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Metal-Free Direct C-H Phosphonation of N-heterocycles with Diphenylphosphine Oxides under Mild Condition

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Supporting Information

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I. General methods and materials

All manipulations were performed under an air atmosphere unless otherwise statement. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AC-P 400 spectrometer (400 MHz for ^{1}H, 100 MHz for ^{13}C) in $CDCl_3$. Chemical shifts (ppm) were recorded with tetramethylsilane (TMS) as the internal reference standard. Multiplicities are given as: s (singlet), d (doublet), t (triplet), dd (doublet of doublets), q (quartet) or m (multiplet). Copies of their ^{1}H NMR and ^{13}C NMR spectra are provided in the Supporting Information. High resolution mass spectra (HRMS) were recorded on quadrupole time-of-flight mass spectrometer (Q-TOF-MS) using electrospray ionization (ESI) as an ionization method. Melting points were obtained on Shanghai Inesa WRS-3 melting point apparatus. Solvents were dried and purified according to the procedure from "Purification of Laboratory Chemicals book". The crude products were purified by flash column chromatography on silica gel and the reported yields are the actual isolated yields of pure products. Unless stated otherwise, commercial reagents were used without further purification. All reagents were weighed and handled in air at room temperature.

II. Synthesis of substrates

General Procedure for the Preparation of various 1,2,4-Triazine-3,5(2*H*,4*H*)-diones

The substrates of various 1,2,4- triazine-3,5(2*H*,4*H*)-diones **1a-1t, 1ai** were synthesized according to procedures described in the previous literature studies.[1, 2]

General Procedure to Prepare Various Quinoxalin-2(1*H*)-ones

The substrates of various quinoxalin-2(1*H*)-ones **1z-1ab,1aj-1am** were synthesized according to procedures described in the previous literature studies.[3]

III. General procedure

Procedure for the Synthesis of 3a

To a solution of 2,4-dibenzyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione **1a** (0.2 mmol) and diphenylphosphine oxide **2a** (0.5 mmol) in DMC (1 mL) were placed in a flame-dried Schlenk-tube equipped with a magnetic stir bar, followed by addition of DBU (2.0 equiv.) *via* syringe. The reaction was stirred at room temperature while the flask was left uncapped for 12 h. The progress of the reaction was monitored by TLC. After completion, the residue was quenched with water, and ethyl acetate was added three times for extraction. The combined organic layers were dried over anhydrous Na_2SO_4. The resulting mixture was then concentrated under reduced pressure and washed by *n*-hexane to give **3a** (97％ yield).

Scale-up experiment

To a solution of 2,4-dibenzyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione **1a** (1.17 g, 4 mmol) and

diphenylphosphine oxide **2a** (2.02 g, 10 mmol) in DMC (0.2 M) were placed in a flame-dried Schlenk-tube equipped with a magnetic stir bar, followed by addition of DBU (2.0 equiv.) *via* syringe. The reaction was stirred at room temperature while the flask was left uncapped for 12 h. The progress of the reaction was monitored by TLC. After completion, the residue was quenched with water, and ethyl acetate was added three times for extraction. The combined organic layers were dried over anhydrous Na_2SO_4. The resulting mixture was then concentrated under reduced pressure and washed by *n*-hexane to give **3a** (1.80 g, 92％ yield).

Procedure for the synthesis of 4[3]

A solution of **3u** (0.2 mmol) in toluene was added CuTc (0.008 mmol), then the mixture was stirred for 3 min at room temperature, followed by addition of TsN_3 (0.24 mmol) *via* syringe. The reaction mixture was stirred for 7 h at room temperature. The residue was purified by column chromatography to afford **4** as white solid in 84% yield.

Procedure for the synthesis of 5[3]

A mixture of **3t** (0.2 mmol) and an excess of *m*-CPBA (0.4 mmol) was stirred at room temperature for 13 h. The mixture was poured into saturated $NaHCO_3$ (aq.) and extracted with ethyl acetate. The combined extracts were dried over anhydrous Na_2SO_4, filtered, and evaporated. The residue was purified by column chromatography to afford **5** as white solid in 41% yield.

Ⅳ. Procedure for the recovery and reuse of remainder DBU, diphenylphosphine oxide and DMC

To a solution of 2,4-dibenzyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione **1a** (0.2 mmol) and diphenylphosphine oxide **2a** (2.5 equiv.) in DMC (1 mL) were placed in a flame-dried Schlenk-tube equipped with a magnetic stir bar, followed by addition of DBU (2.0 equiv.) *via* syringe. The reaction was stirred at room temperature while the Schlenk-tube was left uncapped for 12 h. The progress of the reaction was monitored by TLC. After the reaction completing, the reaction mixture was distillation under reduced pressure (0.1 Mpa, 43℃) and 88 % of DMC was recovered. And the residue was washed with water and *n*-hexane, the expected product was obtained in 89% yield, which was collected by filtration. The water (include excess DBU and diphenylphosphine oxide) was evaporated under reduced pressure. The 0.2 mmol of 2,4-dibenzyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione, diphenylphosphine oxide (2.0 equiv.), DMC (1 mL) and DBU (1.0 equiv.) were added to the residue, and the mixture reacted under standard conditions for 12 hours. The abovementioned post-processing procedure was repeated, and the target product was obtained in 87% yield. This cycle was repeated four times totally, the yield of each cycle is shown in Figure S1.

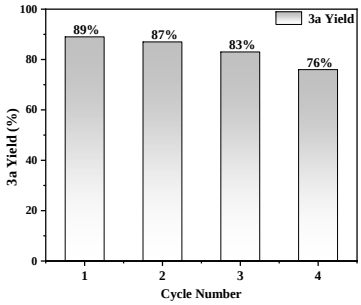
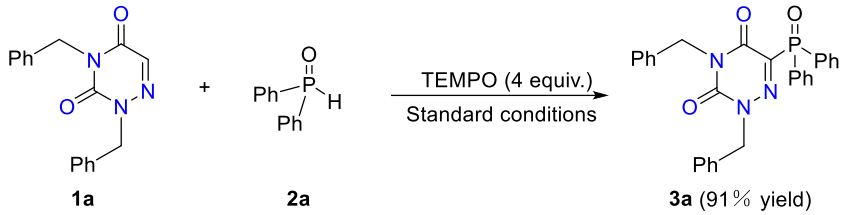


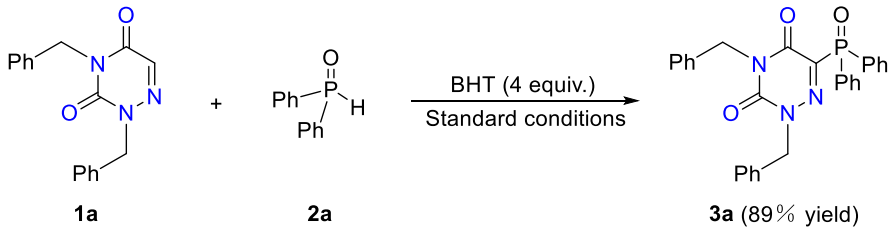
Figure S1 Product yields of recovery and reuse experiments

V. Mechanistic Experiments

Radical Trapping Experiment



To a solution of 2,4-dibenzyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione **1a** (0.2 mmol), diphenylphosphine oxide **2a** (0.5 mmol) and 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) (4.0 equiv.) in DMC (1 mL) were placed in a flame-dried Schlenk-tube equipped with a magnetic stir bar, followed by addition of DBU (2.0 equiv.) *via* syringe. The reaction was stirred at room temperature while the flask was left uncapped for 12 h. The progress of the reaction was monitored by TLC. After completion, the residue was quenched with water, and ethyl acetate was added three times for extraction. The combined organic layers were dried over anhydrous Na_2SO_4. The resulting mixture was then concentrated under reduced pressure and washed by *n*-hexane to give **3a** (91% yield).



To a solution of 2,4-dibenzyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione **1a** (0.2 mmol), diphenylphosphine oxide **2a** (0.5 mmol) and butylated hydroxytoluene (BHT) (4 equiv.) in DMC (1 mL) were placed in a flame-dried Schlenk-tube equipped with a magnetic stir bar, followed by addition of DBU (2.0 equiv.) *via* syringe. The reaction was stirred at room temperature while the flask was left uncapped for 12 h. The progress of the reaction was monitored by TLC. After completion, the residue was quenched with water, and ethyl acetate was added three times for extraction. The combined organic layers were dried over anhydrous Na_2SO_4. The resulting mixture was then concentrated under reduced pressure and

washed by *n*-hexane, the residues were further purified by chromatography on silica gel to afford **3a** (89％ yield).

The detection of intermediate B

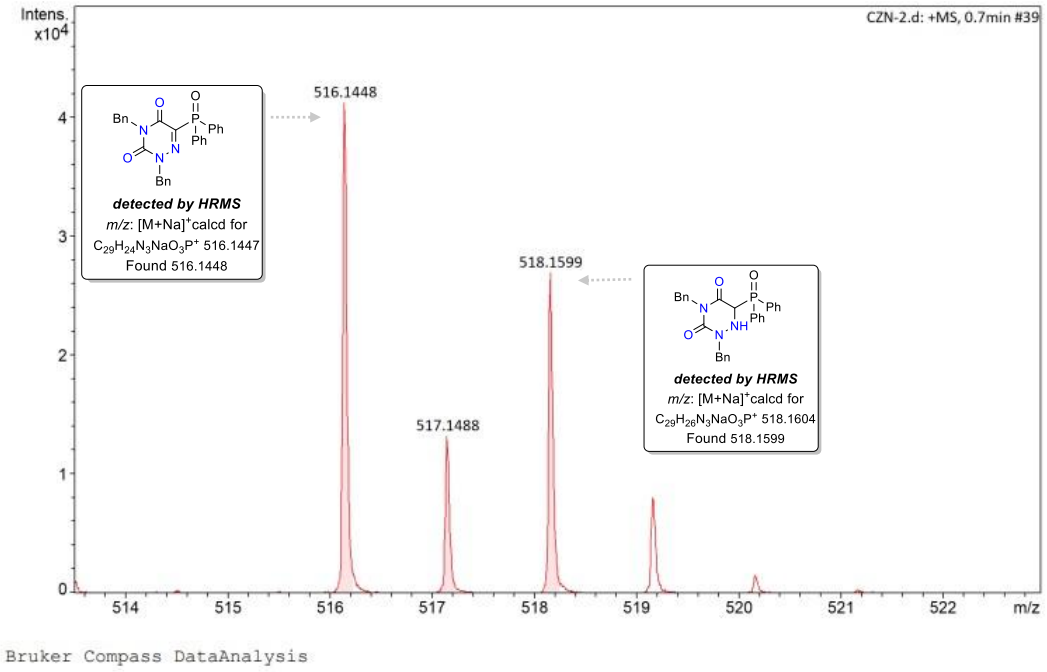
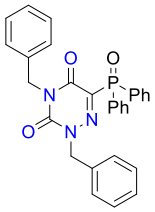


Figure S2 The HRMS analysis of the intermediate **B**

To a solution of 2,4-dibenzyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione **1a** (0.2 mmol) and diphenylphosphine oxide **2a** (0.5 mmol) in DMC (1 mL) were placed in a flame-dried Schlenk-tube equipped with a magnetic stir bar, followed by addition of DBU (2.0 equiv.) *via* syringe. The reaction was stirred at room temperature while the flask was left uncapped. After 1h reaction, the reaction mixture was directly detected by high-resolution mass spectrometer, and the intermediate **B** and target product **3a** can be found respectively.

