

Supporting Information

Harnessing Nitroarenes: Photo-Driven Synergistic Construction of Phosphinic Amides via Hydrogen Transfer and Oxygen Migration

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Table of Contents

Page S1	General Information
Page S1-S2	General procedure for phosphamides
Page S2-S5	Mechanistic Studies
Page S6-S23	Characterization data for the products
Page S24-S90	NMR Spectra

I. General Information

All reactions were run in a tube under air atmosphere. All commercially available reagent grade chemicals and solvents were used as received without further purification. ^1H NMR (400 MHz), ^{13}C NMR (100 MHz), ^{19}F NMR (376 MHz) and ^{31}P NMR (162 MHz), spectra are reported relative to chemical shift of tetramethylsilane (TMS). Chemical shifts (δ) are reported in ppm and coupling constants (J) in hertz (Hz). The following abbreviations are used to explain the multiplicities: s = singlet, d = doublet, dd = doublet of doublet, t = triplet, q = quartet, m = multiplet. For HRMS (ESI) measurements, the mass analyzer is micrOTOF-Q. The UV-Vis measurements were carried out using a UV-Vis spectrophotometer (ULN 2209003, MAPADA P6). Photochemical reactions were performed with a 100 W 390 nm LED purchased from Shanghai 3S Technology Co., Ltd (<https://www.3s-tech.net/en/#>).

II. General procedure for Phosphinic Amides

(1) With the scale of 0.2 mmol

Add nitroarene (0.2 mmol), pentamethyldiethylenetriamine (PMDETA, 2.0 equivalents), and chlorophosphines (1.2 equivalents) sequentially to ethyl acetate (4 mL) in a 10 mL reaction tube. Irradiate the mixture with a 100 W 390 nm LED at room temperature under an air atmosphere for 8 hours, monitoring the reaction progress by thin-layer chromatography (TLC). Upon completion, quench the reaction mixture with water (10 mL) and extract it with ethyl acetate (3 \times). Combine the organic layers, dry over anhydrous Na_2SO_4 , and filter. Concentrate the filtrate under reduced pressure to obtain the crude product. Purify the residue by silica gel column chromatography using a petroleum ether/ethyl acetate gradient to afford the pure product.

(2) With the scale of gram quantities and subsequent derivatization to synthesize inhibitors of Kv1.5

Add 3-nitropyridine (1 g), PMDETA (2.0 equiv.), and chlorophosphines (1.2 equiv.) sequentially to ethyl acetate (20 mL) in a 50 mL reaction tube. Irradiate the mixture with a 100 W 390 nm LED at room temperature under an air atmosphere for 8 hours, monitoring the reaction progress by thin-layer chromatography (TLC). Upon completion, quench the reaction mixture with water (10 mL) and extract it with ethyl acetate (3 \times). Combine the organic layers, dry over anhydrous Na_2SO_4 , and filter. Concentrate the filtrate under reduced pressure to obtain the crude product. Purify the residue by silica gel column chromatography using a petroleum ether/ethyl acetate gradient to afford the pure product.

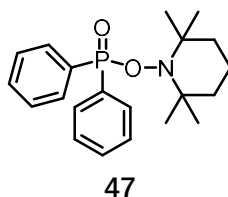
Dissolve compound **44** in anhydrous DMF (15 mL) and cool the mixture to 0 $^\circ\text{C}$. Add sodium hydride (3 equivalents) in batches. After 1 hour, add iodo propane (3 equivalents) dropwise and stir

the mixture at room temperature for 6 hours. Slowly pour the reaction mixture into ice-cold water and extract with ethyl acetate. Wash the organic layer with brine, dry over MgSO_4 , and concentrate under reduced pressure. Purify the residue by silica gel column chromatography (dichloromethane/methanol = 10:1) to obtain compound **45**.

III. Mechanistic Studies

(1) Control experiment

The formation was completely restrained upon adding TEMPO (2.0 equiv.) under standard conditions, and radical adduct **47** was detected by NMR and HRMS.



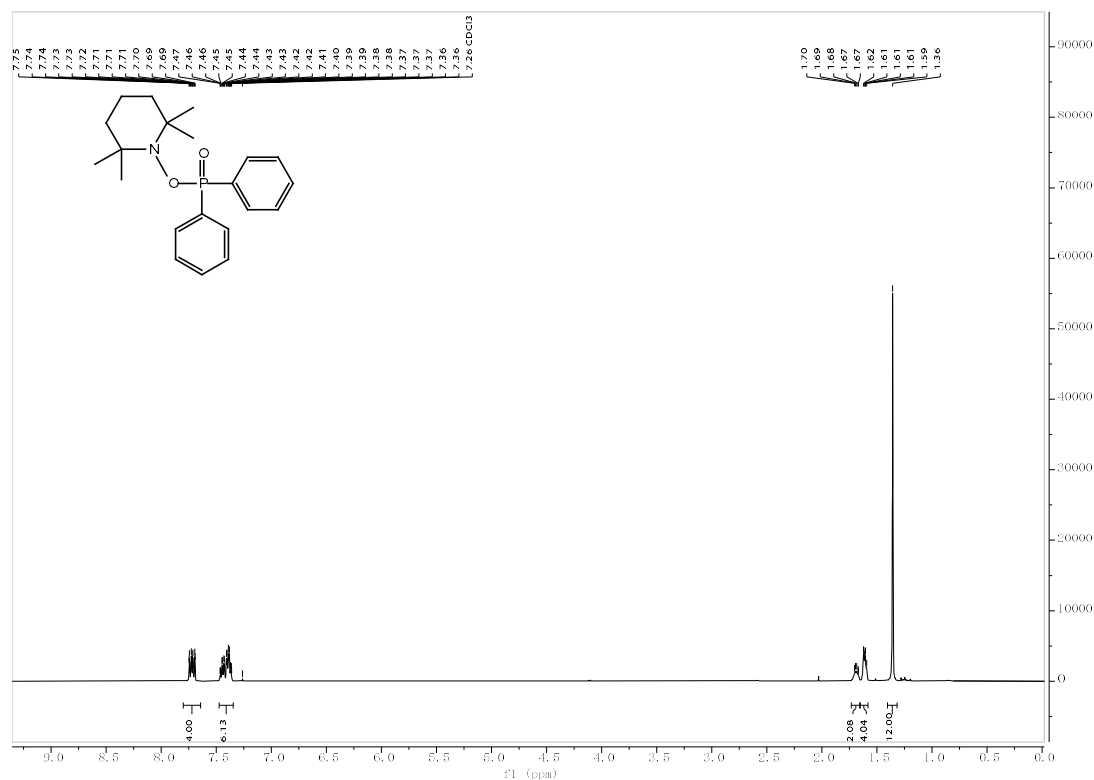
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 5:1) as eluant, white solid (31% yield, 22.2mg).

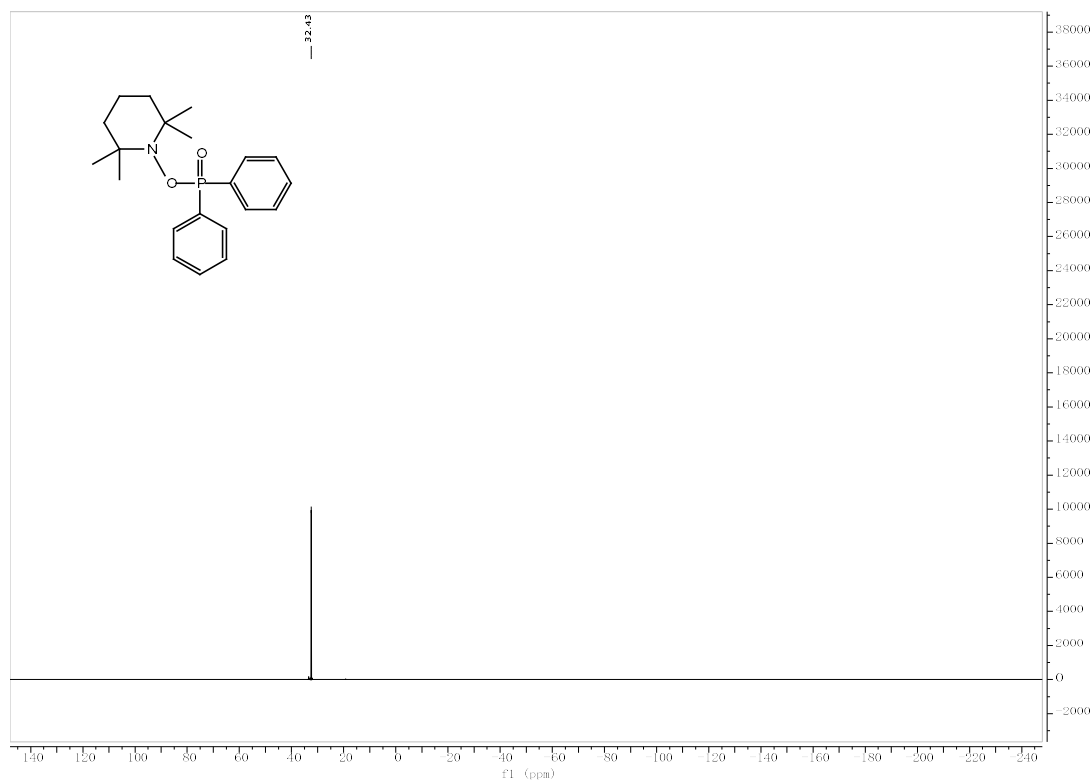
$^1\text{H NMR}$ (400 MHz, Chloroform-*d*) δ 7.75 – 7.69 (m, 4H), 7.47 – 7.26 (m, 6H), 1.70 – 1.67 (m, 2H), 1.62– 1.59 (m, 4H), 1.36 (s, 12H).

$^{31}\text{P NMR}$ (162 MHz, Chloroform-*d*) δ 32.4.

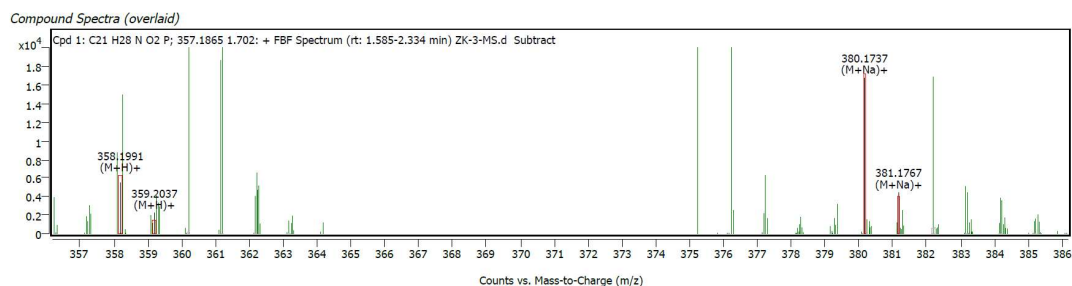
HRMS (ESI) m/z : 358.1991 $[\text{M}+\text{H}]^+$

(Known compound: *Org. Lett.* 2023, 25, 9, 1583–1588)





^{31}P NMR of **47** (162 MHz, CDCl_3)



HRMS (ESI) of **47**

(2) UV-vis experiment

UV-vis experiments were performed to analyze the potential association of PMDETA, with electron acceptors—nitrobenzene

Various combinations of **1**, PMDETA and chlorodiphenylphosphine in EA

- PMDETA (400 μL) was dissolved in EA (4.6 mL).
- chlorodiphenylphosphine (200 μL) was dissolved in EA (4.8 mL).
- 1** (100 μL) was dissolved in EA (4.9 mL).
- 1** (100 μL) and chlorodiphenylphosphine (200 μL) were dissolved in EA (4.70 mL).
- 1** (100 μL) and PMDETA (400 μL) were dissolved in EA (4.50 mL).

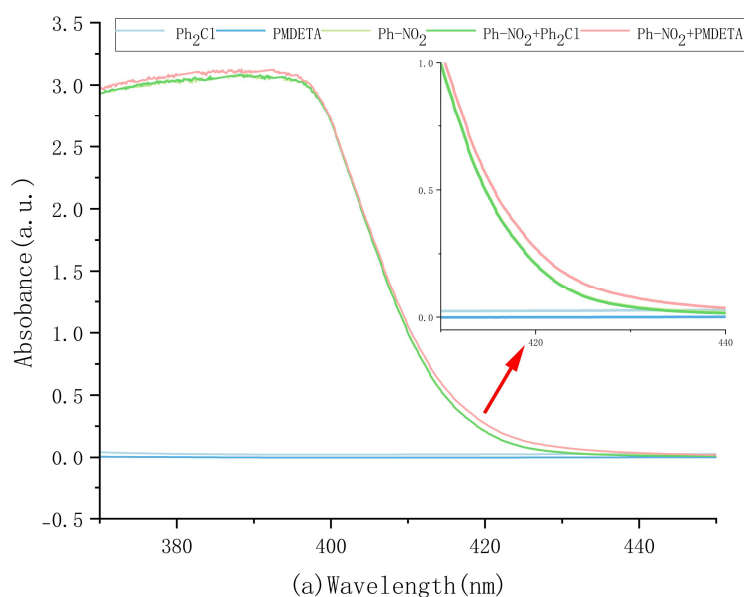


Figure S1 UV-vis spectra of various combinations of **1**, PMDETA and chlorodiphenylphosphine in EA

The results revealed that PMDETA can associate with **1** to form the EDA complex I, resulting a bathochromic shifted compared to the spectra of the individual components.

(3) Quantum yield experiment

Based on the report by Yoon et al. (*Chem. Sci.*, **2015**, 6, 5426-5434), the photon flux of the LED ($\lambda_{\text{max}} = 405 \text{ nm}$) was determined using the standard ferrous oxalate spectrophotometric method. First, potassium oxalate monohydrate (0.737 g) was dissolved in 10 mL of 0.05 M H_2SO_4 to prepare a 0.15 M ferrous oxalate solution. Subsequently, 1,10-phenanthroline (5.0 mg) and sodium acetate (1.13 g) were dissolved in 5.0 mL of 0.5 M H_2SO_4 to prepare the 1,10-phenanthroline buffer solution. Both solutions were stored in the dark.

To determine the photon flux of the LED, 3.0 mL of the ferrous oxalate solution was placed in a test tube and irradiated for 90 seconds with the LED at $\lambda_{\text{max}} = 405 \text{ nm}$. After irradiation, 0.525 mL of the 1,10-phenanthroline solution was added to the test tube, and the mixture was stirred in the dark for 1 hour to ensure complete complexation between ferrous ions and 1,10-phenanthroline. The absorbance of a diluted sample (200 μL of the resulting solution diluted to 3.5 mL with water) was measured at 510 nm. The absorbance measured at 510 nm for a non-irradiated sample was used as a blank control. The obtained absorbance experimental data are as follows:

	Non-irrad	Irrad 1	Irrad 2	Irrad 3
$A_{510 \text{ nm}}$	0.041	0.455	0.446	0.440
Average $A_{510 \text{ nm}}$ of irradiation samples	0.447			

Based on the molar absorptivity (ϵ) of the Fe^{2+} -1,10-phenanthroline complex at 510 nm, the molar amount of Fe^{2+} (mol Fe^{2+}) can be calculated using Equation (2-1).

$$\text{mol Fe}^{2+} = (V \times \Delta A) / (l \times \epsilon) \quad (2-1)$$

$$\text{mol Fe}^{2+} = (V \times \Delta A \times V_2) / (l \times \epsilon \times V_1)$$

$$\text{mol Fe}^{2+} = (3.525 \times 10^{-3} \times 0.406 \times 3.0 \times 10^{-3}) / (1 \times 11100 \times 200 \times 10^{-6}) = 1.93 \times 10^{-6} \text{ mol}$$

(V represents the total volume of the solution after the addition of the phenanthroline solution (3.525×10^{-3} L), V_1 is the volume of the sample taken for measurement before dilution ($200 \mu\text{L} = 200 \times 10^{-6}$ L), V_2 is the total volume after dilution for absorbance measurement ($3.0 \text{ mL} = 3.0 \times 10^{-3}$ L), ΔA is the absorbance difference between the irradiated and non-irradiated solutions at 510 nm, l is the path length (1.00 cm), and ϵ is the molar absorptivity of the ferrous oxalate complex at 510 nm ($11100 \text{ L} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$.)

The photon flux can be calculated using Equation (2-2).

$$\text{photo flux} = \text{mol Fe}^{2+} / \Phi (\text{Fe}^{2+}) \times t \times f \quad (2-2)$$

$$\text{photo flux} = 1.93 \times 10^{-6} / (1.13 \times 90 \times 0.942) = 2.01 \times 10^{-8} \text{ einstein s}^{-1}$$

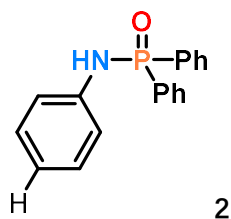
(Where Φ is the quantum yield of the ferrous oxalate complex (the quantum yield of a 0.15 M solution at $\lambda = 405 \text{ nm}$ is 1.13) (*Green Chem.*, **2024**, 26, 7198-7205), t is the exposure time (90.0 s), and f is the fraction of absorbed light at $\lambda = 405 \text{ nm}$ ($f = 1 - 10^{-A_{405 \text{ nm}}} = 0.94246$). The photon flux (average value from three experiments) is calculated to be $2.01 \times 10^{-8} \text{ einstein s}^{-1}$.)

Finally, nitrobenzene (0.2 mmol), PMDETA (0.4 mmol), and diphenylphosphine chloride (0.24 mmol) were tested at $\lambda_{\text{max}} = 405 \text{ nm}$. Under the irradiation of an LED lamp, the reaction lasted for 12000 seconds, and the nuclear magnetic resonance yield was calculated using 1,3,5-trimethoxybenzene as the internal standard. The yield of **2** was 16%. Finally, according to formula (2-3), the quantum yield of the reaction can be obtained.

$$\text{Quantum Yield} = \text{moles of product formed} / (\text{flux} \times f \times t) \quad (2-3)$$

$$\text{Quantum Yield} = 0.024 \times 10^{-3} / (2.01 \times 10^{-8} \times 0.942 \times 12000) = 0.11$$

III. Characterization data for the products



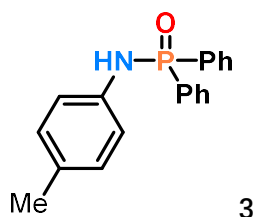
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (73% yield, 44.94 mg).

¹H NMR (400 MHz, Chloroform-*d*) δ 7.94 – 7.84 (m, 4H), 7.56 – 7.50 (m, 2H), 7.50 – 7.42 (m, 4H), 7.14 (t, J = 7.7 Hz, 2H), 6.98 (d, J = 7.9 Hz, 2H), 6.89 (t, J = 7.4 Hz, 1H), 5.37 (s, 1H).

¹³C NMR (100 MHz, Chloroform-*d*) δ 140.3, 132.3 (d, J = 2.7 Hz), 132.0 (d, J = 10.0 Hz), 131.9 (d, J = 133.7 Hz), 129.3, 128.9 (d, J = 13.0 Hz), 121.9, 118.5 (d, J = 6.6 Hz).

³¹P NMR (162 MHz, Chloroform-*d*) δ 18.5.

(Known compound: *J. Org. Chem.* **2024**, 89, 10, 6729–6739).



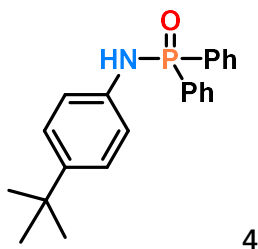
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (65% yield, 39.91 mg).

¹H NMR (400 MHz, DMSO-*d*₆) δ 8.12 (d, J = 11.7 Hz, 1H), 7.85 – 7.73 (m, 4H), 7.59 – 7.44 (m, 6H), 6.99 – 6.87 (m, 4H), 2.13 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 139.4, 133.1 (d, J = 126.6 Hz), 131.8 (d, J = 2.8 Hz), 131.7 (d, J = 9.8 Hz), 129.3, 129.2, 128.6 (d, J = 12.5 Hz), 118.4 (d, J = 6.9 Hz), 20.2.

³¹P NMR (162 MHz, DMSO-*d*₆) δ 16.2.

(Known compound: *J. Org. Chem.* **2024**, 89, 10, 6729–6739).



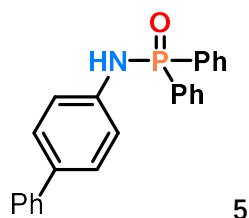
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (62% yield, 44.03 mg).

¹H NMR (400 MHz, Chloroform-*d*) δ 7.94 – 7.82 (m, 4H), 7.55 – 7.48 (m, 2H), 7.48 – 7.41 (m, 4H), 7.16 – 7.11 (m, 2H), 6.89 (d, *J* = 8.6 Hz, 2H), 5.39 (s, 1H), 1.22 (s, 9H).

¹³C NMR (100 MHz, Chloroform-*d*) δ 144.6, 137.5, 132.2 (d, *J* = 2.8 Hz), 132.1 (d, *J* = 129.4 Hz), 132.0 (d, *J* = 10.0 Hz), 128.8 (d, *J* = 13.0 Hz), 126.1, 118.2 (d, *J* = 6.5 Hz), 34.1, 31.4.

³¹P NMR (162 MHz, Chloroform-*d*) δ 18.6.

(Known compound: *Org. Lett.* **2022**, *24*, 49, 9130–9134).



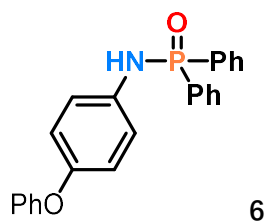
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (49% yield, 36.20 mg).

¹H NMR (400 MHz, DMSO-*d*₆) δ 8.39 (s, 1H), 7.82 (m, 4H), 7.65 – 7.33 (m, 12H), 7.31 – 7.07 (m, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 141.7, 139.8, 133.6, 132.4, 132.0, 131.7 (d, *J* = 10.0 Hz), 131.5 (d, *J* = 9.7 Hz), 128.7 (d, *J* = 11.1 Hz), 127.1, 126.6, 126.0, 118.6 (d, *J* = 7.0 Hz).

³¹P NMR (162 MHz, DMSO-*d*₆) δ 16.47.

(Known compound: *J. Org. Chem.* **2024**, *89*, 11, 7848–7858).



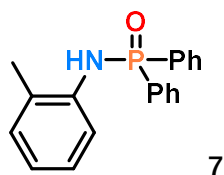
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (66% yield, 50.87 mg).

¹H NMR (400 MHz, Chloroform-*d*) δ 7.95 – 7.86 (m, 4H), 7.59 – 7.52 (m, 2H), 7.51 – 7.43 (m, 4H), 7.32 – 7.26 (m, 2H), 7.09 – 6.97 (m, 3H), 6.95 – 6.89 (m, 2H), 6.87 – 6.79 (m, 2H), 5.55 (s, 1H).

¹³C NMR (100 MHz, Chloroform-*d*) δ 158.0, 151.7, 136.2, 132.4 (d, *J* = 2.8 Hz), 132.1 (d, *J* = 10.0 Hz), 131.9 (d, *J* = 129.2 Hz), 129.7, 128.9 (d, *J* = 12.9 Hz), 122.8, 120.4, 120.3 (d, *J* = 6.3 Hz), 118.1.

³¹P NMR (162 MHz, Chloroform-*d*) δ 18.9.

HRMS: C₂₄H₂₀NO₂P [M+H]⁺; calculated: 386.1310, found: 386.1317.



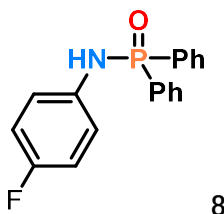
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (63% yield, 38.72mg).

¹H NMR (400 MHz, Chloroform-*d*) δ 7.94 – 7.84 (m, 4H), 7.56 – 7.49 (m, 2H), 7.49 – 7.42 (m, 4H), 7.20 (d, *J* = 8.0 Hz, 1H), 7.11 (d, *J* = 7.5 Hz, 1H), 6.97 – 6.91 (m, 1H), 6.87 – 6.75 (m, 1H), 5.06 (s, 1H), 2.28 (s, 3H).

¹³C NMR (100 MHz, Chloroform-*d*) δ 138.6, 132.2 (d, *J* = 2.9 Hz), 132.1 (d, *J* = 129.5 Hz), 131.9 (d, *J* = 9.9 Hz), 130.5, 128.8 (d, *J* = 12.9 Hz), 127.0, 125.5 (d, *J* = 7.9 Hz), 122.1, 118.9 (d, *J* = 4.2 Hz), 17.8.

³¹P NMR (162 MHz, Chloroform-*d*) δ 18.6.

(Known compound: *J. Org. Chem.* **2024**, *89*, 10, 6729–6739).



Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (84% yield, 52.30 mg).

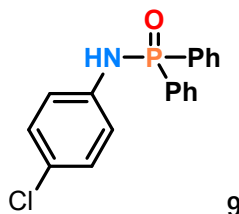
¹H NMR (400 MHz, Chloroform-*d*) δ 7.93 – 7.80 (m, 4H), 7.56 – 7.47 (m, 2H), 7.46 – 7.36 (m, 4H), 7.05 – 6.93 (m, 2H), 6.87 – 6.73 (m, 2H), 5.64 (s, 1H).

¹³C NMR (100 MHz, Chloroform-*d*) δ 158.4 (d, *J* = 240.6 Hz), 136.5, 132.4 (d, *J* = 2.8 Hz), 132.1 (d, *J* = 10.0 Hz), 131.1, 128.9 (d, *J* = 13.0 Hz), 120.9 – 119.9 (m), 116.0 (d, *J* = 22.7 Hz).

³¹P NMR (162 MHz, Chloroform-*d*) δ 18.9.

¹⁹F NMR (376 MHz, Chloroform-*d*) δ -121.8.

(Known compound: *J. Org. Chem.* **2024**, *89*, 11, 7848–7858).



Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (79% yield, 51.78 mg).

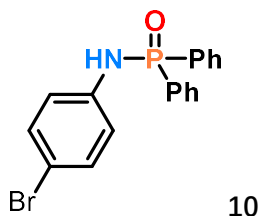
¹H NMR (400 MHz, DMSO-*d*₆) δ 8.42 (d, *J* = 11.6 Hz, 1H), 7.79 (m, 4H), 7.66 – 7.44 (m, 6H), 7.30 –

7.15 (m, 2H), 7.12 – 7.03 (m, 2H).

^{13}C NMR (100 MHz, DMSO- d_6) δ 141.2, 133.2, 132.0 (d, $J = 2.7$ Hz), 132.0, 131.6 (d, $J = 9.9$ Hz), 128.8 (d, $J = 12.2$ Hz), 124.4, 119.7 (d, $J = 7.0$ Hz).

^{31}P NMR (162 MHz, DMSO- d_6) δ 16.9.

(Known compound: *J. Org. Chem.* **2024**, 89, 11, 7848–7858).



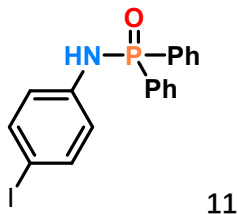
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (62% yield, 46.15 mg).

^1H NMR (400 MHz, DMSO- d_6) δ 8.44 (d, $J = 11.7$ Hz, 1H), 7.83 – 7.73 (m, 4H), 7.63 – 7.46 (m, 6H), 7.32 – 7.25 (m, 2H), 7.06 – 6.99 (m, 2H).

^{13}C NMR (100 MHz, DMSO- d_6) δ 141.6, 133.2, 132.1 (d, $J = 2.7$ Hz), 131.9, 131.7 (d, $J = 9.9$ Hz), 128.8 (d, $J = 12.5$ Hz), 120.2 (d, $J = 7.0$ Hz), 112.3.

^{31}P NMR (162 MHz, DMSO- d_6) δ 16.9.

(Known compound: *J. Org. Chem.* **2024**, 89, 11, 7848–7858).



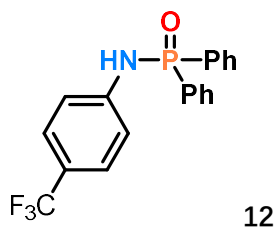
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (62% yield, 51.98 mg).

^1H NMR (400 MHz, DMSO- d_6) δ 8.41 (d, $J = 11.7$ Hz, 1H), 7.83 – 7.73 (m, 4H), 7.61 – 7.48 (m, 6H), 7.47 – 7.41 (m, 2H), 6.91 (d, $J = 8.5$ Hz, 2H).

^{13}C NMR (100 MHz, DMSO- d_6) δ 142.1, 137.4, 132.6 (d, $J = 126.5$ Hz), 132.0 (d, $J = 2.7$ Hz), 131.6 (d, $J = 9.9$ Hz), 128.8 (d, $J = 12.5$ Hz), 120.6 (d, $J = 7.0$ Hz), 83.5.

^{31}P NMR (162 MHz, DMSO- d_6) δ 16.8.

(Known compound: *J. Org. Chem.* **2024**, 89, 11, 7848–7858).



Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (70% yield, 50.58 mg).

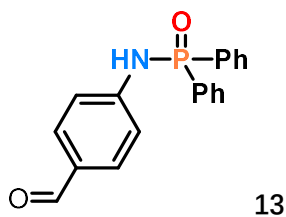
¹H NMR (400 MHz, DMSO-*d*₆) δ 8.76 (d, *J* = 11.8 Hz, 1H), 7.80 (m, 4H), 7.64 – 7.45 (m, 8H), 7.22 (d, *J* = 8.6 Hz, 2H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 146.1, 132.9, 132.2 (d, *J* = 2.9 Hz), 131.6 (d, *J* = 10.2 Hz), 128.8 (d, *J* = 12.7 Hz), 126.2 (d, *J* = 4.0 Hz), 124.6 (d, *J* = 271.1 Hz), 120.7 (q, *J* = 31.9 Hz), 117.9 (d, *J* = 7.2 Hz).

³¹P NMR (162 MHz, DMSO-*d*₆) δ 17.1.

¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -59.9.

(Known compound: *J. Org. Chem.* **2024**, *89*, 11, 7848–7858).



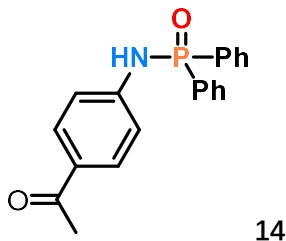
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (93% yield, 59.76 mg).

¹H NMR (400 MHz, Chloroform-*d*) δ 9.74 (s, 1H), 7.88 – 7.79 (m, 4H), 7.66 – 7.59 (m, 2H), 7.58 – 7.50 (m, 2H), 7.49 – 7.40 (m, 4H), 7.13 – 7.07 (m, 2H), 6.27 (s, 1H).

¹³C NMR (100 MHz, Chloroform-*d*) δ 191.0, 147.0, 132.8 (d, *J* = 2.8 Hz), 132.0 (d, *J* = 10.1 Hz), 131.7, 131.2 (d, *J* = 128.9 Hz), 130.4, 129.1 (d, *J* = 13.0 Hz), 118.2 (d, *J* = 6.6 Hz).

³¹P NMR (162 MHz, Chloroform-*d*) δ 19.2.

HRMS: C₂₀H₁₈NOP [M+H]⁺; calculated: 322.0997, found: 322.1002.



Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (88% yield, 59.02 mg).

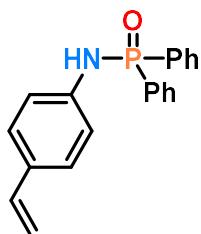
¹H NMR (400 MHz, Chloroform-*d*) δ 7.89 – 7.82 (m, 4H), 7.73 (d, *J* = 8.3 Hz, 2H), 7.58 – 7.50 (m, 2H),

7.48 – 7.41 (m, 4H), 7.02 (d, $J = 8.5$ Hz, 2H), 2.47 (s, 3H).

^{13}C NMR (100 MHz, Chloroform- d) δ 196.8, 145.3, 132.6 (d, $J = 2.9$ Hz), 131.9 (d, $J = 10.3$ Hz), 131.1 (d, $J = 126.4$ Hz), 130.9, 130.1, 129.0 (d, $J = 13.2$ Hz), 117.7 (d, $J = 6.6$ Hz), 26.2.

^{31}P NMR (162 MHz, Chloroform- d) δ 19.2.

(Known compound: *Chin Chem Lett.* **2007**, *18*,1033–1036).



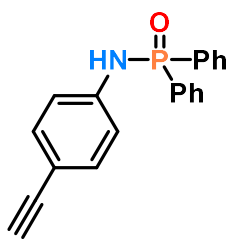
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (74% yield, 47.26 mg).

^1H NMR (400 MHz, Chloroform- d) δ 7.91 – 7.82 (m, 4H), 7.57 – 7.49 (m, 2H), 7.44 (ddd, $J = 8.5, 6.7, 3.4$ Hz, 4H), 7.21 – 7.16 (m, 2H), 6.98 – 6.92 (m, 2H), 6.57 (dd, $J = 17.6, 10.9$ Hz, 1H), 5.60 – 5.50 (m, 2H), 5.08 (dd, $J = 10.9, 0.9$ Hz, 1H).

^{13}C NMR (100 MHz, Chloroform- d) 140.2, 136.3, 132.4 (d, $J = 2.8$ Hz), 132.1 (d, $J = 10.1$ Hz), 131.9 (d, $J = 129.4$ Hz), 131.5, 128.9 (d, $J = 13.0$ Hz), 127.3, 118.6 (d, $J = 6.6$ Hz), 112.0.

^{31}P NMR (162 MHz, Chloroform- d) δ 18.5.

HRMS: $\text{C}_{20}\text{H}_{18}\text{NOP}$ $[\text{M}+\text{H}]^+$; calculated: 320.1204, found: 320.1199.



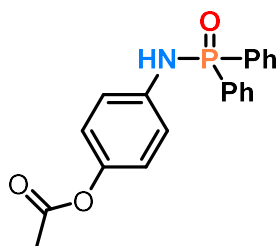
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (79% yield, 50.14 mg).

^1H NMR (400 MHz, DMSO- d_6) δ 7.66 (d, $J = 11.8$ Hz, 1H), 6.97 – 6.82 (m, 4H), 6.74 – 6.64 (m, 6H), 6.37 (d, $J = 8.5$ Hz, 2H), 6.18 (s, 2H), 3.10 (s, 1H).

^{13}C NMR (100 MHz, DMSO- d_6) ^{13}C NMR (100 MHz, DMSO- d_6) δ 143.0, 132.5 (d, $J = 126.5$ Hz), 132.5, 132.1 (d, $J = 2.7$ Hz), 131.6 (d, $J = 9.9$ Hz), 128.8 (d, $J = 12.6$ Hz), 118.1 (d, $J = 7.2$ Hz), 113.5, 83.8, 79.2.

^{31}P NMR (162 MHz, DMSO- d_6) δ 16.7.

HRMS: $\text{C}_{20}\text{H}_{16}\text{NOP}$ $[\text{M}+\text{H}]^+$; calculated: 318.1048, found: 318.1046.



17

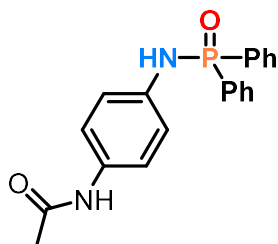
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (80% yield, 56.21 mg).

¹H NMR (400 MHz, DMSO-*d*₆) δ 8.32 (dd, *J* = 11.5, 2.2 Hz, 1H), 7.86 – 7.76 (m, 4H), 7.65 – 7.44 (m, 6H), 7.13 – 7.03 (m, 2H), 6.95 – 6.84 (m, 2H), 2.19 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 169.4, 144.2, 139.7, 133.5, 132.3 131.9 (d, *J* = 2.8 Hz), 131.6 (d, *J* = 9.9 Hz), 128.7 (d, *J* = 12.5 Hz), 122.0, 118.7 (d, *J* = 7.0 Hz), 20.7.

³¹P NMR (162 MHz, DMSO-*d*₆) δ 16.6.

HRMS: C₂₀H₁₈NO₃P [M+H]⁺; calculated: 352.1102, found: 352.1098.



18

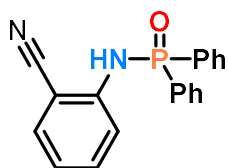
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (78% yield, 54.66mg).

¹H NMR (400 MHz, DMSO-*d*₆) δ 9.68 (s, 1H), 8.13 (d, *J* = 11.5 Hz, 1H), 7.94 – 7.71 (m, 4H), 7.66 – 7.40 (m, 6H), 7.34 – 7.24 (m, 2H), 7.14 – 6.90 (m, 2H), 1.95 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆) ¹³C NMR (100 MHz, DMSO-*d*₆) δ 169.4, 144.2, 139.7, 132.9 (d, *J* = 126.5 Hz), 132.0 (d, *J* = 2.8 Hz), 131.6 (d, *J* = 9.9 Hz), 128.7 (d, *J* = 12.5 Hz), 122.1, 118.8 (d, *J* = 7.0 Hz), 20.8.

³¹P NMR (162 MHz, DMSO-*d*₆) δ 16.3.

HRMS: C₂₀H₁₈N₂O₂P [M+H]⁺; calculated: 351.1262, found: 351.1259.



19

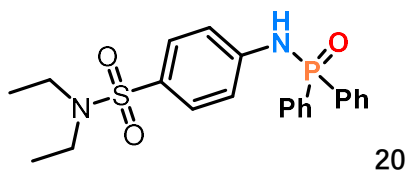
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (74% yield, 47.11mg).

¹H NMR (400 MHz, Chloroform-*d*) δ 7.96 – 7.86 (m, 4H), 7.68 – 7.55 (m, 1H), 7.55 – 7.47 (m, 6H), 7.44 (d, *J* = 8.4 Hz, 1H), 7.36 – 7.30 (m, 1H), 6.98 (d, *J* = 144.7 Hz, 1H), 5.93 (d, *J* = 9.5 Hz, 1H).

¹³C NMR (100 MHz, Chloroform-*d*) δ 144.0, 134.3, 132.9 (d, *J* = 2.9 Hz), 132.7, 131.9 (d, *J* = 10.2 Hz), 130.9 (d, *J* = 129.2 Hz), 129.2 (d, *J* = 13.2 Hz), 122.1, 119.0 (d, *J* = 4.1 Hz), 117.0, 101.5 (d, *J* = 8.1 Hz).

³¹P NMR (162 MHz, Chloroform-*d*) δ 19.6.

(Known compound: *Eur. J. Inorg. Chem.* **2004**, 530-537).



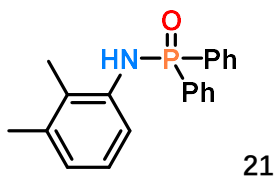
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (84% yield, 71.99mg).

¹H NMR (400 MHz, Methanol-*d*₄) δ 7.91 – 7.79 (m, 4H), 7.65 – 7.48 (m, 8H), 7.22 – 7.12 (m, 2H), 3.14 (q, *J* = 7.1 Hz, 4H), 1.04 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (100 MHz, Methanol-*d*₄) δ 147.1, 133.9 (d, *J* = 2.9 Hz), 133.6, 133.0 (d, *J* = 10.4 Hz), 132.0 (d, *J* = 130.1 Hz), 130.1 (d, *J* = 13.2 Hz), 129.5, 119.4 (d, *J* = 7.3 Hz), 43.2, 14.6.

³¹P NMR (162 MHz, Methanol-*d*₄) δ 21.4.

HRMS: C₂₂H₂₅N₂O₃PS[M+H]⁺; calculated: 429.1401, found: 429.1401.



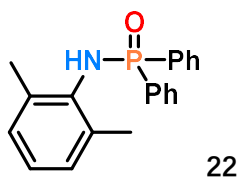
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (58% yield, 37.28mg).

¹H NMR (400 MHz, Chloroform-*d*) δ 7.93 – 7.84 (m, 4H), 7.55 – 7.48 (m, 2H), 7.47 – 7.41 (m, 4H), 7.12 (d, *J* = 8.1 Hz, 1H), 6.83 (t, *J* = 7.7 Hz, 1H), 6.75 (d, *J* = 7.5 Hz, 1H), 5.08 (d, *J* = 8.4 Hz, 1H), 2.27 (s, 3H), 2.19 (s, 3H).

¹³C NMR (100 MHz, Chloroform-*d*) δ 138.4, 137.1, 132.3 (d, *J* = 129.0 Hz), 132.1 (d, *J* = 2.4 Hz), 131.9 (d, *J* = 9.8 Hz), 128.8 (d, *J* = 12.8 Hz), 126.0, 124.5 (d, *J* = 7.5 Hz), 124.2, 117.5 (d, *J* = 3.9 Hz), 20.8, 13.3.

³¹P NMR (162 MHz, Chloroform-*d*) δ 18.6.

HRMS: C₂₀H₁₈N₂O₂P[M+H]⁺; calculated: 322.1361, found: 322.1359.



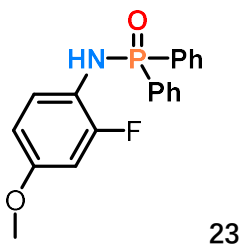
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (20% yield, 12.85mg).

¹H NMR (400 MHz, Chloroform-*d*) δ 7.85 – 7.78 (m, 4H), 7.52 – 7.46 (m, 2H), 7.44 – 7.37 (m, 4H), 6.97 – 6.91 (m, 3H), 4.60 (d, *J* = 5.3 Hz, 1H), 2.27 (s, 6H).

¹³C NMR (100 MHz, Chloroform-*d*) δ 136.3 (d, *J* = 4.2 Hz), 135.1 (d, *J* = 3.6 Hz), 133.3, 132.0 (d, *J* = 1.8 Hz), 131.9, 128.8, 128.5 (d, *J* = 12.8 Hz), 125.6 (d, *J* = 1.5 Hz), 20.2..

³¹P NMR (162 MHz, Chloroform-*d*) δ 20.4.

(Known compound: *New J. Chem.*, **2012**, *36*, 2280-2285).



Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (80% yield, 54.61mg).

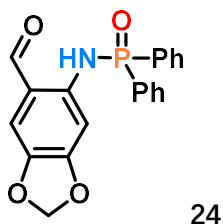
¹H NMR (400 MHz, Chloroform-*d*) δ 7.99 – 7.84 (m, 4H), 7.57 – 7.50 (m, 2H), 7.50 – 7.42 (m, 4H), 7.20 (t, *J* = 9.2 Hz, 1H), 6.63 (d, 1H), 6.50 – 6.37 (m, 1H), 5.21 (d, *J* = 9.1 Hz, 1H), 3.68 (s, 3H).

¹³C NMR (100 MHz, Chloroform-*d*) δ 155.4 (d, *J* = 10.3 Hz), 154.9 (d, *J* = 10.2 Hz), 152.5 (d, *J* = 7.8 Hz), 132.5 (d, *J* = 2.4 Hz), 132.1 (d, *J* = 9.8 Hz), 129.0 (d, *J* = 12.9 Hz), 121.5 (d, *J* = 13.0 Hz), 121.3, 109.5 (d, *J* = 3.1 Hz), 102.4 (d, *J* = 22.8 Hz), 55.8.

³¹P NMR (162 MHz, Chloroform-*d*) δ 19.3.

¹⁹F NMR (376 MHz, Chloroform-*d*) δ -129.1.

HRMS: C₁₉H₁₇FNO₂P [M+H]⁺; calculated: 342.1059, found: 342.1055.



Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA

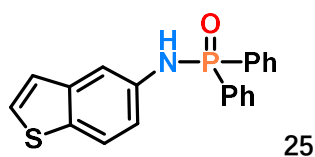
= 1:1) as eluant, white solid (85% yield, 62.11mg).

¹H NMR (400 MHz, Chloroform-*d*) δ 9.72 (s, 1H), 7.87 – 7.78 (m, 4H), 7.60 (d, *J* = 8.1 Hz, 2H), 7.56 – 7.50 (m, 2H), 7.47 – 7.40 (m, 4H), 7.13 – 7.04 (m, 2H), 6.50 (s, 1H).

¹³C NMR (100 MHz, Chloroform-*d*) δ 191.0, 147.1, 132.8 (d, *J* = 2.5 Hz), 132.0 (d, *J* = 10.2 Hz), 131.6, 130.3, 129.1 (d, *J* = 13.1 Hz), 118.2 (d, *J* = 7.0 Hz), 118.1.

³¹P NMR (162 MHz, Chloroform-*d*) δ 19.3.

HRMS: C₂₀H₁₆NO₄P [M+H]⁺; calculated: 366.0895, found: 366.0885.



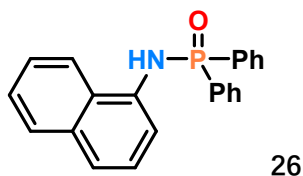
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (68% yield, 47.52mg).

¹H NMR (400 MHz, DMSO-*d*₆) δ 8.34 (d, *J* = 11.7 Hz, 1H), 7.86 – 7.77 (m, 4H), 7.74 (d, *J* = 8.7 Hz, 1H), 7.64 (d, *J* = 5.4 Hz, 1H), 7.59 – 7.46 (m, 7H), 7.25 – 7.18 (m, 2H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 140.0, 139.1, 133.6, 132.3, 131.9 (d, *J* = 2.8 Hz), 131.7 (d, *J* = 10.0 Hz), 128.6 (d, *J* = 12.4 Hz), 128.0, 123.5, 122.7, 117.2 (d, *J* = 7.4 Hz), 112.0 (d, *J* = 6.7 Hz).

³¹P NMR (162 MHz, DMSO-*d*₆) δ 16.8.

HRMS: C₂₀H₁₆NOPS [M+H]⁺; calculated: 350.0748, found: 350.0744.

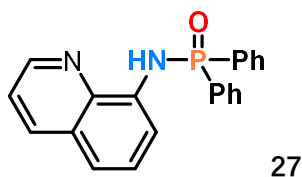


Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (57% yield, 39.17mg).

¹H NMR (400 MHz, Chloroform-*d*) δ 8.02 – 7.89 (m, 5H), 7.87 – 7.80 (m, 1H), 7.58 – 7.37 (m, 10H), 7.23 – 7.14 (m, 1H), 5.96 – 5.69 (m, 1H).

¹³C NMR (100 MHz, Chloroform-*d*) δ 135.5, 134.4, 132.4 (d, *J* = 3.5 Hz), 132.1 (d, *J* = 9.8 Hz), 132.1 (d, *J* = 130.0 Hz), 129.1 (d, *J* = 2.9 Hz), 129.0 (d), 126.2, 123.2, 120.4, 117.0. **³¹P NMR** (162 MHz, Chloroform-*d*) δ 19.0.

(Known compound: *J. Org. Chem.* **2024**, *89*, 10, 6729–6739).



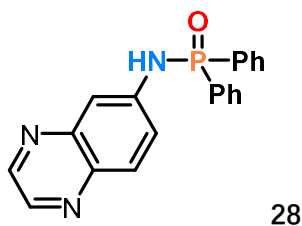
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (87% yield, 57.85mg).

¹H NMR (400 MHz, Chloroform-*d*) δ 8.74 (dd, $J = 4.3, 1.6$ Hz, 1H), 8.09 (dd, $J = 8.3, 1.7$ Hz, 1H), 8.01 (d, $J = 13.3$ Hz, 1H), 7.95 (m, 4H), 7.60 – 7.50 (m, 2H), 7.49 – 7.43 (m, 4H), 7.42 – 7.36 (m, 2H), 7.33 – 7.20 (m, 2H).

¹³C NMR (100 MHz, Chloroform-*d*) δ 148.1, 138.8 (d, $J = 7.3$ Hz), 137.8, 136.4, 132.3 (d, $J = 2.9$ Hz), 132.2 (d, $J = 129.0$ Hz), 131.9 (d, $J = 10.2$ Hz), 128.9 (d, $J = 13.1$ Hz), 128.5, 127.2, 121.8, 119.5, 113.9 (d, $J = 3.8$ Hz).

³¹P NMR (162 MHz, Chloroform-*d*) δ 19.0.

(Known compound: *J. Org. Chem.* **2019**, *84*, 16, 10481–10489).



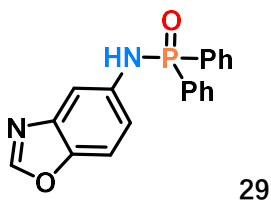
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (79% yield, 54.56mg).

¹H NMR (400 MHz, Chloroform-*d*) δ 8.60 (d, $J = 2.0$ Hz, 1H), 8.56 (d, $J = 2.0$ Hz, 1H), 7.93 – 7.82 (m, 5H), 7.77 (dd, $J = 9.0, 2.6$ Hz, 1H), 7.64 – 7.54 (m, 2H), 7.52 – 7.44 (m, 2H), 7.43 – 7.36 (m, 5H), 6.90 (d, $J = 9.9$ Hz, 1H).

¹³C NMR (100 MHz, Chloroform-*d*) δ 107.5, 106.2, 105.3, 105.1, 101.5, 94.9 (d, $J = 2.8$ Hz), 94.4 (d, $J = 10.3$ Hz), 93.1, 92.8, 91.3 (d, $J = 13.1$ Hz), 86.3 (d, $J = 6.6$ Hz), 76.6 (d, $J = 6.9$ Hz).

³¹P NMR (162 MHz, Chloroform-*d*) δ 19.19.

HRMS: C₂₀H₁₆N₃OP [M+H]⁺; calculated: 346.1109, found: 346.1106.



Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA

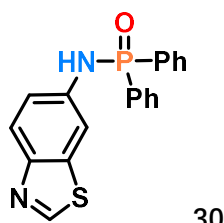
= 1:1) as eluant, white solid (55% yield, 36.77mg).

¹H NMR (400 MHz, Chloroform-*d*) δ 7.95 (s, 1H), 7.93 – 7.84 (m, 4H), 7.46 (dtd, J = 31.6, 7.5, 2.0 Hz, 7H), 7.30 (d, J = 8.7 Hz, 1H), 7.14 (m, 1H), 5.85 (d, J = 9.0 Hz, 1H).

¹³C NMR (100 MHz, Chloroform-*d*) δ 153.3, 145.8, 140.8, 137.8, 132.5 (d, J = 2.7 Hz), 132.2 (d, J = 10.0 Hz), 131.7 (d, J = 129.6 Hz), 129.0 (d, J = 12.9 Hz), 117.9 (d, J = 6.4 Hz), 111.3, 110.4 (d, J = 6.9 Hz).

³¹P NMR (162 MHz, Chloroform-*d*) δ 19.0.

HRMS: C₁₉H₁₅N₂O₂P [M+H]⁺; calculated: 335.0895, found: 335.0947.



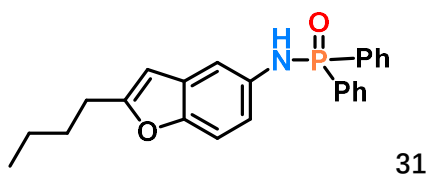
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (41% yield, 28.73mg).

¹H NMR (400 MHz, DMSO-*d*₆) δ 9.11 (s, 1H), 8.33 (s, 1H), 7.91 – 7.77 (m, 5H), 7.73 (d, J = 2.2 Hz, 1H), 7.61 – 7.48 (m, 6H), 7.34 (dd, J = 8.8, 2.2 Hz, 1H).

¹³C NMR (100 MHz, DMSO-*d*₆) δ 153.0, 147.7, 140.4, 133.9 (d, J = 120.2 Hz), 132.1 (d, J = 2.8 Hz), 131.7 (d, J = 9.9 Hz), 128.8 (d, J = 12.5 Hz), 123.2, 118.4 (d, J = 7.6 Hz), 109.9 (d, J = 7.0 Hz), 79.2.

³¹P NMR (162 MHz, DMSO-*d*₆) δ 17.4.

HRMS: C₁₉H₁₅N₂O₂PS [M+H]⁺; calculated: 351.0721, found: 351.0718.



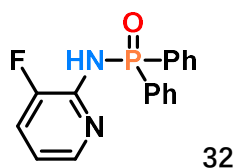
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (53% yield, 41.28mg).

