8 Hz), 127.9, 127.3, 114.9 (d, *J* = 21.3 Hz), 67.8 (d, *J* = 89.9 Hz), 61.1 (d, *J* = 11.4 Hz). HRMS (ESI) m/z Calculated for $C_{26}H_{23}FNO_2P$ $[M\text{-}Na]^+$ 454.1348, found 454.1345.

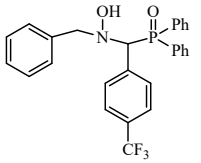


((benzyl(hydroxy)amino)(4-chlorophenyl)methyl)diphenylphosphine oxide (3c): Following the **general procedure**, 3h, 87% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 – 7.81 (m, 2H), 7.59 (td, J = 7.3, 1.5 Hz, 1H), 7.53 – 7.41 (m, 6H), 7.36 (td, *J* = 7.3, 1.4 Hz, 1H), 7.30 – 7.22 (m, 7H), 7.13 – 7.04 (m, 2H), 6.69 (s, 1H), 4.61 (d, *J* = 7.6 Hz, 1H), 4.08 (d, *J* = 13.1 Hz, 1H), 3.60 (d, *J* = 13.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.5, 134.5 (d, *J* = 1.6 Hz), 133.10 (d, *J* = 6.3 Hz), 131.90 (d, *J* = 2.5 Hz), 131.81 (d, *J* = 2.7 Hz), 131.6 (d, *J* = 9.0 Hz), 131.2 (d, *J* = 8.6 Hz), 128.6 (d, *J* = 11.8 Hz), 128.4 (d, *J* = 3.6 Hz), 128.0 (d, *J* = 94.9 Hz), 68.2 (d, *J* = 86.3 Hz), 61.5 (d, *J* = 11.8 Hz). HRMS (ESI) m/z Calculated for $C_{26}H_{23}ClNO_2P$ $[M\text{-}Na]^+$ 447.1053, found 447.1044.

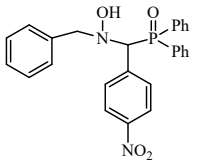


((benzyl(hydroxy)amino)(4-bromophenyl)methyl)diphenylphosphine oxide (3d): Following the **general procedure**, 3h, 87% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.78 (m, 2H), 7.56

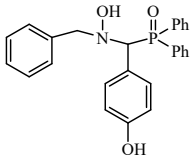
(td, *J* = 7.3, 1.5 Hz, 1H), 7.51 – 7.45 (m, 2H), 7.44 – 7.31 (m, 7H), 7.27 – 7.20 (m, 5H), 7.11 – 7.03 (m, 2H), 6.72 (s, 1H), 4.58 (d, *J* = 7.6 Hz, 1H), 4.05 (d, *J* = 13.1 Hz, 1H), 3.58 (d, *J* = 13.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.5, 133.4 (d, *J* = 6.4 Hz), 131.9 (d, *J* = 2.6 Hz), 131.8 (d, *J* = 2.1 Hz), 131.6 (d, *J* = 8.8 Hz), 131.3, 131.2 (d, *J* = 8.8 Hz), 130.4 (d, *J* = 94.2 Hz), 129.4, 128.6, 128.5 (d, *J* = 4.4 Hz), 128.4 (d, *J* = 11.6 Hz), 127.6, 68.3 (d, *J* = 84.1 Hz), 61.5 (d, *J* = 11.2 Hz). HRMS (ESI) m/z Calculated for $C_{26}H_{23}BrNO_2P$ $[M\text{-}Na]^+$ 514.0547, found 514.0543.



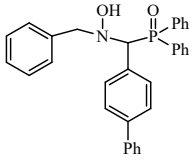
((benzyl(hydroxy)amino)(4-(trifluoromethyl)phenyl)methyl)diphenylphosphine oxide (3e): Following the **general procedure**, 3h, 88% yield, white solid, new compound, (hexane/ethyl acetate = 9/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (m, 2H), 7.50 m 3H), 7.41 (m, 4H), 7.36 – 7.28 (m, 2H), 7.24 (td, *J* = 7.4, 1.5 Hz, 1H), 7.19 – 7.10 (m, 5H), 7.01 (dd, *J* = 7.4, 2.2 Hz, 2H), 6.71 (s, 1H), 4.58 (d, *J* = 7.3 Hz, 1H), 3.99 (d, *J* = 13.1 Hz, 1H), 3.50 (d, *J* = 13.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.5, 135.8, 132.7, 132.1 (d, *J* = 6.3 Hz), 131.9 (d, *J* = 2.8 Hz), 131.8 (d, *J* = 2.8 Hz), 131.7, 131.6 (d, *J* = 8.9 Hz), 131.1 (d, *J* = 8.8 Hz), 130.4 (d, $J_{C\text{-}F}$ = 32.3 Hz), 130.3, 129.4, 128.6 (d, *J* = 11.8 Hz), 128.4 (d, *J* = 5.0 Hz), 128.0 (d, *J* = 92.1 Hz), 124.1 (d, $J_{C\text{-}F}$ = 272.2 Hz), 125.0 (d, *J* = 3.9 Hz), 124.9 (d, *J* = 3.9 Hz), 68.7 (d, *J* = 88.8 Hz), 61.7 (d, *J* = 11.1 Hz). HRMS (ESI) m/z Calculated for $C_{27}H_{23}F_3NO_2P$ $[M\text{-}Na]^+$ 504.1316, found 504.1305.



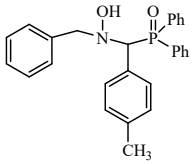
((benzyl(hydroxy)amino)(4-nitrophenyl)methyl)diphenylphosphine oxide (3f): Following the **general procedure**, 4h, 83% yield, white solid, new compound, (hexane/ethyl acetate = 6/4). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 8.4 Hz, 2H), 7.87 – 7.81 (m, 2H), 7.64 (d, *J* = 8.3 Hz, 2H), 7.58 (dd, *J* = 7.5, 1.6 Hz, 1H), 7.49 (td, *J* = 7.6, 2.1 Hz, 2H), 7.44 – 7.36 (m, 3H), 7.33 (dd, *J* = 7.5, 1.6 Hz, 1H), 7.28 – 7.22 (m, 5H), 7.12 – 7.07 (m, 2H), 4.72 (d, *J* = 7.0 Hz, 1H), 4.07 (d, *J* = 13.1 Hz, 1H), 3.59 (d, *J* = 13.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.6, 139.5, 136.1, 132.5 (d, *J* = 6.2 Hz), 132.3, 132.0 (d, *J* = 2.9 Hz), 131.9 (d, *J* = 2.8 Hz), 131.5 (d, *J* = 9.0 Hz), 131.3 (d, *J* = 6.2 Hz), 131.0 (d, *J* = 8.6 Hz), 129.9 (d, *J* = 10.7 Hz), 129.3, 128.5 (d, *J* = 12.1 Hz), 128.4 (d, *J* = 2.4 Hz), 128.2 (d, *J* = 18.9 Hz), 127.9 (d, *J* = 96.1 Hz), 122.9, 68.4 (d, *J* = 87.4 Hz), 62.0 (d, *J* = 11.2 Hz). HRMS (ESI) m/z Calculated for $C_{26}H_{23}N_2O_4P$ $[M\text{-}Na]^+$ 481.1293, found 481.1284.



((benzyl(hydroxy)amino)(4-hydroxyphenyl)methyl)diphenylphosphine oxide (3g): Following the **general procedure**, 4h, 93% yield, white solid, new compound, (hexane). ^{1}H NMR (400 MHz, DMSO) δ 9.46 (s, 1H), 8.07 – 7.92 (m, 3H), 7.64 (m, 2H), 7.53 (dt, *J* = 6.0, 3.9 Hz, 3H), 7.45 – 7.38 (m, 2H), 7.38 – 7.27 (m, 3H), 7.21 – 7.14 (m, 3H), 7.05 (dd, *J* = 7.7, 1.8 Hz, 2H), 6.62 (d, *J* = 8.6 Hz, 2H), 5.20 (d, *J* = 9.6 Hz, 1H), 4.15 (d, *J* = 13.7 Hz, 1H), 3.44 (d, *J* = 13.7 Hz, 1H). ^{13}C NMR (101 MHz, DMSO) δ 166.5, 148.6, 144.1, 143.2 (d, *J* = 13.4 Hz), 142.6 (d, *J* = 7.1 Hz), 142.3, 140.7 (d, *J* = 8.5 Hz), 140.2 (d, *J* = 8.5 Hz), 138.5, 137.9 (d, *J* = 9.1 Hz), 137.8 (d, *J* = 8.8 Hz), 137.5, 136.3, 132.0, 124.1, 78.5 (d, *J* = 85.1 Hz), 70.6 (d, *J* = 5.7 Hz). HRMS (ESI) m/z Calculated for $C_{23}H_{24}NO_3P$[M-Na]$^+$ 452.1391, found 452.1398.

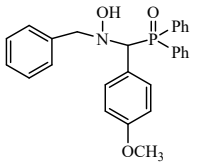


([1,1'-biphenyl]-4-yl(benzyl(hydroxy)amino)methyl)diphenylphosphine oxide (3h): Following the **general procedure**, 5h, 88% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (m, 2H), 7.71 (m, 2H), 7.63 (dd, *J* = 11.7, 7.5 Hz, 2H), 7.55 (d, *J* = 7.5 Hz, 3H), 7.51 – 7.47 (m, 6H), 7.43 (d, *J* = 7.2 Hz, 4H), 7.36 – 7.33 (m, 1H), 7.29 (d, *J* = 7.4 Hz, 1H), 7.25 – 7.22 (m, 3H), 7.13 (dd, *J* = 6.8, 2.6 Hz, 2H), 5.08 (s, 1H), 4.12 (d, *J* = 13.0 Hz, 1H), 3.67 (d, *J* = 13.2 Hz, 1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 141.0 (d, *J* = 1.6 Hz), 140.6, 136.7, 132.7 (d, *J* = 2.5 Hz), 132.2 (d, *J* = 6.7 Hz), 131.8 (d, *J* = 2.2 Hz), 131.7 (d, *J* = 2.1 Hz), 131.6 (d, *J* = 3.6 Hz), 131.3 (d, *J* = 8.8 Hz), 130.8 (d, *J* = 91.5 Hz), 129.4 (d, *J* = 12.9 Hz), 129.2, 129.1 (d, *J* = 3.0 Hz), 129.0 (d, *J* = 2.6 Hz), 128.9, 128.6, 128.4 (d, *J* = 6.2 Hz), 128.2 (d, *J* = 11.7 Hz), 127.9, 127.6, 127.2, 127.1 (d, *J* = 2.9 Hz), 126.8, 70.8 (d, *J* = 88.2 Hz), 61.5 (d, *J* = 12.1 Hz). HRMS (ESI) m/z Calculated for $C_{32}H_{28}NO_2P$[M-Na]$^+$ 512.1755, found 512.1751.

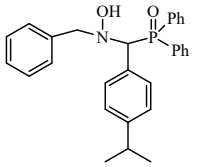


((benzyl(hydroxy)amino)(p-tolyl)methyl)diphenylphosphine oxide (3i): Following the **general procedure**, 3h, 91% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (m, 2H), 7.53 – 7.45 (m, 1H), 7.39 (m, 4H), 7.26 (m, 3H), 7.19 – 7.12 (m, 5H), 7.05 – 7.00 (m, 2H), 6.98 (d, *J* = 7.8 Hz, 2H), 6.60 (s, 1H), 4.50 (d, *J* = 7.4 Hz, 1H), 3.99 (d, *J* = 13.2 Hz, 1H), 3.52 (d, *J* = 13.1 Hz, 1H), 2.22 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.2, 137.0, 133.3, 132.3, 131.7, 131.7 (d, *J* = 3.4 Hz), 131.5 (d, *J* = 4.5 Hz), 131.3 (d, *J* = 8.6 Hz), 129.4, 128.9, 128.5, 128.4 (d, *J* = 4.8 Hz), 128.2, 127.8 (d, *J* = 68.9 Hz), 68.8 (d, *J* = 88.9 Hz), 61.3

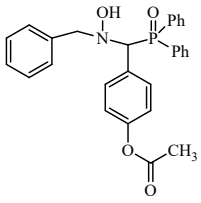
(d, J = 11.5 Hz), 21.3. HRMS (ESI) m/z Calculated for $C_{27}H_{26}NO_2P[M\text{-}Na]^+$ 450.1599, found 450.1590.



((benzyl(hydroxy)amino)(4-methoxyphenyl)methyl)diphenylphosphine oxide (3J): Following the **general procedure**, 4h, 93% yield, white solid, new compound, (hexane/ethyl acetate = 9/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (m, 2H), 7.53 – 7.49 (m, 1H), 7.47 – 7.34 (m, 6H), 7.28 (d, J = 1.7 Hz, 1H), 7.24 – 7.16 (m, 5H), 7.10 – 7.01 (m, 2H), 6.79 – 6.50 (m, 3H), 4.53 (t, J = 7.5 Hz, 1H), 4.04 (d, J = 13.2 Hz, 1H), 3.73 (d, J = 4.3 Hz, 3H), 3.56 (d, J = 13.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.0, 133.1 (d, J = 6.5 Hz), 131.7 (d, J = 2.4 Hz), 131.6 (d, J = 2.4 Hz), 131.3 (d, J = 8.6 Hz), 129.4, 128.5, 128.4 (d, J = 3.6 Hz), 128.2 (d, J = 11.6 Hz), 127.4, 113.6, 68.3 (d, J = 91.1 Hz), 61.3 (d, J = 11.9 Hz), 55.3, 29.8. HRMS (ESI) m/z Calculated for $C_{27}H_{26}NO_3P[M\text{-}Na]^+$ 466.1548, found 466.1550.

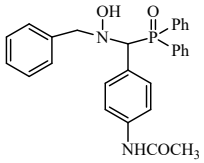


((benzyl(hydroxy)amino)(4-isopropylphenyl)methyl)diphenylphosphine oxide (3k): Following the **general procedure**, 5h, 98% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (m, 1.4 Hz, 2H), 7.57 – 7.51 (m, 1H), 7.46 (m, 2H), 7.42 – 7.34 (m, 4H), 7.31 – 7.26 (m, 1H), 7.24 – 7.15 (m, 5H), 7.14 – 7.05 (m, 4H), 6.66 (s, 1H), 4.59 (d, J = 7.0 Hz, 1H), 4.08 (d, J = 13.1 Hz, 1H), 3.62 (d, J = 13.2 Hz, 1H), 2.83 (p, J = 6.9 Hz, 1H), 1.22 – 1.14 (m, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.0 (d, J = 1.8 Hz), 137.0, 132.1 (d, J = 98.8 Hz), 131.6 (d, J = 3.6 Hz), 131.6 (d, J = 2.1 Hz), 131.3 (d, J = 1.8 Hz), 131.3 (d, J = 13.5 Hz), 130.9, 129.4, 128.5 (d, J = 17.9 Hz), 128.3 (d, J = 5.1 Hz), 128.0 (d, J = 11.6 Hz), 127.4, 126.3, 69.2 (d, J = 90.3 Hz), 61.4 (d, J = 11.5 Hz), 33.9, 24.0 (d, J = 4.5 Hz). HRMS (ESI) m/z Calculated for $C_{29}H_{30}NO_2P[M\text{-}Na]^+$ 478.1912, found 478.1910.

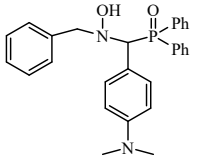


((benzyl(hydroxy)amino)(diphenylphosphoryl)methyl)phenyl acetate (3l): Following the **general procedure**, 4h, 87% yield, white solid, new compound, (hexane/ethyl acetate = 9/1). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 (m, 2H), 7.62 – 7.55 (m, 1H), 7.50 (ddt, J = 10.6, 6.5, 2.2 Hz, 4H), 7.42 (ddt, J = 11.1, 7.0, 1.4 Hz, 2H), 7.36 – 7.32 (m, 1H), 7.24 (ddd, J = 10.4, 6.2, 2.4 Hz, 5H), 7.16 – 7.10 (m, 2H), 7.00

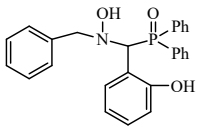
(d, *J* = 8.6 Hz, 2H), 6.56 (s, 1H), 4.63 (d, *J* = 7.4 Hz, 1H), 4.10 (d, *J* = 13.2 Hz, 1H), 3.64 (d, *J* = 13.2 Hz, 1H), 2.29 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.1, 150.8 (d, *J* = 1.8 Hz), 136.9, 132.7 (d, *J* = 6.3 Hz), 132.6 (d, *J* = 100.1 Hz), 131.8 (d, *J* = 2.8 Hz), 131.6 (d, *J* = 8.8 Hz), 131.5, 131.3 (d, *J* = 8.7 Hz), 130.7, 129.4, 129.1, 128.5 (d, *J* = 11.8 Hz), 128.4 (d, *J* = 9.1 Hz), 127.8 (d, *J* = 87.8 Hz), 121.3, 68.8 (d, *J* = 87.2 Hz), 61.6 (d, *J* = 11.4 Hz), 21.3. HRMS (ESI) m/z Calculated for $C_{28}H_{26}NO_4P$[M-Na]$^+$ 494.1497, found 494.1493.



((benzyl(hydroxy)amino)(diphenylphosphoryl)methyl)phenyl)acetamide (3m): Following the **general procedure**, 4h, 85% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, MeOD) δ 8.02 – 7.93 (m, 2H), 7.65 – 7.33 (m, 11H), 7.28 (ddd, *J* = 8.6, 6.7, 3.0 Hz, 2H), 7.25 – 7.18 (m, 3H), 7.14 (dd, *J* = 7.5, 2.0 Hz, 2H), 5.05 (d, *J* = 7.5 Hz, 1H), 4.01 (d, *J* = 13.3 Hz, 1H), 3.62 (d, *J* = 13.3 Hz, 1H), 2.10 (s, 3H), 1.19 (s, 1H). ^{13}C NMR (101 MHz, MeOD) δ 171.6, 140.0, 139.3, 133.7, 133.3 (d, *J* = 6.3 Hz), 132.9 (d, *J* = 17.6 Hz), 132.8 (d, *J* = 9.2 Hz), 132.6 (d, *J* = 2.6 Hz), 132.2 (d, *J* = 8.8 Hz), 130.4, 129.5, 129.4 (d, *J* = 6.4 Hz), 129.1 (d, *J* = 17.8 Hz), 128.5 (d, *J* = 88.6 Hz), 120.3, 71.5 (d, *J* = 87.1 Hz), 63.4 (d, *J* = 9.6 Hz), 32.8, 27.2, 23.8 (d, *J* = 15.7 Hz), 14.4. HRMS (ESI) m/z Calculated for $C_{28}H_{27}N_2O_3P$[M-Na]$^+$ 493.1657, found 493.1664.

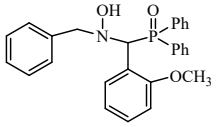


((benzyl(hydroxy)amino)(4-(dimethylamino)phenyl)methyl)diphenylphosphine oxide (3n): Following the **general procedure**, 5h, 91% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.77 (m, 2H), 7.56 – 7.43 (m, 5H), 7.33 – 7.28 (m, 3H), 7.25 – 7.18 (m, 5H), 7.09 (dd, *J* = 6.6, 2.9 Hz, 2H), 6.63 – 6.48 (m, 3H), 4.51 (d, *J* = 8.1 Hz, 1H), 4.06 (d, *J* = 13.1 Hz, 1H), 3.59 (d, *J* = 13.2 Hz, 1H), 2.90 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.3, 133.6, 132.7 (d, *J* = 6.9 Hz), 132.4 (d, *J* = 76.2 Hz), 131.6 (d, *J* = 8.8 Hz), 131.5 (d, *J* = 2.9 Hz), 131.4 (d, *J* = 3.0 Hz), 131.2 (d, *J* = 15.5 Hz), 129.4, 128.3 (d, *J* = 11.6 Hz), 128.2 (d, *J* = 8.4 Hz), 127.7 (d, *J* = 79.2 Hz), 118.2, 111.9, 68.6 (d, *J* = 93.1 Hz), 61.1 (d, *J* = 11.7 Hz), 53.5, 40.4. HRMS (ESI) m/z Calculated for $C_{28}H_{29}N_2O_2P$[M-Na]$^+$ 479.1864, found 479.1860.

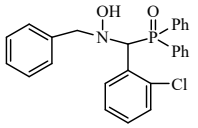


((benzyl(hydroxy)amino)(2-hydroxyphenyl)methyl)diphenylphosphine oxide (3o):

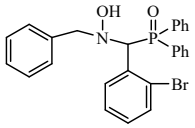
Following the **general procedure**, 4h, 89% yield, white solid, new compound, (hexane). ^{1}H NMR (400 MHz, DMSO) δ 9.81 (s, 1H), 8.07 (s, 1H), 7.98 – 7.88 (m, 2H), 7.82 – 7.74 (m, 1H), 7.63 – 7.51 (m, 5H), 7.37 – 7.26 (m, 3H), 7.22 – 7.15 (m, 3H), 7.11 – 7.01 (m, 3H), 6.78 – 6.67 (m, 2H), 5.59 (d, *J* = 8.2 Hz, 1H), 3.99 (d, *J* = 13.6 Hz, 1H), 3.63 (d, *J* = 13.8 Hz, 1H). ^{13}C NMR (101 MHz, DMSO) δ 165.8 (d, *J* = 7.0 Hz), 148.2, 143.6 (d, *J* = 98.3 Hz), 142.3 (d, *J* = 28.3 Hz), 140.8 (d, *J* = 6.5 Hz), 140.7 (d, *J* = 8.7 Hz), 139.9 (d, *J* = 8.5 Hz), 138.6 (d, *J* = 24.4 Hz), 138.0, 137.8 (d, *J* = 8.4 Hz), 137.5 (d, *J* = 22.3 Hz), 136.3, 128.2 (d, *J* = 85.8 Hz), 124.9, 78.4 (d, *J* = 84.0 Hz), 71.4 (d, *J* = 9.1 Hz). HRMS (ESI) m/z Calculated for $C_{26}H_{24}NO_3P$[M-Na]$^+$ 452.1391, found 452.1383.



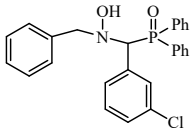
((benzyl(hydroxy)amino)(2-methoxyphenyl)methyl)diphenylphosphine oxide (3p): Following the **general procedure**, 4h, 85% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (d, *J* = 7.7 Hz, 1H), 7.93 – 7.86 (m, 2H), 7.57 – 7.39 (m, 5H), 7.30 – 7.10 (m, 9H), 6.92 (td, *J* = 7.5, 1.1 Hz, 1H), 6.65 (dt, *J* = 7.8, 1.1 Hz, 1H), 6.57 (s, 1H), 5.42 (d, *J* = 6.8 Hz, 1H), 3.95 (d, *J* = 13.5 Hz, 1H), 3.69 (d, *J* = 13.4 Hz, 1H), 3.60 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.1 (d, *J* = 6.8 Hz), 137.5, 133.3 (d, *J* = 99.4 Hz), 131.9, 131.5 (d, *J* = 1.9 Hz), 131.5 (d, *J* = 4.1 Hz), 131.3 (d, *J* = 2.7 Hz), 131.0 (d, *J* = 8.8 Hz), 129.4 (d, *J* = 2.0 Hz), 129.2, 128.4 (d, *J* = 11.7 Hz), 127.9 (d, *J* = 20.7 Hz), 127.4 (d, *J* = 83.2 Hz), 120.7 (d, *J* = 2.0 Hz), 120.5, 109.7, 68.6 (d, *J* = 88.3 Hz), 62.0 (d, *J* = 12.6 Hz), 55.1. HRMS (ESI) m/z Calculated for $C_{27}H_{26}NO_3P$[M-Na]$^+$ 466.1548, found 466.1553.



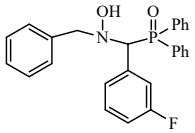
((benzyl(hydroxy)amino)(2-chlorophenyl)methyl)diphenylphosphine oxide (3q): Following the **general procedure**, 4h, 87% yield, white solid, new compound, (hexane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (dt, *J* = 7.8, 1.7 Hz, 1H), 8.01 (m, 2H), 7.64 – 7.60 (m, 1H), 7.60 – 7.54 (m, 3H), 7.53 (dt, *J* = 2.8, 1.6 Hz, 1H), 7.36 (tq, *J* = 6.8, 1.4 Hz, 1H), 7.32 – 7.24 (m, 7H), 7.23 – 7.17 (m, 3H), 6.62 (s, 1H), 5.53 (d, *J* = 6.5 Hz, 1H), 3.99 – 3.79 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) 137.3, 135.0 (d, *J* = 8.6 Hz), 133.7 (d, *J* = 3.7 Hz), 133.4, 132.7 (d, *J* = 2.9 Hz), 132.4, 131.8 (d, *J* = 2.9 Hz), 131.6 (d, *J* = 2.9 Hz), 131.4 (d, *J* = 8.9 Hz), 131.1 (d, *J* = 9.0 Hz), 130.8 (d, *J* = 91.4 Hz), 130.4 (d, *J* = 9.8 Hz), 129.6 (d, *J* = 1.9 Hz), 129.4, 129.1 (d, *J* = 6.1 Hz), 128.6 (d, *J* = 11.8 Hz), 128.2 (d, *J* = 2.3 Hz), 128.0, 127.3, 127.0 (d, *J* = 1.8 Hz), 65.6 (d, *J* = 89.6 Hz), 62.2 (d, *J* = 12.0 Hz).HRMS (ESI) m/z Calculated for $C_{26}H_{23}ClNO_2P$[M-Na]$^+$ 470.1053, found 470.1045.



((benzyl(hydroxy)amino)(2-bromophenyl)methyl)diphenylphosphine oxide (3r): Following the **general procedure**, 3h, 86% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 (d, *J* = 7.9 Hz, 1H), 8.05 (m, 2H), 7.66 – 7.51 (m, 6H), 7.45 – 7.42 (m, 1H), 7.36 – 7.31 (m, 2H), 7.26 (m, 6H), 7.11 (t, *J* = 7.7 Hz, 1H), 6.71 (s, 1H), 5.57 (d, *J* = 6.0 Hz, 1H), 3.97 – 3.87 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.5, 133.7 (d, *J* = 3.8 Hz), 133.5, 132.8, 132.7 (d, *J* = 2.8 Hz), 132.4 (d, *J* = 7.3 Hz), 131.8 (d, *J* = 2.9 Hz), 131.6 (d, *J* = 2.8 Hz), 131.4 (d, *J* = 8.8 Hz), 131.2 (d, *J* = 9.0 Hz), 130.8 (d, *J* = 91.4 Hz), 130.5, 129.9 (d, *J* = 2.0 Hz), 129.5 (d, *J* = 7.2 Hz), 129.1, 129.1 (d, *J* = 26.1 Hz), 128.6 (d, *J* = 11.8 Hz), 128.1, 128.0 (d, *J* = 11.7 Hz), 127.6 (d, *J* = 1.9 Hz), 127.2, 126.3 (d, *J* = 9.0 Hz), 68.7 (d, *J* = 88.6 Hz), 62.2 (d, *J* = 11.9 Hz). HRMS (ESI) m/z Calculated for $C_{26}H_{23}BrNO_2P$ $[M\text{-}Na]^+$ 514.0547, found 514.0551.

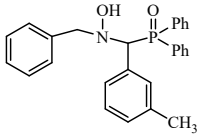


((benzyl(hydroxy)amino)(3-chlorophenyl)methyl)diphenylphosphine oxide (3s): Following the **general procedure**, 3h, 85% yield, white solid, new compound, (hexane/ethyl acetate = 8/2).^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 – 7.83 (m, 2H), 7.60 (td, *J* = 7.4, 1.7 Hz, 1H), 7.54 – 7.42 (m, 6H), 7.36 (td, *J* = 7.4, 1.6 Hz, 1H), 7.31 – 7.23 (m, 6H), 7.22 – 7.13 (m, 3H), 6.99 (s, 1H), 4.62 (d, *J* = 7.1 Hz, 1H), 4.13 (d, *J* = 13.2 Hz, 1H), 3.64 (d, *J* = 13.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.3, 133.6, 133.4, 131.5 (d, *J* = 2.9 Hz), 131.4 (d, *J* = 2.7 Hz), 131.3, 131.2 (d, *J* = 3.0 Hz), 131.0, 130.8 (d, *J* = 8.7 Hz), 130.1, 129.6 (d, *J* = 6.1 Hz), 129.0 (d, *J* = 8.2 Hz), 128.2 (d, *J* = 4.1 Hz), 128.1 (d, *J* = 5.9 Hz), 128.0, 127.9, 127.2, 68.4 (d, *J* = 88.5 Hz), 61.3 (d, *J* = 11.3 Hz). HRMS (ESI) m/z Calculated for $C_{26}H_{23}ClNO_2P$ $[M\text{-}Na]^+$ 470.1053, found 470.1043.

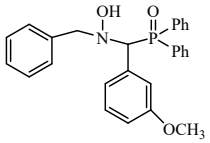


((benzyl(hydroxy)amino)(3-fluorophenyl)methyl)diphenylphosphine oxide (3t): Following the **general procedure**, 3h, 87% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 – 7.82 (m, 2H), 7.62 – 7.58 (m, 1H), 7.57 – 7.43 (m, 5H), 7.36 (td, *J* = 7.3, 1.5 Hz, 1H), 7.30 – 7.22 (m, 7H), 7.13 (dd, *J* = 7.2, 2.2 Hz, 2H), 6.98 (m, 1H), 6.77 (s, 1H), 4.63 (d, *J* = 7.4 Hz, 1H), 4.12 (d, *J* = 13.1 Hz, 1H), 3.65 (d, *J* = 13.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.4 (d, J_{CF} = 246.4 Hz), 136.6, 133.9 (d, *J* = 7.1 Hz), 132.9, 132.7 (d, *J* = 3.0 Hz), 131.9 (d, *J* = 2.8 Hz), 131.7 (d, *J* = 2.8 Hz), 131.6 (d, *J* = 8.8 Hz), 131.4, 131.2 (d, *J* = 8.7 Hz), 130.8 (d, J_{CF} = 8.4 Hz), 130.4, 129.6, 129.5 (d, *J* = 6.1 Hz), 129.0 (d, *J* =

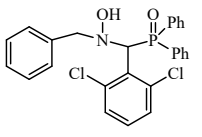
12.8 Hz), 128.6, 128.5 (d, J = 5.3 Hz), 128.3 (d, J = 11.7 Hz), 127.6 (d, J = 9.7 Hz), 118.8 (d, J = 6.4 Hz), 118.6 (d, J = 6.3 Hz), 115.5, 115.3, 68.6 (d, J = 88.4 Hz), 61.6 (d, J = 11.3 Hz). HRMS (ESI) m/z Calculated for $C_{26}H_{23}FNO_2P$ $[M\text{-}Na]^+$ 454.1348, found 454.1347.



((benzyl(hydroxy)amino)(m-tolyl)methyl)diphenylphosphine oxide (3u): Following the **general procedure**, 3h, 90% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (500 MHz, DMSO) δ 8.11 – 8.03 (m, 3H), 7.72 – 7.65 (m, 2H), 7.59 – 7.51 (m, 3H), 7.48 – 7.42 (m, 2H), 7.37 – 7.27 (m, 3H), 7.22 – 7.11 (m, 4H), 7.11 – 7.06 (m, 2H), 7.03 (d, J = 7.6 Hz, 1H), 5.30 (d, J = 9.4 Hz, 1H), 4.23 (d, J = 13.6 Hz, 1H), 3.47 (d, J = 13.6 Hz, 1H), 2.22 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.8, 137.0, 133.3 (d, J = 10.0 Hz), 132.7 (d, J = 2.9 Hz), 132.4 (d, J = 6.5 Hz), 132.2 (d, J = 88.1 Hz), 131.7 (d, J = 3.1 Hz), 131.6 (d, J = 8.8 Hz), 131.5 (d, J = 2.6 Hz), 131.3 (d, J = 8.7 Hz), 130.9, 130.8 (d, J = 3.3 Hz), 129.4 (d, J = 6.5 Hz), 129.2 (d, J = 2.0 Hz), 129.1, 129.0, 128.8 (d, J = 6.7 Hz), 128.5 (d, J = 6.3 Hz), 128.4 (d, J = 6.6 Hz), 128.1 (d, J = 11.5 Hz), 127.4, 69.5 (d, J = 89.1 Hz), 61.5 (d, J = 11.6 Hz), 21.50. HRMS (ESI) m/z Calculated for $C_{27}H_{26}NO_2P[M\text{-}Na]^+$ 450.1599, found 450.1602.

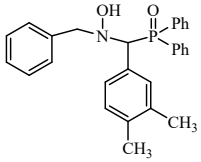


((benzyl(hydroxy)amino)(3-methoxyphenyl)methyl)diphenylphosphine oxide (3v): Following the **general procedure**, 5h, 92% yield, white solid, new compound, (hexane/ethyl acetate = 9/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.77 (m, 2H), 7.69 (m, 2H), 7.54 (dt, J = 3.8, 1.7 Hz, 1H), 7.51 (dd, J = 4.1, 2.2 Hz, 1H), 7.48 – 7.45 (m, 3H), 7.41 (dt, J = 3.7, 1.2 Hz, 1H), 7.39 (d, J = 1.6 Hz, 1H), 7.31 – 7.28 (m, 1H), 7.21 (dd, J = 5.2, 2.1 Hz, 3H), 7.12 – 7.09 (m, 2H), 7.05 – 7.00 (m, 1H), 6.98 – 6.94 (m, 1H), 6.79 – 6.74 (m, 1H), 4.59 (d, J = 6.8 Hz, 1H), 4.07 (d, J = 13.2 Hz, 1H), 3.81 (s, 1H), 3.70 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.3, 136.8, 132.7 (d, J = 2.8 Hz), 131.8 (d, J = 2.7 Hz), 131.6 (d, J = 8.7 Hz), 131.3 (d, J = 8.8 Hz), 130.8 (d, J = 11.6 Hz), 129.5, 129.4 (d, J = 1.9 Hz), 129.1 (d, J = 2.6 Hz), 129.0, 128.5, 128.4 (d, J = 8.4 Hz), 128.1 (d, J = 11.7 Hz), 127.5, 123.0 (d, J = 98.7 Hz), 117.7, 114.6, 112.3, 70.7 (d, J = 91.4 Hz), 61.6 (d, J = 11.7 Hz), 55.4 (d, J = 10.4 Hz). HRMS (ESI) m/z Calculated for $C_{27}H_{26}NO_3P[M\text{-}Na]^+$ 466.1548, found 466.1552.

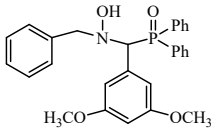


((benzyl(hydroxy)amino)(2,6-dichlorophenyl)methyl)diphenylphosphine oxide

(3w): Following the **general procedure**, 3h, 85% yield, white solid, new compound, (hexane/ethyl acetate = 9/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 – 8.07 (m, 2H), 7.72 – 7.64 (m, 2H), 7.57 (m, 3H), 7.46 – 7.40 (m, 2H), 7.36 – 7.22 (m, 7H), 7.13 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.03 (td, *J* = 8.0, 1.5 Hz, 1H), 6.70 (s, 1H), 6.09 (d, *J* = 4.2 Hz, 1H), 3.92 – 3.76 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.7, 137.1 (d, *J* = 4.7 Hz), 136.5 (d, *J* = 7.4 Hz), 135.9, 134.9, 131.8 (d, *J* = 3.5 Hz), 131.6 (d, *J* = 2.9 Hz), 131.4 (d, *J* = 2.8 Hz), 131.3 (d, *J* = 1.6 Hz), 131.2 (d, *J* = 92.8 Hz), 130.5 (d, *J* = 2.0 Hz), 129.7 (d, *J* = 2.3 Hz), 129.2, 128.7 (d, *J* = 11.8 Hz), 128.2 (d, *J* = 5.7 Hz), 127.6 (d, *J* = 11.9 Hz), 127.2, 71.1 (d, *J* = 84.5 Hz), 62.7 (d, *J* = 11.3 Hz). HRMS (ESI) m/z Calculated for $C_{26}H_{22}Cl_2NO_2P[M-H]^+$ 482.0843, found 482.0834.

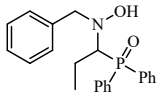


((benzyl(hydroxy)amino)(3,4-dimethylphenyl)methyl)diphenylphosphine oxide (3x): Following the **general procedure**, 4h, 92% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 – 7.82 (m, 2H), 7.75 (m, 1H), 7.61 – 7.58 (m, 1H), 7.58 – 7.55 (m, 1H), 7.51 (dq, *J* = 6.9, 4.0, 3.0 Hz, 4H), 7.44 (d, *J* = 6.9 Hz, 1H), 7.38 – 7.33 (m, 1H), 7.26 (dd, *J* = 5.1, 2.0 Hz, 4H), 7.20 (d, *J* = 8.0 Hz, 1H), 7.15 – 7.13 (m, 1H), 7.04 (d, *J* = 7.7 Hz, 1H), 6.66 (s, 1H), 4.60 (d, *J* = 7.5 Hz, 1H), 4.11 (d, *J* = 13.2 Hz, 1H), 3.64 (d, *J* = 13.2 Hz, 1H), 2.22 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.0, 136.7 (d, *J* = 2.0 Hz), 132.8 (d, *J* = 6.6 Hz), 132.6 (d, *J* = 2.9 Hz), 131.6 (d, *J* = 3.3 Hz), 131.5 (d, *J* = 2.8 Hz), 131.4 (d, *J* = 2.7 Hz), 131.3 (d, *J* = 8.8 Hz), 130.7 (d, *J* =91.4 Hz), 129.7 (d, *J* = 5.0 Hz), 129.3 (d, *J* = 2.8 Hz), 129.2 (d, *J* = 3.7 Hz), 129.1, 129.0 (d, *J* = 4.4 Hz), 128.9 (d, *J* = 2.9 Hz), 128.4, 128.2 (d, *J* = 4.9 Hz), 128.0 (d, *J* = 11.7 Hz), 127.3, 69.0 (d, *J* = 93.5 Hz), 61.2 (d, *J* = 11.5 Hz), 19.8, 19.5. HRMS (ESI) m/z Calculated for $C_{28}H_{28}NO_2P[M-Na]^+$ 464.1755, found 464.1741.

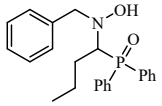


((benzyl(hydroxy)amino)(3,5-dimethoxyphenyl)methyl)diphenylphosphine oxide (3y): Following the **general procedure**, 5h, 93% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 – 7.77 (m, 2H), 7.60 – 7.39 (m, 7H), 7.37 – 7.31 (m, 1H), 7.23 (dt, *J* = 4.9, 2.4 Hz, 4H), 7.13 (dd, *J* = 6.6, 2.9 Hz, 2H), 6.63 – 6.56 (m, 2H), 6.33 (d, *J* = 1.2 Hz, 1H), 4.55 (d, *J* = 6.7 Hz, 1H), 4.09 (d, *J* = 13.2 Hz, 1H), 3.79 (s, 1H), 3.68 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.4, 136.9, 133.6, 133.1, 132.7 (d, *J* = 2.9 Hz), 132.1 (d, *J* = 4.6 Hz), 131.8 (d, *J* = 2.9 Hz), 131.6 (d, *J* = 3.7 Hz), 131.6, 131.4 (d, *J* = 98.8 Hz), 130.9, 130.8 (d, *J* = 4.8 Hz), 129.4 (d, *J* = 9.6 Hz), 129.1 (d, *J* = 3.8 Hz), 129.0, 128.5, 128.4 (d, *J* = 8.6 Hz), 128.2 (d, *J* = 11.7 Hz), 127.5, 109.6 (d, *J* = 6.4 Hz), 106.3, 104.0, 101.0, 69.8 (d, *J* =

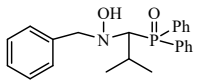
91.5 Hz), 61.6 (d, J = 11.6 Hz), 55.59, 55.47. HRMS (ESI) m/z Calculated for $C_{28}H_{28}NO_4P[M\text{-}Na]^+$ 496.1654, found 496.1650.



(1-(benzyl(hydroxy)amino)propyl)diphenylphosphine oxide (3z): Following the **general procedure**, 5h, 85% yield, white solid, new compound, (hexane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (m, 4H), 7.52 – 7.40 (m, 7H), 7.21 (dd, J = 5.0, 1.9 Hz, 3H), 7.09 – 7.03 (m, 2H), 4.25 (d, J = 13.2 Hz, 1H), 3.99 (d, J = 13.2 Hz, 1H), 3.64 (td, J = 7.0, 5.2 Hz, 1H), 2.23 (ddt, J = 14.9, 11.4, 7.4 Hz, 1H), 1.76 (ddt, J = 14.9, 11.4, 7.5 Hz, 1H), 0.92 (t, J = 7.5 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.5, 133.1, 132.7 (d, J = 3.0 Hz), 132.3 (d, J = 88.2 Hz), 131.9 (d, J = 2.9 Hz), 131.7 (d, J = 2.9 Hz), 131.5 (d, J = 25.9 Hz), 131.3 (d, J = 9.9 Hz), 130.8 (d, J = 11.4 Hz), 129.3, 129.0 (d, J = 4.9 Hz), 128.8 (d, J = 20.9 Hz), 128.6 (d, J = 11.1 Hz), 128.3, 127.4, 65.6 (d, J = 81.5 Hz), 61.6 (d, J = 9.7 Hz), 17.3 (d, J = 4.2 Hz), 13.9 (d, J = 9.0 Hz). HRMS (ESI) m/z Calculated for $C_{22}H_{24}NO_2P[M\text{-}Na]^+$ 388.1442, found 388.1439.

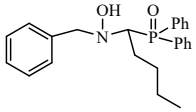


(1-(benzyl(hydroxy)amino)butyl)diphenylphosphine oxide (3aa): Following the **general procedure**, 5h, 88% yield, white solid, new compound, (hexane/ethyl acetate = 9/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 – 7.72 (m, 4H), 7.51 – 7.47 (m, 2H), 7.44 (m, 4H), 7.38 (s, 1H), 7.20 (dd, J = 5.0, 2.0 Hz, 2H), 7.05 (dd, J = 5.1, 2.0 Hz, 2H), 6.40 (s, 1H), 4.24 (d, J = 13.2 Hz, 1H), 3.97 (d, J = 13.2 Hz, 1H), 3.73 (td, J = 7.1, 5.1 Hz, 1H), 2.26 – 2.10 (m, 1H), 1.73 – 1.60 (m, 1H), 1.51 – 1.41 (m, 1H), 1.22 – 1.11 (m, 1H), 0.79 (t, J = 7.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.46, 133.01 (d, J = 5.9 Hz), 132.67 (d, J = 2.9 Hz), 132.44 (d, J = 12.1 Hz), 131.91 (d, J = 2.9 Hz), 131.70 (d, J = 2.9 Hz), 131.51 (d, J = 85.5 Hz), 131.31 (d, J = 6.2 Hz), 131.19, 130.79 (d, J = 11.5 Hz), 129.33 (d, J = 10.3 Hz), 128.99 (d, J = 2.5 Hz), 128.99 (d, J = 12.8 Hz), 128.60 (d, J = 2.3 Hz), 128.62 (d, J = 20.5 Hz), 128.29, 127.35, 63.83 (d, J = 81.8 Hz), 61.61 (d, J = 9.5 Hz), 25.89 (d, J = 3.7 Hz), 22.22 (d, J = 8.6 Hz), 14.13 (d, J = 12.8 Hz). HRMS (ESI) m/z Calculated for $C_{23}H_{26}NO_2P[M\text{-}Na]^+$ 402.1599, found 402.1597.

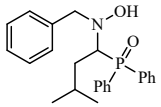


(1-(benzyl(hydroxy)amino)-2-methylpropyl)diphenylphosphine oxide (3ab): Following the **general procedure**, 5h, 86% yield, white solid, new compound, (hexane/ethyl acetate = 9/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (m, 4H), 7.62 – 7.34 (m, 7H), 7.19 (m, 3H), 7.04 – 6.93 (m, 2H), 6.73 (s, 1H), 4.24 (d, J = 13.2 Hz, 1H), 4.09 (d, J = 13.2 Hz, 1H), 3.63 (dd, J = 7.9, 4.6 Hz, 1H), 2.45 (dpd, J = 11.4, 6.9, 4.6 Hz, 1H), 1.06 (dd, J = 24.9, 6.9 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.5, 134.2 (d, J = 94.2 Hz), 132.7 (d, J = 31.2 Hz), 131.7 (d, J = 2.8 Hz), 131.5 (d, J = 2.9 Hz),

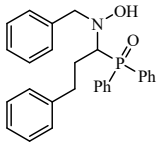
131.3 (d, *J* = 9.2 Hz), 131.0 (d, *J* = 8.9 Hz), 130.7 (d, *J* = 11.4 Hz), 129.1, 129.0 (d, *J* = 5.8 Hz), 128.6 (d, *J* = 4.7 Hz), 128.5 (d, *J* = 4.6 Hz), 127.7 (d, *J* = 95.5 Hz), 68.8 (d, *J* = 82.6 Hz), 62.6 (d, *J* = 7.2 Hz), 28.1 (d, *J* = 5.2 Hz), 22.2 (d, *J* = 7.4 Hz), 21.8 (d, *J* = 4.3 Hz). HRMS (ESI) m/z Calculated for $C_{23}H_{26}NO_2P$[M-Na]$^+$ 402.1599, found 402.1593.



(1-(benzyl(hydroxy)amino)pentyl)diphenylphosphine oxide (3ac): Following the **general procedure**, 5h, 91% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (m, 4H), 7.51 – 7.47 (m, 2H), 7.43 (m, 4H), 7.37 (s, 1H), 7.20 (m, 3H), 7.07 – 7.04 (m, 1H), 6.46 (s, 1H), 4.24 (d, *J* = 13.1 Hz, 1H), 3.96 (d, *J* = 13.2 Hz, 1H), 3.70 (q, *J* = 6.5 Hz, 1H), 2.20 (dtt, *J* = 13.2, 8.7, 5.7 Hz, 1H), 1.78 – 1.61 (m, 1H), 1.43 (ddt, *J* = 13.2, 8.7, 5.3 Hz, 1H), 1.24 – 1.05 (m, 3H), 0.75 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.5, 133.0, 132.7 (d, *J* = 2.9 Hz), 132.4 (d, *J* = 15.7 Hz), 132.1 (d, *J* = 20.4 Hz), 131.9 (d, *J* = 2.9 Hz), 131.7 (d, *J* = 2.7 Hz), 131.5 (d, *J* = 16.1 Hz), 131.3 (d, *J* = 6.0 Hz), 131.2, 130.8 (d, *J* = 11.4 Hz), 129.3 (d, *J* = 8.4 Hz), 129.0 (d, *J* = 12.8 Hz), 128.8 (d, *J* = 2.8 Hz), 128.6 (d, *J* = 2.7 Hz), 128.5 (d, *J* = 20.2 Hz), 127.8 (d, *J* = 95.7 Hz), 64.0 (d, *J* = 82.1 Hz), 61.5 (d, *J* = 9.6 Hz), 31.2 (d, *J* = 8.1 Hz), 23.3 (d, *J* = 3.8 Hz), 22.7 (d, *J* = 6.1 Hz), 13.8. HRMS (ESI) m/z Calculated for $C_{24}H_{28}NO_2P$[M-Na]$^+$ 416.1755, found 416.1750.

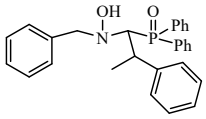


(benzyl(hydroxy)amino)-3-methylbutyl)diphenylphosphine oxide (3ad): Following the **general procedure**, 5h, 96% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 – 7.81 (m, 3H), 7.74 (m, 1H), 7.63 – 7.49 (m, 7H), 7.43 (s, 1H), 7.26 – 7.24 (m, 2H), 7.16 – 7.07 (m, 2H), 4.31 (d, *J* = 13.3 Hz, 1H), 4.05 (d, *J* = 13.3 Hz, 1H), 3.91 (td, *J* = 7.6, 4.9 Hz, 1H), 2.19 (dddd, *J* = 14.1, 10.8, 7.8, 4.9 Hz, 1H), 1.71 – 1.44 (m, 2H), 0.90 (d, *J* = 6.5 Hz, 3H), 0.77 (d, *J* = 6.5 Hz, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 137.6, 133.1 (d, *J* = 7.4 Hz), 132.7 (d, *J* = 2.9 Hz), 132.3 (d, *J* = 29.7 Hz), 131.9 (d, *J* = 2.9 Hz), 131.8 (d, *J* = 2.7 Hz), 131.5 (d, *J* = 11.5 Hz), 131.3 (d, *J* = 8.6 Hz), 130.8 (d, *J* = 11.5 Hz), 129.3 (d, *J* = 16.2 Hz), 129.0 (d, *J* = 4.5 Hz), 129.0 (d, *J* = 5.0 Hz), 128.7 (d, *J* = 3.4 Hz), 128.6 (d, *J* = 3.6 Hz), 127.8 (d, *J* = 96.7 Hz), 62.3 (d, *J* = 80.0 Hz), 61.6 (d, *J* = 8.8 Hz), 33.0 (d, *J* = 4.1 Hz), 26.2 (d, *J* = 8.5 Hz), 23.0, 21.9. HRMS (ESI) m/z Calculated for $C_{24}H_{28}NO_2P$[M-Na]$^+$ 416.1755, found 416.1753.