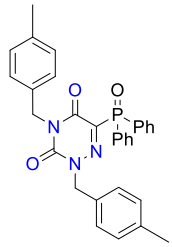
VI. Characterization data of 3a–5

Compounds **3z**, **3aa**, **3ad**, **3ae**, **3af**, **3ag** are known compounds[4-7], so only ^{1}H NMR spectrum were included in the Supporting Information.

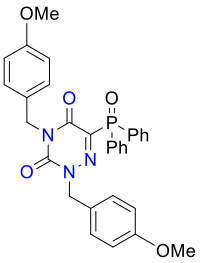


*2,4-dibenzyl-6-(diphenylphosphoryl)-1,2,4-triazine-3,5(2H,4H)-dione (**3a**)*. a white solid (97% yield, 96.1 mg). m. p. = 178-179 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.82–7.78 (m, 4H), 7.63–7.60 (m,

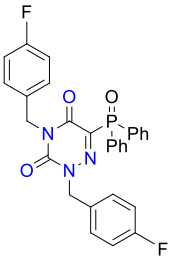
2H), 7.54–7.50 (m, 4H), 7.44 (s, 2H), 7.34–7.30 (m, 6H), 7.22–7.20 (m, 2H), 5.05 (s, 2H), 5.03 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.8, 154.6, 148.1, 141.0, 139.8, 134.8, 134.5, 132.4(d, *J* = 2.2 Hz), 131.8(d, *J* = 10.0 Hz), 130.2, 129.6, 129.1, 128.7, 128.5, 128.4, 128.2, 55.8, 44.1; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.8 (s); HRMS (ESI): m/z calcd for $C_{29}H_{24}N_3NaO_3P^+$ $[M+Na]^+$ 516.1447. Found 516.1447.



6-(diphenylphosphoryl)-2,4-bis(4-methylbenzyl)-1,2,4-triazine-3,5(2H,4H)-dione ***(3b)***. a white solid (80% yield, 83.6 mg). m. p. = 183-184 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.80–7.75 (m, 4H), 7.61–7.57 (m, 2H), 7.51–7.47 (m, 4H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.11–7.07 (m, 6H), 4.97 (s, 2H), 4.95 (s, 2H), 2.34 (s, 3H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.9, 154.7, 148.0, 140.9, 139.6, 138.4, 138.0, 132.4(d, *J* = 3.0 Hz), 132.0, 131.8(d, *J* = 9.7 Hz), 131.5, 130.3, 129.6, 129.4, 129.2(d, *J* = 7.0 Hz), 128.4(d, *J* = 12.9 Hz), 55.7, 43.9, 21.2, 21.1; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.8 (s); HRMS (ESI): m/z calcd for $C_{31}H_{28}N_3NaO_3P^+$ $[M+Na]^+$ 544.1760. Found 544.1761.

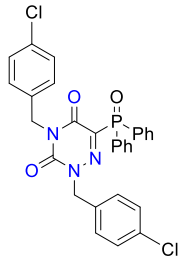


6-(diphenylphosphoryl)-2,4-bis(4-methoxybenzyl)-1,2,4-triazine-3,5(2H,4H)-dione ***(3c)***. a white solid (82% yield, 90.3 mg). m. p. = 172-173 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.79–7.74 (m, 4H), 7.60–7.57 (m, 2H), 7.50–7.47 (m, 4H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.11 (d, *J* = 8.0 Hz, 2H), 6.81–6.77 (m, 4H), 4.95 (s, 2H), 4.93 (s, 2H), 3.79 (s, 3H), 3.76 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 159.7, 159.4, 154.8, 154.6, 148.0,140.8, 139.5, 132.3(d, *J* = 2.5 Hz), 131.8(d, *J* = 9.9 Hz), 131.2, 130.6, 130.3, 129.2, 128.4(d, *J* = 12.9 Hz), 127.1, 126.6, 113.9(d, *J* = 17.5 Hz), 55.3, 55.2, 55.2, 43.6; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.9 (s); HRMS (ESI): m/z calcd for $C_{31}H_{28}N_3NaO_5P^+$ $[M+Na]^+$ 576.1659. Found 576.1658.

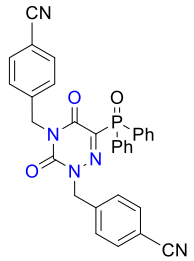


6-(diphenylphosphoryl)-2,4-bis(4-fluorobenzyl)-1,2,4-triazine-3,5(2H,4H)-dione ***(3d)***. a white solid (63% yield, 66.2 mg). m. p. = 158-159 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.78–7.73 (m, 4H), 7.62–7.58 (m, 2H), 7.51–7.48 (m, 4H), 7.42–7.39 (m, 2H), 7.15–7.12 (m, 2H), 7.79 (q, *J* = 8.0 Hz, 4H), 4.98 (s, 2H), 4.95 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.0, 163.8, 161.6, 161.4, 154.7, 154.5, 148.0, 141.3, 140.0, 132.5(d, *J* = 3.3 Hz), 131.8, 131.7, 131.7, 131.1(d, *J* = 7.6 Hz), 130.7(d, *J* = 2.5 Hz), 130.2(d, *J* = 3.5 Hz), 130.1, 129.0, 128.5(d, *J* = 13.2 Hz), 115.8, 115.6(d, *J* = 1.0 Hz), 115.3, 55.0,

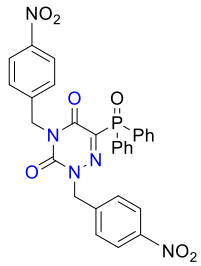
43.5; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.9 (s); HRMS (ESI): m/z calcd for $C_{29}H_{22}F_2N_3NaO_3P^+$ $[M+Na]^+$ 552.1259. Found 552.1257.



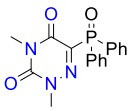
2,4-bis(4-chlorobenzyl)-6-(diphenylphosphoryl)-1,2,4-triazine-3,5(2H,4H)-dione ***(3e)***. a white solid (72% yield, 80.3 mg). m. p. = 160-161 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.72–7.67 (m, 4H), 7.57–7.53 (m, 2H), 7.46–7.42 (m, 4H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.21–7.17 (m, 4H), 7.03 (d, *J* = 8.0 Hz, 2H), 4.92 (s, 2H), 4.89 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.6, 154.4, 148.0, 141.4, 140.1, 134.7, 134.3, 133.2, 132.7, 132.5(d, *J* = 3.3 Hz), 131.8(d, *J* = 10.0 Hz), 131.1, 130.6, 130.0, 128.9, 128.7, 128.6, 128.5, 55.0, 43.5; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.9 (s); HRMS (ESI): m/z calcd for $C_{29}H_{22}Cl_2N_3NaO_3P^+$ $[M+Na]^+$ 584.0668. Found 584.0667.



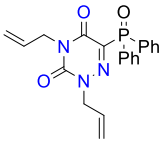
4,4'-((6-(diphenylphosphoryl)-3,5-dioxo-1,2,4-triazine-2,4(3H,5H)-diyl)bis(methylene))dibenzonit rile ***(3f)***. a white solid (51% yield, 55.7 mg). m. p. = 201-202 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.79–7.74 (m, 4H), 7.64 (t, *J* = 8.0 Hz, 2H), 7.59–7.49 (m, 11H), 7.28 (s, 1H), 5.08 (s, 2H), 5.03 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.5, 154.3, 148.0, 142.0, 140.7, 140.0, 139.0, 132.7(d, *J* = 2.6 Hz), 132.5, 132.4, 131.7(d, *J* = 11.0 Hz), 130.3, 129.8, 128.6(d, *J* = 12.6 Hz), 118.3, 118.1, 112.7, 112.4, 55.2, 43.9; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 23.0 (s); HRMS (ESI): m/z calcd for $C_{31}H_{22}N_5NaO_3P^+$ $[M+Na]^+$ 566.1352. Found 566.1353.



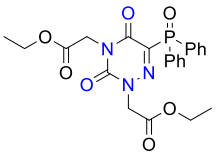
6-(diphenylphosphoryl)-2,4-bis(4-nitrobenzyl)-1,2,4-triazine-3,5(2H,4H)-dione ***(3g)***. a yellow solid (58% yield, 67.7 mg). m. p. = 118-119 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.07 (d, *J* = 8.0 Hz, 4H), 7.79–7.74 (m, 4H), 7.63–7.60 (m, 2H), 7.56–7.48 (m, 6H), 7.32 (d, *J* = 8.0 Hz, 2H), 5.11 (s, 2H), 5.06 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.5, 154.3, 148.0, 148.0, 147.8, 142.2, 141.4, 141.0, 140.9, 132.8(d, *J* = 2.6 Hz), 131.8(d, *J* = 9.5 Hz), 130.5, 130.1, 129.8, 128.6(d, *J* = 13.1 Hz), 123.9(d, *J* = 16.4 Hz), 54.9, 43.6; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.9 (s); HRMS (ESI): m/z calcd for $C_{29}H_{22}N_5NaO_7P^+$ $[M+Na]^+$ 606.1149. Found 606.1148.



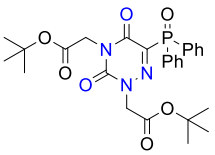
*6-(diphenylphosphoryl)-2,4-dimethyl-1,2,4-triazine-3,5(2H,4H)-dione (**3h**)*. a white solid (74% yield, 50.4 mg). m. p. = 198-199 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.87–7.82 (m, 4H), 7.58–7.55 (m, 2H), 7.49–7.46 (m, 4H), 3.69 (s, 3H), 3.27 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.4 155.2, 148.5, 139.9, 138.6, 132.5(d, *J* = 2.9 Hz), 131.9(d, *J* = 10.0 Hz), 130.2, 129.1, 128.4(d, *J* = 12.8 Hz), 40.5, 27.1; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 21.5 (s); HRMS (ESI): m/z calcd for $C_{17}H_{16}N_3NaO_3P^+$ $[M+Na]^+$ 364.0821. Found 364.0821.



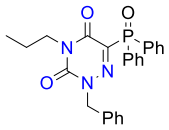
*2,4-diallyl-6-(diphenylphosphoryl)-1,2,4-triazine-3,5(2H,4H)-dione (**3i**)*. a white solid (76% yield, 59.6 mg). m. p. = 152-153 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.84–7.79 (m, 4H), 7.59–7.55 (m, 2H), 7.50–7.46 (m, 4H), 5.89–5.74 (m, 2H), 5.29–5.19 (m, 4H), 4.56 (d, *J* = 4.0 Hz, 2H), 4.45 (d, *J* = 4.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.6, 154.4, 147.7, 140.8, 139.5, 132.4(d, *J* = 3.2 Hz), 131.9(d, *J* = 9.7 Hz), 130.4, 130.2, 129.6, 129.1, 128.5(d, *J* = 12.3 Hz), 120.4, 120.0, 54.9, 42.9; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.1 (s); HRMS (ESI): m/z calcd for $C_{21}H_{20}N_3NaO_3P^+$ $[M+Na]^+$ 416.1134. Found 416.1130.