¹H NMR (400 MHz, Chloroform-*d*) δ 7.97 – 7.87 (m, 4H), 7.57 – 7.41 (m, 6H), 7.21 – 7.15 (m, 2H), 6.95 – 6.87 (m, 1H), 6.20 (s, 1H), 5.39 (d, J = 8.8 Hz, 1H), 2.70 (t, J = 7.6 Hz, 2H), 1.71 – 1.64 (m, 2H), 1.45 – 1.33 (m, 2H), 0.94 (t, J = 7.3 Hz, 3H).

¹³C NMR (100 MHz, Chloroform-*d*) δ 160.7, 150.8, 135.3, 132.2 (d, J = 3.4 Hz), 132.2, 129.8, 128.9 (d, J = 12.8 Hz), 115.8 (d, J = 6.7 Hz), 111.5, 111.1, 110.7, 102.0, 29.8, 28.3, 22.4, 13.9.

³¹P NMR (162 MHz, Chloroform-*d*) δ 18.7.

HRMS: C₂₄H₂₄NO₂P [M+H]⁺; calculated: 390.1623, found: 390.1618.



Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (86% yield, 53.71mg).

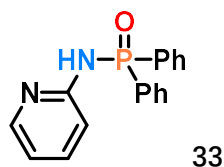
¹H NMR (400 MHz, Chloroform-*d*) δ 7.98 – 7.86 (m, 4H), 7.82 (d, *J* = 5.0 Hz, 1H), 7.59 – 7.49 (m, 2H), 7.43 (d, *J* = 3.9 Hz, 4H), 7.33 – 7.22 (m, 1H), 6.82 – 6.66 (m, 1H), 6.05 (s, 1H).

¹³C NMR (100 MHz, Chloroform-*d*) δ 147.5 (dd, *J* = 254.1, 6.5 Hz), 143.8 (d, *J* = 11.8 Hz), 143.3 (d, *J* = 6.0 Hz), 132.2 (d, *J* = 2.9 Hz), 132.0 (d, *J* = 131.0 Hz), 131.8 (d, *J* = 10.3 Hz), 128.7 (d, *J* = 13.3 Hz), 122.3 (d, *J* = 15.8 Hz), 117.2.

¹⁹F NMR (376 MHz, Chloroform-*d*) δ -137.39.

³¹P NMR (162 MHz, Chloroform-*d*) δ 19.3.

HRMS: C₁₇H₁₄FN₂OP [M+H]⁺; calculated: 314.0906, found: 314.0908.



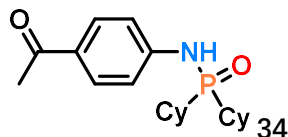
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (81% yield, 47.68mg).

¹H NMR (400 MHz, Chloroform-*d*) δ 8.04 – 7.88 (m, 4H), 7.73 – 7.65 (m, 1H), 7.61 – 7.53 (m, 2H), 7.53 – 7.44 (m, 4H), 7.43 – 7.35 (m, 1H), 7.06 (d, *J* = 8.3 Hz, 1H), 6.73 – 6.62 (m, 1H).

¹³C NMR (100 MHz, Chloroform-*d*) δ 154.2 (d, *J* = 2.5 Hz), 147.7 (d, *J* = 1.9 Hz), 138.0, 132.3, 131.9 (d, *J* = 10.3 Hz), 131.0, 128.8 (d, *J* = 13.1 Hz), 117.0, 112.1 (d, *J* = 2.9 Hz).

³¹P NMR (162 MHz, Chloroform-*d*) δ 18.9.

(Known compound: *Org. Lett.* **2023**, 25, 31, 5724–5729).



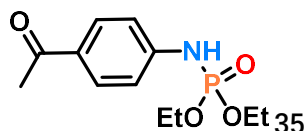
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (77% yield, 53.51mg).

¹H NMR (400 MHz, DMSO-*d*₆) δ 7.75 (d, *J* = 8.8 Hz, 2H), 7.46 (d, *J* = 12.6 Hz, 1H), 7.28 (d, *J* = 8.7 Hz, 2H), 2.45 (s, 3H), 1.95 – 1.66 (m, 11H), 1.37 – 1.02 (m, 11H).

^{13}C NMR (100 MHz, DMSO- d_6) δ 196.0, 150.1, 129.7, 128.5, 116.7 (d, J = 5.1 Hz), 35.9 (d, J = 82.1 Hz), 26.2, 25.9, 25.8 (d, J = 4.0 Hz), 25.6 (d, J = 7.4 Hz), 25.1 (d, J = 3.3 Hz), 24.8 (d, J = 2.9 Hz).

^{31}P NMR (162 MHz, DMSO- d_6) δ 46.1.

HRMS: $\text{C}_{20}\text{H}_{30}\text{NO}_2\text{P}$ [M+H] $^+$; calculated: 348.2092, found: 348.2106.



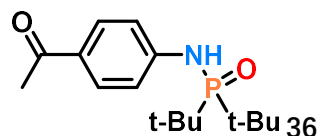
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, Yellow oily liquid (77% yield, 48.83mg).

^1H NMR (400 MHz, DMSO- d_6) δ 8.55 (d, J = 9.4 Hz, 1H), 7.84 (d, J = 8.7 Hz, 2H), 7.10 (d, J = 8.7 Hz, 2H), 4.13 – 3.91 (m, 4H), 2.48 (s, 3H), 1.22 (t, J = 7.1 Hz, 6H).

^{13}C NMR (100 MHz, DMSO- d_6) δ 196.2, 145.9, 129.9, 129.6, 116.4 (d, J = 7.8 Hz), 62.3 (d, J = 5.2 Hz), 26.3, 16.0 (d, J = 6.6 Hz).

^{31}P NMR (162 MHz, DMSO- d_6) δ 1.29.

HRMS: $\text{C}_{12}\text{H}_{18}\text{N O}_4\text{P}$ [M+H] $^+$; calculated: 271.1051, found: 272.1046.



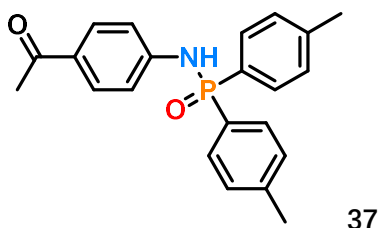
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (37% yield, 21.86 mg).

^1H NMR (400 MHz, DMSO- d_6) δ 8.91 (s, 1H), 6.97 (d, J = 8.5 Hz, 2H), 6.35 (d, J = 8.6 Hz, 2H), 1.61 (s, 3H), 0.39 (d, J = 13.2 Hz, 18H).

^{13}C NMR (100 MHz, DMSO- d_6) ^{13}C NMR (100 MHz, DMSO- d_6) δ 195.9, 155.7 (d, J = 6.7 Hz), 129.5, 128.4, 112.8, 50.0 (d, J = 65.6 Hz), 35.2 (d, J = 56.8 Hz), 26.0.

^{31}P NMR (162 MHz, DMSO- d_6) δ 59.42.

HRMS: $\text{C}_{16}\text{H}_{26}\text{NO}_2\text{P}$ [M+H] $^+$; calculated: 296.1179, found: 292.1175.



Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (56% yield, 40.70 mg)

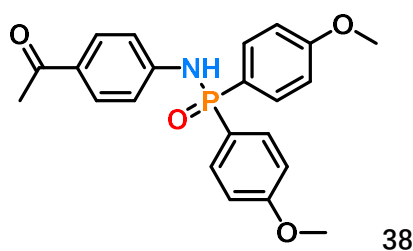
^1H NMR (400 MHz, Chloroform- d) δ 7.70 – 7.64 (m, 6H), 7.19 – 7.16(m, 4H), 7.03 (d, J = 8.4

Hz, 2H), 6.83 (s, 1H), 2.40 (s, 3H), 2.33 (s, 6H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 197.0, 146.2, 142.9 (d, $J = 2.9$ Hz), 132.0 (d, $J = 10.6$ Hz), 130.4, 130.1, 129.6 (d, $J = 13.4$ Hz), 129.0, 117.7 (d, $J = 6.8$ Hz), 26.2, 21.7.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 19.5.

HRMS: $\text{C}_{22}\text{H}_{22}\text{NO}_2\text{P}$ [M+H] $^+$; calculated: 363.1388, found: 363.1386.



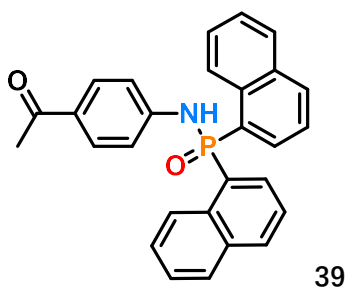
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (56% yield, 39.54 mg)

^1H NMR (400 MHz, Chloroform-*d*) δ 7.75 – 7.69 (m, 6H), 7.04 (d, $J = 8.7$ Hz, 2H), 6.91 (m, 4H), 6.54 (d, $J = 10.2$ Hz, 1H), 3.80 (s, 6H), 2.45 (s, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 197.0, 162.8 (d, $J = 3.0$ Hz), 146.2, 133.8 (d, $J = 11.5$ Hz), 130.5, 130.1, 122.9 (d, $J = 136.7$ Hz), 117.7 (d, $J = 6.8$ Hz), 114.4 (d, $J = 14.1$ Hz), 55.4,

^{31}P NMR (162 MHz, Chloroform-*d*) δ 19.4

HRMS: $\text{C}_{22}\text{H}_{22}\text{NO}_4\text{P}$ [M+H] $^+$; calculated: 395.1286, found: 365.1277.



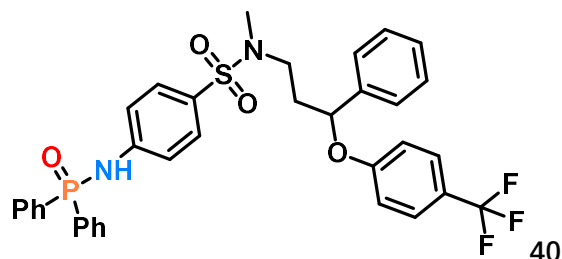
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (63% yield, 54.87 mg)

^1H NMR (400 MHz, DMSO-*d*₆) δ 9.04 (d, $J = 13.1$ Hz, 1H), 8.87 – 8.85 (m, 2H), 8.21 (d, $J = 8.2$ Hz, 2H), 8.19 – 8.06 (m, 2H), 7.77 (d, $J = 8.7$ Hz, 2H), 7.69 – 7.52 (m, 8H), 7.32 – 7.29 (m, 2H), 2.44 (s, 3H).

^{13}C NMR (100 MHz, DMSO-*d*₆) δ 196.1, 147.8, 133.6 (d, $J = 9.9$ Hz), 133.4, 133.2 (d, $J = 23.8$ Hz), 133.1 (d, $J = 2.9$ Hz), 129.8, 129.4, 129.0, 128.7, 127.4, 126.9 (d, $J = 71.1$ Hz), 126.9 (d, $J = 4.9$ Hz), 124.9 (d, $J = 14.8$ Hz), 117.3 (d, $J = 6.7$ Hz), 26.2.

^{31}P NMR (162 MHz, DMSO- d_6) δ 23.4.

HRMS: $\text{C}_{22}\text{H}_{22}\text{NO}_4\text{P}$ $[\text{M}+\text{H}]^+$; calculated: 435.1388, found: 435.1393.



Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (70% yield, 93.06mg).

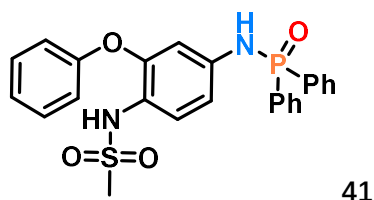
^1H NMR (400 MHz, DMSO- d_6) δ 8.86 (d, J = 11.8 Hz, 1H), 7.89 – 7.40 (m, 15H), 7.40 – 7.11 (m, 6H), 7.05 (d, J = 8.5 Hz, 2H), 5.45 (dd, J = 8.5, 4.3 Hz, 1H), 3.19 – 3.09 (m, 1H), 3.03 – 2.94 (m, 1H), 2.61 (s, 3H), 2.14 – 2.02 (m, 1H), 1.98 – 1.87 (m, 1H).

^{13}C NMR (100 MHz, DMSO- d_6) δ 160.3, 146.7, 140.4, 132.8, 132.2 (d, J = 2.8 Hz), 131.7 – 131.33 (m), 130.2 (d, J = 11.4 Hz), 129.0, 128.9, 128.8, 128.6, 128.5, 127.8 (d, J = 4.1 Hz), 126.8 (d, J = 4.0 Hz), 126.0, 117.8 (d, J = 7.2 Hz), 116.2, 76.6, 46.4, 35.8, 34.8.

^{31}P NMR (162 MHz, DMSO- d_6) δ 17.29.

^{19}F NMR (376 MHz, DMSO- d_6) δ -59.88.

HRMS: $\text{C}_{35}\text{H}_{32}\text{F}_3\text{N}_2\text{O}_4\text{PS}$ $[\text{M}+\text{H}]^+$; calculated: 665.1850, found: 665.1836.



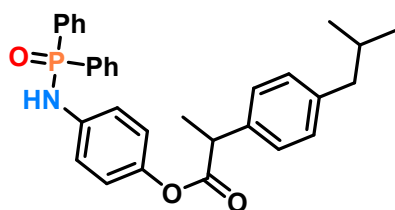
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (65% yield, 62.21mg).

^1H NMR (400 MHz, DMSO- d_6) δ 9.03 (s, 1H), 8.39 (d, J = 11.5 Hz, 1H), 7.78 – 7.69 (m, 4H), 7.62 – 7.46 (m, 6H), 7.32 (t, J = 7.9 Hz, 2H), 7.13 (dd, J = 10.2, 8.0 Hz, 2H), 6.84 (d, J = 8.0 Hz, 2H), 6.79 (dd, J = 8.6, 2.5 Hz, 1H), 6.63 (d, J = 2.5 Hz, 1H), 2.87 (s, 3H).

^{13}C NMR (100 MHz, DMSO- d_6) δ 153.6 (d, J = 421.1 Hz), 141.6, 133.1, 132.5, 132.0 (d, J = 2.7 Hz), 131.8, 131.6 (d, J = 9.9 Hz), 130.3 (d, J = 11.5 Hz), 129.9, 129.1, 129.0 (d, J = 12.5 Hz), 128.7 (d, J = 12.6 Hz), 123.6, 120.1, 118.7, 110.6 (dd, J = 514.1, 6.9 Hz).

^{31}P NMR (162 MHz, DMSO- d_6) δ 16.4.

HRMS: $\text{C}_{25}\text{H}_{23}\text{N}_2\text{O}_4\text{PS}$ $[\text{M}+\text{H}]^+$; calculated: 479.1194, found: 479.1192.



42

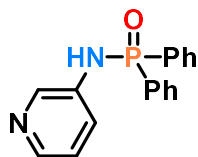
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (67% yield, 66.68mg).

$^1\text{H NMR}$ (400 MHz, Chloroform-*d*) δ 7.92 – 7.77 (m, 4H), 7.54 – 7.45 (m, 2H), 7.42 (q, J = 3.8 Hz, 4H), 7.31 – 7.20 (m, 2H), 7.16 – 7.04 (m, 2H), 7.01 – 6.87 (m, 2H), 6.75 (dd, J = 8.9, 2.4 Hz, 2H), 5.85 – 5.49 (m, 1H), 3.86 (q, J = 7.1 Hz, 1H), 2.45 (d, J = 7.2 Hz, 2H), 1.85 (dt, J = 13.5, 6.8 Hz, 1H), 1.55 (d, J = 7.1 Hz, 3H), 0.89 (d, J = 6.6 Hz, 6H).

$^{13}\text{C NMR}$ (100 MHz, Chloroform-*d*) δ 173.5, 145.5, 140.9, 138.2, 137.3, 132.4, 132.1 (d, J = 9.9 Hz), 131.1, 129.6, 128.9 (d, J = 13.0 Hz), 127.3, 122.1, 119.4 (d, J = 6.5 Hz), 45.3, 45.1, 30.3, 22.5, 18.6.

$^{31}\text{P NMR}$ (162 MHz, Chloroform-*d*) δ 18.7.

HRMS: $\text{C}_{31}\text{H}_{32}\text{N}_2\text{O}_3\text{P}$ [M+H] $^+$; calculated: 498.2198, found: 498.2198.



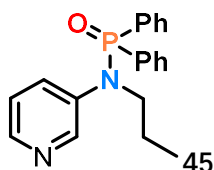
44

Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, white solid (87% yield, 2.06 g).

$^1\text{H NMR}$ (400 MHz, DMSO-*d*₆) δ 8.52 (d, J = 11.2 Hz, 1H), 8.34 (d, J = 2.7 Hz, 1H), 8.02 (dd, J = 4.7, 1.4 Hz, 1H), 7.86 – 7.77 (m, 4H), 7.66 – 7.48 (m, 6H), 7.44 – 7.34 (m, 1H), 7.13 (dd, J = 8.3, 4.6 Hz, 1H).

$^{13}\text{C NMR}$ (100 MHz, DMSO-*d*₆) δ 141.8, 140.1 (d, J = 7.4 Hz), 138.8, 133.0, 132.2 (d, J = 2.9 Hz), 131.7 (d, J = 9.9 Hz), 128.9 (d, J = 12.6 Hz), 124.6 (d, J = 6.6 Hz), 123.6..

(Known compound: *Bioorg. Med. Chem. Lett.* **2013**, *23*, 706-710).



45

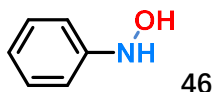
Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 1:1) as eluant, pale yellow solid (95% yield, 2.24 g).

¹H NMR (400 MHz, Chloroform-*d*) δ 8.51 (d, *J* = 2.5 Hz, 1H), 8.24 (d, *J* = 4.5 Hz, 1H), 7.89 – 7.80 (m, 4H), 7.71 – 7.65 (m, 1H), 7.47 – 7.33 (m, 6H), 7.08 (dd, *J* = 8.3, 4.7 Hz, 1H), 3.41 – 3.30 (m, 2H), 1.55 – 1.49 (m, 2H), 0.78 – 0.70 (m, 3H).

¹³C NMR (100 MHz, Chloroform-*d*) ¹³C NMR (100 MHz, Chloroform-*d*) δ 149.2, 146.2, 140.2, 135.6 (d, *J* = 4.3 Hz), 132.6 (d, *J* = 9.2 Hz), 132.0 (d, *J* = 2.9 Hz), 131.4 (d, *J* = 129.5 Hz), 128.6 (d, *J* = 12.7 Hz), 123.5, 52.9 (d, *J* = 3.2 Hz), 22.3 (d, *J* = 3.1 Hz), 11.2.

³¹P NMR (162 MHz, Chloroform-*d*) δ 26.92.

(Known compound: *Bioorg. Med. Chem. Lett.* **2013**, *23*, 706-710).

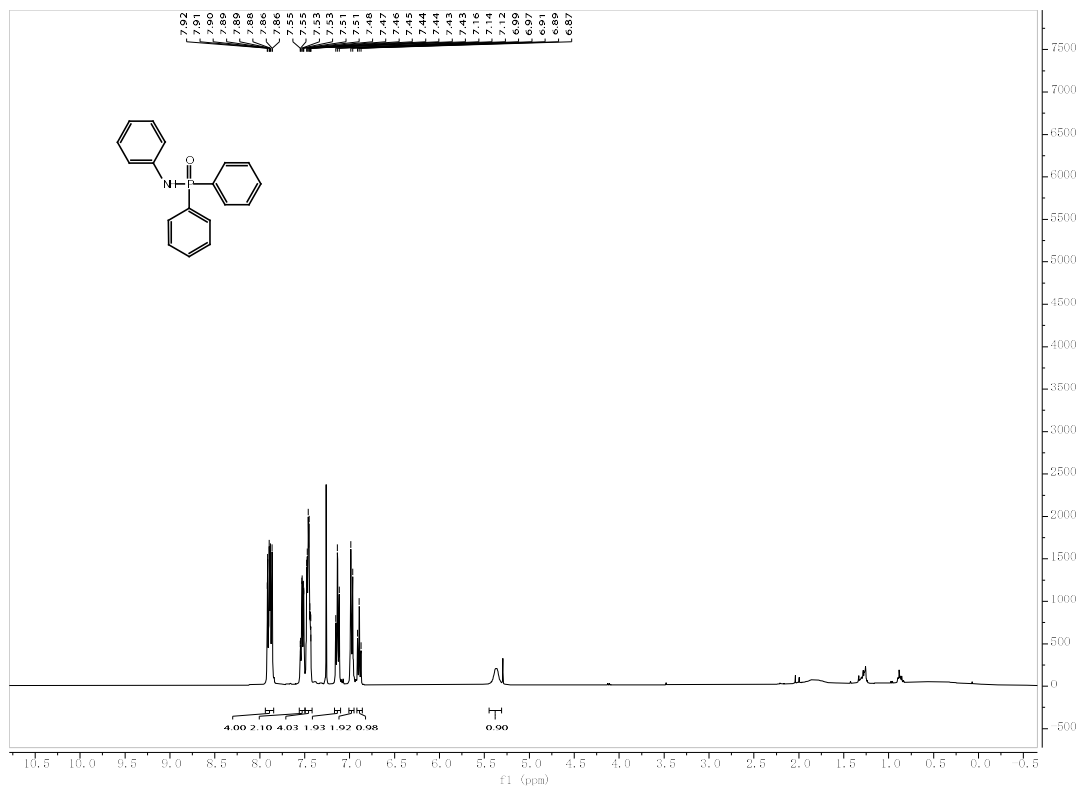


Purified by column chromatography using the mixture of petroleum ether and ethyl acetate (PE/EA = 3:1) as eluant, yellow solid.

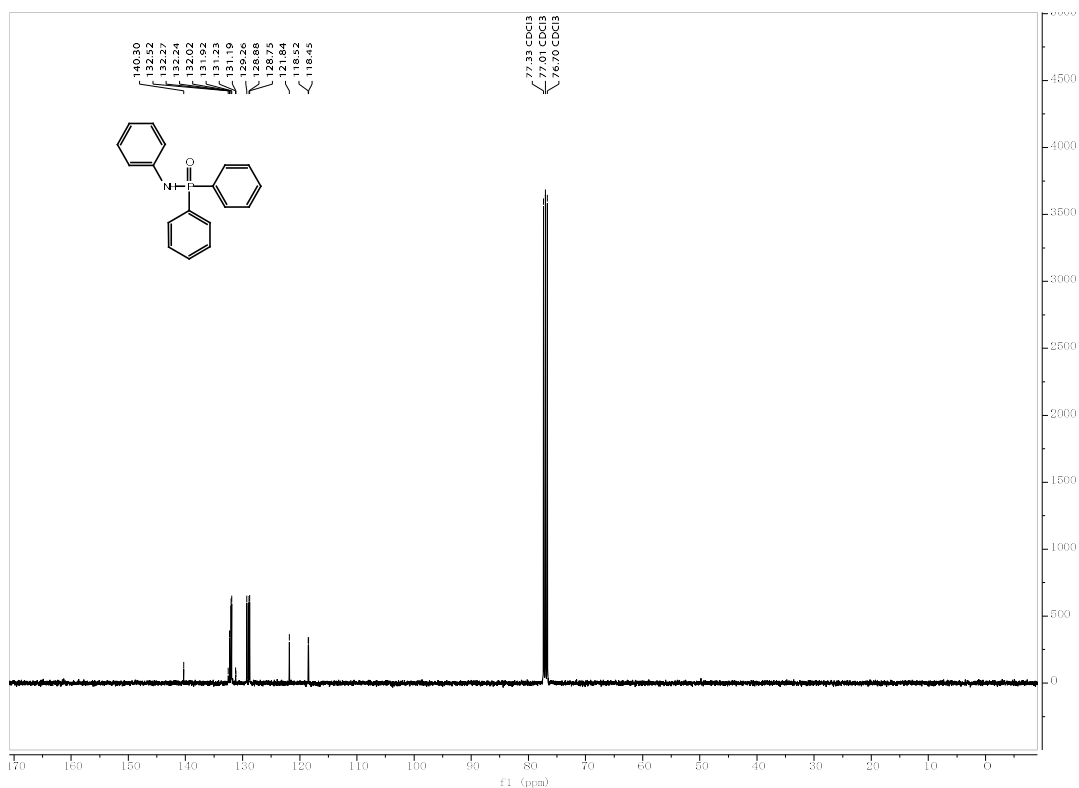
¹H NMR (400 MHz, Chloroform-*d*) δ 7.36 – 7.28 (m, 2H), 7.09 – 6.99 (m, 3H), 6.37 (s, 1H), 6.13 (s, 1H).

(Known compound: *Chem. Sci.*, **2021**, *12*, 13730-13736)

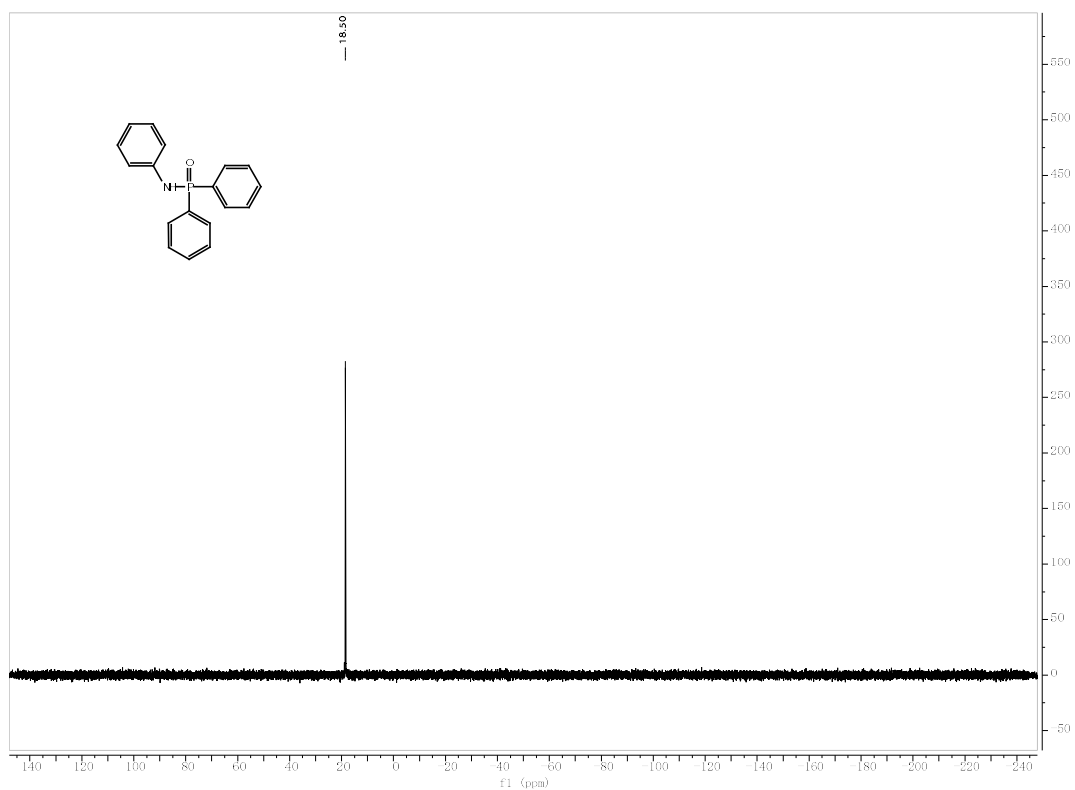
IV. NMR Spectra



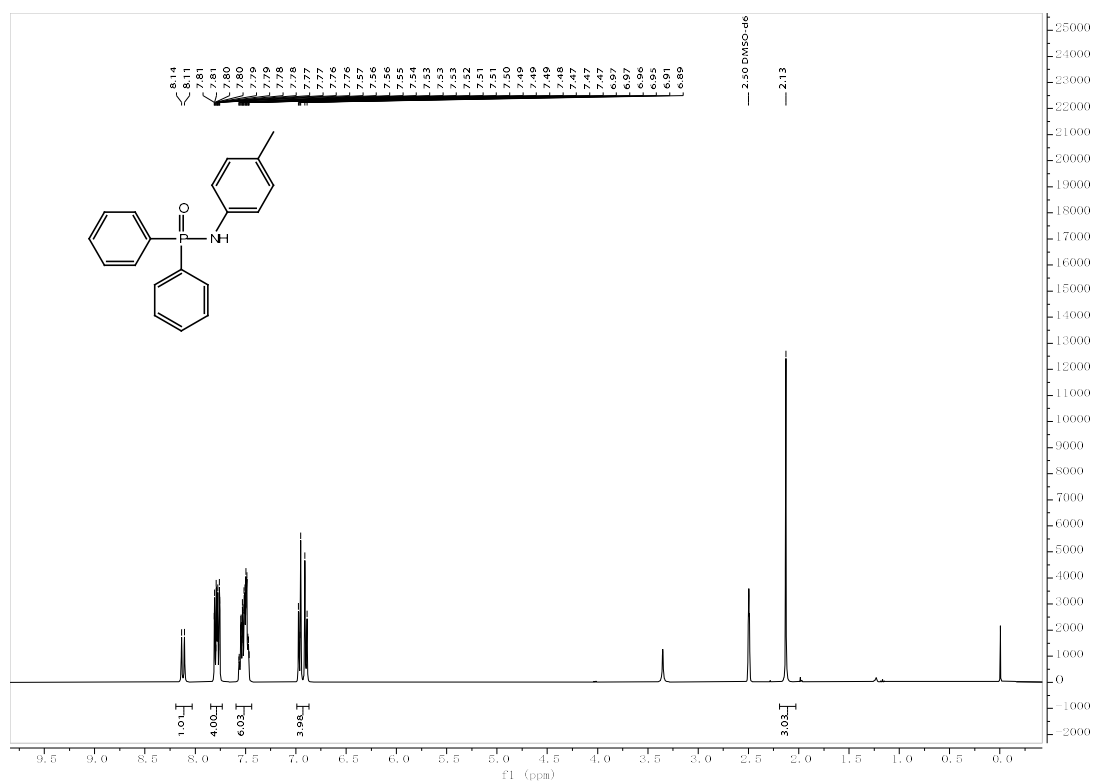
¹H NMR of **2** (400 MHz, CDCl₃)



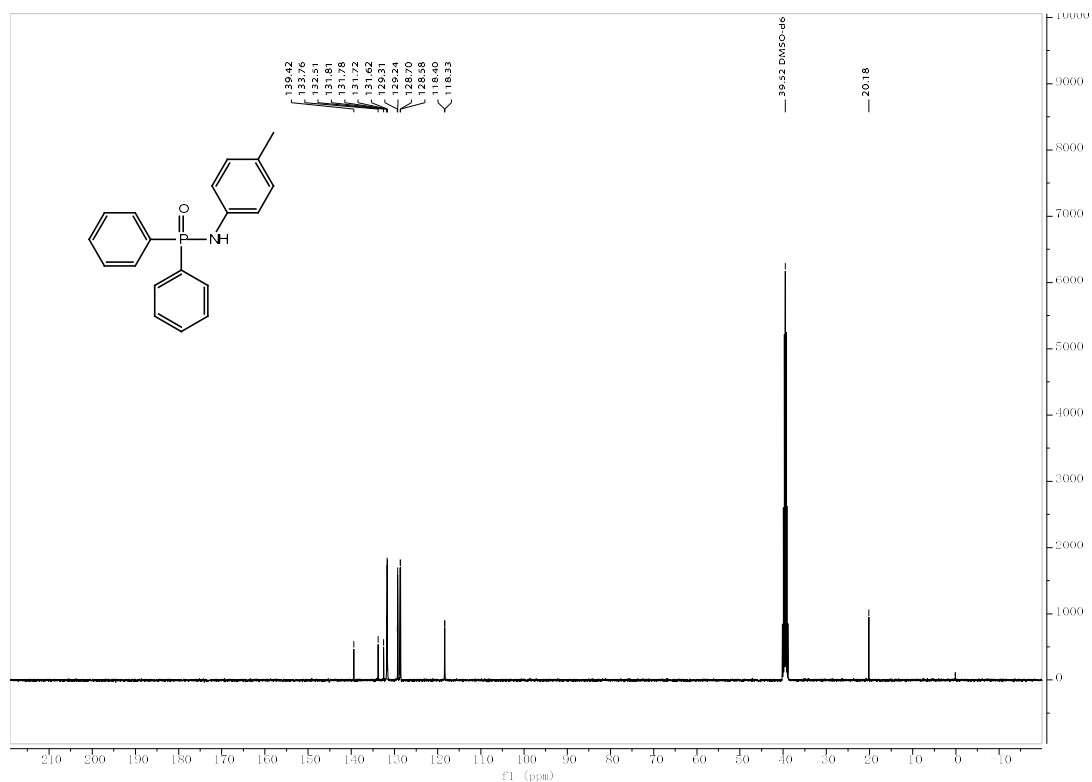
¹³C NMR of **2** (100 MHz, CDCl₃)



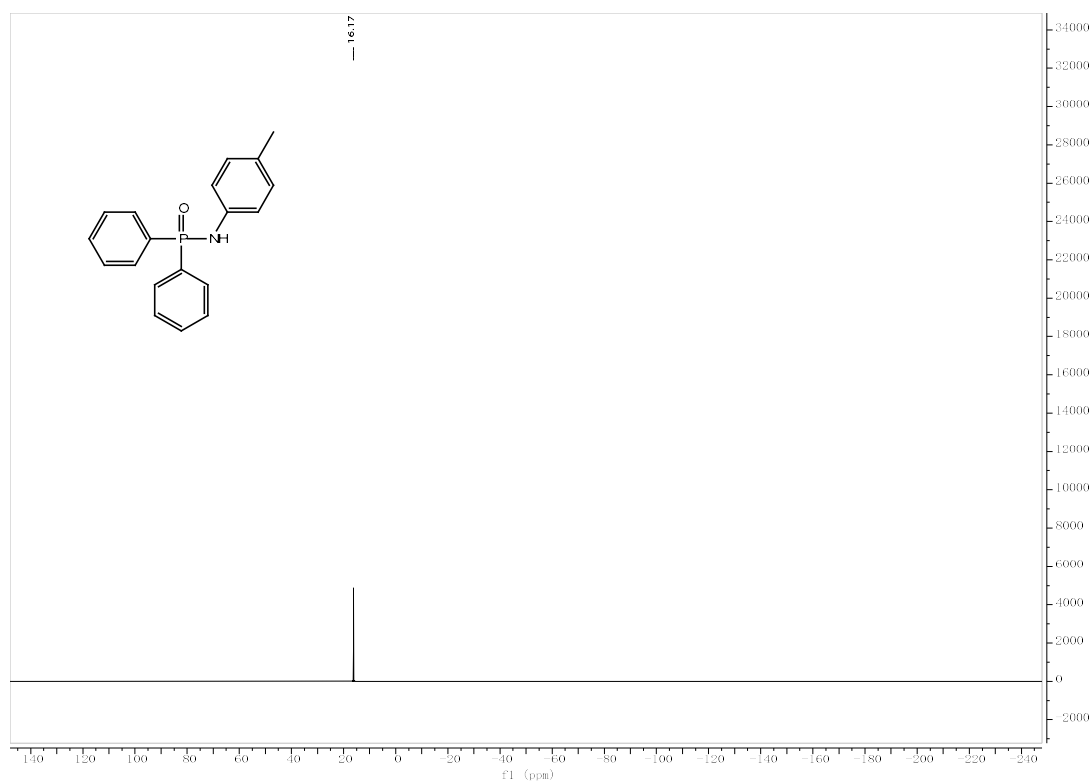
^{31}P NMR of 2 (162 MHz, CDCl_3)



^1H NMR of 3 (400 MHz, CDCl_3)



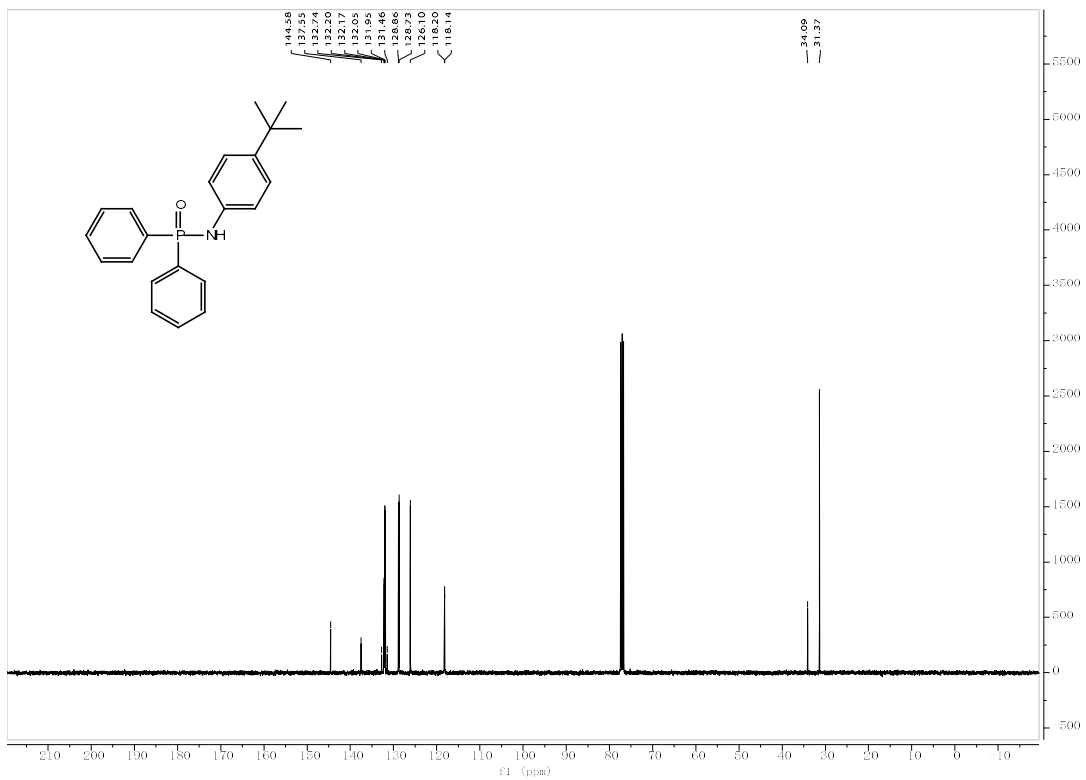
^{13}C NMR of **3** (100 MHz, CDCl_3)



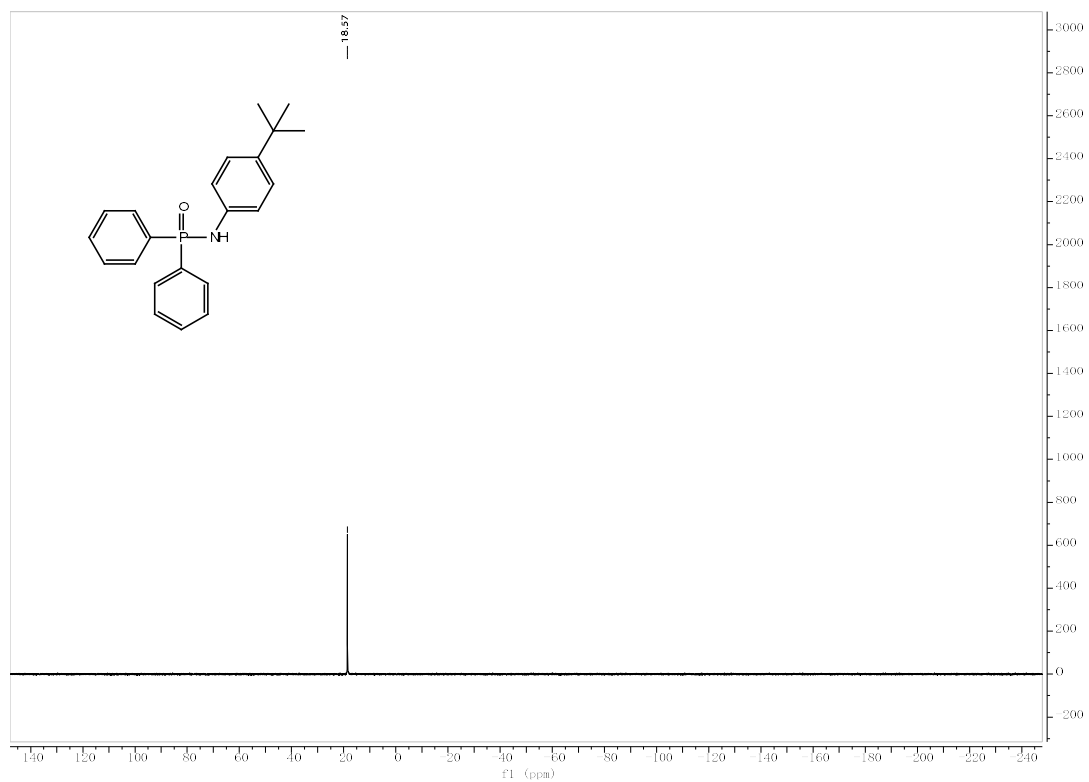
^{31}P NMR of **3** (162 MHz, CDCl_3)



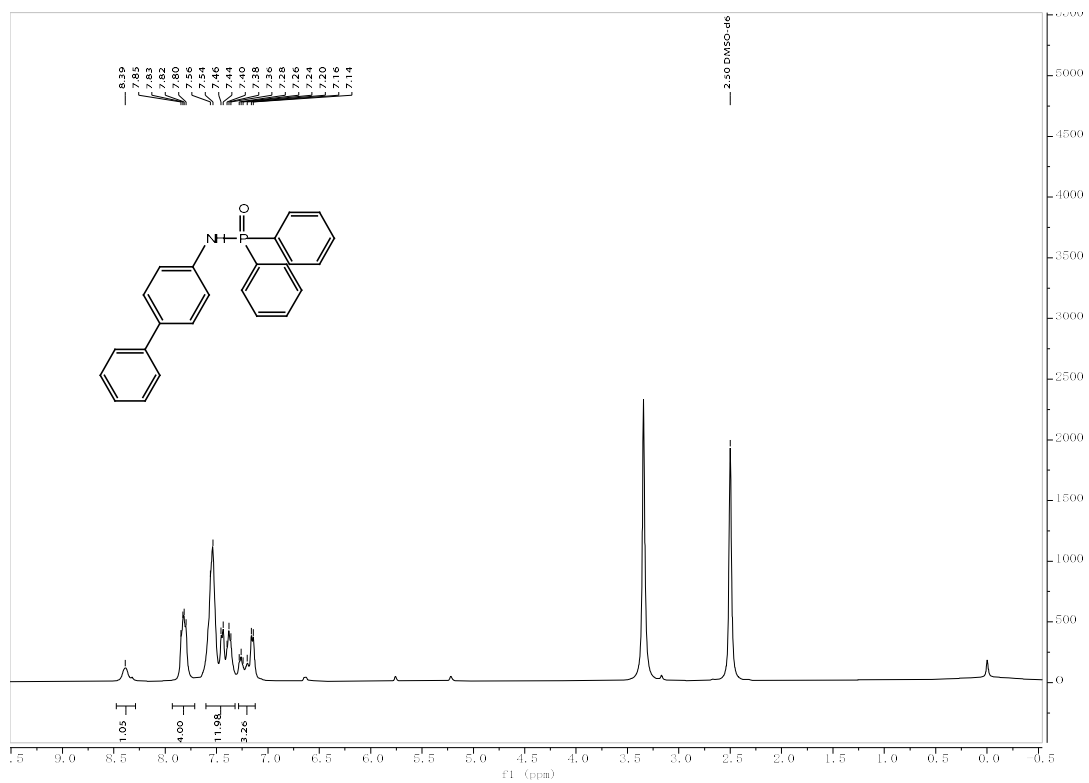
¹H NMR of 4 (400 MHz, CDCl₃)



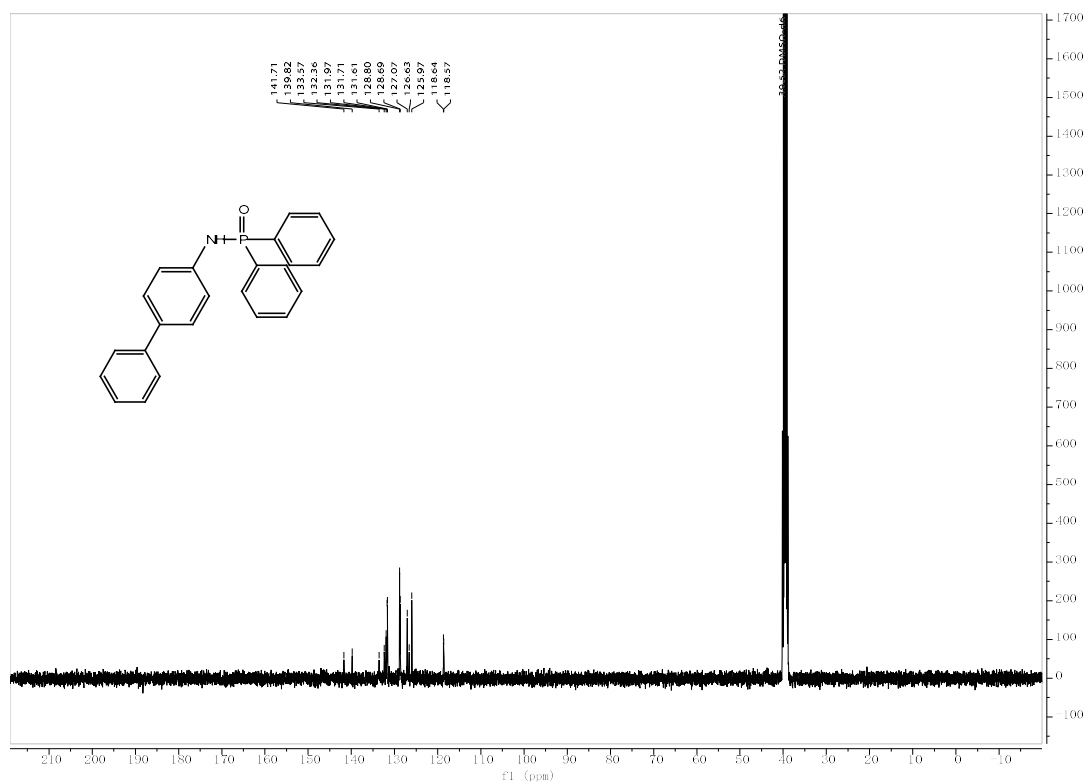
¹³C NMR of 4 (100 MHz, CDCl₃)



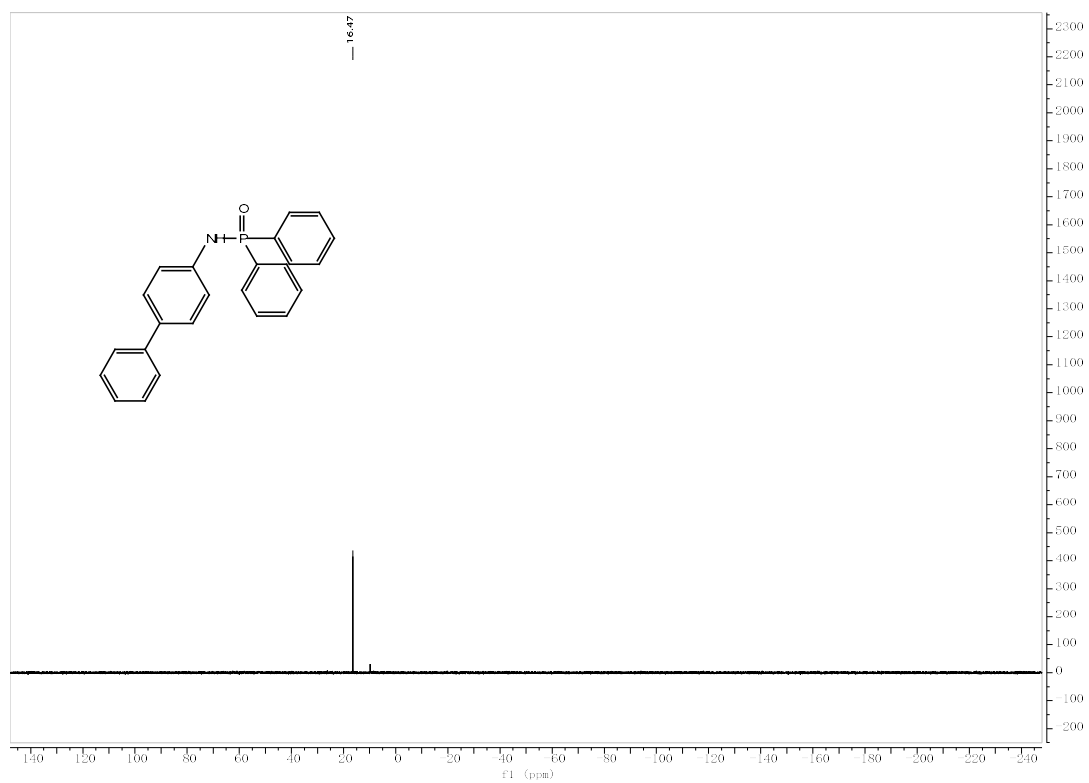
^{31}P NMR of 4 (162 MHz, CDCl_3)



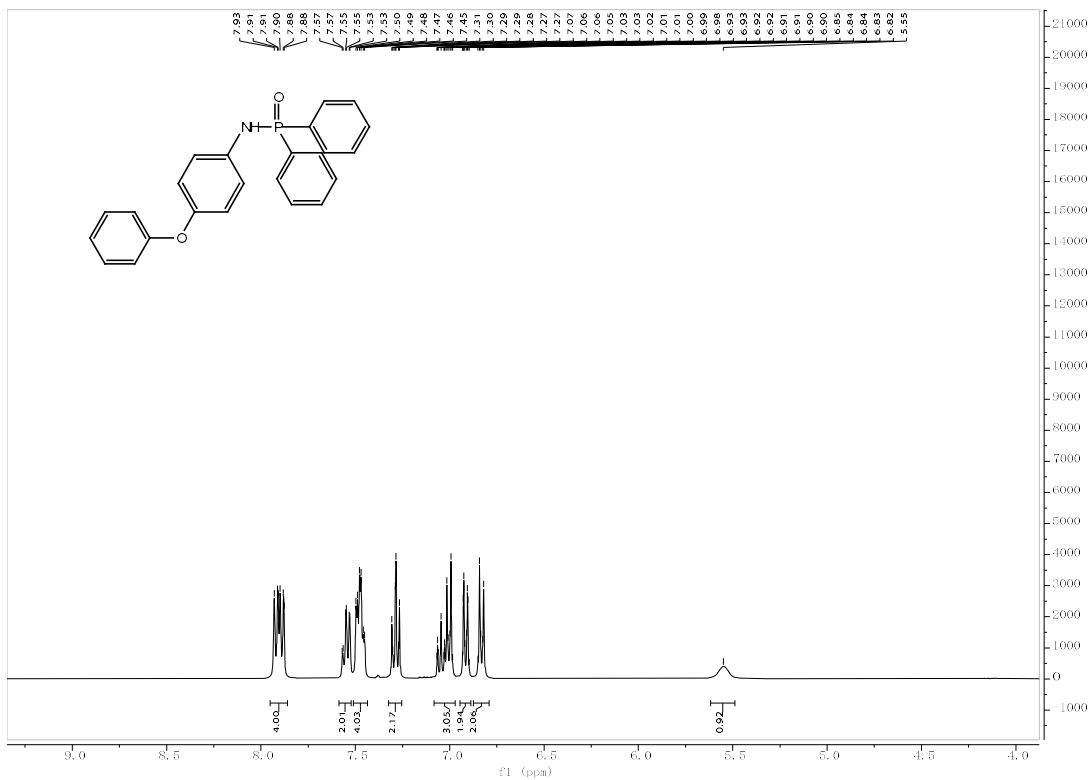
^1H NMR of 5 (400 MHz, DMSO-d_6)