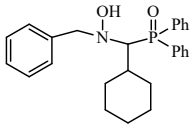


(1-(benzyl(hydroxy)amino)-3-phenylpropyl)diphenylphosphine oxide (3ae): Following the **general procedure**, 6h, 86% yield, white solid, new compound,

(hexane/ethyl acetate = 9/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (m, 4H), 7.55 – 7.49 (m, 2H), 7.47 – 7.38 (m, 4H), 7.26 – 7.16 (m, 6H), 7.04 – 6.91 (m, 4H), 6.32 (s, 1H), 4.23 (d, *J* = 13.1 Hz, 1H), 3.97 (d, *J* = 13.2 Hz, 1H), 3.73 (td, *J* = 6.6, 4.9 Hz, 1H), 2.69 – 2.41 (m, 3H), 2.10 – 1.93 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.3, 137.4, 132.8 (d, *J* = 90.3 Hz), 132.0 (d, *J* = 2.9 Hz), 131.8 (d, *J* = 20.7 Hz), 131.8 (d, *J* = 2.9 Hz), 131.4 (d, *J* = 5.6 Hz), 131.3 (d, *J* = 5.4 Hz), 129.4, 128.8 (d, *J* = 8.4 Hz), 128.7 (d, *J* = 1.8 Hz), 128.5 (d, *J* = 19.2 Hz), 126.8 (d, *J* = 124.6 Hz), 62.5 (d, *J* = 80.5 Hz), 61.8 (d, *J* = 9.2 Hz), 34.2 (d, *J* = 8.2 Hz), 26.0 (d, *J* = 5.0 Hz). HRMS (ESI) m/z Calculated for $C_{28}H_{28}NO_2P$[M-Na]$^+$ 464.1755, found 464.1758.

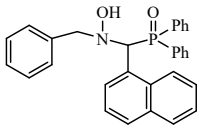


(benzyl(hydroxy)amino)-2-phenylpropyl)diphenylphosphine oxide (3af): Following the **general procedure**, 6h, 85% yield, white solid, new compound, (hexane/ethyl acetate = 9/1). ^{1}H NMR (400 MHz, DMSO) δ 8.15 (s, 1H), 7.98 – 7.90 (m, 2H), 7.71 (t, *J* = 9.0 Hz, 2H), 7.47 (dt, *J* = m, 4H), 7.40 – 7.30 (m, 3H), 7.20 (d, *J* = 5.9 Hz, 1H), 7.15 – 7.09 (m, 4H), 7.02 (t, *J* = 7.4 Hz, 2H), 6.89 – 6.78 (m, 2H), 4.42 (t, *J* = 6.7 Hz, 1H), 3.83 (q, *J* = 13.1 Hz, 2H), 3.42 (q, *J* = 6.9, 6.5 Hz, 1H), 1.55 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 144.9 (d, *J* = 7.5 Hz), 139.6, 135.9 (d, *J* = 74.2 Hz), 135.0 (d, *J* = 72.8 Hz), 131.8 (d, *J* = 9.4 Hz), 131.2 (d, *J* = 23.7 Hz), 130.9 (d, *J* = 8.7 Hz), 128.6 (d, *J* = 21.5 Hz), 128.6 (t, *J* = 4.9 Hz), 128.2 (d, *J* = 6.6 Hz), 128.2 (d, *J* = 27.4 Hz), 126.8 (d, *J* = 41.0 Hz), 126.1, 71.3 (d, *J* = 70.9 Hz), 62.25 (d, *J* = 82.7 Hz), 20.0 (d, *J* = 6.3 Hz). HRMS (ESI) m/z Calculated for $C_{28}H_{28}NO_2P$[M-Na]$^+$ 464.1755, found 464.1749.

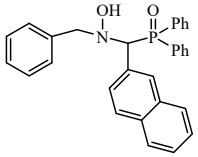


((benzyl(hydroxy)amino)(cyclohexyl)methyl)diphenylphosphine oxide (3ag): Following the **general procedure**, 3h, 97% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (m, 4H), 7.54 – 7.41 (m, 6H), 7.24 – 7.13 (m, 3H), 7.03 – 6.92 (m, 2H), 6.75 (s, 1H), 4.25 (d, *J* = 13.1 Hz, 1H), 4.09 (d, *J* = 13.2 Hz, 1H), 3.60 (dd, *J* = 8.1, 4.5 Hz, 1H), 2.16 – 2.01 (m, 1H), 1.94 – 1.88 (m, 1H), 1.73 (d, *J* = 12.5 Hz, 1H), 1.68 – 1.50 (m, 3H), 1.27 (qt, *J* = 12.3, 3.3 Hz, 2H), 1.05 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.2, 137.5, 134.8, 133.8(d, *J* = 9.4 Hz), 133.0 (d, *J* = 33.3 Hz), 132.6 (d, *J* = 2.9 Hz), 132.0, 131.7 (d, *J* = 2.9 Hz), 131.4 (d, *J* = 2.7 Hz), 131.2 (d, *J* = 9.2 Hz), 131.0 (d, *J* = 9.0 Hz), 130.7 (d, *J* = 11.5 Hz), 129.0 (d, *J* = 6.3 Hz), 128.9 (d, *J* = 3.8 Hz), 128.7 (d, *J* = 18.9 Hz), 128.5 (d, *J* = 3.5 Hz), 128.4, 127.7 (d, *J* = 97.2 Hz), 68.6 (d, *J* = 72.6 Hz), 62.5 (d, *J* = 7.5 Hz), 38.2 (d, *J* = 5.0 Hz), 35.0, 32.6 (d, *J* = 7.2 Hz), 32.0 (d, *J* = 4.1 Hz), 28.8, 26.8 (d, *J* = 12.5 Hz), 26.0 (d, *J* = 13.2 Hz), 25.2. HRMS (ESI) m/z Calculated for

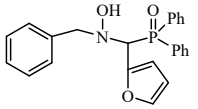
$C_{26}H_{30}NO_2P$ [M-Na]$^+$ 442.1912, found 442.1911.



((benzyl(hydroxy)amino)(naphthalen-1-yl)methyl)diphenylphosphine oxide (3ah): Following the **general procedure**, 4h, 89% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 (s, 1H), 8.04 – 7.85 (m, 2H), 7.69 (t, *J* = 9.1 Hz, 3H), 7.57 – 7.32 (m, 6H), 7.26 – 6.96 (m, 8H), 6.89 (s, 2H), 6.63 (s, 1H), 5.65 (s, 1H), 3.99 (d, *J* = 13.8 Hz, 1H), 3.59 (d, *J* = 13.6 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.4, 133.1 (d, *J* = 112.7 Hz), 131.6 (d, *J* = 9.1 Hz), 131.0 (d, *J* = 25.4 Hz), 130.9 (d, *J* = 8.6 Hz), 129.3, 128.6 (d, *J* = 11.7 Hz), 128.1, 126.7 (d, *J* = 109.1 Hz), 125.4, 122.7, 63.9 (d, *J* = 83.9 Hz), 62.0 (d, *J* = 8.2 Hz). HRMS (ESI) m/z Calculated for $C_{30}H_{26}NO_2P$ [M-Na]$^+$ 486.1599, found 486.1592.

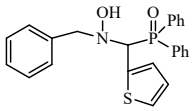


((benzyl(hydroxy)amino)(naphthalen-2-yl)methyl)diphenylphosphine oxide (3ai): Following the **general procedure**, 4h, 95% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 – 7.85 (m, 3H), 7.80 – 7.76 (m, 2H), 7.74 – 7.67 (m, 2H), 7.63 (dd, *J* = 8.5, 1.6 Hz, 1H), 7.56 (d, *J* = 7.4 Hz, 1H), 7.52 – 7.43 (m, 8H), 7.25 – 7.23 (m, 2H), 7.12 (m, 3H), 6.77 (s, 1H), 4.80 (d, *J* = 7.4 Hz, 1H), 4.09 (d, *J* = 13.2 Hz, 1H), 3.63 (d, *J* = 13.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.9, 134.4 (d, *J* = 24.0 Hz), 133.3 (d, *J* = 11.1 Hz), 133.0 (d, *J* = 14.1 Hz), 132.7 (d, *J* = 2.7 Hz), 131.8 (d, *J* = 2.8 Hz), 131.6 (d, *J* = 9.1 Hz), 131.4 (d, *J* = 7.8 Hz), 131.3 (d, *J* = 8.8 Hz), 130.9, 130.7 (d, *J* = 21.0 Hz), 129.4, 129.1 (d, *J* = 4.6 Hz), 128.9 (d, *J* = 19.9 Hz), 128.6, 128.4 (d, *J* = 7.2 Hz), 128.3 (d, *J* = 2.9 Hz), 128.1, 127.8 (d, *J* = 28.0 Hz), 127.7 (d, *J* = 4.4 Hz), 127.5 (d, *J* = 3.1 Hz), 126.3 (d, *J* = 92.8 Hz), 126.2 (d, *J* = 28.6 Hz), 69.4 (d, *J* = 87.8 Hz), 61.5 (d, *J* = 9.6 Hz). HRMS (ESI) m/z Calculated for $C_{30}H_{26}NO_2P$ [M-Na]$^+$ 486.1599, found 486.1594.

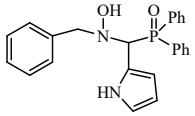


((benzyl(hydroxy)amino)(furan-2-yl)methyl)diphenylphosphine oxide (3aJ): Following the **general procedure**, 3h, 94% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 – 7.60 (m, 2H), 7.50 – 7.30 (m, 7H), 7.27 – 7.14 (m, 6H), 7.10 – 7.01 (m, 2H), 6.61 (t, *J* = 2.6 Hz, 1H), 6.25 (dd, *J* = 3.3, 1.9 Hz, 1H), 4.73 (d, *J* = 11.1 Hz, 1H), 4.07 (d, *J* = 12.6 Hz, 1H), 3.58 (d, *J* = 12.7 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.8 (d, *J* = 4.7 Hz), 142.5 (d, *J* = 2.1 Hz), 136.2, 131.7, 131.6 (d, *J* = 2.8 Hz), 131.3 (d, *J* = 9.1 Hz), 130.7 (d, *J* = 8.9 Hz), 130.7 (d, *J* = 26.6 Hz), 113.5 (d, *J* = 3.9 Hz), 110.8 (d, *J* = 1.3 Hz), 62.10(d,

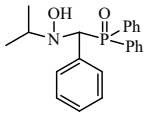
J = 64.8 Hz), 61.50 (d, J = 10.2 Hz). HRMS (ESI) m/z Calculated for $C_{24}H_{22}NO_3P$ $[M\text{-}Na]^+$ 426.1235, found 426.1233.



((benzyl(hydroxy)amino)(thiophen-2-yl)methyl)diphenylphosphine oxide (3ak): Following the **general procedure**, 4h, 87% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (500 MHz, DMSO) δ 8.21 (s, 1H), 8.04 (m, 2H), 7.74 (m, 2H), 7.60 – 7.53 (m, 3H), 7.44 (dd, J = 5.2, 1.1 Hz, 1H), 7.42 – 7.38 (m, 1H), 7.35 (td, J = 7.5, 1.9 Hz, 2H), 7.25 – 7.22 (m, 1H), 7.22 – 7.16 (m, 3H), 7.06 (dd, J = 7.6, 1.8 Hz, 2H), 6.94 (dd, J = 5.1, 3.5 Hz, 1H), 5.70 (d, J = 11.6 Hz, 1H), 4.23 (d, J = 13.5 Hz, 1H), 3.50 (d, J = 13.5 Hz, 1H). ^{13}C NMR (101 MHz, DMSO) δ 139.1, 133.8, 133.4, 132.8, 132.4, 132.0 (d, J = 6.0 Hz), 131.9, 131.9 (d, J = 2.5 Hz), 131.7 (d, J = 8.8 Hz), 131.0 (d, J = 8.7 Hz), 130.8 (d, J = 6.3 Hz), 129.3, 128.8 (d, J = 21.1 Hz), 128.8 (d, J = 1.6 Hz), 128.3, 127.9, 127.2, 126.6, 65.4 (d, J = 85.8 Hz), 61.3 (d, J = 5.0 Hz). HRMS (ESI) m/z Calculated for $C_{24}H_{22}NO_2PS$ $[M\text{-}Na]^+$ 442.1007, found 442.1006.

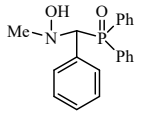


((benzyl(hydroxy)amino)(1H-pyrrol-2-yl)methyl)diphenylphosphine oxide (3al): Following the **general procedure**, 4h, 87% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.74 (s, 1H), 7.77 – 7.67 (m, 2H), 7.58 (td, J = 7.3, 1.4 Hz, 1H), 7.47 (m, 2H), 7.36 (td, J = 7.2, 1.5 Hz, 1H), 7.31 – 7.28 (m, 1H), 7.27 – 7.23 (m, 5H), 7.11 (dd, J = 6.6, 2.8 Hz, 2H), 6.83 (tt, J = 2.7, 1.3 Hz, 1H), 6.63 (s, 1H), 6.07 (q, J = 2.9 Hz, 1H), 5.91 (d, J = 1.9 Hz, 1H), 4.58 (d, J = 9.8 Hz, 1H), 4.04 (d, J = 12.6 Hz, 1H), 3.69 (d, J = 12.6 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.6, 131.8 (d, J = 2.7 Hz), 131.7 (d, J = 4.0 Hz), 131.6 (d, J = 4.4 Hz), 130.9 (d, J = 9.0 Hz), 129.6, 128.4 (d, J = 13.4 Hz), 128.1 (d, J = 11.6 Hz), 127.5, 120.1 (d, J = 84.2 Hz), 112.4 (d, J = 8.1 Hz), 107.4, 61.6 (d, J = 92.1 Hz), 61.4 (d, J = 11.9 Hz). HRMS (ESI) m/z Calculated for $C_{24}H_{23}N_2O_2P[M\text{-}Na]^+$ 425.1395, found 425.1386.

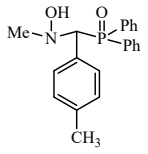


((hydroxy(isopropyl)amino)(phenyl)methyl)diphenylphosphine oxide (3am): Following the **general procedure**, 6h, 87% yield, white solid, new compound, (hexane/ethyl acetate = 6/4). ^{1}H NMR (400 MHz, MeOD) δ 8.03 (m, 2H), 7.77 (m, 1H), 7.58 – 7.54 (m, 2H), 7.49 – 7.29 (m, 7H), 7.23 (m, 2H), 7.17 – 7.13 (m, 2H), 5.15 (d, J = 3.3 Hz, 1H), 2.80 (hept, J = 6.4 Hz, 1H), 1.03 (dd, J = 6.4, 3.3 Hz, 6H). ^{13}C NMR (101 MHz, MeOD) δ 141.3, 140.0, 134.8, 134.7 (d, J = 4.1 Hz), 134.3, 133.8, 133.3, 132.6 (d, J = 4.6 Hz), 132.3, 132.3 (d, J = 3.1 Hz), 132.2 (d, J = 3.0 Hz), 132.0 (d, J = 5.8 Hz), 131.0 (d, J = 2.7 Hz), 130.7, 130.1 (d, J = 3.3 Hz), 129.3 (d, J = 11.9 Hz), 129.0 (d, J = 8.8 Hz), 128.9, 128.8 (d, J = 12.1 Hz), 70.2 (d, J = 89.2 Hz),

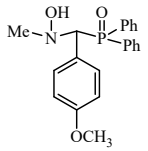
55.2 (d, J = 10.9 Hz), 45.1, 21.0 (d, J = 28.2 Hz), 15.21. HRMS (ESI) m/z Calculated for $C_{22}H_{24}NO_2P[M\text{-}Na]^+$ 388.1442, found 388.1450.



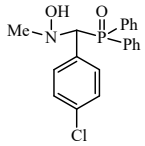
((hydroxy(methyl)amino)(phenyl)methyl)diphenylphosphine oxide (3an): Following the **general procedure**, 5h, 88% yield, white solid, new compound, (hexane/ethyl acetate = 6/4). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 – 7.86 (m, 2H), 7.54 – 7.41 (m, 5H), 7.31 (m, 3H), 7.25 – 7.05 (m, 6H), 4.61 (d, J = 5.3 Hz, 1H), 2.59 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 133.4, 132.4, 132.1, 131.8 (d, J = 2.9 Hz), 131.7 (d, J = 6.4 Hz), 131.5 (d, J = 3.4 Hz), 131.5 (d, J = 2.0 Hz), 131.2 (d, J = 6.3 Hz), 130.7, 128.6 (d, J = 11.6 Hz), 128.3 (d, J = 1.9 Hz), 128.2 (d, J = 4.5 Hz), 128.0, 74.0 (d, J = 86.8 Hz), 46.7 (d, J = 11.0 Hz). HRMS (ESI) m/z Calculated for $C_{20}H_{20}NO_2P[M\text{-}Na]^+$ 360.1129, found 360.1126.



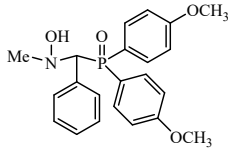
((hydroxy(methyl)amino)(p-tolyl)methyl)diphenylphosphine oxide (3ao): Following the **general procedure**, 5h, 87% yield, white solid, new compound, (hexane/ethyl acetate = 6/4). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (ddd, J = 11.1, 8.2, 1.5 Hz, 2H), 7.55 – 7.45 (m, 5H), 7.37 – 7.32 (m, 1H), 7.25 (d, J = 5.7 Hz, 2H), 7.21 – 7.16 (m, 2H), 6.98 (d, J = 7.8 Hz, 2H), 6.76 (s, 1H), 4.53 (d, J = 5.9 Hz, 1H), 2.55 (s, 3H), 2.25 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.2, 132.4, 131.9 (d, J = 2.8 Hz), 131.7 (d, J = 2.5 Hz), 131.6 (d, J = 1.9 Hz), 131.2 (d, J = 6.6 Hz), 128.8 (d, J = 27.8 Hz), 128.5, 128.1 (d, J = 11.6 Hz), 70.7 (d, J = 83.1 Hz), 46.5 (d, J = 11.4 Hz), 21.3. HRMS (ESI) m/z Calculated for $C_{21}H_{22}NO_2P[M\text{-}Na]^+$ 374.1286, found 374.1282.



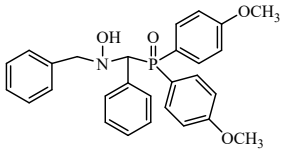
((hydroxy(methyl)amino)(4-methoxyphenyl)methyl)diphenylphosphine oxide (3ap): Following the **general procedure**, 6h, 92% yield, white solid, new compound, (hexane/ethyl acetate = 6/4). ^{1}H NMR (400 MHz, MeOD) δ 8.02 (m, 2H), 7.67 – 7.47 (m, 6H), 7.41 – 7.34 (m, 3H), 7.33 – 7.26 (m, 2H), 6.76 (d, J = 8.7 Hz, 2H), 4.92 (d, J = 5.8 Hz, 1H), 3.74 (s, 3H), 2.56 (s, 3H). ^{13}C NMR (101 MHz, MeOD) δ 138.2 (d, J = 1.7 Hz), 133.6 (d, J = 8.7 Hz), 133.4 (d, J = 2.7 Hz), 133.3 (d, J = 2.8 Hz), 132.9 (d, J = 15.5 Hz), 132.4 (d, J = 92.3 Hz), 130.4, 129.6 (d, J = 11.5 Hz), 129.5, 129.3 (d, J = 11.7 Hz), 129.0 (d, J = 2.9 Hz), 128.8, 128.8 (d, J = 2.0 Hz), 74.1 (d, J = 88.3 Hz). HRMS (ESI) m/z Calculated for $C_{21}H_{22}NO_3P[M\text{-}Na]^+$ 390.1235, found 390.1239.



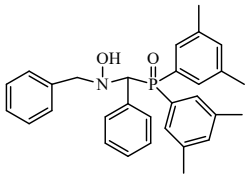
((4-chlorophenyl)(hydroxy(methyl)amino)methyl)diphenylphosphine oxide (3aq): Following the **general procedure**, 6h, 85% yield, white solid, new compound, (hexane/ethyl acetate = 6/4). ^{1}H NMR (400 MHz, DMSO) δ 8.27 (s, 1H), 8.01 (m, 2H), 7.63 (ddd, *J* = 10.8, 8.2, 1.6 Hz, 2H), 7.58 – 7.49 (m, 5H), 7.38 – 7.22 (m, 5H), 5.22 (d, *J* = 7.9 Hz, 1H), 2.44 (s, 3H). ^{13}C NMR (101 MHz, DMSO) δ 134.6, 133.9, 133.8 (d, *J* = 3.0 Hz), 133.7, 132.9 (d, *J* = 1.4 Hz), 132.9, 131.7 (d, *J* = 2.5 Hz), 131.6 (d, *J* = 2.7 Hz), 131.4 (d, *J* = 8.9 Hz), 131.0 (d, *J* = 8.5 Hz), 128.9, 128.7 (d, *J* = 6.6 Hz), 128.2 (d, *J* = 67.4 Hz), 69.8 (d, *J* = 85.0 Hz), 46.6 (d, *J* = 7.3 Hz). HRMS (ESI) m/z Calculated for $C_{20}H_{19}ClNO_2P$[M-Na]$^+$ 394.0740, found 394.0738.



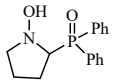
((hydroxy(methyl)amino)(phenyl)methyl)bis(4-methoxyphenyl)phosphine oxide (3ar): Following the **general procedure**, 6h, 85% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, MeOD) δ 7.88 (m, 2H), 7.45 – 7.35 (m, 4H), 7.19 (dd, *J* =m 3H), 7.06 (dd, *J* = 8.9, 2.4 Hz, 2H), 6.79 (dd, *J* = 8.9, 2.3 Hz, 2H), 4.81 (d, *J* = 6.8 Hz, 1H), 3.84 (s, 3H), 3.71 (s, 3H), 2.56 (s, 3H). ^{13}C NMR (101 MHz, MeOD) δ 161.1, 133.7 (d, *J* = 6.6 Hz), 132.7 (d, *J* = 3.2 Hz), 132.6, 132.5 (d, *J* = 1.4 Hz), 132.2 (d, *J* = 8.6 Hz), 130.0, 130.0 (d, *J* = 11.7 Hz), 129.4 (d, *J* = 11.7 Hz), 129.2 (d, *J* = 11.8 Hz), 125.4, 114.4, 73.4 (d, *J* = 86.9 Hz), 55.6, 47.2 (d, *J* = 9.6 Hz). HRMS (ESI) m/z Calculated for $C_{22}H_{24}NO_4P$[M-Na]$^+$ 420.1341, found 420.1340.



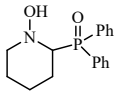
((benzyl(hydroxy)amino)(phenyl)methyl)bis(4-methoxyphenyl)phosphine oxide (3as): Following the **general procedure**, 5h, 89% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, MeOD) δ 7.87 (dd, *J* = 10.8, 8.8 Hz, 2H), 7.53 – 7.46 (m, 2H), 7.38 (dd, *J* = 10.7, 8.8 Hz, 2H), 7.30 – 7.13 (m, 8H), 7.09 (dd, *J* = 8.9, 2.4 Hz, 2H), 6.81 (dd, *J* = 9.0, 2.3 Hz, 2H), 4.95 (d, *J* = 8.8 Hz, 1H), 4.02 (d, *J* = 13.3 Hz, 1H), 3.88 (s, 3H), 3.74 (s, 3H), 3.62 (d, *J* = 13.3 Hz, 1H). ^{13}C NMR (101 MHz, MeOD) δ 164.0 (d, *J* = 2.9 Hz), 163.7 (d, *J* = 2.9 Hz), 139.4, 134.8 (d, *J* = 10.2 Hz), 134.2 (d, *J* = 10.2 Hz), 133.7, 132.9 (d, *J* = 6.2 Hz), 130.4, 129.2 (d, *J* = 1.5 Hz), 129.0 (d, *J* = 11.0 Hz), 128.1, 124.8 (d, *J* = 7.7 Hz), 123.8 (d, *J* = 6.5 Hz), 115.0 (d, *J* = 12.9 Hz), 114.7 (d, *J* = 12.7 Hz), 72.5 (d, *J* = 87.2 Hz), 63.4 (d, *J* = 9.2 Hz), 55.9 (d, *J* = 17.3 Hz). HRMS (ESI) m/z Calculated for $C_{28}H_{28}NO_4P$[M-Na]$^+$ 496.1654, found 496.1645.



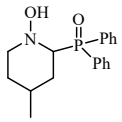
((benzyl(hydroxy)amino)(phenyl)methyl)bis(3,5-dimethylphenyl)phosphine oxide (3at): Following the **general procedure**, 4h, 86% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.23 (m, 5H), 7.13 – 7.08 (m, 6H), 7.05 – 6.96 (m, 3H), 6.84 (m, 2H), 6.76 (s, 1H), 4.39 (d, *J* = 8.0 Hz, 1H), 3.92 (d, *J* = 13.1 Hz, 1H), 3.47 (d, *J* = 13.2 Hz, 1H), 2.22 (s, 6H), 1.99 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.9, 138.7, 138.1 (d, *J* = 12.4 Hz), 137.7 (d, *J* = 12.2 Hz), 137.0, 134.4 (d, *J* = 2.8 Hz), 133.5 (d, *J* = 2.8 Hz), 133.3, 132.8, 132.0 (d, *J* = 6.4 Hz), 131.7 (d, *J* = 27.3 Hz), 130.7 (d, *J* = 97.1 Hz), 129.4 (d, *J* = 6.7 Hz), 129.2, 129.1 (d, *J* = 3.4 Hz), 128.9 (d, *J* = 8.7 Hz), 128.7 (d, *J* = 16.5 Hz), 128.4, 128.3 (d, *J* = 4.5 Hz), 127.7 (d, *J* = 98.7 Hz), 68.8 (d, *J* = 87.6 Hz), 61.3 (d, *J* = 11.8 Hz), 21.4 (d, *J* = 29.9 Hz). HRMS (ESI) m/z Calculated for $C_{30}H_{32}NO_2P$[M-Na]$^+$ 492.2068, found 492.2066.



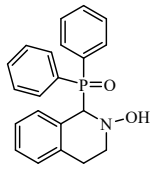
hydroxypyrrolidin-2-yl)diphenylphosphine oxide (5a): Following the **general procedure**, 6h, 91% yield, white solid, new compound, (hexane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 – 7.86 (m, 2H), 7.85 – 7.74 (m, 2H), 7.47 (m, 6H), 5.94 (s, 1H), 3.99 (dd, *J* = 10.2, 6.9 Hz, 1H), 3.11 (dt, *J* = 10.9, 6.5 Hz, 1H), 2.98 (dt, *J* = 11.0, 6.6 Hz, 1H), 2.24 – 2.11 (m, 1H), 1.99 – 1.81 (m, 2H), 1.72 – 1.60 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 132.6, 132.0, 132.0 (d, *J* = 3.8 Hz), 131.9 (d, *J* = 6.6 Hz), 131.6 (d, *J* = 8.9 Hz), 128.7, 128.6 (d, *J* = 1.8 Hz), 128.5, 68.2 (d, *J* = 91.3 Hz), 60.2 (d, *J* = 10.0 Hz), 24.9 (d, *J* = 1.8 Hz), 22.7 (d, *J* = 4.4 Hz). HRMS (ESI) m/z Calculated for $C_{16}H_{18}NO_2P$[M-Na]$^+$ 310.0973, found 310.0981.



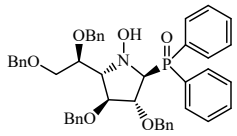
hydroxypiperidin-2-yl)diphenylphosphine oxide(5b): Following the **general procedure**, 6h, 88% yield, white solid, new compound, (hexane). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.91 – 7.85 (m, 2H), 7.78 – 7.72 (m, 2H), 7.46 (m, 6H), 7.05 (s, 1H), 3.54 (d, *J* = 13.8 Hz, 1H), 3.43 – 3.36 (m, 1H), 2.60 (s, 1H), 1.73 – 1.47 (m, 5H), 1.21 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 132.0 (d, *J* = 7.4 Hz), 131.8, 131.7 (d, *J* = 1.9 Hz), 131.7 (d, *J* = 1.7 Hz), 131.4 (d, *J* = 8.7 Hz), 128.5 (d, *J* = 4.9 Hz), 128.4 (d, *J* = 4.9 Hz), 67.0 (d, *J* = 102.2 Hz), 59.4 (d, *J* = 8.8 Hz), 26.4, 24.9, 23.6 (d, *J* = 12.6 Hz). HRMS (ESI) m/z Calculated for $C_{17}H_{20}NO_2P$[M-Na]$^+$ 324.1129, found 324.1128.



hydroxy-4-methylpiperidin-2-yl)diphenylphosphine oxide (5c): Following the **general procedure**, 6h, 85% yield, white solid, new compound, (hexane). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 – 7.83 (m, 2H), 7.79 – 7.70 (m, 2H), 7.44 (m, 6H), 7.07 (s, 1H), 3.94 (dt, *J* = 11.7, 5.9 Hz, 1H), 3.33 (ddd, *J* = 12.1, 7.0, 3.9 Hz, 1H), 2.94 (ddd, *J* = 12.2, 8.7, 3.8 Hz, 1H), 2.07 – 1.96 (m, 1H), 1.78 – 1.63 (m, 2H), 1.57 – 1.39 (m, 2H), 0.90 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 131.6 (d, *J* = 2.7 Hz), 131.6 (d, *J* = 2.8 Hz), 131.4, 131.3 (d, *J* = 9.1 Hz), 128.6, 128.4 (d, *J* = 11.3 Hz), 61.9 (d, *J* = 83.9 Hz), 54.0 (d, *J* = 7.0 Hz), 30.4, 29.0, 25.3 (d, *J* = 7.3 Hz), 19.3. HRMS (ESI) m/z Calculated for $C_{18}H_{22}NO_2P[M\text{-}Na]^+$ 338.1286, found 338.1280.

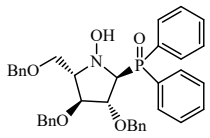


hydroxy-1,2,3,4-tetrahydroisoquinolin-1-yl)diphenylphosphine oxide (5d): Following the **general procedure**, 6h, 85% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 – 7.78 (m, 2H), 7.75 – 7.67 (m, 1H), 7.61 – 7.55 (m, 2H), 7.54 – 7.45 (m, 3H), 7.41 (m, 3H), 7.15 – 7.05 (m, 2H), 7.05 – 6.93 (m, 2H), 5.01 (d, *J* = 9.9 Hz, 1H), 3.10 (dt, *J* = 11.5, 5.5 Hz, 1H), 2.87 (ddd, *J* = 11.6, 7.9, 5.7 Hz, 1H), 2.71 (q, *J* = 7.9, 5.8 Hz, 1H), 2.66 – 2.57 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.0, 132.6, 130.7 (d, *J* = 11.5 Hz), 129.3 (d, *J* = 2.3 Hz), 128.9 (d, *J* = 12.9 Hz), 128.5, 128.4 (d, *J* = 4.8 Hz), 128.3, 127.7 (d, *J* = 3.8 Hz), 126.8 (d, *J* = 2.9 Hz), 125.6 (d, *J* = 2.9 Hz), 58.2 (d, *J* = 74.0 Hz), 41.2 (d, *J* = 7.1 Hz), 29.3. HRMS (ESI) m/z Calculated for $C_{21}H_{20}NO_2P[M\text{-}Na]^+$ 372.1129, found 372.1128.

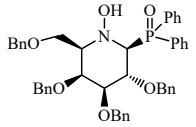


((2S,3R,4S,5S)-3,4-bis(benzyloxy)-5-((S)-1,2-bis(benzyloxy)ethyl)-1-hydroxypyrrolidin-2-yl)diphenylphosphine oxide (5e): Following the **general procedure**, 6h, 85% yield, white solid, new compound, (ethyl acetate/petroleum ether=1/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 – 7.78 (m, 4H), 7.51 (dd, *J* = 7.5, 2.1 Hz, 1H), 7.43 (m, 3H), 7.36 (dd, *J* = 7.6, 2.2 Hz, 2H), 7.26 (m, 17H), 7.18 (dd, *J* = 7.6, 1.9 Hz, 2H), 7.12 (dd, *J* = 7.6, 2.1 Hz, 2H), 6.07 (s, 1H), 4.69 (d, *J* = 11.6 Hz, 1H), 4.57 (dt, *J* = 11.5, 5.3 Hz, 1H), 4.47 – 4.41 (m, 5H), 4.40 – 4.31 (m, 2H), 4.22 (d, *J* = 11.6 Hz, 1H), 3.81 (q, *J* = 4.8 Hz, 1H), 3.72 – 3.57 (m, 2H), 3.18 (dd, *J* = 8.0, 4.7 Hz, 1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 138.5, 138.2 (d, *J* = 5.1 Hz), 138.0, 132.0 (d, *J* = 4.2 Hz), 131.9 (d, *J* = 5.9 Hz), 131.7, 128.7, 128.6 (d, *J* = 10.1 Hz), 128.4 (d, *J* = 2.1 Hz), 128.3, 128.0 (d, *J* = 4.5 Hz), 127.8, 127.8 (d, *J* = 2.4 Hz), 127.7 (d, *J* = 12.0 Hz),

73.2 (d, *J* = 73.0 Hz), 72.5 (d, *J* = 9.6 Hz), 72.0 (d, *J* = 9.0 Hz), 71.1 (d, *J* = 7.7 Hz). HRMS (ESI) m/z Calculated for $C_{46}H_{46}NO_6P[M\text{-}Na]^+$ 762.2960, found 762.2965.



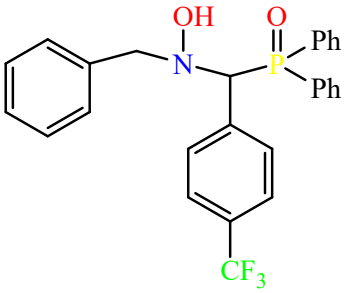
((2S,3R,4S,5S)-3,4-bis(benzyloxy)-5-((benzyloxy)methyl)-1-hydroxypyrrolidin-2-yl)diphenylphosphine oxide (5f): Following the **general procedure**, 6h, 86% yield, white solid, new compound, (hexane/ethyl acetate = 8/2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 – 7.78 (m, 4H), 7.55 – 7.46 (m, 2H), 7.46 – 7.36 (m, 4H), 7.30 – 7.23 (m, 13H), 7.20 (dd, *J* = 7.4, 2.1 Hz, 2H), 7.08 (dd, *J* = 7.6, 2.1 Hz, 2H), 4.53 – 4.43 (m, 3H), 4.43 – 4.32 (m, 3H), 4.29 (d, *J* = 11.5 Hz, 1H), 4.24 (dd, *J* = 7.1, 3.9 Hz, 1H), 4.12 (d, *J* = 11.4 Hz, 1H), 3.80 (dd, *J* = 10.4, 5.0 Hz, 1H), 3.66 (dd, *J* = 10.4, 5.1 Hz, 1H), 3.54 (q, *J* = 6.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.2 (d, *J* = 11.3 Hz), 137.8, 132.3 (d, *J* = 12.9 Hz), 132.1, 132.0 (d, *J* = 3.4 Hz), 131.9 (d, *J* = 2.5 Hz), 131.8 (d, *J* = 2.3 Hz), 128.7 (d, *J* = 11.3 Hz), 128.5 (d, *J* = 6.0 Hz), 128.4 (d, *J* = 8.5 Hz), 129.0, 127.8 (d, *J* = 4.3 Hz), 127.7 (d, *J* = 1.9 Hz), 84.2 (d, *J* = 4.3 Hz), 83.9 (d, *J* = 5.7 Hz), 72.7 (d, *J* = 71.2 Hz), 71.8 (d, *J* = 10.6 Hz), 66.9. HRMS (ESI) m/z Calculated for $C_{38}H_{38}NO_5P[M\text{-}Na]^+$ 642.2385, found 642.2390.



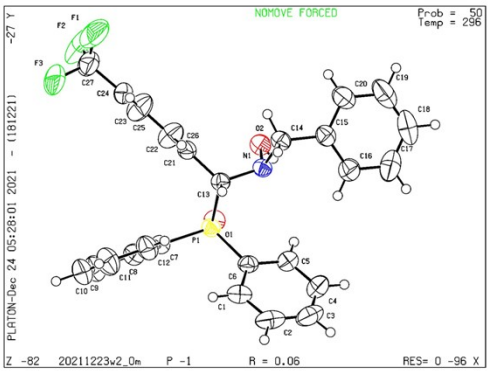
diphenyl((2S,3R,4S,5S,6R)-3,4,5-tris(benzyloxy)-6-((benzyloxy)methyl)-1-hydroxypiperidin-2-yl)phosphine oxide (5g): Following the **general procedure**, 6h, 85% yield, white solid, new compound, (ethyl acetate/petroleum ether=1/1). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.00 – 7.88 (m, 4H), 7.55 – 7.50 (m, 1H), 7.45 (m, 3H), 7.40 – 7.27 (m, 18H), 7.19 (m 4H), 5.73 (s, 1H), 4.65 (d, *J* = 12.2 Hz, 1H), 4.58 (d, *J* = 12.1 Hz, 1H), 4.49 (dd, *J* = 16.8, 12.1 Hz, 2H), 4.36 (d, *J* = 11.6 Hz, 1H), 4.31 – 4.22 (m, 3H), 4.08 – 3.97 (m, 3H), 3.93 (dd, *J* = 10.5, 5.1 Hz, 1H), 3.82 (dd, *J* = 10.6, 5.1 Hz, 1H), 3.57 (s, 1H), 3.25 (s, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.7, 138.4, 138.2 (d, *J* = 8.1 Hz), 134.0 (d, *J* = 85.4 Hz), 133.2 (d, *J* = 85.8 Hz), 132.3 (d, *J* = 9.1 Hz), 132.1 (d, *J* = 9.5 Hz), 131.5, 131.3 (d, *J* = 2.8 Hz), 128.5, 128.4 (d, *J* = 2.3 Hz), 128.3 (d, *J* = 14.3 Hz), 128.2 (d, *J* = 5.4 Hz), 128.0 (d, *J* = 15.5 Hz), 127.8 (d, *J* = 2.9 Hz), 127.7 (d, *J* = 7.7 Hz), 127.6, 73.1, 72.6 (d, *J* = 87.8 Hz), 71.4, 67.0, 29.7. HRMS (ESI) m/z Calculated for $C_{46}H_{46}NO_6P[M\text{-}Na]^+$ 762.2960, found 762.2965.

Structure determination (X-ray crystallographic data for 3e)

Determination of the basic skeleton of the compound. ((benzyl(hydroxy)amino)(4-(trifluoromethyl)phenyl)methyl)diphenylphosphine oxide (**3e**) was recrystallized by mixed solvent ethyl acetate/hexane and its basic skeleton was determined by X-ray diffraction analysis.The X-ray data have been deposited at the Cambridge Crystallographic Data Center (**CCDC 2232462**).



3e



Datablock:

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               alpha=92.062(7) beta=106.913(7)    gamma=109.626(7)
Temperature:  296 K
                    Calculated          Reported
Volume              1220.6(11)          1220.6(11)
Space group         P -1                 P -1
Hall group          -P 1                 -P 1
Moiety formula      C27 H23 F3 N O2 P    ?
Sum formula         C27 H23 F3 N O2 P    C27 H23 F3 N O2 P
Mr                  481.43               481.43
Dx,g cm-3           1.310                1.310
Z                   2                    2
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F000'               500.48
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Nref                5715                 5435
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Tmin'               0.967
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Data completeness= 0.951          Theta(max)= 27.687
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S = 1.086               Npar= 30
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References

1. A. Dondoni, S. Franco, F. Junquera, F. Merchan, P. Merino and T. TeJero, *Synth. Commun.*, 1994, **24**, 2537-2550.

2. I. A. Dmitriev, V. I. Supranovich, V. V. Levin, M. I. Struchkova and A. D. Dilman, *Adv. Synth. Catal.*, 2018, **360**, 3788-3792.

3. S. Cicchi, M. Marradi, A. Goti and A. Brandi, *Tetrahedron Lett.*, 2001, **42**, 6503-6505.

4. A. Brar, D. K. Unruh, N. Ling and C. Krempner, *Org. Lett.*, 2019, **21**, 6305-6309.

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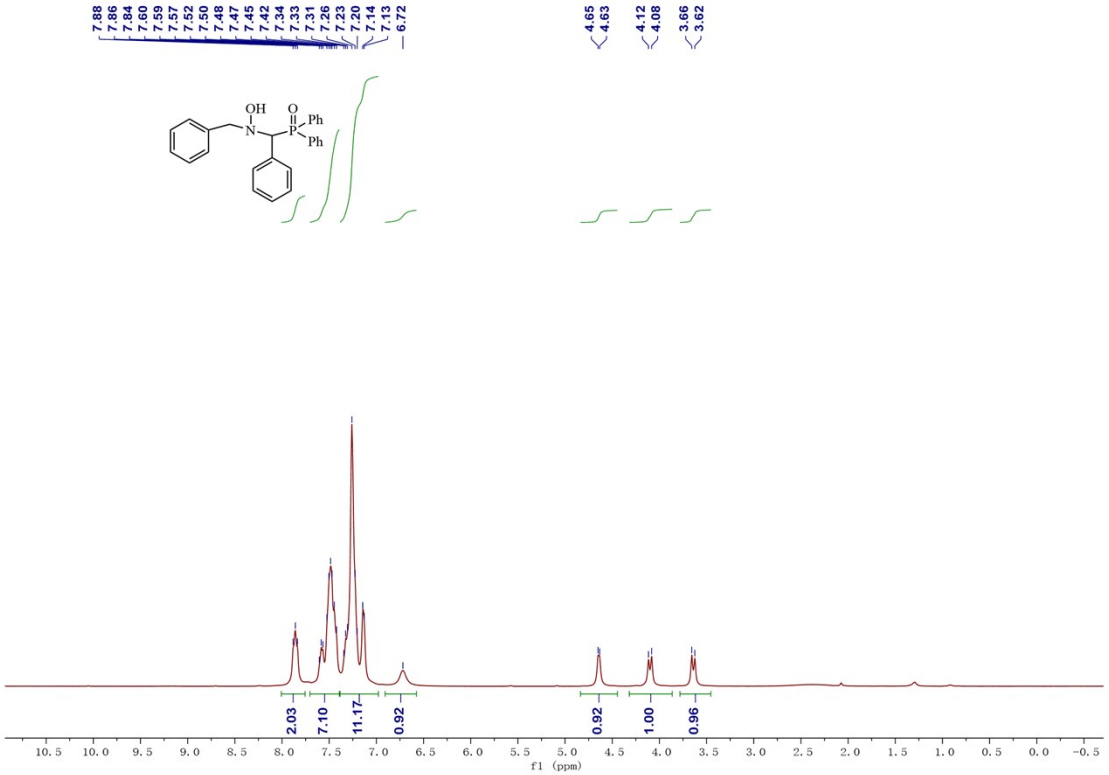
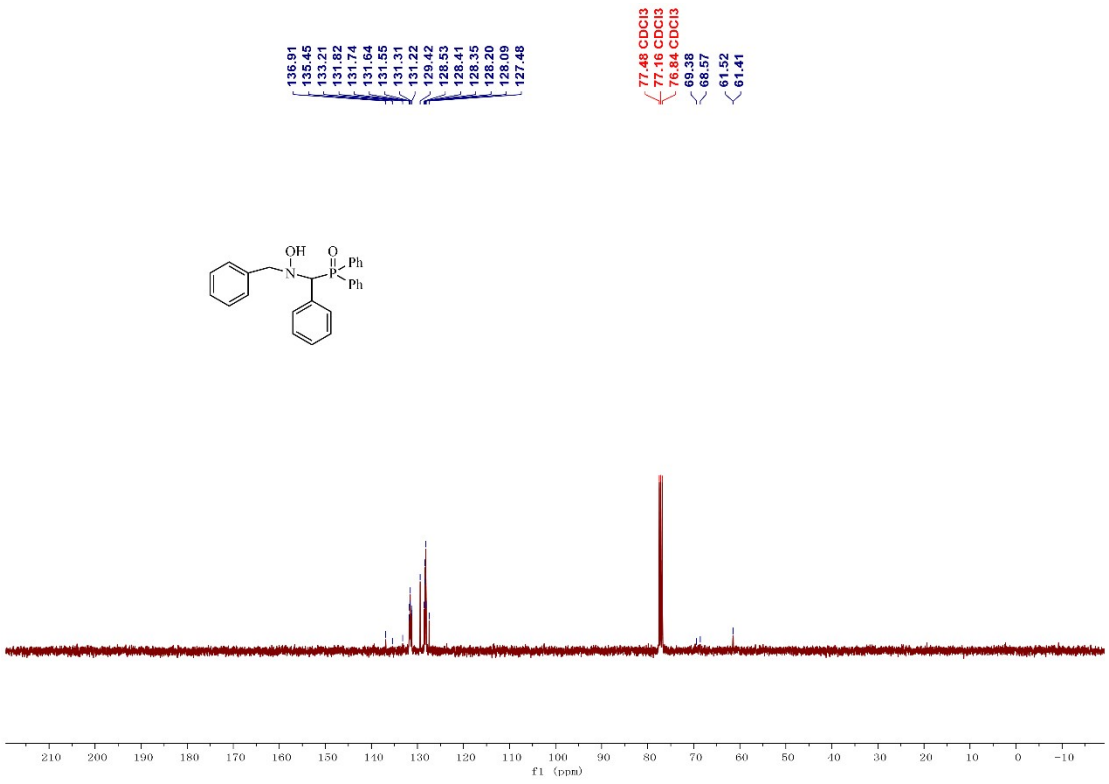
6. C. Liu, J. Gao, G. Yang, R. H. Wightman and S. Jiang, *Lett. Org. Chem.*, 2007, **4**, 556-558.