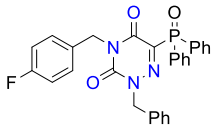
*diethyl 2,2'-(6-(diphenylphosphoryl)-3,5-dioxo-1,2,4-triazine-2,4(3H,5H)-diyl)diacetate (**3j**)*. a white solid (74% yield, 72.2 mg). m. p. = 183-184 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.86–7.81 (m, 4H), 7.60–7.56 (m, 2H), 7.51–7.47 (m, 4H), 4.78 (s, 2H), 4.58 (s, 2H), 4.26–4.15 (m, 4H), 1.28 (t, *J* = 8.0 Hz, 3H), 1.23 (t, *J* = 8.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.6, 165.9, 154.5, 154.3, 148.0, 141.5, 140.3, 132.7(d, *J* = 2.0 Hz), 132.0(d, *J* = 10.4 Hz), 130.0, 128.9, 128.6(d, *J* = 12.8 Hz), 62.2, 62.1, 53.4, 41.4, 14.1, 14.0; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 21.6 (s); HRMS (ESI): m/z calcd for $C_{23}H_{24}N_3NaO_7P^+$ $[M+Na]^+$ 508.1244. Found 508.1245.



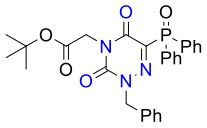
*di-tert-butyl 2,2'-(6-(diphenylphosphoryl)-3,5-dioxo-1,2,4-triazine-2,4(3H,5H)-diyl)diacetate (**3k**)*. a white solid (80% yield, 86.7 mg). m. p. = 161-162 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.86–7.81 (m, 4H), 7.58–7.55 (m, 2H), 7.49–7.45 (m, 4H), 4.67 (s, 2H), 4.47 (s, 2H), 1.45 (s, 9H), 1.40 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.5, 164.9, 154.6, 154.4, 148.0, 141.0, 140.0, 132.5(d, *J* = 2.4 Hz), 132.0(d, *J* = 9.9 Hz), 130.2, 129.0, 128.5(d, *J* = 12.9 Hz), 83.3, 83.1, 54.1, 42.1, 27.9, 27.8; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 21.5 (s); HRMS (ESI): m/z calcd for $C_{27}H_{32}N_3NaO_7P^+$ $[M+H]^+$ 564.1870. Found 564.1870.



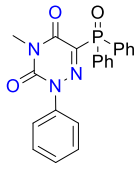
2-benzyl-6-(diphenylphosphoryl)-4-propyl-1,2,4-triazine-3,5(2H,4H)-dione ***(3l)***. a white solid (87% yield, 77.6 mg). m. p. = 145-146 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.83–7.78 (m, 4H), 7.59–7.55 (m, 2H), 7.49–7.46 (m, 4H), 7.41–7.40 (m, 2H), 7.27–7.26 (m, 3H), 5.02 (s, 2H), 3.91 (t, *J* = 8.0 Hz, 2H), 1.70–1.61 (m, 2H), 0.84 (t, *J* = 8.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.0, 154.8, 148.2, 140.3, 139.1, 135.0, 132.4(d, *J* = 2.7 Hz), 131.8(d, *J* = 9.9 Hz), 130.4, 129.5, 129.3, 128.5(d, *J* = 3.2 Hz), 128.4, 128.2, 54.1, 44.1, 21.3, 10.8; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.4 (s); HRMS (ESI): m/z calcd for $C_{25}H_{24}N_3NaO_3P^+$ $[M+Na]^+$ 468.1447. Found 468.1447.



2-benzyl-6-(diphenylphosphoryl)-4-(4-fluorobenzyl)-1,2,4-triazine-3,5(2H,4H)-dione ***(3m)***. a white solid (81% yield, 82.3 mg). m. p. = 146-147 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.79–7.73 (m, 4H), 7.61–7.58 (m, 2H), 7.51–7.47 (m, 4H), 7.40–7.39 (m, 2H), 7.27–7.26 (m, 3H), 7.14 (t, *J* = 8.0 Hz, 2H), 6.96 (t, *J* = 8.0 Hz, 2H), 4.99 (s, 2H), 4.99 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.0, 161.6, 154.7, 154.6, 148.1, 141.3, 140.0, 134.8, 132.5(d, *J* = 3.3 Hz), 131.8(d, *J* = 9.7 Hz), 131.1(d, *J* = 8.7 Hz), 130.3(d, *J* = 3.0 Hz), 130.2, 129.6, 129.1, 128.6(d, *J* = 1.2 Hz), 128.4, 128.3, 115.8, 115.6, 55.0, 44.2; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.8 (s); HRMS (ESI): m/z calcd for $C_{29}H_{23}FN_3NaO_3P^+$ $[M+Na]^+$ 534.1353. Found 534.1353.

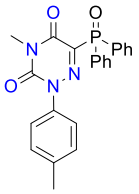


tert-butyl 2-(2-benzyl-6-(diphenylphosphoryl)-3,5-dioxo-2,5-dihydro-1,2,4-triazin-4(3H)-yl)acetat e ***(3n)***. a white solid (75% yield, 77.8 mg). m. p. = 144-145 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.85–7.80 (m, 4H), 7.59–7.56 (m, 2H), 7.49–7.45 (m, 4H), 7.37–7.36 (m, 2H), 7.27–7.26 (m, 3H), 5.02 (s, 2H), 4.62 (s, 2H), 1.42 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.7, 155. 0, 154.8, 148.3, 141.3, 140.0, 134.8, 132.5(d, *J* = 2.8 Hz), 131.9(d, *J* = 10.0 Hz), 130.2, 129. 3, 129.1, 128.6, 128.4, 128.2, 83.3, 54.0, 44.1, 27.9; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 21.8 (s); HRMS (ESI): m/z calcd for $C_{28}H_{28}N_3NaO_5P^+$ $[M+Na]^+$ 540.1659. Found 540.1659.

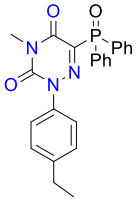


6-(diphenylphosphoryl)-4-methyl-2-phenyl-1,2,4-triazine-3,5(2H,4H)-dione ***(3o)***. a white solid (88% yield, 71.3 mg). m. p. = 236-237 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.92–7.87 (m, 4 H), 7.59–7.55 (m, 2H), 7.50–7.44 (m, 7H), 7.17 (d, *J* = 4.0 Hz, 2H), 3.76 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.1, 154.9, 148.2, 141.2, 139.9, 132.6(d, *J* = 1.7 Hz), 132.0(d, *J* = 1 0.8 Hz), 130.2, 129.5(d, *J* = 3.3 Hz), 129.1, 128.5(d, *J* = 12.7 Hz), 127.6, 40.7; ^{31}P NMR (16

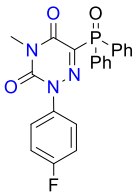
2 MHz, $CDCl_3$) δ: 21.2 (s); HRMS (ESI): m/z calcd for $C_{22}H_{18}N_3NaO_3P^+$ $[M+Na]^+$ 426.0978. Found 426.0978.



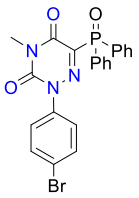
6-(diphenylphosphoryl)-4-methyl-2-(p-tolyl)-1,2,4-triazine-3,5(2H,4H)-dione (***3p***). a white solid (94% yield, 78.2 mg). m. p. = 210-211 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.93–7.88 (m, 4 H), 7.58–7.55 (m, 2H), 7.50–7.46 (m, 4H), 7.25 (d, *J* = 8.0 Hz, 2H), 7.04 (d, *J* = 8.0 Hz, 2 H), 3.75 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.2, 155.0, 148.3, 141.0, 13 9.7(d, *J* = 7.2 Hz), 132.5(d, *J* = 2.3 Hz), 132.0(d, *J* = 9.3 Hz), 130.2, 129.3, 129.1, 128.5(d, *J* = 12.4 Hz), 127.3, 40.7, 21.2; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 21.2 (s); HRMS (ESI): m/z calcd for $C_{23}H_{20}N_3NaO_3P^+$ $[M+Na]^+$ 440.1134. Found 440.1133.



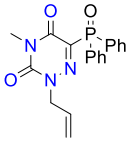
6-(diphenylphosphoryl)-2-(4-ethylphenyl)-4-methyl-1,2,4-triazine-3,5(2H,4H)-dione (***3q***). a whit e solid (92% yield, 79.7 mg). m. p. = 170-171 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.93–7.88 (m, 4H), 7.58–7.55 (m, 2H), 7.50–7.46 (m, 4H), 7.28 (d, *J* = 12.0 Hz, 2H), 7.07 (d, *J* = 8.0 Hz, 2H), 3.76 (s, 3H), 2.66 (q, *J* = 8.0 Hz, 2H), 1.22 (t, *J* = 8.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.3, 155.1, 148.4, 145.8, 141.0, 139.8, 132.5(d, *J* = 3.6 Hz), 132.0(d, *J* = 11.2 Hz), 130.2, 129.5, 129.1, 129.0, 128.5(d, *J* = 11.6 Hz), 127.3, 40.7, 28.5, 15.1; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 21.1 (s); HRMS (ESI): m/z calcd for $C_{24}H_{22}N_3NaO_3P^+$ $[M+Na]^+$ 454.12 91. Found 454.1291.



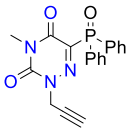
6-(diphenylphosphoryl)-2-(4-fluorophenyl)-4-methyl-1,2,4-triazine-3,5(2H,4H)-dione (***3r***). a whi te solid (90% yield, 75.8 mg). m. p. = 184-185 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.91–7.86 (m, 4H), 7.60–7.56 (m, 2H), 7.50–7.47 (m, 4H), 7.16–7.11 (m, 4H), 3.74 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.0, 161.6, 155.1, 154.9, 148.2, 141.2, 139.9, 132.6(d, *J* = 2.7 Hz), 1 32.0(d, *J* = 9.9 Hz), 130.1, 129.7(d, *J* = 9.3 Hz), 129.0, 128.6(d, *J* = 13.1 Hz), 127.8, 116.7, 116.5, 40.7; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 21.3 (s); HRMS (ESI): m/z calcd for $C_{22}H_{17}FN_3NaO_3P^+$ $[M+Na]^+$ 444.0884. Found 444.0882.