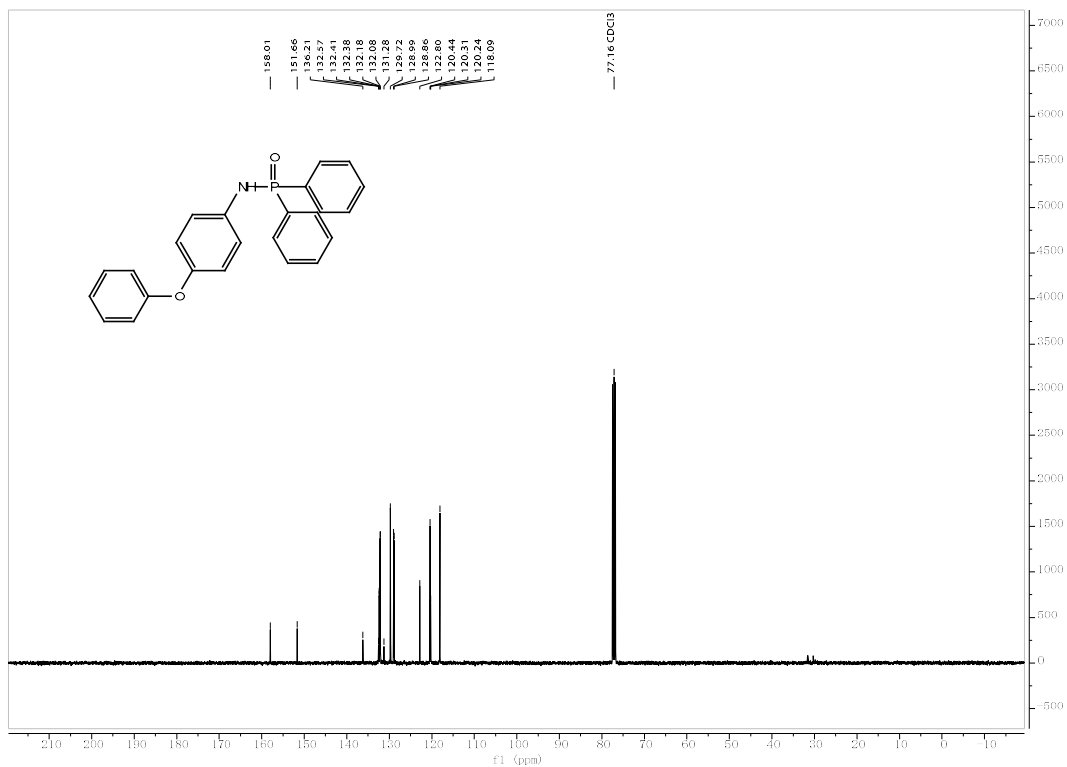
¹³C NMR of 5 (100 MHz, DMSO-d₆)



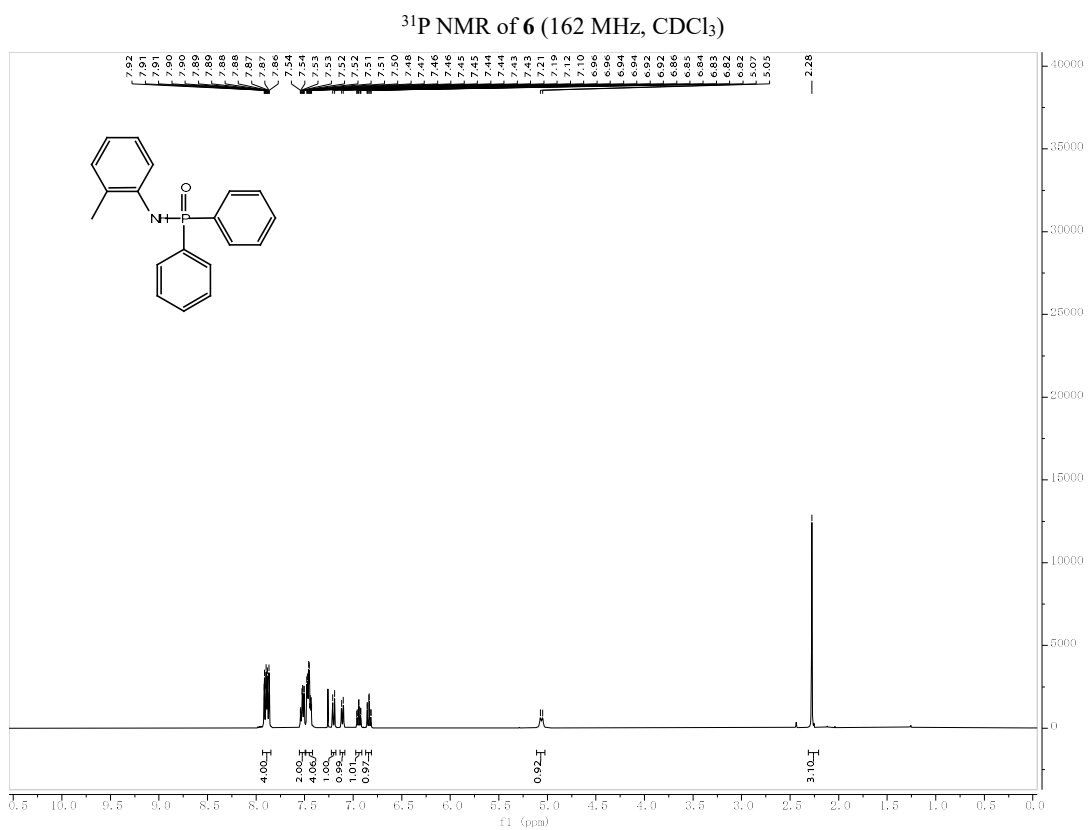
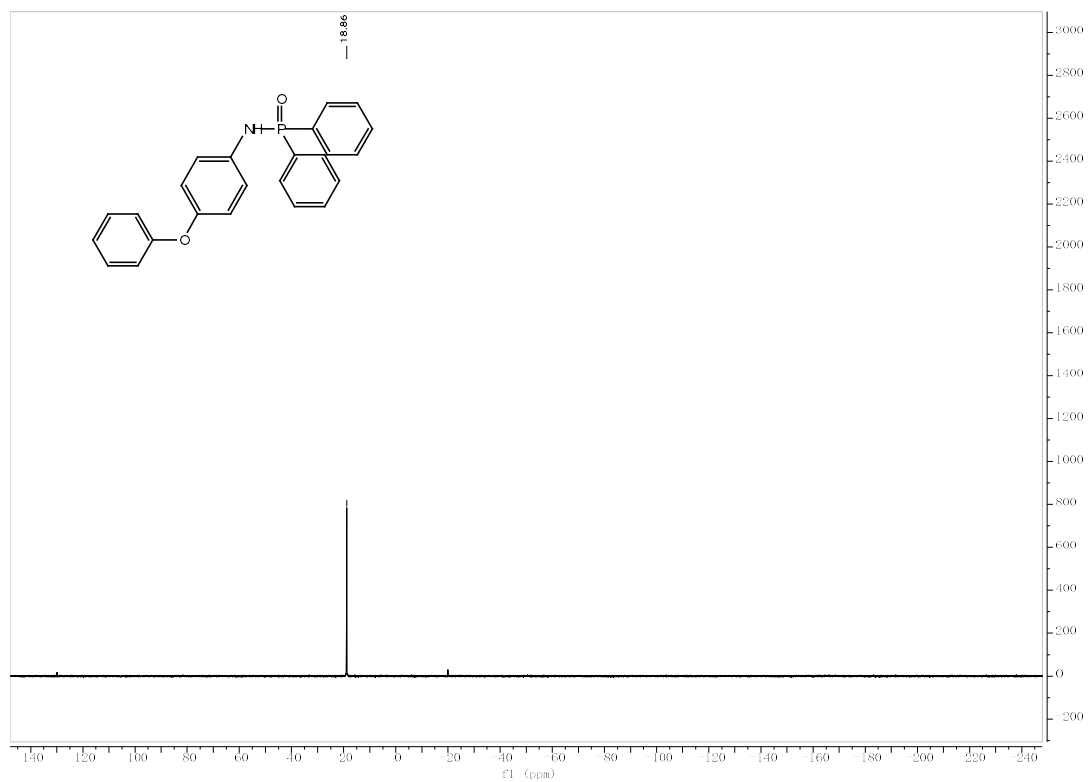
³¹P NMR of 5 (162 MHz, DMSO-d₆)

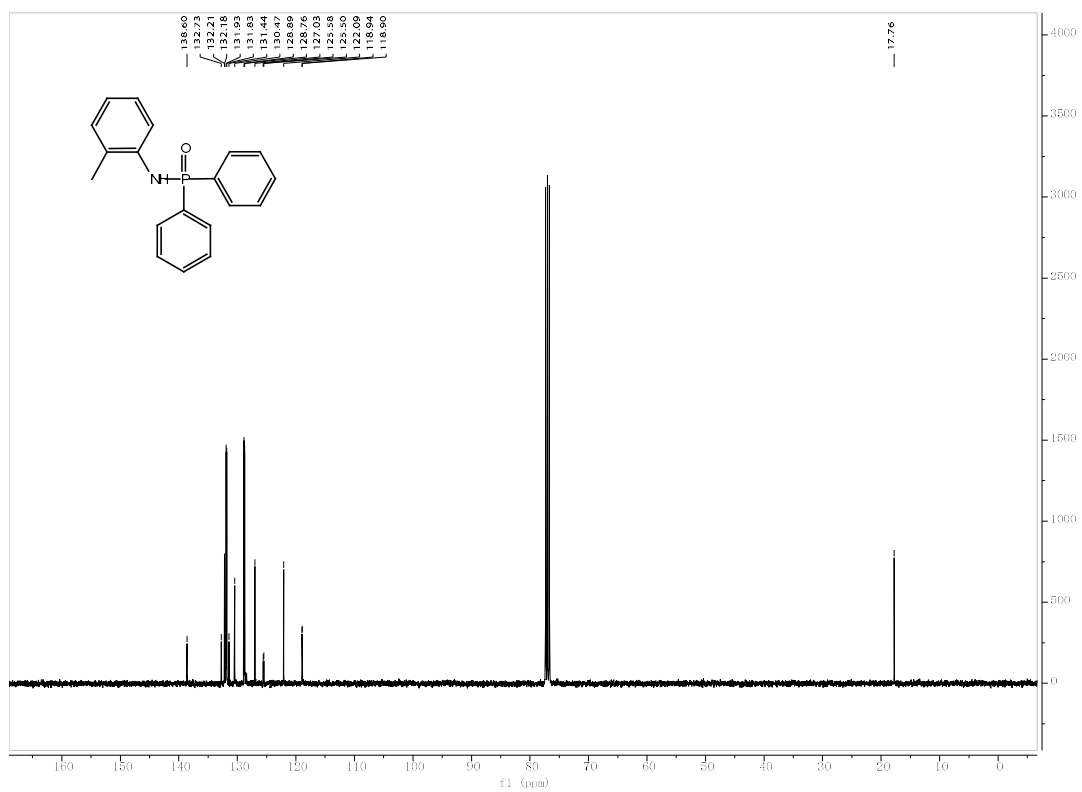


¹H NMR of 6 (400 MHz, CDCl₃)



¹³C NMR of 6 (100 MHz, CDCl₃)

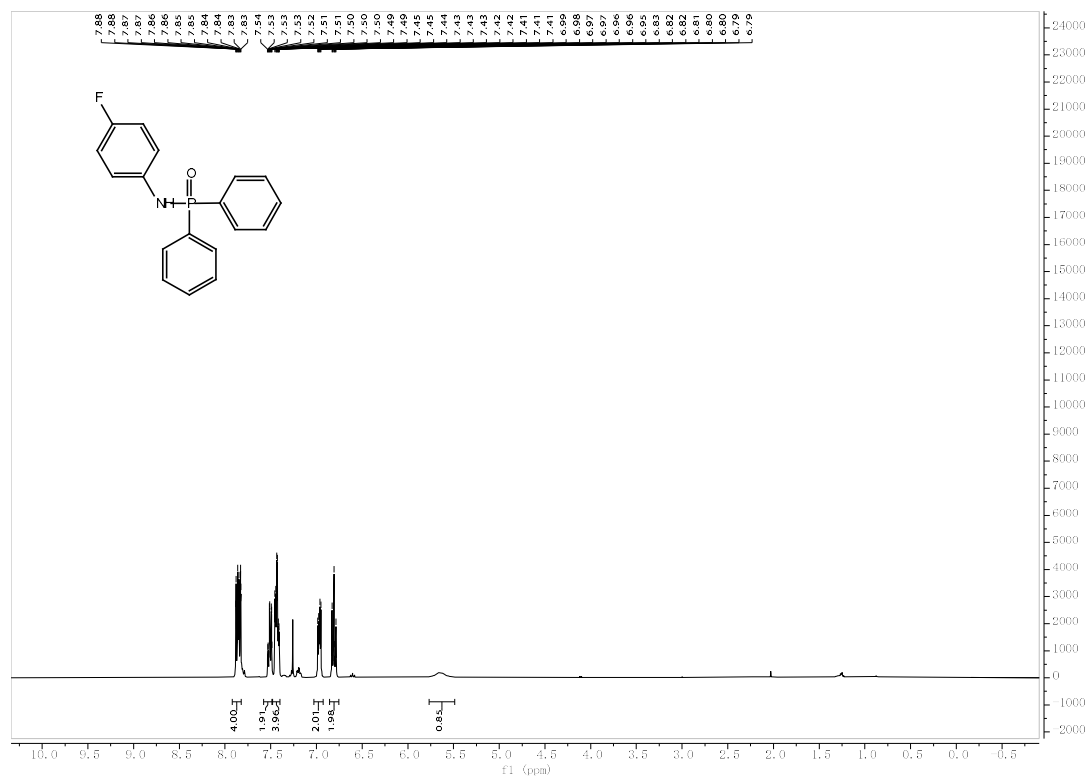




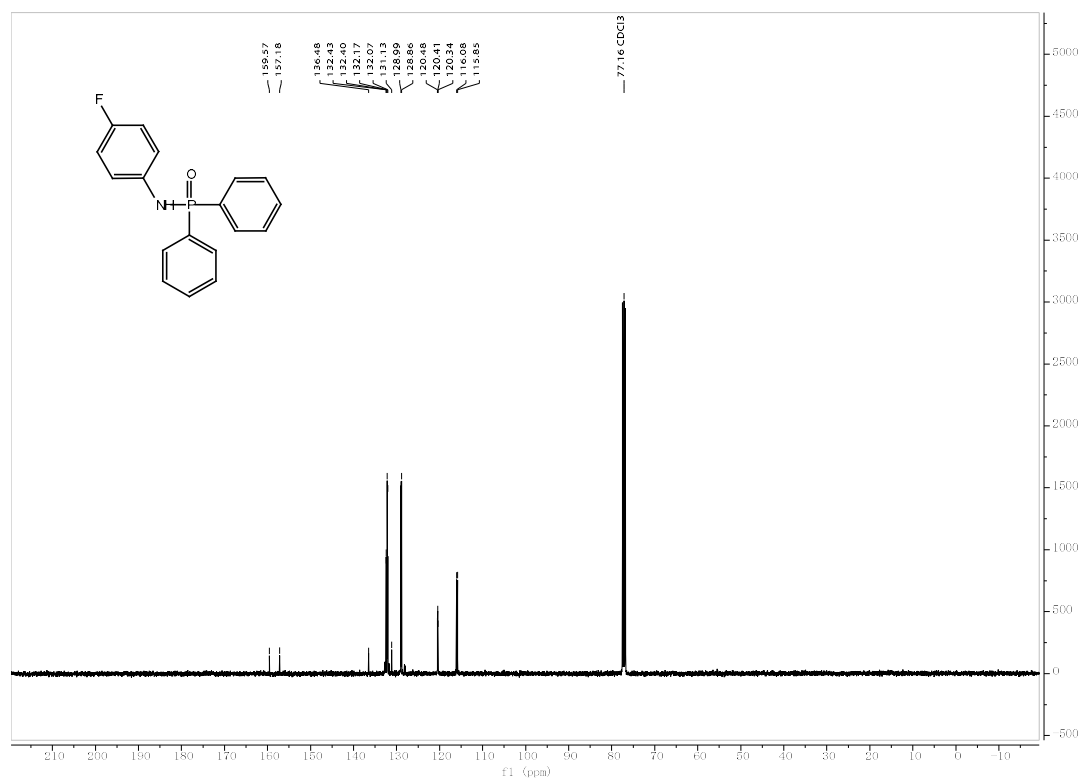
^{13}C NMR of 7 (100 MHz, CDCl_3)



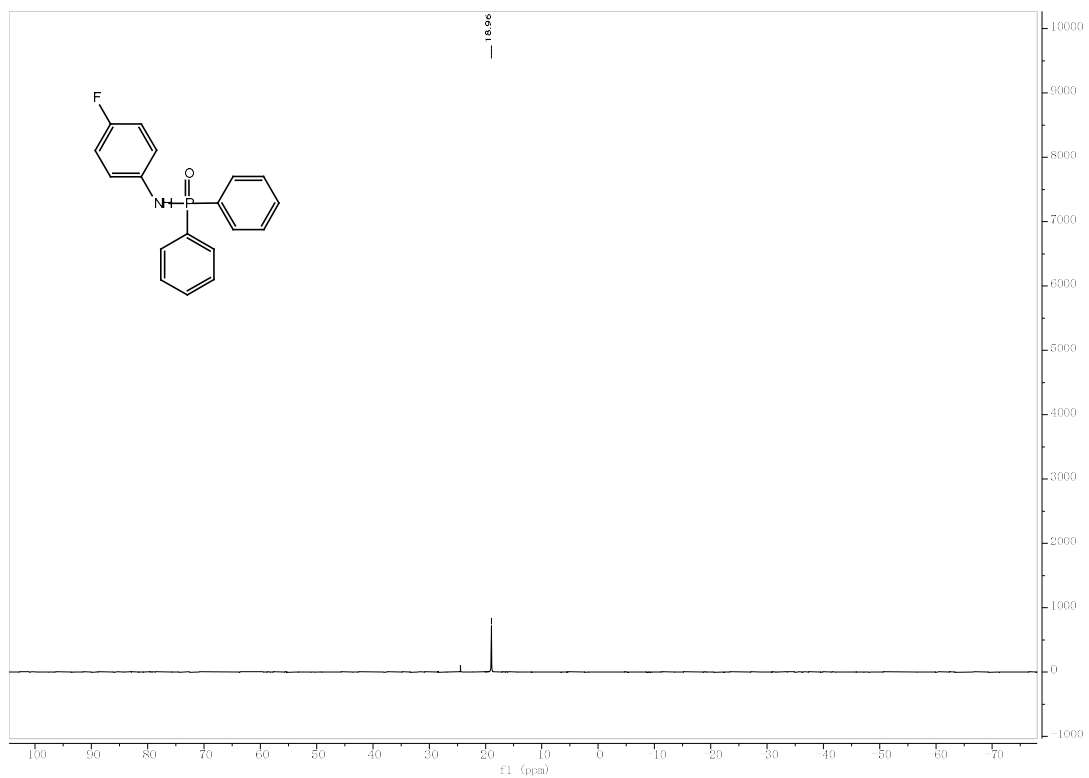
^{31}P NMR of 7 (162 MHz, CDCl_3)



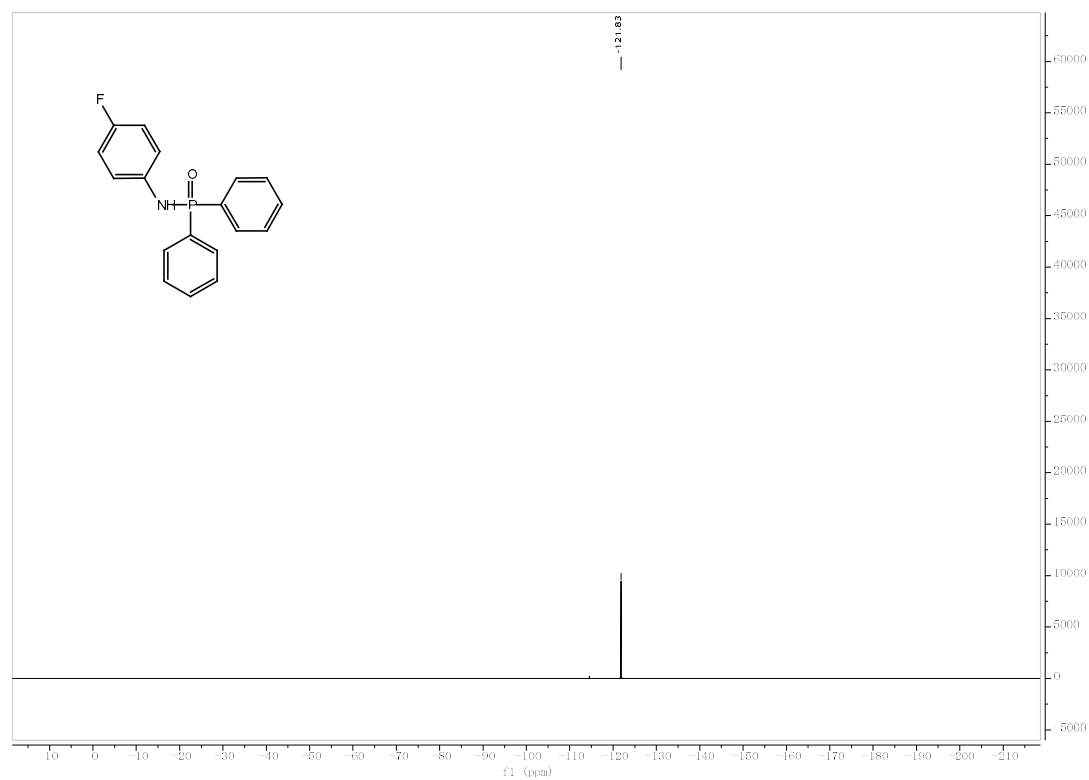
¹H NMR of 8 (400 MHz, CDCl₃)



¹³C NMR of 8 (100 MHz, CDCl₃)



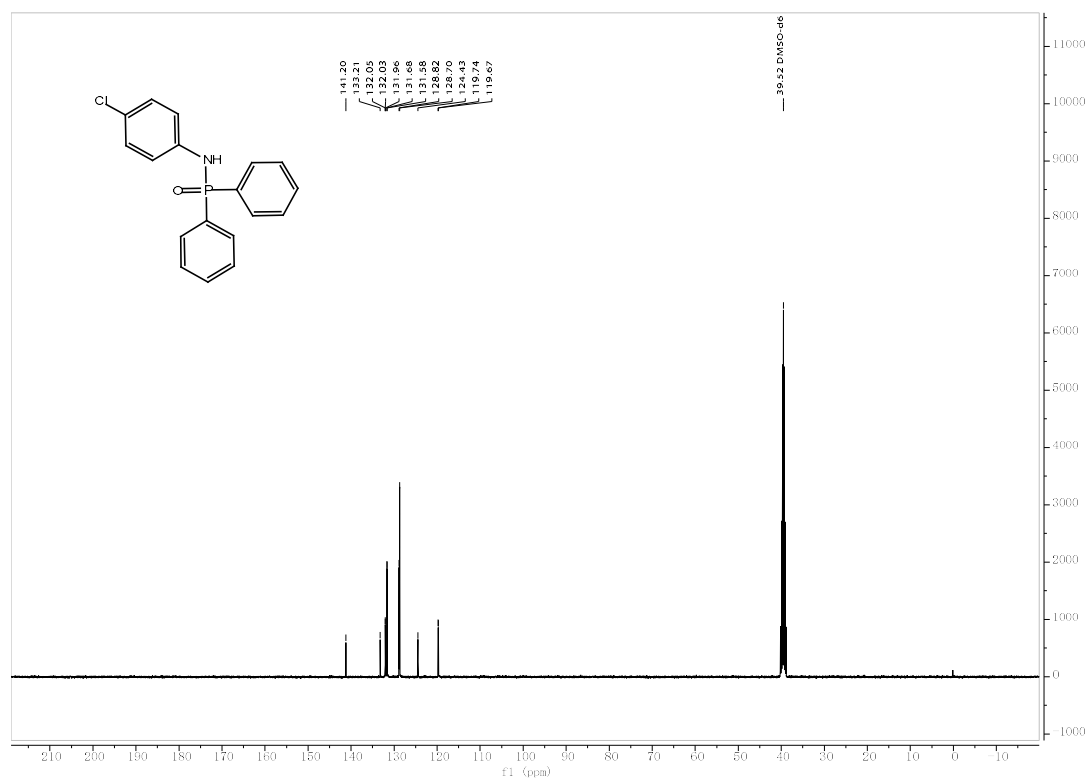
^{31}P NMR of **8** (162 MHz, CDCl_3)



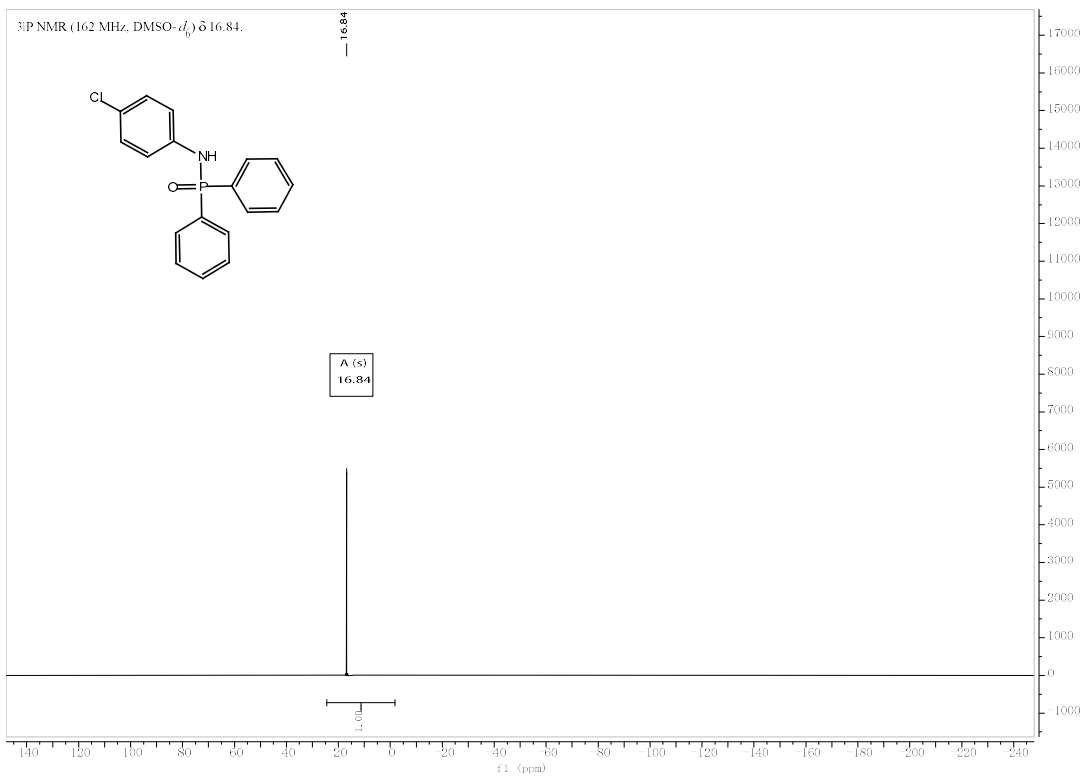
^{19}F NMR of **8** (376 MHz, CDCl_3)



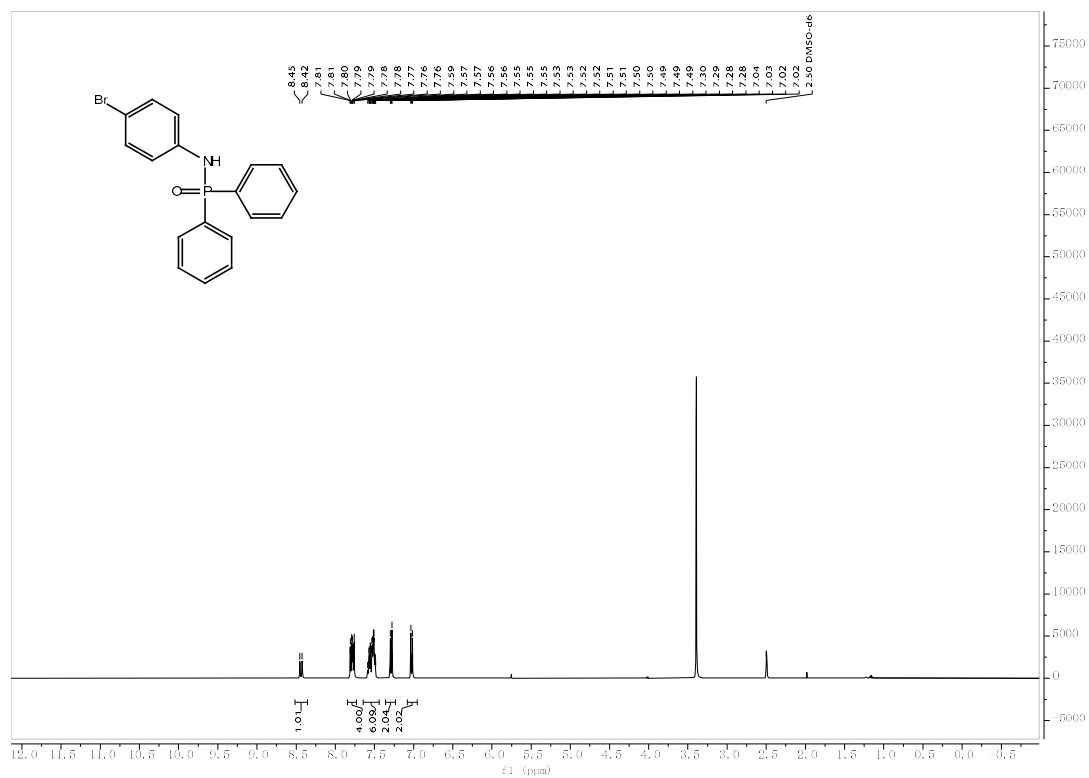
¹H NMR of 9 (400 MHz, DMSO-d₆)



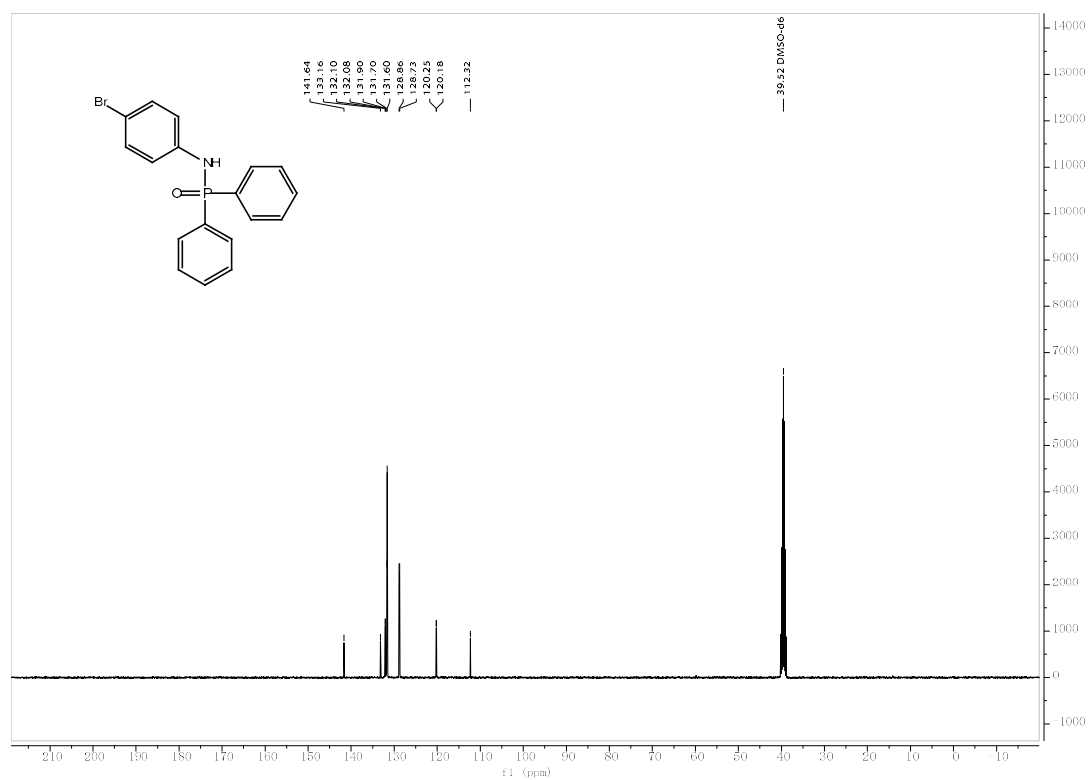
¹³C NMR of 9 (100 MHz, DMSO-d₆)



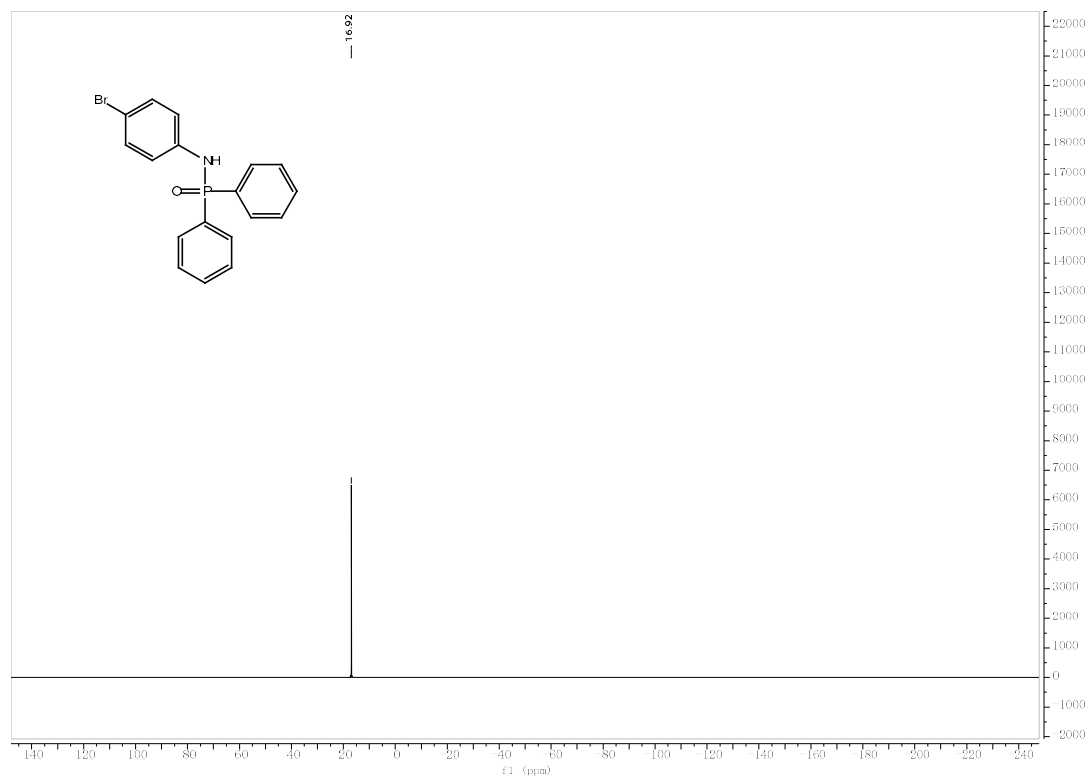
^{31}P NMR of **9** (162 MHz, $\text{DMSO-}d_6$)



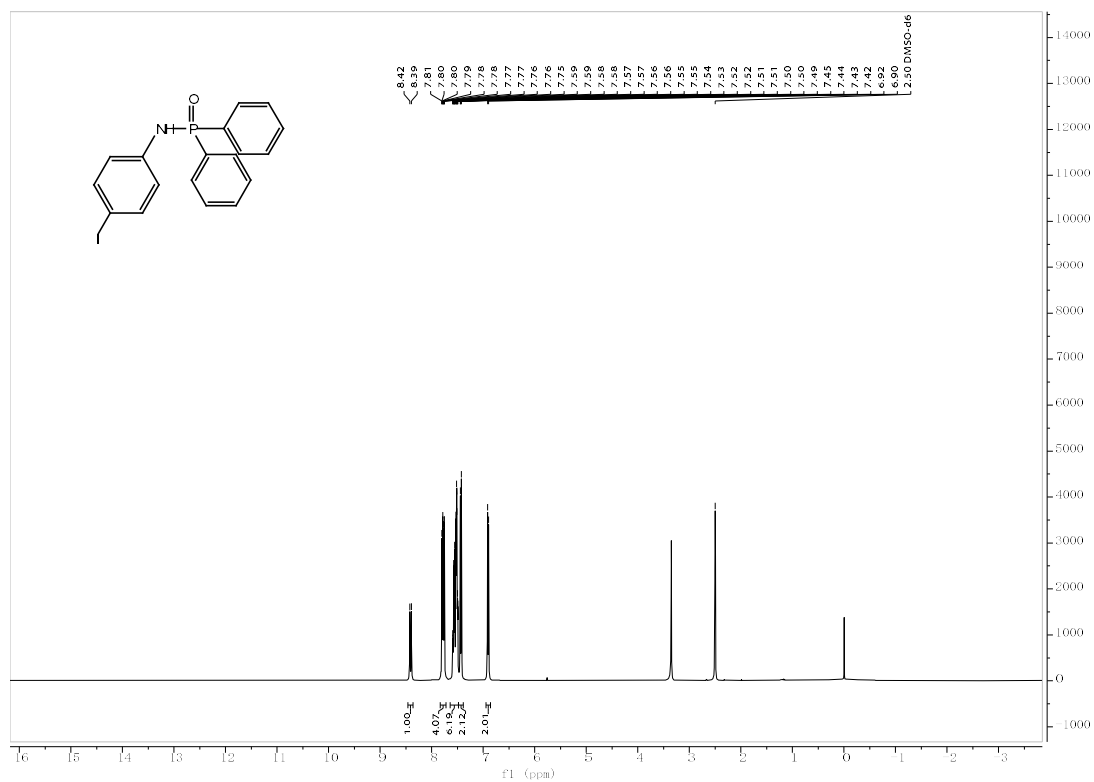
^1H NMR of **10** (400 MHz, $\text{DMSO-}d_6$)



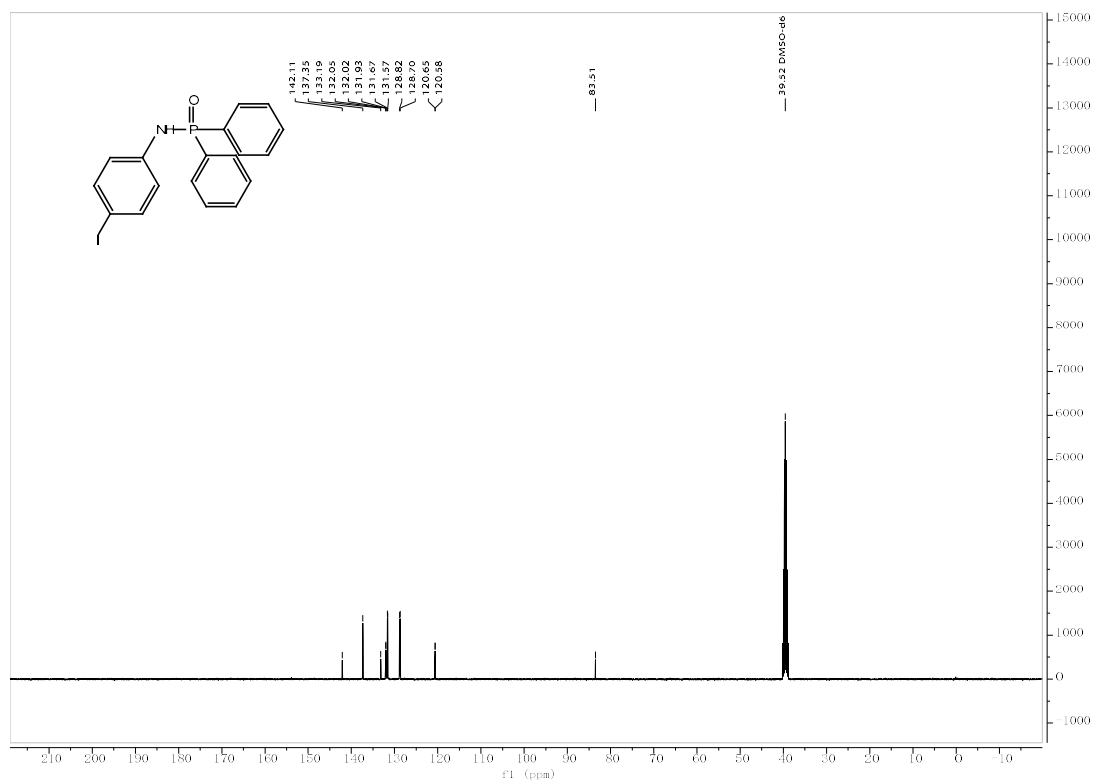
¹³C NMR of **10** (100 MHz, DMSO-d₆)



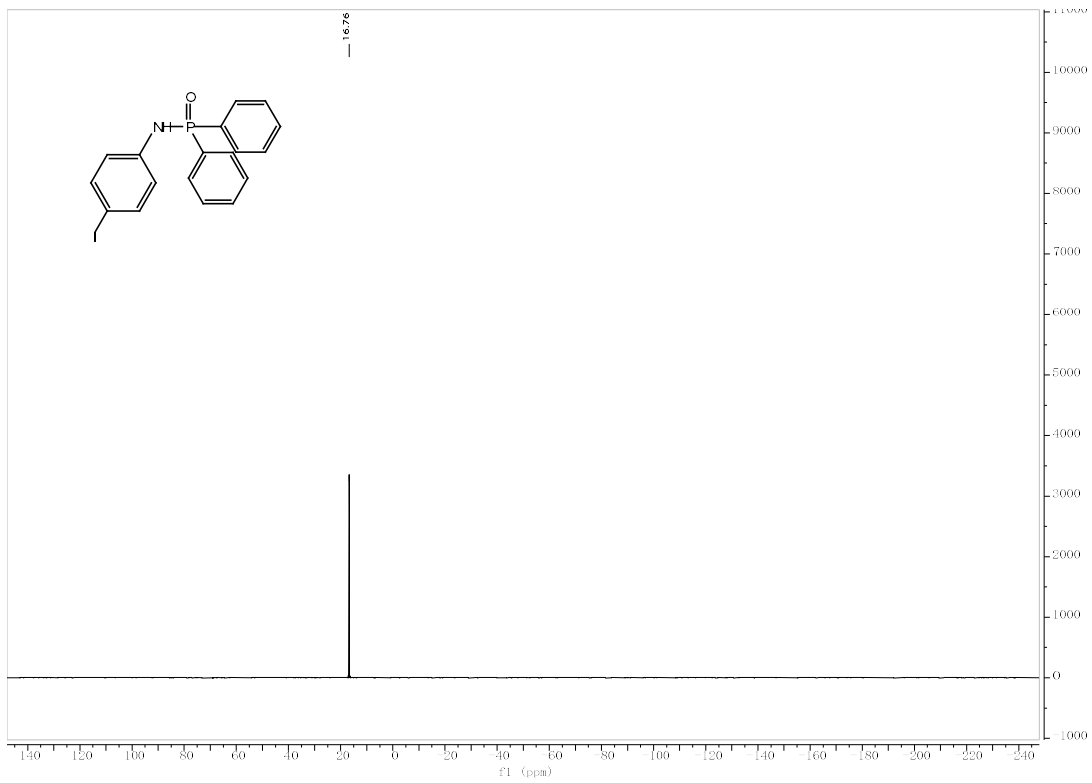
³¹P NMR of **10** (162 MHz, DMSO-d₆)



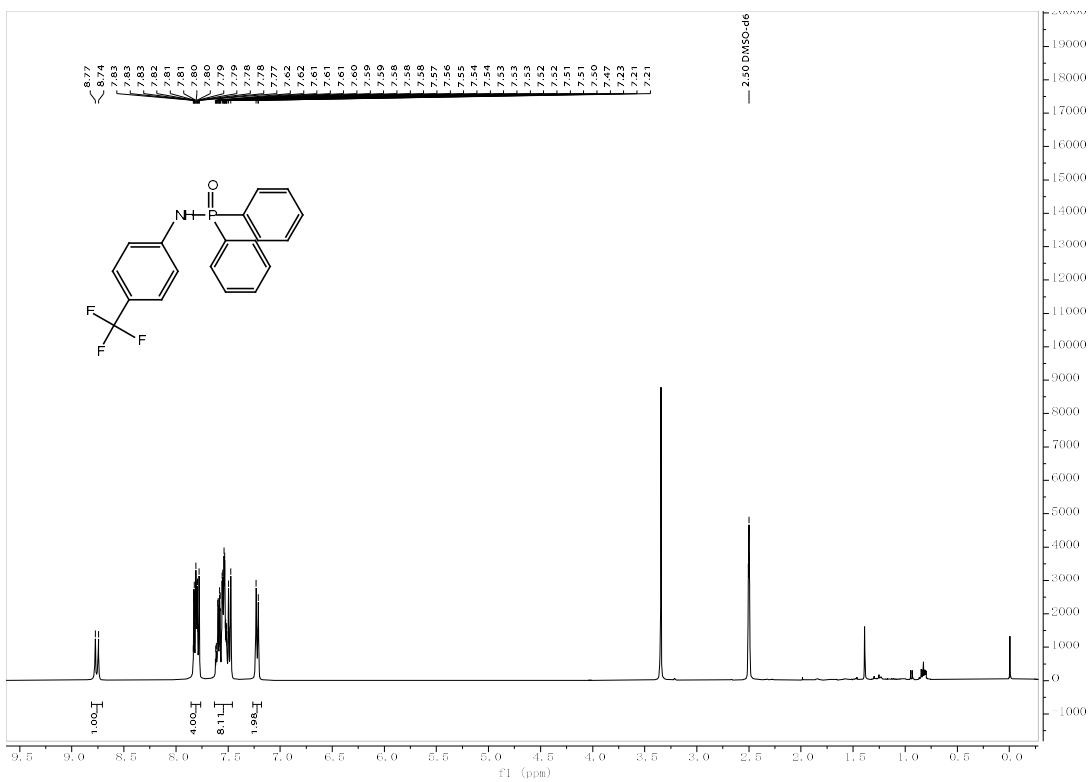
¹H NMR of 11 (400 MHz, DMSO-d₆)



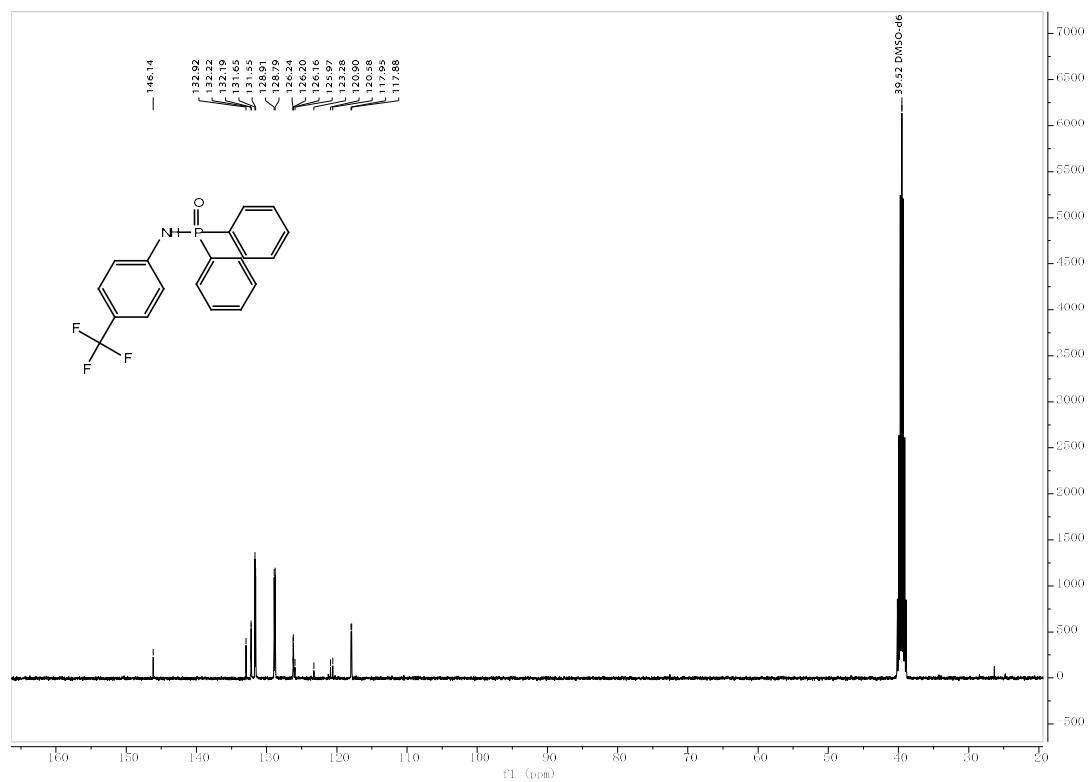
¹³C NMR of 11 (100 MHz, DMSO-d₆)



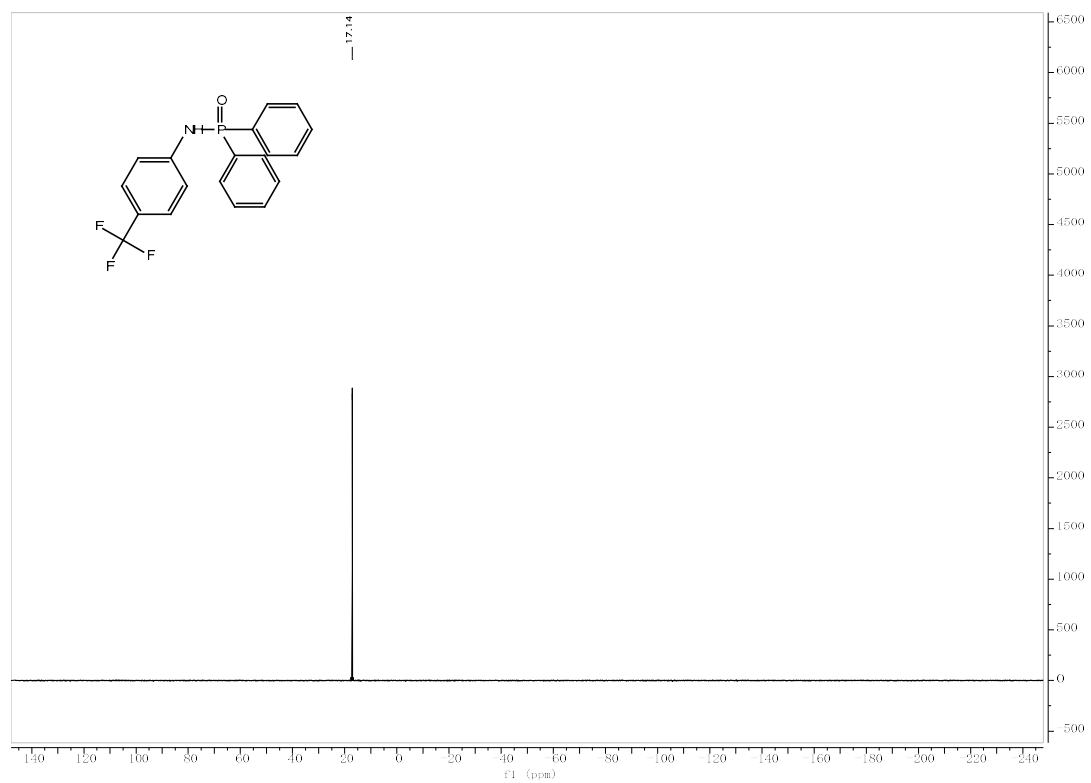
^{31}P NMR of 11 (162 MHz, DMSO- d_6)



^1H NMR of 12 (400 MHz, DMSO- d_6)