NMR Spectra for Products

3a





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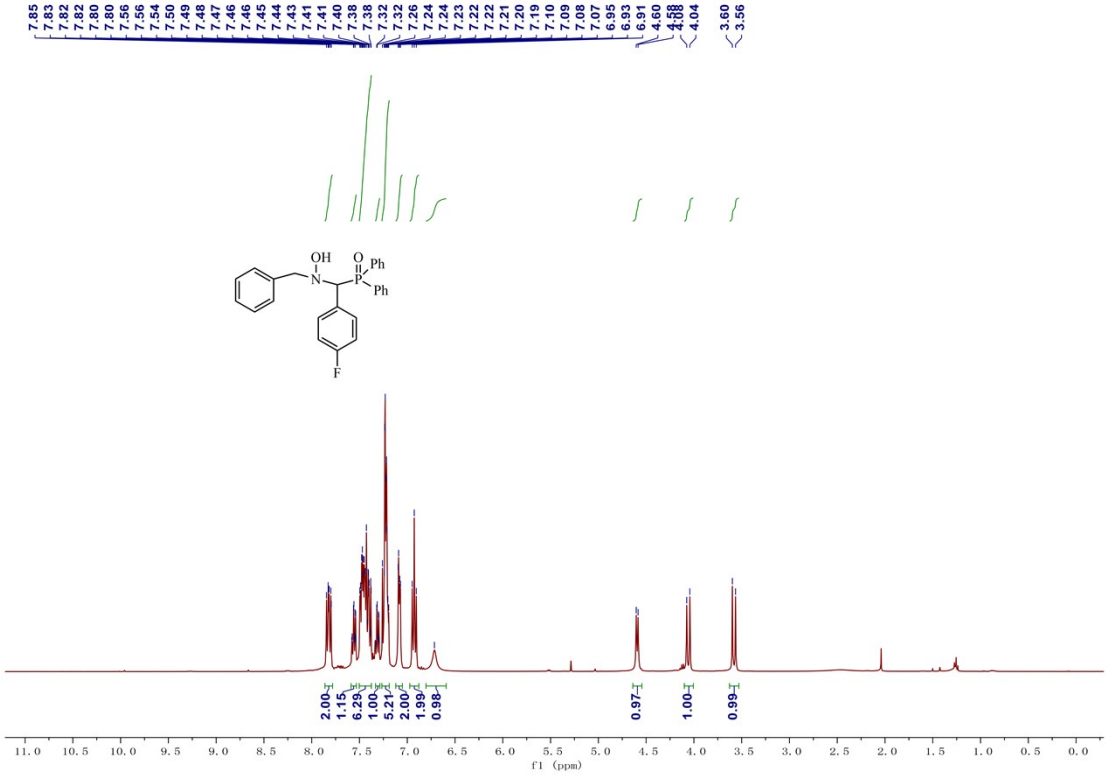
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O
P
Ph
Ph
F
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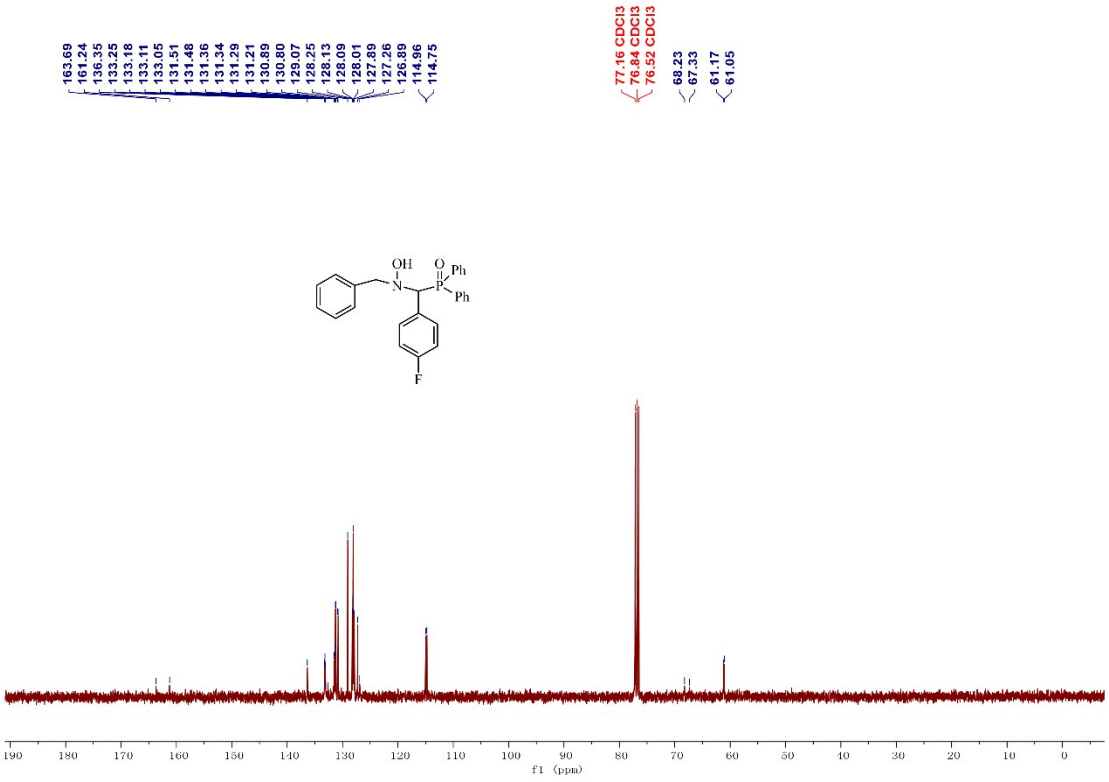
f1 (ppm)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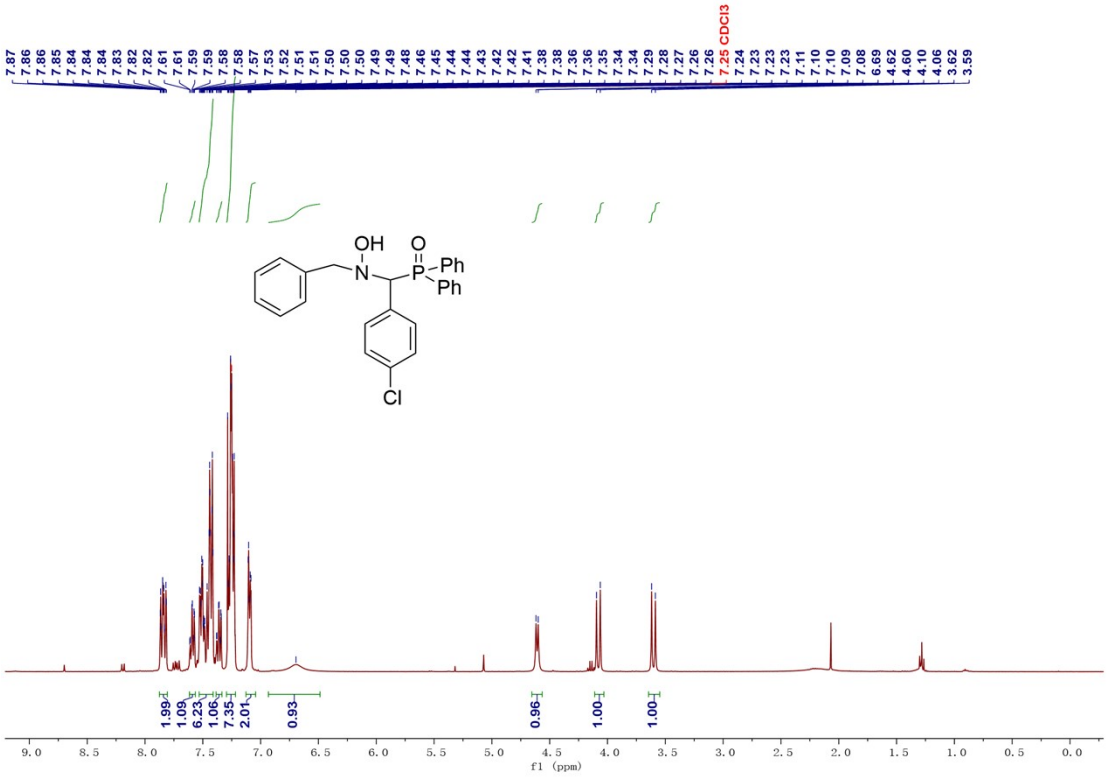
O
P
Ph
Ph
F
f1 (ppm)

3c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O
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P
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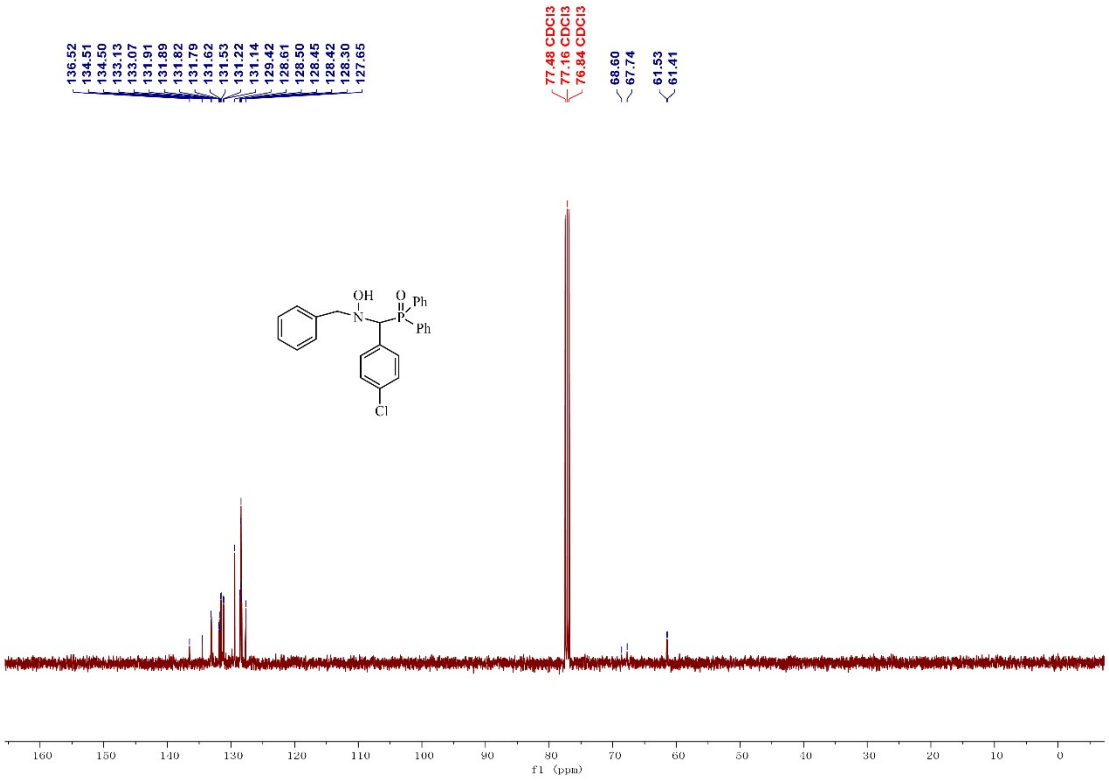
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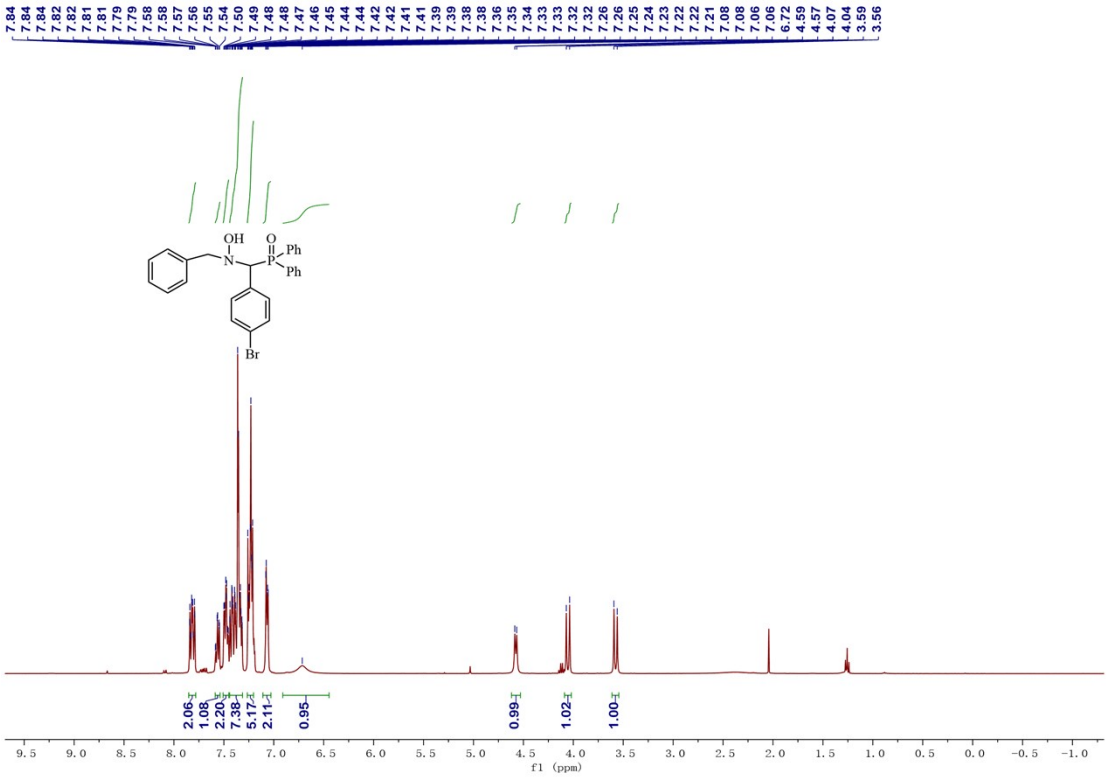
P
Ph
Ph
Cl
f1 (ppm)

3d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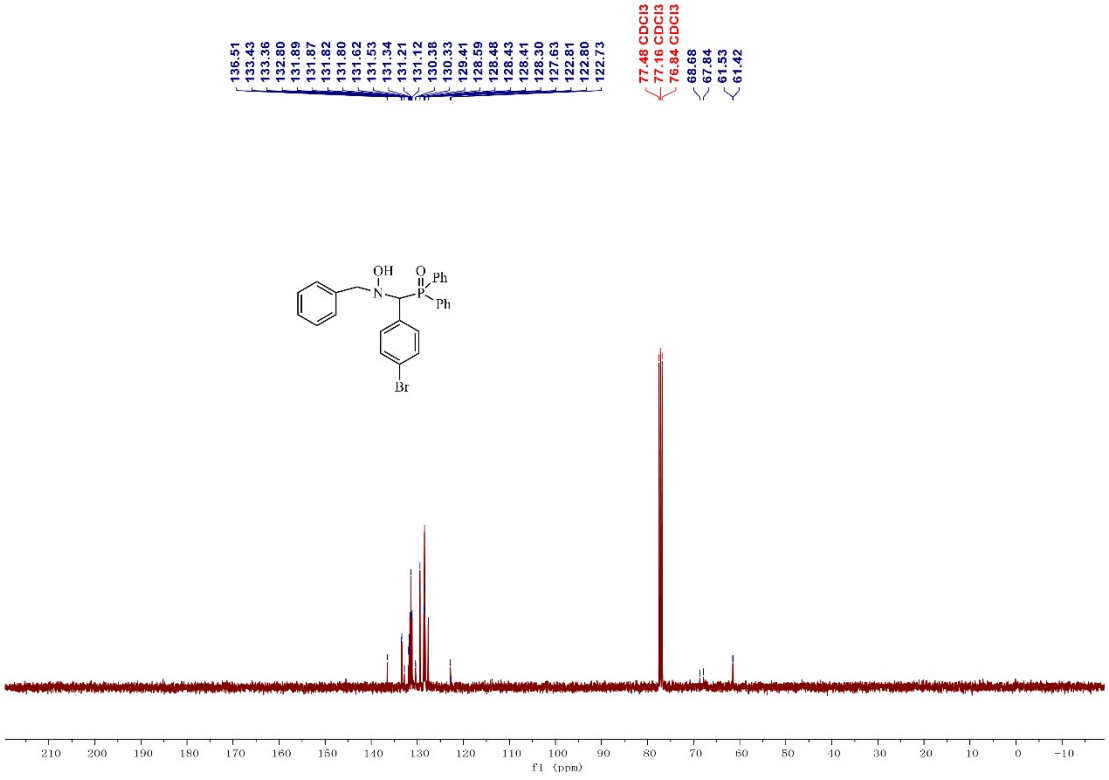
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P
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Ph
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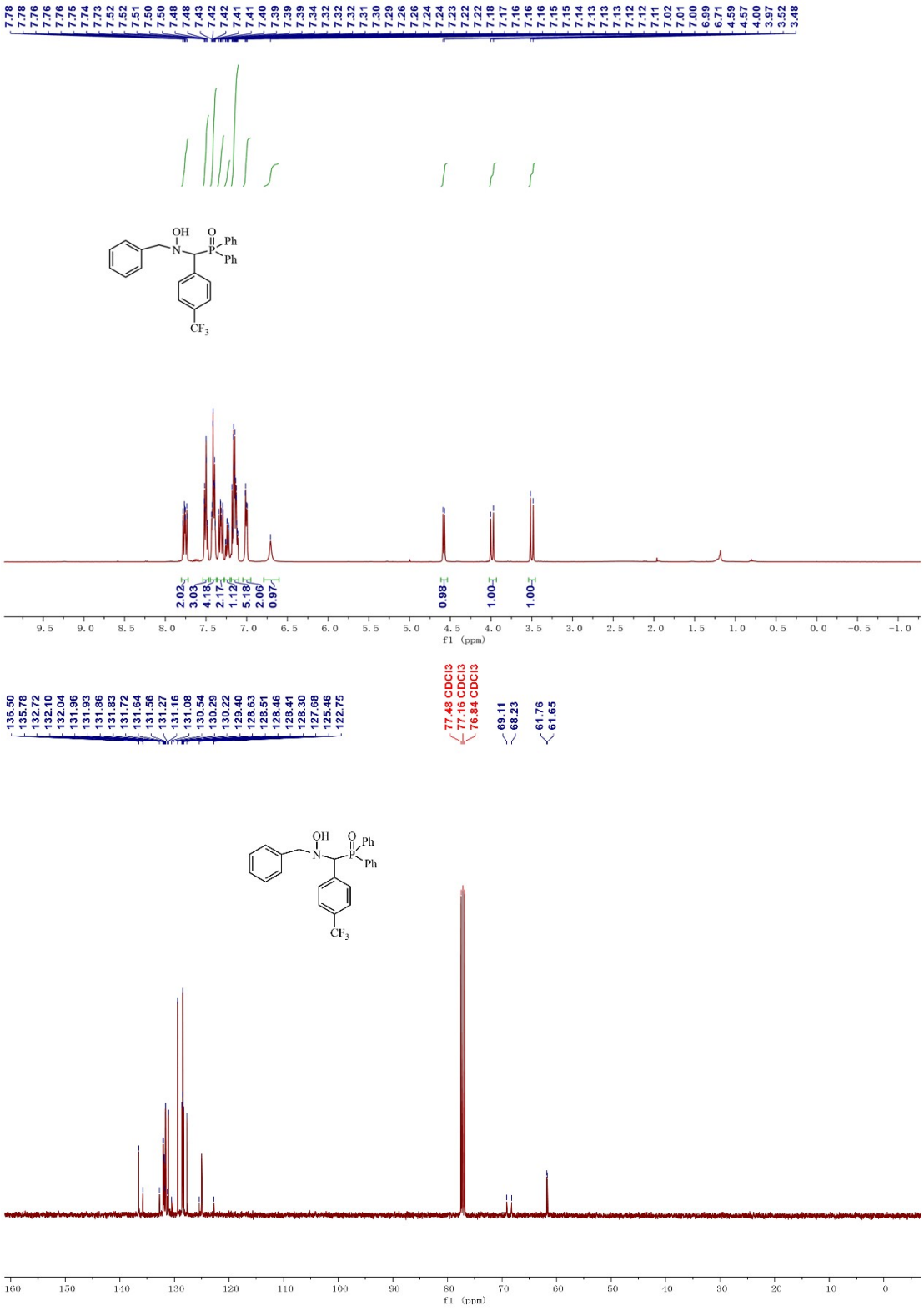
f1 (ppm)

3e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O
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Ph
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P
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f1 (ppm)
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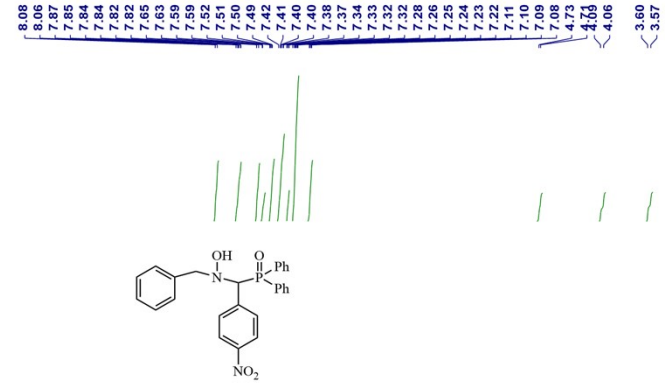
f1 (ppm)

3f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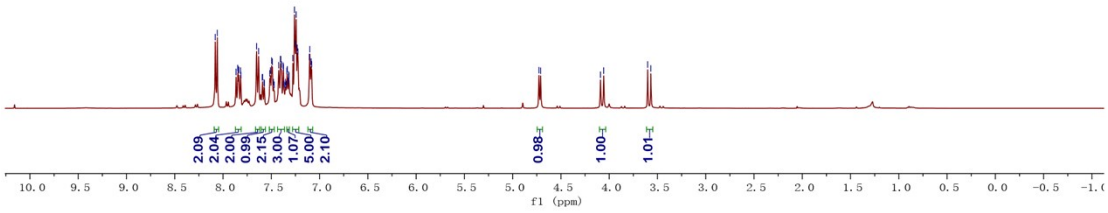
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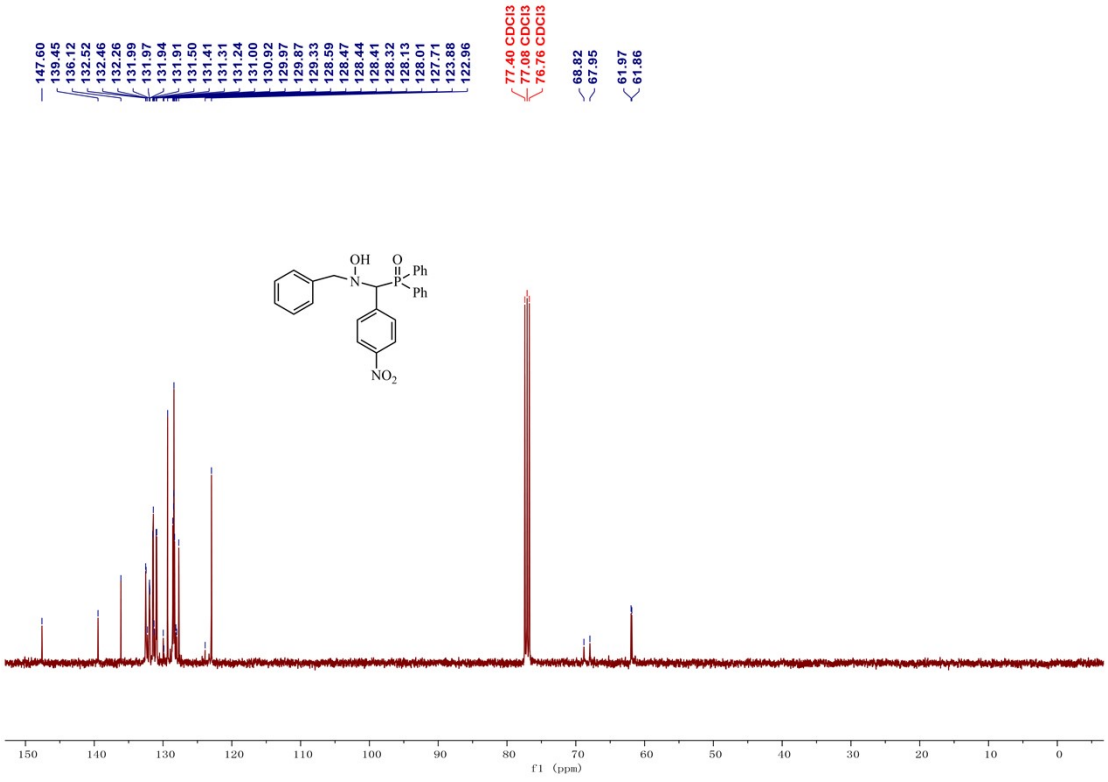
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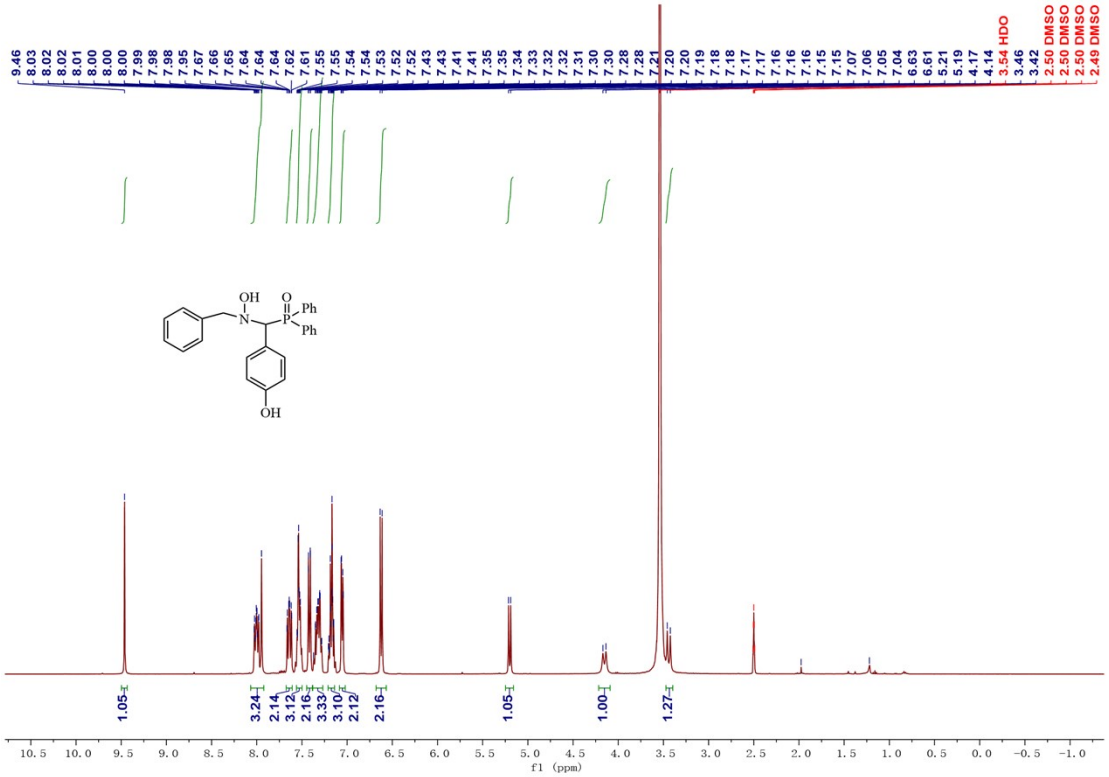
f1 (ppm)

3g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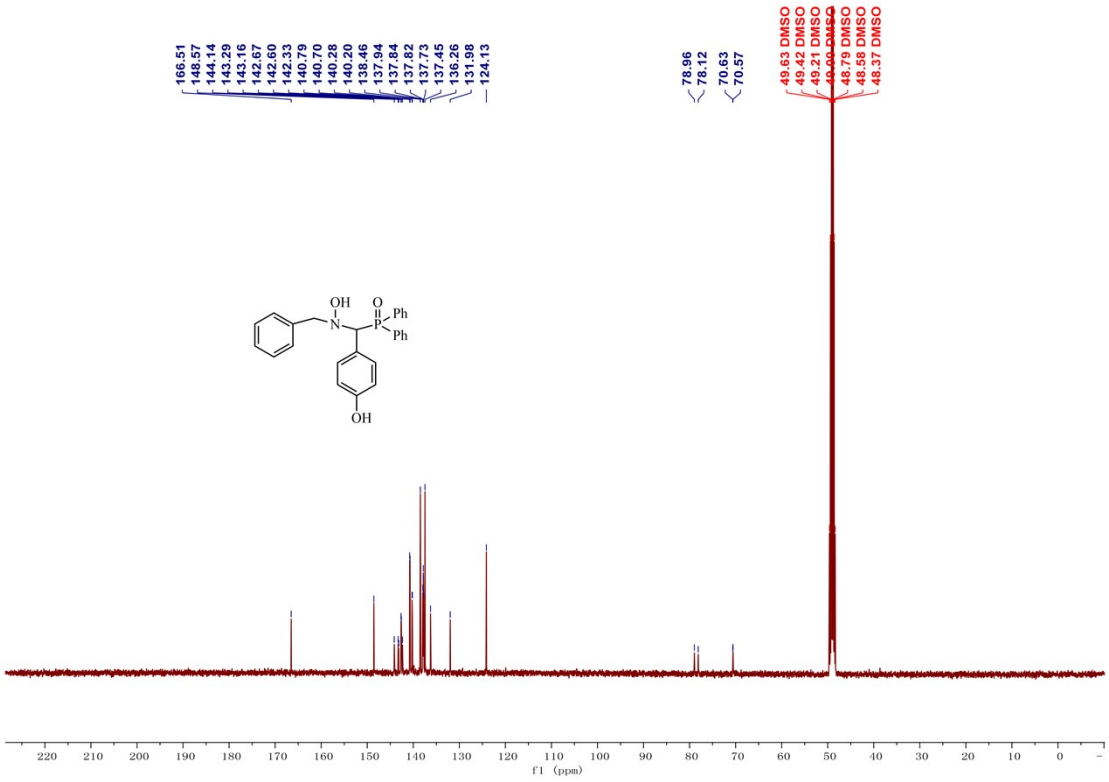
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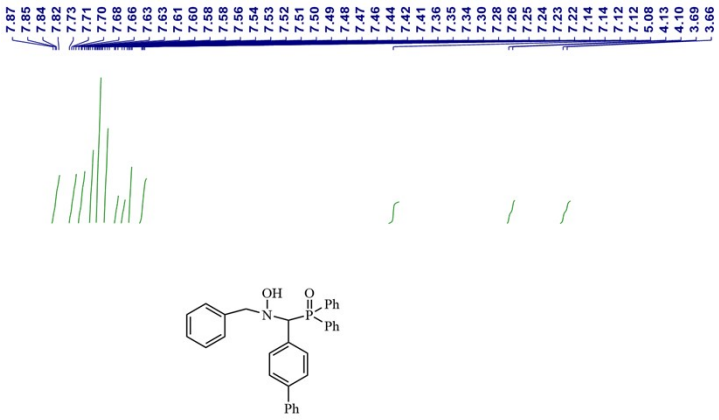
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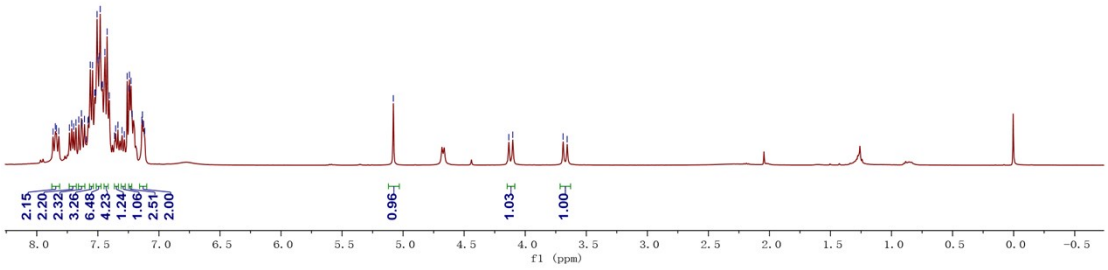
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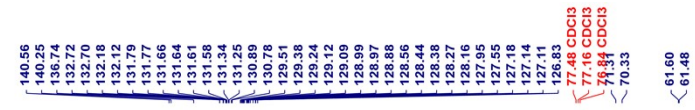
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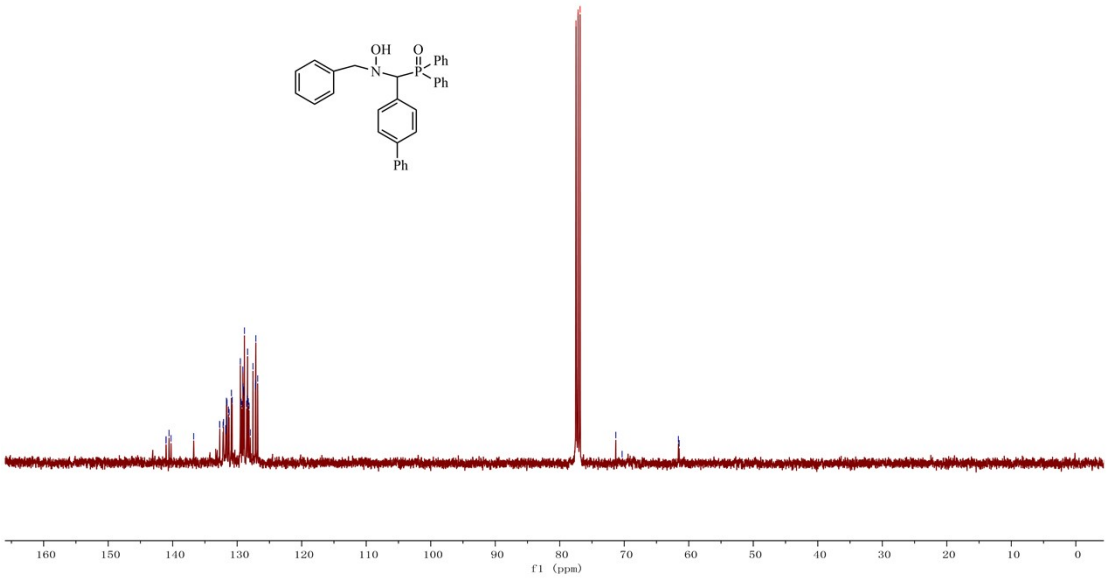
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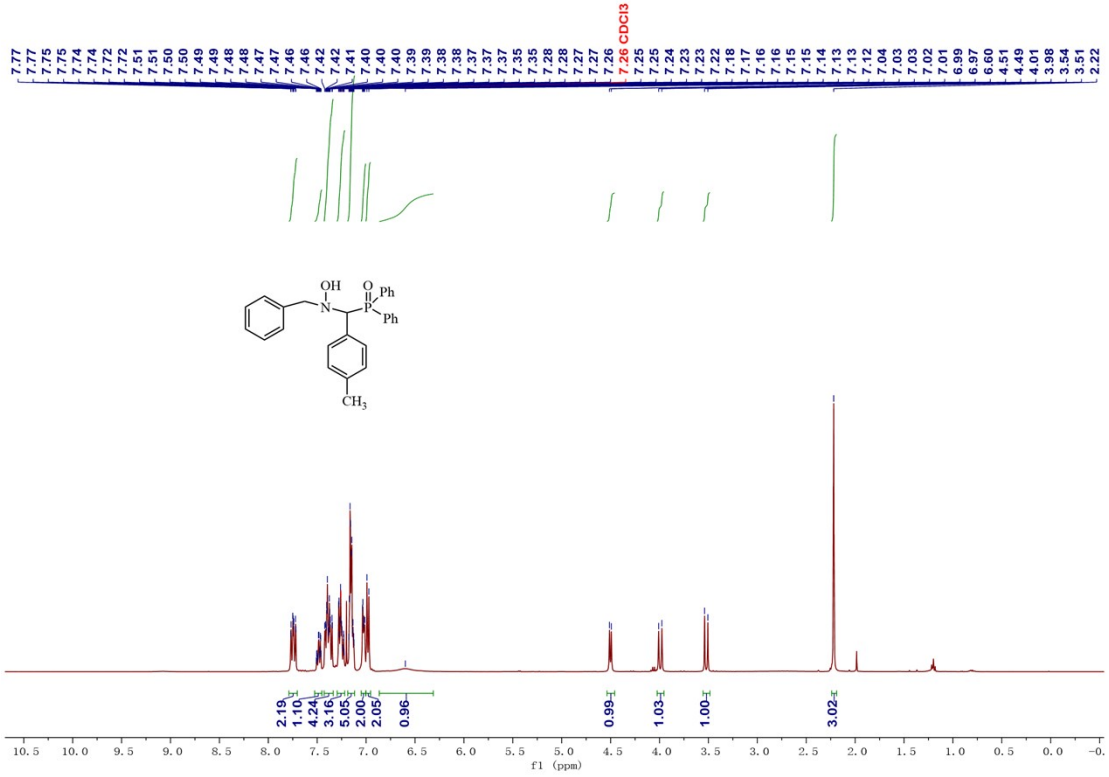
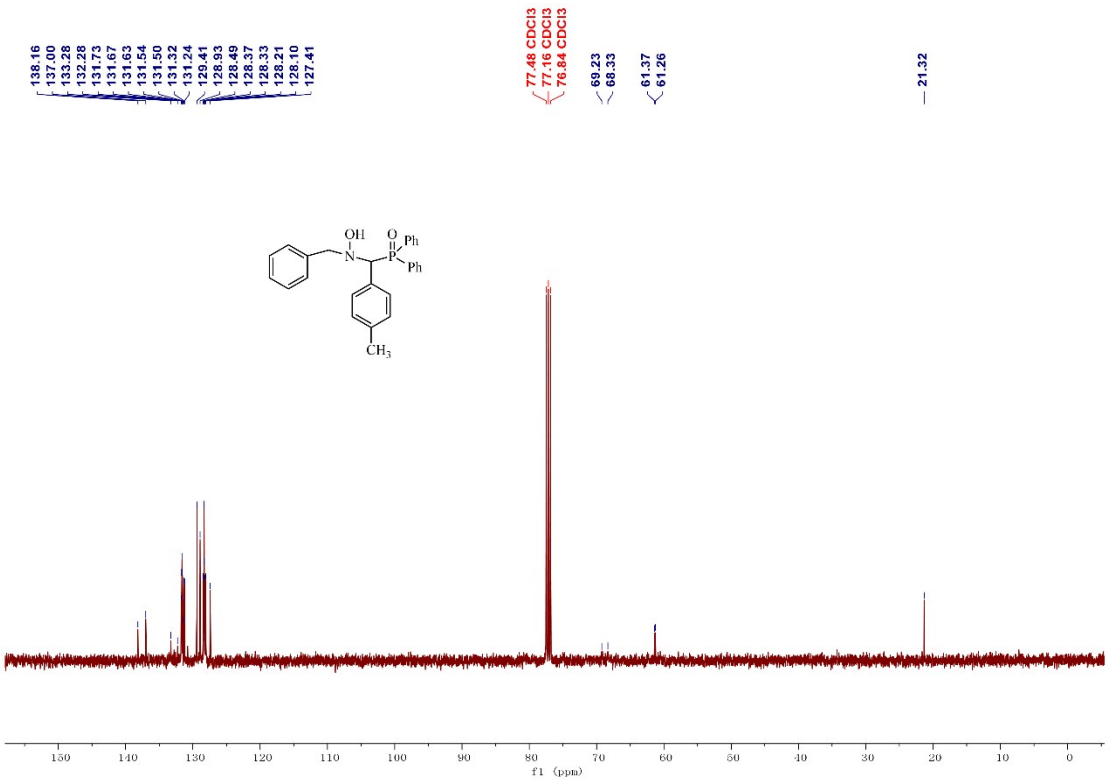
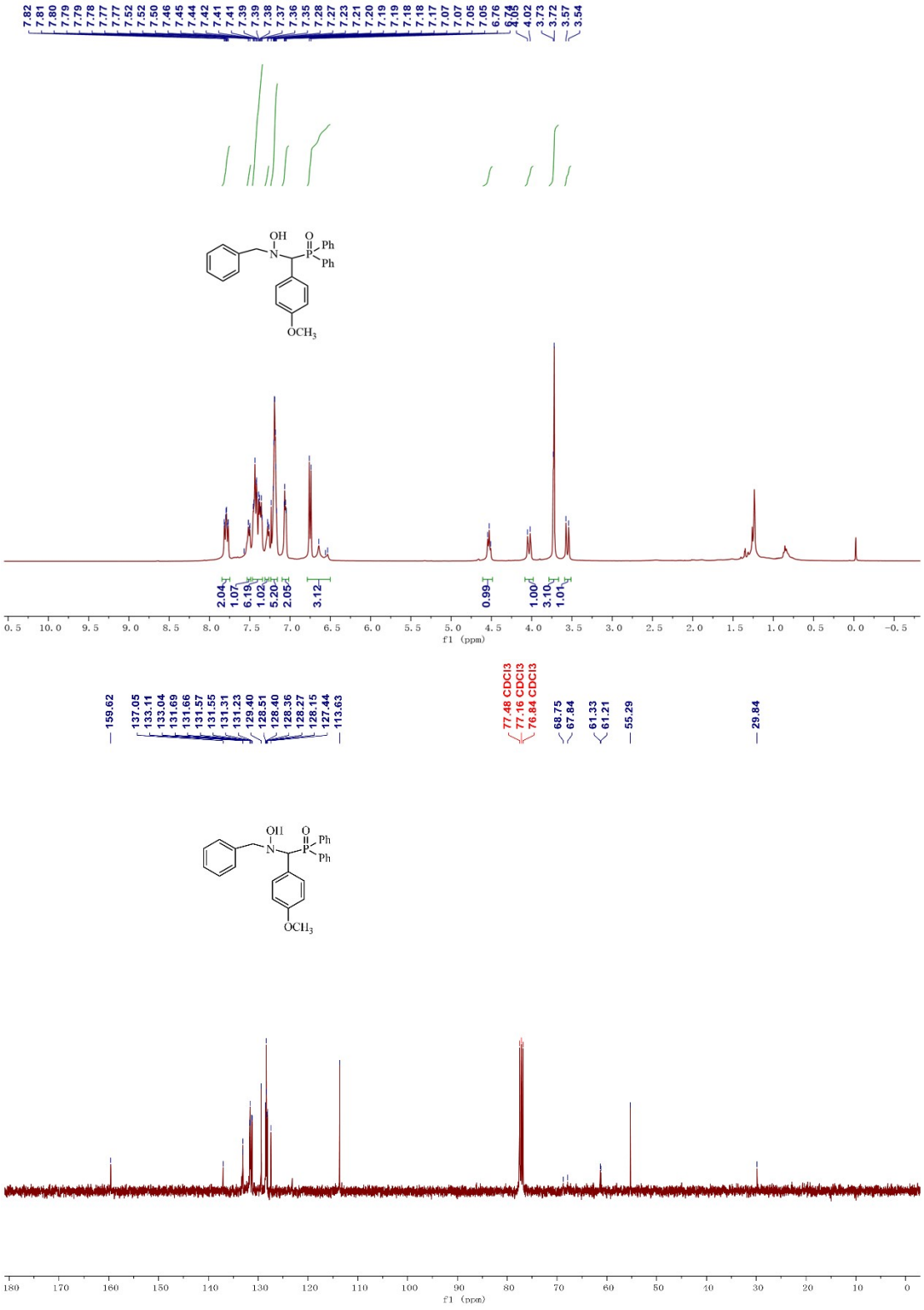
3i

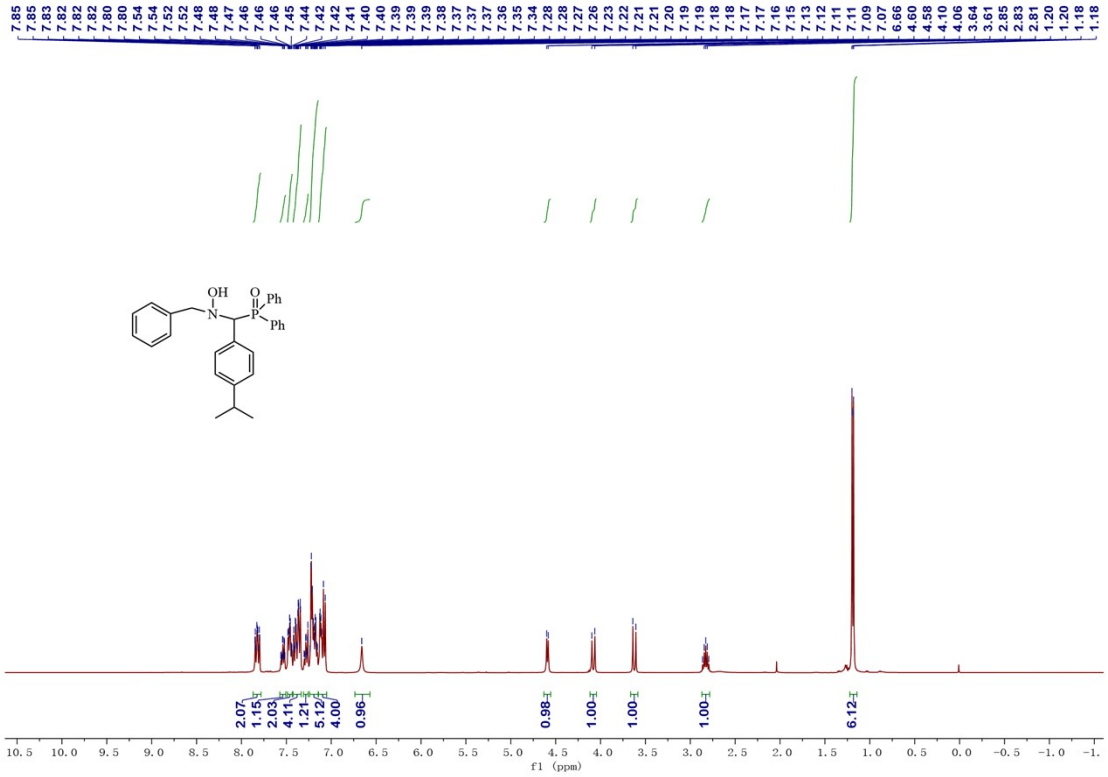

7.77 7.77 7.75 7.75 7.74 7.74 7.72 7.72 7.51 7.51 7.50 7.50 7.49 7.49 7.48 7.48 7.47 7.47 7.46 7.46 7.42 7.42 7.41 7.40 7.40 7.40 7.39 7.39 7.38 7.38 7.37 7.37 7.37 7.35 7.35 7.28 7.28 7.27 7.27 7.26 7.26 CDCl3 7.25 7.25 7.24 7.23 7.23 7.22 7.18 7.17 7.16 7.16 7.15 7.15 7.14 7.13 7.13 7.12 7.04 7.03 7.03 7.02 7.01 6.99 6.97 6.60 4.51 4.49 4.01 3.98 3.54 3.51 2.22
OH
N
O
P
Ph
Ph
CH3
2.19 1.10 4.24 3.16 5.05 2.00 2.05 0.96 0.99 1.03 1.00 3.02
f1 (ppm)

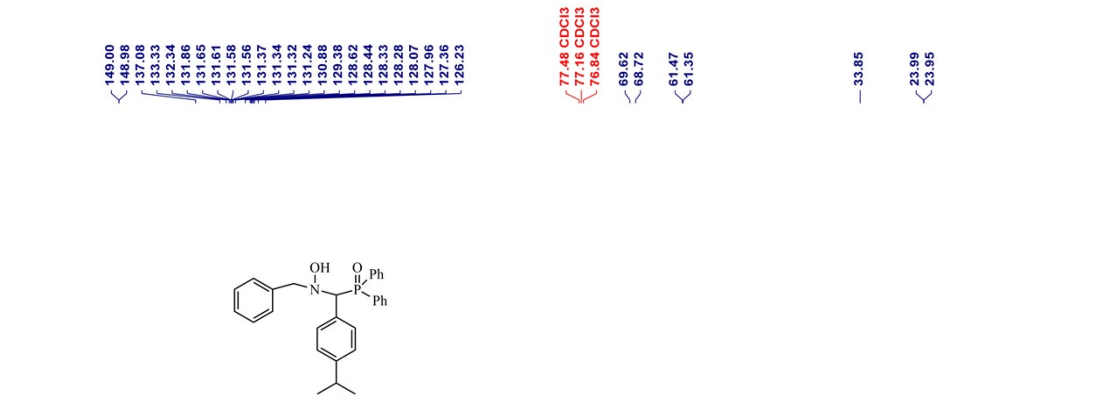

138.16 137.00 133.28 132.28 131.73 131.67 131.63 131.54 131.50 131.32 131.24 129.41 128.93 128.49 128.37 128.33 128.21 128.10 127.41
77.48 CDCl3 77.16 CDCl3 76.84 CDCl3 69.23 68.33 61.37 61.26 21.32
OH
N
O
P
Ph
Ph
CH3
f1 (ppm)