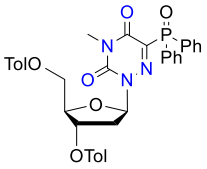
2-(4-bromophenyl)-6-(diphenylphosphoryl)-4-methyl-1,2,4-triazine-3,5(2H,4H)-dione (***3s***). a white solid (93% yield, 89.3 mg). m. p. = 155-156 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.91–7.86 (m, 4H), 7.58–7.57 (m, 4H), 7.51–7.47 (m, 4H), 7.05 (d, *J* = 8.0 Hz, 2H), 3.75 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.9, 154.7, 148.0, 141.2, 140.0, 132.7, 132.6(d, *J* = 2.8 Hz), 132.0(d, *J* = 11.4 Hz), 131.0, 130.1, 129.4, 129.0, 128.6(d, *J* = 13.8 Hz), 123.7, 40.7; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 21.2 (s); HRMS (ESI): m/z calcd for $C_{22}H_{17}BrN_3NaO_3P^+$ $[M+Na]^+$ 504.0083. Found 504.0082.



2-allyl-6-(diphenylphosphoryl)-4-methyl-1,2,4-triazine-3,5(2H,4H)-dione (***3t***). a white solid (94% yield, 69.0 mg). m. p. = 152-153 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.87–7.82 (m, 4H), 7.59–7.55 (m, 2H), 7.51–7.47 (m, 4H), 5.83–5.73 (m, 1H), 5.29–5.19 (m, 4H), 4.45 (d, *J* = 4.0 Hz, 2H), 3.68 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.9, 154.7, 148.1, 140.3, 139.1, 132.5(d, *J* = 1.9 Hz), 131.9(d, *J* = 11.2 Hz), 130.3, 129.6, 129.2, 128.5(d, *J* = 11.8 Hz), 120.0, 42.9, 40.5; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 21.6 (s); HRMS (ESI): m/z calcd for $C_{19}H_{18}N_3NaO_3P^+$ $[M+Na]^+$ 390.0978. Found 390.0977.

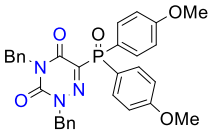


6-(diphenylphosphoryl)-4-methyl-2-(prop-2-yn-1-yl)-1,2,4-triazine-3,5(2H,4H)-dione (***3u***). a white solid (95% yield, 69.3 mg). m. p. = 155-156 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.88–7.83 (m, 4H), 7.60–7.56 (m, 2H), 7.51–7.47 (m, 4H), 4.60 (s, 2H), 3.71 (s, 3H), 2.19–2.17 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.3, 154.1, 147.6, 140.4, 139.2, 132.6(d, *J* = 2.4 Hz), 131.9(d, *J* = 9.9 Hz), 130.7, 130.6, 130.1, 129.0, 128.8, 128.6(d, *J* = 13.2 Hz), 75.9, 72.0, 40.5, 29.9; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 21.4 (s); HRMS (ESI): m/z calcd for $C_{19}H_{16}N_3NaO_3P^+$ $[M+Na]^+$ 388.0821. Found 388.0821.

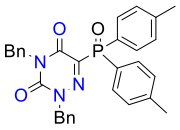


(2R,3S,5S)-5-(6-(diphenylphosphoryl)-4-methyl-3,5-dioxo-4,5-dihydro-1,2,4-triazin-2(3H)-yl)-2-(((4-methylbenzoyl)oxy)methyl)tetrahydrofuran-3-yl 4-methylbenzoate (***3v***). a white solid (83% yield, 112.6 mg). m. p. = 115-116 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.94–7.89 (m, 6H), 7.84–7.79 (m, 2H), 7.60–7.51 (m, 6H), 7.22 (t, *J* = 8.0 Hz, 4H), 6.67 (t, *J* = 8.0 Hz, 1H), 4.81–4.77 (m, 1H), 4.40–4.36 (m, 1H), 4.13–4.08 (m, 1H), 3.90–3.86 (m, 1H), 3.31 (s, 3H), 2.67–2.60

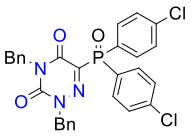
(m, 1H), 2.43–2.36 (m, 7H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.1, 165.7, 154.6, 154.4, 148.4, 144.3, 143.9, 142.5, 141.2, 132.7(d, *J* = 2.0 Hz), 132.6(d, *J* = 3.0 Hz), 132.0(d, *J* = 10.2 Hz), 131.6(d, *J* = 10.3 Hz), 130.3, 129.7(d, *J* = 1.3 Hz), 129.3, 129.2(d, *J* = 10.7 Hz), 129.0, 128.8, 128.7, 128.2, 126.9, 126.4, 87.7, 82.8, 75.0, 64.4, 35.0, 27.2, 21.7, 21.6; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 24.2 (s); HRMS (ESI): m/z calcd for $C_{37}H_{34}N_3NaO_8P^+$ $[M+Na]^+$ 702.1976. Found 702.1977.



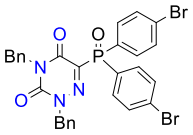
*2,4-dibenzyl-6-(bis(4-methoxyphenyl)phosphoryl)-1,2,4-triazine-3,5(2H,4H)-dione (**3w**)*. a white solid (90% yield, 99.2 mg). m. p. = 173-174 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.70–7.65 (m, 4H), 7.41–7.39 (m, 2H), 7.31–7.23 (m, 8H), 6.97 (d, *J* = 8.0 Hz, 4H), 5.04 (s, 2H), 4.99 (s, 2H), 3.86 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.8(d, *J* = 2.8 Hz), 154.8, 154.7, 148.2, 141.6, 140.3, 135.0, 134.6, 133.8(d, *J* = 12.8 Hz), 129.6, 129.1, 128.7, 128.5(d, *J* = 3.7 Hz), 128.2, 121.6, 120.5, 114.1(d, *J* = 14.4 Hz), 55.8, 55.3, 44.1; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.7 (s); HRMS (ESI): m/z calcd for $C_{31}H_{28}N_3NaO_5P^+$ $[M+Na]^+$ 576.1659. Found 576.1660.



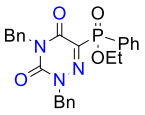
*2,4-dibenzyl-6-(di-p-tolylphosphoryl)-1,2,4-triazine-3,5(2H,4H)-dione (**3x**)*. a white solid (93% yield, 97.4 mg). m. p. = 177-178 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.71–7.66 (m, 4H), 7.45–7.44 (m, 2H), 7.35–7.31 (m, 10H), 7.25–7.23 (m, 2H), 5.07 (s, 2H), 5.03 (s, 2H), 2.46 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.8, 154.6, 148.1, 142.9, 142.9, 141.4, 140.1, 134.9, 134.6, 131.8(d, *J* = 11.4 Hz), 129.6, 129.3, 129.1, 128.7, 128.5(d, *J* = 3.5 Hz), 128.2, 127.1, 126.0, 55.8, 44.1, 21.7; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 23.1 (s); HRMS (ESI): m/z calcd for $C_{31}H_{28}N_3NaO_5P^+$ $[M+Na]^+$ 544.1760. Found 544.1759.



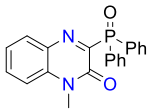
*2,4-dibenzyl-6-(bis(4-chlorophenyl)phosphoryl)-1,2,4-triazine-3,5(2H,4H)-dione (**3y**)*. a white solid (33% yield, 36.5 mg). m. p. = 225-226 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.69–7.64 (m, 4H), 7.47–7.45 (m, 4H), 7.41–7.40 (m, 2H), 7.33–7.26 (m, 6H), 7.19–7.17 (m, 2H), 5.05 (s, 2H), 5.00 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.8, 154.6, 148.0, 140.2, 139.4, 139.3, 138.9, 134.7, 134.3, 133.1(d, *J* = 11.7 Hz), 129.6, 129.2, 129.1, 128.9, 128.8, 128.7(d, *J* = 5.6 Hz), 128.4(d, *J* = 5.4 Hz), 127.3, 55.9, 44.3; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 21.0 (s); HRMS (ESI): m/z calcd for $C_{29}H_{22}Cl_2N_3NaO_3P^+$ $[M+Na]^+$ 584.0668. Found 584.0669.



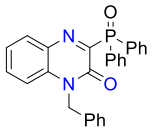
2,4-dibenzyl-6-(bis(4-bromophenyl)phosphoryl)-1,2,4-triazine-3,5(2H,4H)-dione ***(3z)***. a white solid (28% yield, 35.2 mg). m. p. = 247-248 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.61–7.55 (m, 8H), 7.41–7.39 (m, 2H), 7.32–7.26 (m, 6H), 7.18–7.17 (m, 2H), 5.04 (s, 2H), 5.00 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.7, 154.5, 148.0, 140.1, 138.8, 134.7, 134.2, 133.2(d, *J* = 10.3 Hz), 133.0, 132.1, 131.9(d, *J* = 14.0 Hz), 129.6, 129.2, 128.8(d, *J* = 6.1 Hz), 128.6(d, *J* = 6.1 Hz), 128.4, 128.0(d, *J* = 4.1 Hz), 127.7, 55.9, 44.3; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 21.4 (s); HRMS (ESI): m/z calcd for $C_{29}H_{22}Br_2N_3NaO_3P^+$ $[M+Na]^+$ 671.9658. Found 671.9657.



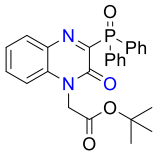
ethyl (2,4-dibenzyl-3,5-dioxo-2,3,4,5-tetrahydro-1,2,4-triazin-6-yl)(phenyl)phosphinate ***(3aa)***. a white solid (49% yield, 45.6 mg). m. p. = 132-133 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.94–7.88 (m, 2H), 7.63–7.58 (m, 1H), 7.52–7.47 (m, 2H), 7.46–7.43 (m, 2H), 7.31–7.27 (m, 6H), 7.24–7.22 (m, 2H), 5.14–5.11 (m, 1H), 5.01 (s, 2H), 4.99–4.96 (m, 1H), 4.38–4.23 (m, 2H), 1.38 (t, *J* = 8.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.6, 154.4, 148.2, 140.5, 138.9, 134.9, 134.6, 132.9(d, *J* = 3.4 Hz), 132.5(d, *J* = 9.5 Hz), 129.6, 129.5, 129.0, 128.7, 128.5(d, *J* = 5.4 Hz), 128.4, 128.2(d, *J* = 3.9 Hz), 128.0, 62.8, 55.8, 44.2, 16.5; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.9 (s); HRMS (ESI): m/z calcd for $C_{25}H_{24}N_3NaO_4P^+$ $[M+Na]^+$ 484.1397. Found 484.1394.