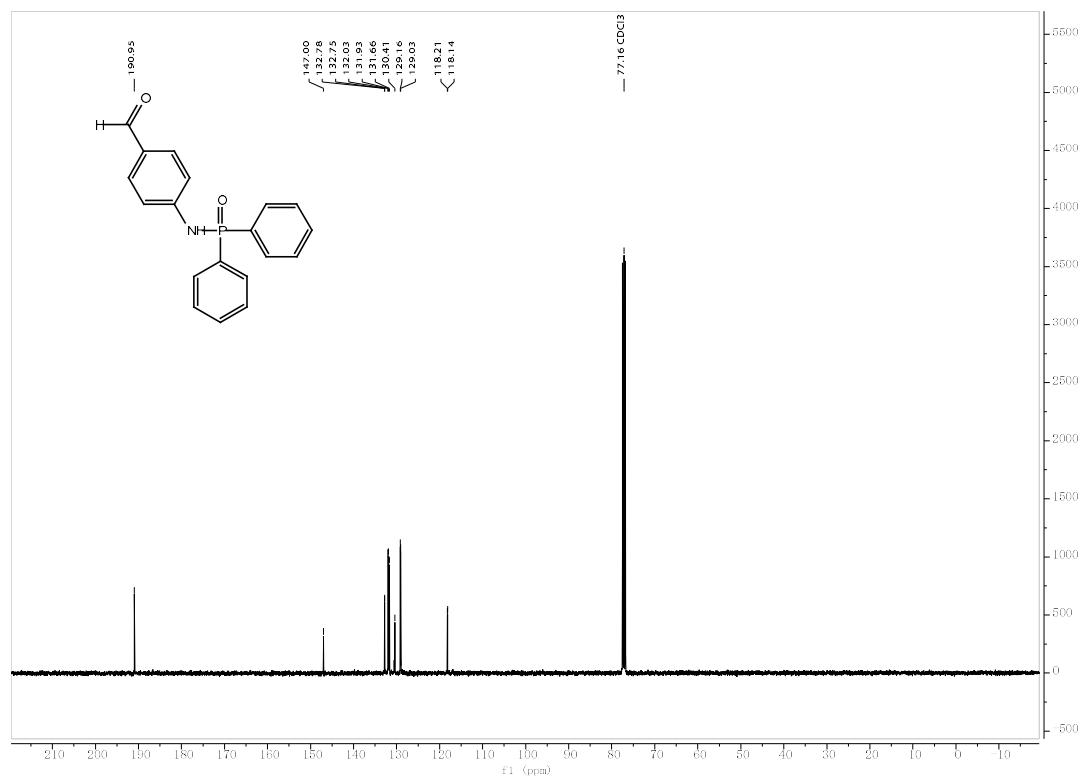
¹³C NMR of **12** (100 MHz, DMSO-d₆)



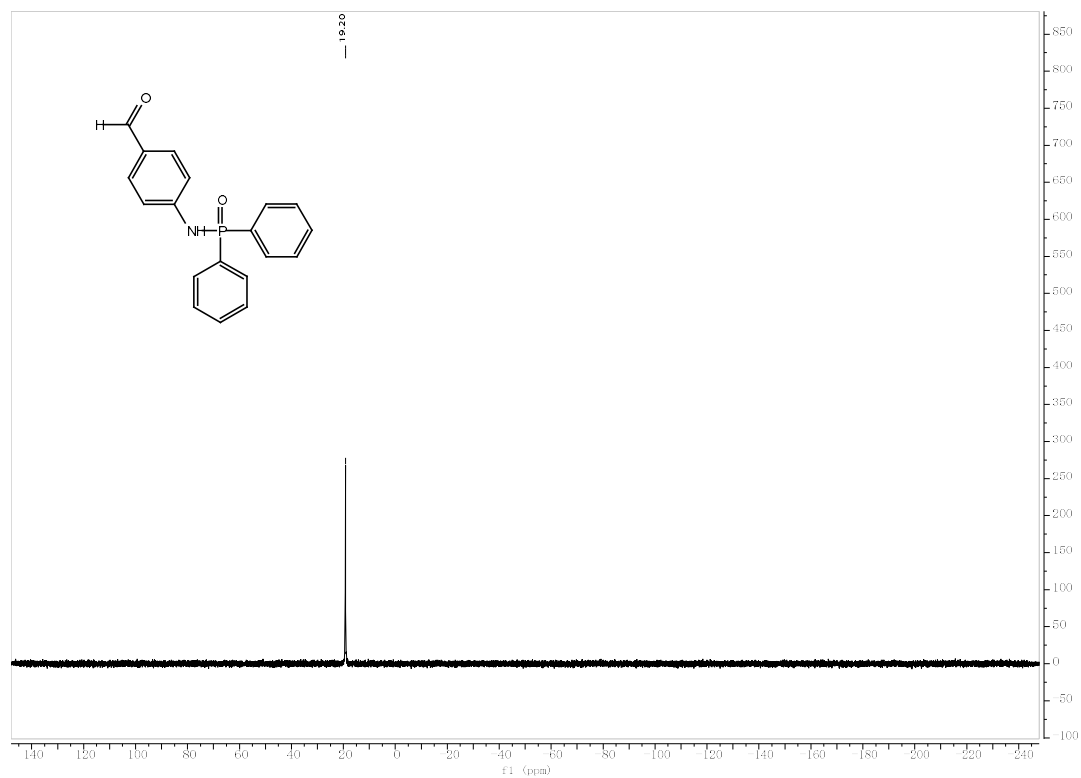
³¹P NMR of **12** (162 MHz, DMSO-d₆)



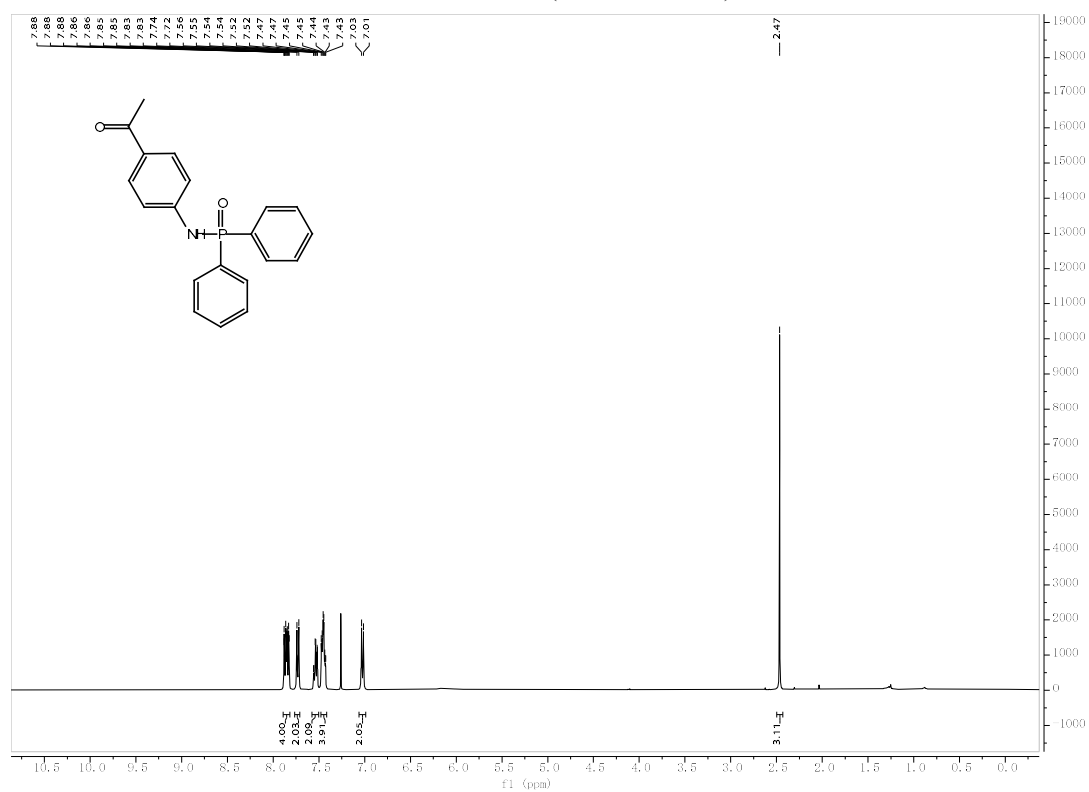
^1H NMR of **13** (400 MHz, CDCl_3)



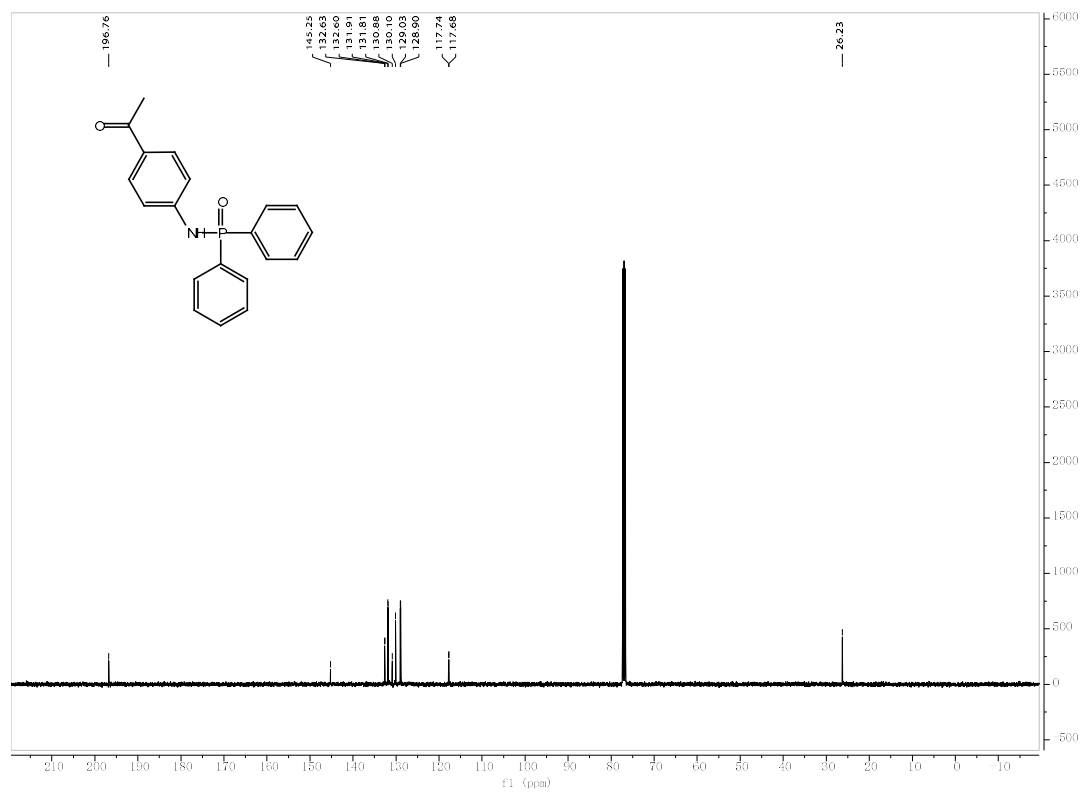
^{13}C NMR of **13** (100 MHz, CDCl_3)



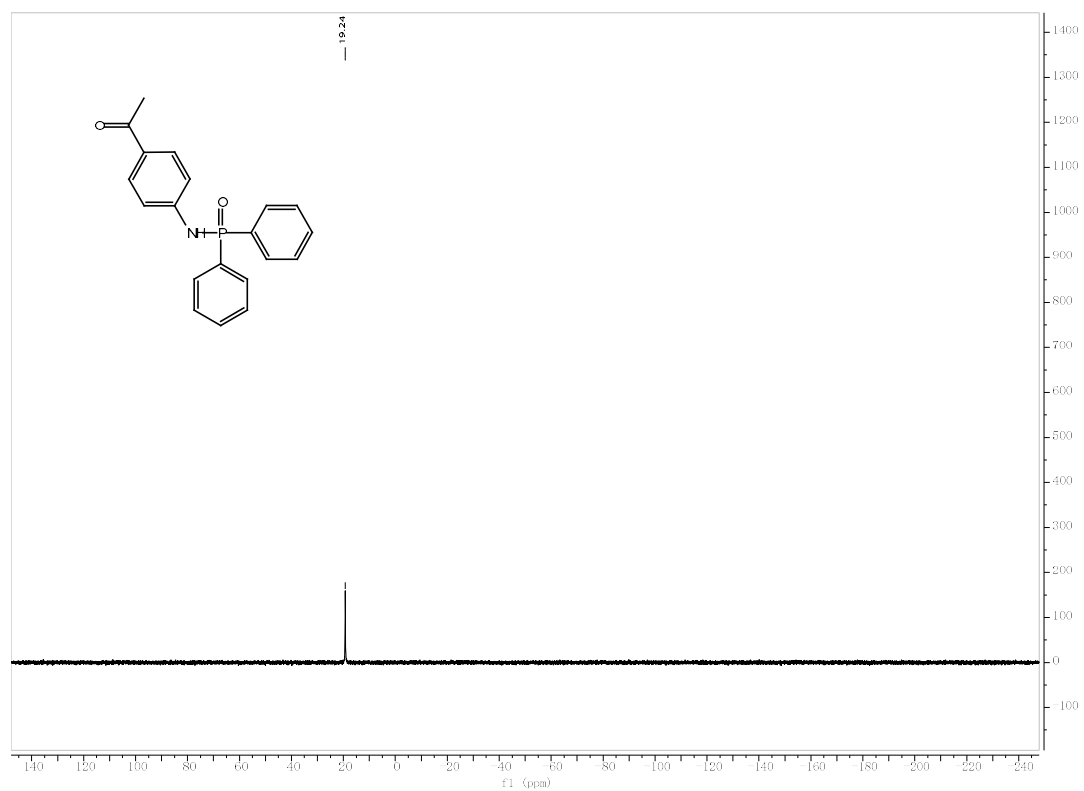
^{31}P NMR of 13 (162 MHz, CDCl_3)



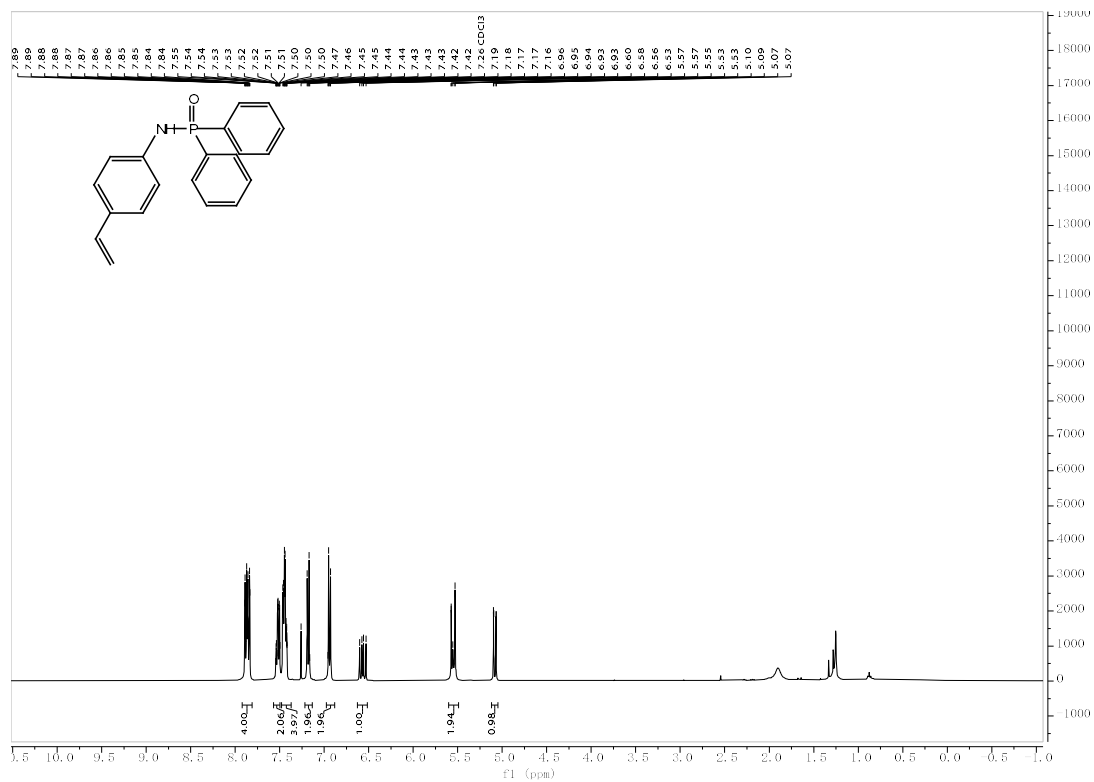
^1H NMR of 14 (400 MHz, CDCl_3)



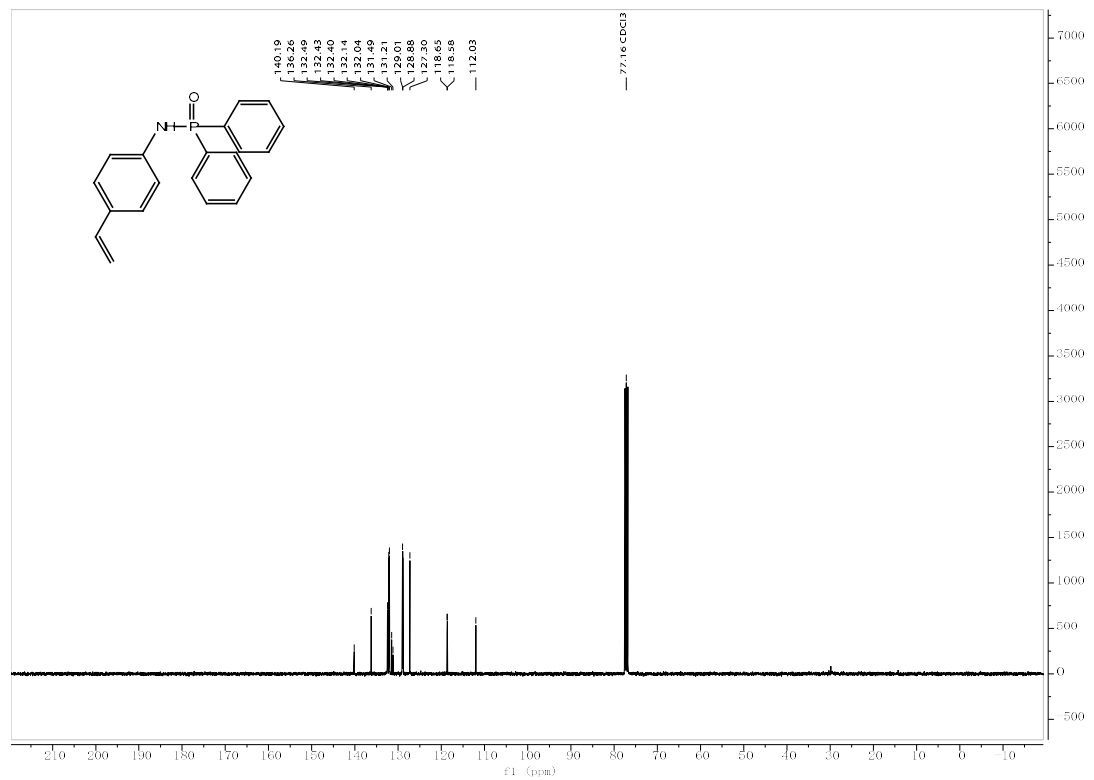
¹³C NMR of 14 (100 MHz, CDCl₃)



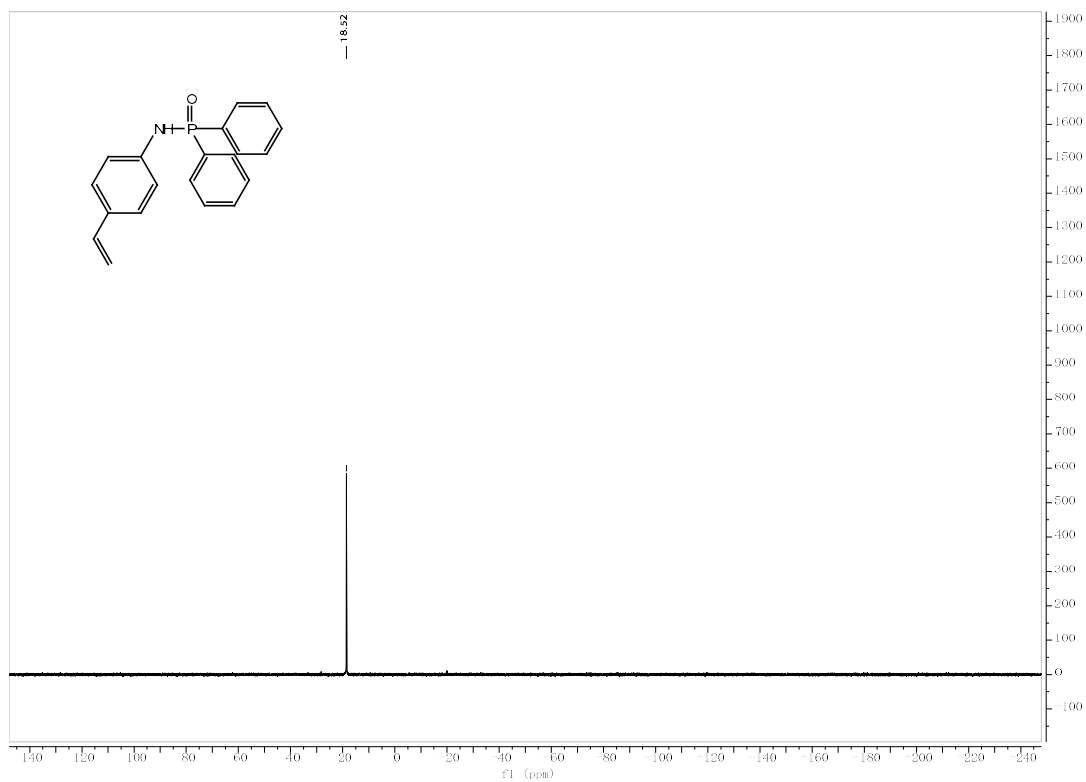
³¹P NMR of 14 (162 MHz, CDCl₃)



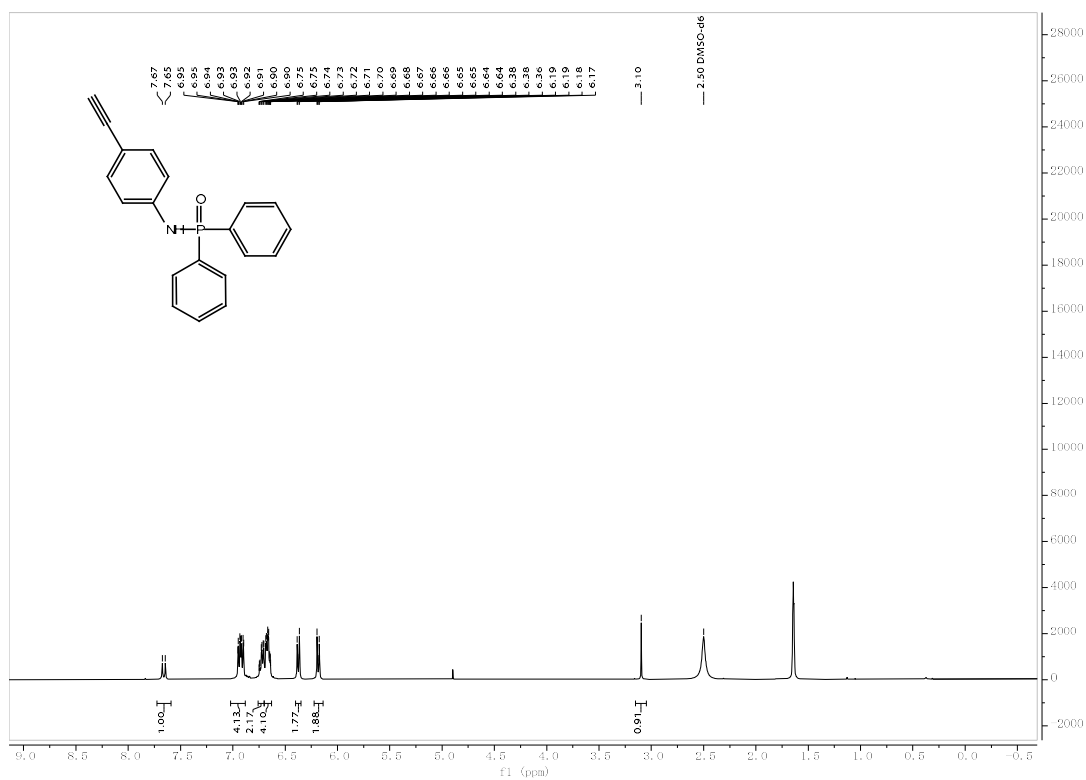
¹H NMR of 15 (400 MHz, CDCl₃)



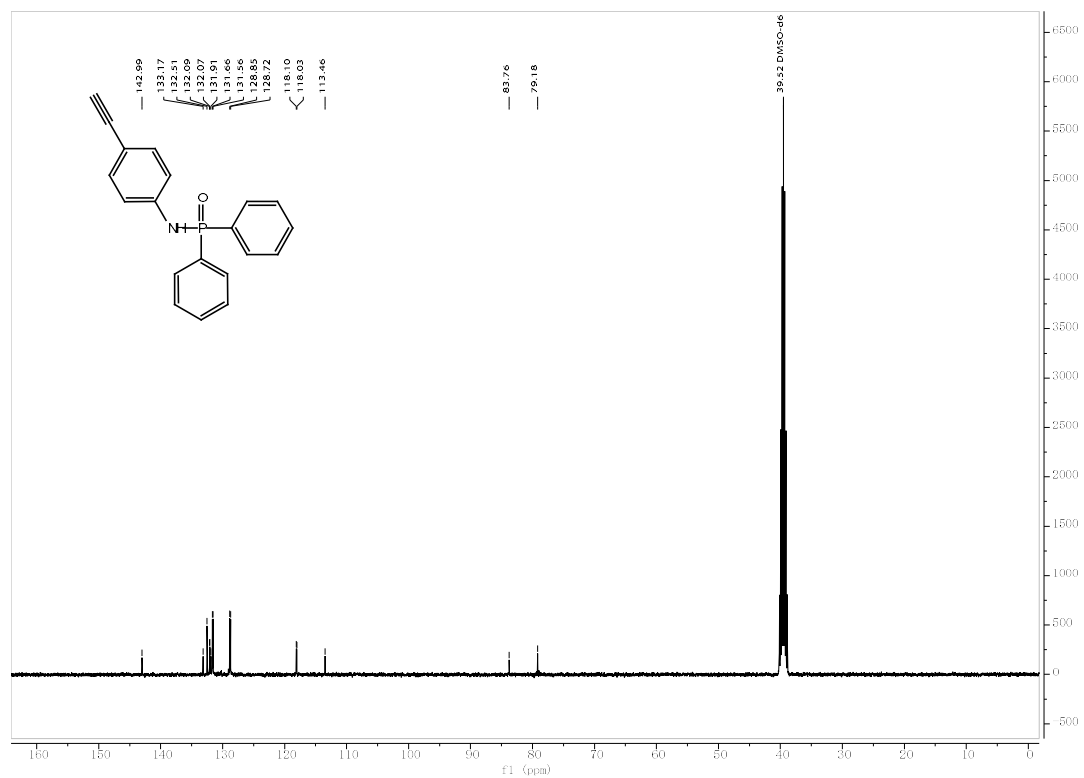
¹³C NMR of 15 (100 MHz, CDCl₃)



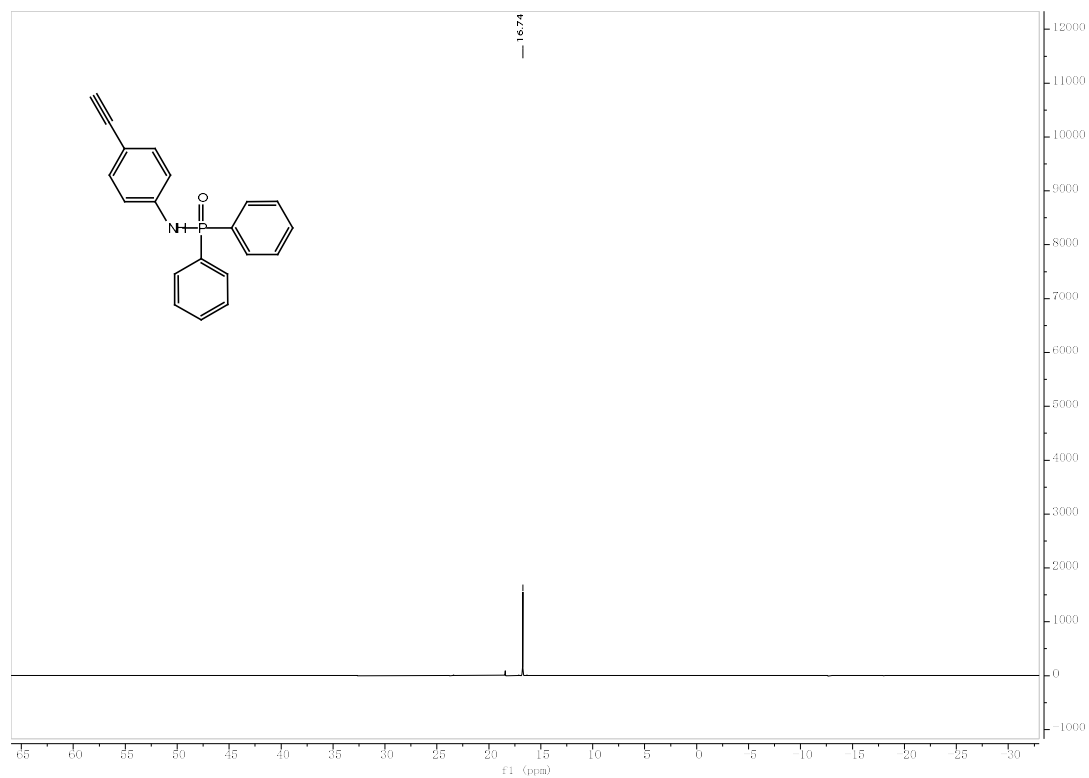
^{31}P NMR of 15 (162 MHz, CDCl_3)



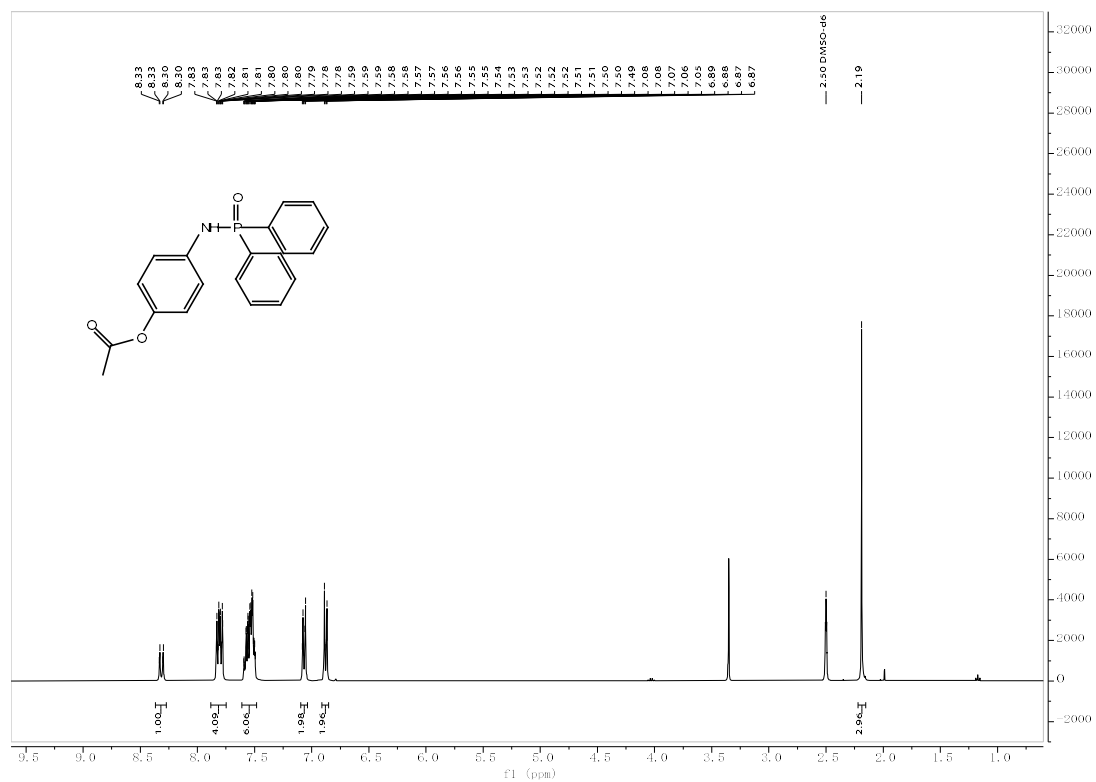
^1H NMR of 16 (400 MHz, DMSO-d_6)



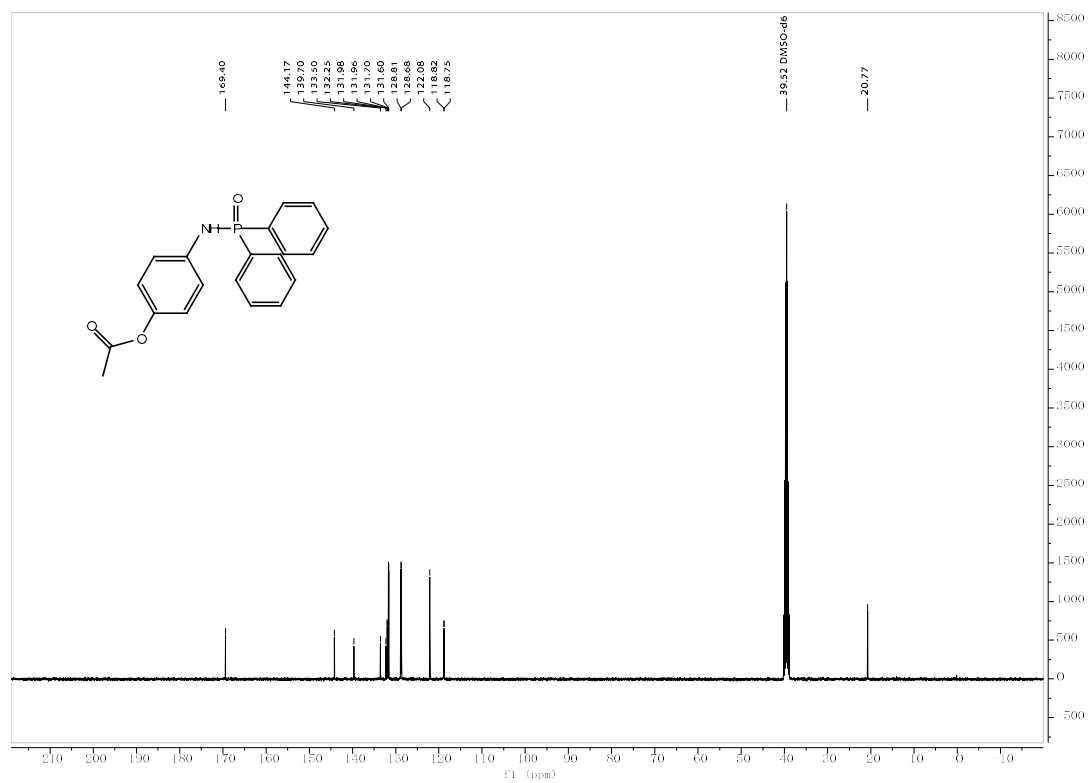
¹³C NMR of **16** (100 MHz, DMSO-d₆)



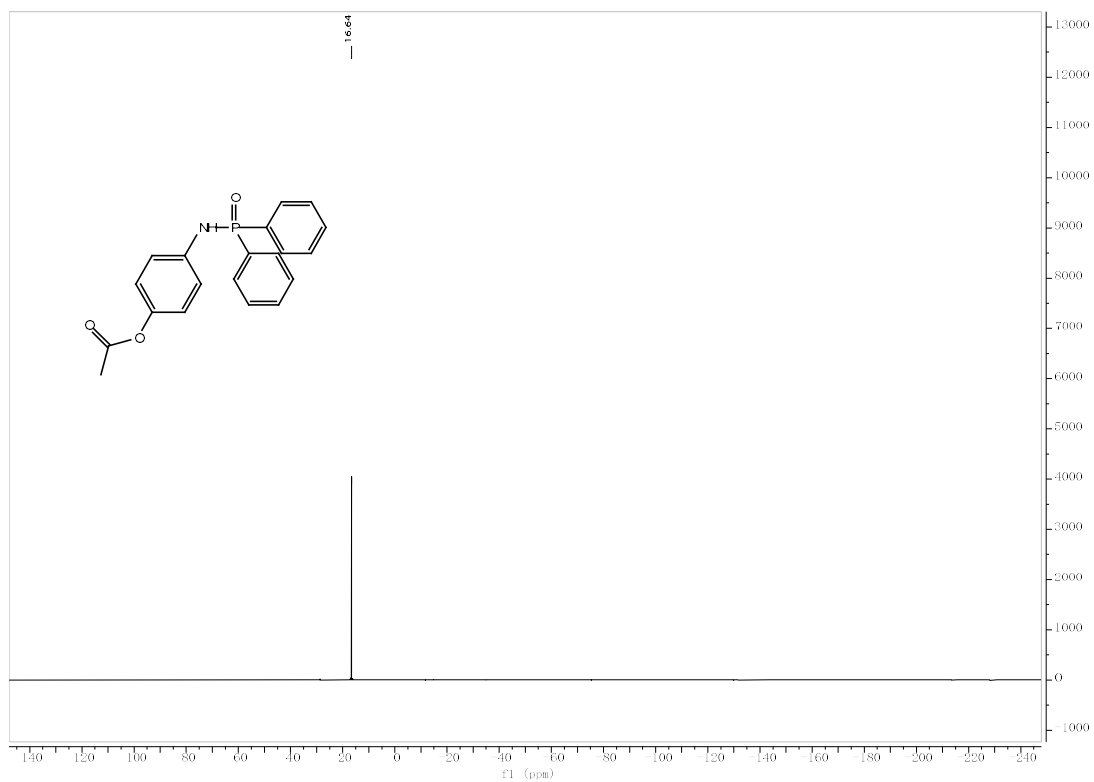
³¹P NMR of **16** (162 MHz, DMSO-d₆)



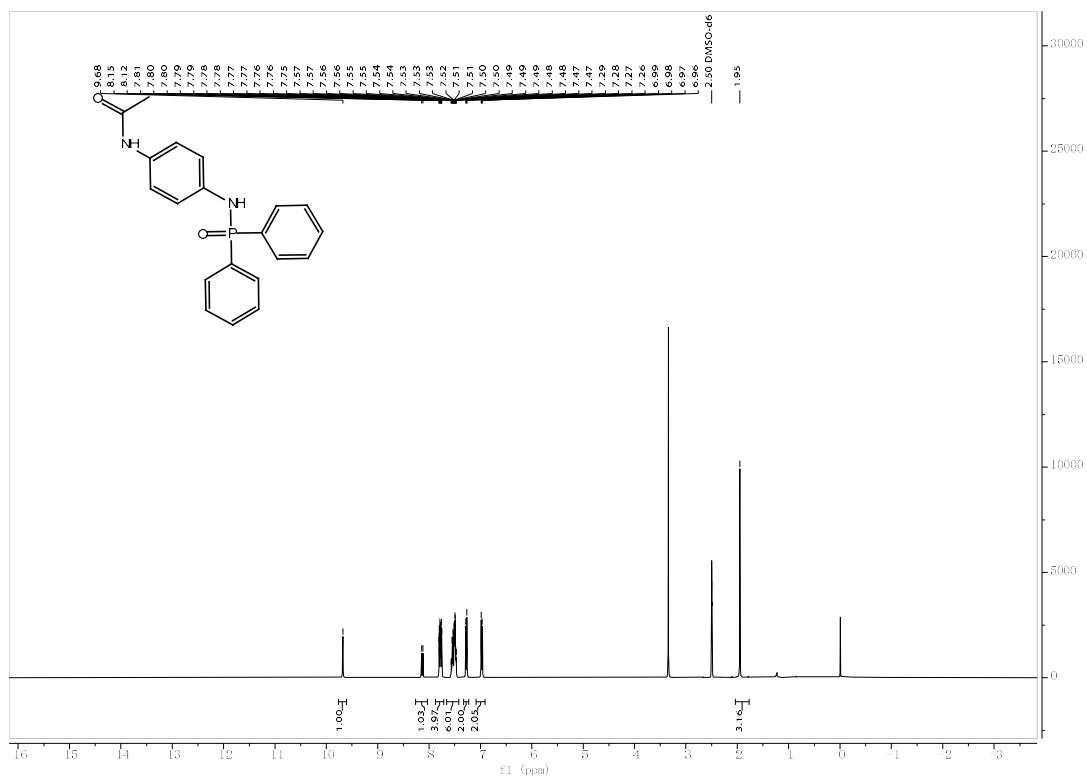
¹H NMR of 17 (400 MHz, DMSO-d₆)



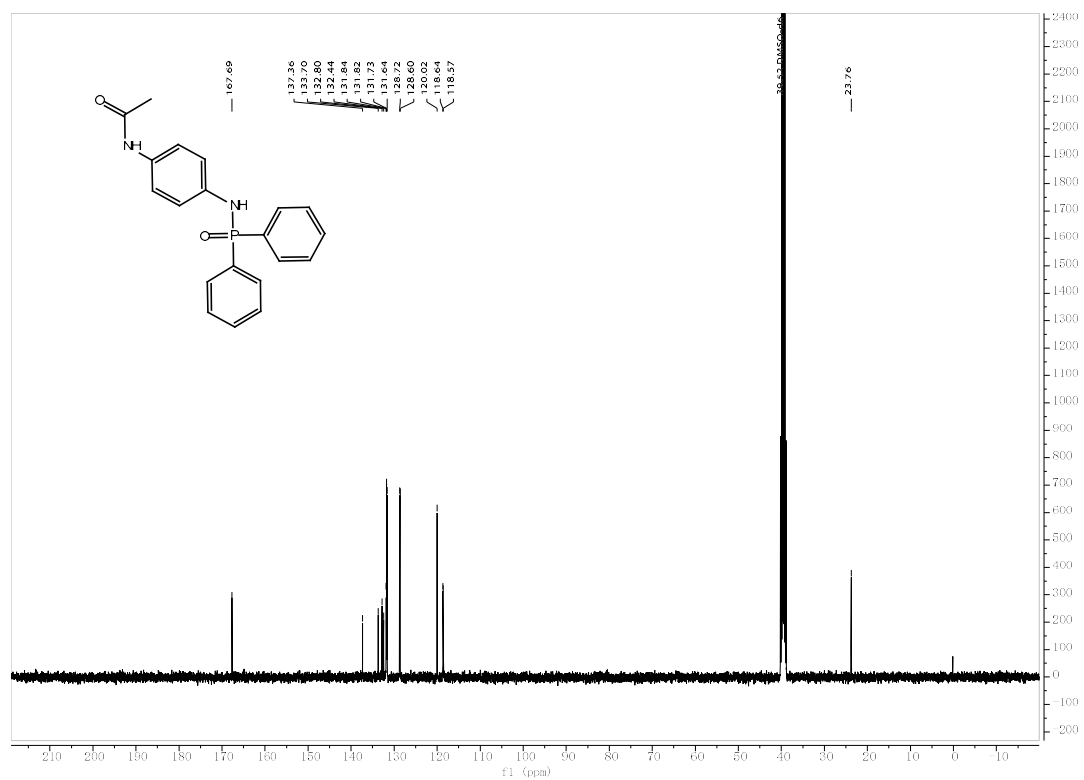
¹³C NMR of 17 (100 MHz, DMSO-d₆)



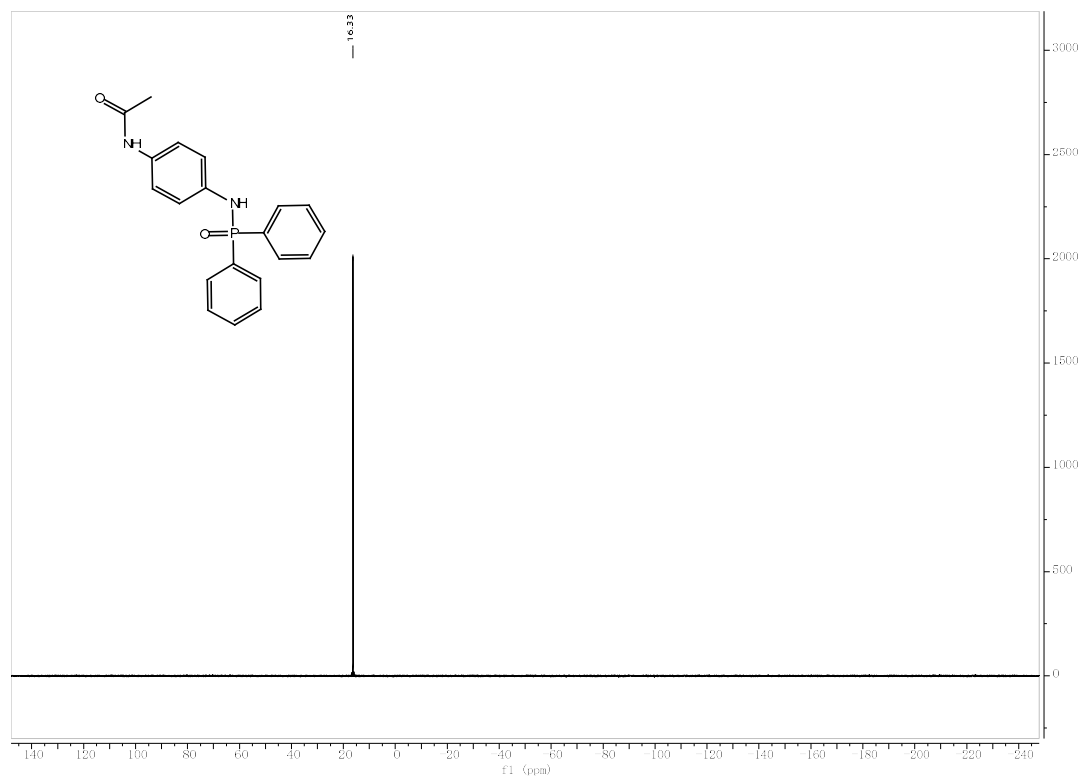
^{31}P NMR of 17 (162 MHz, DMSO- d_6)



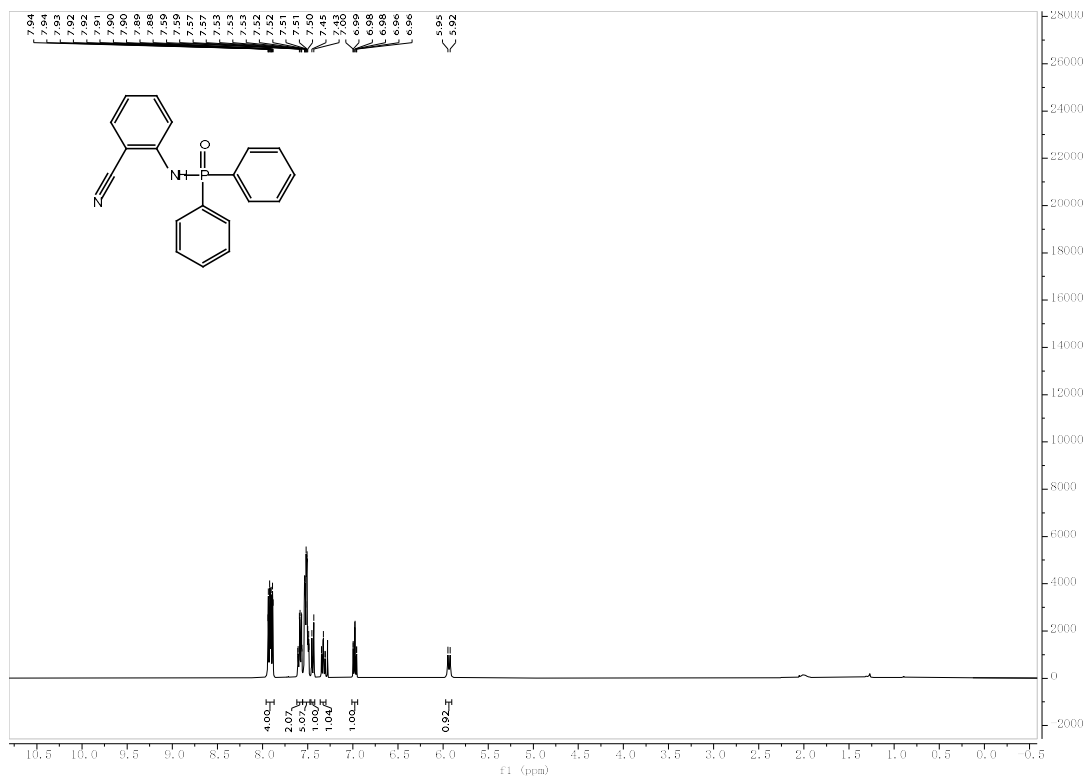
^1H NMR of 18 (400 MHz, DMSO- d_6)



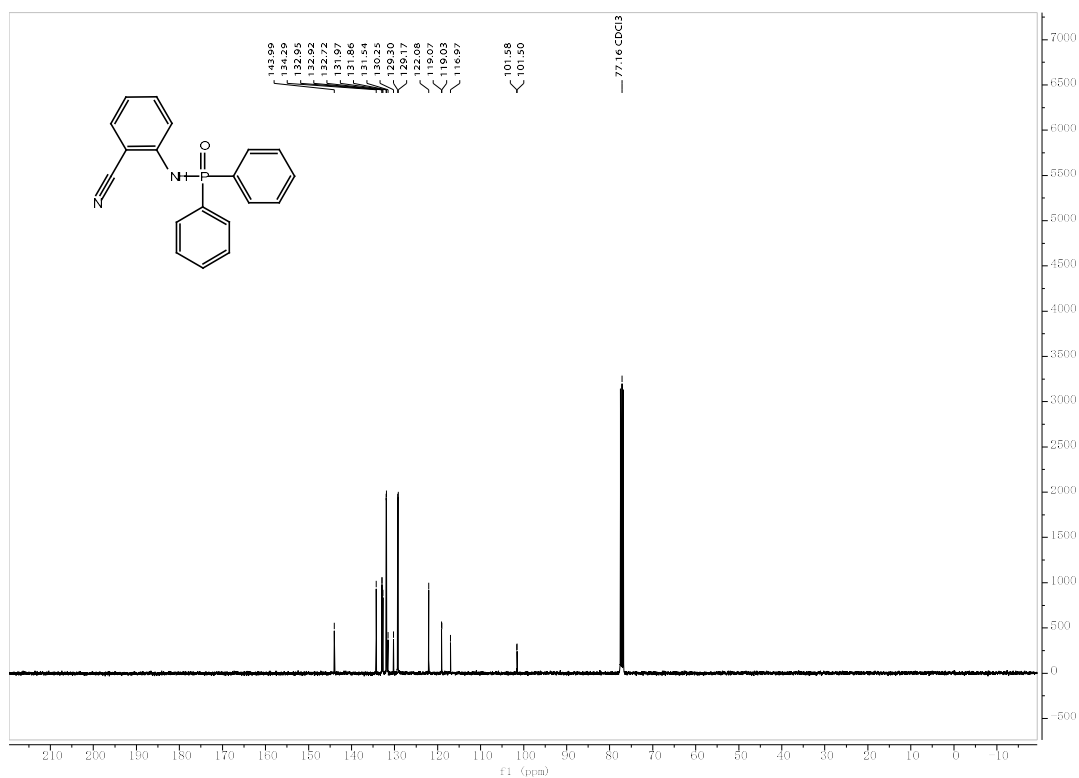
¹³C NMR of **18** (100 MHz, DMSO-d₆)