3J

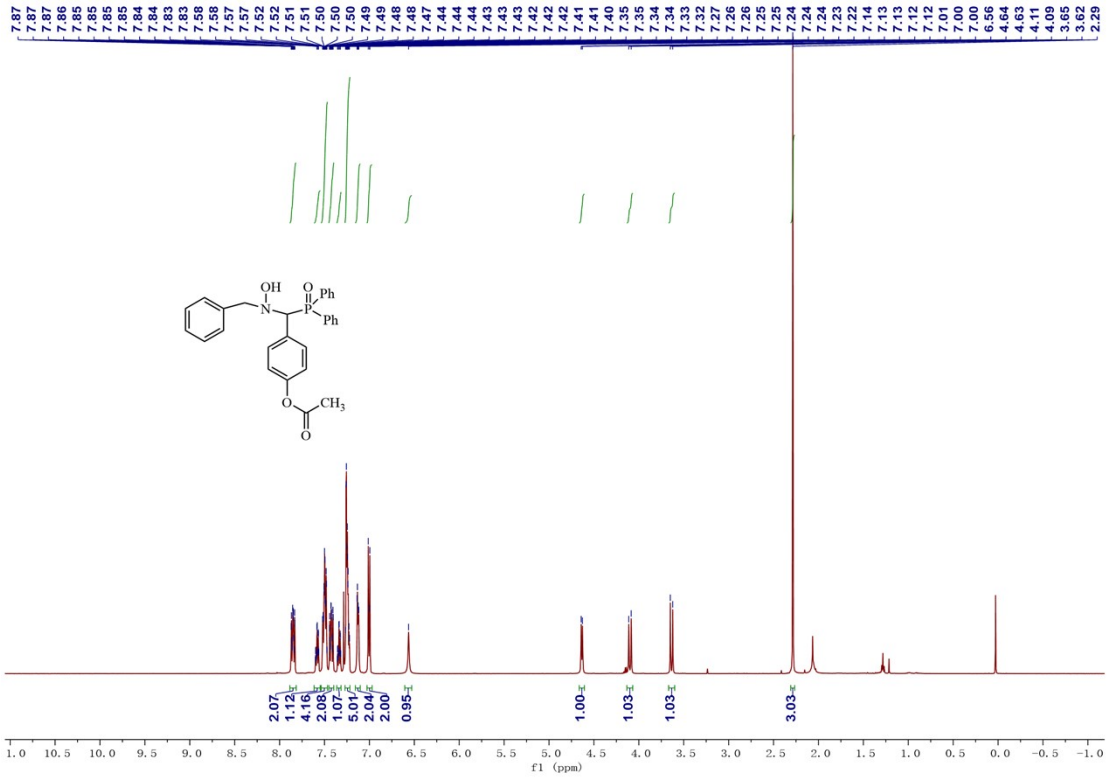

7.82 7.81 7.80 7.79 7.79 7.78 7.77 7.77 7.52 7.52 7.50 7.46 7.45 7.44 7.42 7.41 7.41 7.39 7.39 7.38 7.37 7.36 7.35 7.28 7.27 7.23 7.21 7.20 7.19 7.19 7.18 7.18 7.17 7.07 7.07 7.05 7.05 6.76 6.74 4.05 4.02 3.73 3.72 3.57 3.54
OH
O
N
P
Ph
Ph
OCH3
2.04 1.07 6.19 1.02 5.20 2.05 3.12 0.99 1.00 3.10 1.01
f1 (ppm)
159.62 137.05 133.11 133.04 131.69 131.66 131.57 131.55 131.31 131.23 129.40 128.51 128.40 128.36 128.27 128.15 127.44 113.63
77.48 CDCl3 77.16 CDCl3 76.84 CDCl3
68.75 67.84 61.33 61.21 55.29 29.84
OH
O
N
P
Ph
Ph
OCH3
f1 (ppm)

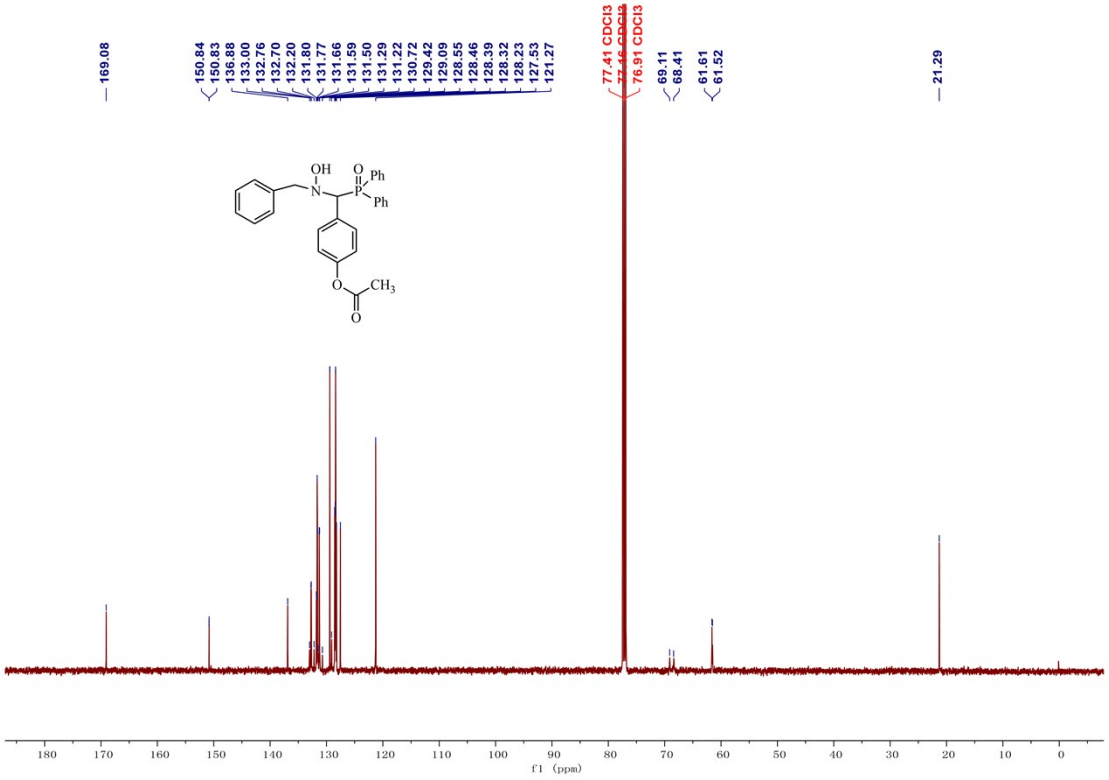
3k


7.85 7.85 7.83 7.82 7.82 7.82 7.80 7.80 7.54 7.52 7.52 7.48 7.48 7.47 7.46 7.46 7.46 7.45 7.44 7.42 7.42 7.41 7.40 7.40 7.39 7.39 7.38 7.37 7.37 7.36 7.35 7.34 7.28 7.28 7.27 7.26 7.23 7.22 7.21 7.21 7.20 7.19 7.19 7.18 7.18 7.17 7.17 7.16 7.15 7.13 7.12 7.11 7.11 7.09 7.07 6.66 4.60 4.58 4.10 4.06 3.64 3.61 2.85 2.83 2.81 1.20 1.20 1.18 1.18
OH
N
O
P
Ph
Ph
2.07 1.15 2.03 4.11 1.21 5.12 4.00 0.96 0.98 1.00 1.00 1.00 6.12
f1 (ppm)

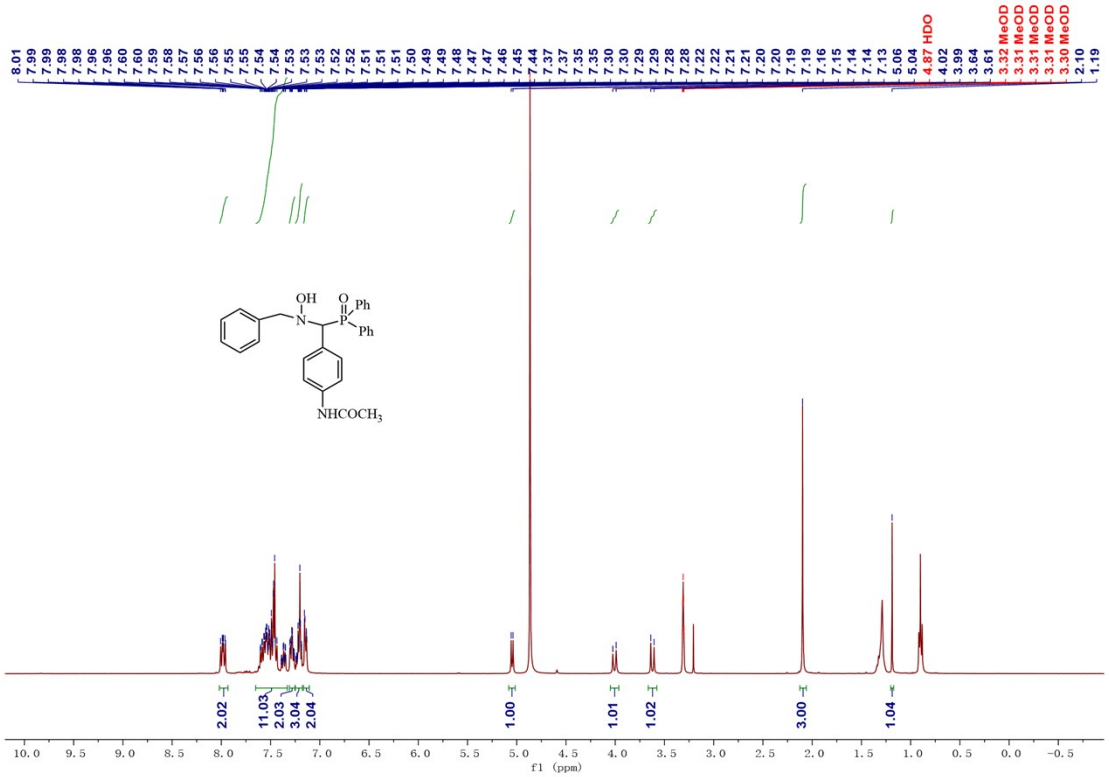

149.00 148.98 137.08 133.33 132.34 131.86 131.65 131.61 131.58 131.56 131.37 131.34 131.32 131.24 130.88 129.38 128.62 128.44 128.33 128.28 128.07 127.96 127.36 126.23
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
69.62 68.72 61.47 61.35 33.85 23.99 23.95
OH
N
O
P
Ph
Ph
f1 (ppm)

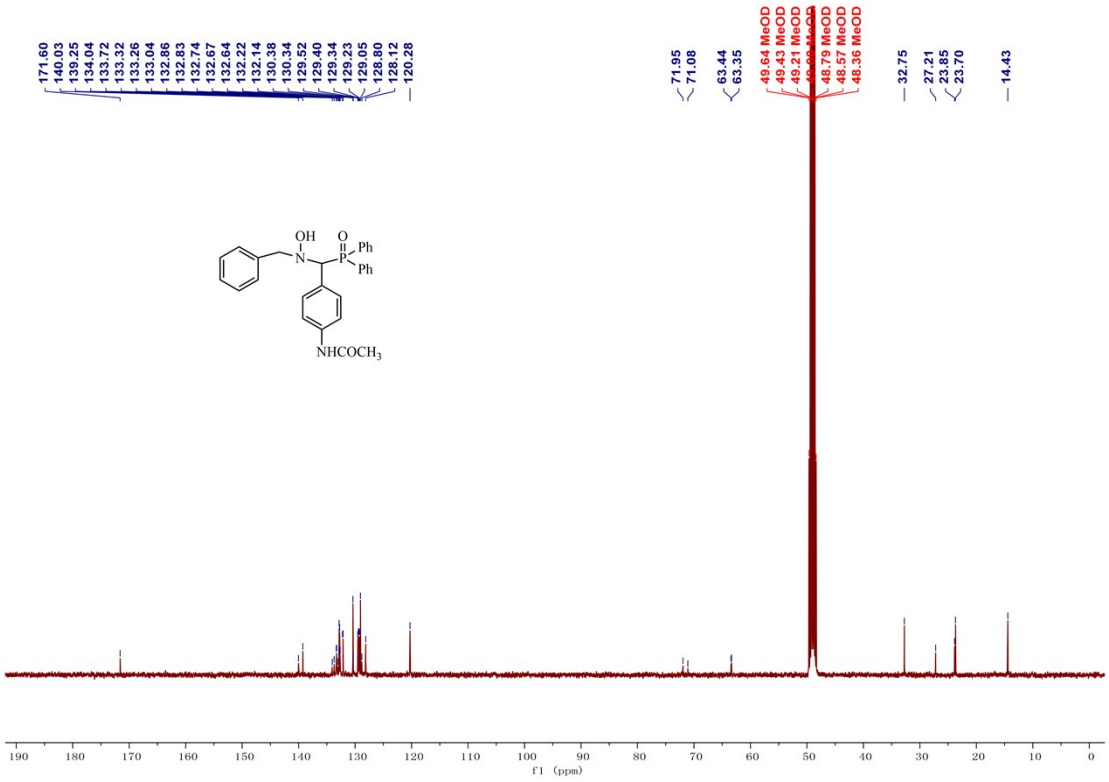
3l


7.87 7.87 7.87 7.86 7.85 7.85 7.85 7.84 7.84 7.83 7.83 7.58 7.57 7.57 7.52 7.52 7.51 7.51 7.50 7.50 7.50 7.49 7.49 7.48 7.48 7.47 7.44 7.44 7.44 7.43 7.43 7.42 7.42 7.42 7.41 7.41 7.40 7.35 7.35 7.34 7.34 7.33 7.32 7.27 7.26 7.26 7.25 7.25 7.24 7.24 7.24 7.23 7.22 7.14 7.13 7.13 7.12 7.12 7.01 7.00 7.00 6.56 4.64 4.63 4.11 4.09 3.65 3.62 2.29
OH
N
O
P
Ph
Ph
O
CH3
O
2.07 1.12 4.16 2.05 1.07 5.01 2.04 2.00 0.95 1.00 1.03 1.03 3.03
f1 (ppm)

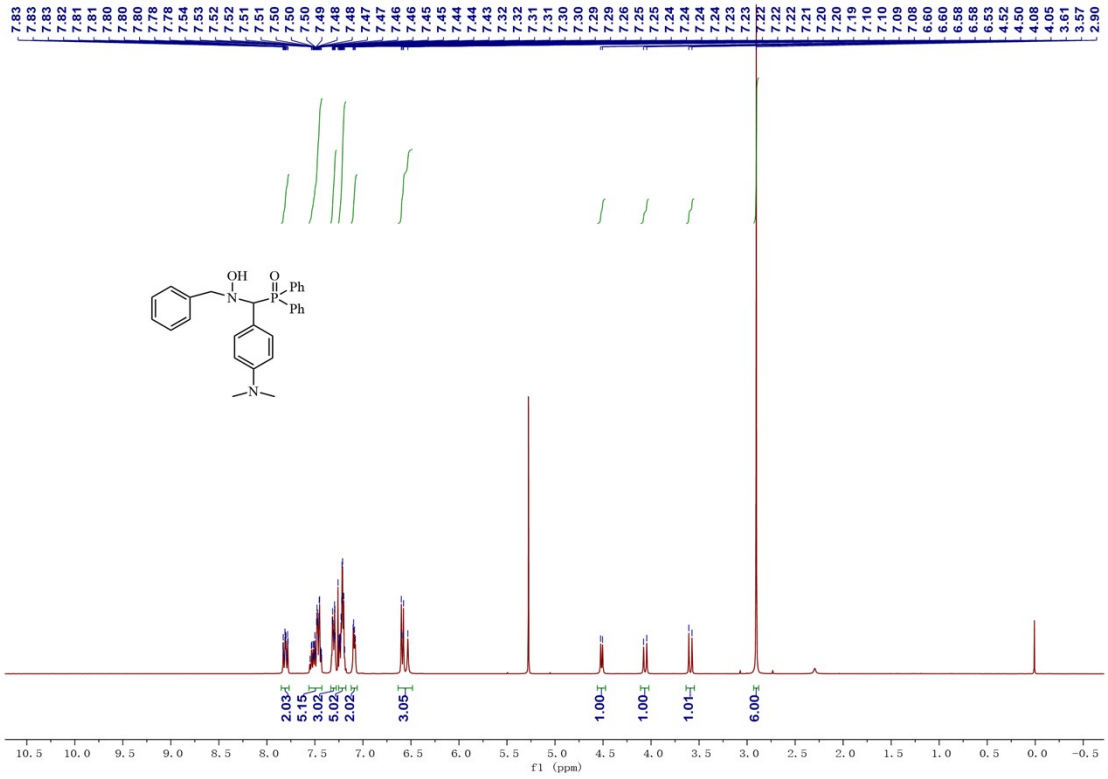

169.08
150.84 150.83 136.88 133.00 132.76 132.70 132.20 131.80 131.77 131.66 131.59 131.50 131.29 131.22 130.72 129.42 129.09 128.55 128.46 128.39 128.32 128.23 127.53 121.27
77.41 CDCl3
77.16 CDCl3
76.91 CDCl3
69.11 68.41 61.61 61.52
21.29
OH
N
O
P
Ph
Ph
O
CH3
O
f1 (ppm)

3m


8.01 7.99 7.99 7.98 7.98 7.96 7.96 7.60 7.59 7.58 7.57 7.56 7.56 7.55 7.54 7.54 7.53 7.53 7.52 7.52 7.51 7.51 7.51 7.50 7.49 7.49 7.48 7.47 7.47 7.46 7.45 7.44 7.37 7.37 7.35 7.35 7.30 7.29 7.29 7.28 7.28 7.22 7.21 7.21 7.20 7.20 7.19 7.19 7.16 7.15 7.14 7.14 7.13 5.06 5.04 4.87 HDO 4.02 3.99 3.64 3.61 3.32 MeOD 3.31 MeOD 3.31 MeOD 3.31 MeOD 3.30 MeOD 2.10 1.19
OH
N
O
P
Ph
Ph
NHCOCH3
2.02 11.03 2.03 3.04 2.04 1.00 1.01 1.02 3.00 1.04
f1 (ppm)


171.60 140.03 139.25 134.04 133.72 133.32 133.26 133.04 132.86 132.83 132.74 132.67 132.64 132.22 132.14 130.38 130.34 129.52 129.40 129.34 129.23 129.05 128.80 128.12 120.28
71.95 71.08 63.44 63.35
49.64 MeOD 49.43 MeOD 49.21 MeOD 48.79 MeOD 48.57 MeOD 48.36 MeOD
32.75 27.21 23.85 23.70 14.43
OH
N
O
P
Ph
Ph
NHCOCH3
f1 (ppm)

3n


7.83 7.83 7.82 7.81 7.81 7.80 7.80 7.80 7.78 7.78 7.54 7.53 7.52 7.52 7.51 7.51 7.50 7.50 7.50 7.49 7.48 7.48 7.47 7.47 7.46 7.46 7.45 7.45 7.44 7.44 7.43 7.32 7.31 7.31 7.30 7.30 7.29 7.29 7.26 7.25 7.25 7.24 7.24 7.24 7.23 7.23 7.22 7.22 7.22 7.21 7.20 7.20 7.19 7.10 7.10 7.09 7.08 6.60 6.60 6.58 6.58 6.53 4.52 4.50 4.08 4.05 3.61 3.57 2.90
OH
O
Ph
Ph
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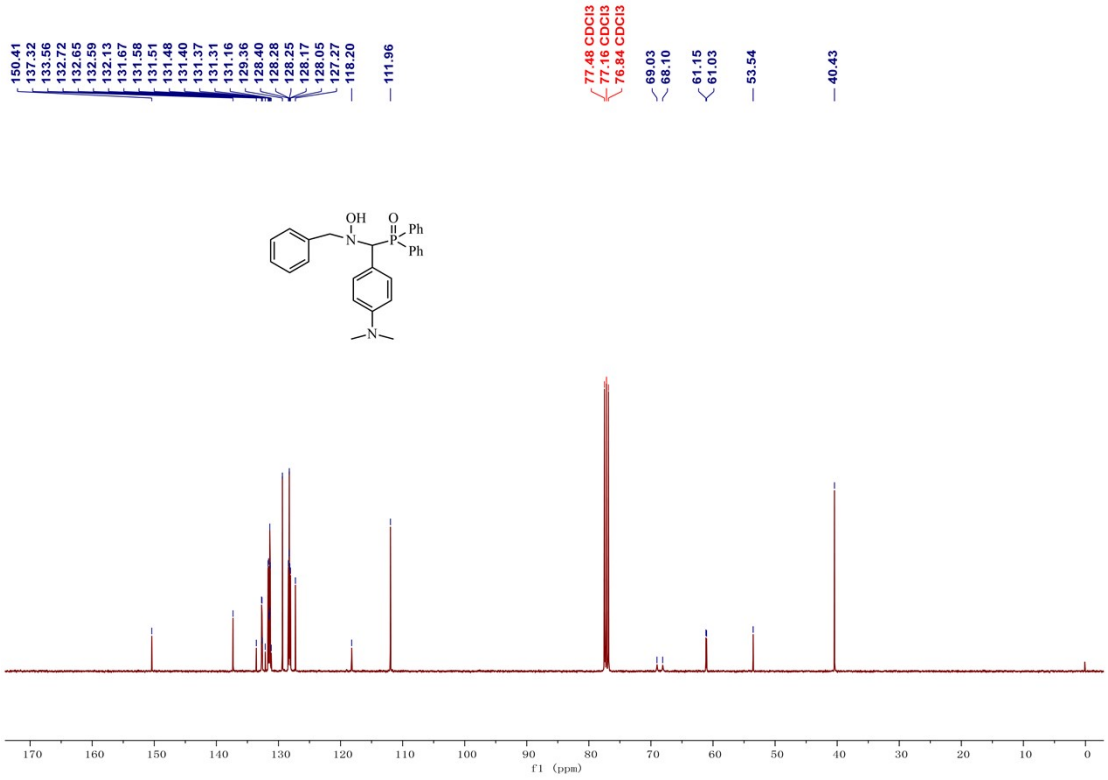
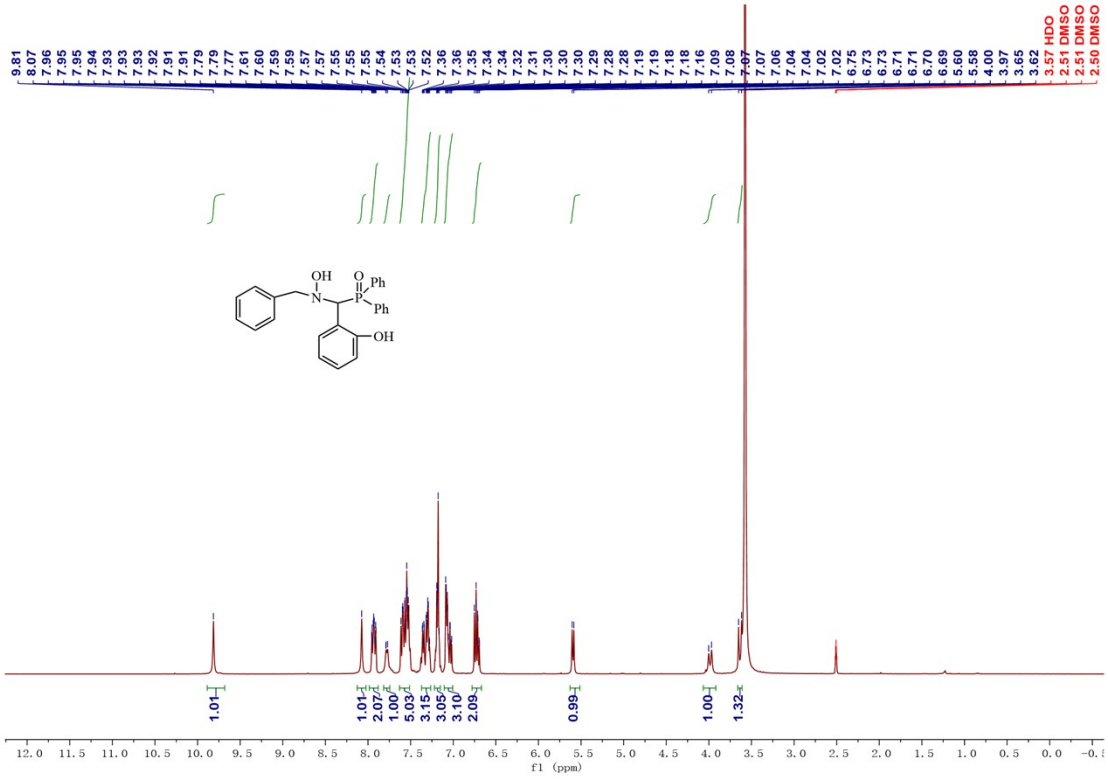
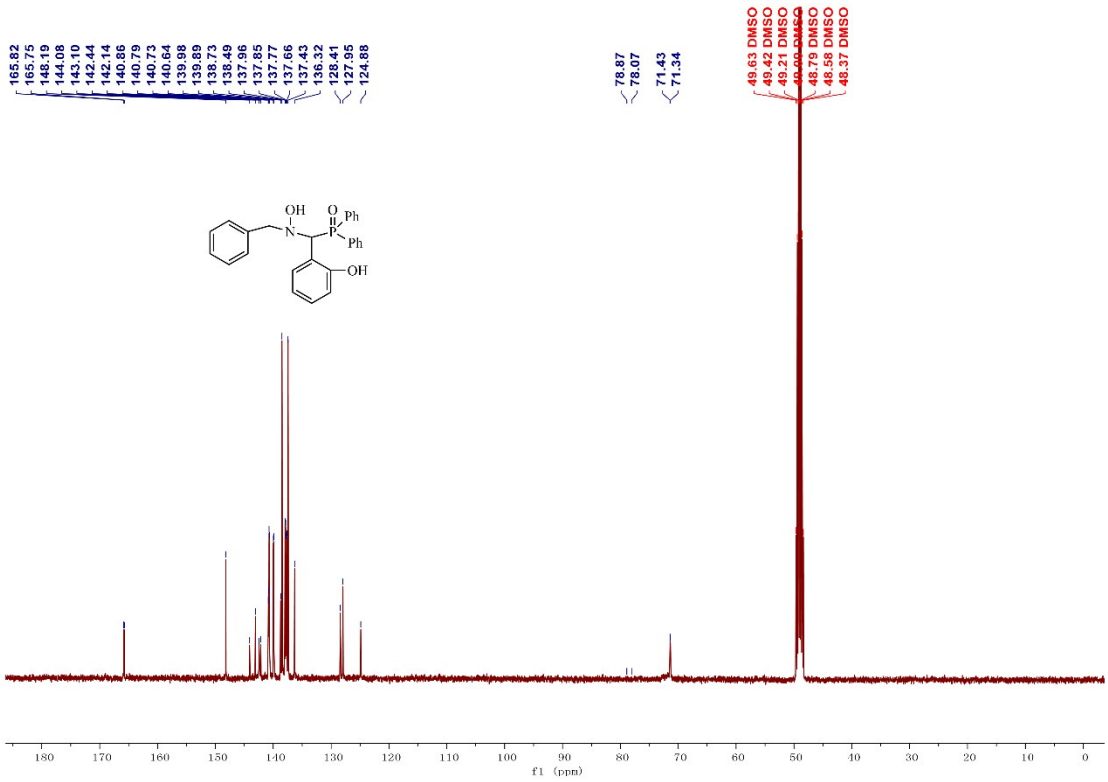
3.05
1.00
1.00
1.01
6.00
f1 (ppm)

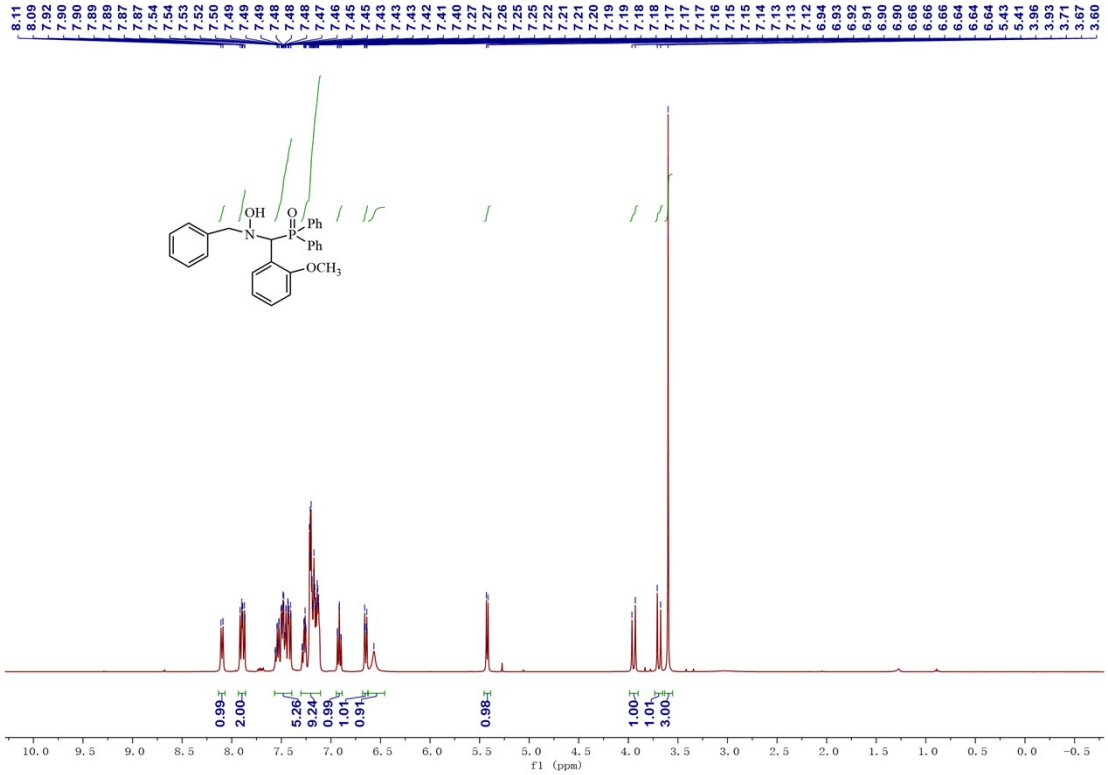

150.41 137.32 133.56 132.72 132.65 132.59 132.13 131.67 131.58 131.51 131.48 131.40 131.37 131.31 131.16 129.36 128.40 128.28 128.25 128.17 128.05 127.27 118.20 111.96
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
69.03 68.10 61.15 61.03 53.54 40.43
OH
O
Ph
Ph
N
f1 (ppm)

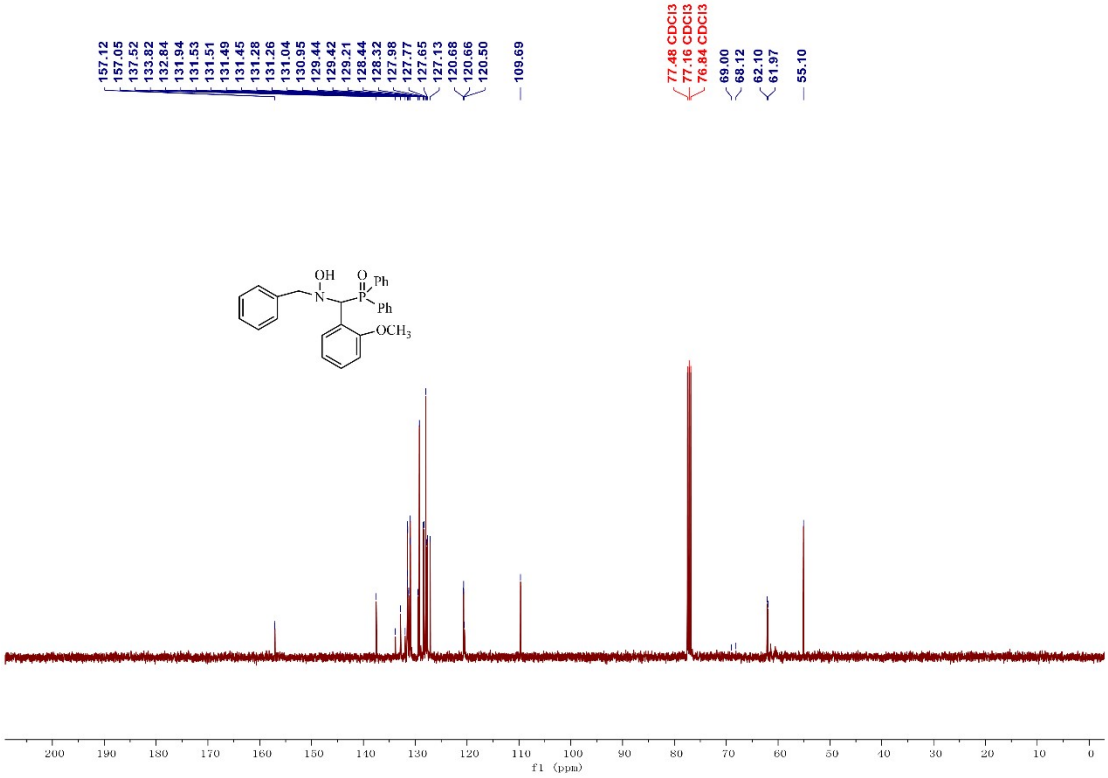
3o


9.81 8.07 7.96 7.95 7.95 7.94 7.93 7.93 7.92 7.91 7.91 7.79 7.79 7.77 7.61 7.60 7.59 7.59 7.57 7.57 7.55 7.55 7.55 7.54 7.53 7.53 7.52 7.36 7.36 7.35 7.34 7.34 7.32 7.31 7.30 7.30 7.30 7.29 7.28 7.28 7.19 7.19 7.18 7.18 7.16 7.09 7.08 7.07 7.07 7.06 7.04 7.04 7.02 7.02 6.75 6.73 6.73 6.71 6.70 6.69 5.60 5.58 4.00 3.97 3.65 3.62 3.57 HDO 2.51 DMSO 2.51 DMSO 2.50 DMSO
OH
N
O
P
Ph
Ph
OH
1.01 1.01 2.07 1.00 5.03 3.15 3.05 3.10 2.09 0.99 1.00 1.32
f1 (ppm)


165.82 165.75 148.19 144.08 143.10 142.44 142.14 140.86 140.79 140.73 140.64 139.98 139.89 138.73 138.49 137.96 137.85 137.77 137.66 137.43 136.32 128.41 127.95 124.88
78.87 78.07 71.43 71.34
49.63 DMSO 49.42 DMSO 49.21 DMSO 48.79 DMSO 48.58 DMSO 48.37 DMSO
OH
N
O
P
Ph
Ph
OH
f1 (ppm)

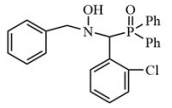
3p

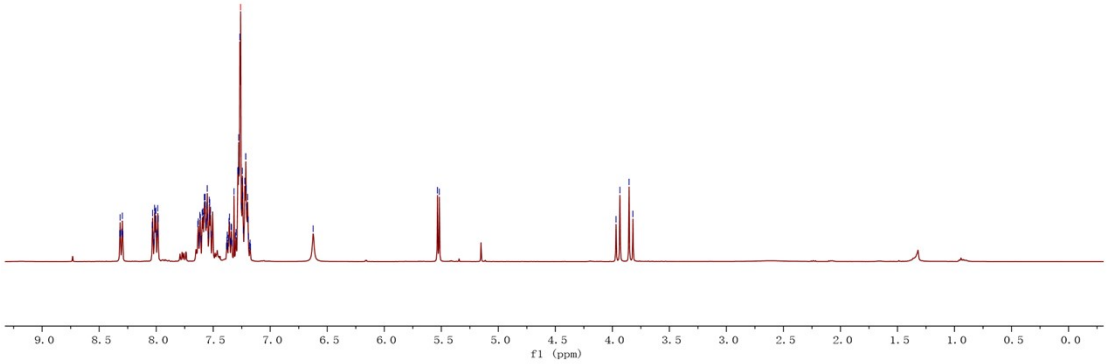

8.11 8.09 7.92 7.90 7.90 7.89 7.89 7.87 7.87 7.54 7.54 7.53 7.52 7.50 7.49 7.49 7.49 7.48 7.48 7.48 7.47 7.46 7.45 7.45 7.43 7.43 7.43 7.42 7.41 7.40 7.27 7.27 7.26 7.25 7.25 7.22 7.21 7.21 7.20 7.19 7.19 7.18 7.18 7.17 7.17 7.17 7.16 7.15 7.15 7.14 7.13 7.13 7.12 6.94 6.93 6.92 6.91 6.90 6.90 6.66 6.66 6.64 6.64 6.64 5.43 5.41 3.96 3.93 3.71 3.67 3.60
OH
N
O
P
Ph
Ph
OCH3
0.99 2.00 5.26 9.24 0.99 1.01 0.91 0.98 1.00 1.01 3.00
10.0 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 −0.5
f1 (ppm)

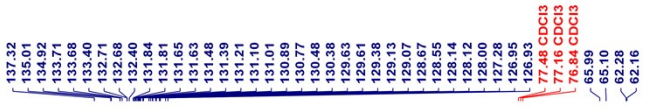

157.12 157.05 137.52 133.82 132.84 131.94 131.53 131.51 131.49 131.45 131.28 131.26 131.04 130.95 129.44 129.42 129.21 128.44 128.32 127.98 127.77 127.65 127.13 120.68 120.66 120.50 109.69
77.48 CDCl3 77.16 CDCl3 76.84 CDCl3 69.00 68.12 62.10 61.97 55.10
OH
N
O
P
Ph
Ph
OCH3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

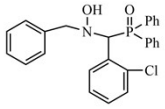
3q

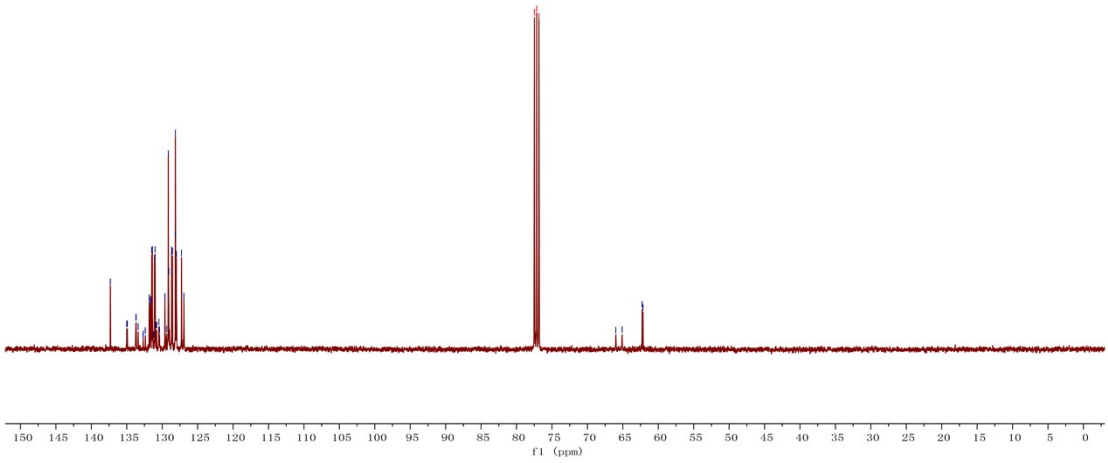

8.32 8.32 8.31 8.30 8.30 8.29 8.03 8.03 8.03 8.01 8.01 8.01 8.00 8.00 7.99 7.99 7.98 7.63 7.63 7.62 7.62 7.61 7.60 7.59 7.58 7.58 7.57 7.57 7.57 7.56 7.56 7.55 7.55 7.54 7.53 7.53 7.53 7.52 7.52 7.38 7.36 7.36 7.34 7.34 7.32 7.30 7.30 7.29 7.28 7.28 7.27 7.27 7.26 CDCl3 7.25 7.25 7.24 7.24 7.24 7.22 7.21 7.21 7.21 7.20 7.19 7.19 6.62 5.53 5.52 3.97 3.93 3.85 3.82


OH
O
N
P
Ph
Ph
Cl

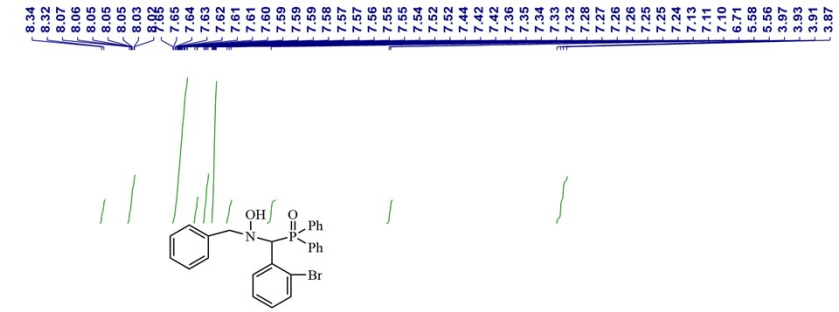

9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


137.32 135.01 134.92 133.71 133.68 133.40 132.71 132.68 132.40 131.84 131.81 131.65 131.63 131.48 131.39 131.21 131.10 131.01 130.89 130.77 130.48 130.38 129.63 129.61 129.38 129.13 129.07 128.67 128.55 128.14 128.12 128.00 127.28 126.95 126.93 77.48 CDCl3 77.16 CDCl3 76.84 CDCl3 65.99 65.10 62.28 62.16


OH
O
N
P
Ph
Ph
Cl


150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

3r


8.34
8.32
8.07
8.06
8.05
8.05
8.05
8.03
8.02
7.65
7.64
7.63
7.62
7.61
7.61
7.60
7.59
7.59
7.59
7.58
7.57
7.57
7.56
7.55
7.55
7.54
7.52
7.52
7.44
7.42
7.36
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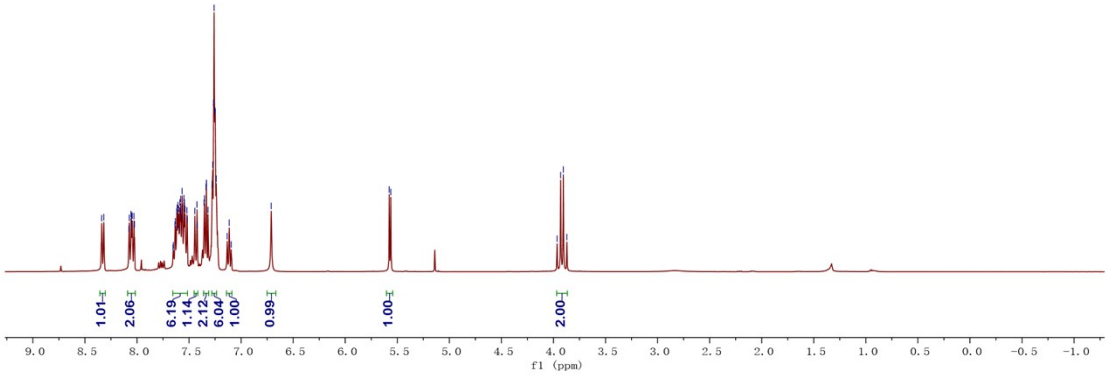
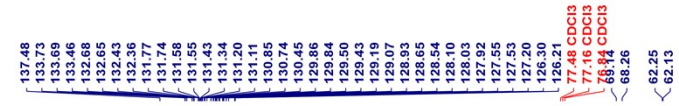
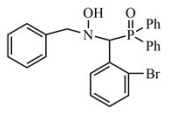
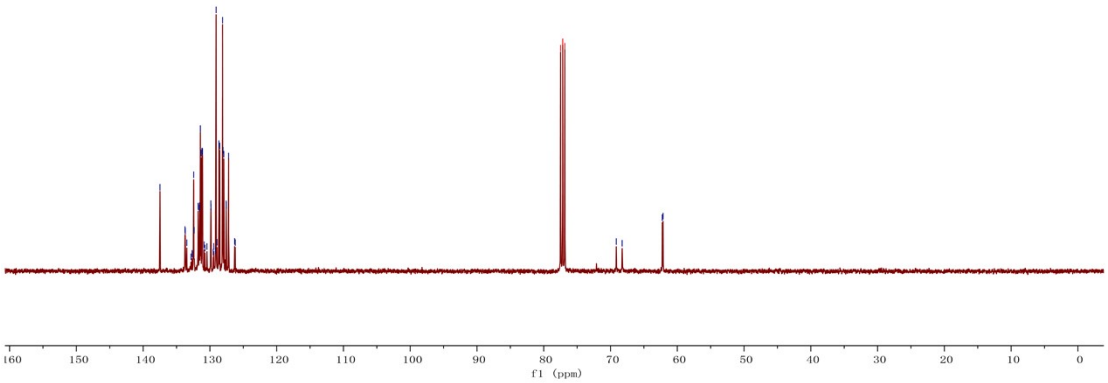
7.34
7.33
7.32
7.28
7.27
7.26
7.26
7.25
7.25
7.24
7.13
7.11
7.10
6.71
5.58
5.56
3.97
3.93
3.91
3.87
OH
N
O
P
Ph
Ph
Br


1.01
2.06
6.19
1.14
2.12
6.04
1.00
0.99
1.00
2.00
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
-0.5
-1.0
f1 (ppm)

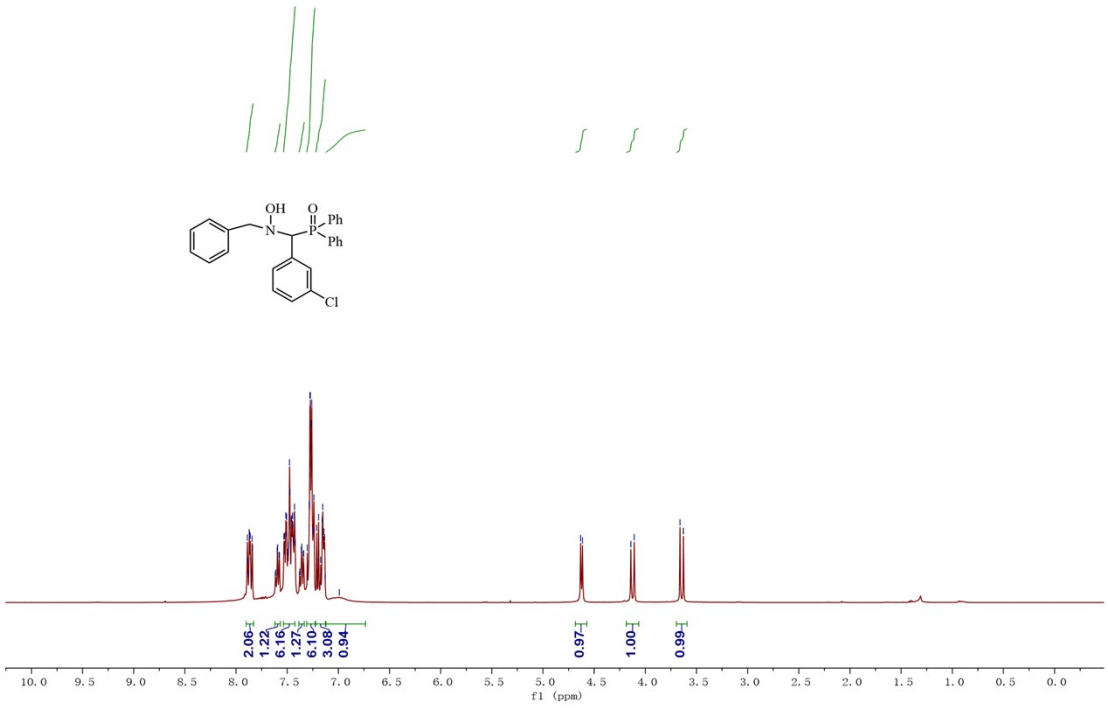

137.48
133.73
133.69
133.46
132.68
132.65
132.43
132.36
131.77
131.74
131.58
131.55
131.43
131.34
131.20
131.11
130.85
130.74
130.45
129.86
129.84
129.50
129.43
129.19
129.07
128.93
128.65
128.54
128.10
128.03
127.92
127.55
127.53
127.20
126.30
126.21
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
68.44
68.26
62.25
62.13


OH
N
O
P
Ph
Ph
Br


160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

3s


7.89 7.89 7.88 7.87 7.87 7.86 7.84 7.84 7.62 7.61 7.60 7.59 7.58 7.58 7.53 7.53 7.51 7.51 7.50 7.49 7.49 7.48 7.47 7.46 7.46 7.45 7.45 7.44 7.43 7.43 7.42 7.38 7.36 7.36 7.34 7.34 7.30 7.29 7.28 7.27 7.27 7.26 7.25 7.24 7.21 7.19 7.17 7.16 7.15 7.14 7.13 7.13 6.99 4.63 4.61 4.14 4.11 3.66 3.63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O
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P
Ph
Ph
Cl
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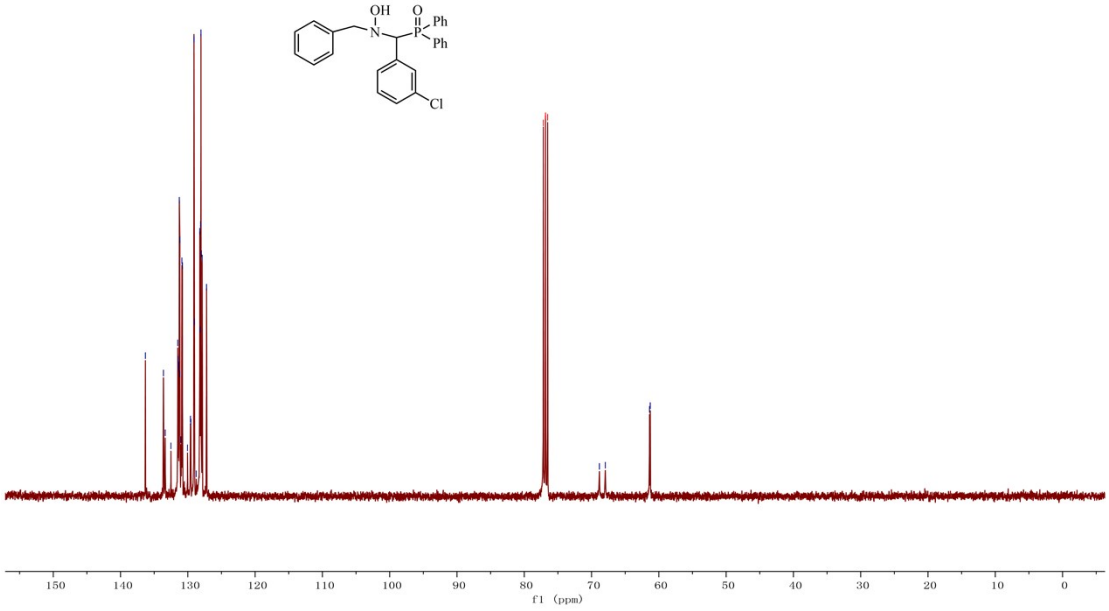
1.27
6.10
3.08
0.94
0.97
1.00
0.99
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