3-(diphenylphosphoryl)-1-methylquinoxalin-2(1H)-one ***(3ab)***. a yellow solid (98% yield, 70.8 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.02 (d, *J* = 8.0 Hz, 1H), 7.94 (t, *J* = 8.0 Hz, 4H), 7.64 (t, *J* = 8.0 Hz, 1H), 7.53 (d, *J* = 8.0 Hz, 2H), 7.48–7.47 (m, 4H), 7.38–7.32 (m, 2H), 3.61 (s, 3H). The spectral characteristics data were consistent with it reported previously in the literature.[7]

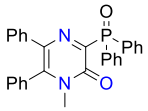


1-benzyl-3-(diphenylphosphoryl)quinoxalin-2(1H)-one ***(3ac)***. a yellow solid (97% yield, 84.8 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.02–7.97 (m, 4H), 7.95 (s, 1H), 7.58–7.55 (m, 2H), 7.51–7.48 (m, 5H), 7.33–7.25 (m, 5H), 7.15 (d, *J* = 8.0 Hz, 2H), 5.41 (s, 2H). The spectral characteristics data were consistent with it reported previously in the literature.[7]

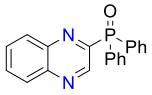


tert-butyl 2-(3-(diphenylphosphoryl)-2-oxoquinoxalin-1(2H)-yl)acetate ***(3ad)***. a yellow solid (98% yield, 90.1 mg). m. p. = 183-184 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.04 (d, *J* = 8.0 Hz, 1H), 7.96–7.90 (m, 4H), 7.61 (t, *J* = 8.0 Hz, 1H), 7.56–7.52 (m, 2H), 7.48–7.44 (m, 4H), 7.37 (t, *J* = 8.0 Hz, 1H), 7.10 (d, *J* = 8.0 Hz, 1H), 4.85 (s, 2H), 1.37 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.5, 156.4, 155.2, 153.7, 153.5, 133.5, 133.4, 133.2(d, *J* = 1.4 Hz), 133.

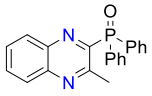
1, 132.3, 132.1(d, *J* = 10.8 Hz),131.0, 129.9, 128.3(d, *J* = 12.6 Hz), 124.2, 113.3, 83.3, 43.9, 27.8; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 23.8 (s); HRMS (ESI): m/z calcd for $C_{26}H_{25}N_2NaO_4P^+$ $[M+Na]^+$ 483.1444. Found 483.1444.



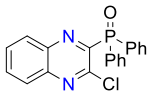
*3-(diphenylphosphoryl)-1-methyl-5,6-diphenylpyrazin-2(1H)-one (**3ae**)*. a yellow solid (65% yi eld, 60.2 mg). m. p. = 198-200 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.02–7.97 (m, 4H), 7.90–7.86 (m, 1H), 7.57 (d, *J* = 8.0 Hz, 2H), 7.54–7.50 (m, 4H), 7.47–7.45 (m, 2H), 7.35–7.30 (m, 1H), 7.24 (d, *J* = 8.0 Hz, 1H), 7.13–7.05 (m, 3H), 7.00 (d, *J* = 8.0 Hz, 2H), 3.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.3, 155.1, 150.8, 149.5, 142.0, 136.6, 134.3, 134.1, 132.1(d, *J* = 9.6 Hz), 131.9(d, *J* = 2.4 Hz), 131.8, 131.7, 131.5(d, *J* = 2.7 Hz), 131.4, 130.4, 130.1, 129.4(d, *J* = 7.2 Hz), 129.1, 128.9, 128.8, 128.6, 128.5, 128.2(d, *J* = 12.4 Hz), 127.6, 127.3, 127.2, 34.2; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 24.7 (s); HRMS (ESI): m/z calcd for $C_{29}H_{23}N_2NaO_2P^+$ $[M+Na]^+$ 485.1389. Found 485.1389.



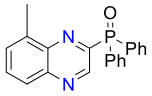
*diphenyl(quinoxalin-2-yl)phosphine oxide (**3af**)*. a white solid (40% yield, 26.4 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.67 (s, 1H), 8.21–7.18 (m, 2H), 8.02–7.97 (m, 4H), 7.91–7.83 (m, 2H), 7.60–7.57 (m, 2H), 7.53–7.49 (m, 4H). The spectral characteristics data were consistent with it reported previously in the literature.[4]



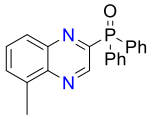
*(3-methylquinoxalin-2-yl)diphenylphosphine oxide (**3ag**)*. a white solid (60% yield, 41.0 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.02 (d, *J* = 8.0 Hz, 1H), 7.93 (d, *J* = 8.0 Hz, 1H), 7.85–7.77 (m, 5H), 7.69–7.66 (m, 1H), 7.57–7.53 (m, 2H), 7.49–7.45 (m, 4H), 2.99 (s, 3H). The spectral characteristics data were consistent with it reported previously in the literature.[4, 5]



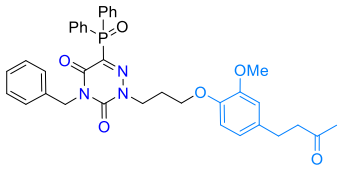
*(3-chloroquinoxalin-2-yl)diphenylphosphine oxide (**3ah**)*. a white solid (23% yield, 16.8 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.05 (d, *J* = 8.0 Hz, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.89–7.86 (m, 1H), 7.83–7.75 (m, 5H), 7.62–7.58 (m, 2H), 7.53–7.49 (m, 4H). The spectral characteristics data were consistent with it reported previously in the literature.[4]



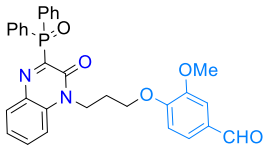
*(8-methylquinoxalin-2-yl)diphenylphosphine oxide (**3ai**)*. a white solid (28% yield, 19.2 mg). (PET/EtOAc = 4:1 as the eluet). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.64 (s, 1H), 8.01–7.96 (m, 5H), 7.74 (t, *J* = 8.0 Hz, 1H), 7.64 (d, *J* = 8.0 Hz, 1H), 7.56–7.52 (m, 2H), 7.49–7.45 (m, 4H), 2.73 (s, 3H). The spectral characteristics data were consistent with it reported previously in the literature.[4]



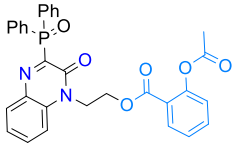
*(5-methylquinoxalin-2-yl)diphenylphosphine oxide (**3ai′**)*. a white solid (25% yield, 17.4 m g). (PET/EtOAc = 1:1 as the eluet). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.60 (s, 1H), 7.98–7.93 (m, 5H), 7.71-7.68 (m, 2H), 7.56-7.52 (m, 2H), 7.49-7.45 (m, 4H), 2.81 (s, 3H). The spectral characteristics data were consistent with it reported previously in the literature.[4]



*4-benzyl-6-(diphenylphosphoryl)-2-(3-(2-methoxy-4-(3-oxobutyl)phenoxy)propyl)-1,2,4-triazine-3, 5(2H,4H)-dione (**3ak**)*. a white solid (54% yield, 34.4 mg). m. p. = 132-133 °C. (PET/EtOAc = 1:4 as the eluet). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.62–7.57 (m, 4H), 7.41–7.37 (m, 2H), 7. 29–7.24 (m, 4H), 7.12–7.09 (m, 3H), 7.05–7.03 (m, 2H), 6.52–6.46 (m, 3H), 4.87 (s, 2H), 3.9 0 (t, *J* = 6.0 Hz, 2H), 3.81 (t, *J* = 6.0 Hz, 2H), 3.50 (s, 3H), 2.68–2.64 (m, 2H), 2.58–2.54 (m, 2H), 1.97 (s, 3H), 1.95–1.92 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 208.0, 155.1, 154. 9, 149.3, 148.3, 146.4, 140.6, 139.3, 134.6, 134.0, 132.3(d, *J* = 2.5 Hz), 131.8(d, *J* = 9.9 Hz), 130.4, 129.3, 129.1, 128.7, 128.5(d, *J* = 3.0 Hz), 128.4, 120.0, 113.3, 112.1, 67.2, 56.0, 55.6, 45.4, 39.1, 30.1, 29.3, 26.9; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.7 (s); HRMS (ESI): m/z calc d for $C_{36}H_{36}N_3NaO_6P^+$ $[M+Na]^+$ 660.2234. Found 660.2236.

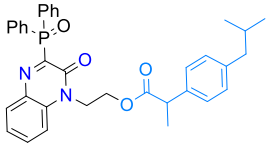


*4-(3-(3-(diphenylphosphoryl)-2-oxoquinoxalin-1(2H)-yl)propoxy)-3-methoxybenzaldehyde (**3al**)*. a white solid (66% yield, 71.3 mg). m. p. = 131-132 °C. (PET/EtOAc = 1:4 as the eluet). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.84 (s, 1H), 8.03 (d, *J* = 8.0 Hz, 1H), 7.94–7.89 (m, 4H), 7.59 –7.58 (m, 2H), 7.55–7.52 (m, 2H), 7.46–7.41 (m, 5H), 7.38–7.35 (m, 2H), 6.84 (d, *J* = 8.0 H z, 1H), 4.46 (t, *J* = 8.0 Hz, 2H), 4.11–4.09 (m, 2H), 3.86 (s, 3H), 2.34–2.28 (m, 2H); ^{13}C N MR (100 MHz, $CDCl_3$) δ: 190.9, 156.3, 155.1, 154.3, 154.0, 153.3, 149.7, 133.8, 133.6, 133. 2, 133.0, 132.3, 132.1(d, *J* = 4.2 Hz), 131.9, 131.1, 130.4, 130.0, 128.3(d, *J* = 12.1 Hz), 126. 6, 124.0, 113.9, 111.5, 109.1, 66.2, 55.8, 39.4, 27.0; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 23.9 (s); HRMS (ESI): m/z calcd for $C_{31}H_{27}N_2NaO_5P^+$ $[M+Na]^+$ 561.1550. Found 561.1551.

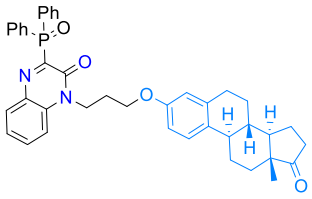


*2-(3-(diphenylphosphoryl)-2-oxoquinoxalin-1(2H)-yl)ethyl 2-acetoxybenzoate (**3am**)*. a white s olid (62% yield, 68.7 mg). m. p. = 130-131 °C. (PET/EtOAc = 1:4 as the eluet). ^{1}H NMR (4 00 MHz, $CDCl_3$) δ: 8.01 (d, *J* = 8.0 Hz, 1H), 7.95–7.90 (m, 4H), 7.84–7.74 (m, 1H), 7.65–7. 61 (m, 1H), 7.59–7.52 (m, 3H), 7.48–7.42 (m, 6H), 7.37–7.32 (m, 2H), 4.45–4.42 (m, 2H), 4.

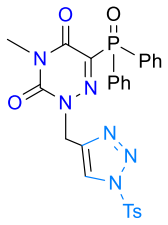
36–4.33 (m, 2H), 1.83 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 170.7, 156.3, 155.0, 154.1, 15 3.8, 133.6, 133.4, 133.0, 132.5, 132.3, 132.1(d, *J* = 4.4 Hz), 131.9, 131.6, 131.5(d, *J* = 2.5 H z), 131.4, 131.4, 131.3, 130.9, 129.8, 128.8, 128.6, 128.5, 128.3(d, *J* = 5.9 Hz), 128.2, 127.2, 126.8, 124.1, 113.7, 60.3, 40.8, 20.5; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 23.9 (s); HRMS (ESI): m/z calcd for $C_{31}H_{25}N_2NaO_6P^+$ $[M+NH_4]^+$ 570.1788. Found 570.1786.