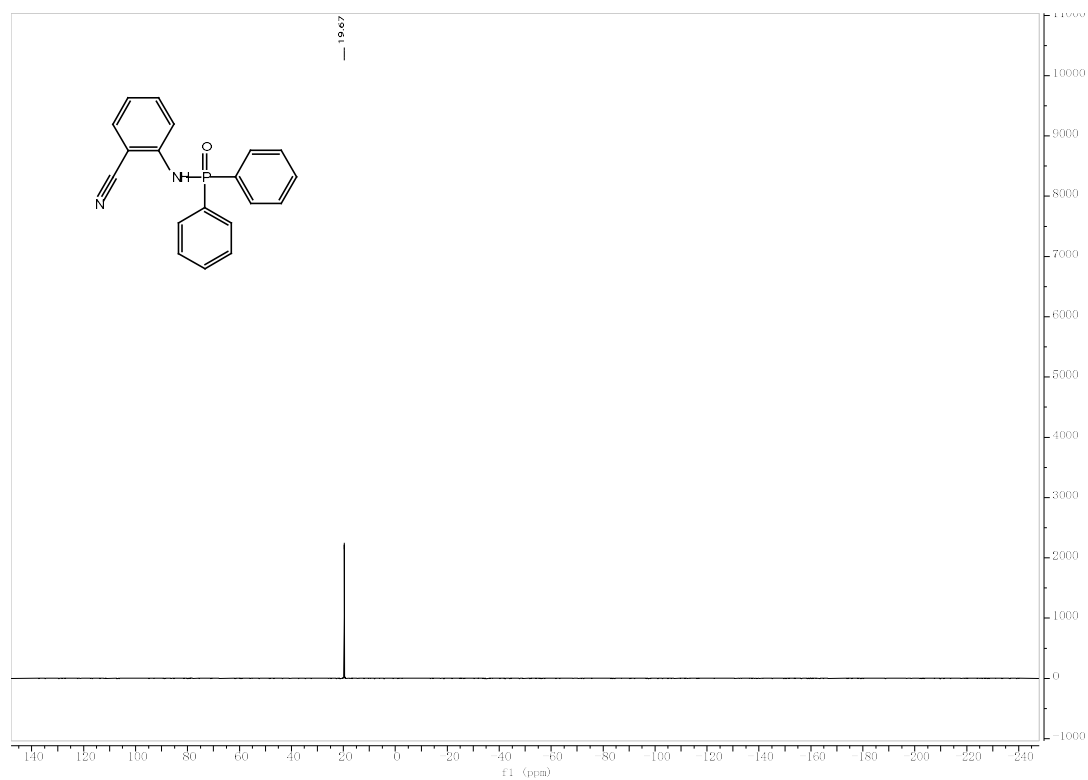
³¹P NMR of **18** (162 MHz, DMSO-d₆)



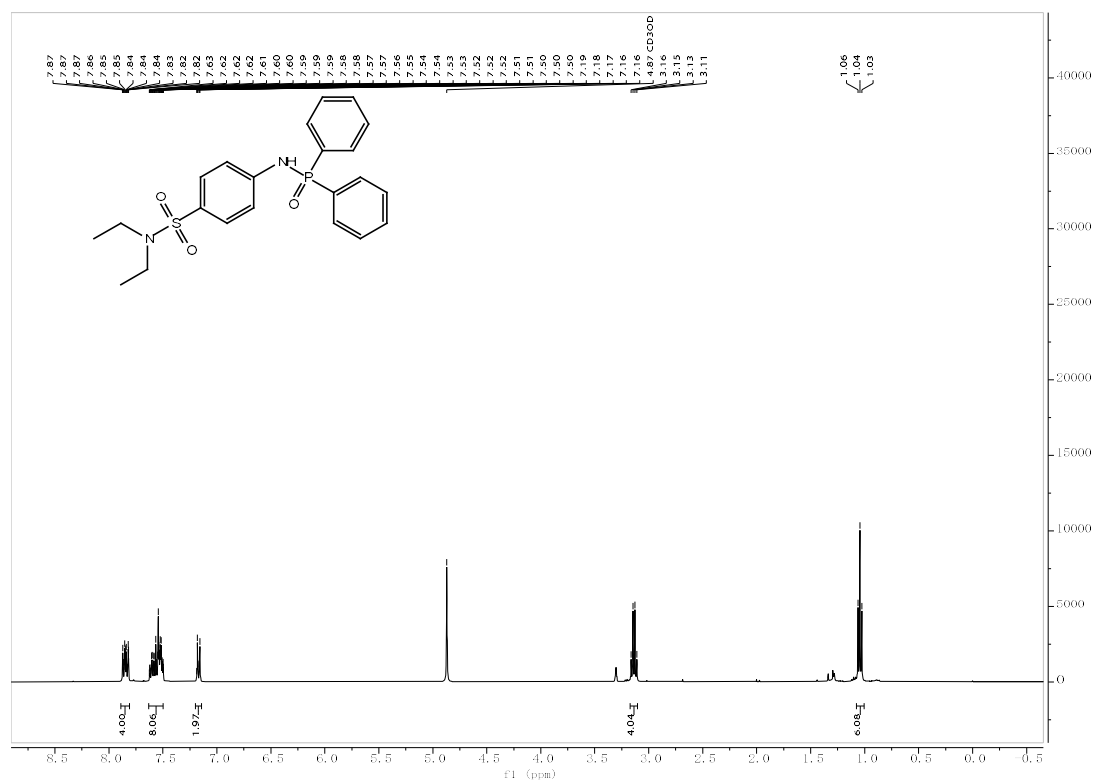
¹H NMR of 19 (400 MHz, CDCl₃)



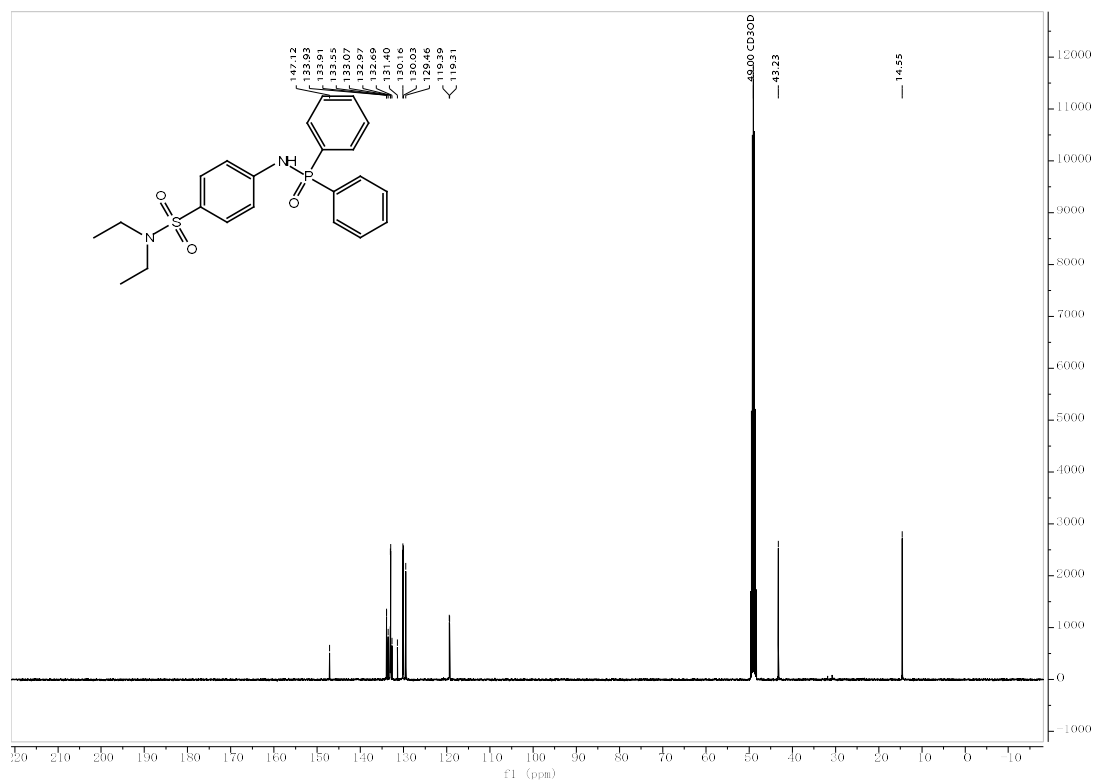
¹³C NMR of 19 (100 MHz, CDCl₃)



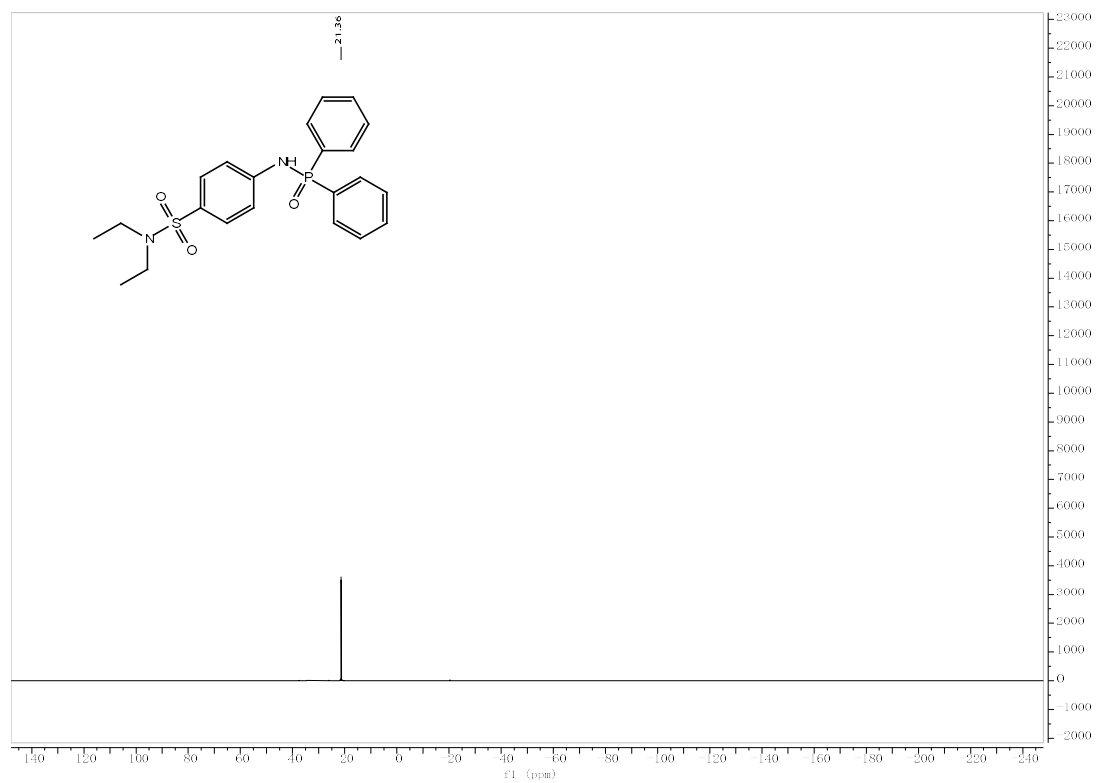
^{31}P NMR of 19 (162 MHz, CDCl_3)



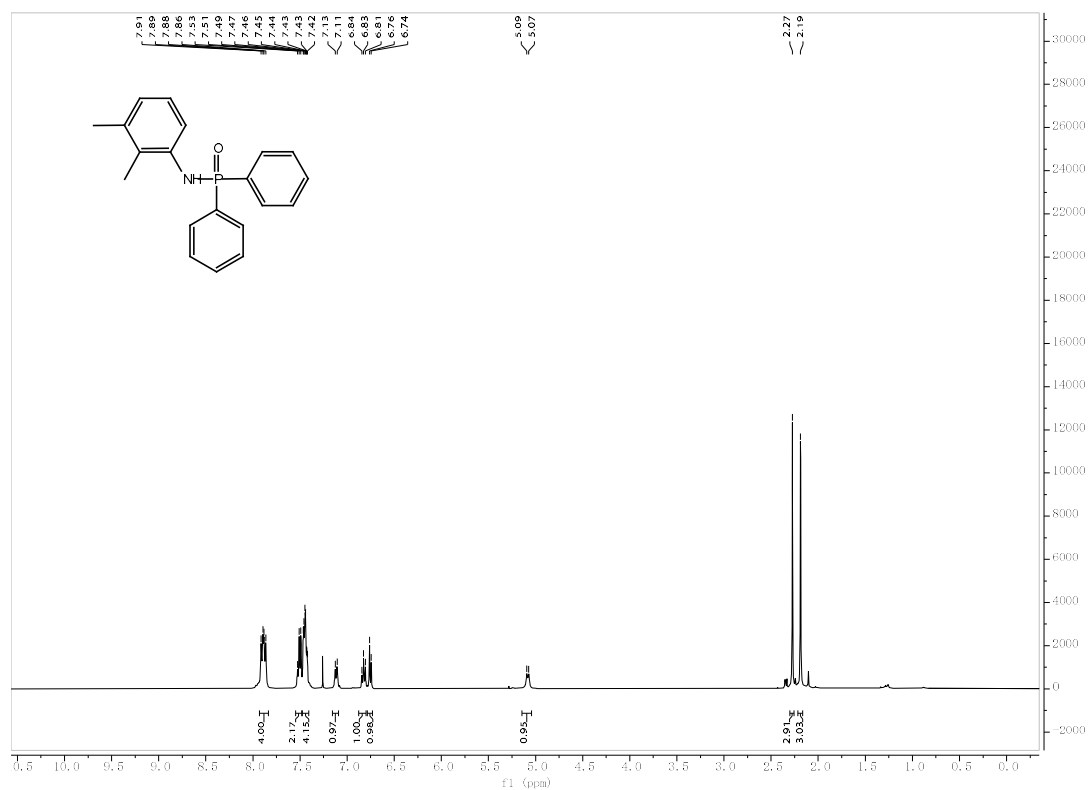
^1H NMR of 20 (400 MHz, CDCl_3)



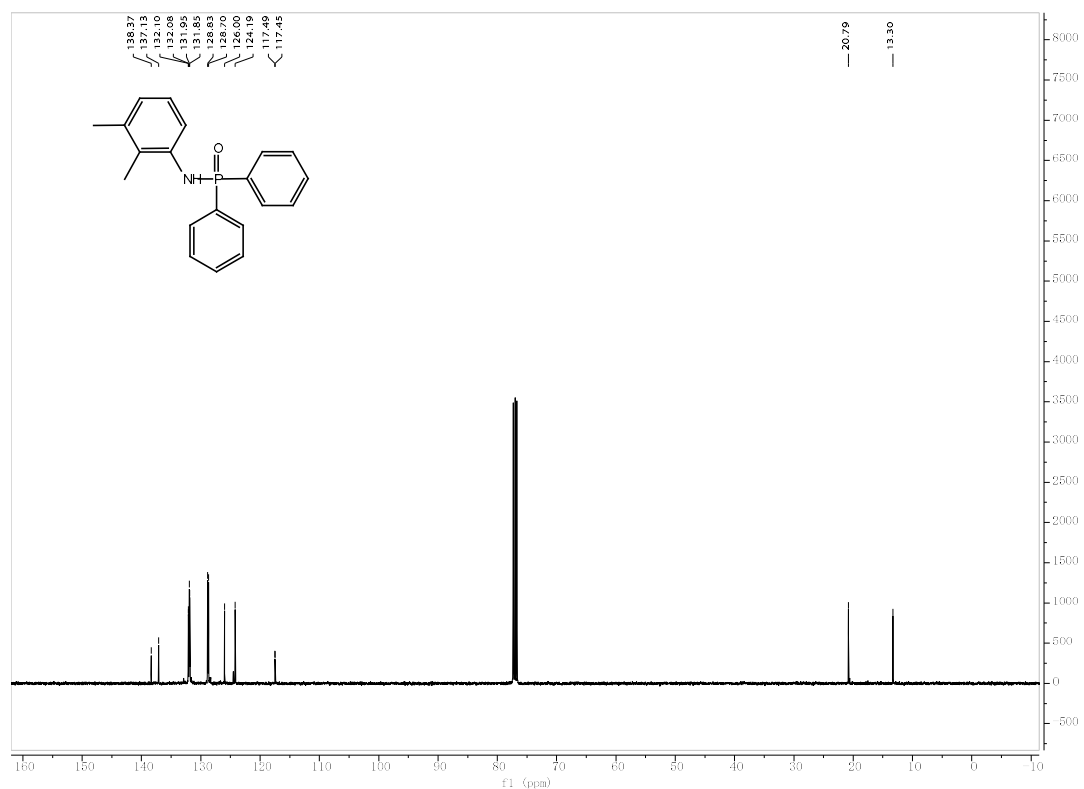
^{13}C NMR of **20** (100 MHz, CDCl₃)



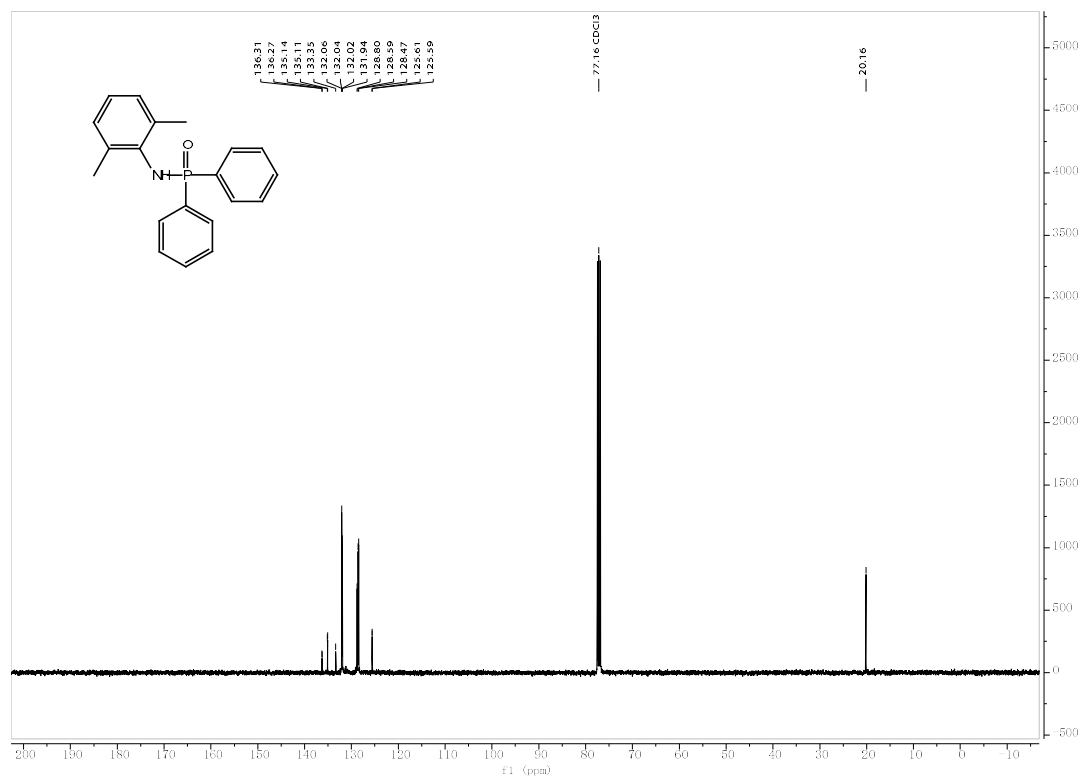
^{31}P NMR of **20** (162 MHz, CDCl₃)



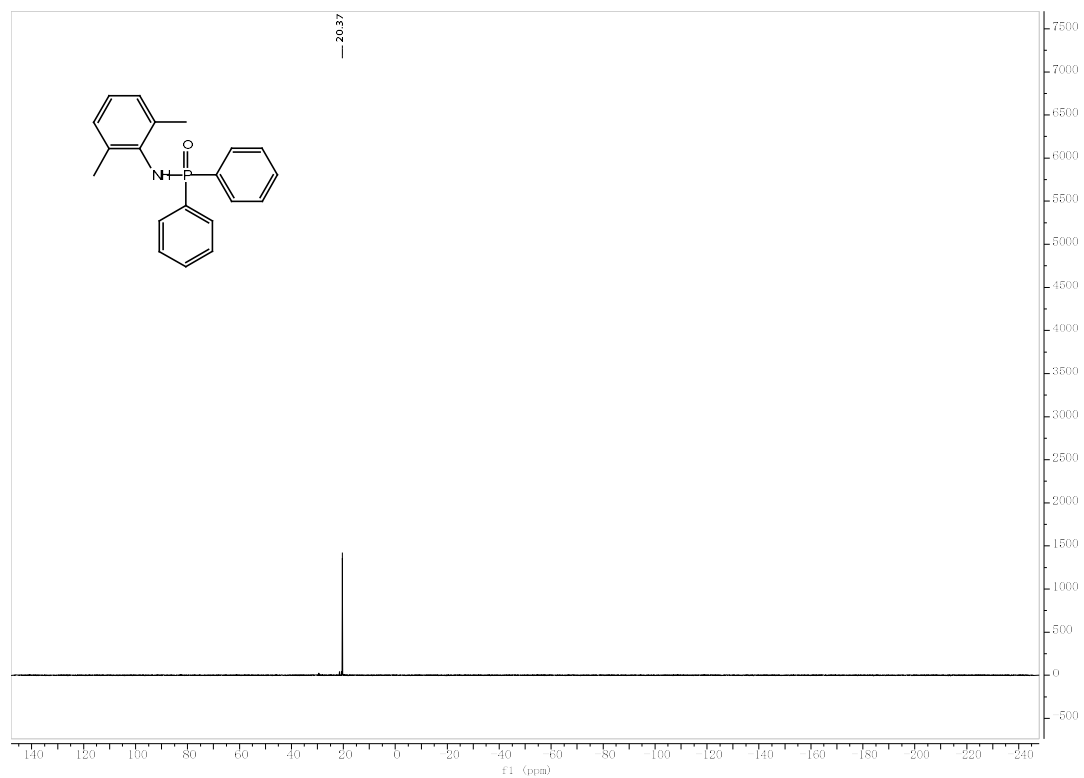
¹H NMR of 21 (400 MHz, CDCl₃)



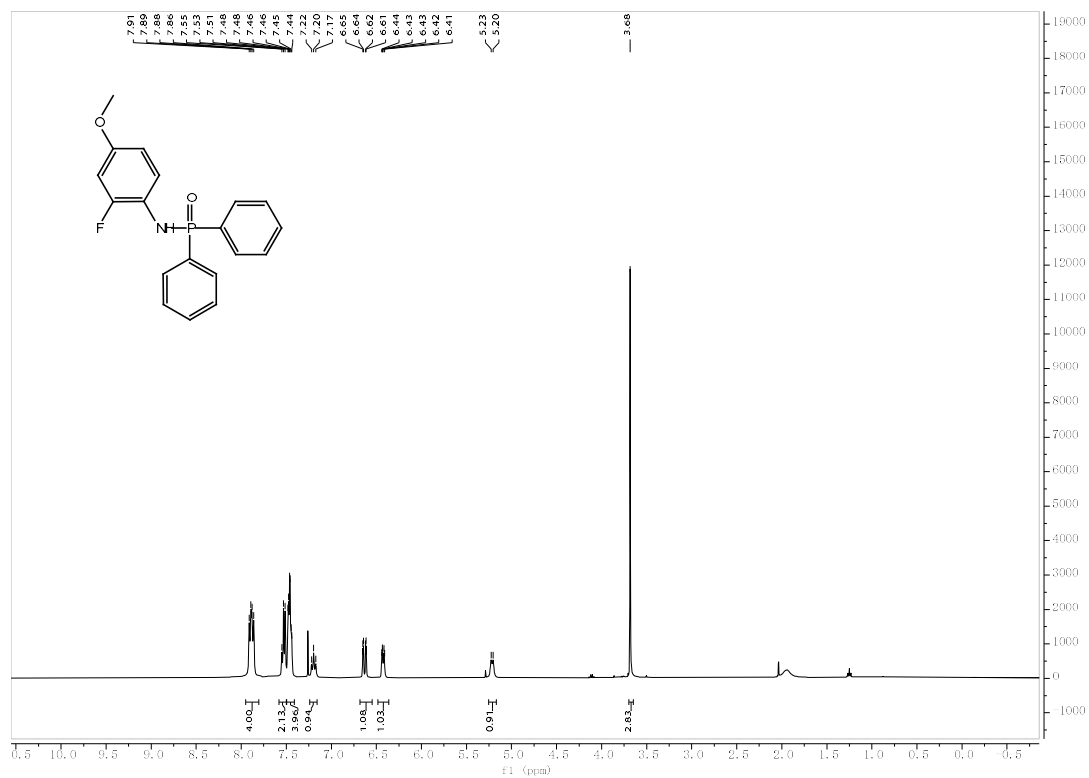
¹³C NMR of 21 (100 MHz, CDCl₃)



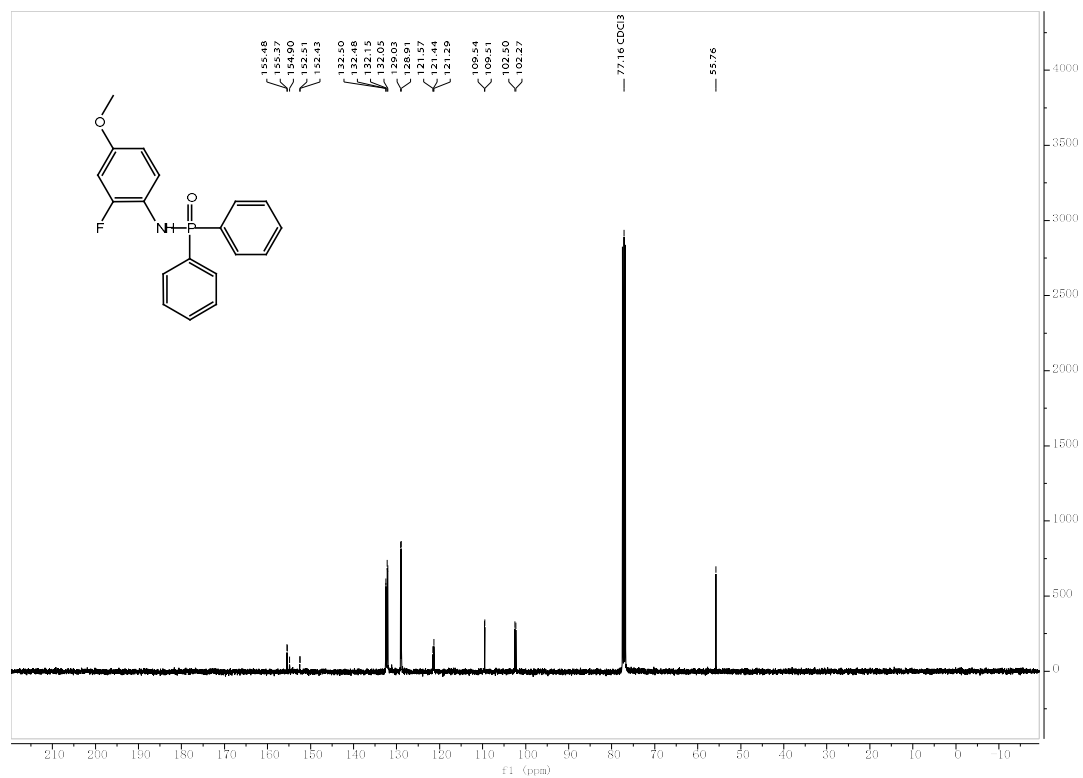
¹³C NMR of **22** (100 MHz, CDCl₃)



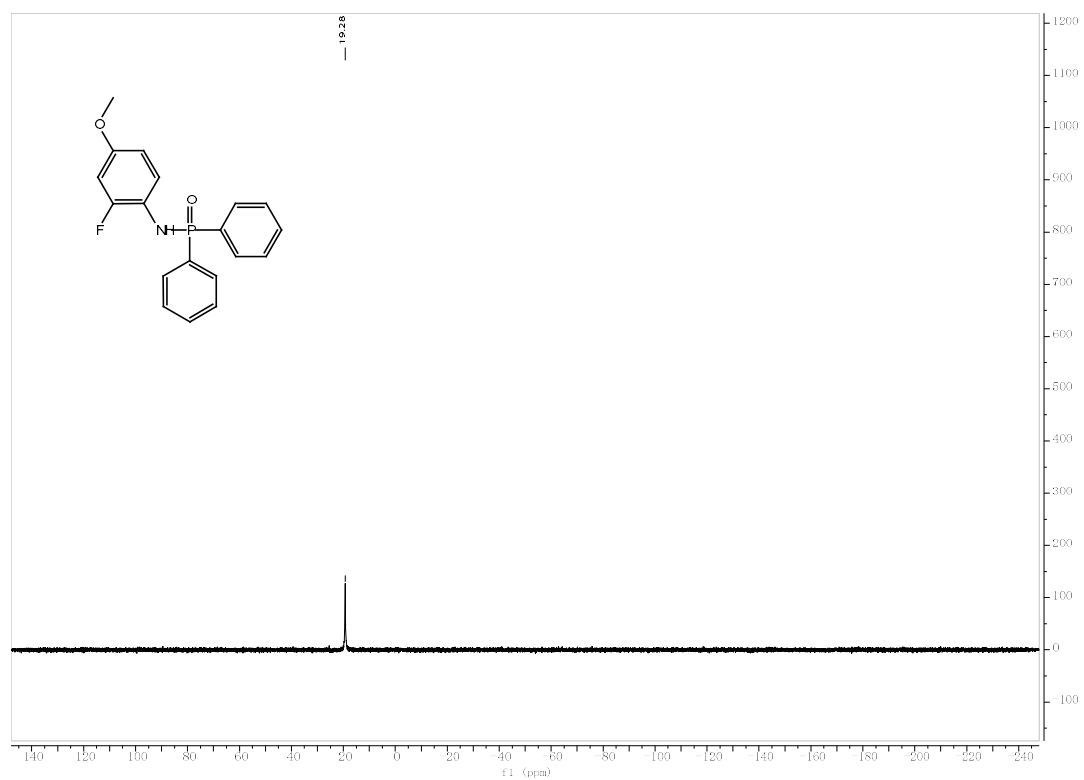
³¹P NMR of **22** (162 MHz, CDCl₃)



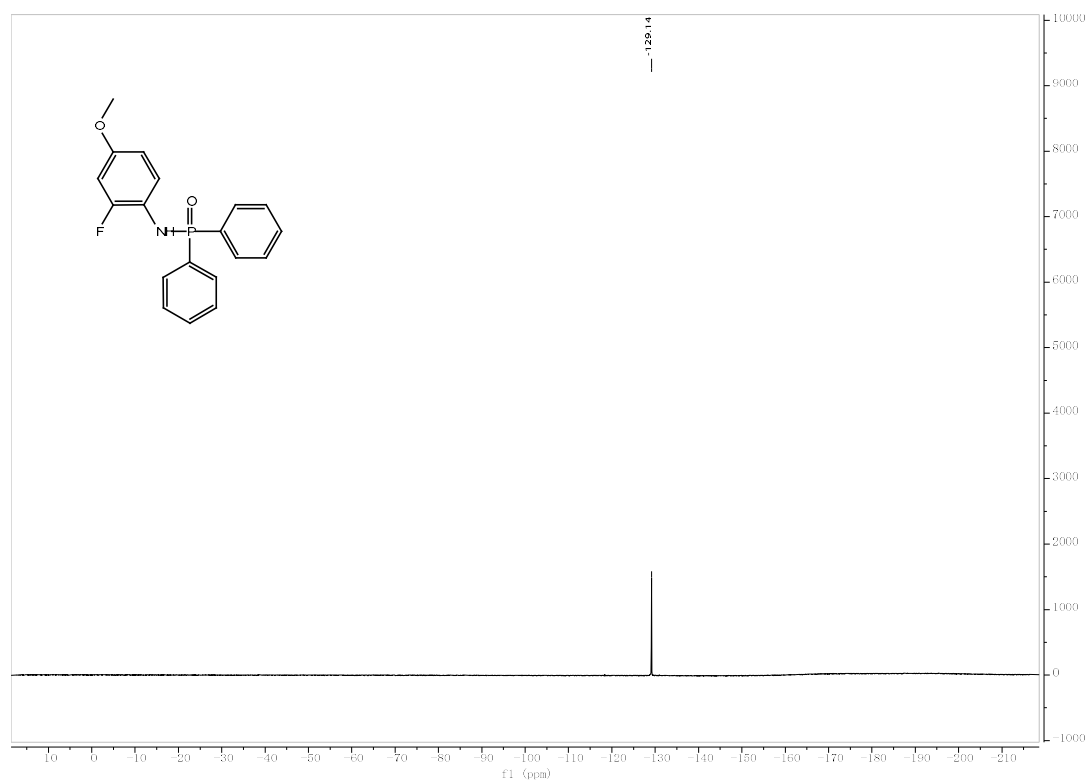
¹H NMR of 23 (400 MHz, CDCl₃)



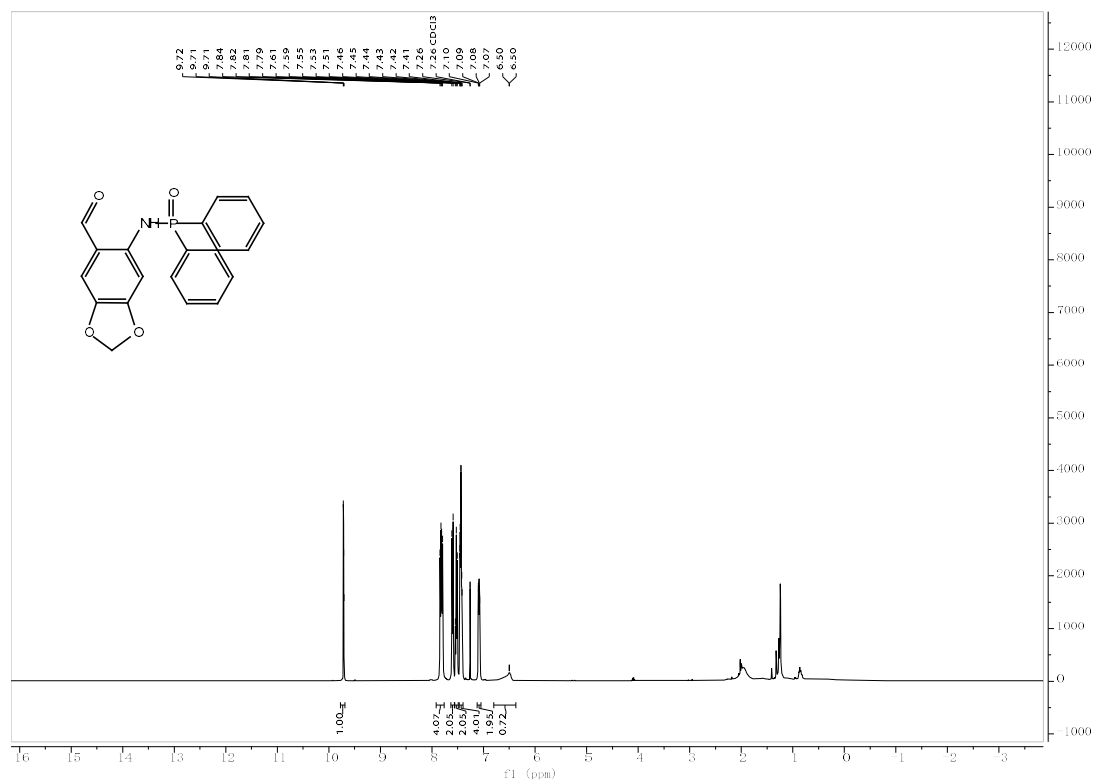
¹³C NMR of 23 (100 MHz, CDCl₃)



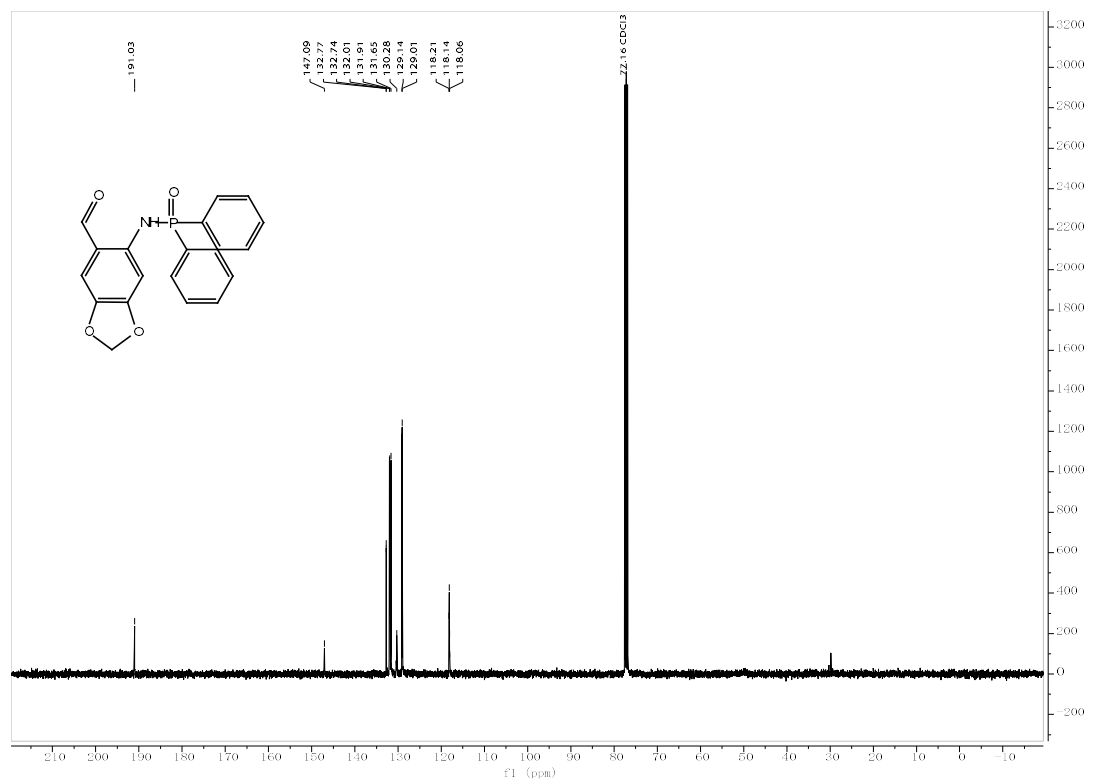
^{31}P NMR of **23** (162 MHz, CDCl_3)



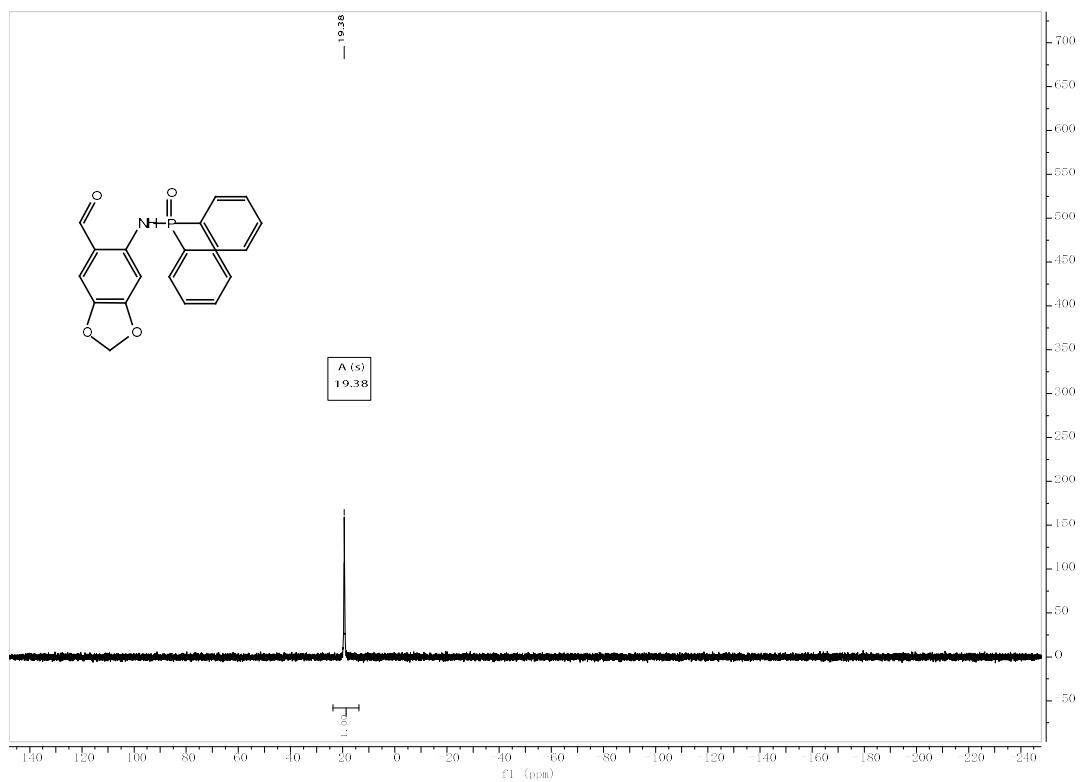
^{18}F NMR of **23** (376 MHz, CDCl_3)



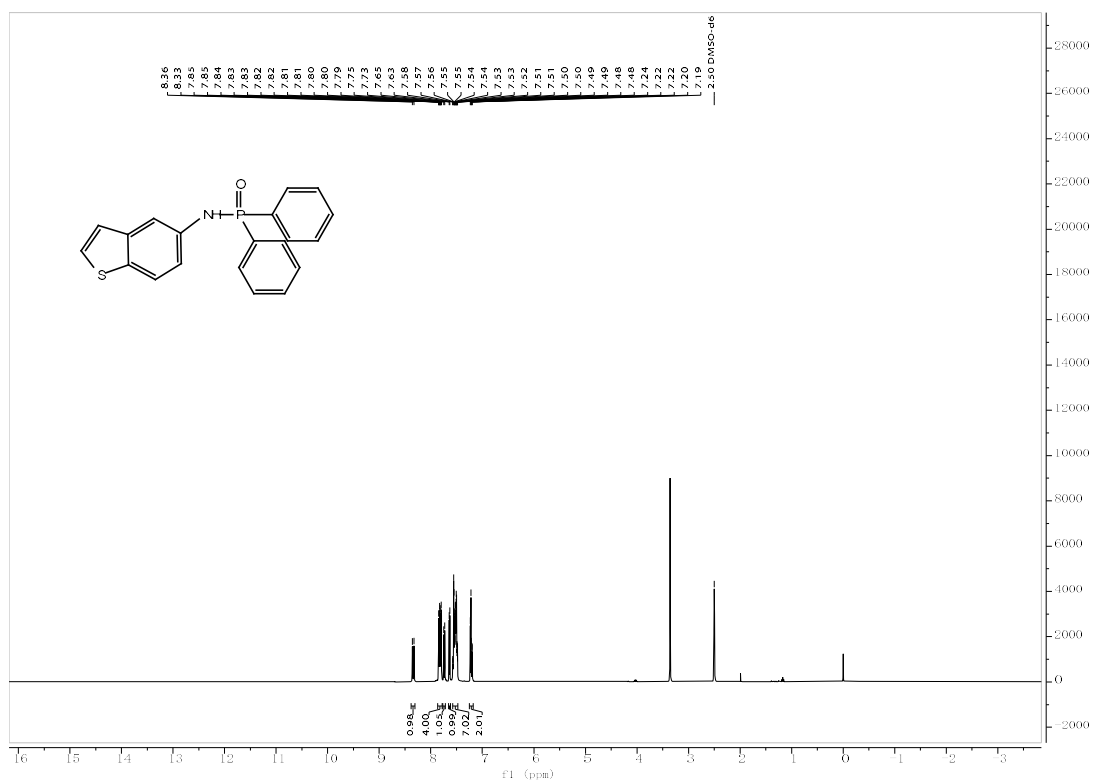
¹H NMR of 24 (400 MHz, CDCl₃)



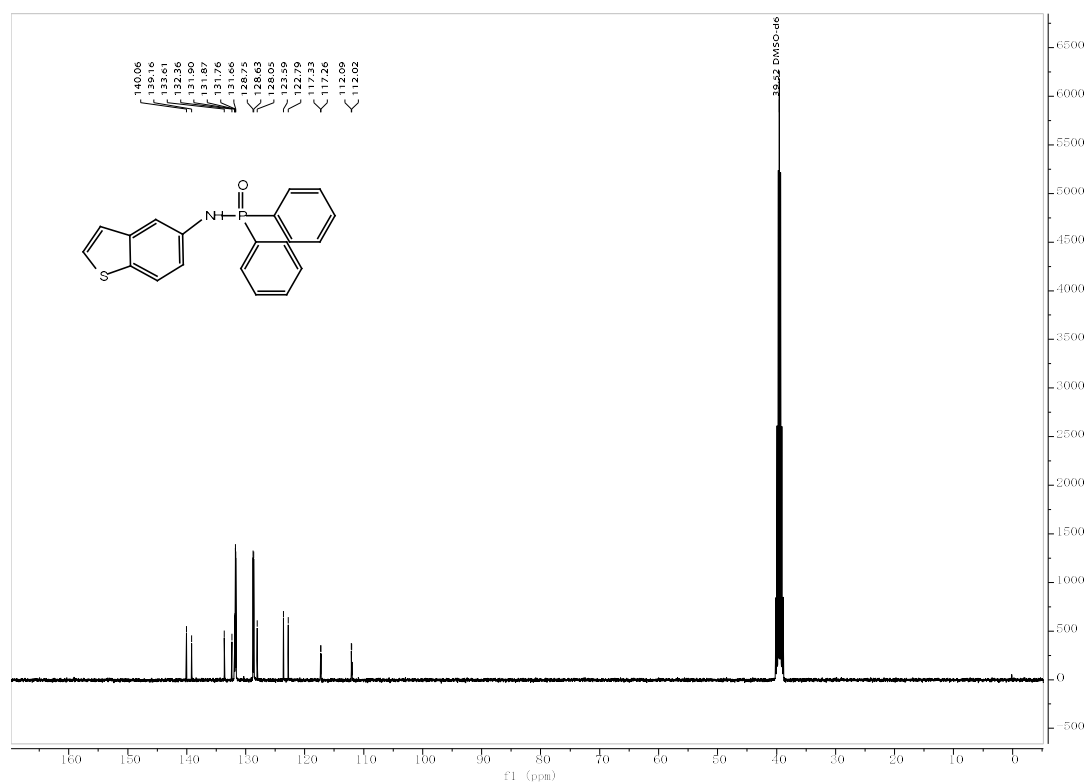
¹³C NMR of 24 (100 MHz, CDCl₃)



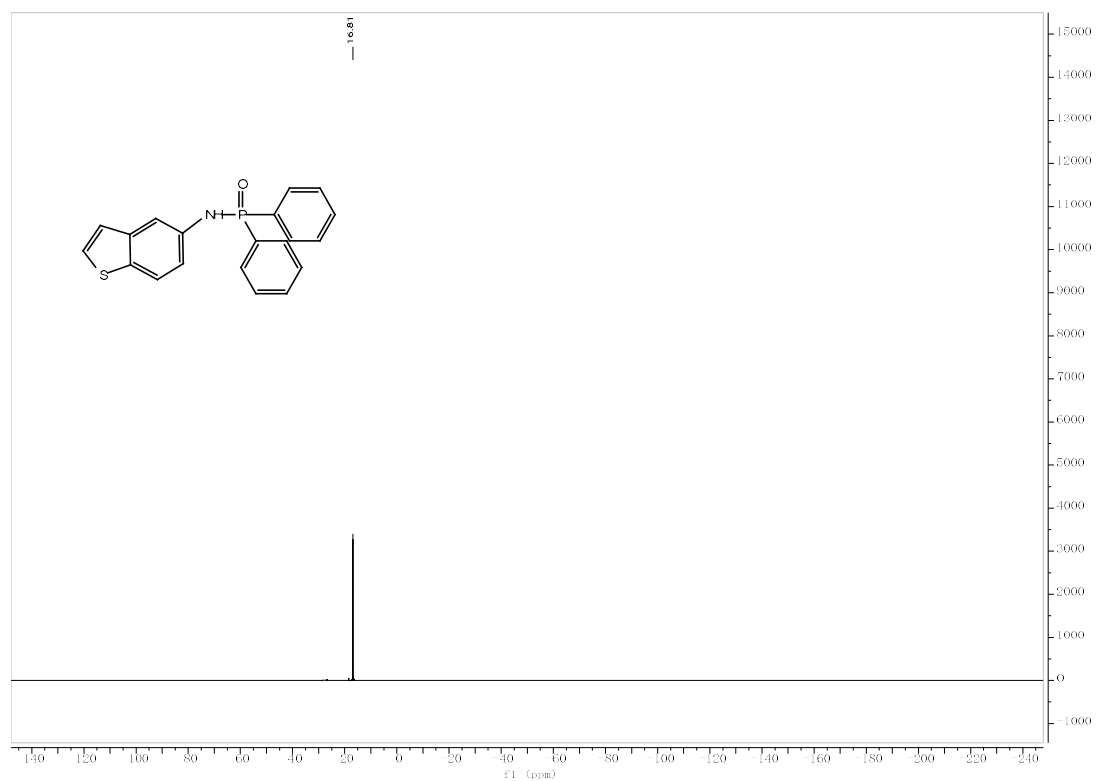
^{31}P NMR of 24 (162 MHz, CDCl_3)



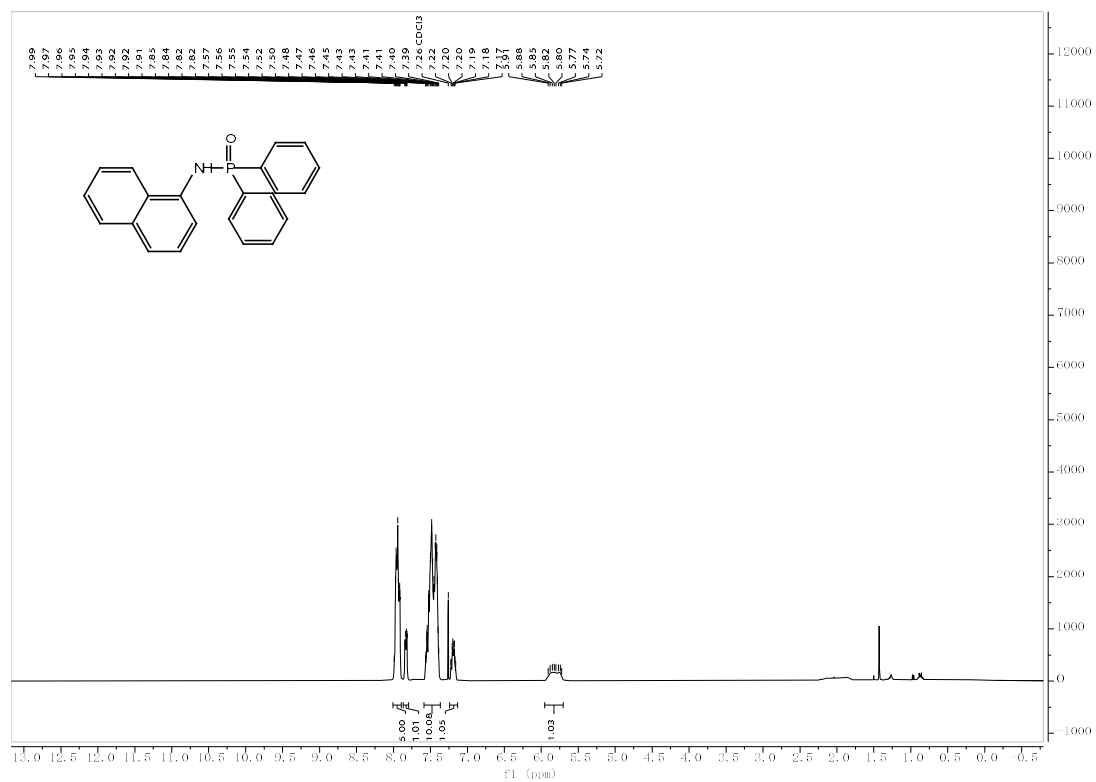
^1H NMR of 25 (400 MHz, DMSO-d_6)