136.31 133.62 133.37 132.51 131.49 131.47 131.40 131.37 131.27 131.21 131.18 131.03 130.88 130.80 130.05 129.61 129.55 129.09 129.01 128.74 128.23 128.18 128.11 128.05 127.98 127.86 127.22
77.16 CDCl3
76.84 CDCl3
76.52 CDCl3
68.83
67.95
61.39
61.28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N
P
Ph
Ph
Cl
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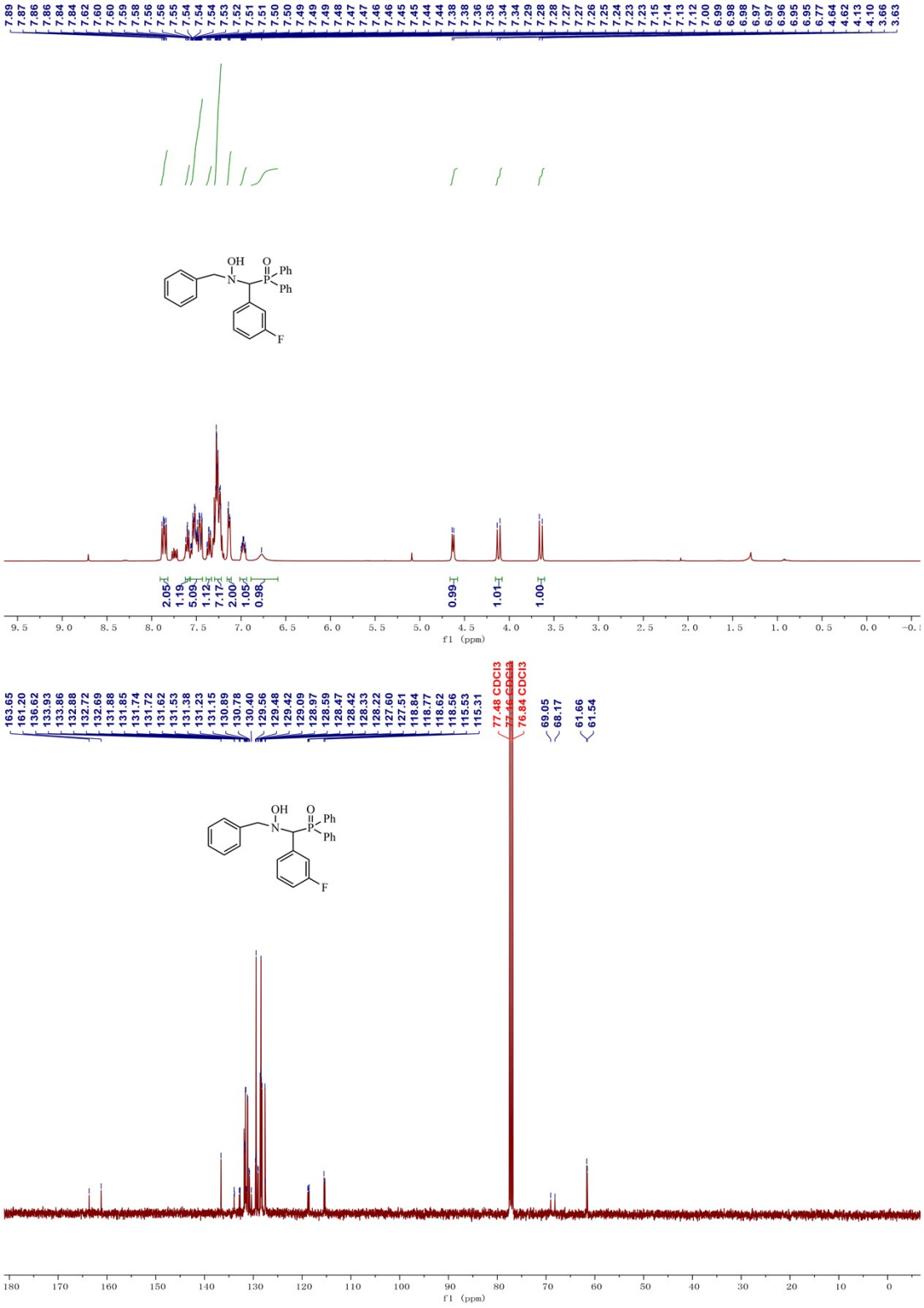
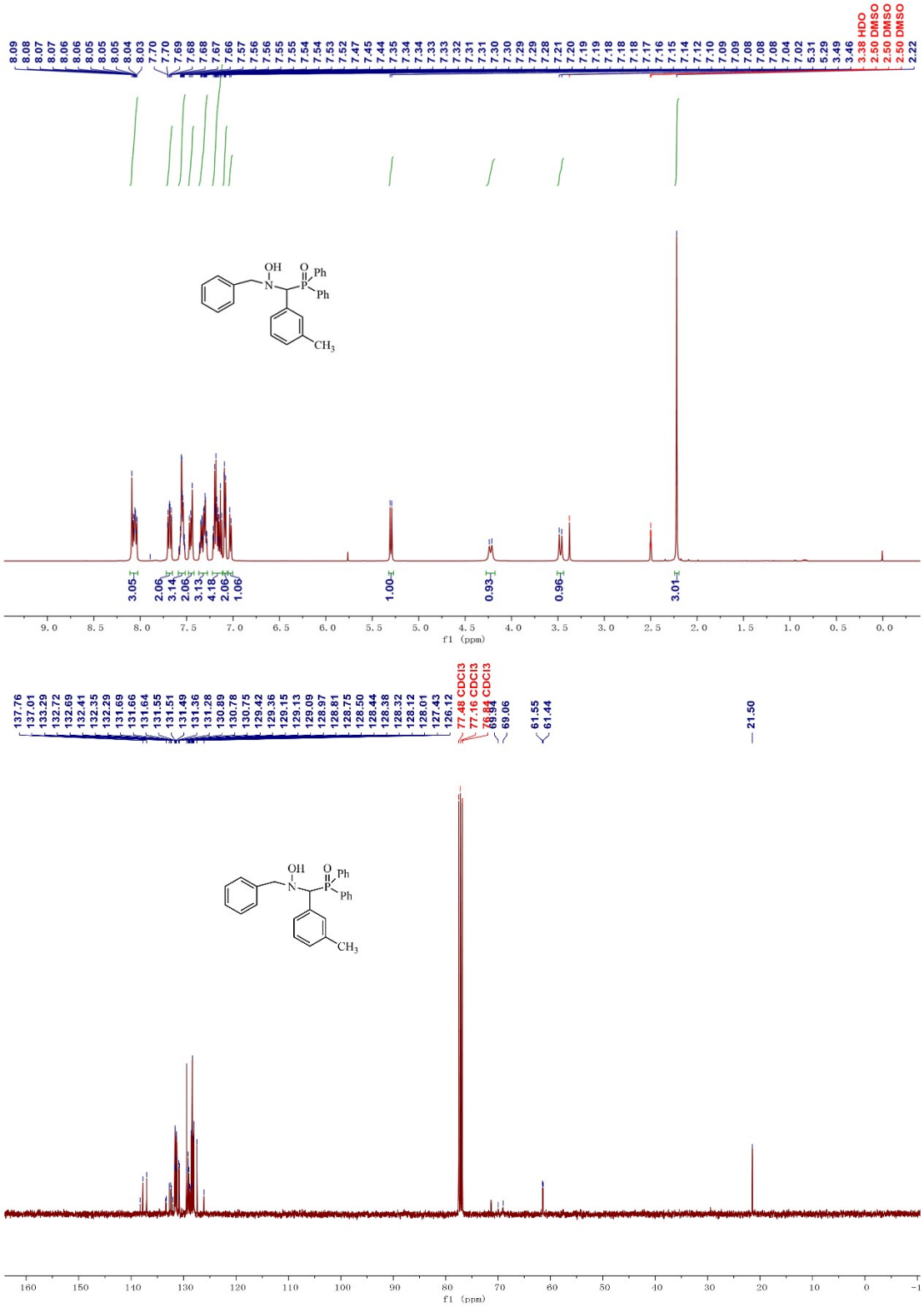
f1 (ppm)

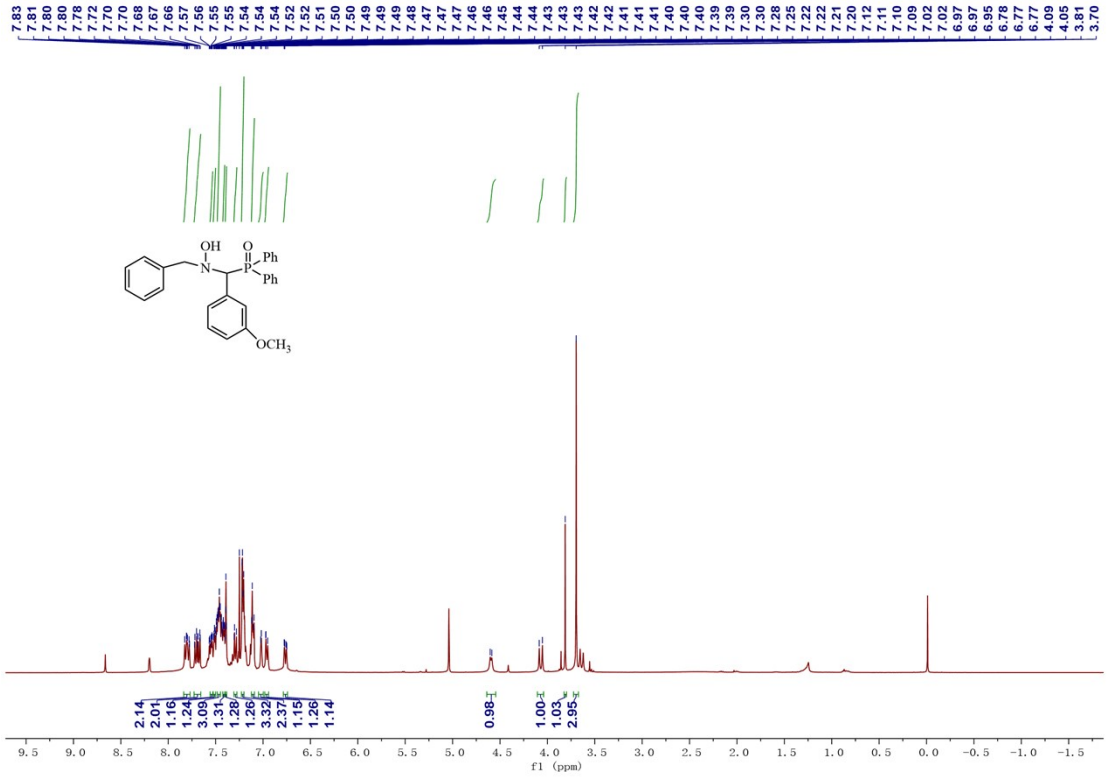
3t

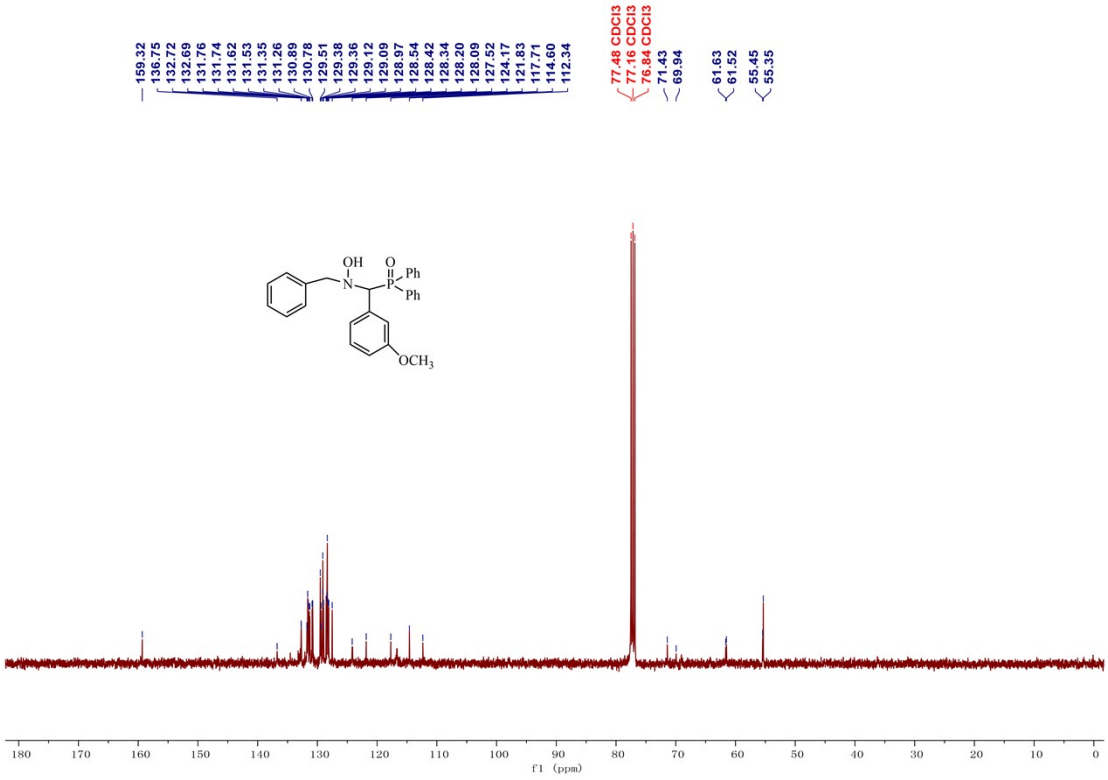

7.89 7.87 7.86 7.86 7.84 7.84 7.62 7.60 7.59 7.58 7.56 7.56 7.55 7.54 7.54 7.54 7.53 7.52 7.51 7.51 7.50 7.50 7.49 7.49 7.49 7.48 7.47 7.47 7.46 7.46 7.45 7.45 7.44 7.44 7.38 7.38 7.36 7.36 7.34 7.34 7.29 7.28 7.28 7.27 7.27 7.26 7.25 7.24 7.23 7.23 7.15 7.14 7.13 7.12 7.00 6.99 6.98 6.98 6.97 6.97 6.96 6.95 6.95 6.77 4.64 4.62 4.13 4.10 3.66 3.63
OH
O
N
P
Ph
Ph
F
2.05 1.19 5.09 1.12 7.17 2.00 1.05 0.98 0.99 1.01 1.00
f1 (ppm)
163.65 161.20 136.62 133.93 133.86 132.88 132.72 132.69 131.88 131.85 131.74 131.72 131.62 131.53 131.38 131.23 131.15 130.89 130.78 130.40 129.56 129.48 129.42 129.09 128.97 128.59 128.47 128.42 128.33 128.22 127.60 127.51 118.84 118.77 118.62 118.56 115.53 115.31 77.48 CDCl3 77.16 CDCl3 76.84 CDCl3 69.05 68.17 61.66 61.54
f1 (ppm)

3u

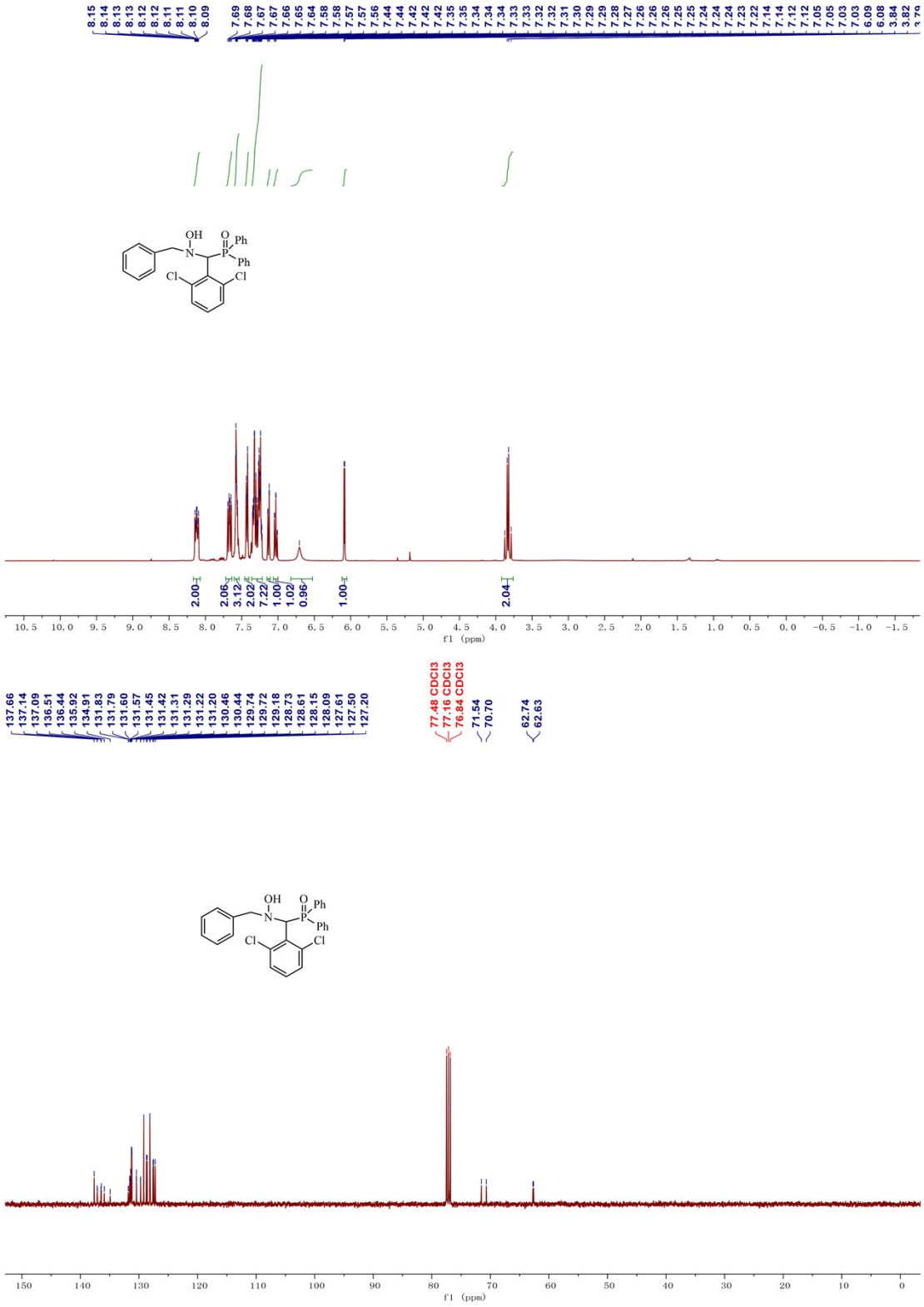

8.09 8.08 8.07 8.07 8.06 8.06 8.05 8.05 8.05 8.04 8.03 7.70 7.70 7.69 7.68 7.68 7.67 7.66 7.57 7.56 7.56 7.55 7.55 7.54 7.54 7.53 7.52 7.47 7.45 7.44 7.35 7.34 7.34 7.33 7.33 7.32 7.31 7.31 7.30 7.30 7.29 7.29 7.28 7.21 7.20 7.19 7.19 7.18 7.18 7.18 7.17 7.16 7.15 7.14 7.12 7.10 7.09 7.09 7.08 7.08 7.04 7.02 5.31 5.29 3.49 3.46 3.38 HDO 2.50 DMSO 2.50 DMSO 2.50 DMSO 2.22
OH
N
O
P
Ph
Ph
CH3
3.05 2.06 3.14 2.06 3.15 4.18 2.06 1.06 1.00 0.93 0.96 3.01
f1 (ppm)
137.76 137.01 133.29 132.72 132.69 132.41 132.35 132.29 131.69 131.66 131.64 131.55 131.51 131.49 131.36 131.28 130.89 130.78 130.75 129.42 129.36 129.15 129.13 129.09 128.97 128.81 128.75 128.50 128.44 128.38 128.32 128.12 128.01 127.43 126.12 77.48 CDCl3 77.16 CDCl3 76.84 CDCl3 69.94 69.06 61.55 61.44 21.50
f1 (ppm)

3v

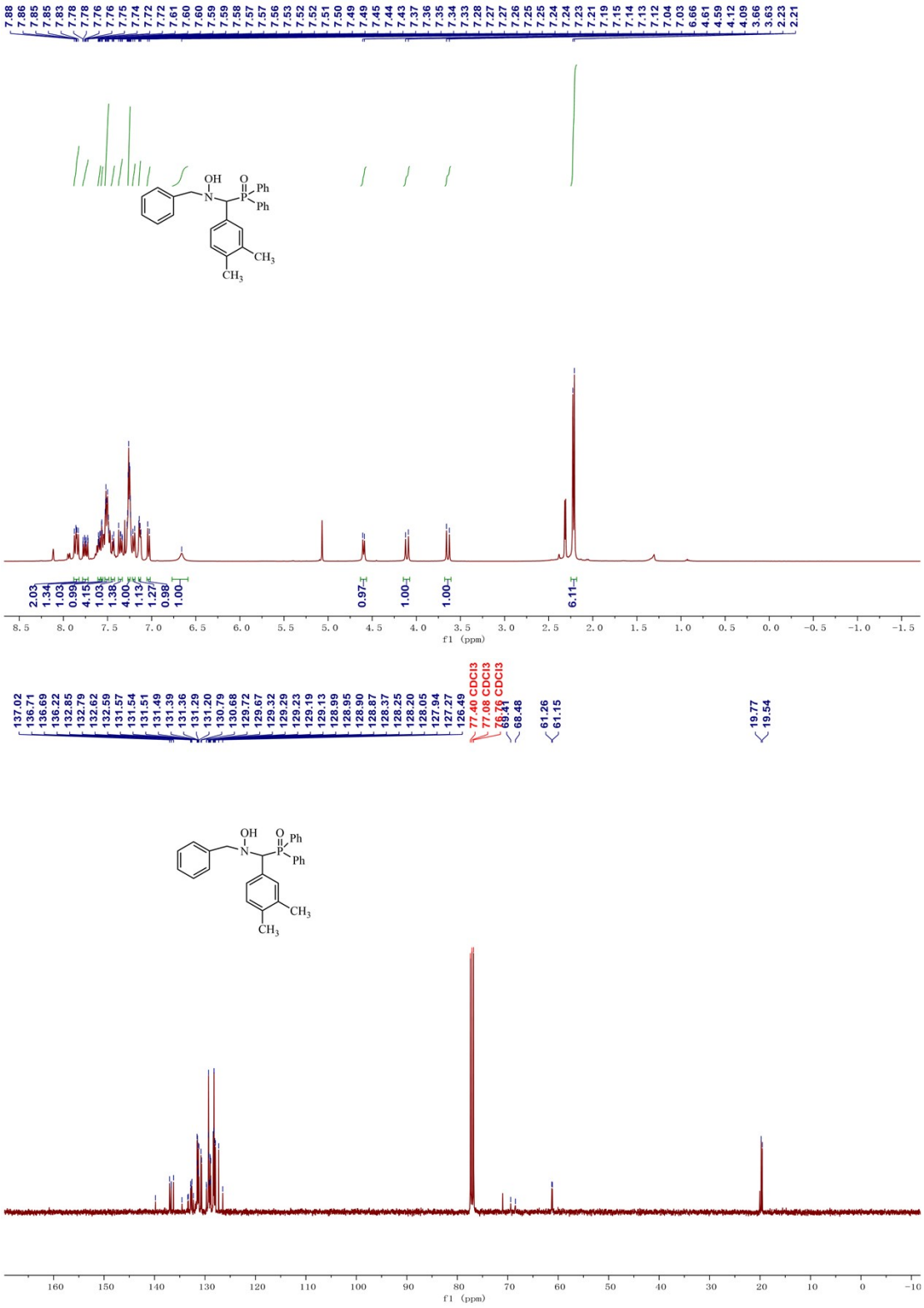

7.83 7.81 7.80 7.80 7.78 7.72 7.70 7.68 7.67 7.66 7.57 7.56 7.55 7.55 7.54 7.54 7.54 7.52 7.52 7.51 7.50 7.50 7.49 7.49 7.49 7.48 7.47 7.47 7.47 7.46 7.46 7.45 7.44 7.44 7.43 7.43 7.42 7.42 7.41 7.41 7.41 7.40 7.40 7.40 7.39 7.39 7.30 7.28 7.25 7.22 7.22 7.21 7.20 7.12 7.11 7.10 7.09 7.02 7.02 6.97 6.97 6.95 6.78 6.77 6.77 4.09 4.05 3.81 3.70
OH
O
Ph
Ph
N
P
OCH3
2.14 2.01 1.16 1.24 3.09 1.31 1.28 1.26 3.32 2.37 1.15 1.26 1.14
0.98
1.00 1.03 2.95
f1 (ppm)


159.32 136.75 132.72 132.69 131.76 131.74 131.62 131.53 131.35 131.26 130.89 130.78 129.51 129.38 129.36 129.12 129.09 128.97 128.54 128.42 128.34 128.20 128.09 127.52 124.17 121.83 117.71 114.60 112.34
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
71.43
69.94
61.63
61.52
55.45
55.35
OH
O
Ph
Ph
N
P
OCH3
f1 (ppm)

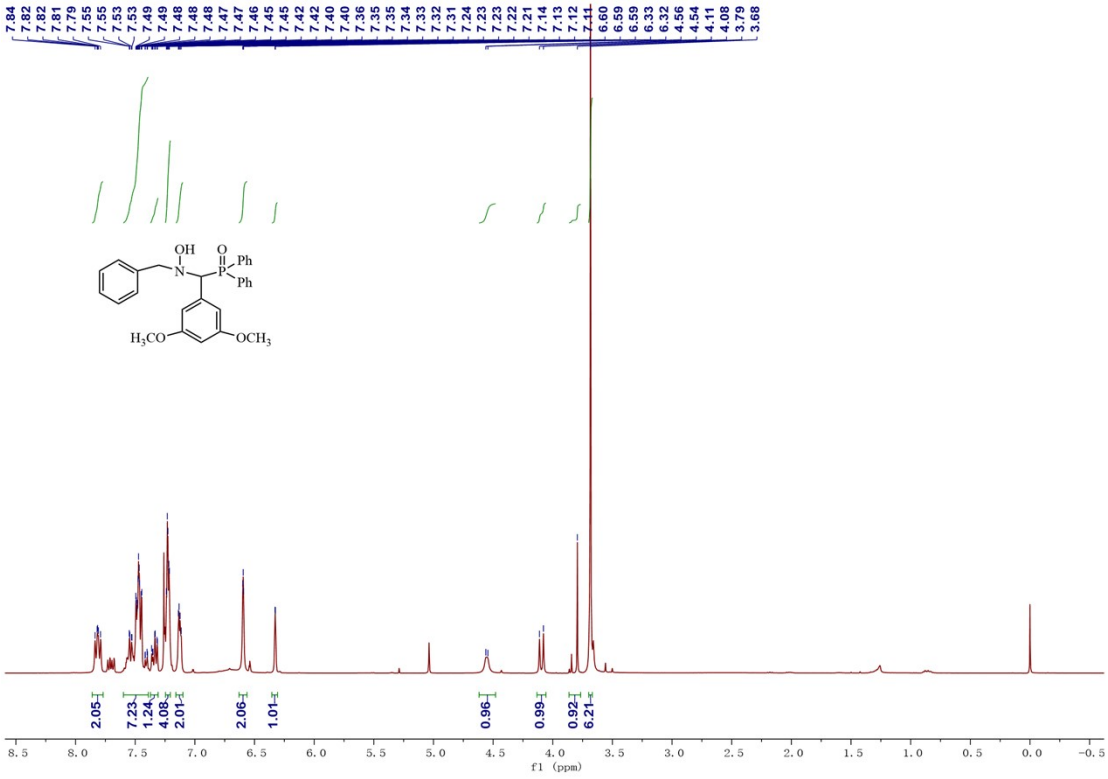
3w

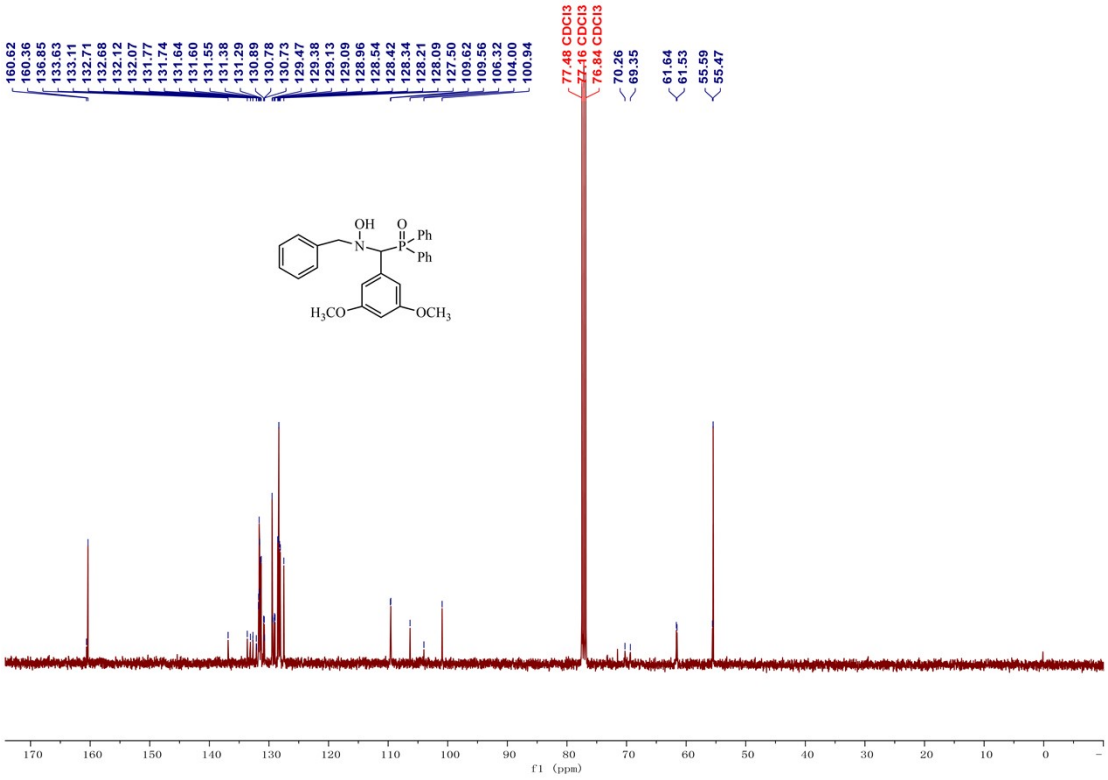

8.15 8.14 8.13 8.13 8.12 8.12 8.11 8.11 8.10 8.09
7.69 7.68 7.67 7.67 7.66 7.65 7.64 7.58 7.58 7.57 7.57 7.56 7.44 7.44 7.42 7.42 7.35 7.35 7.34 7.34 7.33 7.33 7.32 7.31 7.30 7.29 7.29 7.28 7.27 7.26 7.26 7.25 7.25 7.24 7.24 7.24 7.23 7.22 7.14 7.14 7.12 7.12 7.05 7.05 7.03 7.03 6.09 6.08 3.84 3.82
OH
N
O
P
Ph
Ph
Cl
Cl
2.00 2.06 3.12 2.02 7.22 1.00 1.02 0.96 1.00 2.04
f1 (ppm)
137.66 137.14 137.09 136.51 136.44 135.92 134.91 131.83 131.79 131.60 131.57 131.45 131.42 131.31 131.29 131.22 131.20 130.46 130.44 129.74 129.72 129.18 128.73 128.61 128.15 128.09 127.61 127.50 127.20
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
71.54
70.70
62.74
62.63
f1 (ppm)

3x

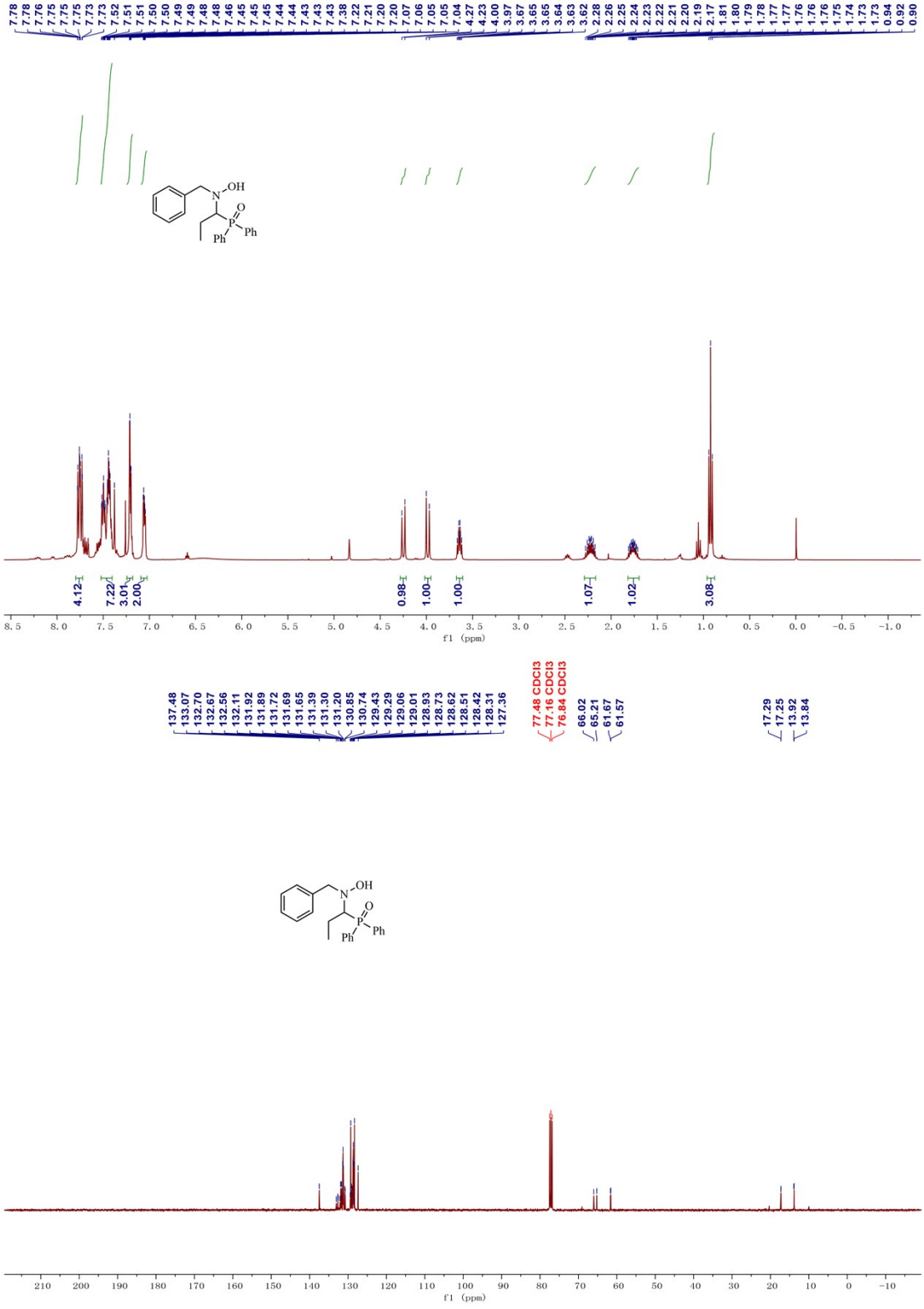

7.88 7.86 7.85 7.85 7.83 7.78 7.78 7.76 7.76 7.75 7.74 7.72 7.72 7.61 7.60 7.60 7.59 7.59 7.58 7.57 7.57 7.56 7.53 7.52 7.52 7.51 7.50 7.49 7.49 7.45 7.44 7.43 7.37 7.36 7.35 7.34 7.33 7.28 7.27 7.27 7.26 7.25 7.25 7.24 7.24 7.23 7.21 7.19 7.15 7.14 7.13 7.12 7.04 7.03 6.66 4.61 4.59 4.12 4.09 3.66 3.63 2.23 2.21
OH
N
O
P
Ph
Ph
CH3
CH3
2.03 1.34 1.03 0.99 4.15 1.03 1.38 4.00 1.13 1.27 0.98 1.00 0.97 1.00 1.00 6.11
f1 (ppm)
137.02 136.71 136.69 136.22 132.85 132.79 132.62 132.59 131.57 131.54 131.51 131.49 131.39 131.36 131.29 131.20 130.79 130.68 129.72 129.67 129.32 129.29 129.23 129.19 129.13 128.99 128.95 128.90 128.87 128.37 128.25 128.20 128.05 127.94 127.27 126.49 77.40 CDCl3 77.08 CDCl3 76.76 CDCl3 69.41 68.48 61.26 61.15 19.77 19.54
f1 (ppm)

3y

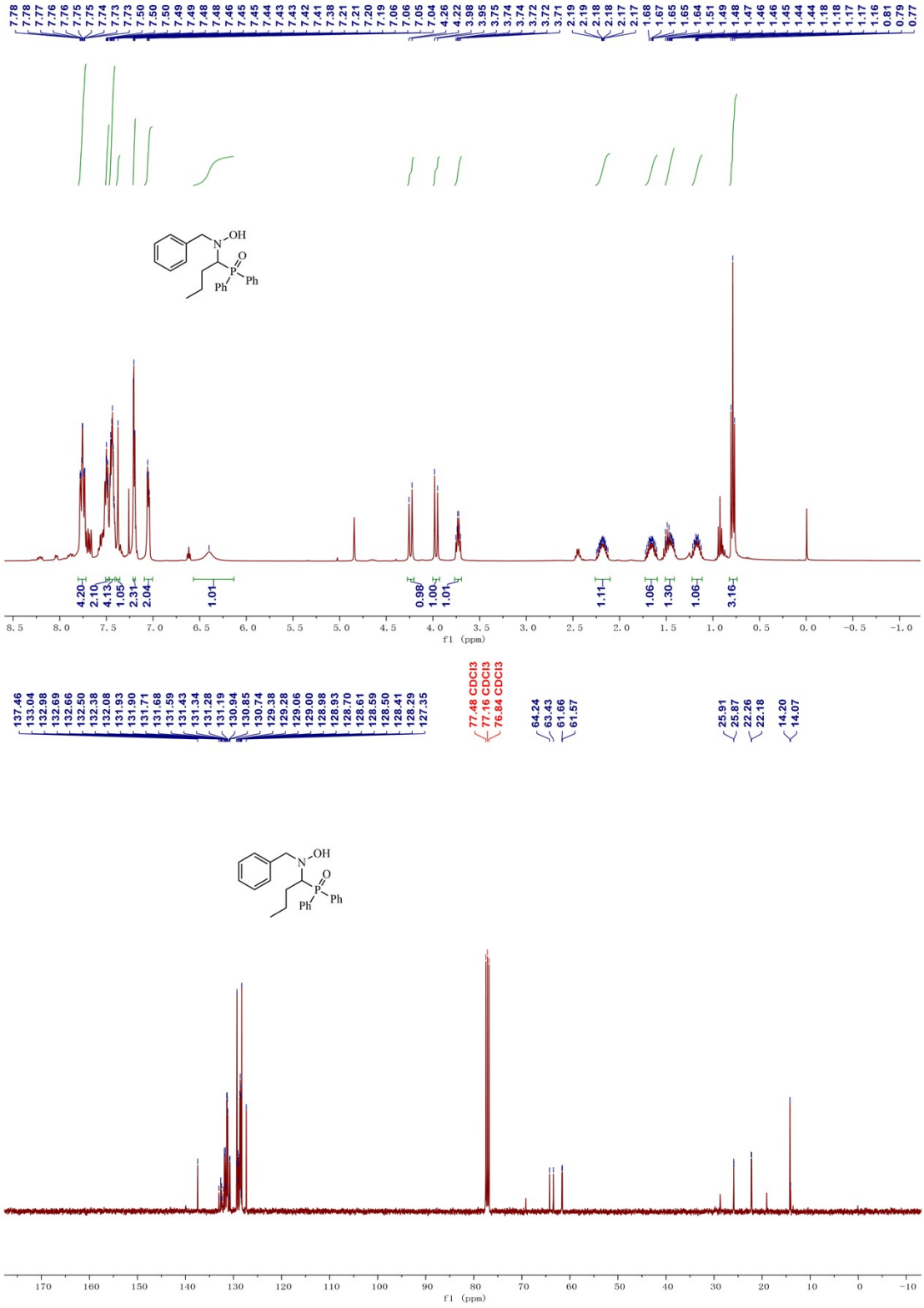

7.84 7.82 7.82 7.81 7.79 7.55 7.55 7.53 7.53 7.49 7.49 7.48 7.48 7.48 7.47 7.47 7.46 7.45 7.45 7.42 7.42 7.40 7.40 7.36 7.35 7.35 7.34 7.33 7.32 7.31 7.24 7.23 7.23 7.22 7.21 7.14 7.13 7.12 7.11 6.60 6.59 6.59 6.33 6.32 4.56 4.54 4.11 4.08 3.79 3.68
OH
O
Ph
Ph
N
P
H3CO
OCH3
2.05 7.23 1.24 4.08 2.01 2.06 1.01 0.96 0.99 0.92 6.21
f1 (ppm)


160.62 160.36 136.85 133.63 133.11 132.71 132.68 132.12 132.07 131.77 131.74 131.64 131.60 131.55 131.38 131.29 130.89 130.78 130.73 129.47 129.38 129.13 129.09 128.96 128.54 128.42 128.34 128.21 128.09 127.50 109.62 109.56 106.32 104.00 100.94
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
70.26
69.35
61.64
61.53
55.59
55.47
OH
O
Ph
Ph
N
P
H3CO
OCH3
f1 (ppm)

3z


7.78 7.78 7.76 7.75 7.75 7.73 7.73 7.52 7.51 7.51 7.50 7.50 7.49 7.49 7.48 7.48 7.46 7.45 7.45 7.45 7.44 7.44 7.43 7.43 7.43 7.38 7.22 7.21 7.20 7.20 7.07 7.06 7.05 7.05 7.04 4.27 4.23 4.00 3.97 3.67 3.65 3.65 3.64 3.63 3.62 2.28 2.26 2.25 2.24 2.23 2.22 2.21 2.20 2.19 2.17 1.81 1.80 1.79 1.78 1.77 1.77 1.76 1.76 1.76 1.75 1.74 1.73 1.73 0.94 0.92 0.90
OH
N
O
P
Ph
Ph
4.12
7.22
3.01
2.00
0.98
1.00
1.00
1.07
1.02
3.08
f1 (ppm)
137.48 133.07 132.70 132.67 132.56 132.11 131.92 131.89 131.72 131.69 131.65 131.39 131.30 131.20 130.85 130.74 129.43 129.29 129.06 129.01 128.93 128.73 128.62 128.51 128.42 128.31 127.36
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
66.02 65.21 61.67 61.57
17.29 17.25 13.92 13.84
f1 (ppm)