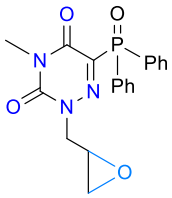
*2-(3-(diphenylphosphoryl)-2-oxoquinoxalin-1(2H)-yl)ethyl 2-(4-isobutylphenyl)propanoate (**3an**)*. a white solid (82% yield, 94.6 mg). m. p. = 145-147 °C. (PET/EtOAc = 1:4 as the eluet). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.01 (d, *J* = 8.0 Hz, 1H), 7.95–7.90 (m, 4H), 7.57–7.53 (m, 3 H), 7.48–7.45 (m, 3H), 7.38 (d, *J* = 8.0 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.20–7.06 (m, 2 H), 7.02 (s, 3H), 4.49–4.27 (m, 4H), 3.49–3.41 (m, 1H), 2.41 (d, *J* = 8.0 Hz, 2H), 1.87–1.78 (m, 1H), 1.34 (d, *J* = 4.0 Hz, 3H), 0.89 (s, 3H), 0.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 174.6, 156.3, 155.0, 154.0, 153.8, 140.7, 137.0, 133.6, 133.5(d, *J* = 1.5 Hz), 133.4, 133.0, 132.2, 132.1(d, *J* = 3.9 Hz), 132.0, 131.0(d, *J* = 4.4 Hz), 129.9(d, *J* = 4.3 Hz), 129.4, 129.3, 128.3(d, *J* = 13.0 Hz), 127.1, 126.9(d, *J* = 7.1 Hz), 124.0, 113.9, 60.8, 44.9, 44.8, 40.8, 30.1, 22.3, 18.2; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 23.7 (s); HRMS (ESI): m/z calcd for $C_{35}H_{35}N_2NaO_4P^+$ $[M+Na]^+$ 601.2227. Found 601.2221.



*3-(diphenylphosphoryl)-1-(3-(((8S,9R,13R,14R)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-dec ahydro-6H-cyclopenta[a]phenanthren-3-yl)oxy)propyl)quinoxalin-2(1H)-one (**3ao**)*. a white solid (4 7% yield, 61.2 mg). m. p. = 156-157 °C. (PET/EtOAc = 1:4 as the eluet). ^{1}H NMR (400 MH z, $CDCl_3$) δ: 8.03 (d, *J* = 8.0 Hz, 1H), 7.96–7.91 (m, 4H), 7.62 (t, *J* = 8.0 Hz, 1H), 7.56–7.5 3 (m, 2H), 7.50–7.45 (m, 5H), 7.36 (t, *J* = 8.0 Hz, 1H), 7.18 (d, *J* = 8.0 Hz, 1H), 6.66 (d, *J* = 8.0 Hz, 1H), 6.59 (s, 1H), 4.39 (t, *J* = 8.0 Hz, 2H), 3.99 (t, *J* = 8.0 Hz, 2H), 2.94–2.82 (m, 2H), 2.50 (dd, *J* = 16.0 Hz, *J* = 8.0 Hz, 1H), 2.40–2.34 (m, 1H), 2.27–2.14 (m, 4H), 2.1 1–2.02 (m, 2H), 2.00–1.94 (m, 2H), 1.67–1.59 (m, 1H), 1.54–1.53 (m, 1H), 1.50–1.38 (m, 3H), 0.91 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 220.8, 156.4, 155.2, 154.1, 153.9, 137.8, 133. 7, 133.5, 133.3(d, *J* = 2.0 Hz), 133.1, 132.5, 132.3, 132.0(d, *J* = 10.1 Hz), 131.2, 130.1, 128. 3(d, *J* = 13.0 Hz), 126.4, 123.9, 114.5, 113.9, 112.1, 65.1, 50.4, 48.0, 43.9, 39.7, 38.3, 35.8, 3 1.5, 29.6, 27.1, 26.5, 25.9, 21.5, 13.8; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 23.8 (s); HRMS (ESI): m/z calcd for $C_{41}H_{41}N_2NaO_4P^+$ $[M+Na]^+$ 679.2696. Found 679.2697.



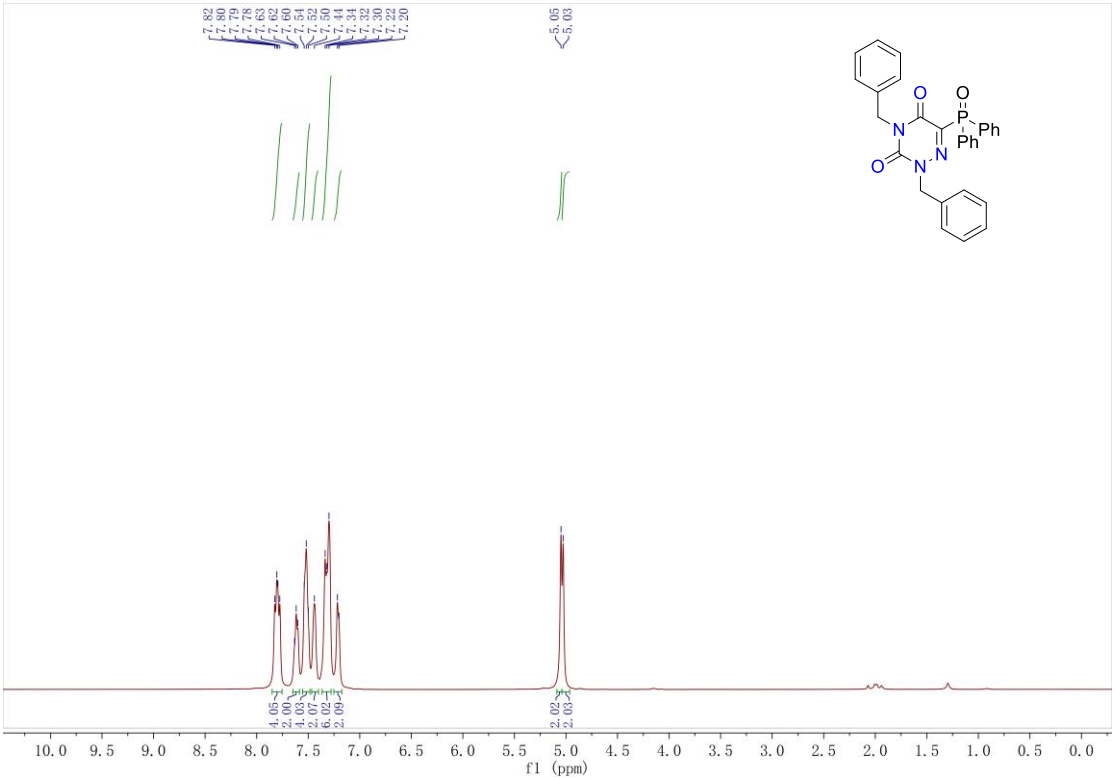
6-(diphenylphosphoryl)-4-methyl-2-((1-tosyl-1H-1,2,3-triazol-4-yl)methyl)-1,2,4-triazine-3,5(2H,4H)-dione ***(4)***. a white solid (84% yield, 93.9 mg). m. p. = 179-180 °C. (PET/EtOAc = 1:5 as the eluet).^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.10 (s, 1H), 7.95 (d, *J* = 8.0 Hz, 2H), 7.84–7.79 (m, 4H), 7.59–7.56 (m, 2H), 7.50–7.46 (m, 4H), 7.37 (d, *J* = 8.0 Hz, 2H), 5.14 (s, 2H), 3.66 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.8, 154.6, 147.9, 147.5, 140.7, 140.4, 139.1, 132.7, 132.6(d, *J* = 1.9 Hz), 131.9(d, *J* = 10.4 Hz), 130.4, 130.1, 129.0, 128.8, 128.5(d, *J* = 13.6 Hz), 123.5, 40.6, 35.2, 21.8; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 21.6 (s); HRMS (ESI): m/z calcd for $C_{26}H_{23}N_6NaO_5PS^+$ $[M+Na]^+$ 585.1080. Found 585.1078.



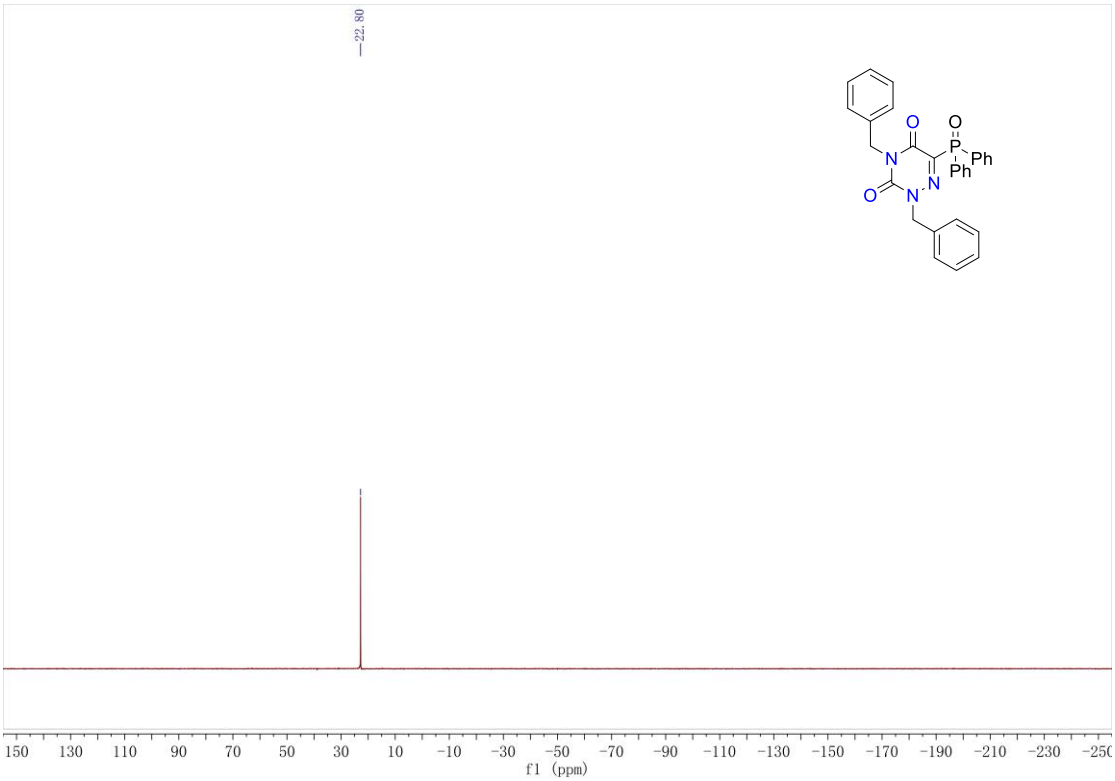
6-(diphenylphosphoryl)-4-methyl-2-(oxiran-2-ylmethyl)-1,2,4-triazine-3,5(2H,4H)-dione ***(5)***. a white solid (41% yield, 31.6 mg). m. p. = 104-106 °C. (PET/EtOAc = 1:5 as the eluet). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.88–7.83 (m, 4H), 7.59–7.57 (m, 2H), 7.51–7.47 (m, 4H), 4.16–4.12 (m, 1H), 3.99–3.94 (m, 1H), 3.69 (s, 3H), 3.59–3.58 (m, 1H), 2.75–2.73 (m, 1H), 2.62–2.61 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.2, 155.0, 148.4, 140.2, 139.0, 132.6(d, *J* = 3.0 Hz), 132.0(d, *J* = 1.7 Hz), 131.9(d, *J* = 1.7 Hz), 130.0, 129.7, 129.7, 128.9, 128.6(d, *J* = 11.8 Hz), 48.0, 46.3, 42.5, 40.6; ^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.0 (s); HRMS (ESI): m/z calcd for $C_{19}H_{18}N_3NaO_4P^+$ $[M+Na]^+$ 406.0927. Found 406.0923.