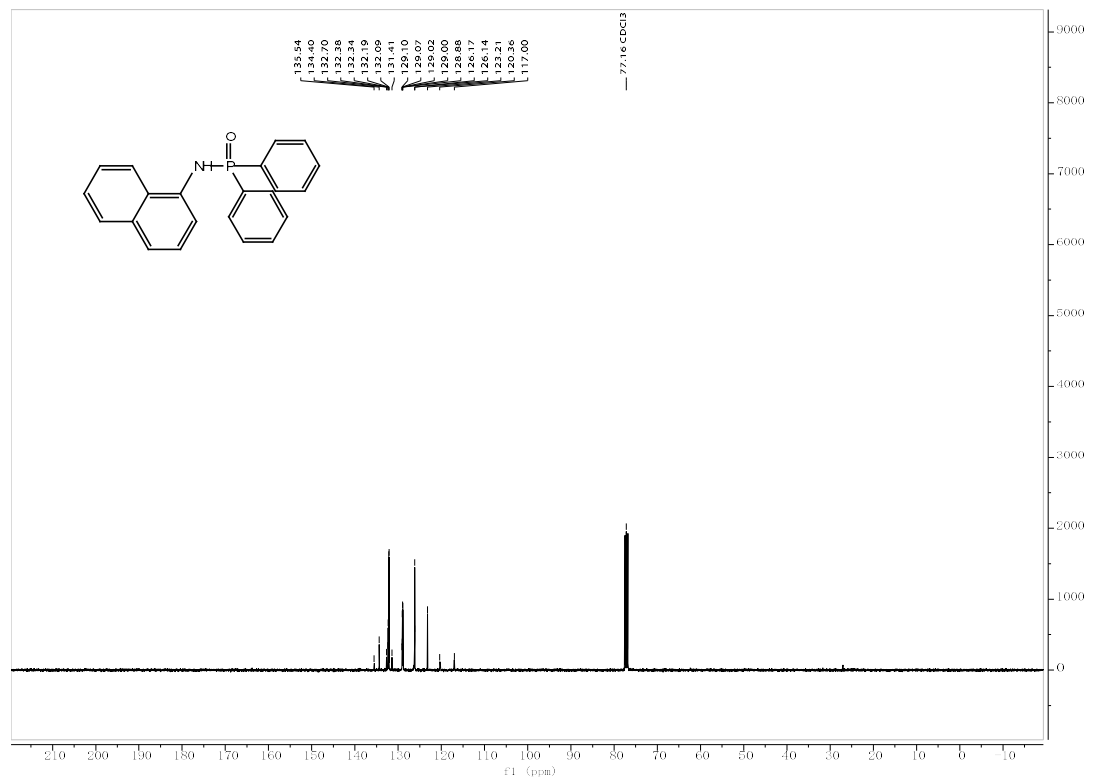
¹³C NMR of 25 (100 MHz, DMSO-d₆)



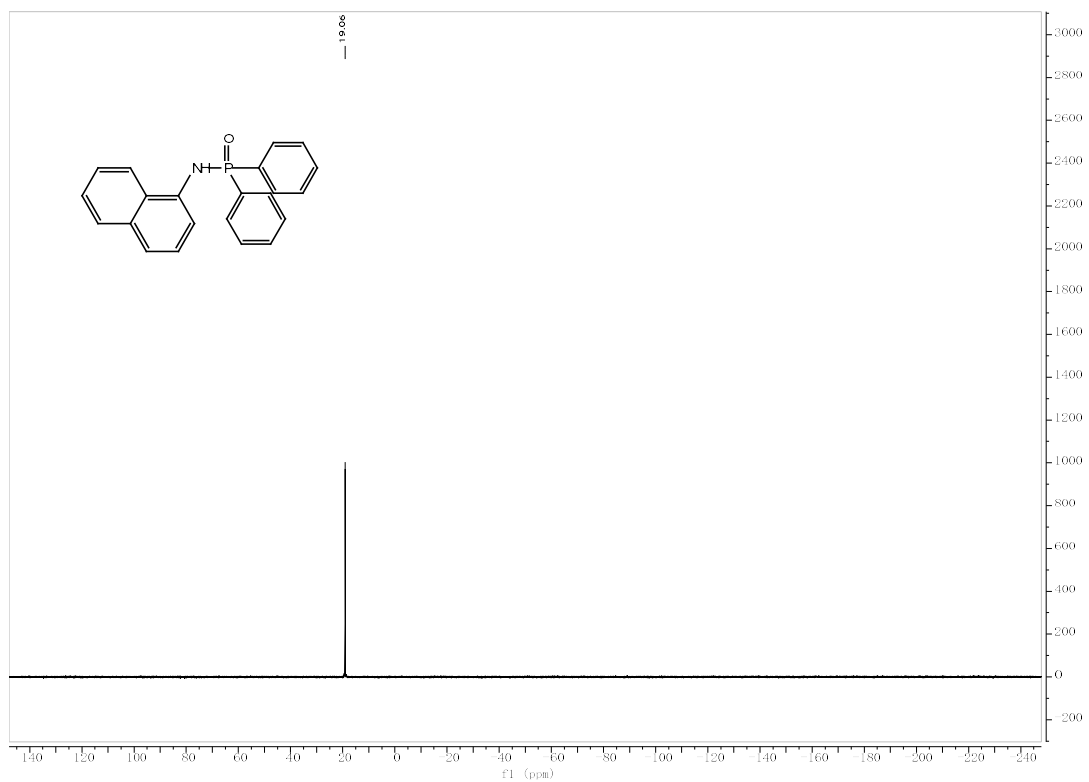
³¹P NMR of 25 (162 MHz, DMSO-d₆)



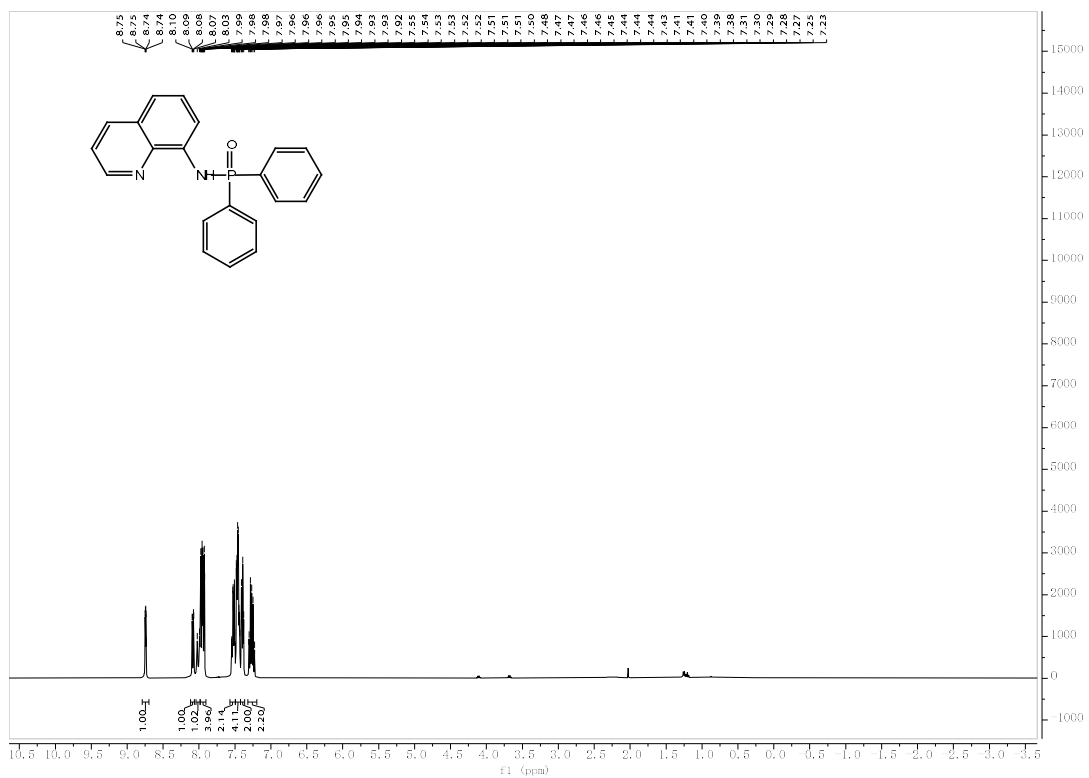
¹H NMR of 26 (400 MHz, CDCl₃)



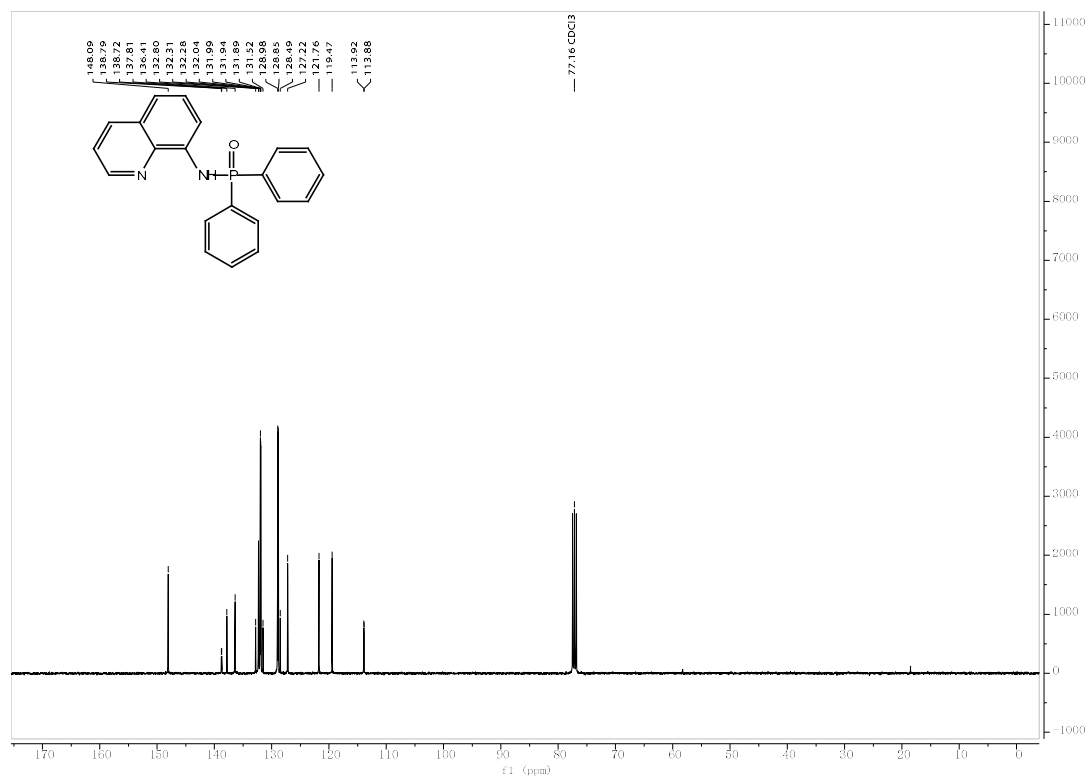
¹³C NMR of 26 (100 MHz, CDCl₃)



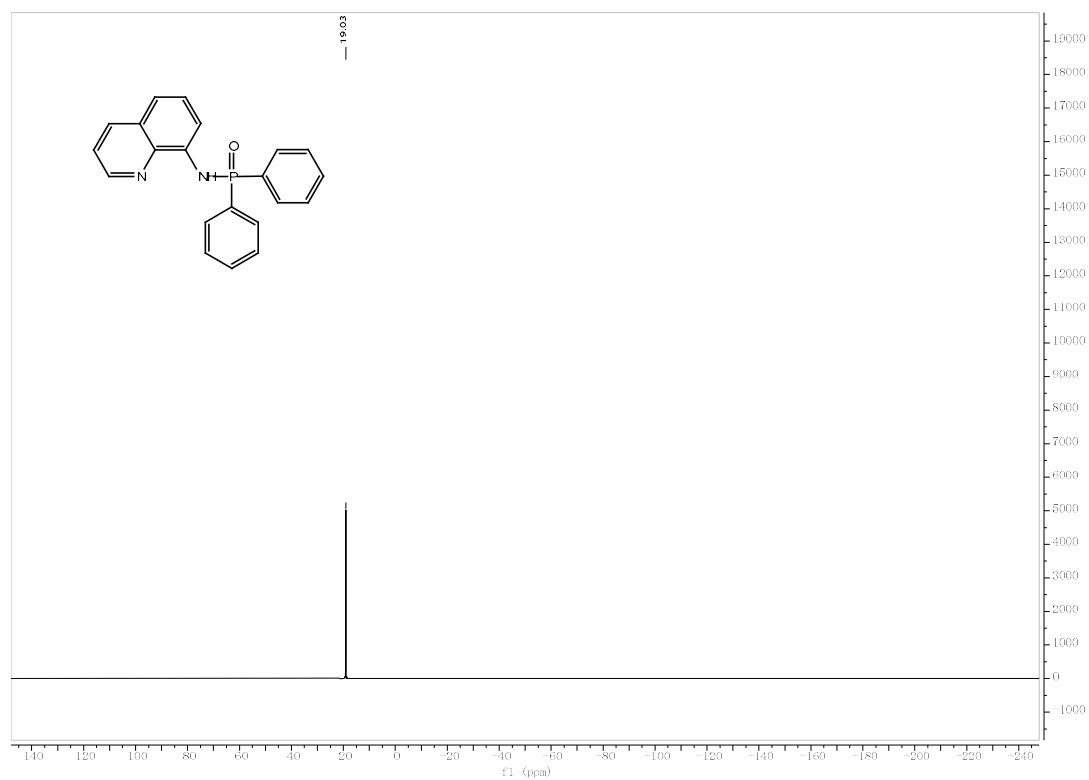
^{31}P NMR of **26** (162 MHz, CDCl_3)



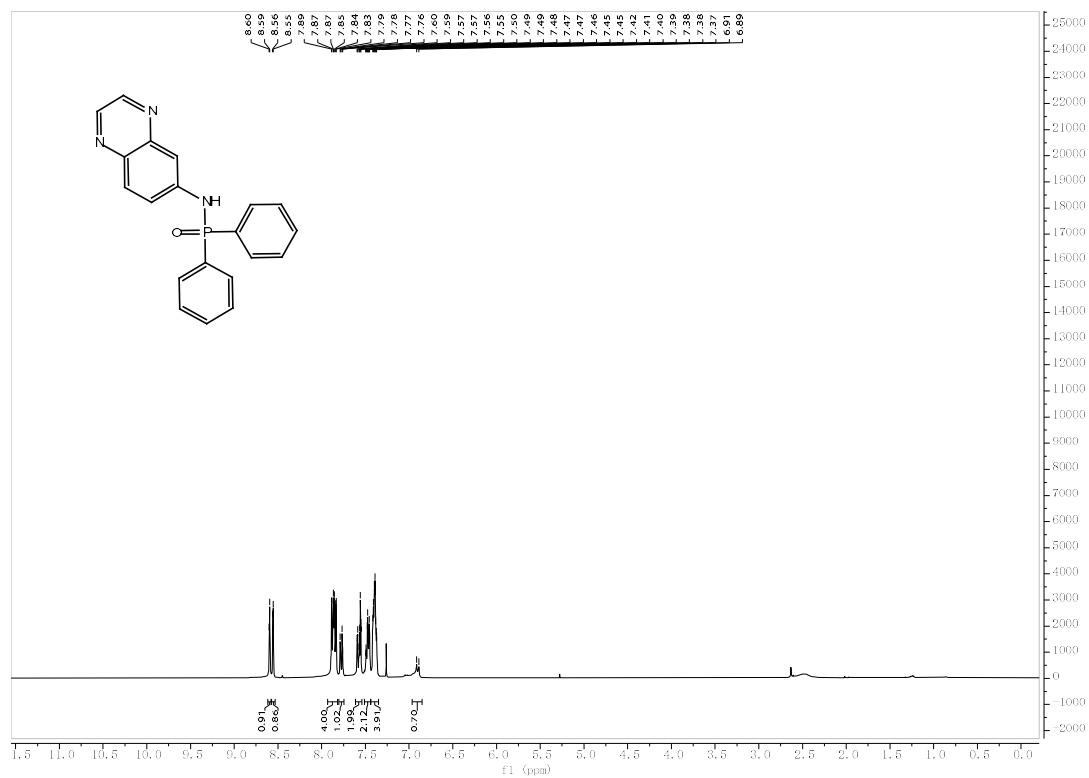
^1H NMR of **27** (400 MHz, CDCl_3)



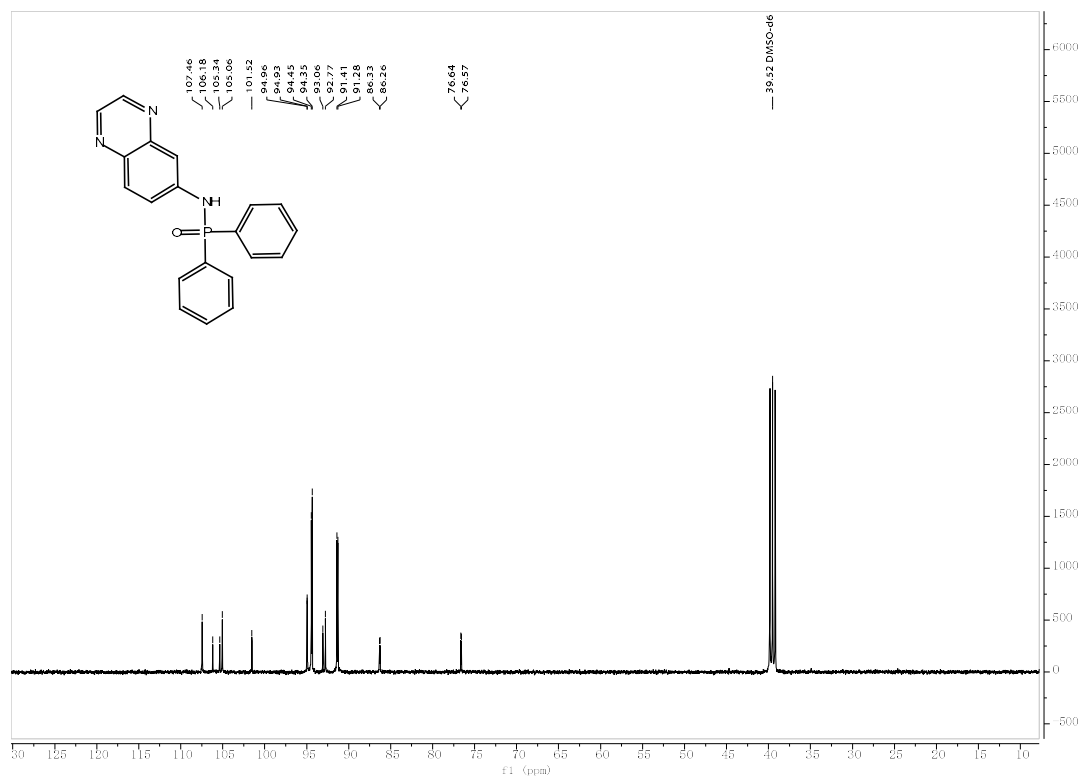
^{13}C NMR of 27 (100 MHz, CDCl₃)



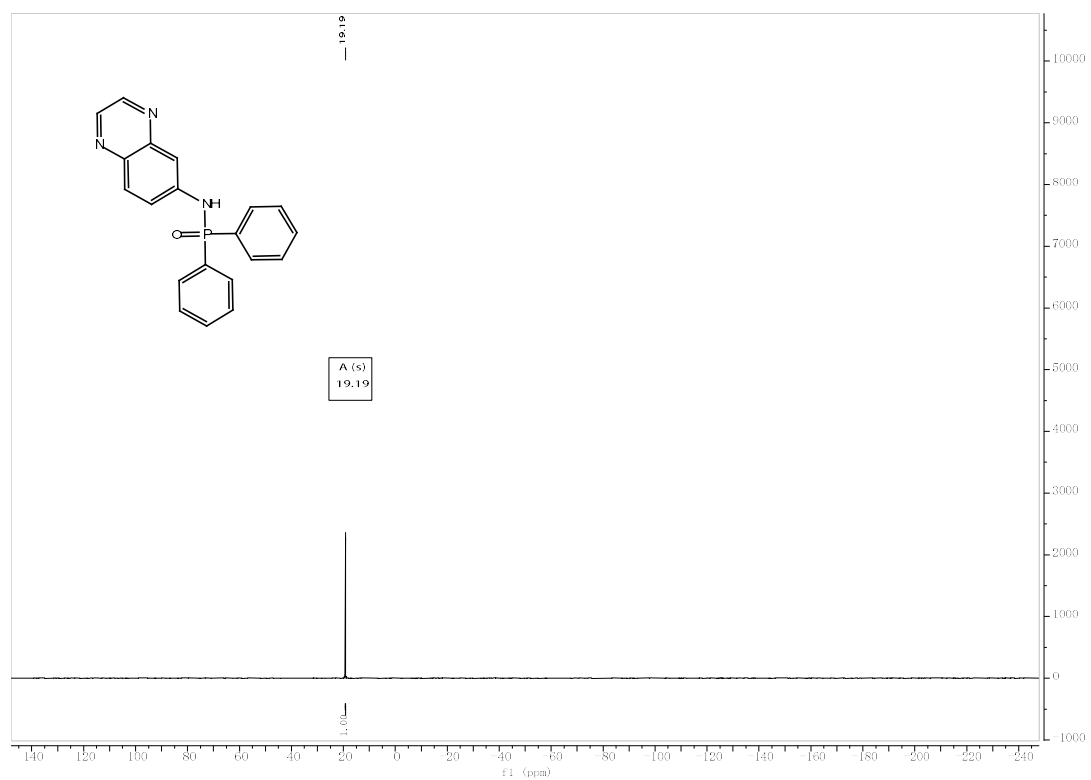
^{31}P NMR of 27 (162 MHz, CDCl₃)



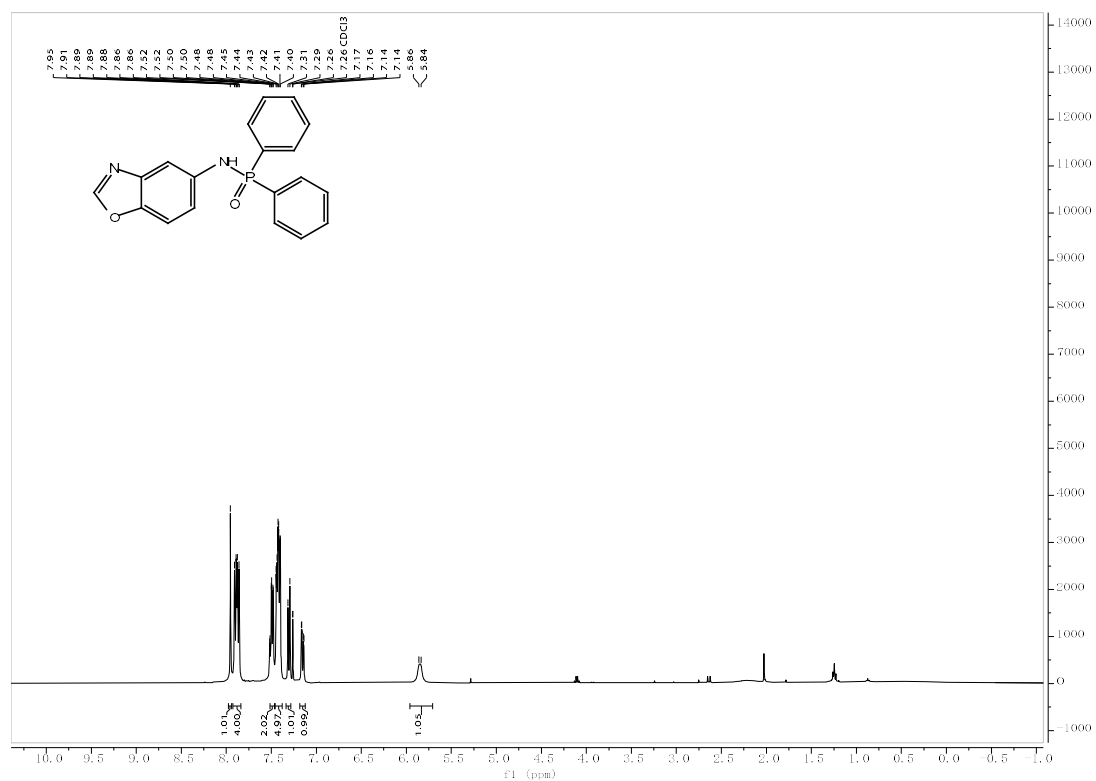
¹H NMR of 28 (400 MHz, CDCl₃)



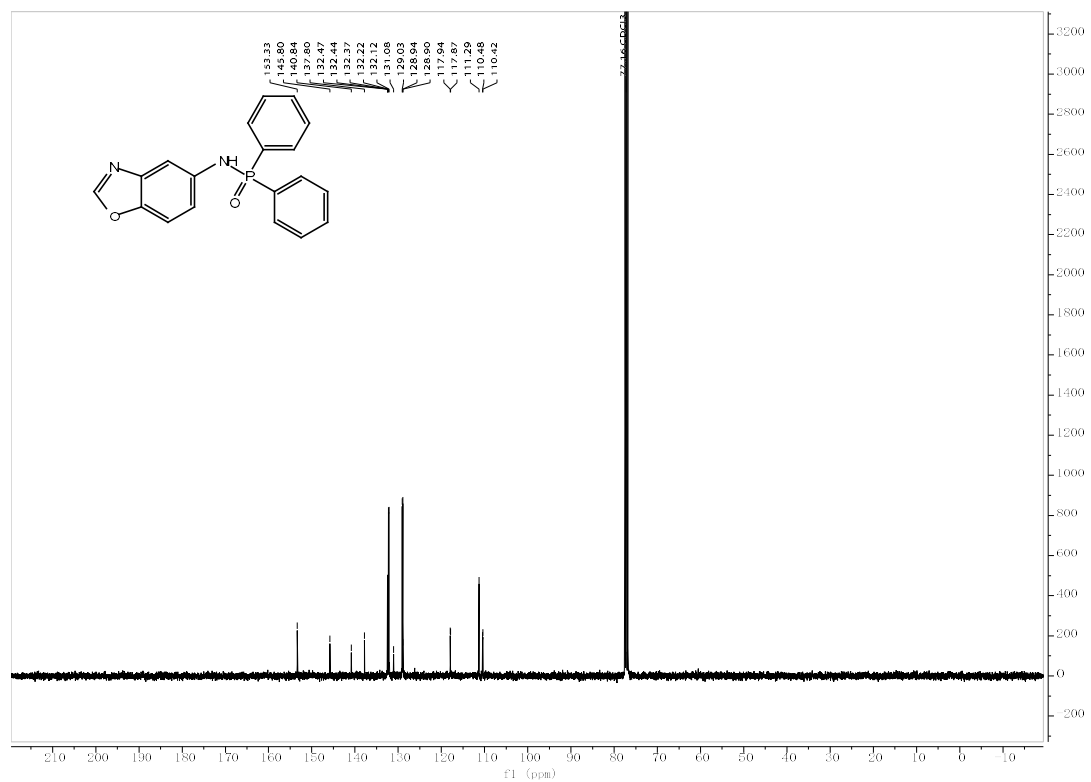
¹³C NMR of 28 (400 MHz, CDCl₃)



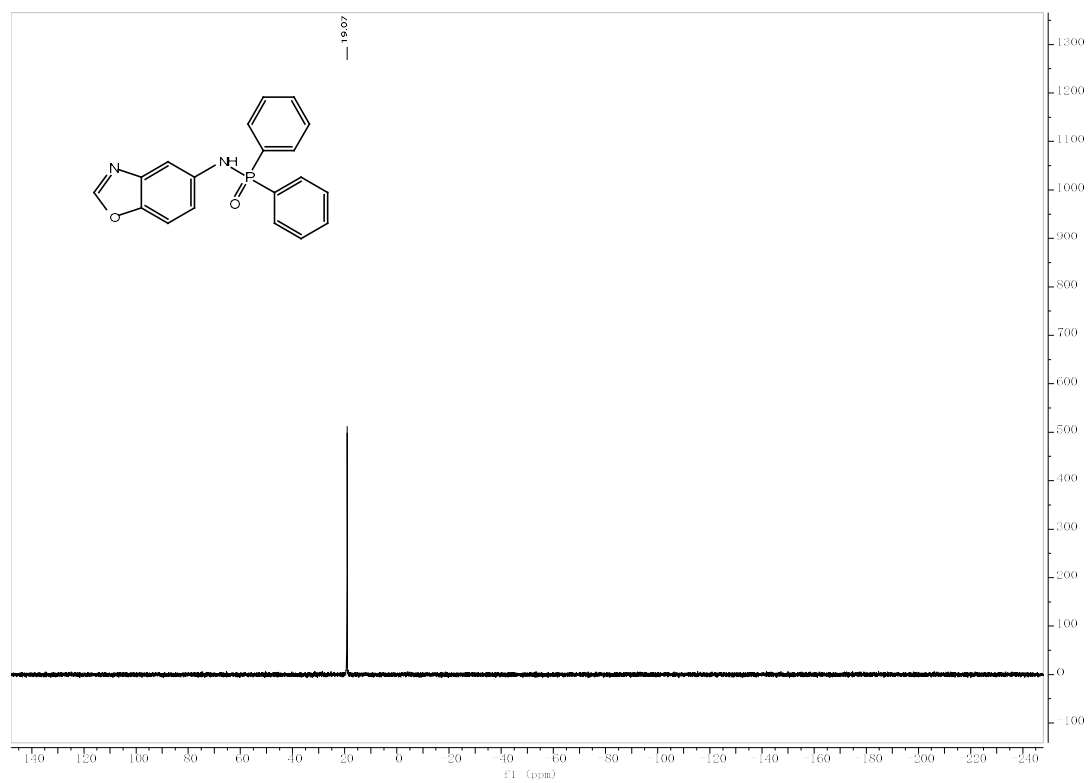
³¹P NMR of 28 (162 MHz, CDCl₃)



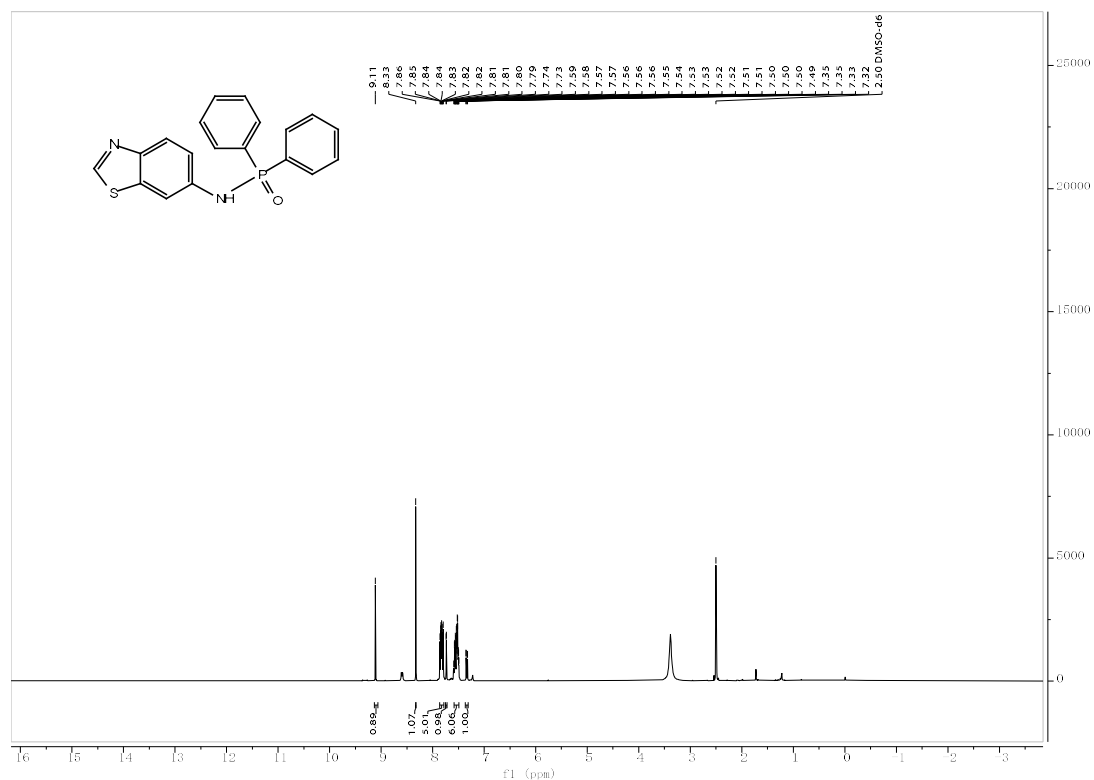
¹H NMR of 29 (400 MHz, CDCl₃)



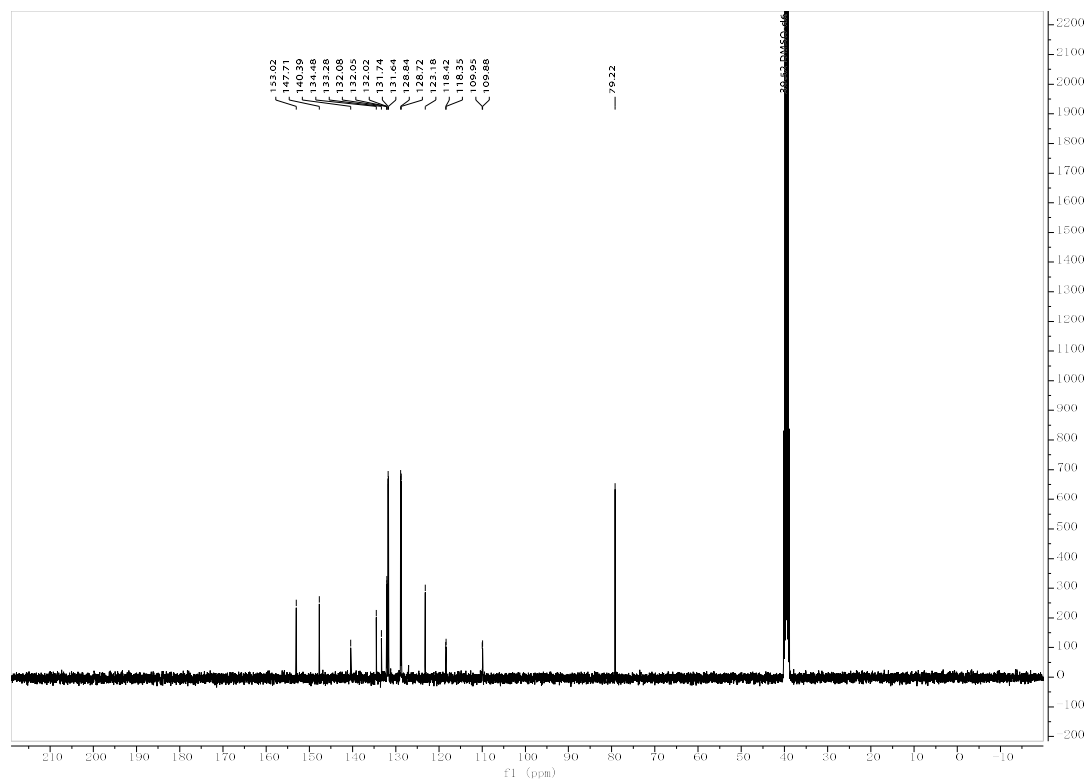
¹³C NMR of **29** (100 MHz, CDCl₃)



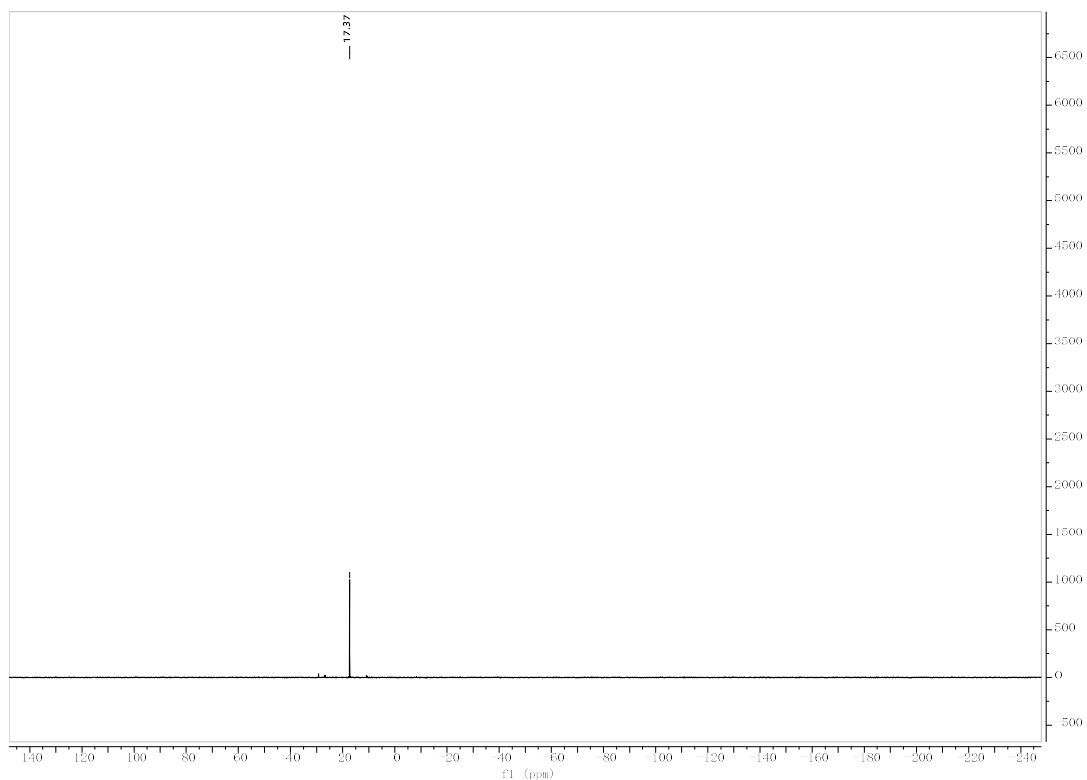
³¹P NMR of **29** (162 MHz, CDCl₃)



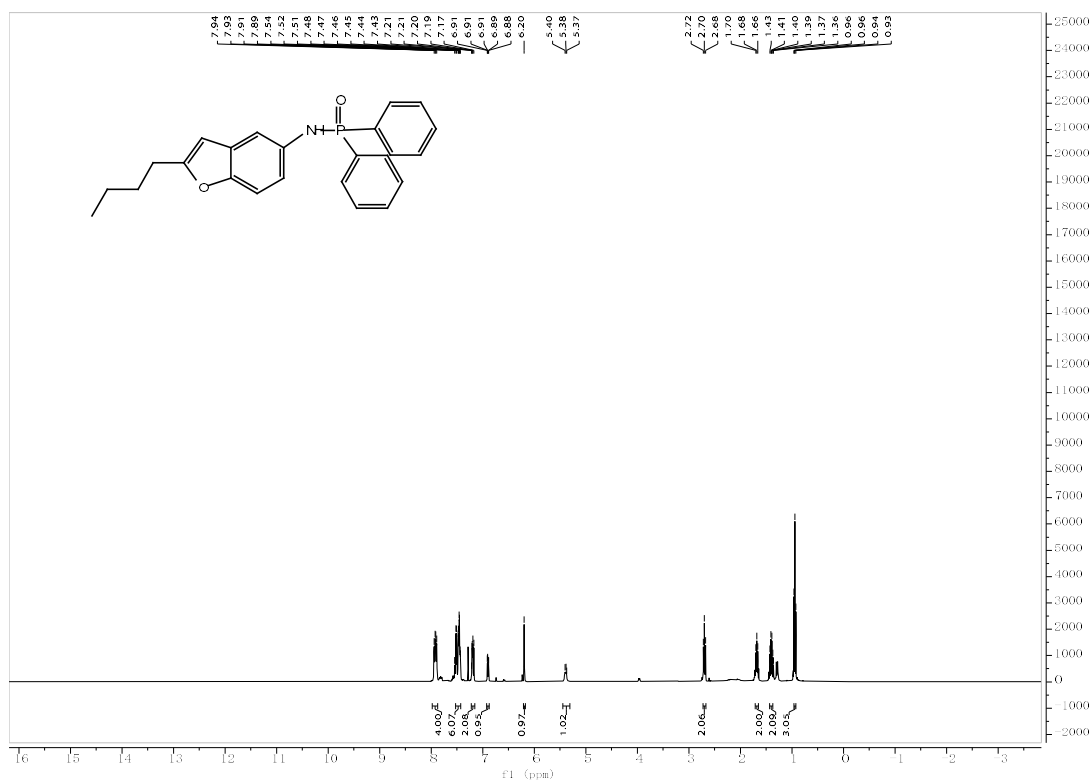
¹H NMR of **30** (400 MHz, DMSO-d₆)



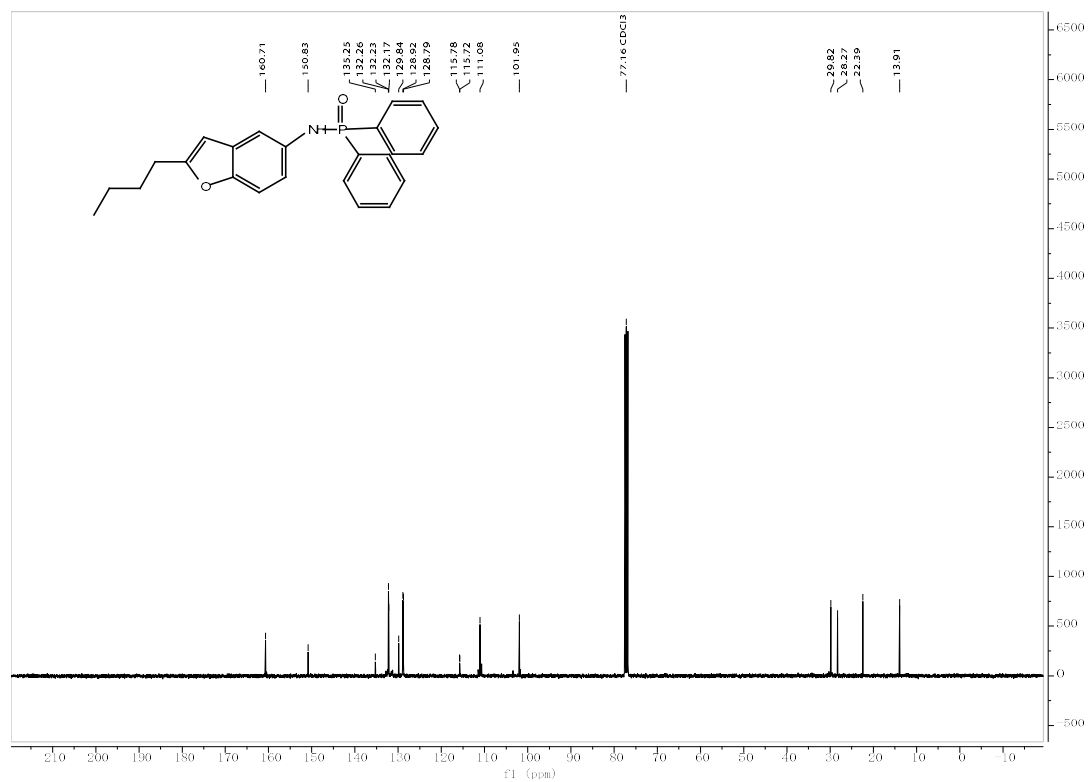
¹³C NMR of **30** (100 MHz, DMSO-d₆)



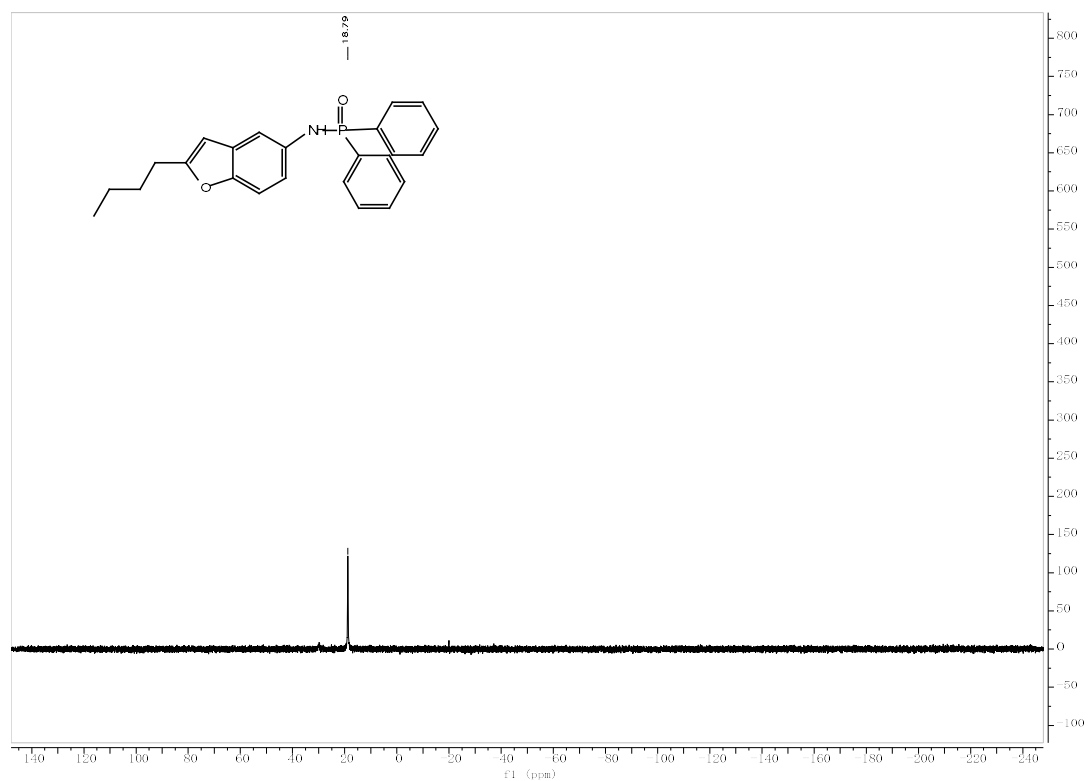
³¹P NMR of **30** (162 MHz, DMSO-d₆)



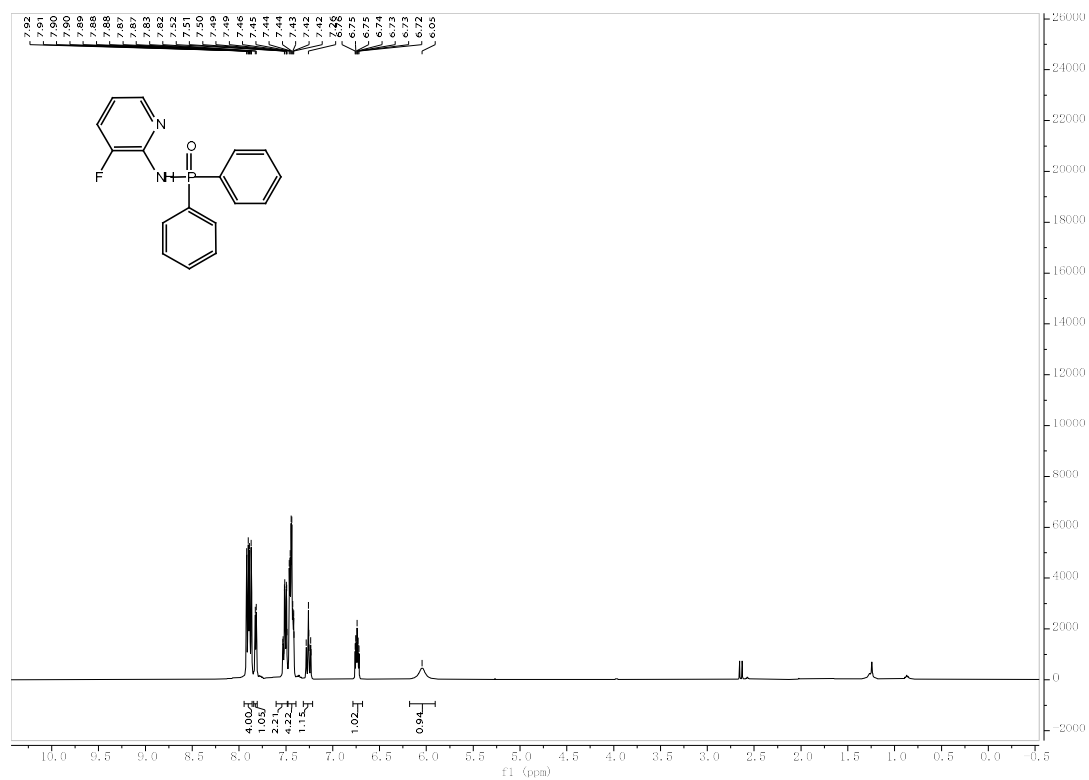
¹H NMR of **31** (400 MHz, DMSO-d₆)



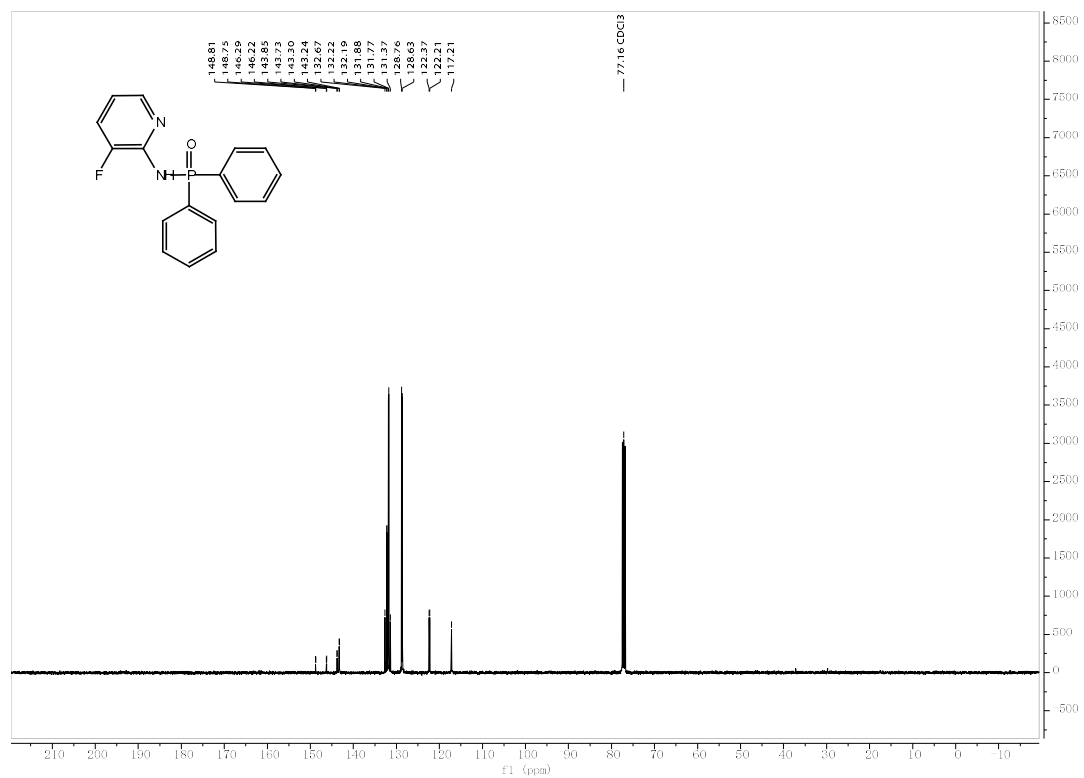
^{13}C NMR of **31** (100 MHz, DMSO- d_6)



^{31}P NMR of **31** (162 MHz, DMSO- d_6)