3aa



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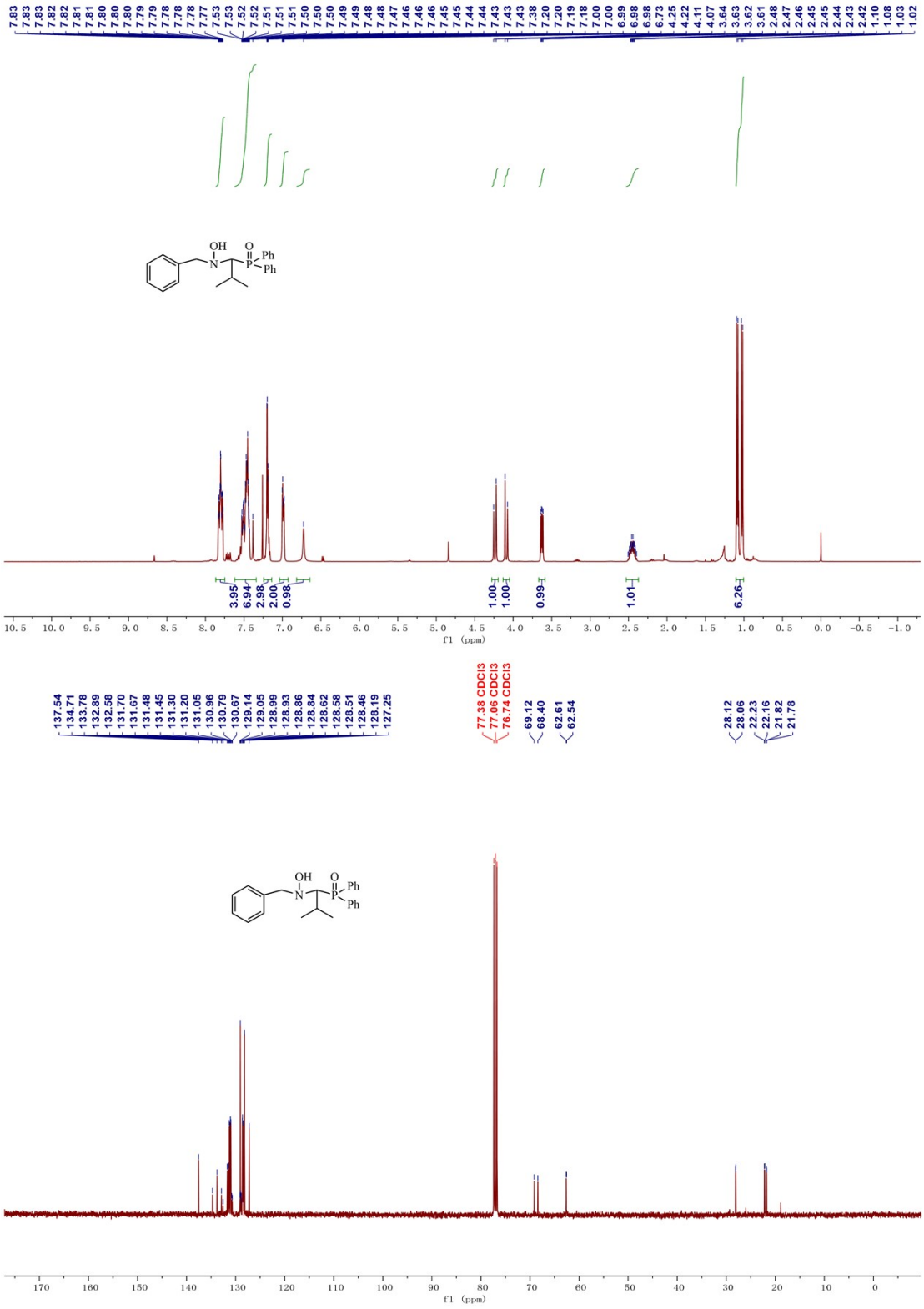
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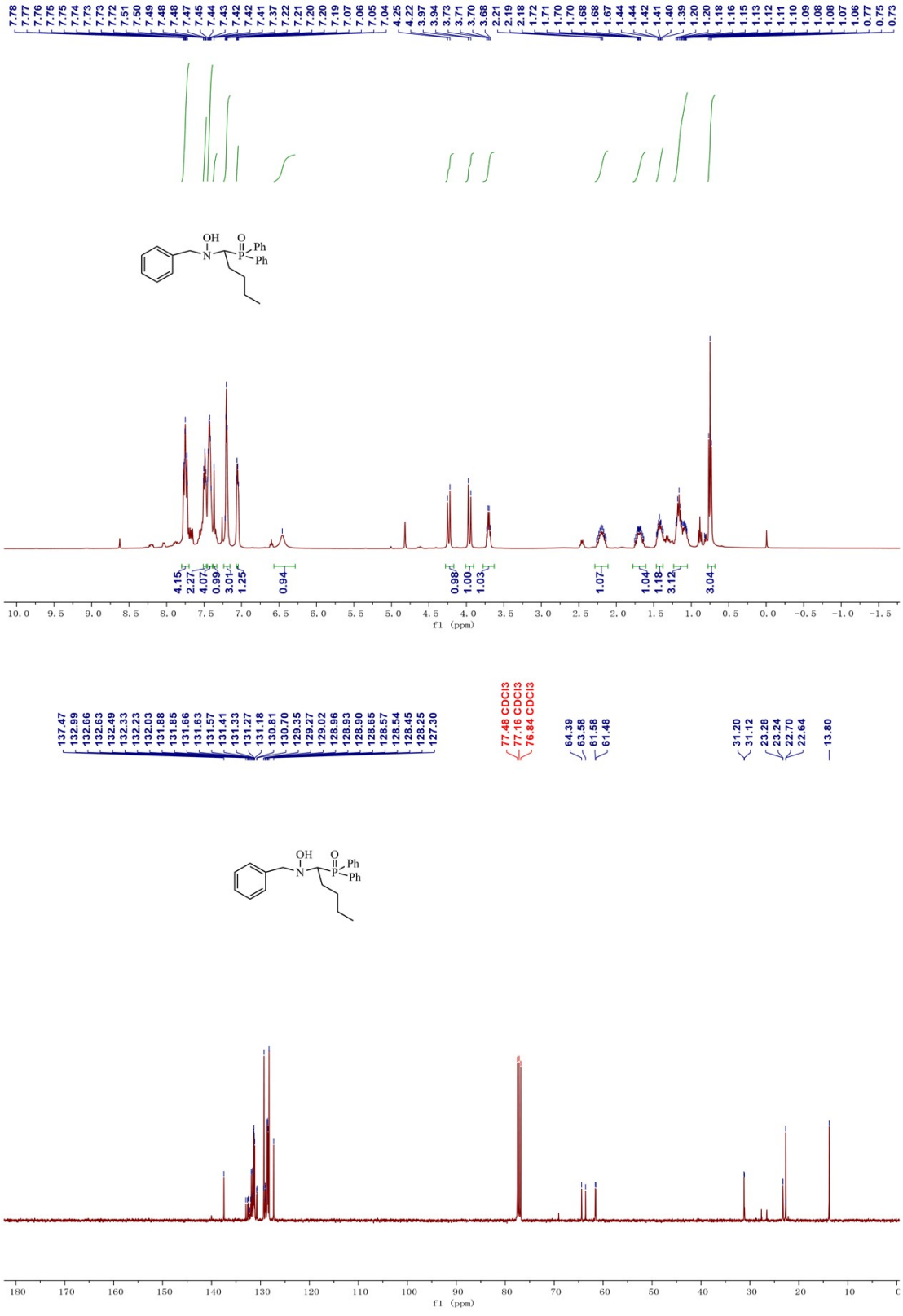
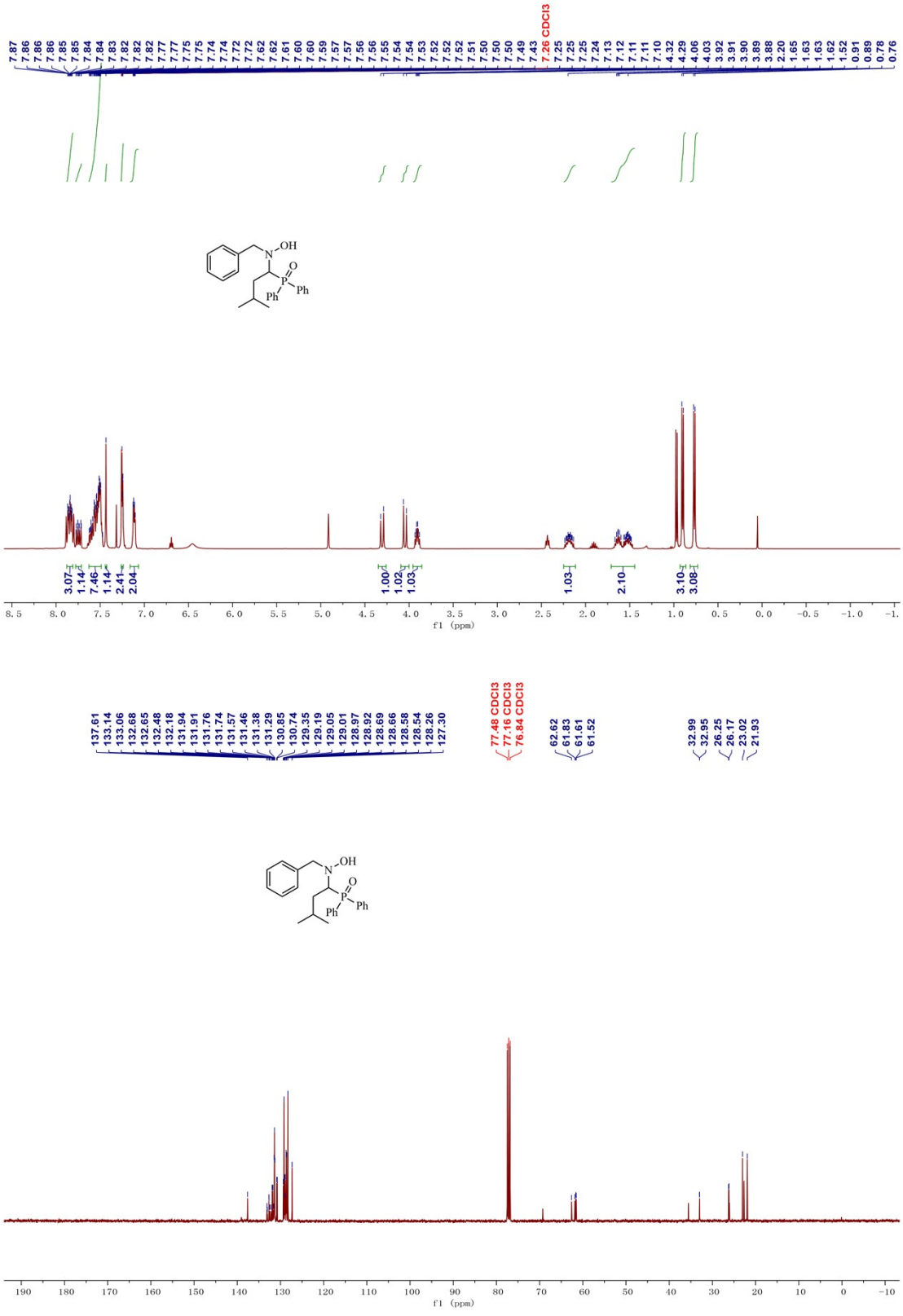
OH
O
Ph
Ph
3.95 6.94 2.98 2.00 0.98 1.00 1.00 0.99 1.01 6.26
f1 (ppm)
137.54 134.71 133.78 132.89 132.58 131.70 131.67 131.48 131.45 131.30 131.20 131.05 130.96 130.79 130.67 129.14 129.05 128.99 128.93 128.86 128.84 128.62 128.58 128.51 128.46 128.19 127.25
77.38 CDCl3
77.06 CDCl3
76.74 CDCl3
69.12 68.40 62.61 62.54
28.12 28.06 22.23 22.16 21.82 21.78
f1 (ppm)

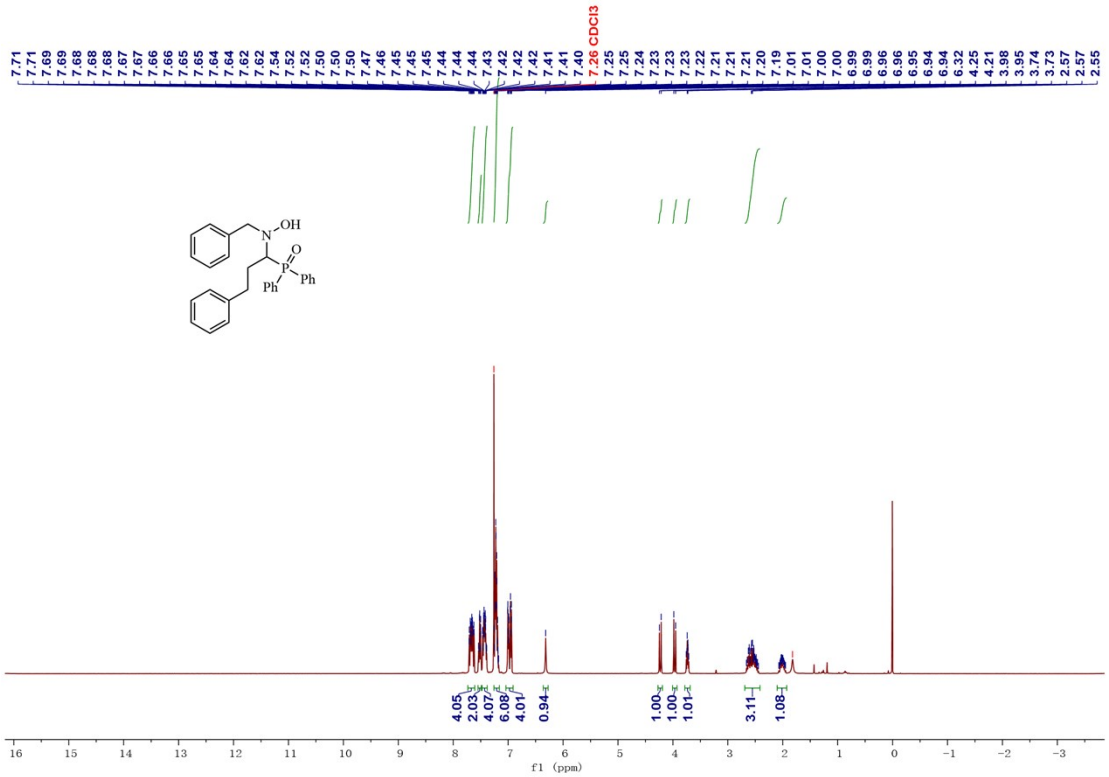
3ac


7.78 7.77 7.76 7.75 7.74 7.73 7.72 7.51 7.50 7.49 7.48 7.47 7.45 7.44 7.43 7.42 7.41 7.37 7.22 7.21 7.20 7.19 7.07 7.06 7.05 7.04 4.25 4.22 3.97 3.94 3.73 3.71 3.70 3.68 2.21 2.19 2.18 1.72 1.71 1.70 1.70 1.68 1.67 1.44 1.42 1.41 1.40 1.39 1.20 1.20 1.18 1.16 1.15 1.13 1.12 1.11 1.10 1.09 1.08 1.08 1.07 1.06 0.77 0.75 0.73
OH
N
O
P
Ph
Ph
4.15 2.27 4.07 0.99 3.01 1.25 0.94 0.98 1.00 1.03 1.07 1.04 1.18 3.12 3.04
f1 (ppm)
137.47 132.99 132.66 132.63 132.49 132.33 132.23 132.03 131.88 131.85 131.66 131.63 131.57 131.41 131.33 131.27 131.18 130.81 130.70 129.35 129.27 129.02 128.96 128.93 128.90 128.65 128.57 128.54 128.45 128.25 127.30
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
64.39 63.58 61.56 61.48
31.20 31.12 23.28 23.24 22.70 22.64 13.80
f1 (ppm)

3ad


7.87 7.86 7.86 7.86 7.85 7.85 7.84 7.84 7.83 7.82 7.82 7.82 7.77 7.77 7.75 7.75 7.74 7.74 7.72 7.72 7.62 7.62 7.61 7.60 7.60 7.59 7.57 7.57 7.56 7.56 7.55 7.54 7.54 7.53 7.52 7.52 7.52 7.51 7.50 7.50 7.50 7.49 7.43 7.26 CDCl3 7.25 7.25 7.24 7.13 7.12 7.11 7.10 4.32 4.29 4.06 4.03 3.92 3.91 3.90 3.89 3.88 2.20 1.65 1.63 1.63 1.62 1.52 0.91 0.89 0.78 0.76
OH
N
O
P
Ph
Ph
3.07 1.14 7.46 1.14 2.41 2.04 1.00 1.02 1.03 1.03 2.10 3.10 3.08
f1 (ppm)
137.61 133.14 133.06 132.68 132.65 132.48 132.18 131.94 131.91 131.76 131.74 131.57 131.46 131.38 131.29 130.85 130.74 129.35 129.19 129.05 129.01 128.97 128.92 128.69 128.66 128.58 128.54 128.26 127.30
77.48 CDCl3 77.16 CDCl3 76.84 CDCl3
62.62 61.83 61.61 61.52
32.99 32.95 26.25 26.17 23.02 21.93
f1 (ppm)

3ae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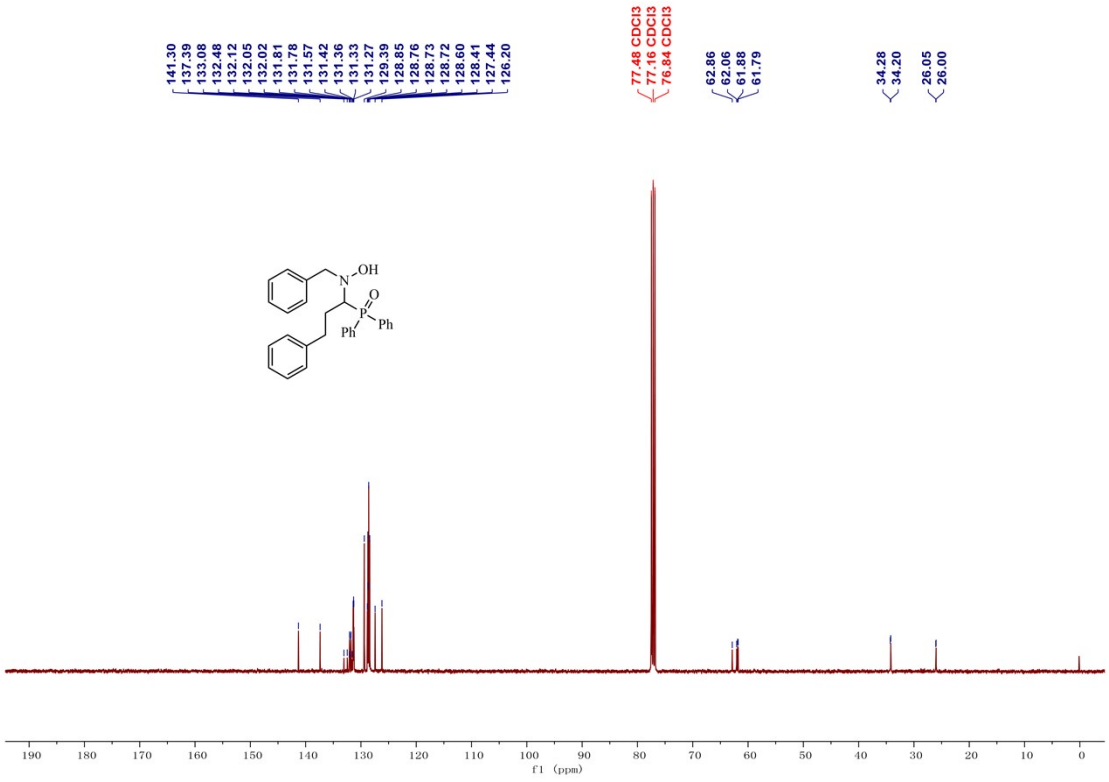
OH
N
O
P
Ph
Ph
4.05 2.03 4.07 6.08 4.01 0.94 1.00 1.00 1.01 3.11 1.08
f1 (ppm)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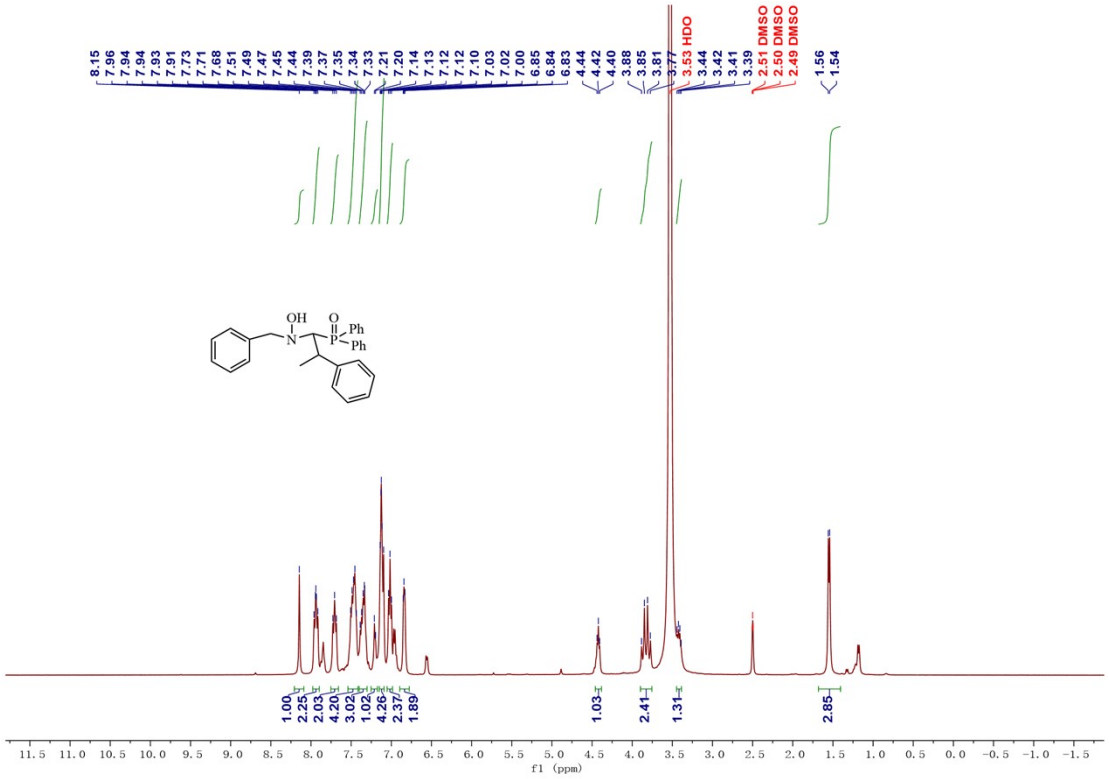
77.48 CDCl3 77.16 CDCl3 76.84 CDCl3
62.86 62.06 61.88 61.79
34.28 34.20
26.05 26.00
OH
N
O
P
Ph
Ph
f1 (ppm)

3af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7.94
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7.91
7.73
7.71
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7.51
7.49
7.47
7.45
7.44
7.39
7.37
7.35
7.34
7.33
7.21
7.20
7.14
7.13
7.12
7.12
7.10
7.03
7.02
7.00
6.85
6.84
6.83
4.44
4.42
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3.81
3.77
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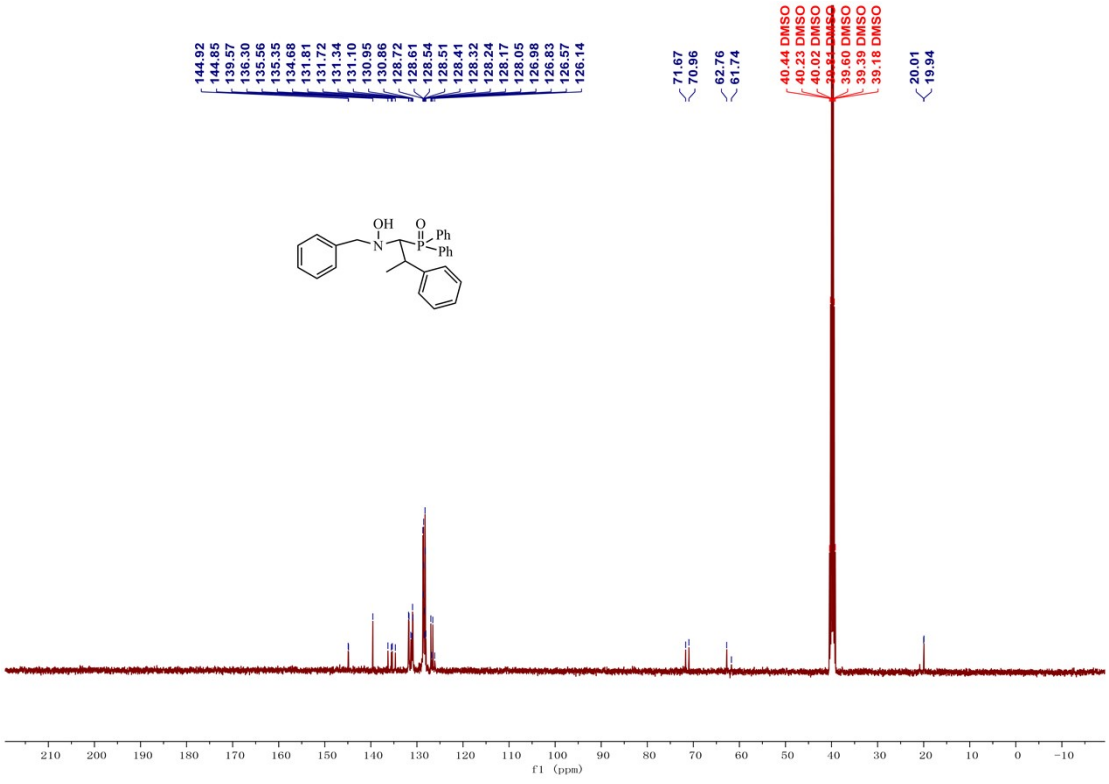
3.44
3.42
3.41
3.39
2.51 DMSO
2.50 DMSO
2.49 DMSO
1.56
1.54
OH
O
N
P
Ph
Ph
1.00
2.25
2.03
4.20
3.02
1.02
4.26
2.37
1.89
1.03
2.41
1.31
2.85
f1 (ppm)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144.85
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136.30
135.56
135.35
134.68
131.81
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131.34
131.10
130.95
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128.72
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128.51
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128.32
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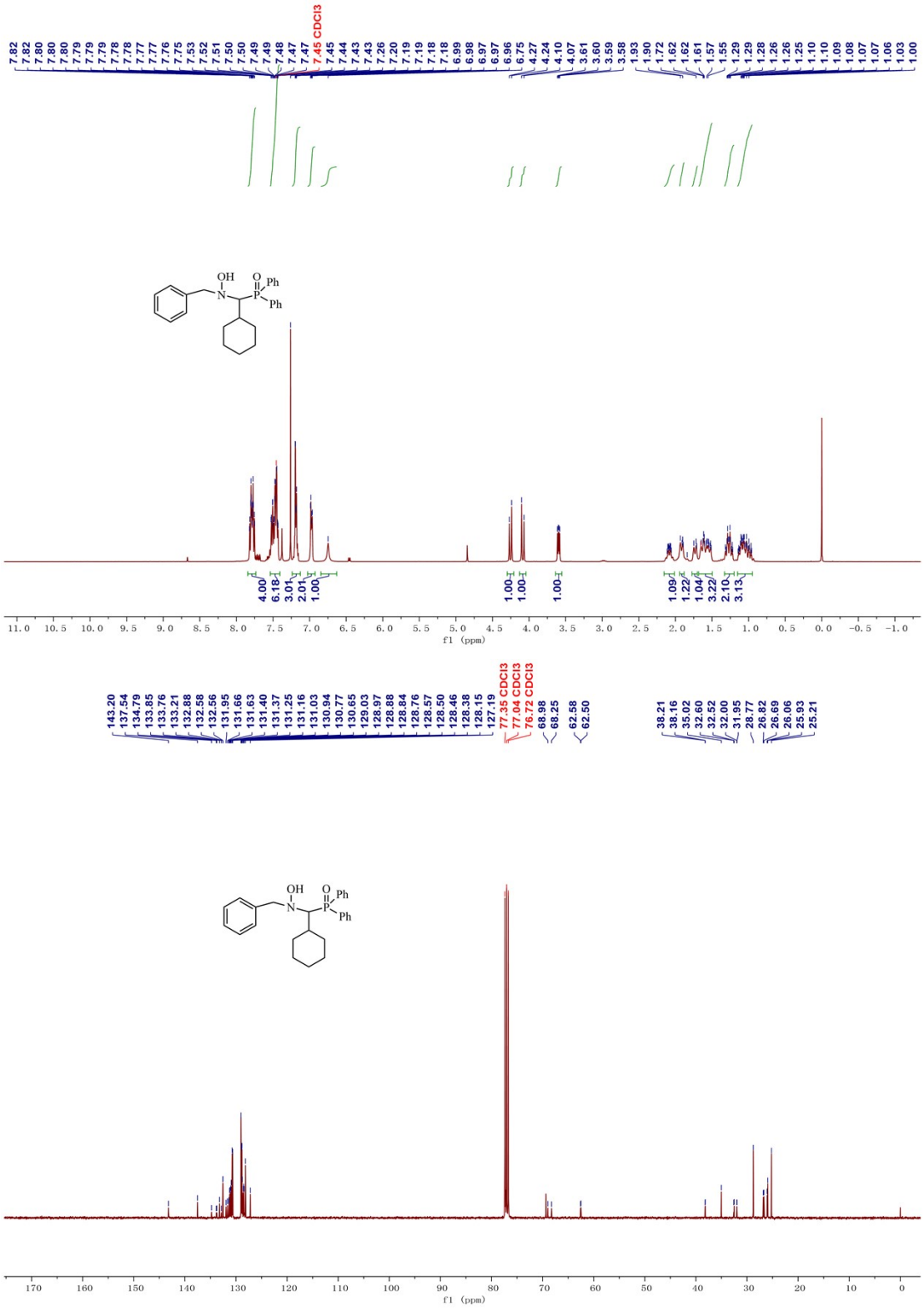
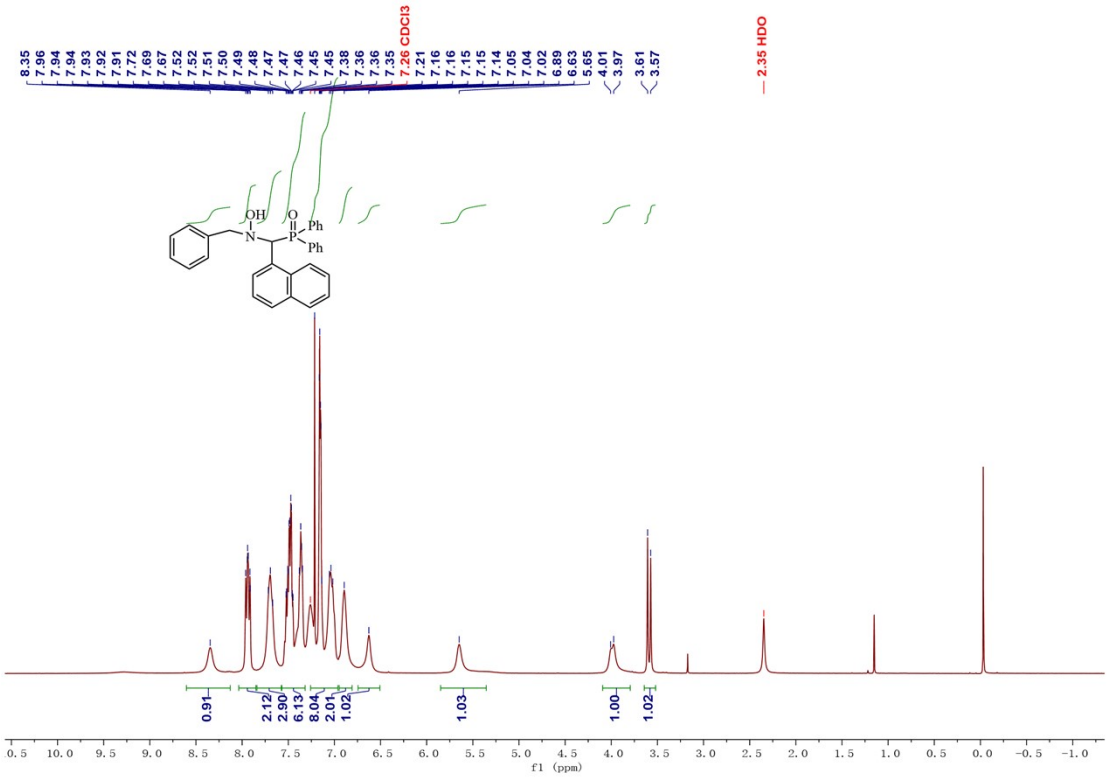
126.14
71.67
70.96
62.76
61.74
40.44 DMSO
40.23 DMSO
40.02 DMSO
39.81 DMSO
39.60 DMSO
39.39 DMSO
39.18 DMSO
20.01
19.94
OH
O
N
P
Ph
Ph
f1 (ppm)

3ag


7.82 7.82 7.80 7.80 7.80 7.79 7.79 7.79 7.78 7.78 7.77 7.77 7.76 7.75 7.53 7.52 7.51 7.50 7.50 7.49 7.49 7.48 7.47 7.47 7.45 CDCl3 7.45 7.44 7.43 7.43 7.26 7.20 7.19 7.19 7.18 7.18 6.99 6.98 6.97 6.97 6.96 6.75 4.27 4.24 4.10 4.07 3.61 3.60 3.59 3.58 1.93 1.90 1.72 1.62 1.62 1.61 1.57 1.55 1.29 1.29 1.28 1.26 1.25 1.10 1.10 1.09 1.08 1.07 1.07 1.06 1.03 1.00
OH
N
O
P
Ph
Ph
4.00 6.18 3.01 2.01 1.00 1.00 1.00 1.00 1.09 1.22 1.04 3.22 2.10 3.13
f1 (ppm)
143.20 137.54 134.79 133.85 133.76 133.21 132.88 132.58 132.56 131.95 131.66 131.63 131.40 131.37 131.25 131.16 131.03 130.94 130.77 130.65 129.03 128.97 128.88 128.84 128.76 128.57 128.50 128.46 128.38 128.15 127.19 77.35 CDCl3 77.04 CDCl3 76.72 CDCl3 68.98 68.25 62.58 62.50 38.21 38.16 35.02 32.60 32.52 32.00 31.95 28.77 26.82 26.69 26.06 25.93 25.21
f1 (ppm)

3ah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7.94
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7.92
7.91
7.72
7.69
7.67
7.52
7.52
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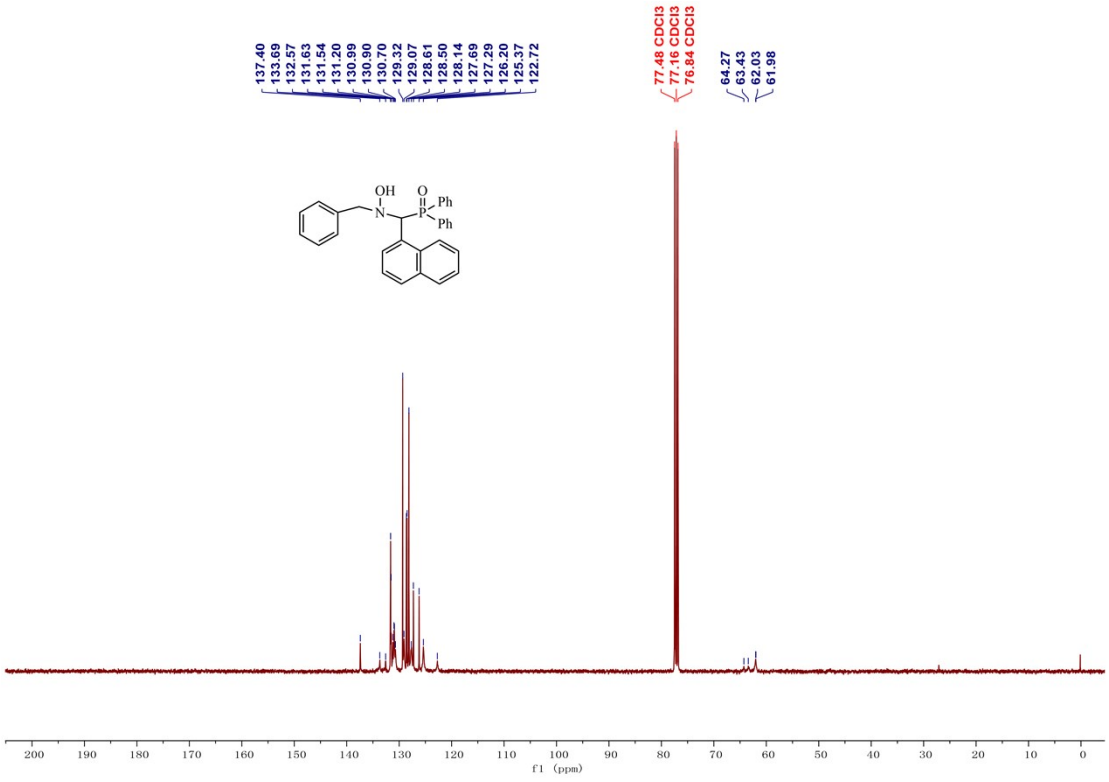
7.50
7.49
7.48
7.47
7.47
7.46
7.45
7.45
7.38
7.36
7.36
7.35
7.26 CDCl3
7.21
7.16
7.16
7.15
7.15
7.14
7.05
7.04
7.02
6.89
6.63
5.65
4.01
3.97
3.61
3.57
2.35 HDO
OH
O
N
P
Ph
Ph
0.91
2.12
2.90
6.13
8.04
2.01
1.02
1.03
1.00
1.02
f1 (ppm)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131.54
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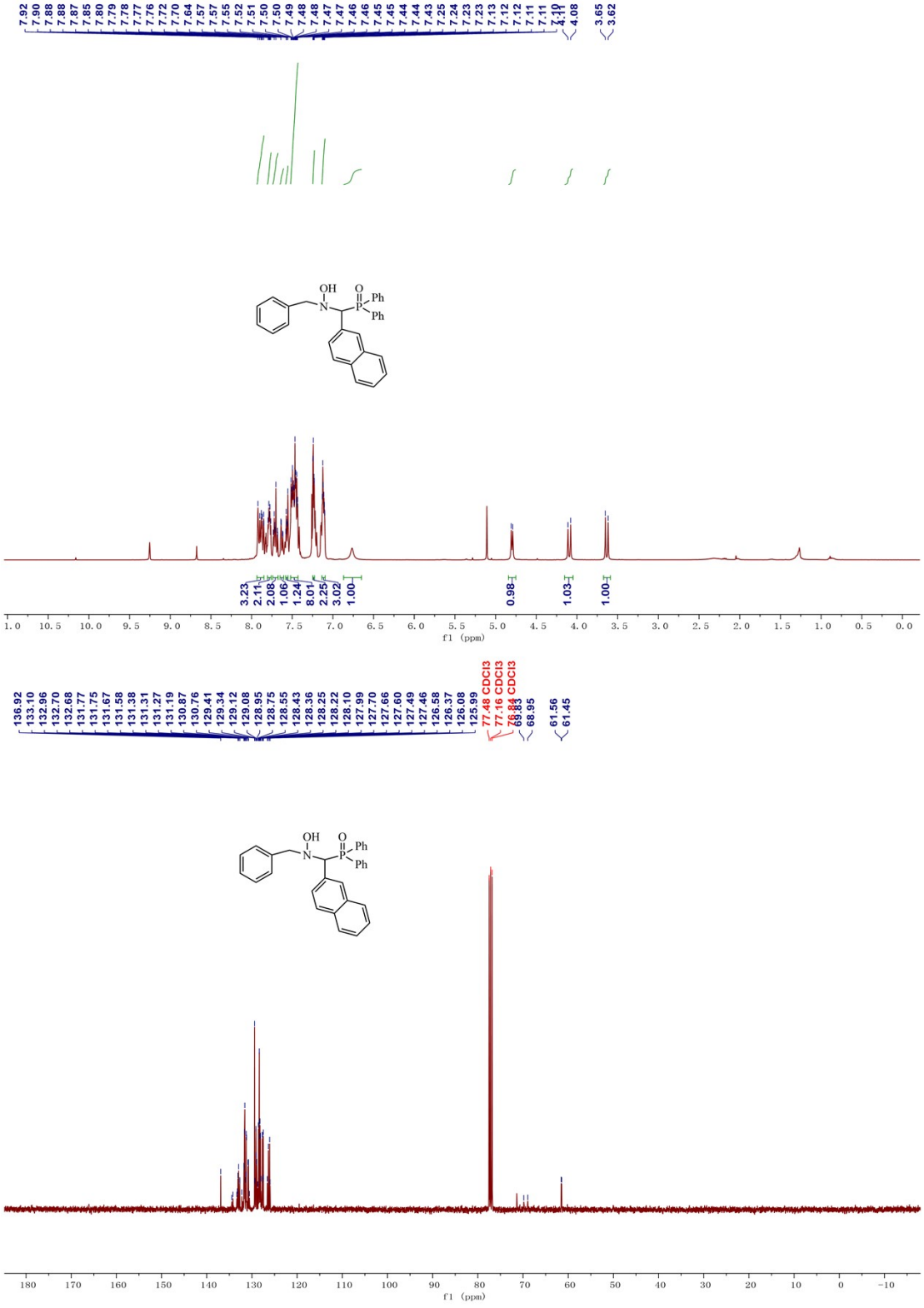
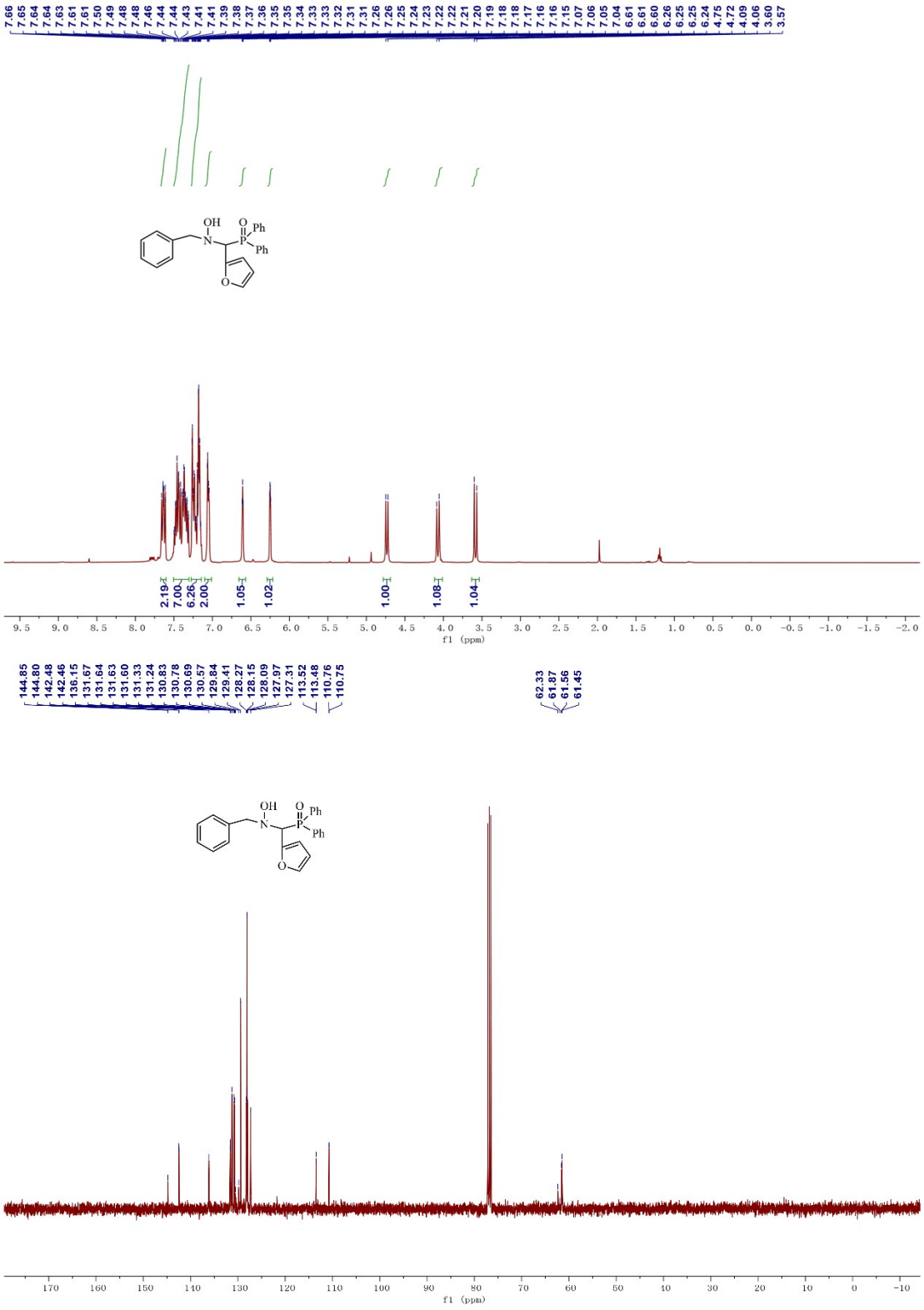
130.99
130.90
130.70
129.32
129.07
128.61
128.50
128.14
127.69
127.29
126.20
125.37
122.72
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
64.27
63.43
62.03
61.98
OH
O
N
P
Ph
Ph
f1 (ppm)

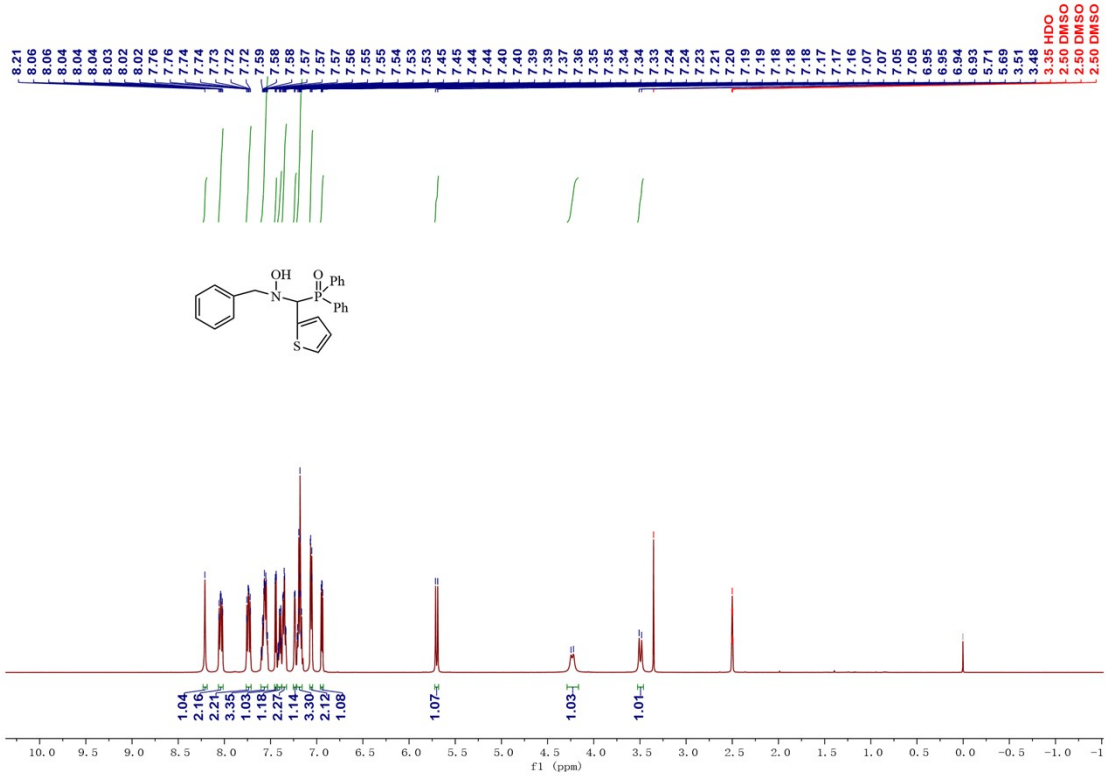
3ai

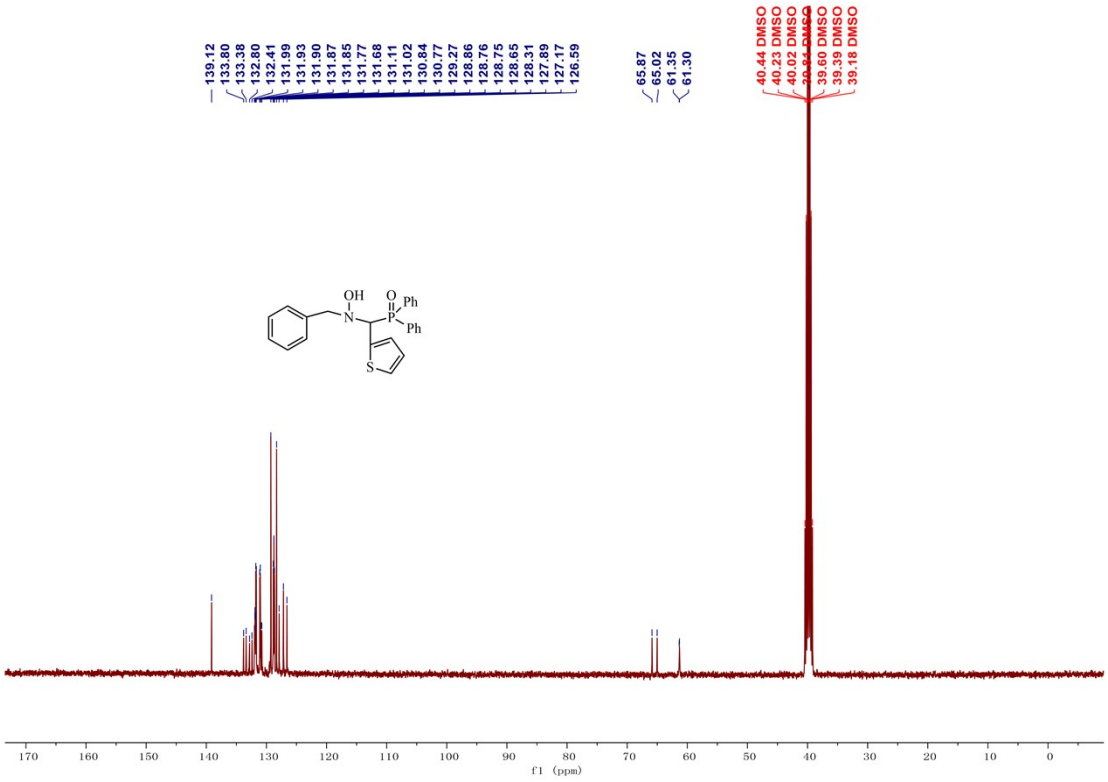

7.92 7.90 7.88 7.88 7.87 7.85 7.80 7.79 7.78 7.77 7.76 7.72 7.70 7.64 7.57 7.57 7.55 7.52 7.51 7.50 7.50 7.49 7.48 7.48 7.47 7.47 7.46 7.46 7.45 7.45 7.44 7.44 7.43 7.25 7.24 7.23 7.23 7.13 7.12 7.12 7.11 7.11 7.10 4.11 4.08 3.65 3.62
OH
O
N
P
Ph
Ph
3.23 2.11 2.08 1.06 1.24 8.01 2.25 3.02 1.00 0.98 1.03 1.00
f1 (ppm)
136.92 133.10 132.96 132.70 132.68 131.77 131.75 131.67 131.58 131.38 131.31 131.27 131.19 130.87 130.76 129.41 129.34 129.12 129.08 128.95 128.75 128.55 128.43 128.36 128.25 128.22 128.10 127.99 127.70 127.66 127.60 127.49 127.46 126.58 126.37 126.08 125.99 77.48 CDCl3 77.16 CDCl3 76.84 CDCl3 69.83 68.95 61.56 61.45
f1 (ppm)