VII. NMR charts of 3a–5

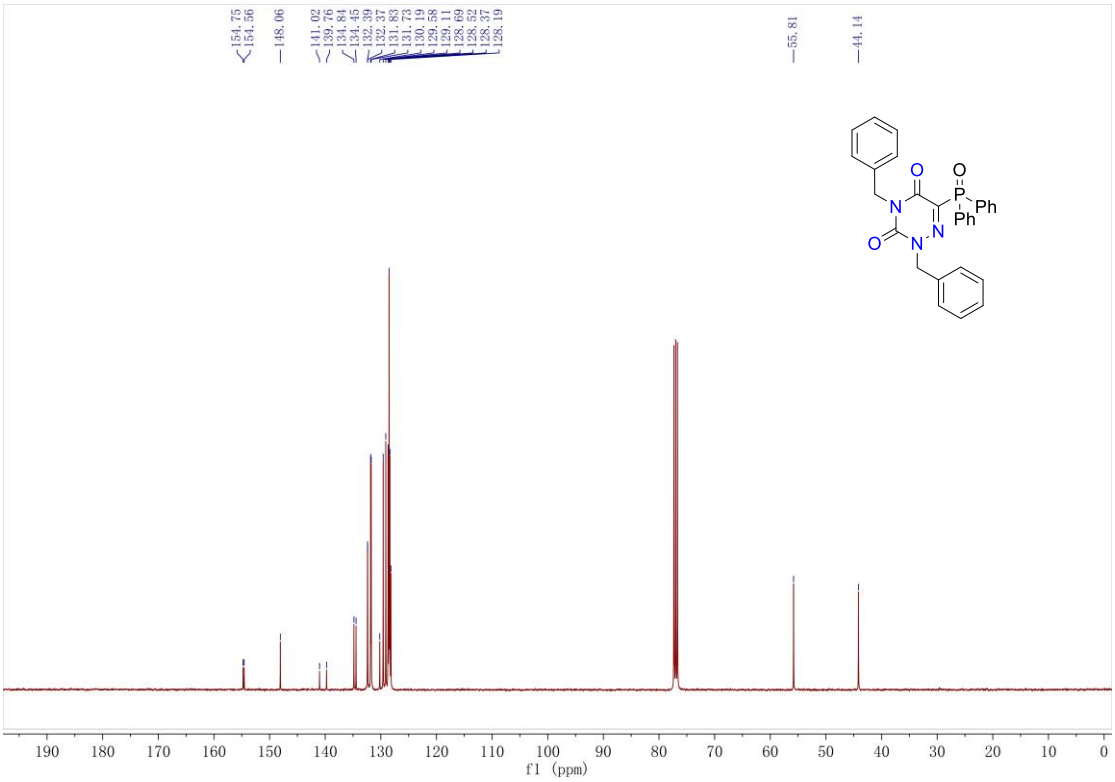
3a-^{1}H



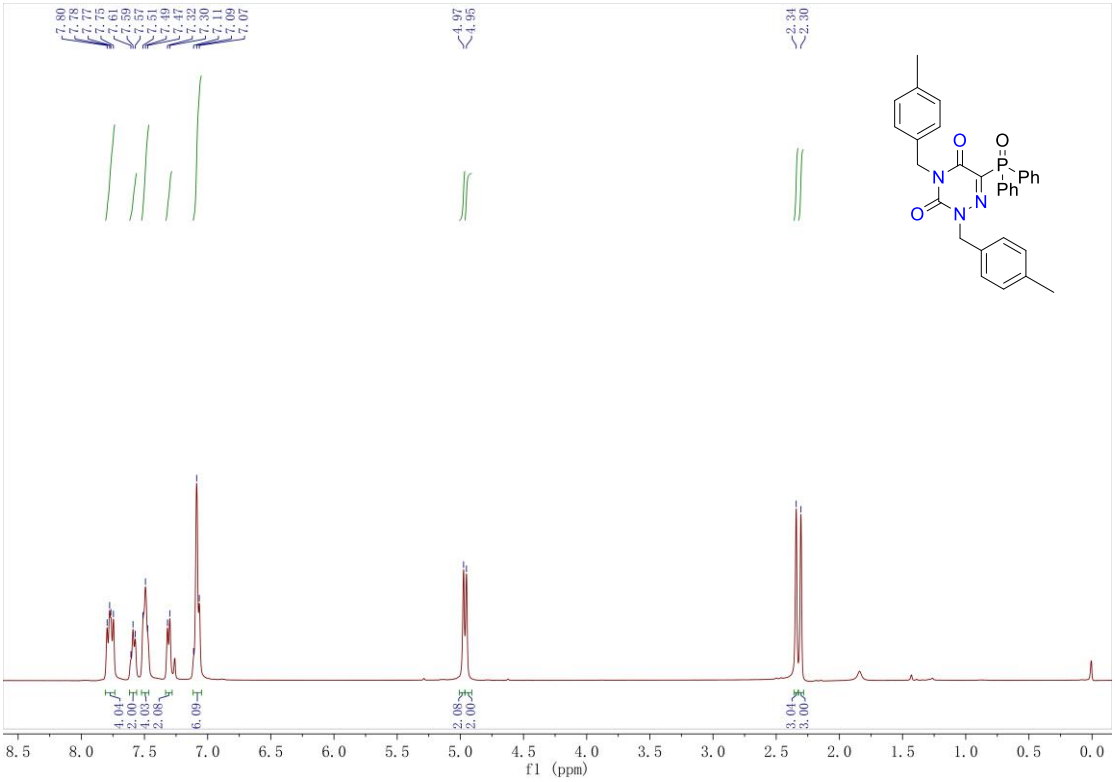
3a-^{31}P



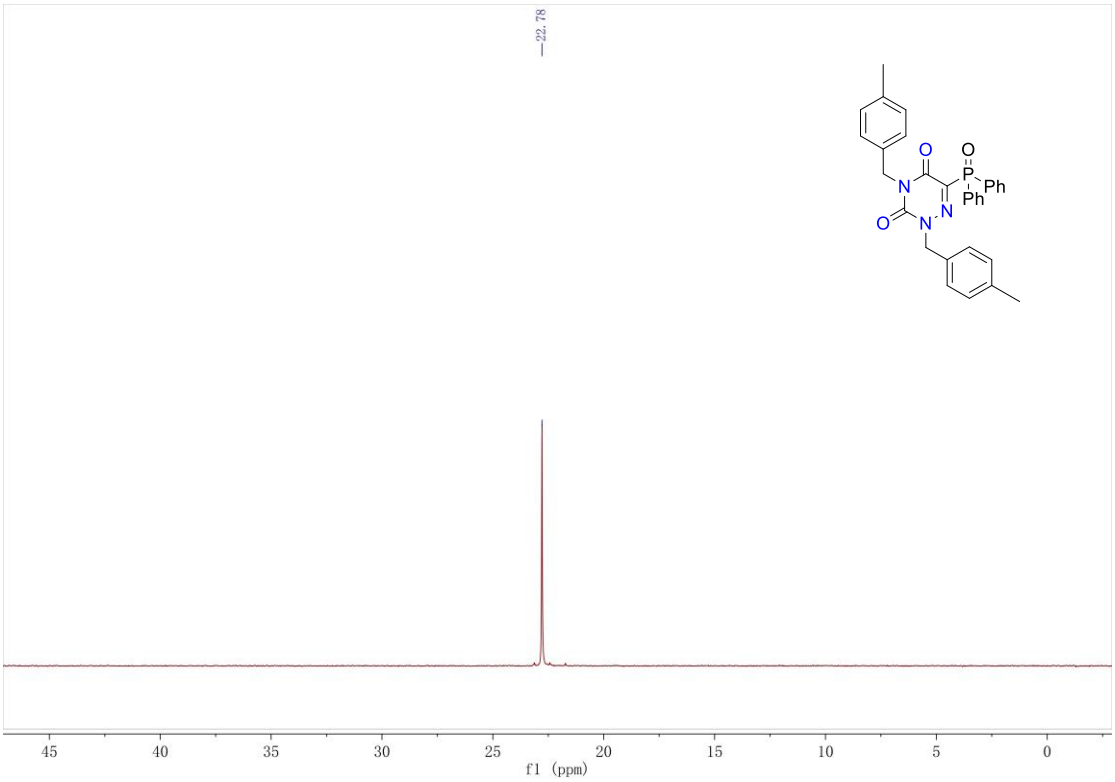
3a-^{13}C


154.75
154.56
148.06
141.02
139.76
134.84
134.45
132.39
132.37
131.83
131.73
130.19
129.58
129.11
128.69
128.52
128.37
128.19
55.81
44.14
O
O
P
Ph
Ph
N
N
N
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