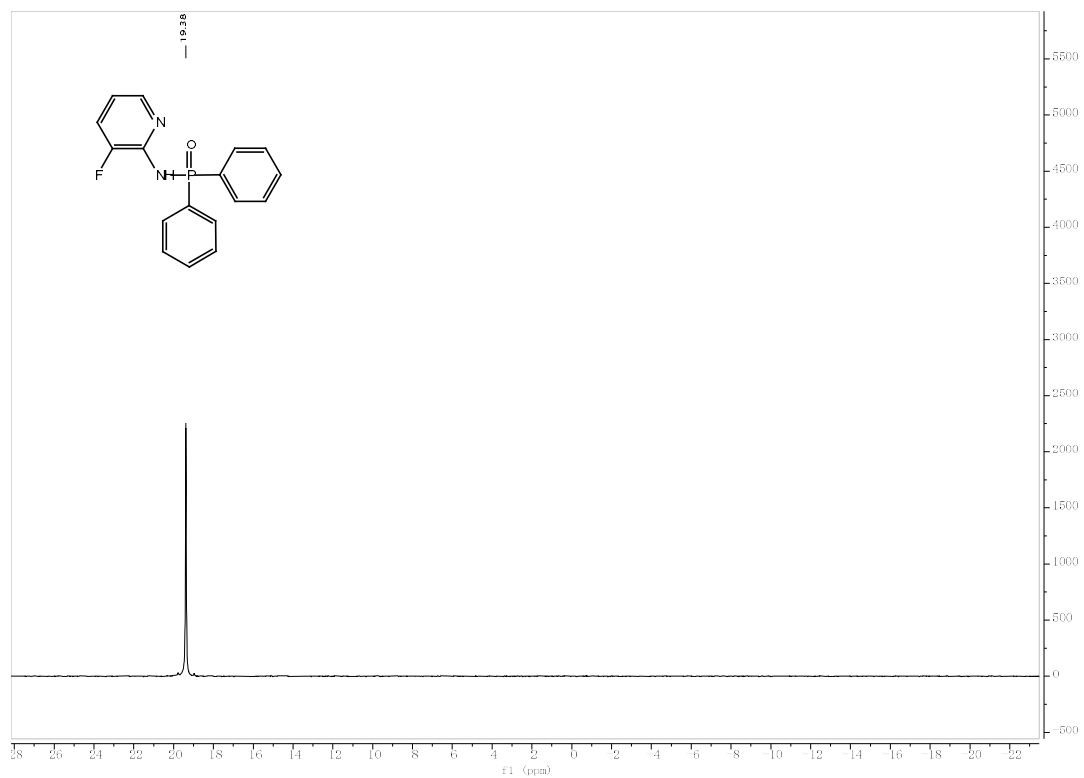
¹H NMR of 32 (400 MHz, CDCl₃)



¹³C NMR of 32 (100 MHz, CDCl₃)



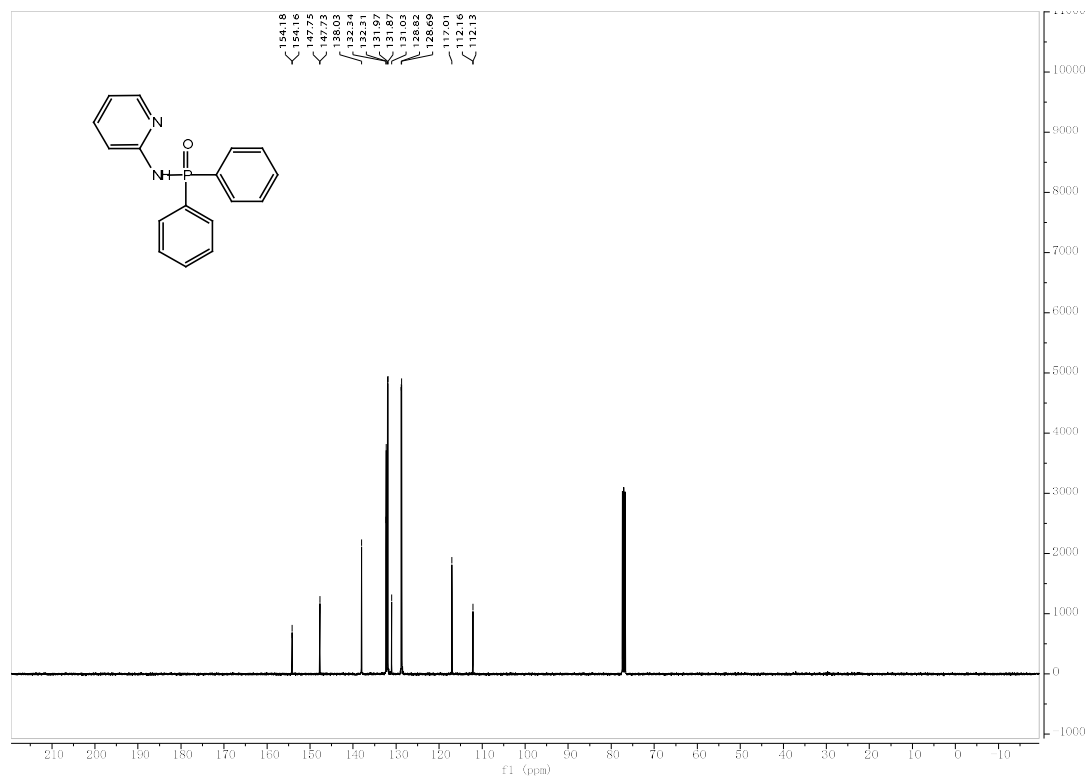
^{31}P NMR of **32** (162 MHz, CDCl_3)



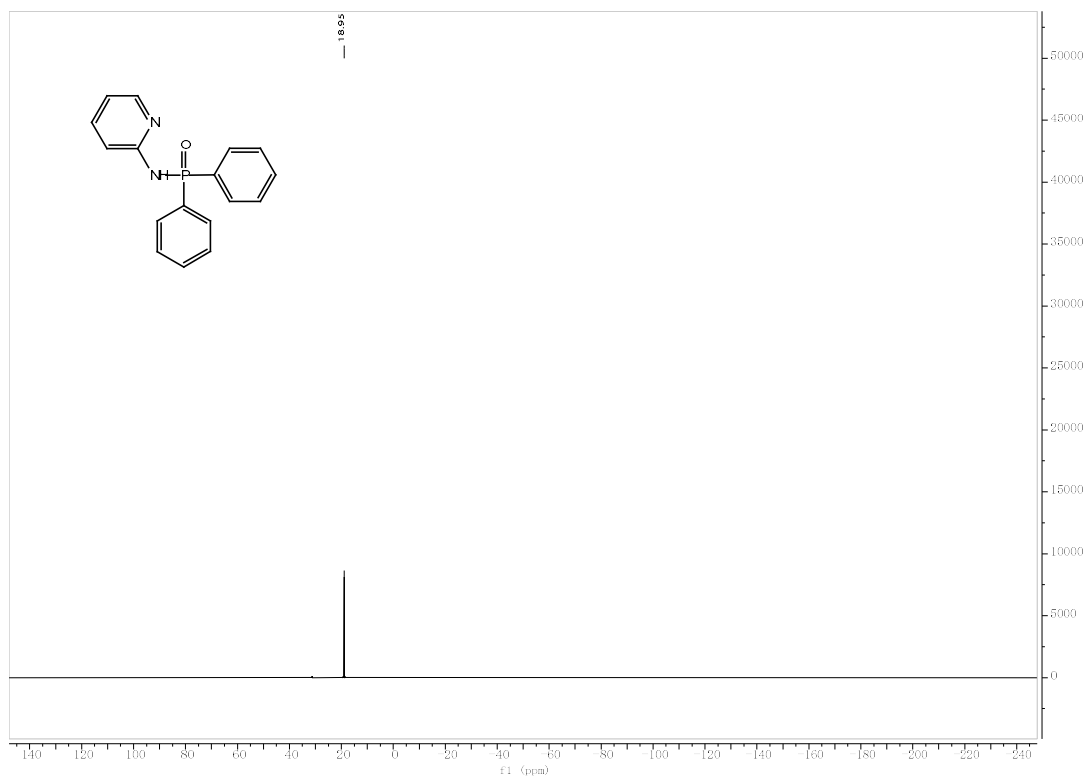
^{19}F NMR of **32** (376 MHz, CDCl_3)



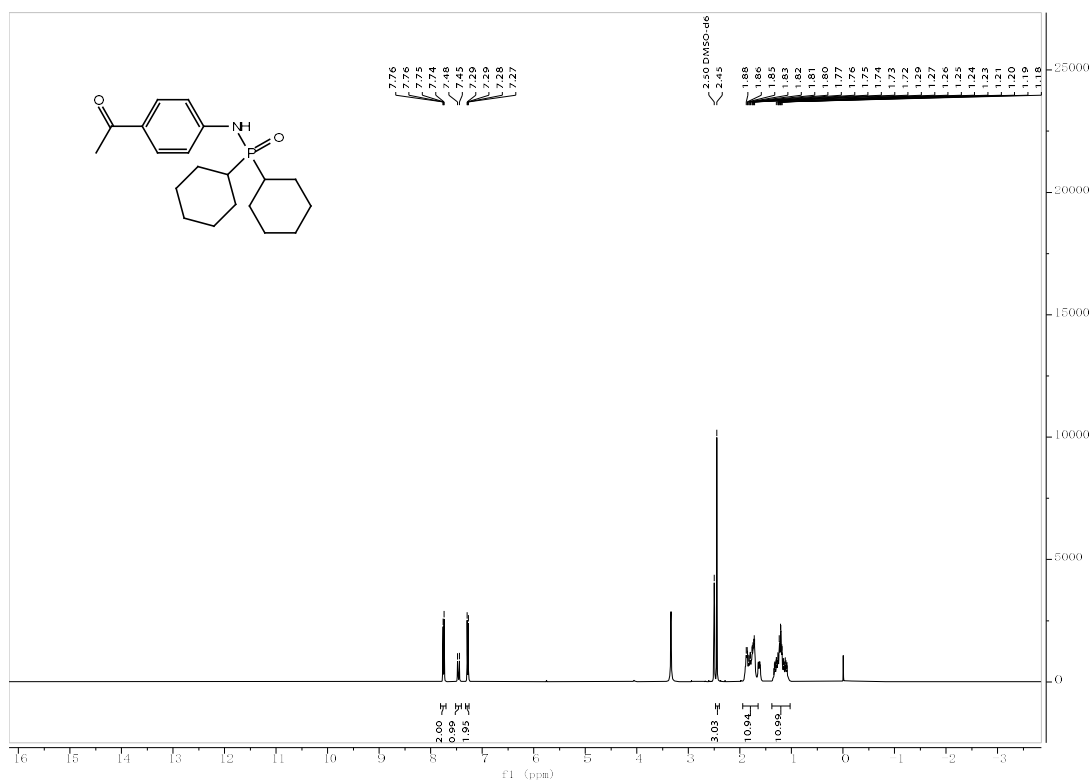
¹H NMR of 33 (400 MHz, CDCl₃)



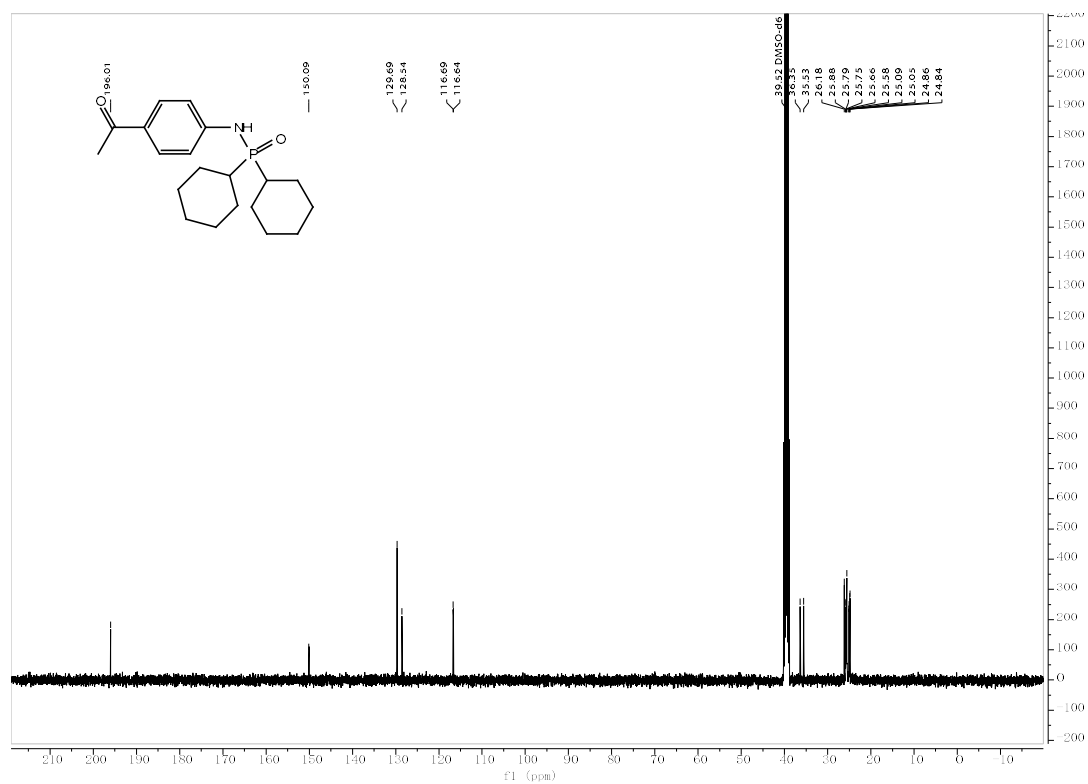
¹³C NMR of 33 (100 MHz, CDCl₃)



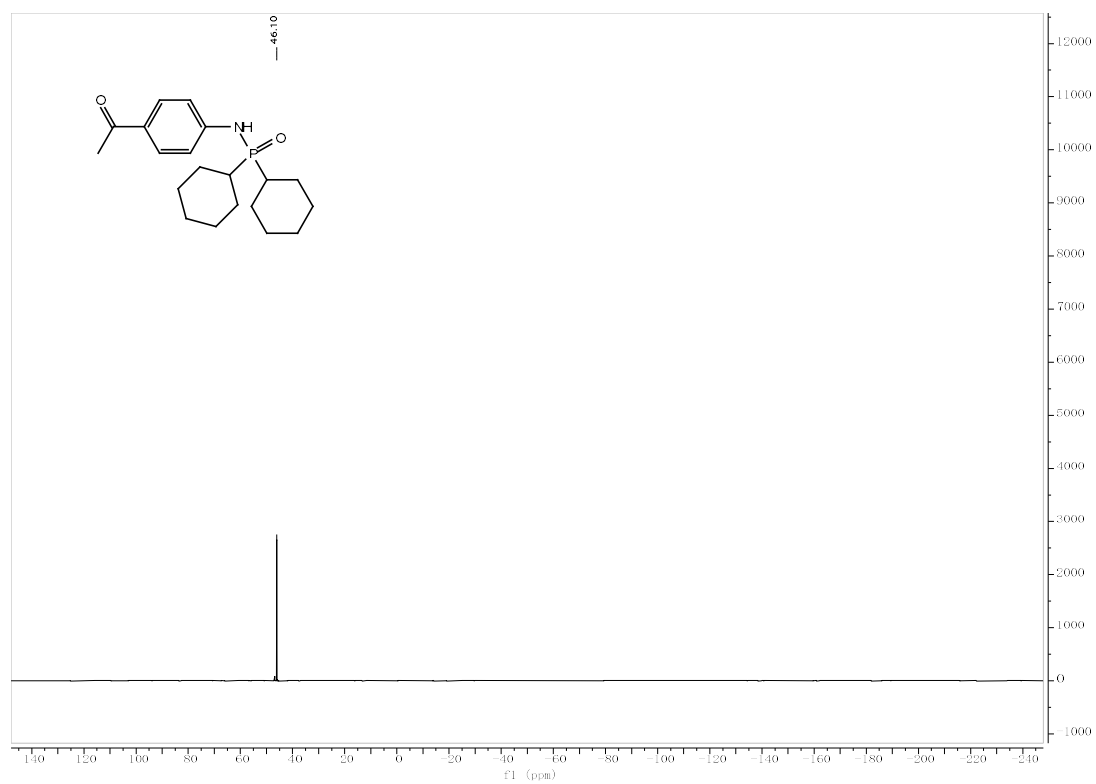
^{31}P NMR of 33 (162 MHz, CDCl_3)



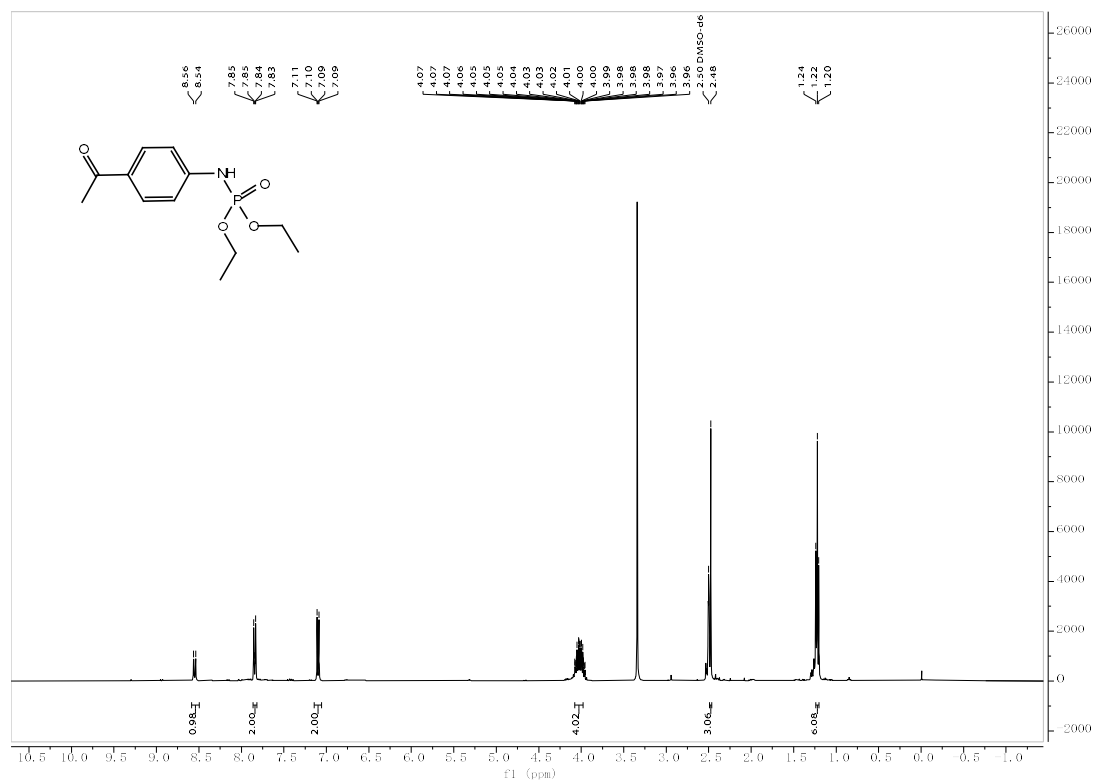
^1H NMR of 34 (400 MHz, DMSO-d_6)



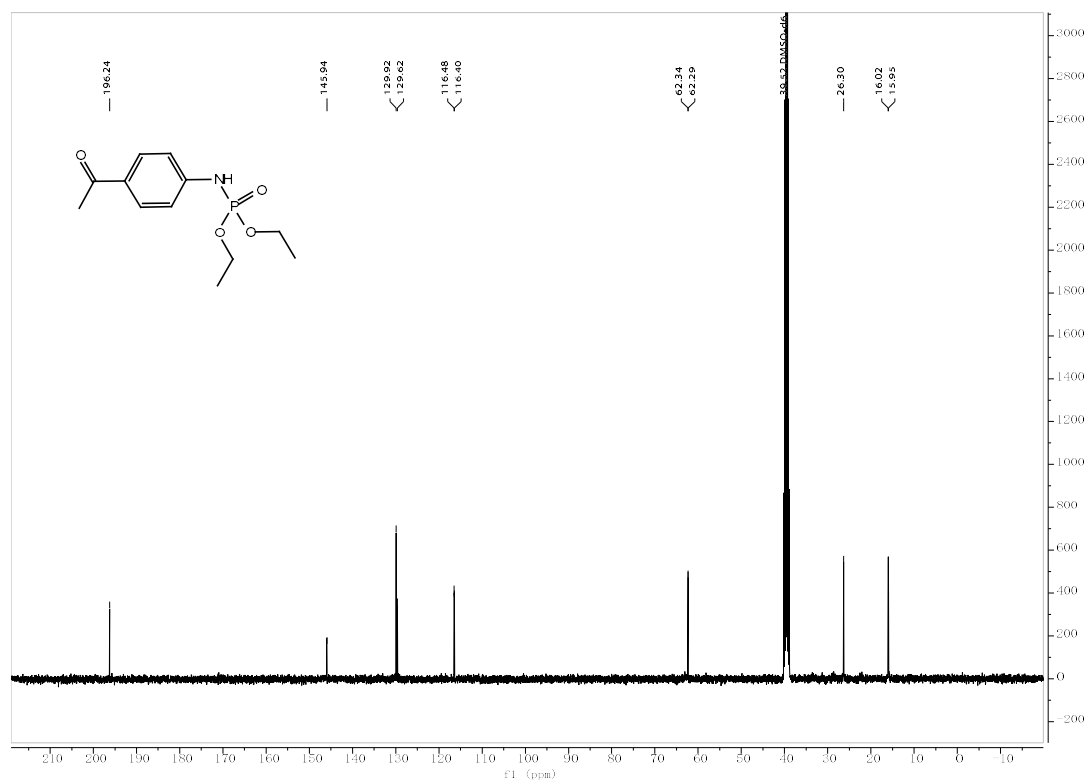
¹³C NMR of **34** (100 MHz, DMSO-d₆)



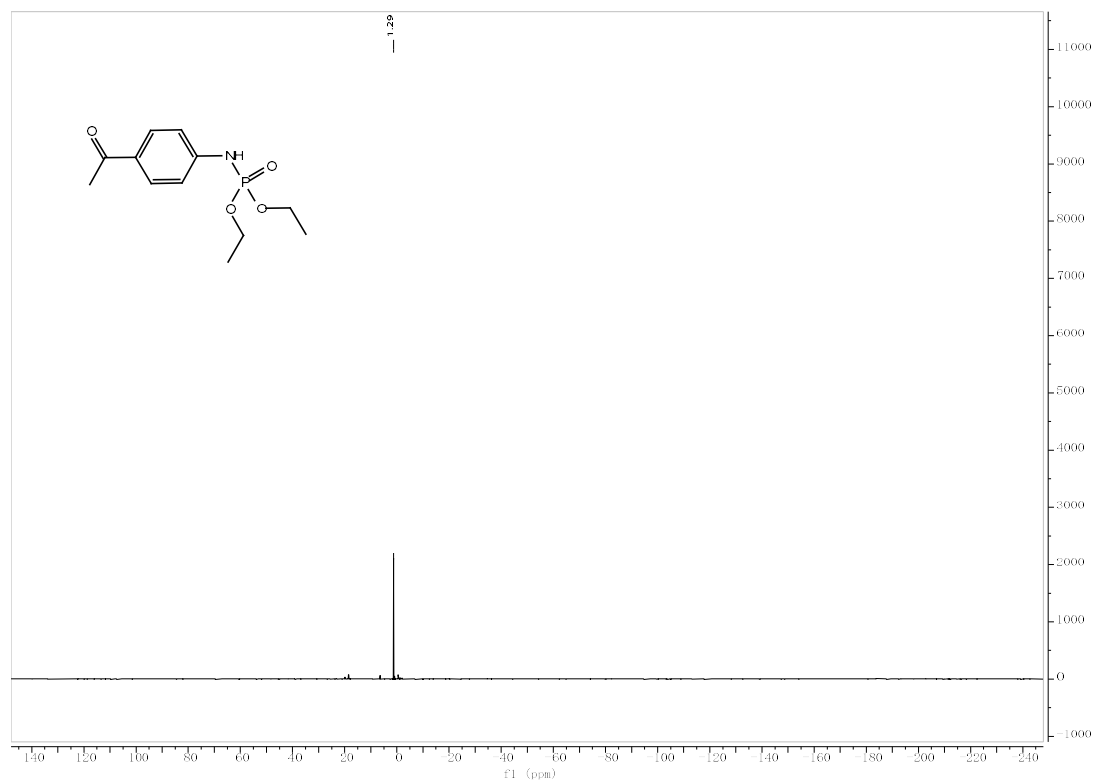
³¹P NMR of **34** (162 MHz, DMSO-d₆)



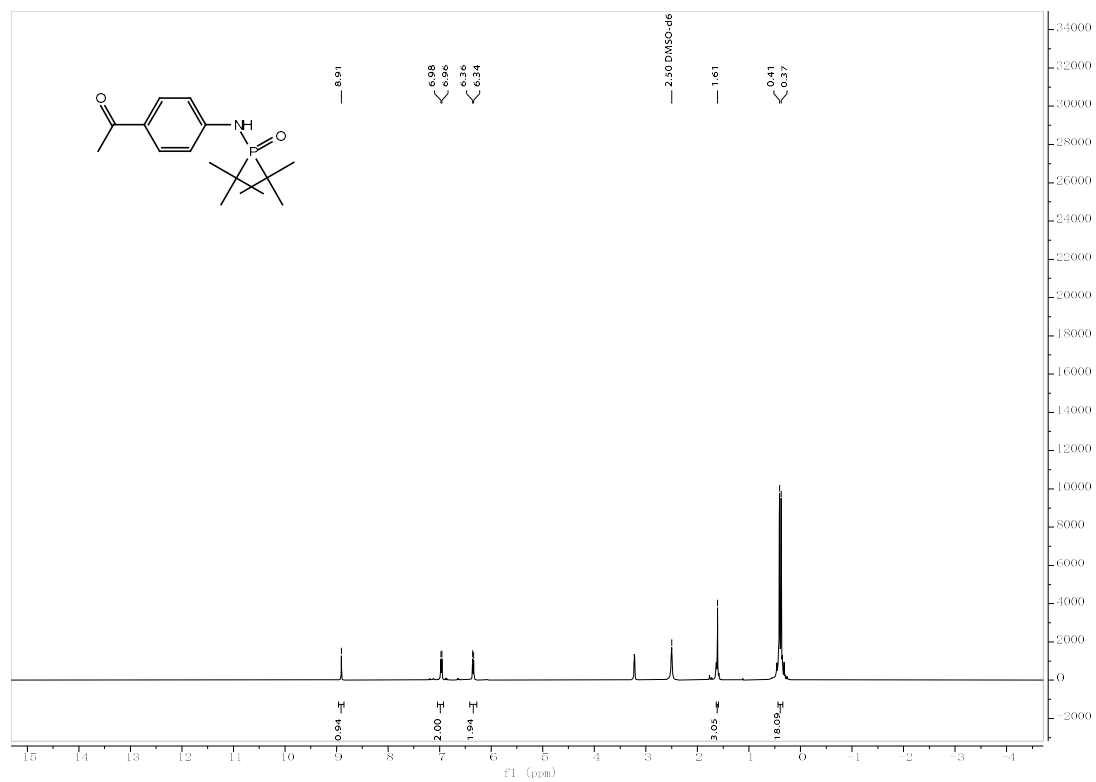
¹H NMR of 35 (400 MHz, DMSO-d₆)



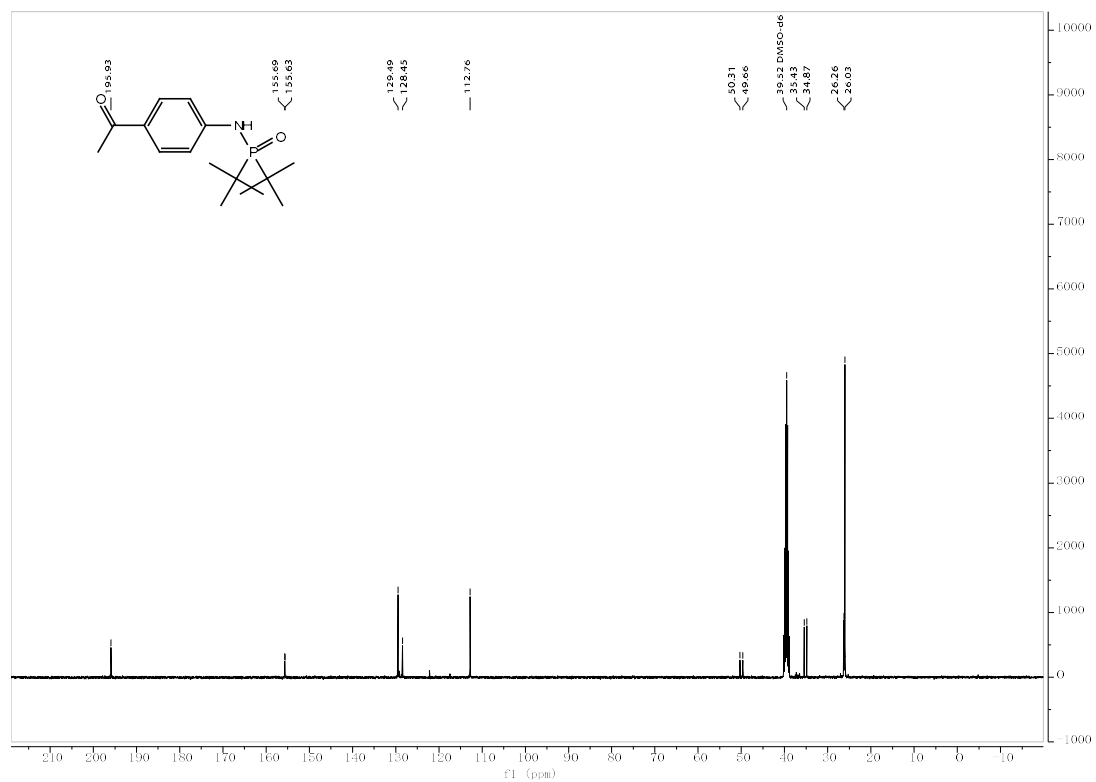
¹³C NMR of 35 (100 MHz, DMSO-d₆)



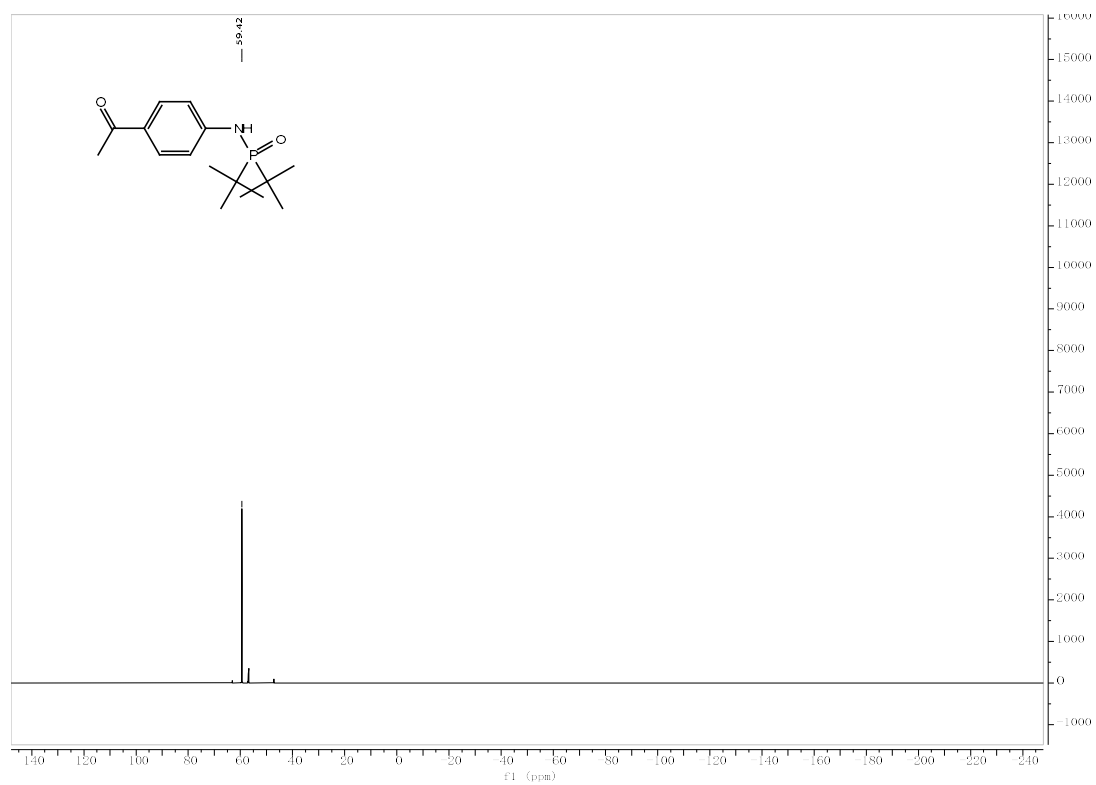
^{31}P NMR of 35 (162 MHz, DMSO- d_6)



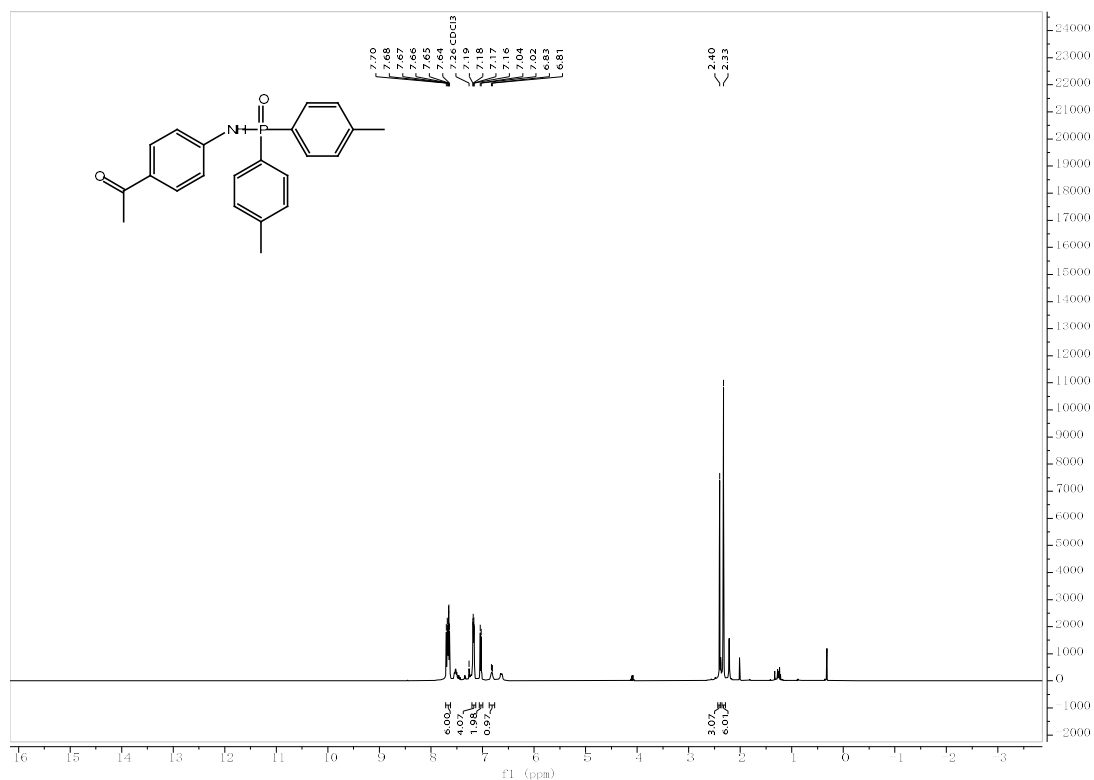
^1H NMR of 36 (400 MHz, DMSO- d_6)



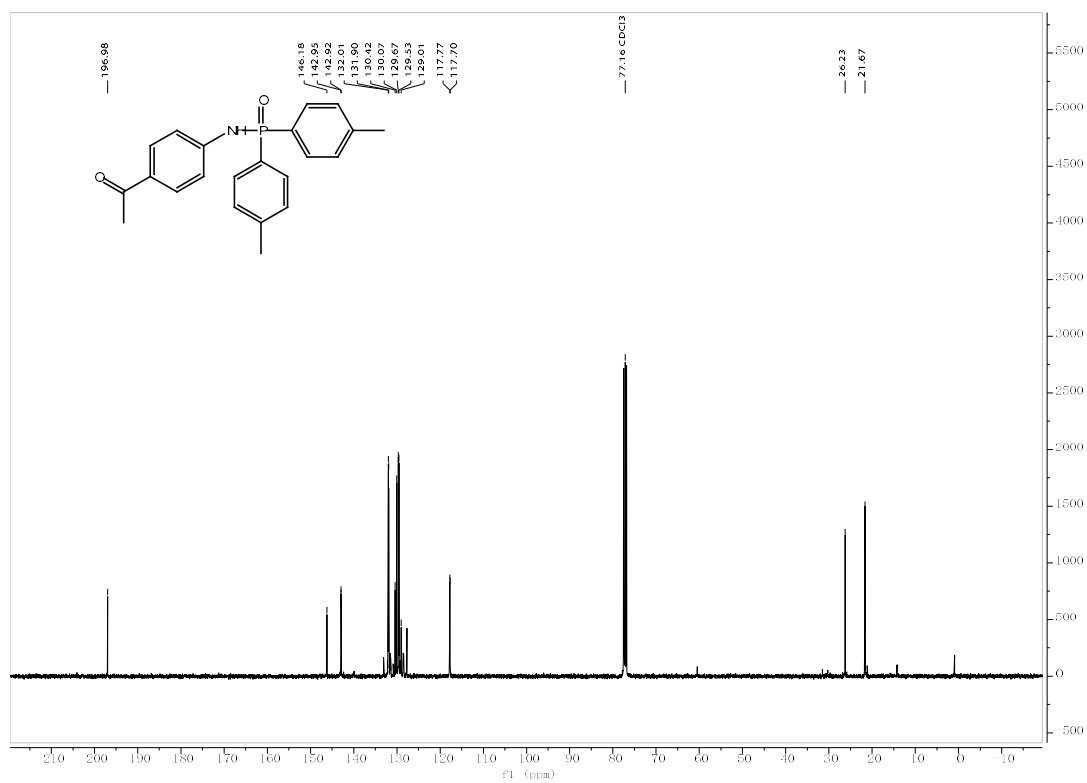
^{13}C NMR of **36** (100 MHz, DMSO- d_6)



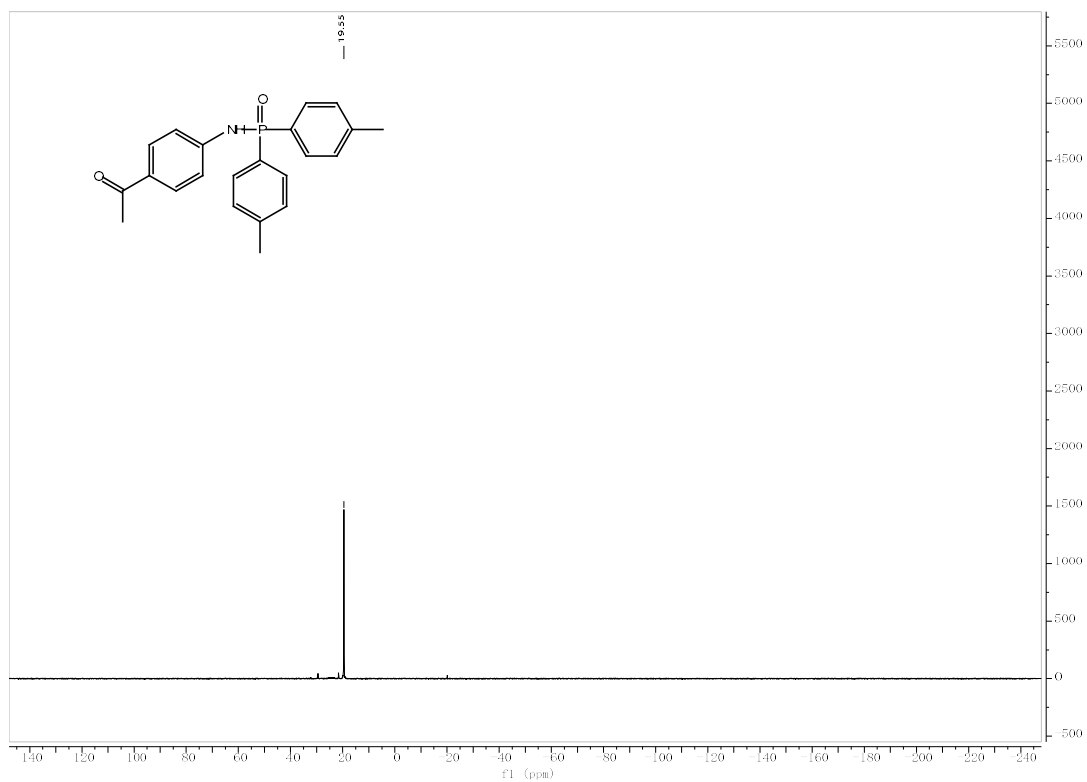
^{31}P NMR of **36** (162 MHz, DMSO- d_6)



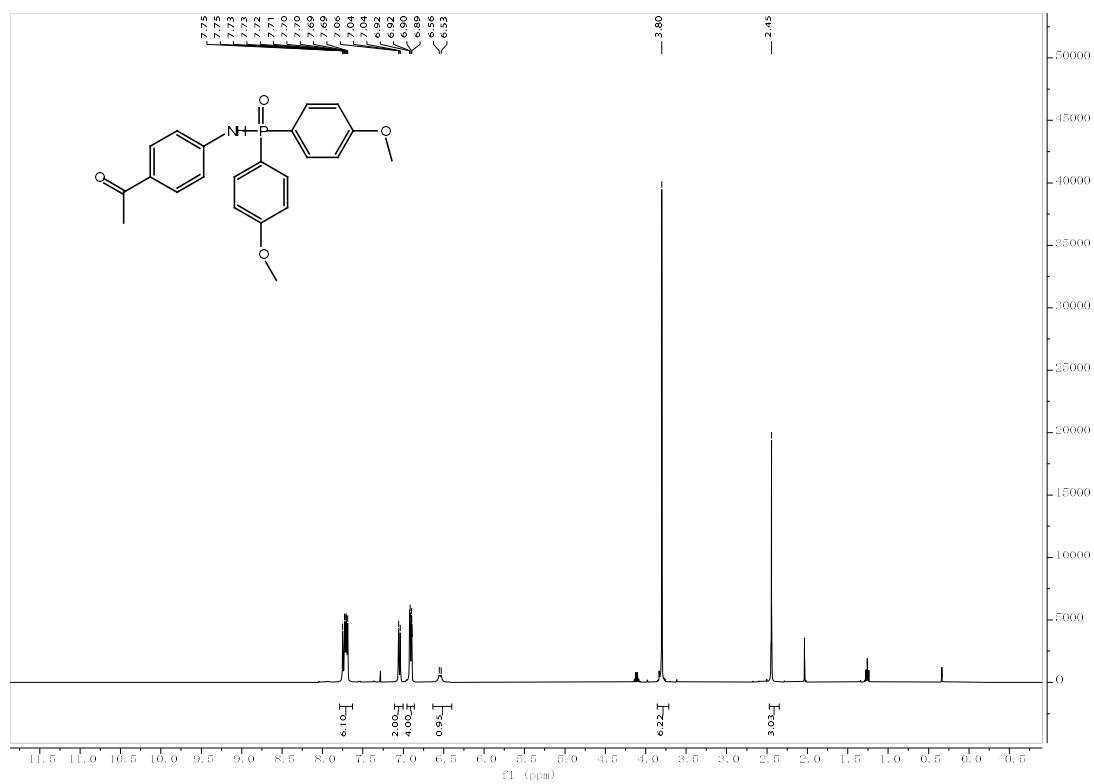
¹H NMR of 37 (400 MHz, CDCl₃)



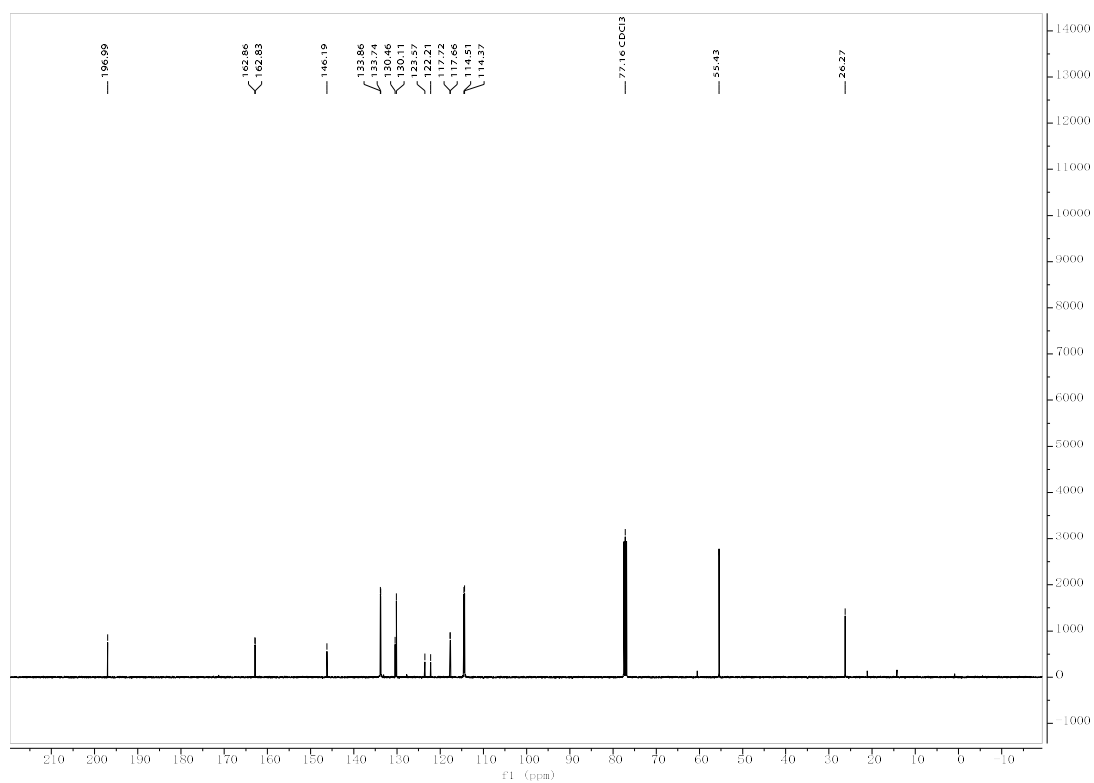
¹³C NMR of 37 (100 MHz, CDCl₃)



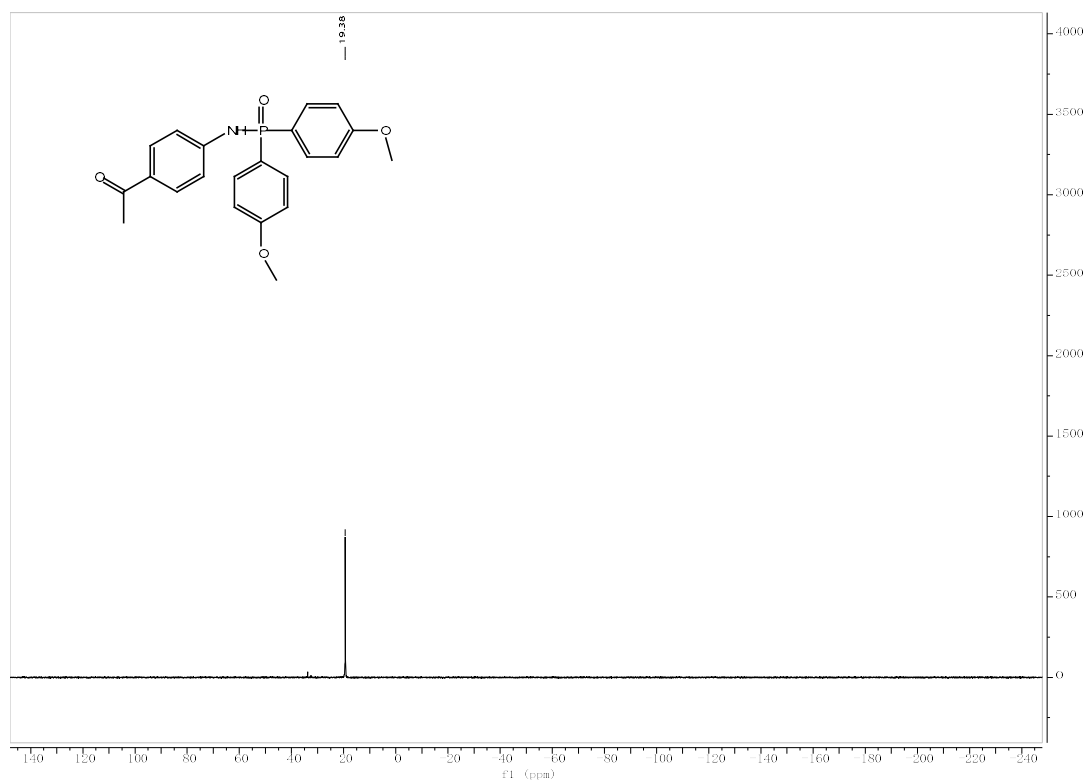
^{31}P NMR of 37 (162 MHz, CDCl_3)



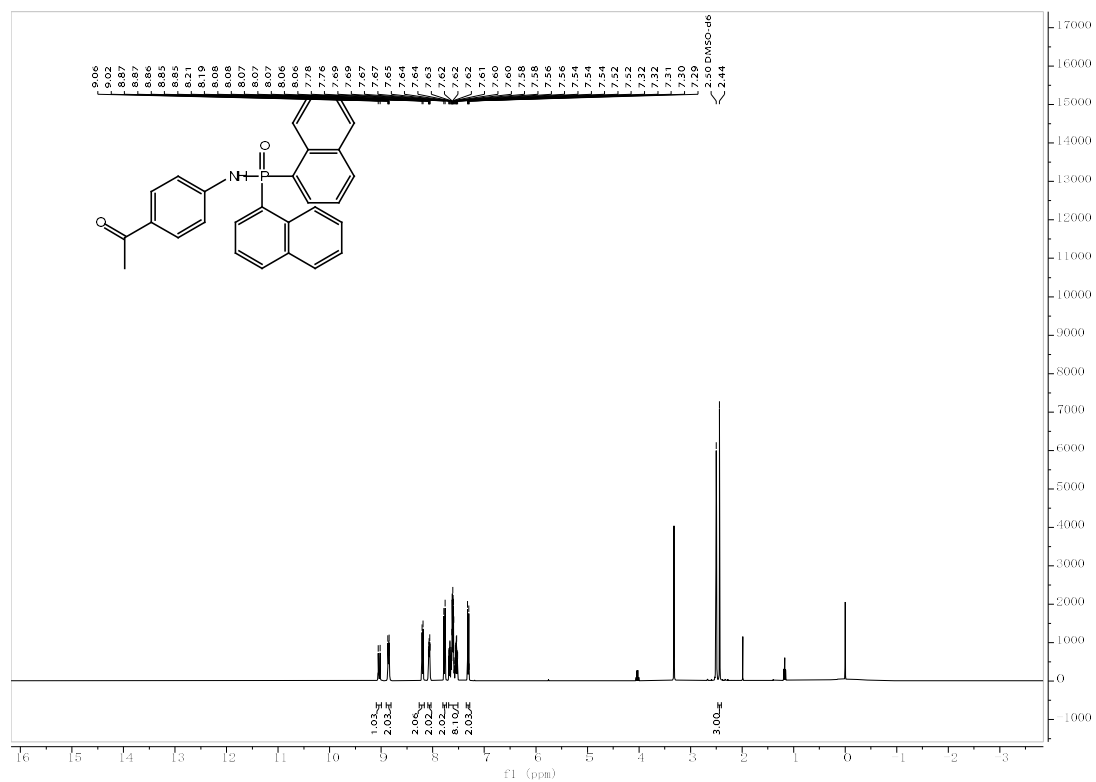
^1H NMR of 38 (400 MHz, CDCl_3)