3aJ

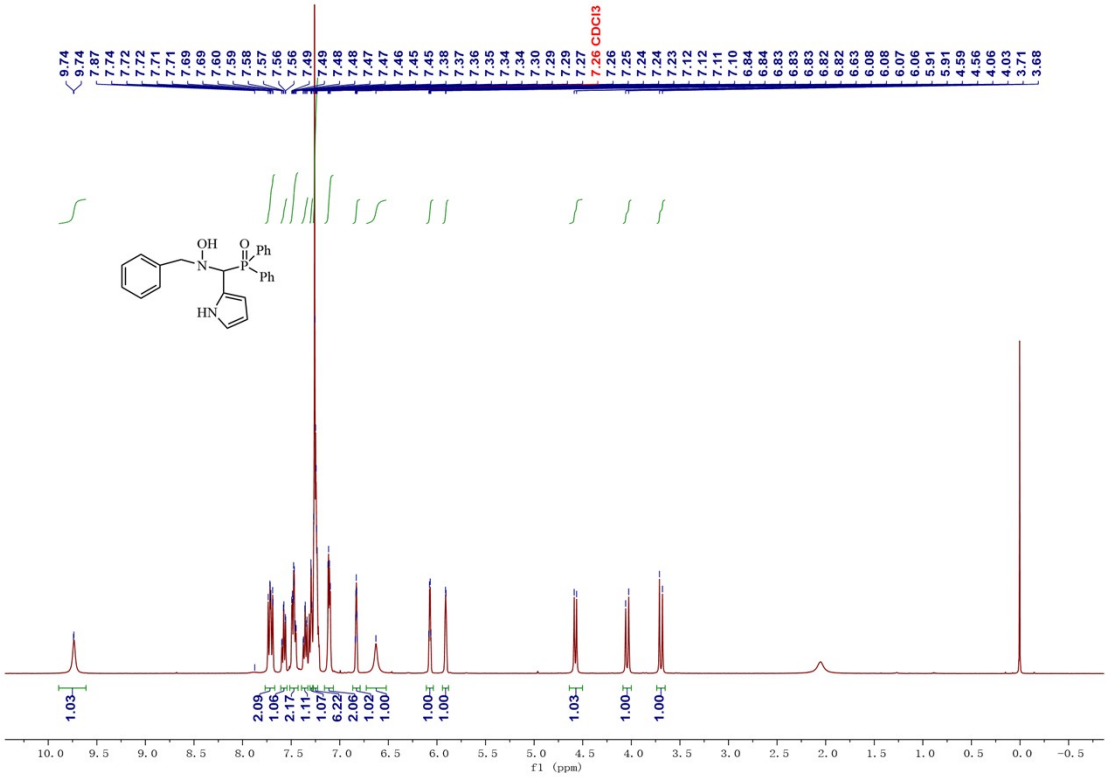

7.66 7.65 7.64 7.64 7.63 7.61 7.61 7.50 7.49 7.48 7.48 7.46 7.44 7.44 7.43 7.41 7.41 7.39 7.38 7.37 7.36 7.35 7.35 7.34 7.33 7.33 7.32 7.31 7.31 7.26 7.25 7.24 7.23 7.22 7.22 7.21 7.20 7.19 7.18 7.18 7.17 7.16 7.16 7.15 7.07 7.06 7.05 7.04 6.61 6.61 6.60 6.26 6.25 6.25 6.24 4.75 4.72 4.09 4.06 3.60 3.57
OH
N
O
P
Ph
Ph
O
2.19 7.00 6.26 2.00 1.05 1.02 1.00 1.08 1.04
f1 (ppm)
144.85 144.80 142.48 142.46 136.15 131.67 131.64 131.63 131.60 131.33 131.24 130.83 130.78 130.69 130.57 129.84 129.41 128.27 128.15 128.09 127.97 127.31 113.52 113.48 110.76 110.75
62.33 61.87 61.56 61.45
f1 (ppm)

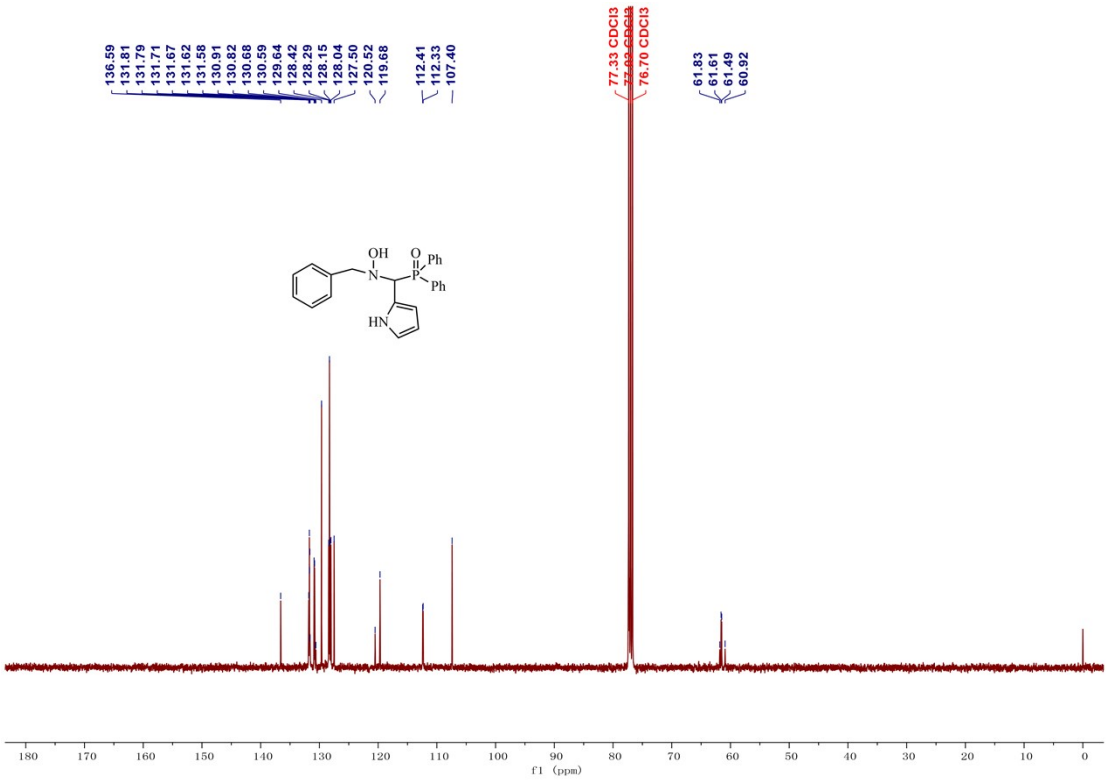
3ak


8.21 8.06 8.06 8.04 8.04 8.04 8.03 8.02 8.02 7.76 7.76 7.74 7.74 7.73 7.72 7.72 7.59 7.58 7.58 7.57 7.57 7.57 7.56 7.55 7.55 7.54 7.53 7.53 7.45 7.45 7.44 7.44 7.40 7.39 7.39 7.37 7.36 7.35 7.35 7.34 7.34 7.33 7.24 7.24 7.23 7.21 7.20 7.19 7.19 7.18 7.18 7.17 7.17 7.16 7.07 7.05 6.95 6.95 6.94 6.93 5.71 5.69 3.51 3.48 3.35 HDO 2.50 DMSO 2.50 DMSO 2.50 DMSO
OH
N
O
P
Ph
Ph
S
1.04 2.16 2.21 3.35 1.03 1.18 2.27 1.14 3.30 2.12 1.08 1.07 1.03 1.01
f1 (ppm)

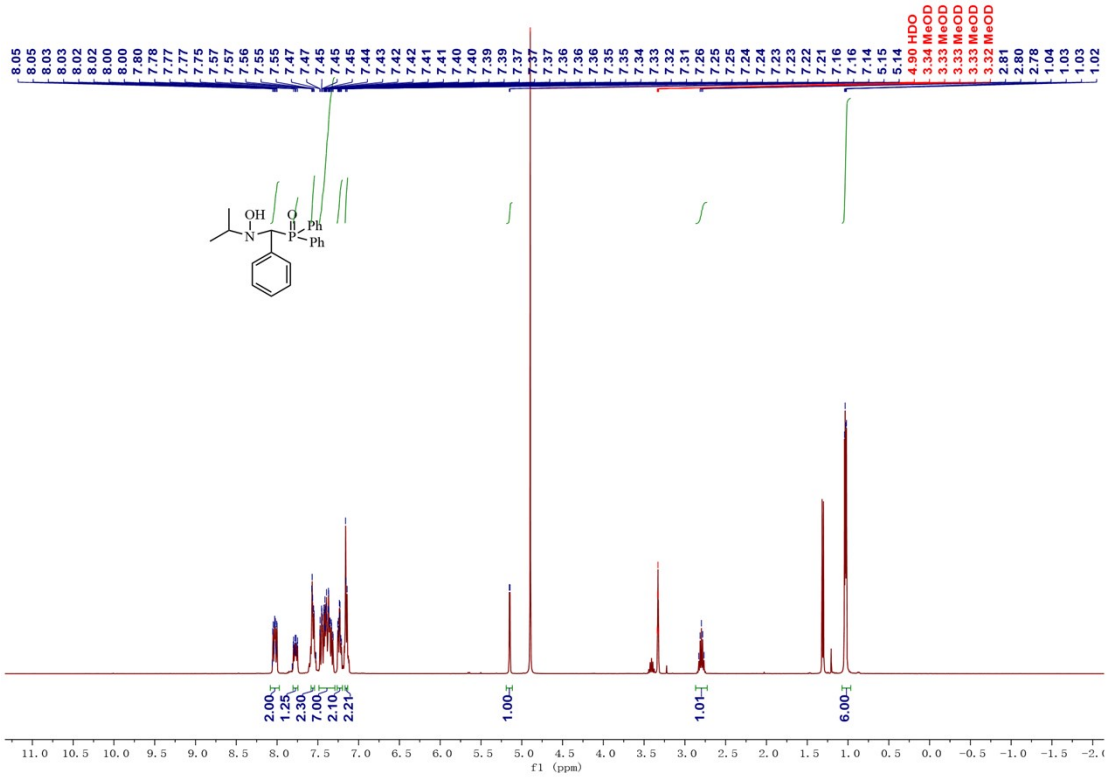

139.12 133.80 133.38 132.80 132.41 131.99 131.93 131.90 131.87 131.85 131.77 131.68 131.11 131.02 130.84 130.77 129.27 128.86 128.76 128.75 128.65 128.31 127.89 127.17 126.59
65.87 65.02 61.35 61.30
40.44 DMSO 40.23 DMSO 40.02 DMSO 39.60 DMSO 39.39 DMSO 39.18 DMSO
OH
N
O
P
Ph
Ph
S
f1 (ppm)

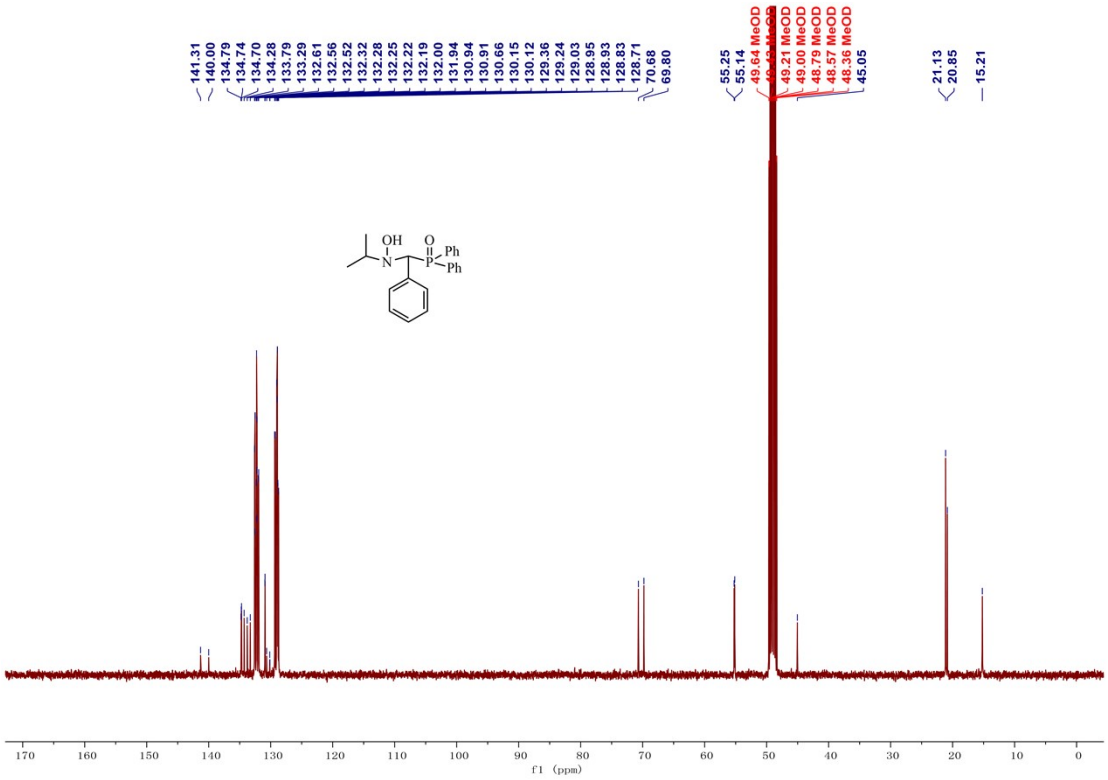
3al


9.74 9.74 7.87 7.74 7.72 7.72 7.71 7.71 7.69 7.69 7.60 7.59 7.58 7.57 7.56 7.56 7.49 7.49 7.48 7.48 7.47 7.47 7.46 7.45 7.45 7.38 7.37 7.36 7.35 7.34 7.34 7.30 7.29 7.29 7.27 7.26 CDCl3 7.26 7.25 7.24 7.24 7.23 7.12 7.12 7.11 7.10 6.84 6.84 6.83 6.83 6.83 6.82 6.82 6.63 6.08 6.08 6.07 6.06 5.91 5.91 4.59 4.56 4.06 4.03 3.71 3.68
OH
N
O
P
Ph
Ph
HN
1.03 2.09 1.06 2.17 1.11 1.07 6.22 2.06 1.02 1.00 1.00 1.00 1.03 1.00 1.00
10.0 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 −0.5
f1 (ppm)

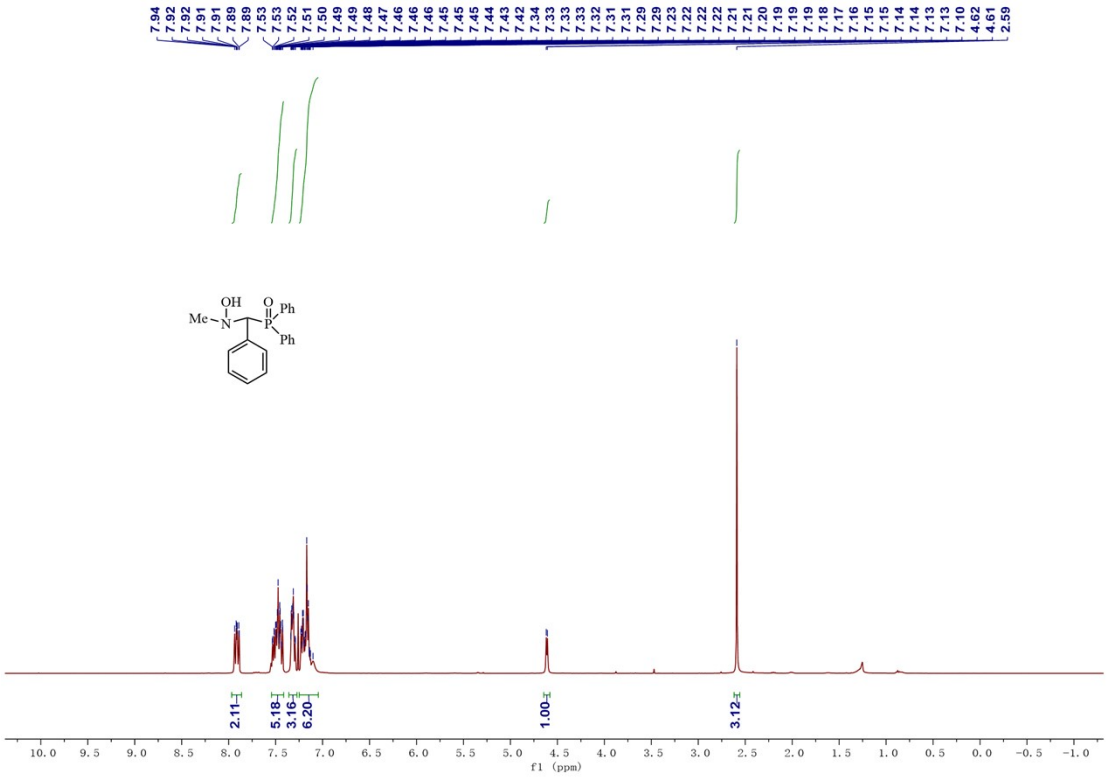

136.59 131.81 131.79 131.71 131.67 131.62 131.58 130.91 130.82 130.68 130.59 129.64 128.42 128.29 128.15 128.04 127.50 120.52 119.68 112.41 112.33 107.40
77.33 CDCl3 76.70 CDCl3
61.83 61.61 61.49 60.92
OH
N
O
P
Ph
Ph
HN
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

3am


8.05 8.05 8.03 8.03 8.02 8.02 8.00 8.00 7.80 7.78 7.77 7.77 7.75 7.57 7.57 7.56 7.55 7.55 7.47 7.47 7.45 7.45 7.45 7.44 7.43 7.42 7.42 7.41 7.41 7.40 7.40 7.39 7.39 7.37 7.37 7.37 7.36 7.36 7.35 7.35 7.34 7.33 7.32 7.31 7.26 7.25 7.25 7.24 7.24 7.23 7.23 7.22 7.21 7.16 7.16 7.14 5.15 5.14 4.90 HDO 3.34 MeOD 3.33 MeOD 3.33 MeOD 3.33 MeOD 3.32 MeOD 2.81 2.80 2.78 1.04 1.03 1.02
OH
N
O
P
Ph
Ph
2.00 1.25 2.30 7.00 2.10 2.21 1.00 1.01 6.00
11.0 10.5 10.0 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 −0.5 −1.0 −1.5
f1 (ppm)


141.31 140.00 134.79 134.74 134.70 134.28 133.79 133.29 132.61 132.56 132.52 132.32 132.28 132.25 132.22 132.19 132.00 131.94 130.94 130.91 130.66 130.15 130.12 129.36 129.24 129.03 128.95 128.93 128.83 128.71 70.68 69.80
55.25 55.14 49.64 MeOD 49.21 MeOD 49.00 MeOD 48.79 MeOD 48.57 MeOD 48.36 MeOD 45.05
21.13 20.85 15.21
OH
N
O
P
Ph
Ph
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

3an


7.94 7.92 7.92 7.91 7.91 7.89 7.53 7.52 7.51 7.50 7.49 7.49 7.48 7.47 7.46 7.46 7.46 7.45 7.45 7.44 7.43 7.42 7.34 7.33 7.33 7.32 7.31 7.31 7.29 7.29 7.23 7.22 7.22 7.22 7.21 7.21 7.20 7.19 7.19 7.19 7.18 7.17 7.16 7.15 7.15 7.14 7.14 7.13 7.13 7.10 4.62 4.61 2.59
OH
Me
N
O
P
Ph
Ph
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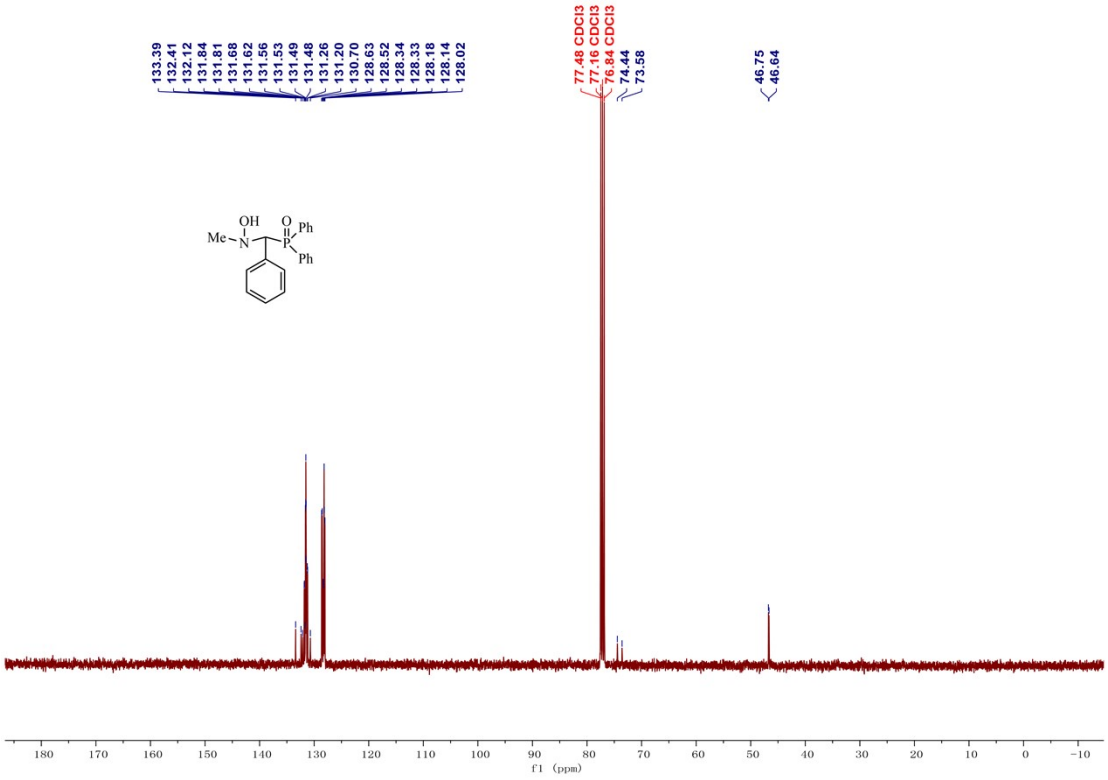
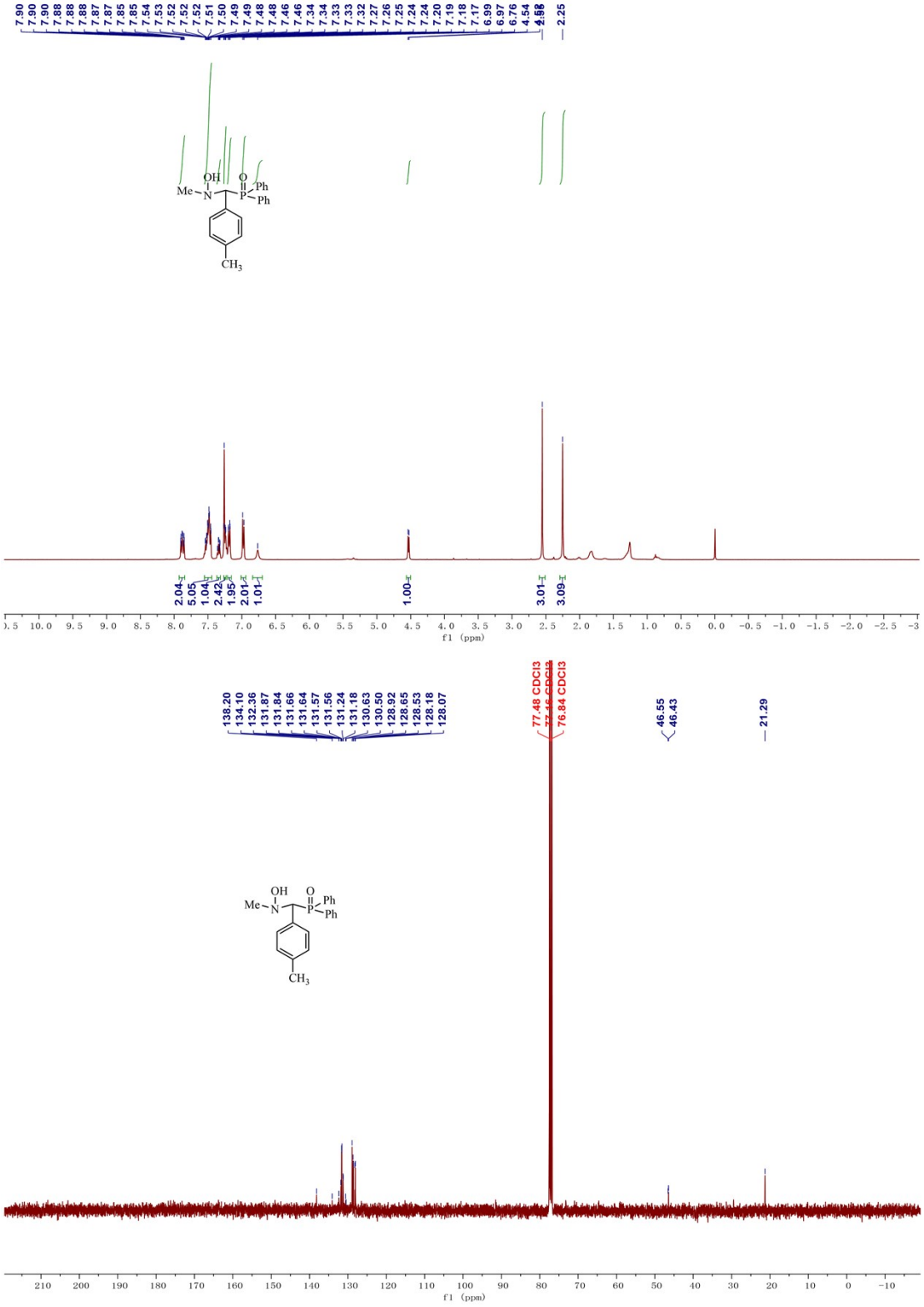
5.18
3.16
6.20
1.00
3.12
f1 (ppm)


133.39 132.41 132.12 131.84 131.81 131.68 131.62 131.56 131.53 131.49 131.48 131.26 131.20 130.70 128.63 128.52 128.34 128.33 128.18 128.14 128.02
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
74.44
73.58
46.75
46.64
OH
Me
N
O
P
Ph
Ph
f1 (ppm)

3ao


7.90
7.90
7.90
7.88
7.88
7.88
7.87
7.87
7.85
7.85
7.54
7.53
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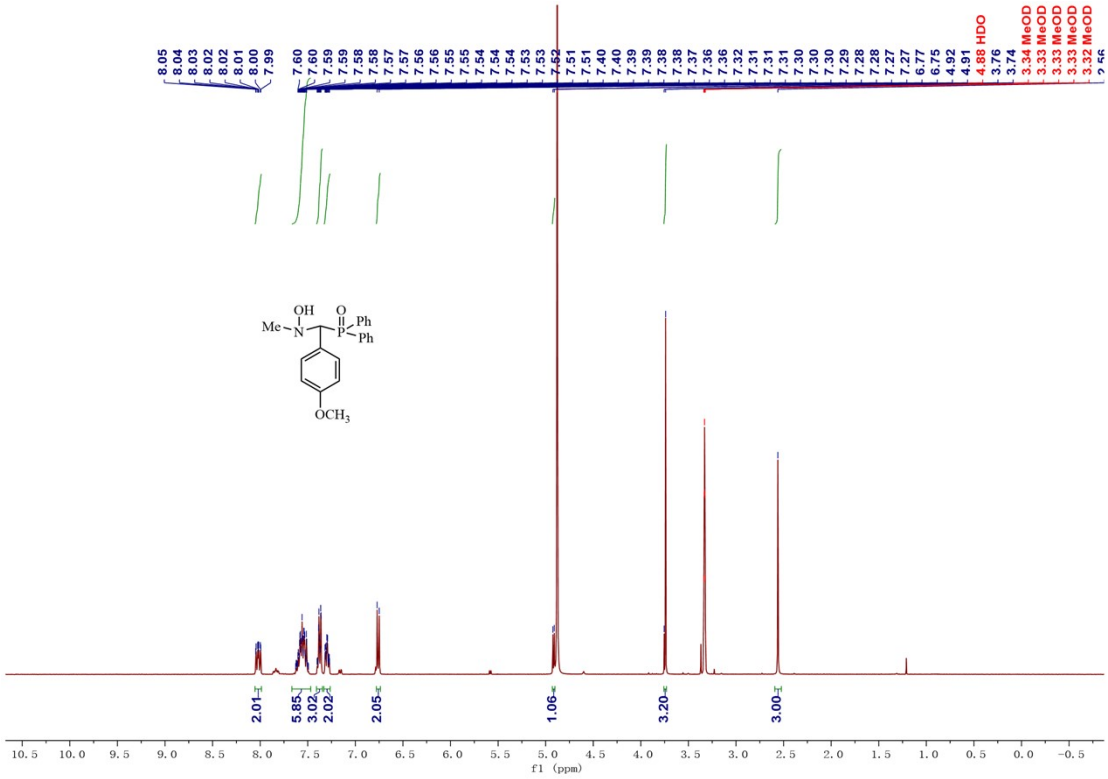
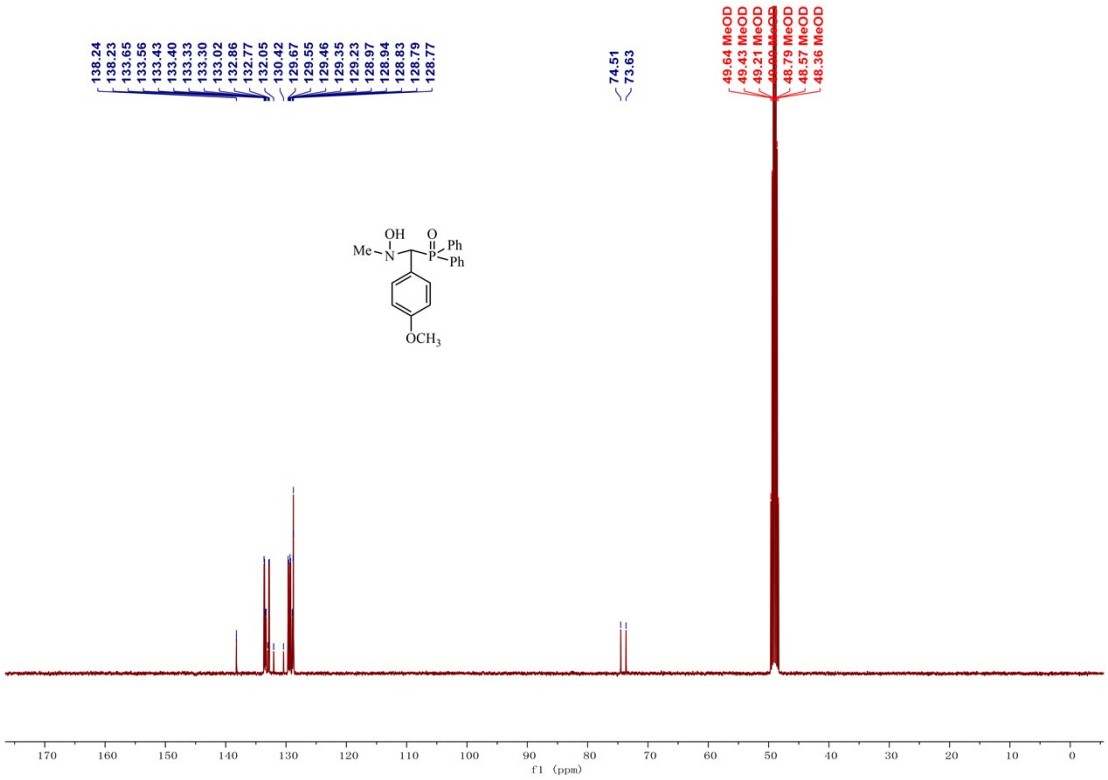
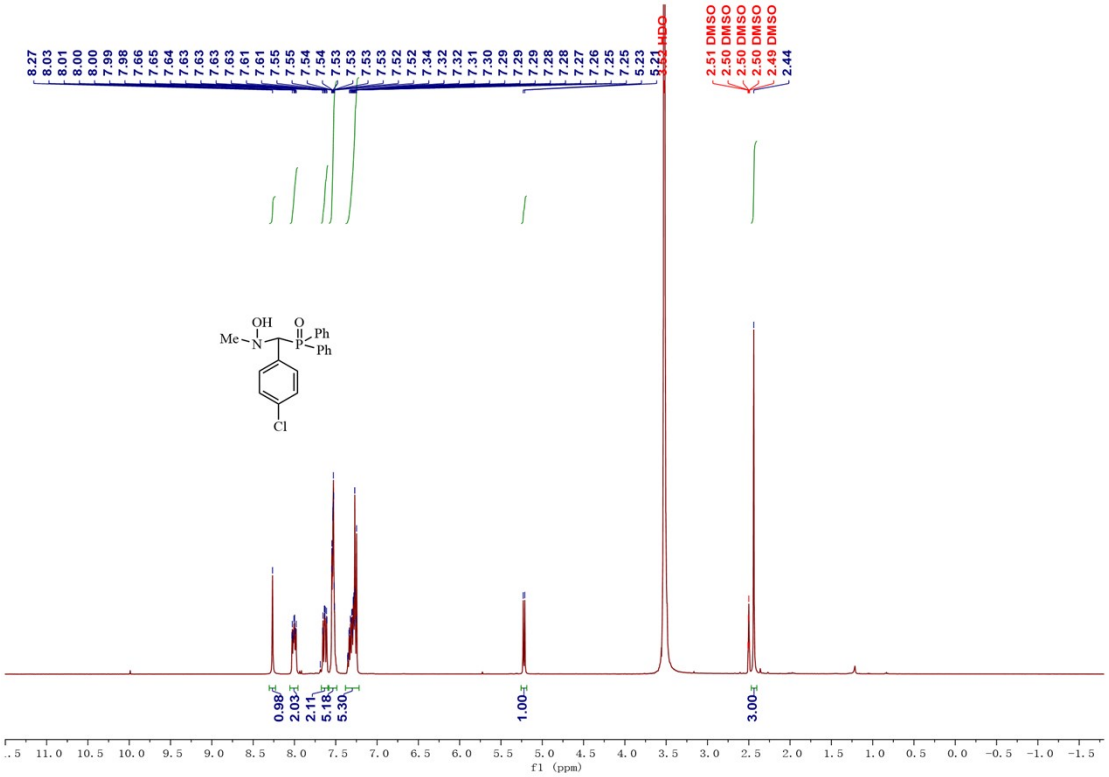
7.52
7.52
7.51
7.50
7.49
7.49
7.48
7.48
7.46
7.46
7.34
7.34
7.33
7.33
7.32
7.27
7.26
7.25
7.24
7.24
7.20
7.19
7.18
7.17
6.99
6.97
6.76
4.54
2.25
OH
Me
N
O
P
Ph
Ph
CH3
2.04
5.05
1.04
2.42
1.95
2.01
1.01
1.00
3.01
3.09
f1 (ppm)
138.20
134.10
132.36
131.87
131.84
131.66
131.64
131.57
131.56
131.24
131.18
130.63
130.50
128.92
128.65
128.53
128.18
128.07
77.48 CDCl3
76.84 CDCl3
46.55
46.43
21.29
f1 (ppm)

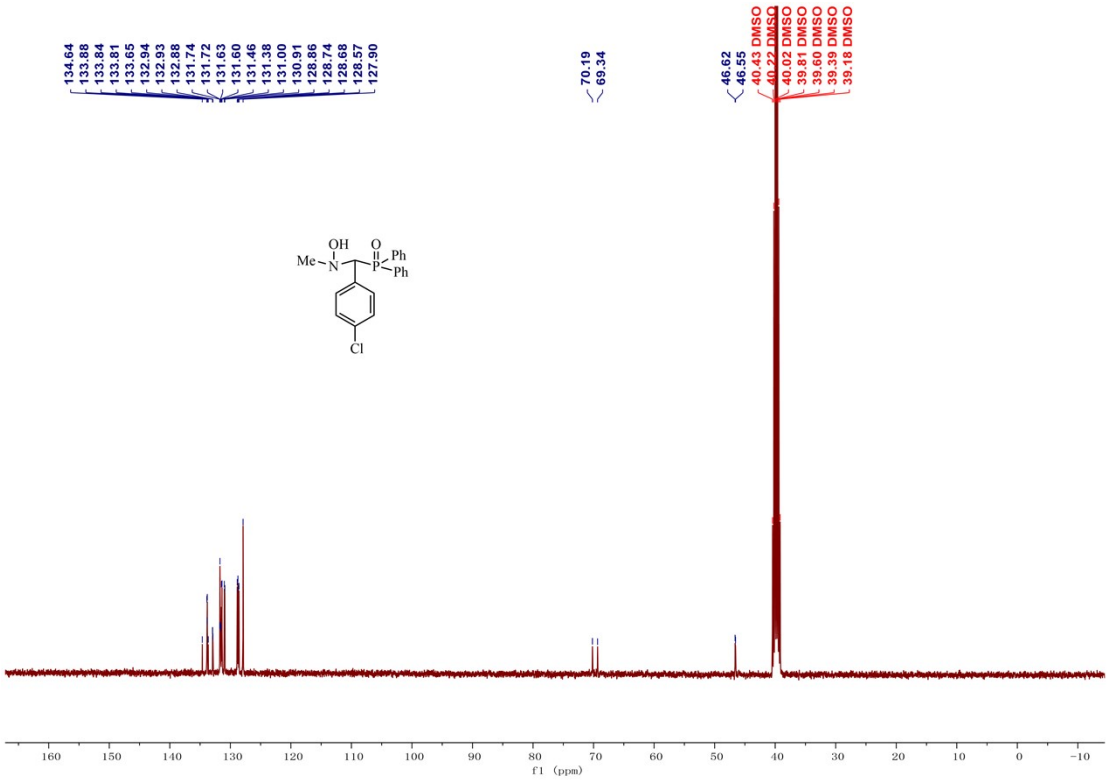
3ap


8.05 8.04 8.03 8.02 8.02 8.01 8.00 7.99
7.60 7.60 7.59 7.59 7.58 7.58 7.57 7.57 7.56 7.56 7.55 7.54 7.54 7.53 7.53 7.52 7.51 7.51 7.40 7.40 7.39 7.39 7.38 7.38 7.37 7.36 7.32 7.31 7.31 7.31 7.30 7.30 7.29 7.28 7.28 7.27 7.27 6.77 6.75 4.92 4.91 4.88 HDO 3.76 3.74 3.34 MeOD 3.33 MeOD 3.33 MeOD 3.33 MeOD 3.32 MeOD
OH
Me
N
O
P
Ph
Ph
OCH3
2.01 5.85 3.02 2.02 2.05 1.06 3.20 3.00
f1 (ppm)

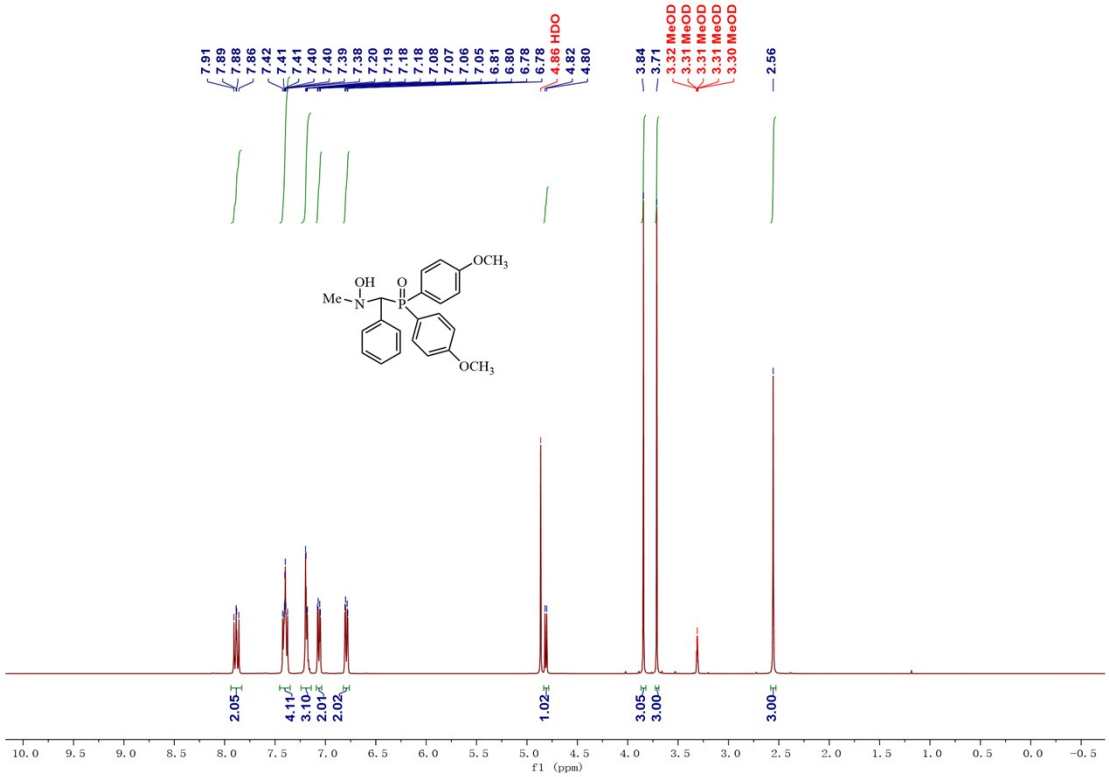

138.24 138.23 133.65 133.56 133.43 133.40 133.33 133.30 133.02 132.86 132.77 132.05 130.42 129.67 129.55 129.46 129.35 129.23 128.97 128.94 128.83 128.79 128.77
74.51 73.63
49.64 MeOD 49.43 MeOD 49.21 MeOD 48.79 MeOD 48.57 MeOD 48.36 MeOD
OH
Me
N
O
P
Ph
Ph
OCH3
f1 (ppm)

3aq


8.27 8.03 8.01 8.00 8.00 7.99 7.98 7.66 7.65 7.64 7.63 7.63 7.63 7.63 7.61 7.61 7.55 7.55 7.54 7.54 7.53 7.53 7.53 7.53 7.52 7.52 7.34 7.32 7.32 7.31 7.30 7.29 7.29 7.29 7.28 7.28 7.27 7.26 7.25 7.25 5.23 5.21 3.51 HDO
2.51 DMSO 2.50 DMSO 2.50 DMSO 2.50 DMSO 2.49 DMSO 2.44
OH O Me N P Ph Ph Cl
0.98 2.03 2.11 5.18 5.30 1.00 3.00
f1 (ppm)


134.64 133.88 133.84 133.81 133.65 132.94 132.93 132.88 131.74 131.72 131.63 131.60 131.46 131.38 131.00 130.91 128.86 128.74 128.68 128.57 127.90
70.19 69.34
46.62 46.55
40.43 DMSO 40.22 DMSO 40.02 DMSO 39.81 DMSO 39.60 DMSO 39.39 DMSO 39.18 DMSO
OH O Me N P Ph Ph Cl
f1 (ppm)

3ar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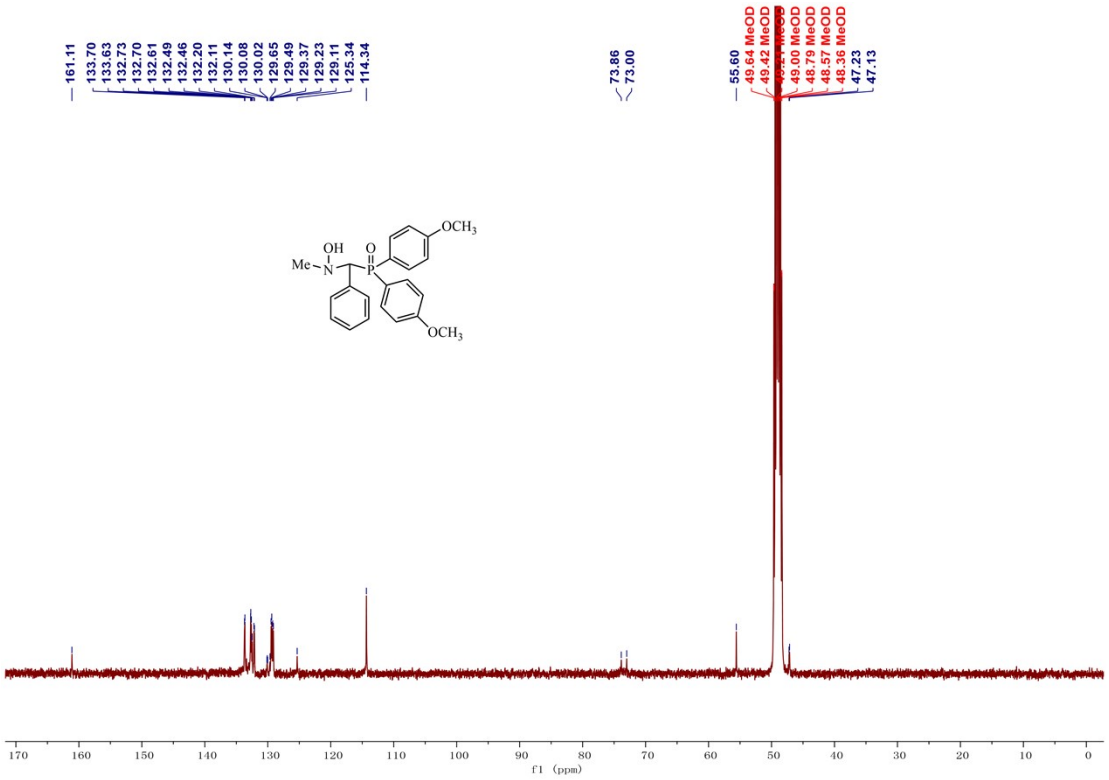
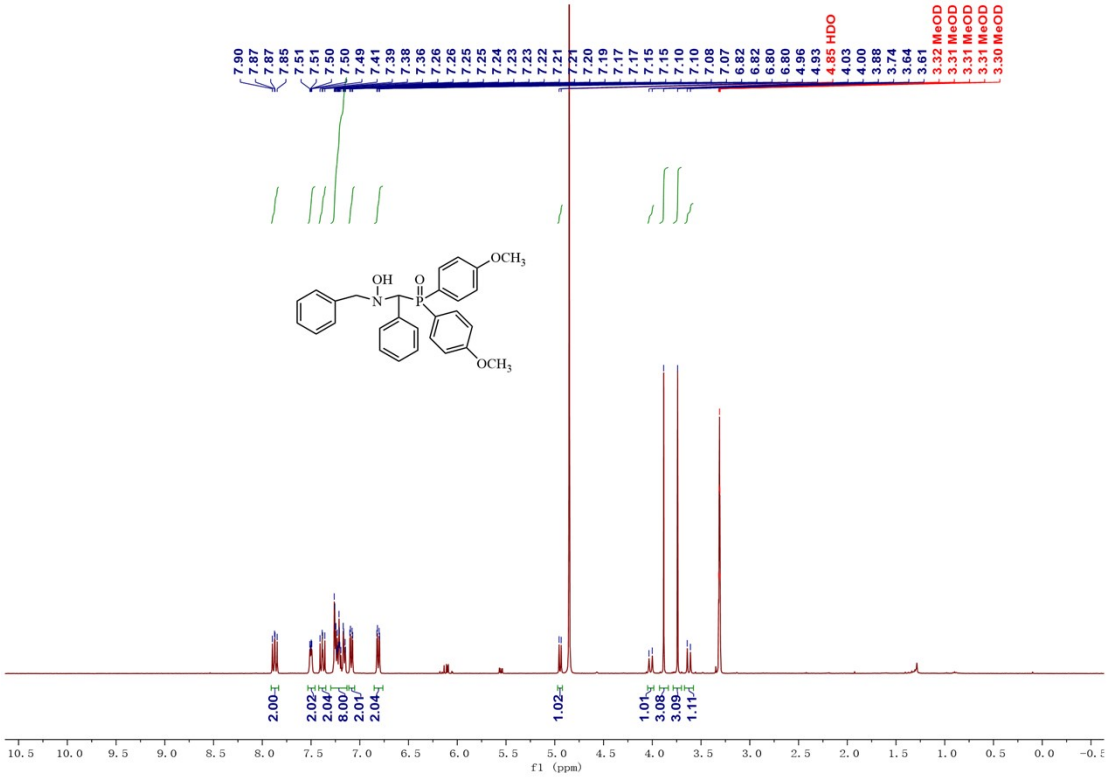
7.86
7.42
7.41
7.41
7.40
7.40
7.39
7.38
7.20
7.19
7.18
7.18
7.08
7.07
7.06
7.05
6.81
6.80
6.78
6.78
4.86 HDO
4.82
4.80
3.84
3.71
3.32 MeOD
3.31 MeOD
3.31 MeOD
3.31 MeOD
3.30 MeOD
2.56
OCH3
OH
O
Me
N
P
OCH3
2.05
4.11
3.10
2.01
2.02
1.02
3.05
3.00
3.00
f1 (ppm)


161.11
133.70
133.63
132.73
132.70
132.61
132.49
132.46
132.20
132.11
130.14
130.08
130.02
129.65
129.49
129.37
129.23
129.11
125.34
114.34
73.86
73.00
55.60
49.64 MeOD
49.42 MeOD
49.00 MeOD
48.79 MeOD
48.57 MeOD
48.36 MeOD
47.23
47.13
OCH3
OH
O
Me
N
P
OCH3
f1 (ppm)