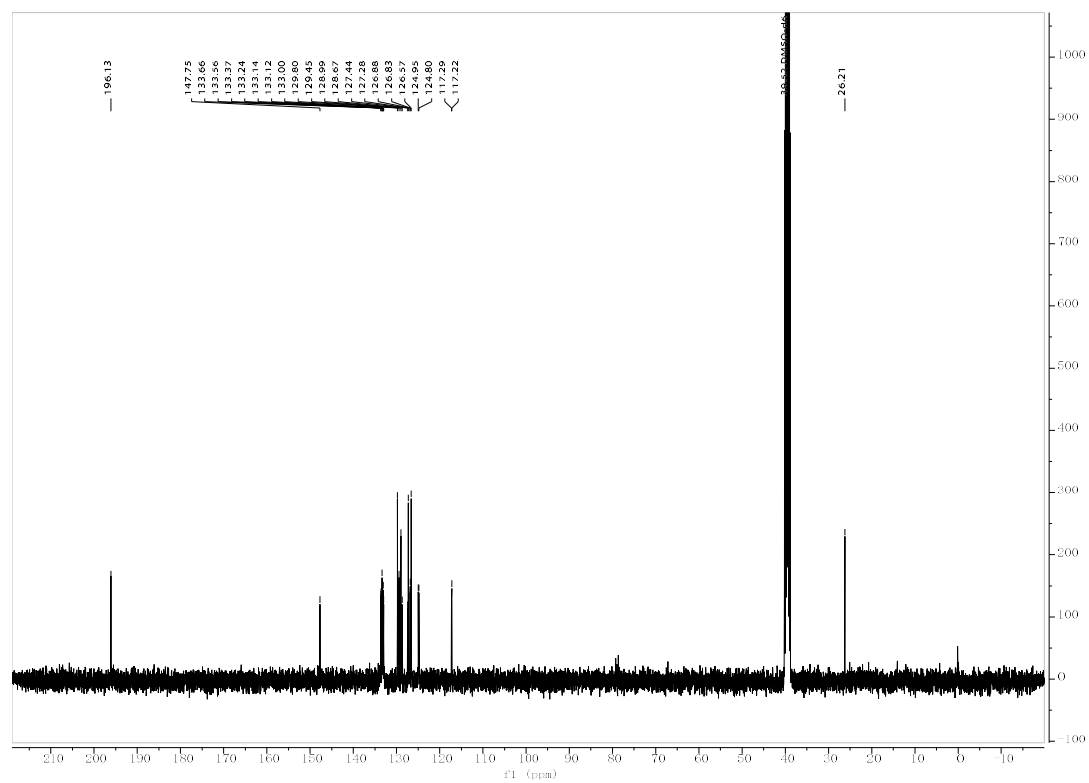
¹³C NMR of **38** (100 MHz, CDCl₃)



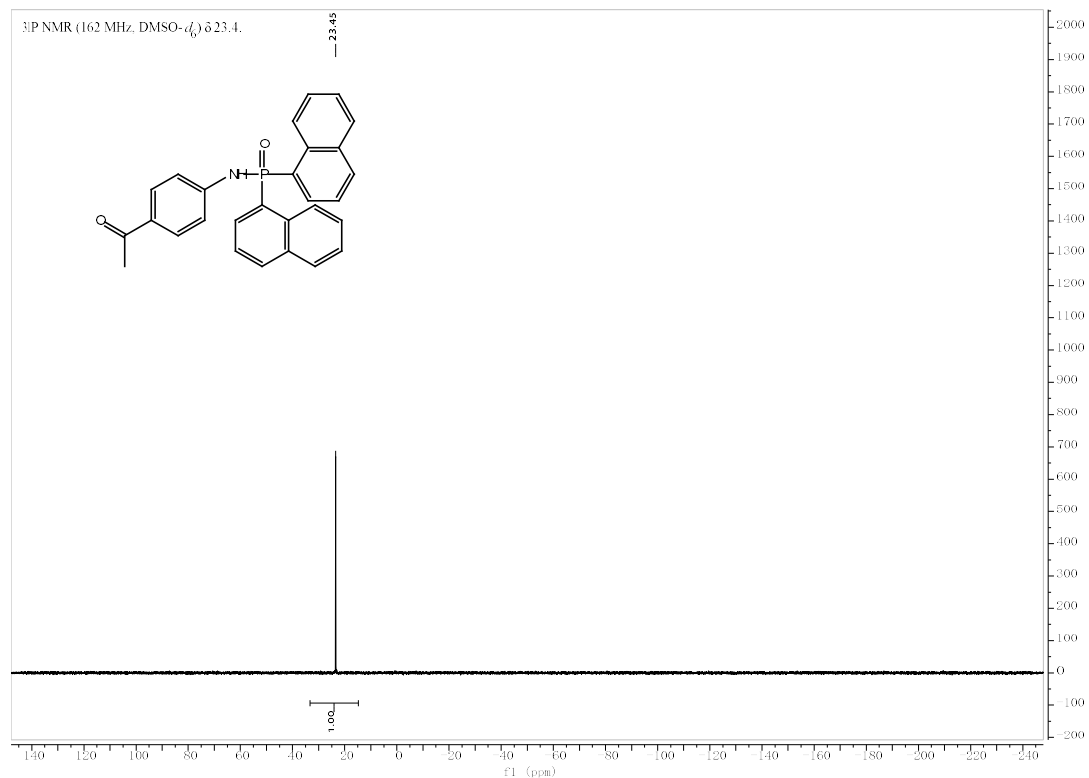
³¹P NMR of **38** (162 MHz, CDCl₃)



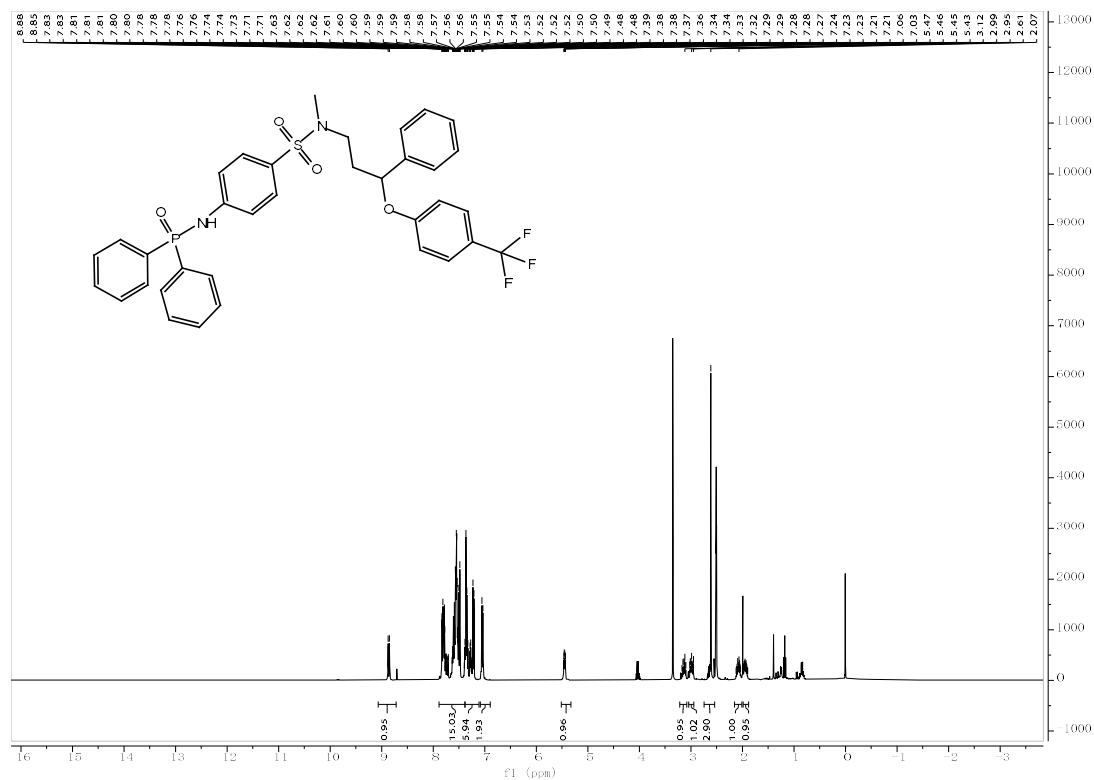
¹H NMR of 39 (400 MHz, DMSO-d₆)



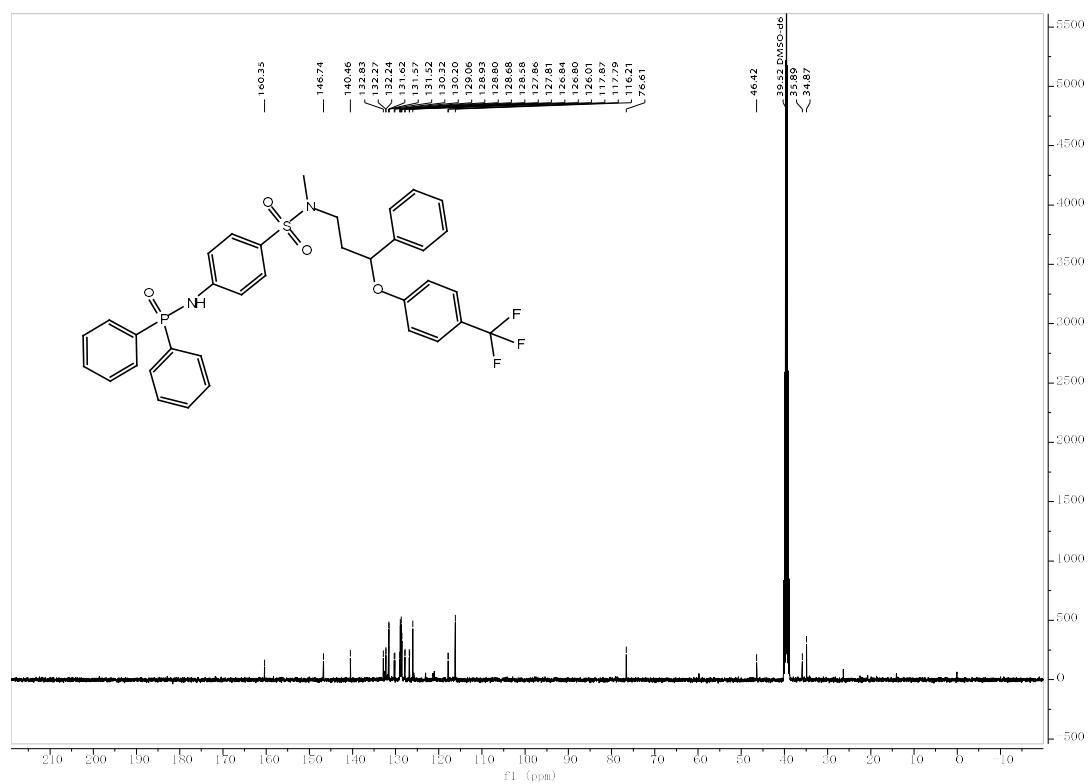
¹³C NMR of 39 (100 MHz, DMSO-d₆)



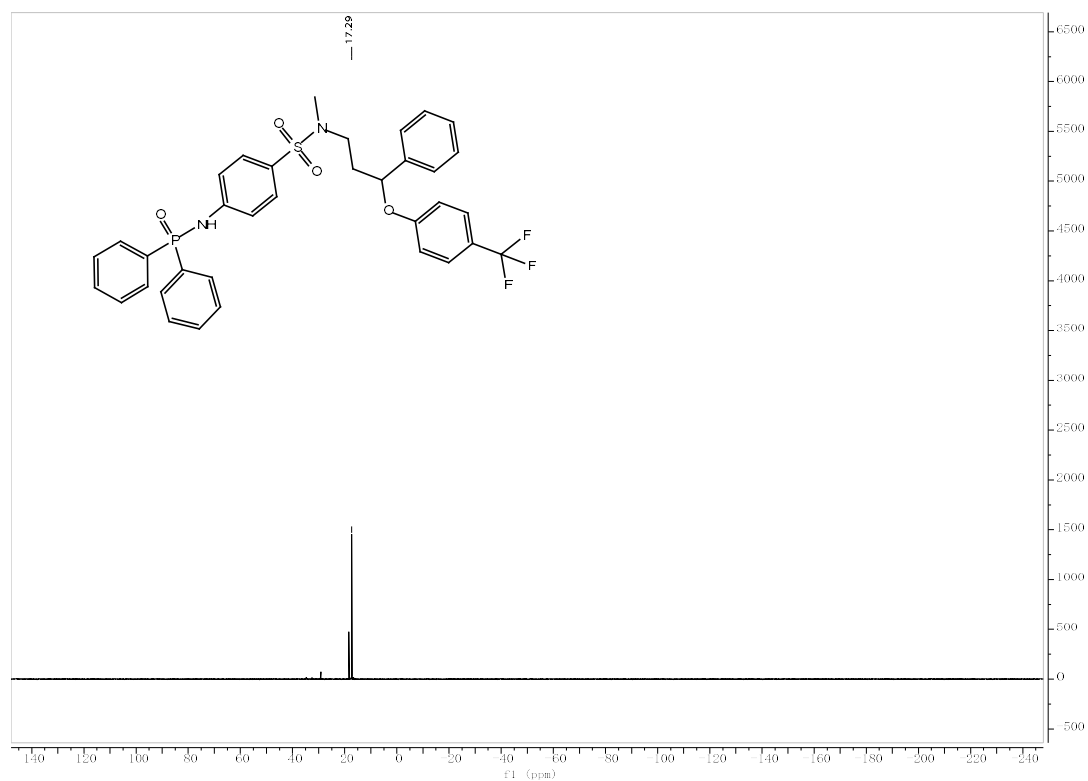
^{31}P NMR of 39 (162 MHz, $\text{DMSO}-d_6$)



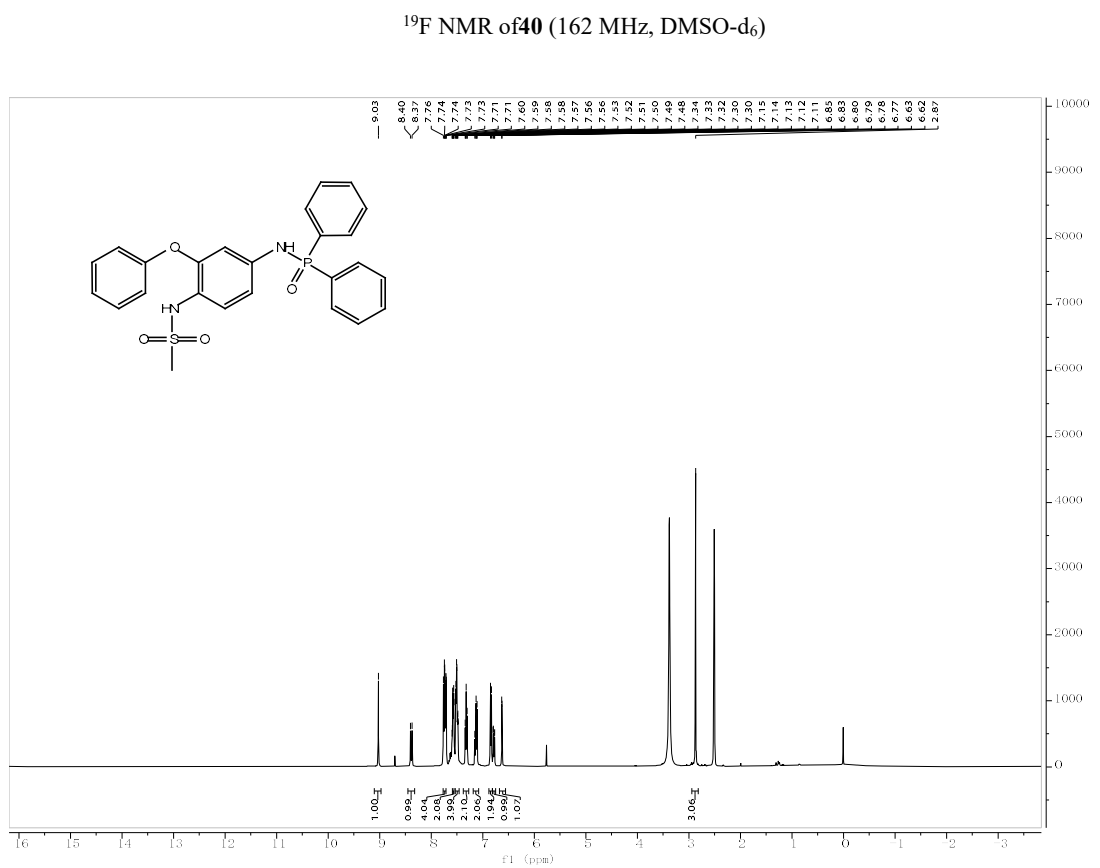
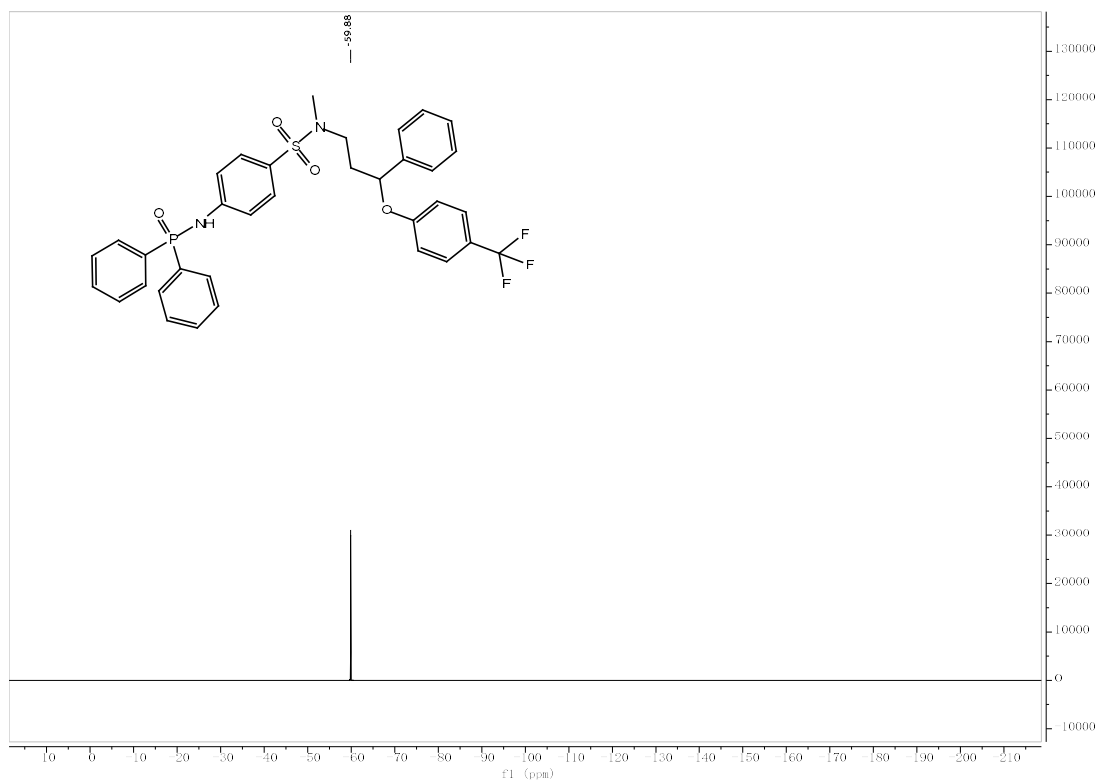
^1H NMR of 40 (400 MHz, $\text{DMSO}-d_6$)



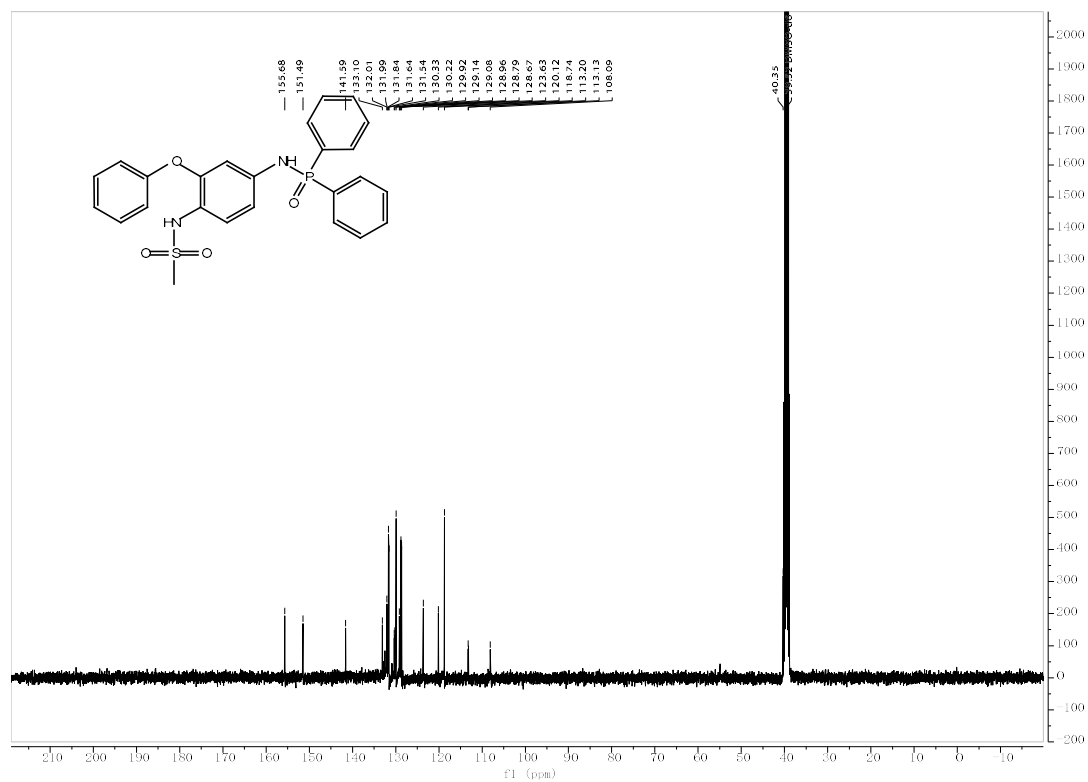
¹³C NMR of **40** (100 MHz, DMSO-d₆)



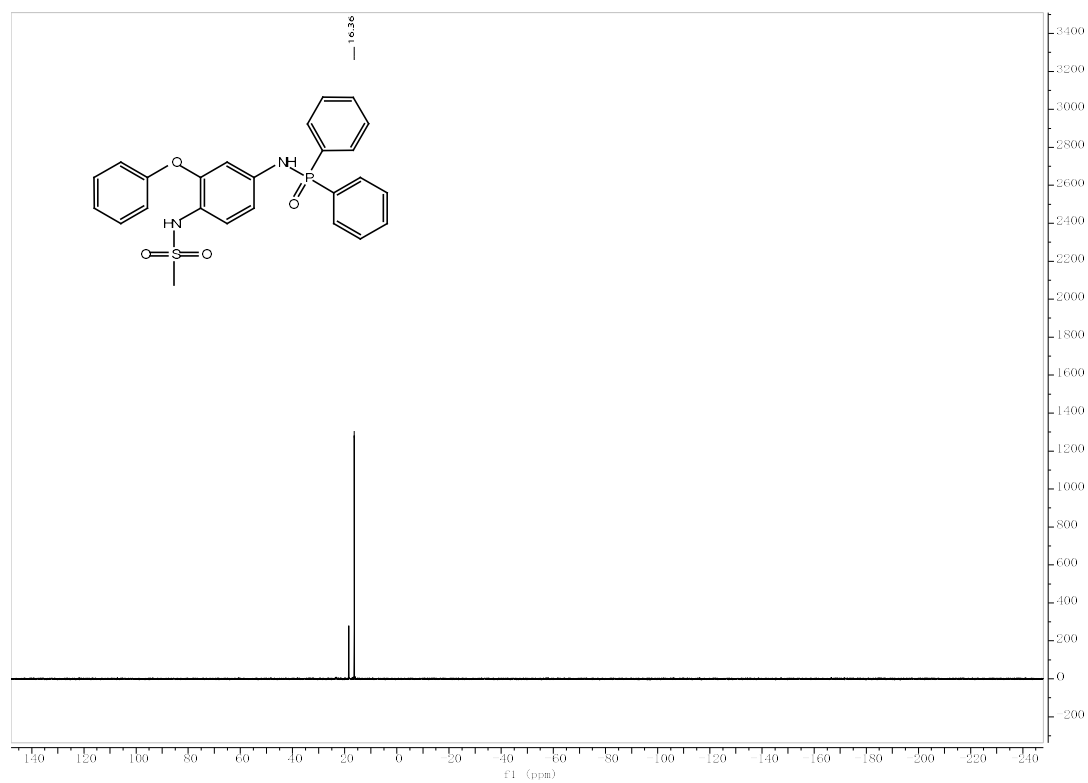
³¹P NMR of **40** (162 MHz, DMSO-d₆)



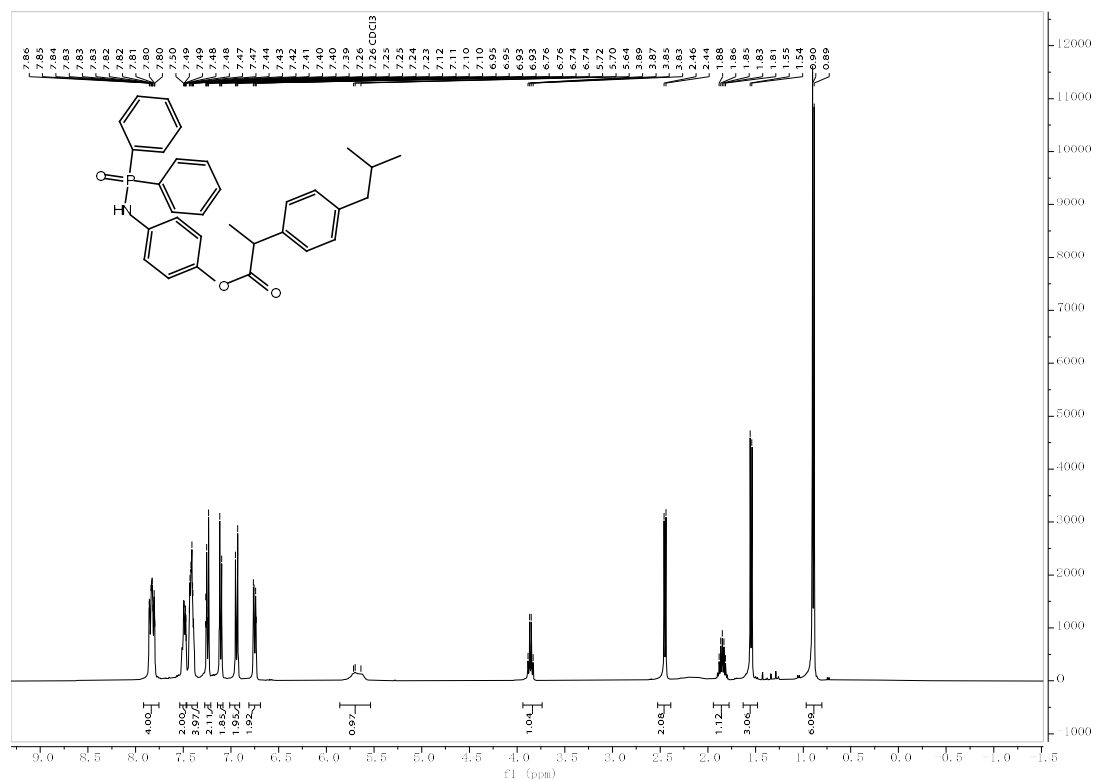
^1H NMR of 41 (400 MHz, DMSO- d_6)



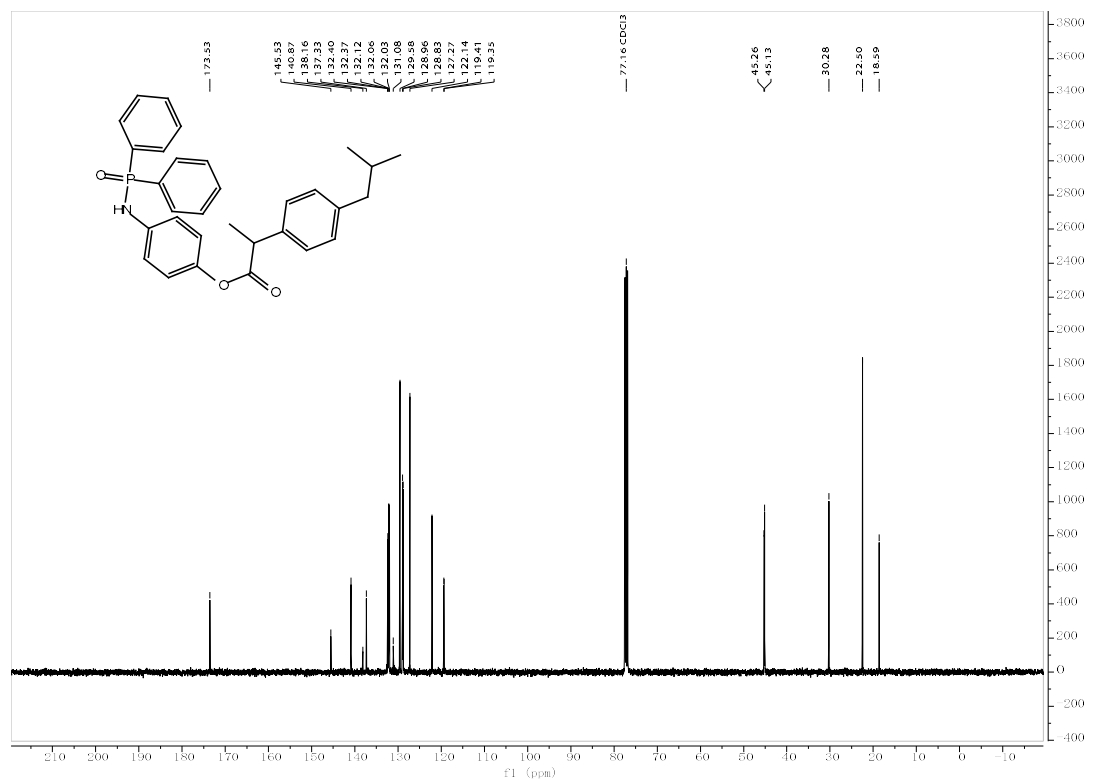
¹³C NMR of **41** (100 MHz, DMSO-d₆)



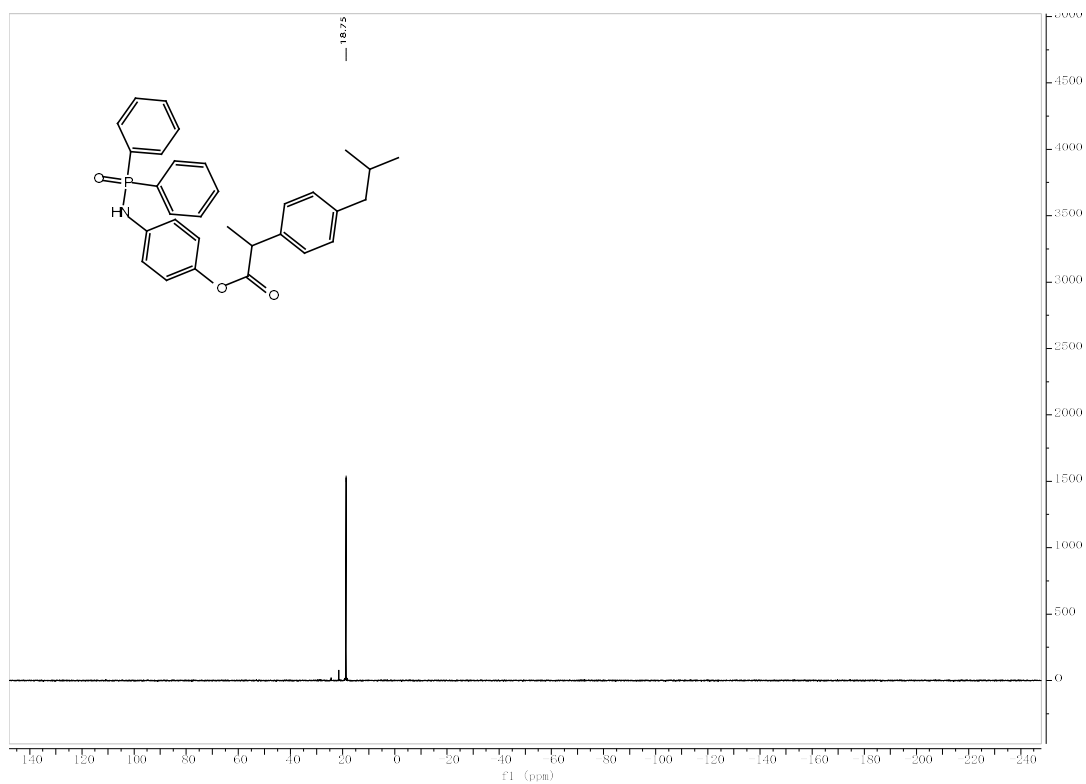
³¹P NMR of **41** (162 MHz, DMSO-d₆)



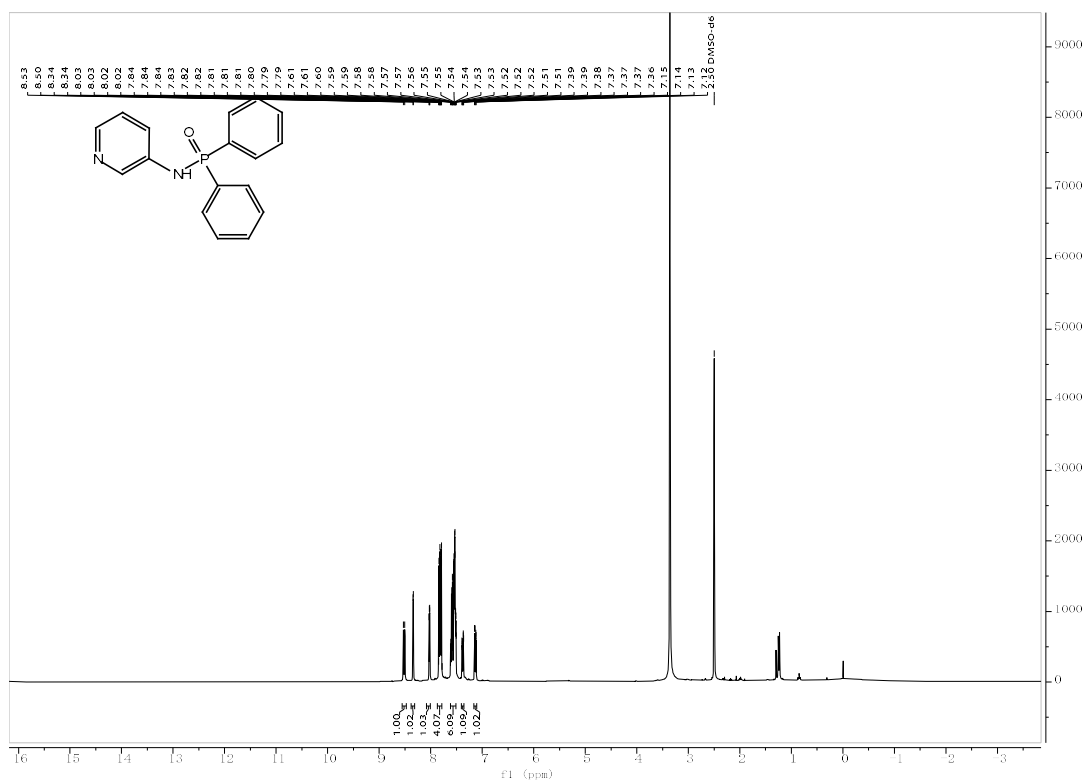
¹H NMR of 42 (400 MHz, CDCl₃)



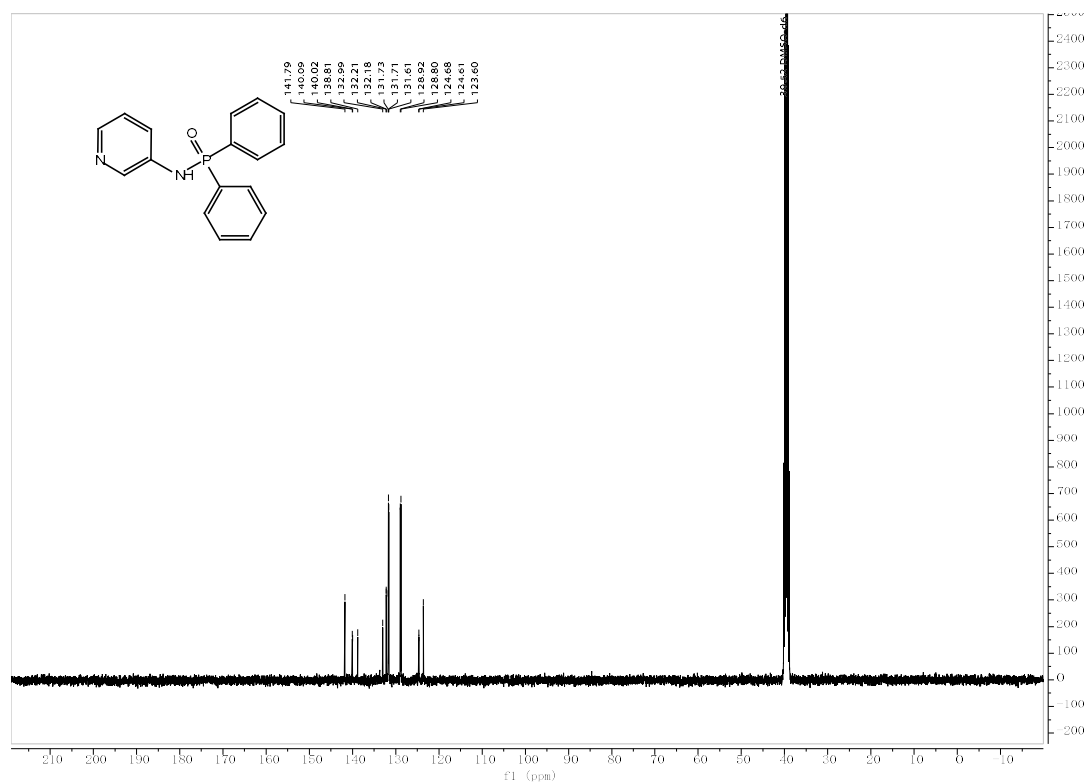
¹³C NMR of 42 (100 MHz, CDCl₃)



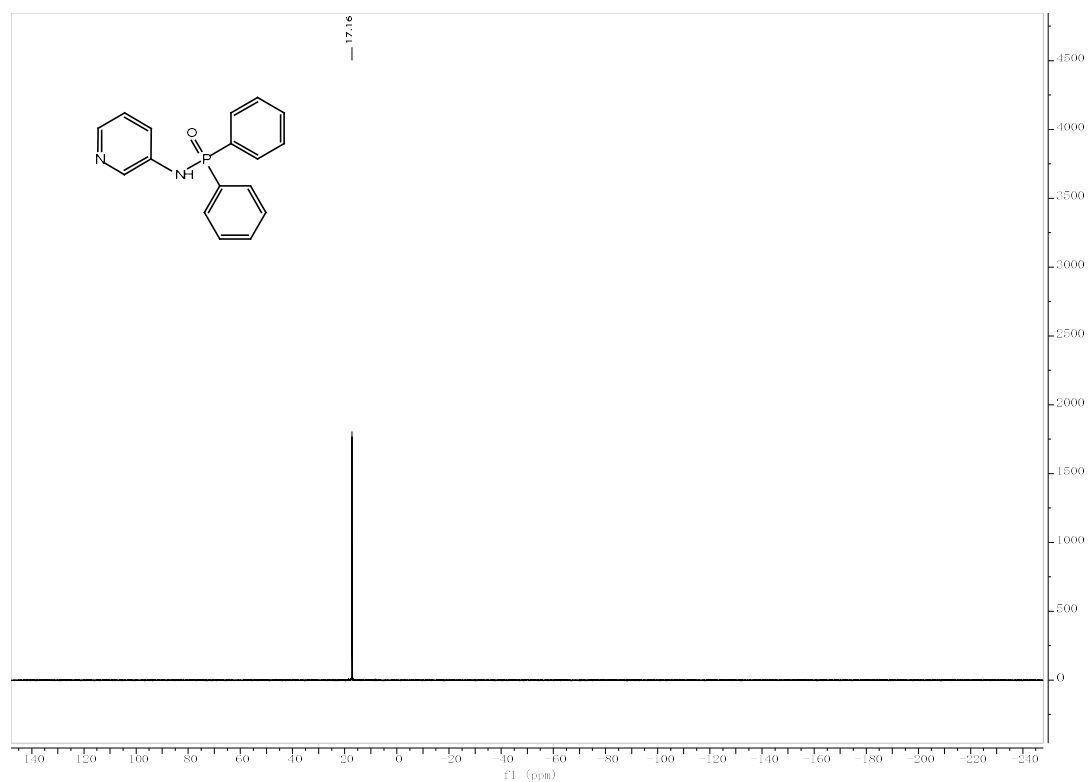
^{31}P NMR of 42 (162 MHz, CDCl_3)



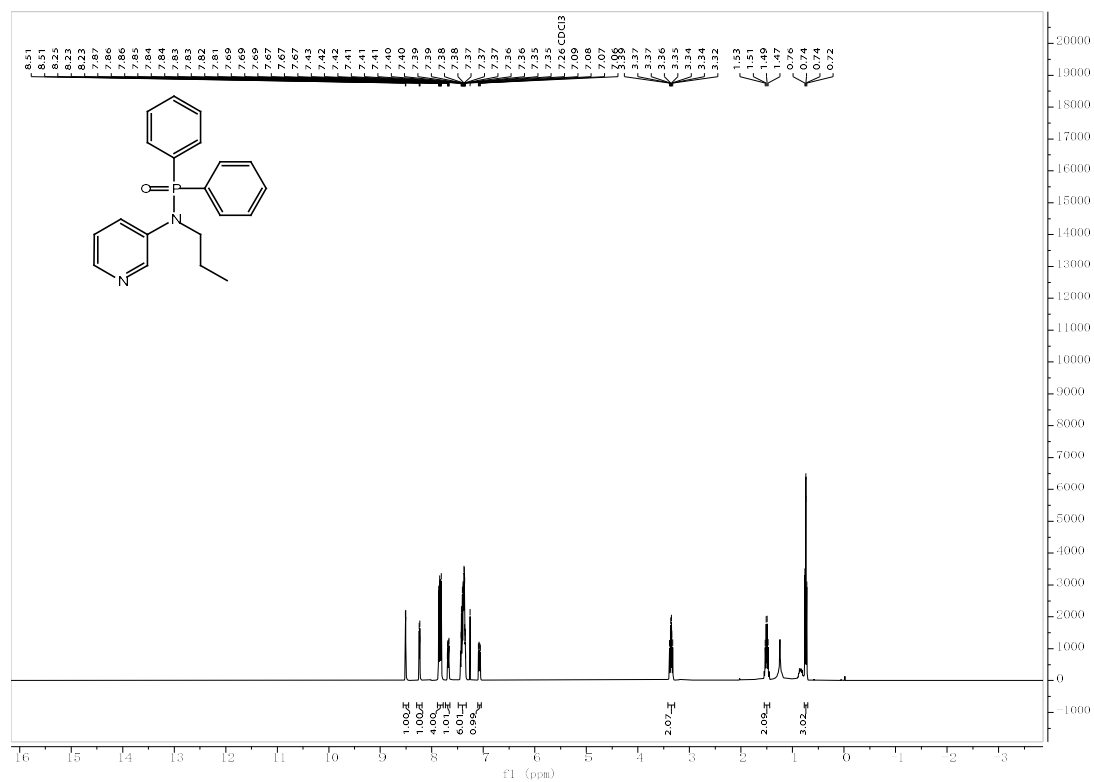
^1H NMR of 44 (400 MHz, DMSO-d_6)



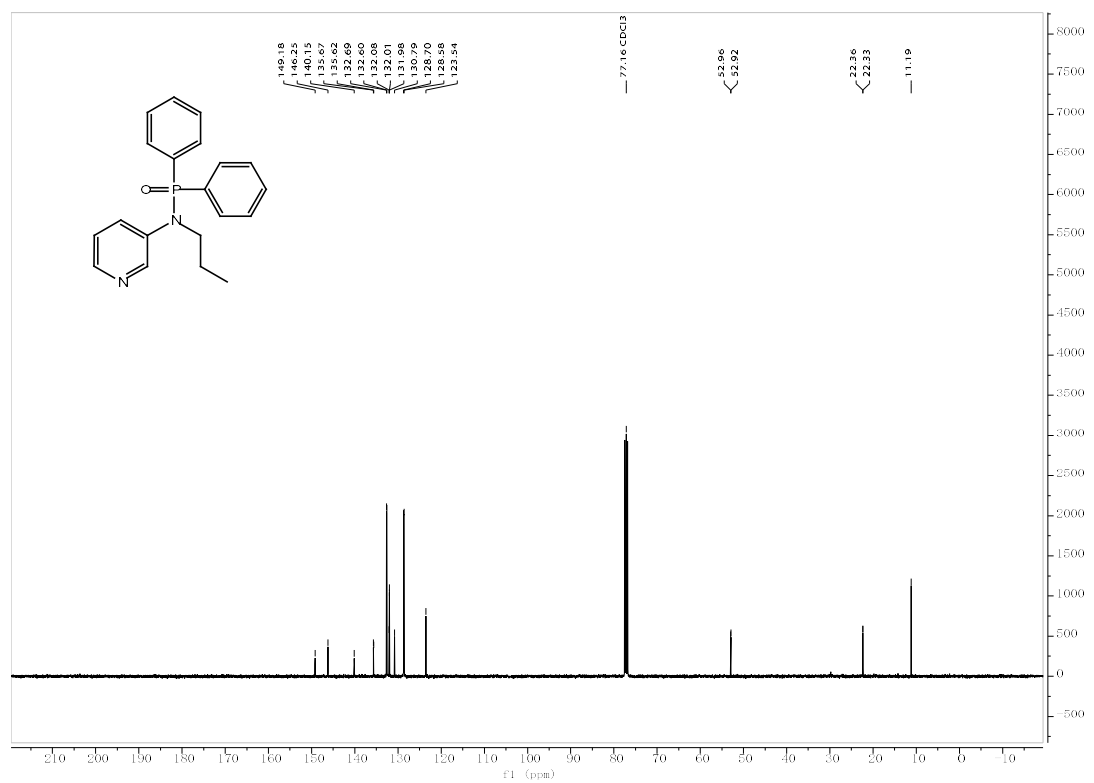
^{13}C NMR of **44** (100 MHz, DMSO- d_6)



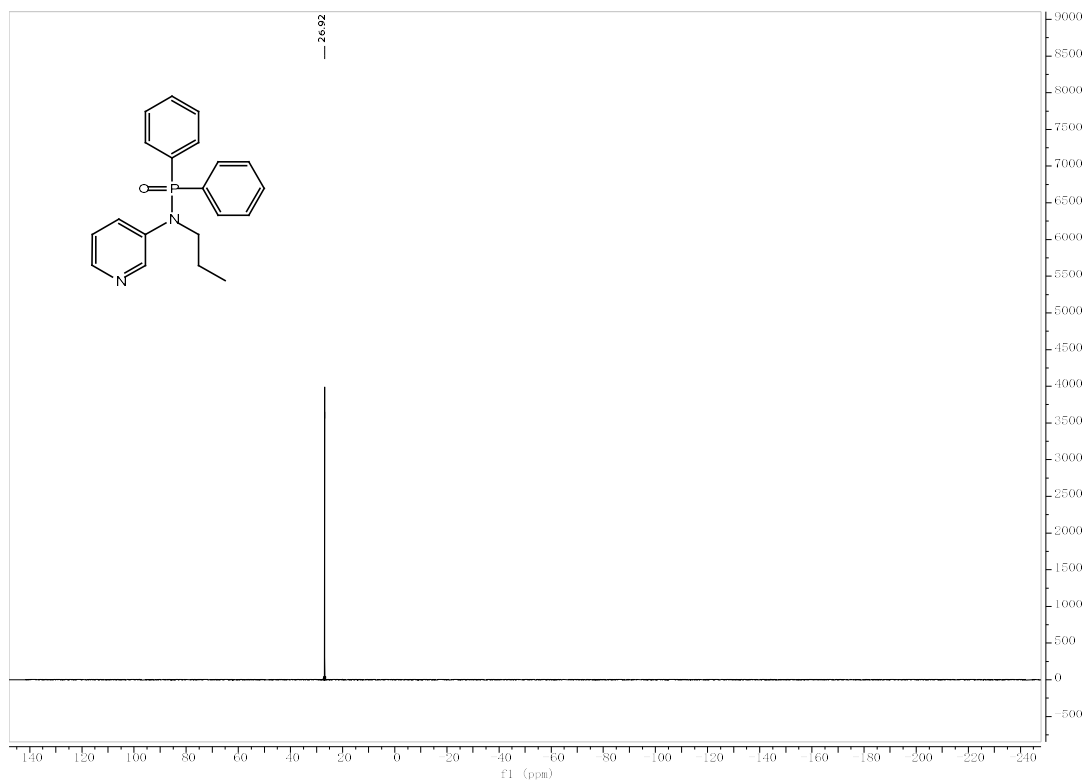
^{31}P NMR of **44** (162 MHz, DMSO- d_6)



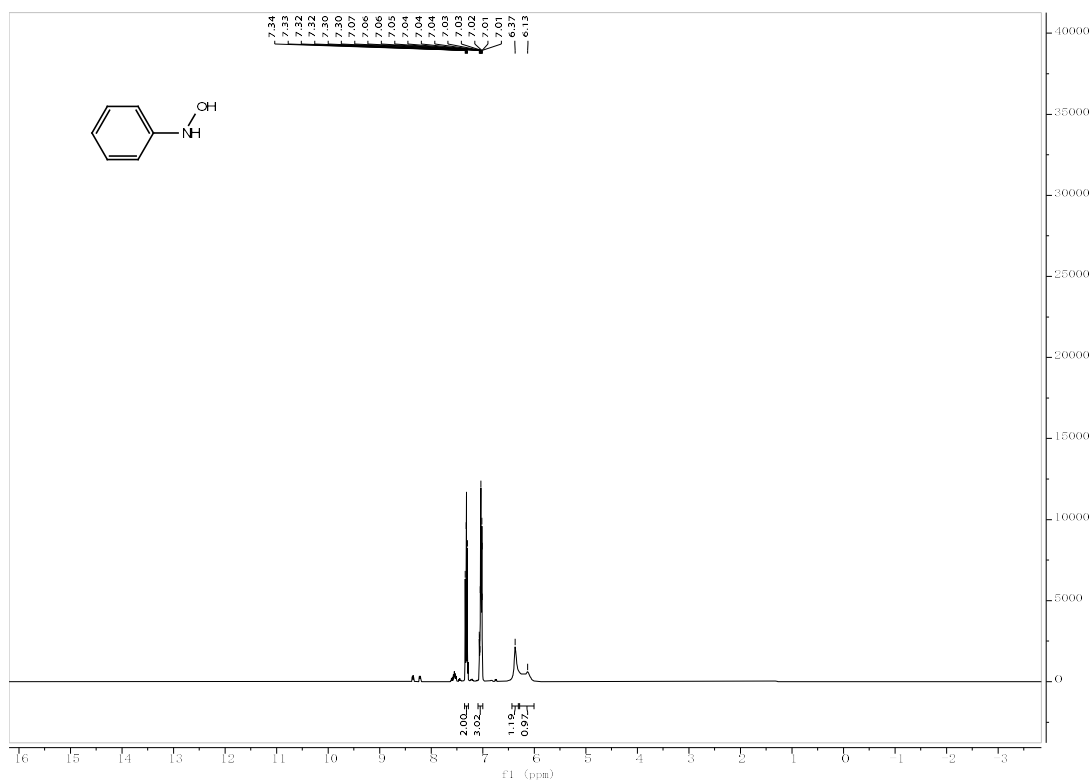
¹H NMR of 45 (400 MHz, CDCl₃)



¹³C NMR of 45 (100 MHz, CDCl₃)



^{31}P NMR of 45 (162 MHz, CDCl_3)



^1H NMR of 46 (400 MHz, CDCl_3)