3as


7.90
7.87
7.87
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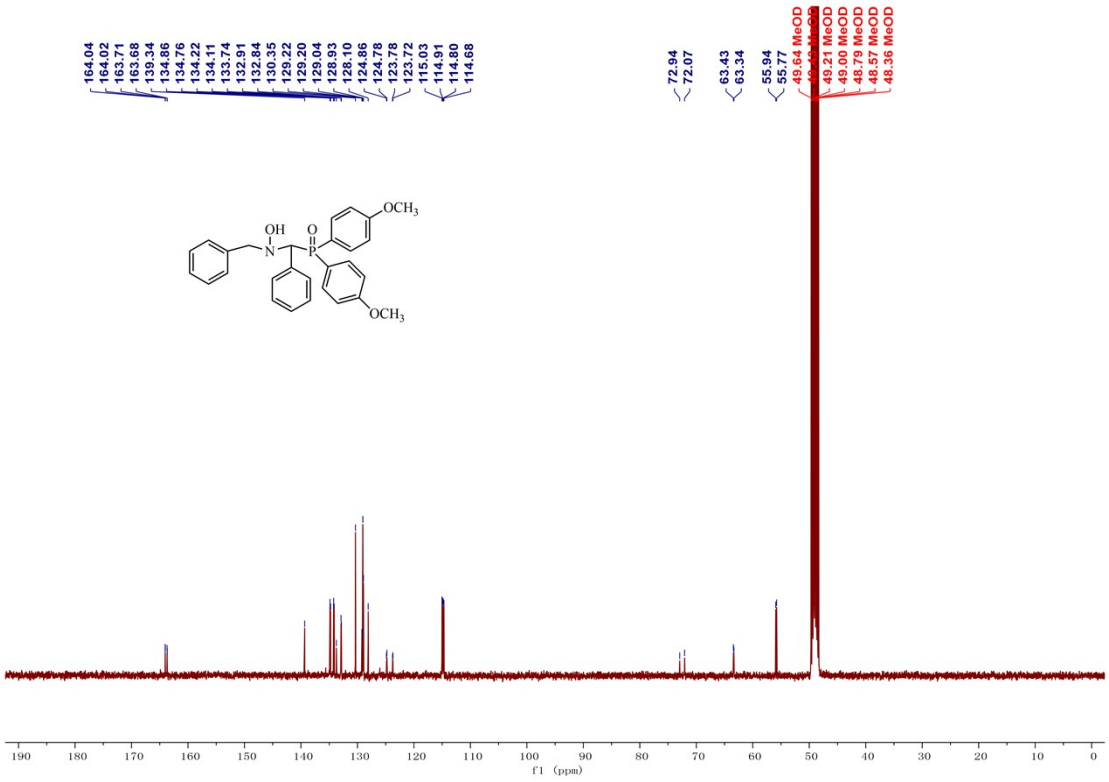
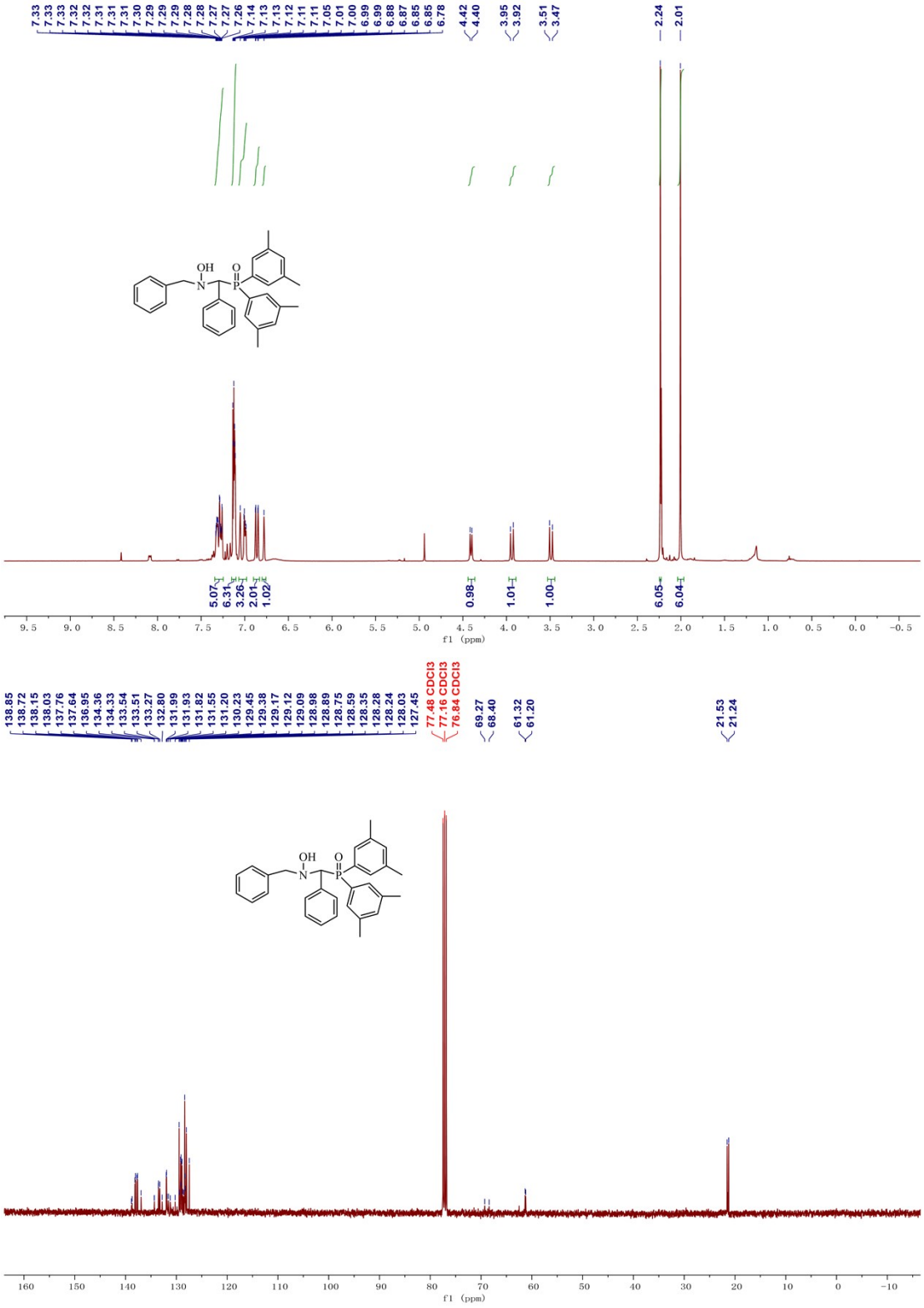
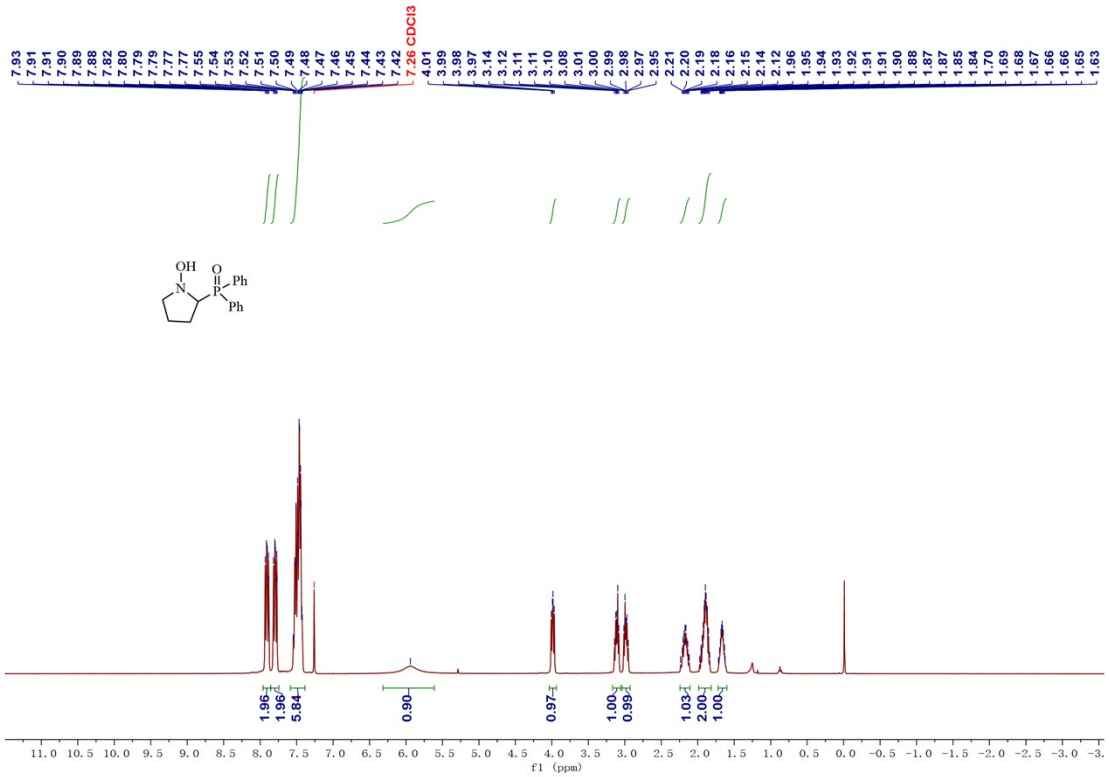
7.51
7.51
7.50
7.50
7.49
7.41
7.39
7.38
7.36
7.26
7.26
7.25
7.25
7.24
7.23
7.23
7.22
7.21
7.21
7.20
7.19
7.17
7.17
7.15
7.15
7.10
7.10
7.08
7.07
6.82
6.82
6.80
6.80
4.96
4.93
4.85 HDO
4.03
4.00
3.88
3.74
3.64
3.61
3.32 MeOD
3.31 MeOD
3.31 MeOD
3.31 MeOD
3.30 MeOD
OCH3
OH
N
O
P
OCH3
2.00
2.02
2.04
8.00
2.01
2.04
1.02
1.01
3.08
3.09
1.11
f1 (ppm)

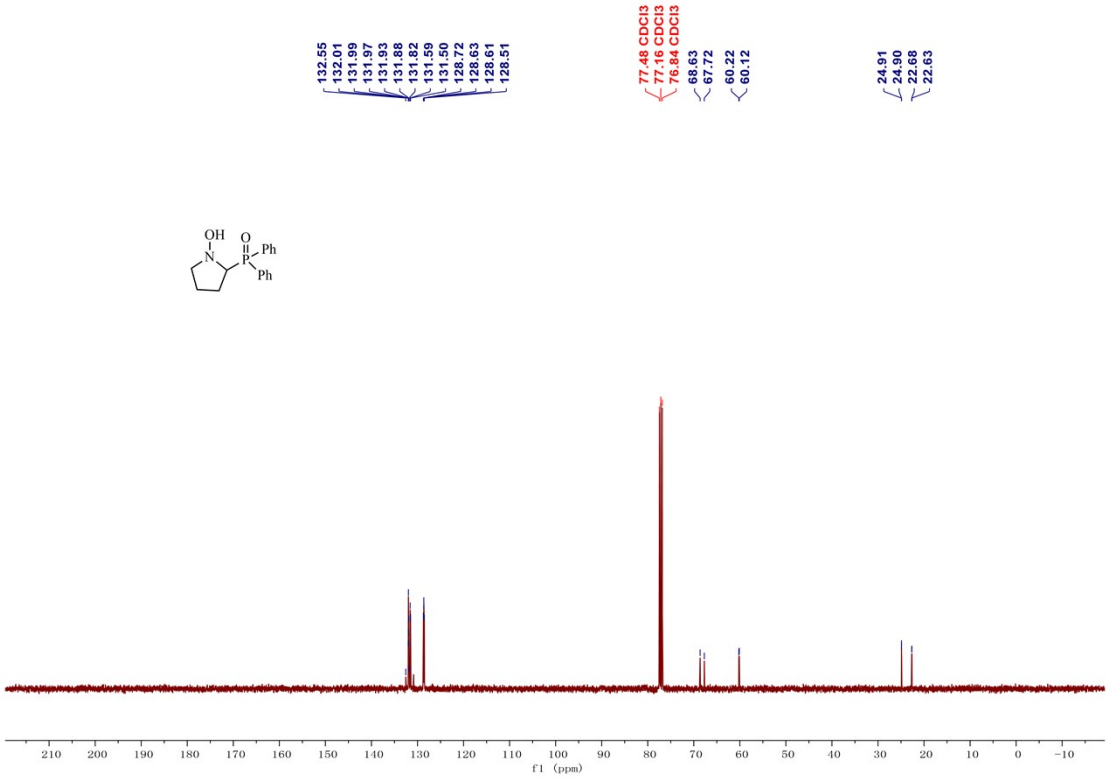

164.04
164.02
163.71
163.68
139.34
134.86
134.76
134.22
134.11
133.74
132.91
132.84
130.35
129.22
129.20
129.04
128.93
128.10
124.86
124.78
123.78
123.72
115.03
114.91
114.80
114.68
72.94
72.07
63.43
63.34
55.94
55.77
49.64 MeOD
49.21 MeOD
49.00 MeOD
48.79 MeOD
48.57 MeOD
48.36 MeOD
OCH3
OH
N
O
P
OCH3
f1 (ppm)

3at

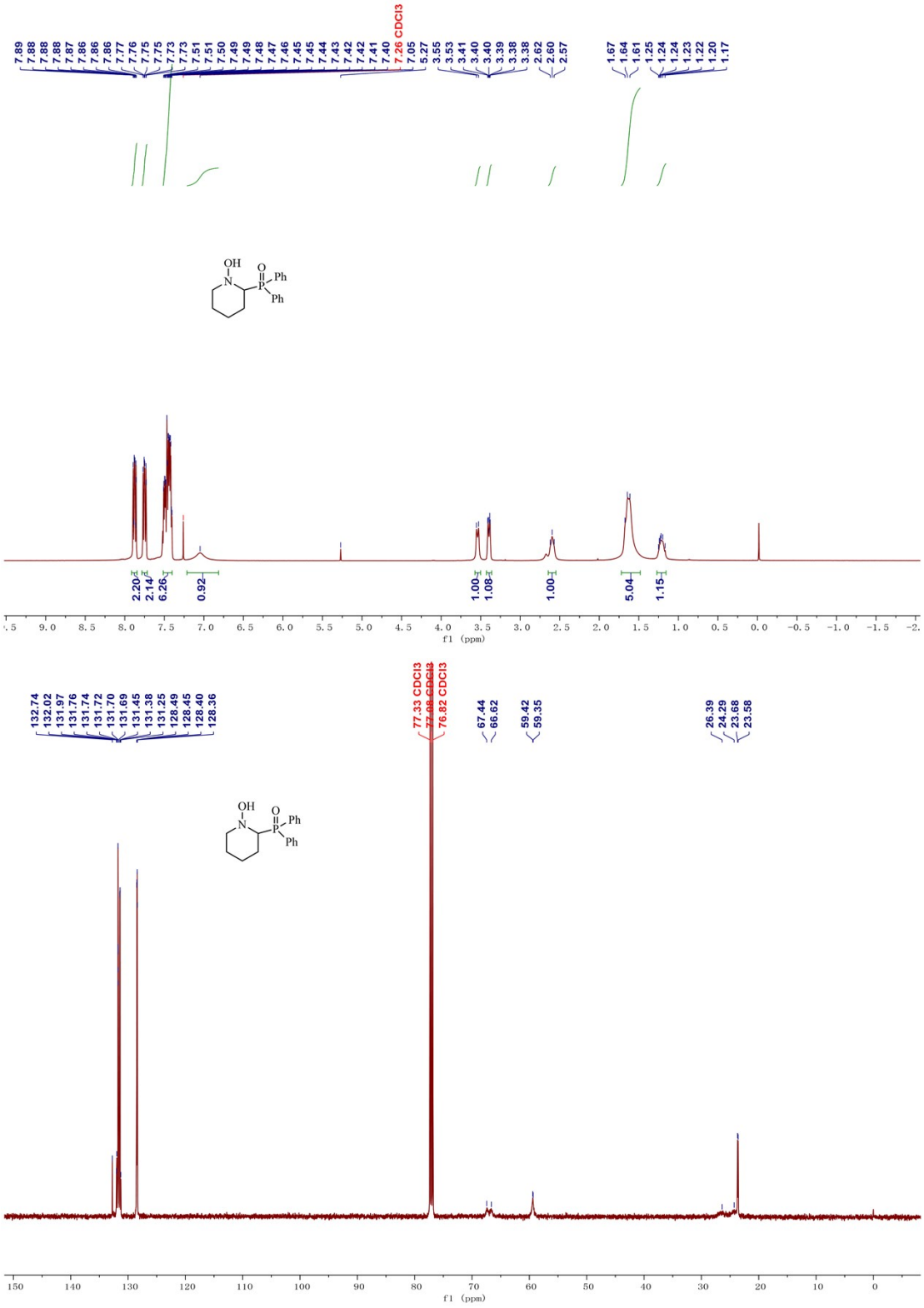

7.33 7.33 7.33 7.32 7.32 7.31 7.31 7.31 7.30 7.29 7.29 7.29 7.28 7.28 7.27 7.27 7.26 7.14 7.13 7.13 7.12 7.11 7.11 7.05 7.01 7.00 6.99 6.88 6.87 6.85 6.85 6.78
4.42 4.40
3.95 3.92
3.51 3.47
2.24 2.01
OH
N
O
P
5.07 6.31 3.26 2.01 1.02
0.98 1.01 1.00
6.05 6.04
f1 (ppm)
138.85 138.72 138.15 138.03 137.76 137.64 136.95 134.36 134.33 133.54 133.51 133.27 132.80 131.99 131.93 131.82 131.55 131.20 130.23 129.45 129.38 129.17 129.12 129.09 128.98 128.89 128.75 128.59 128.35 128.28 128.24 128.03 127.45
77.48 CDCl3 77.16 CDCl3 76.84 CDCl3
69.27 68.40
61.32 61.20
21.53 21.24
OH
N
O
P
f1 (ppm)

5a

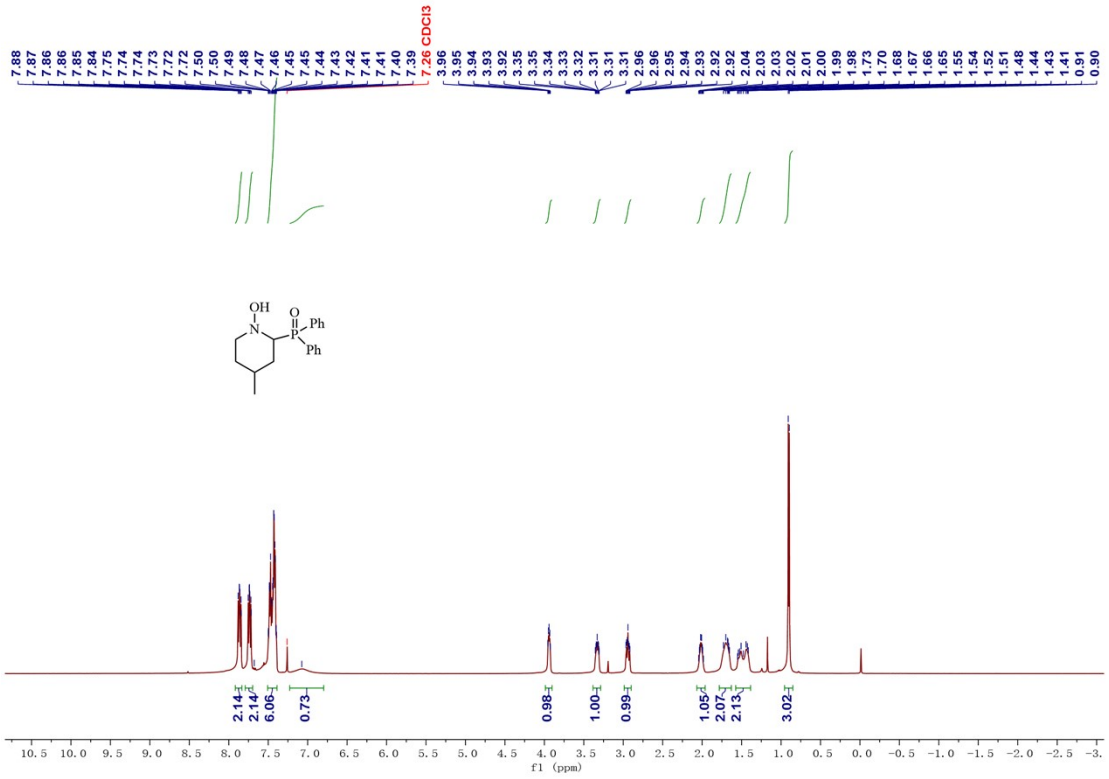

7.93 7.91 7.91 7.90 7.89 7.88 7.82 7.80 7.79 7.79 7.77 7.77 7.55 7.54 7.53 7.52 7.51 7.50 7.49 7.48 7.47 7.46 7.45 7.44 7.43 7.42 7.26 CDCl3
4.01 3.99 3.98 3.97 3.14 3.12 3.11 3.11 3.10 3.08 3.01 3.00 2.99 2.98 2.97 2.95 2.21 2.20 2.19 2.18 2.16 2.15 2.14 2.12 1.96 1.95 1.94 1.93 1.92 1.91 1.91 1.90 1.88 1.87 1.87 1.85 1.84 1.70 1.69 1.68 1.67 1.66 1.66 1.65 1.63
OH
N
O
P
Ph
Ph
1.96 1.96 5.84 0.90 0.97 1.00 0.99 1.03 2.00 1.00
f1 (ppm)

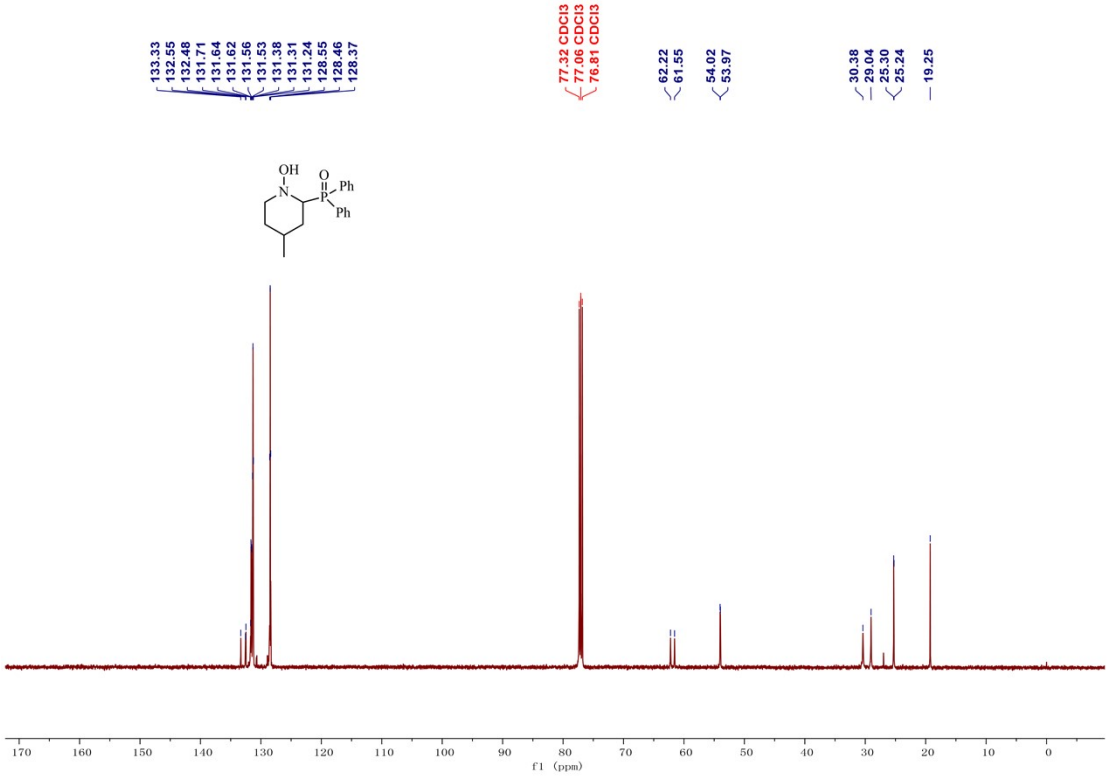

132.55 132.01 131.99 131.97 131.93 131.88 131.82 131.59 131.50 128.72 128.63 128.61 128.51
77.48 CDCl3 77.16 CDCl3 76.84 CDCl3 68.63 67.72 60.22 60.12
24.91 24.90 22.68 22.63
OH
N
O
P
Ph
Ph
f1 (ppm)

5b

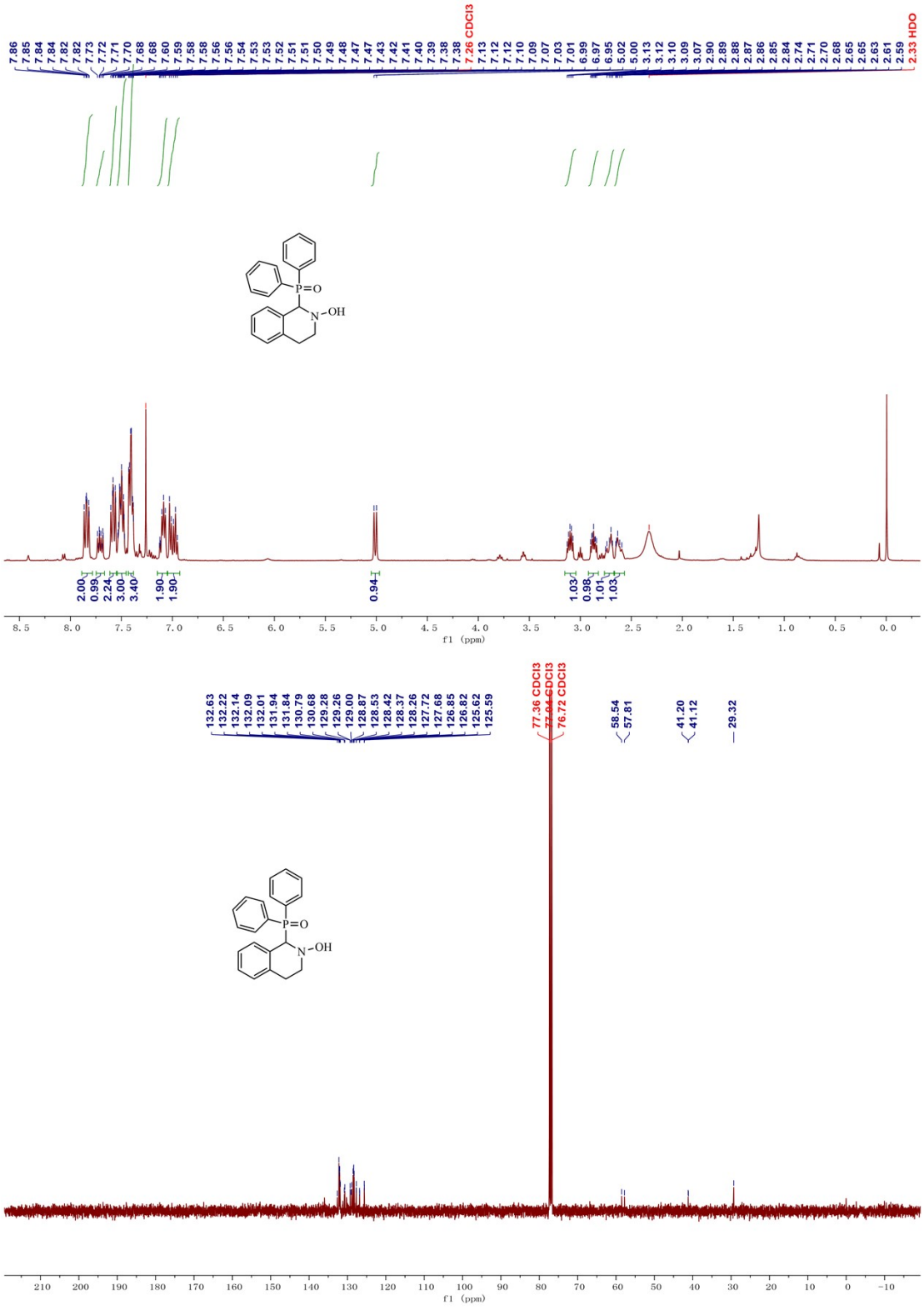

7.89 7.88 7.88 7.88 7.87 7.86 7.86 7.86 7.77 7.76 7.75 7.73 7.73 7.51 7.51 7.50 7.49 7.49 7.48 7.47 7.46 7.45 7.45 7.44 7.43 7.42 7.42 7.41 7.40 7.26 CDCl3 7.05 5.27 3.55 3.53 3.41 3.40 3.40 3.39 3.38 3.38 2.62 2.60 2.57 1.67 1.64 1.61 1.25 1.24 1.24 1.23 1.22 1.20 1.17
OH
N
O
P
Ph
Ph
2.20 2.14 6.26 0.92 1.00 1.08 1.00 5.04 1.15
f1 (ppm)
132.74 132.02 131.97 131.76 131.74 131.72 131.70 131.69 131.45 131.38 131.25 128.49 128.45 128.40 128.36
77.33 CDCl3 76.82 CDCl3
67.44 66.62 59.42 59.35 26.39 24.29 23.68 23.58
f1 (ppm)

5c

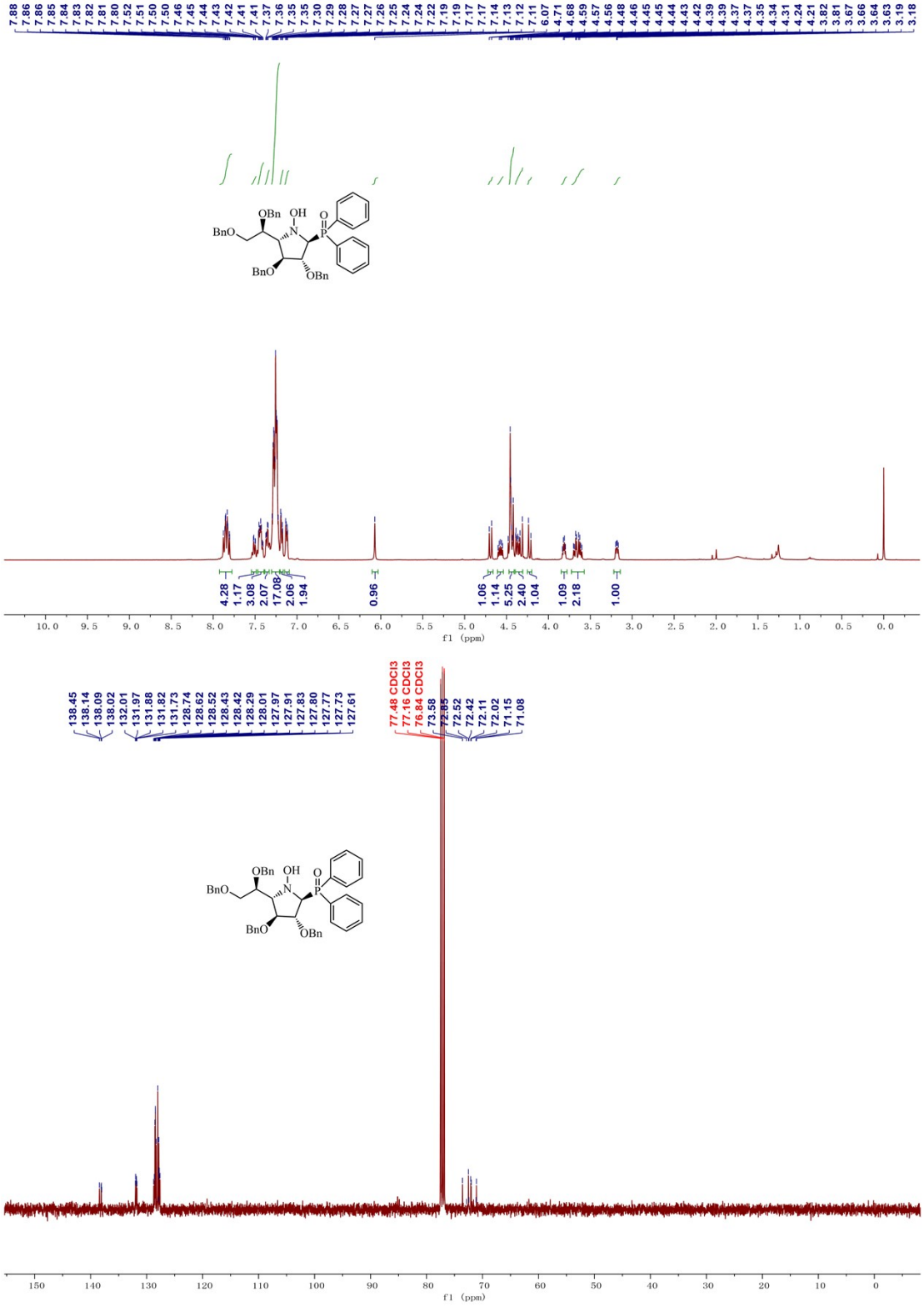

7.88 7.87 7.86 7.86 7.85 7.84 7.75 7.74 7.74 7.73 7.72 7.72 7.50 7.50 7.49 7.48 7.47 7.46 7.45 7.45 7.44 7.43 7.42 7.41 7.41 7.40 7.39 7.26 CDCl3
3.96 3.95 3.94 3.93 3.92 3.35 3.35 3.34 3.33 3.32 3.31 3.31 3.31 2.96 2.96 2.95 2.94 2.93 2.92 2.92 2.04 2.03 2.03 2.02 2.01 2.00 1.99 1.98 1.73 1.70 1.68 1.67 1.66 1.65 1.55 1.54 1.52 1.51 1.48 1.44 1.43 1.41 0.91 0.90
OH
O
Ph
Ph
2.14 2.14 6.06 0.73 0.98 1.00 0.99 1.05 2.07 2.13 3.02
f1 (ppm)


133.33 132.55 132.48 131.71 131.64 131.62 131.56 131.53 131.38 131.31 131.24 128.55 128.46 128.37
77.32 CDCl3 77.06 CDCl3 76.81 CDCl3
62.22 61.55 54.02 53.97
30.38 29.04 25.30 25.24 19.25
OH
O
Ph
Ph
f1 (ppm)

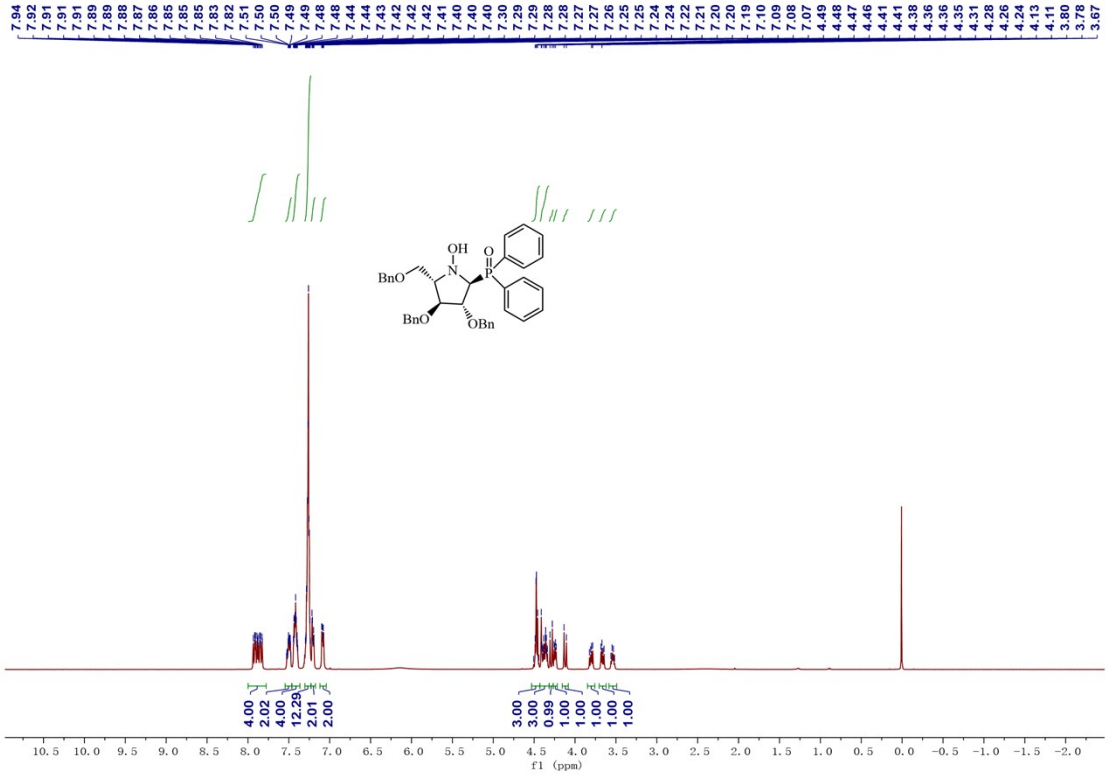
5d

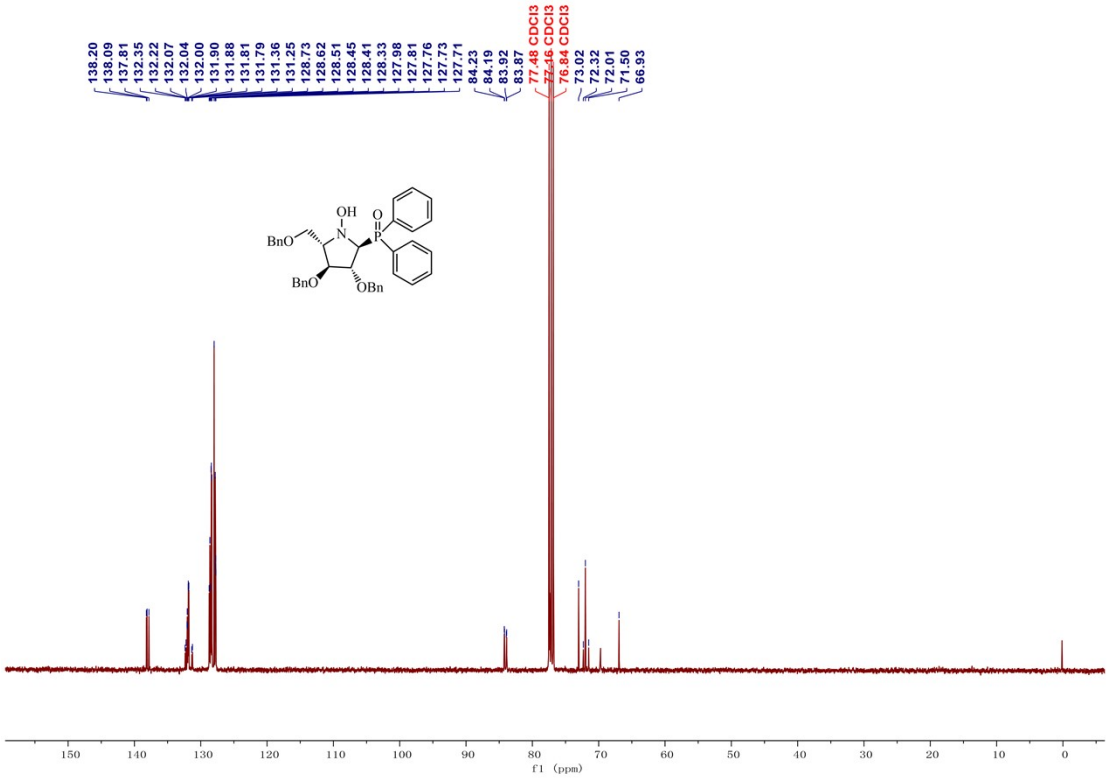

7.86 7.85 7.84 7.84 7.82 7.82 7.73 7.72 7.71 7.70 7.68 7.68 7.60 7.59 7.58 7.58 7.56 7.56 7.54 7.53 7.53 7.52 7.51 7.51 7.50 7.49 7.48 7.47 7.47 7.43 7.42 7.41 7.40 7.39 7.38 7.38 7.26 CDCl3 7.13 7.12 7.12 7.10 7.09 7.07 7.03 7.01 6.99 6.97 6.95 5.02 5.00 3.13 3.12 3.10 3.09 3.07 2.90 2.89 2.88 2.87 2.86 2.85 2.84 2.74 2.71 2.70 2.68 2.65 2.65 2.63 2.61 2.59 2.33 HDO
P=O
N-OH
2.00 0.99 2.24 3.00 3.40 1.90 1.90 0.94 1.03 0.98 1.01 1.03
f1 (ppm)
132.63 132.22 132.14 132.09 132.01 131.94 131.84 130.79 130.68 129.28 129.26 129.00 128.87 128.53 128.42 128.37 128.26 127.72 127.68 126.85 126.82 125.62 125.59
77.36 CDCl3 77.04 CDCl3 76.72 CDCl3
58.54 57.81 41.20 41.12 29.32
P=O
N-OH
f1 (ppm)

5e

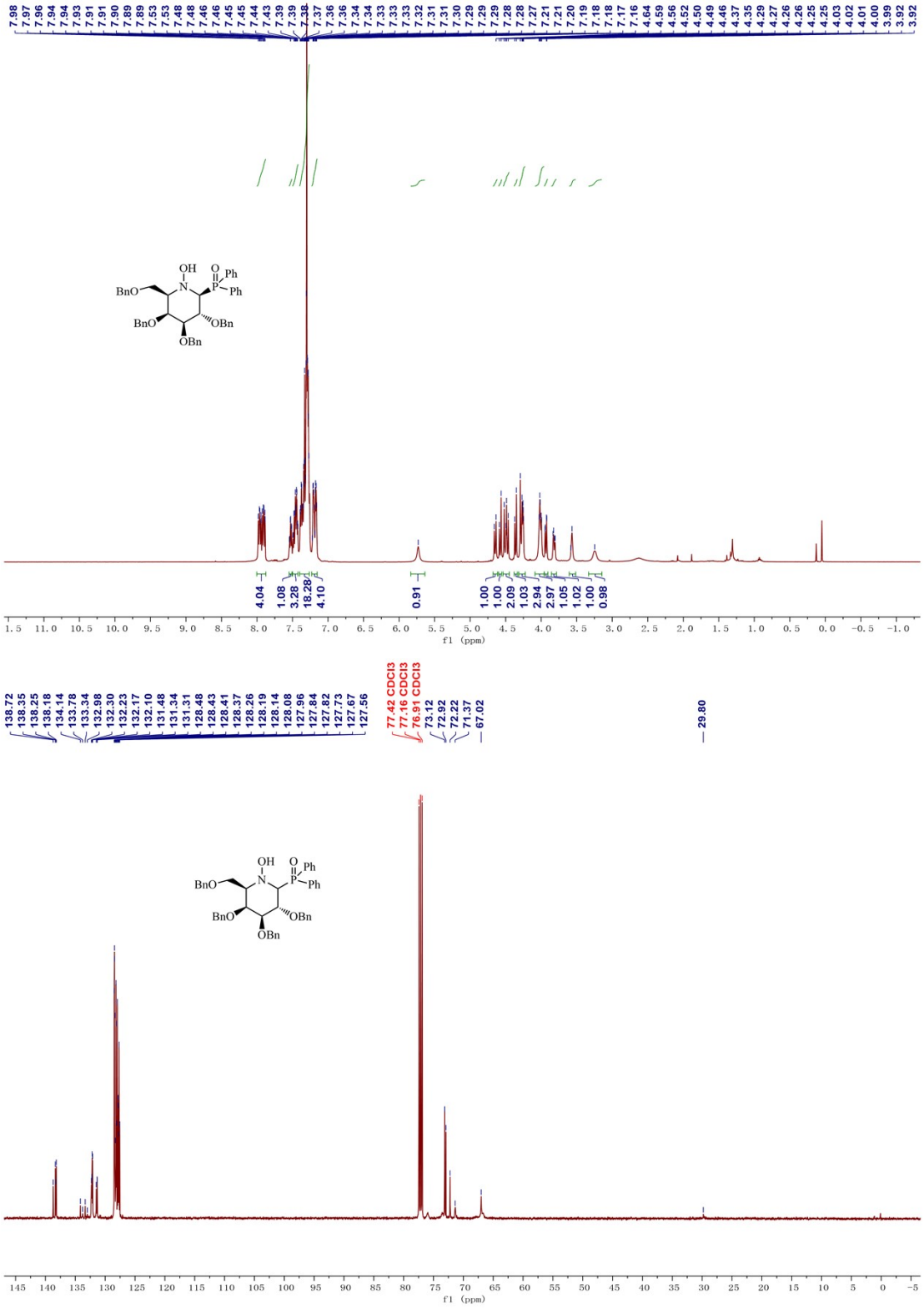

7.88 7.86 7.86 7.85 7.84 7.83 7.82 7.81 7.80 7.52 7.51 7.50 7.50 7.46 7.45 7.44 7.43 7.42 7.41 7.41 7.37 7.36 7.35 7.35 7.30 7.29 7.28 7.27 7.27 7.26 7.25 7.24 7.24 7.22 7.19 7.19 7.17 7.17 7.14 7.13 7.12 7.11 6.07 4.71 4.68 4.59 4.57 4.56 4.48 4.46 4.45 4.45 4.44 4.43 4.42 4.39 4.39 4.37 4.37 4.35 4.34 4.31 4.24 4.21 3.82 3.81 3.67 3.66 3.64 3.63 3.19 3.18
OBn OH O
BnO N P
BnO OBn
4.28 1.17 3.08 2.07 17.08 2.06 1.94 0.96 1.06 1.14 5.25 2.40 1.04 1.09 2.18 1.00
10.0 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
f1 (ppm)
138.45 138.14 138.09 138.02 132.01 131.97 131.88 131.82 131.73 128.74 128.62 128.52 128.43 128.42 128.29 128.01 127.97 127.91 127.83 127.80 127.77 127.73 127.61
77.48 CDCl3 77.16 CDCl3 76.84 CDCl3 73.58 72.85 72.52 72.42 72.11 72.02 71.15 71.08
OBn OH O
BnO N P
BnO OBn
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

5f

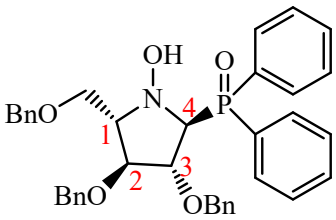

7.94 7.92 7.91 7.91 7.91 7.89 7.89 7.88 7.87 7.86 7.85 7.85 7.85 7.83 7.82 7.51 7.50 7.50 7.49 7.49 7.48 7.48 7.44 7.44 7.43 7.42 7.42 7.41 7.40 7.40 7.40 7.30 7.29 7.29 7.28 7.28 7.27 7.27 7.26 7.25 7.25 7.24 7.24 7.22 7.21 7.20 7.20 7.19 7.10 7.09 7.08 7.07 4.49 4.48 4.47 4.46 4.41 4.41 4.38 4.36 4.35 4.31 4.28 4.26 4.24 4.13 4.11 3.80 3.78 3.67
OH
O
P
N
BnO
BnO
OBn
4.00 2.02 4.00 12.29 2.01 2.00
3.00 3.00 0.99 1.00 1.00 1.00 1.00 1.00
10.5 10.0 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 -0.5 -1.0 -1.5 -2.0
f1 (ppm)


138.20 138.09 137.81 132.35 132.22 132.07 132.04 132.00 131.90 131.88 131.81 131.79 131.36 131.25 128.73 128.62 128.51 128.45 128.41 128.33 127.98 127.81 127.76 127.73 127.71 84.23 84.19 83.92 83.87 77.48 CDCl3 77.16 CDCl3 76.84 CDCl3 73.02 72.32 72.01 71.50 66.93
OH
O
P
N
BnO
BnO
OBn
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

5g


7.98 7.97 7.96 7.94 7.94 7.93 7.91 7.91 7.90 7.89 7.89 7.53 7.48 7.48 7.46 7.46 7.45 7.45 7.44 7.43 7.39 7.39 7.38 7.37 7.36 7.36 7.34 7.34 7.33 7.33 7.32 7.31 7.31 7.30 7.29 7.29 7.29 7.28 7.28 7.27 7.21 7.21 7.20 7.19 7.18 7.18 7.17 7.16 4.64 4.59 4.56 4.52 4.50 4.49 4.46 4.37 4.35 4.29 4.27 4.26 4.26 4.25 4.25 4.03 4.02 4.01 4.00 3.99 3.92
OH
O
Ph
P
Ph
N
BnO
BnO
OBn
OBn
4.04 1.08 3.28 18.28 4.10 0.91 1.00 1.00 2.09 1.03 2.94 2.97 1.05 1.02 1.00 0.98
f1 (ppm)
138.72 138.35 138.25 138.18 134.14 133.78 133.34 132.98 132.30 132.23 132.17 132.10 131.48 131.34 131.31 128.48 128.43 128.41 128.37 128.26 128.19 128.14 128.08 127.96 127.84 127.82 127.73 127.67 127.56
77.42 CDCl3
77.16 CDCl3
76.91 CDCl3
73.12 72.92 72.22 71.37 67.02
29.80
f1 (ppm)

5f



The H on the five-membered ring is at δ 3.54 (q, *J* = 6.0 Hz, 1H) at position 1, at δ 4.24 (dd, *J* = 7.1, 3.9 Hz, 1H) at position 2, at δ 4.43 - 4.32 (m, 3H) at position 3, and at δ 4.12 (d, *J* = 11.4 Hz, 1H) at position 4. To determine the orientation of the hydrogen at the 4-position, the H at the 4-position is related to the H at the 2-position, independent of the H at the 1-position and 3-position, according to the NOESY spectrum. The results confirmed that the hydrogens at the 2 and 4 positions are on the same side. Therefore, we determined the structure of 5f.