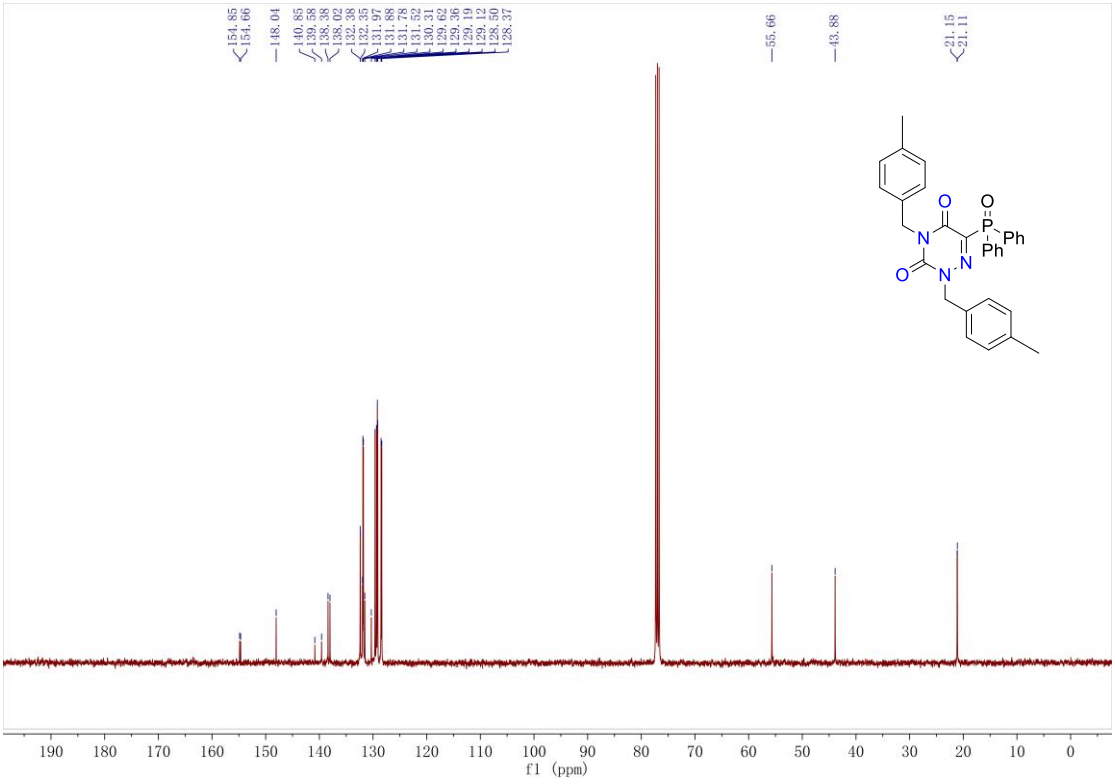
3b-^{1}H


7.80
7.78
7.77
7.75
7.61
7.59
7.57
7.51
7.49
7.47
7.32
7.30
7.11
7.09
7.07
4.97
4.95
2.34
2.30
O
O
P
Ph
Ph
N
N
N
O
4.04
2.00
4.03
2.08
6.09
2.08
2.00
3.04
3.00
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)

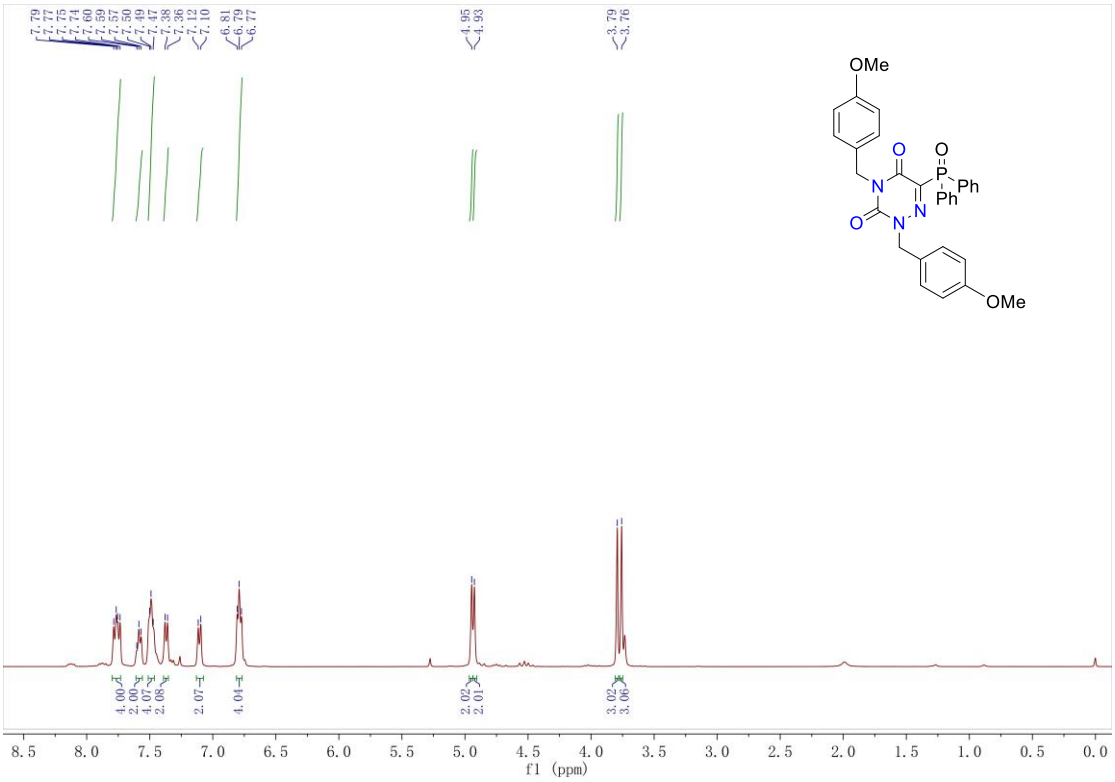
3b-^{31}P


22.78
O
O
P
Ph
Ph
N
N
O
N
45
40
35
30
25
20
15
10
5
0
f1 (ppm)

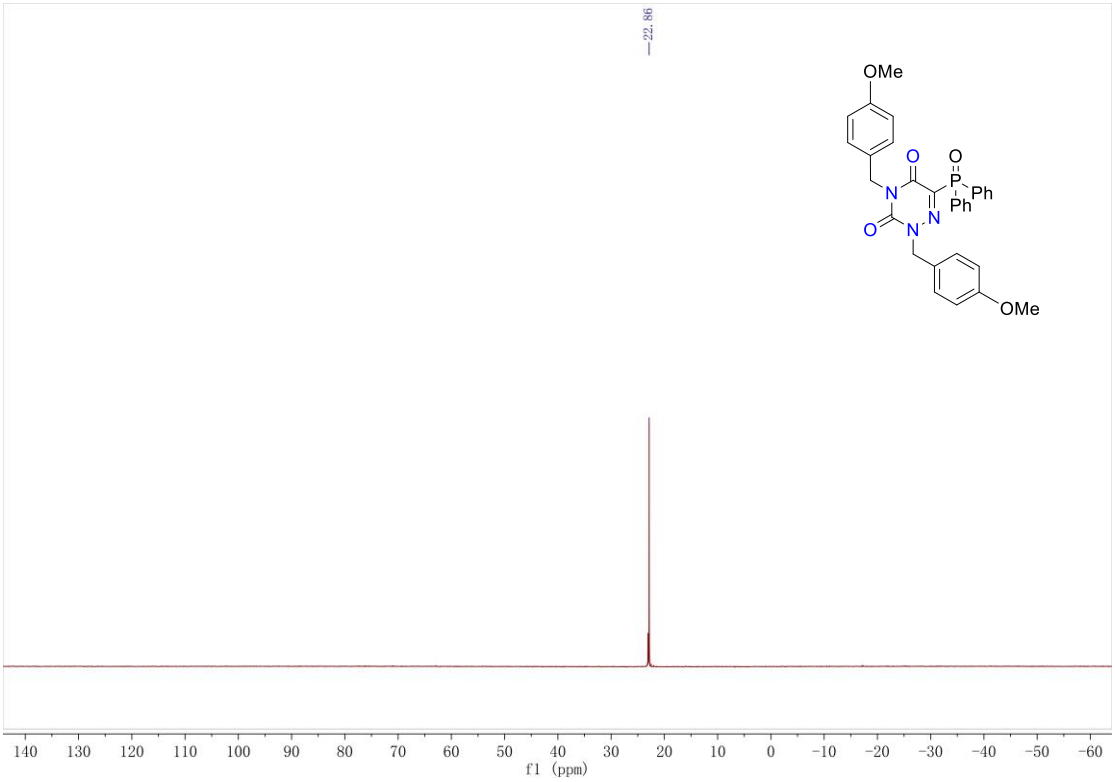
3b-^{13}C


154.85
154.66
148.04
140.85
139.58
138.38
138.02
132.38
132.35
131.97
131.88
131.78
131.52
130.31
129.62
129.36
129.19
129.12
128.90
128.37
55.66
43.88
21.15
21.11
O
O
P
Ph
Ph
N
N
O
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

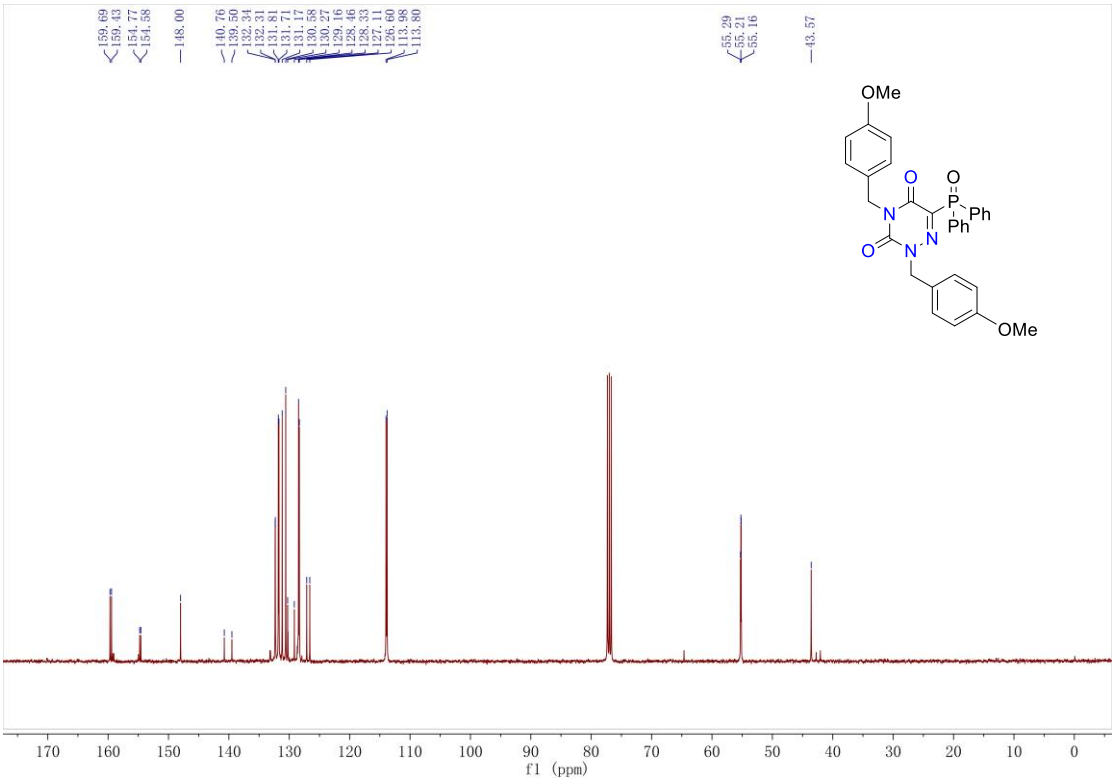
3c-^{1}H


7.79
7.77
7.75
7.74
7.60
7.59
7.57
7.56
7.49
7.47
7.38
7.36
7.12
7.10
6.81
6.79
6.77
4.95
4.93
3.79
3.76
OMe
O
P
Ph
Ph
N
O
N
N
OMe
4.00
2.00
4.07
2.08
2.07
4.04
2.02
2.01
3.02
3.06
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)

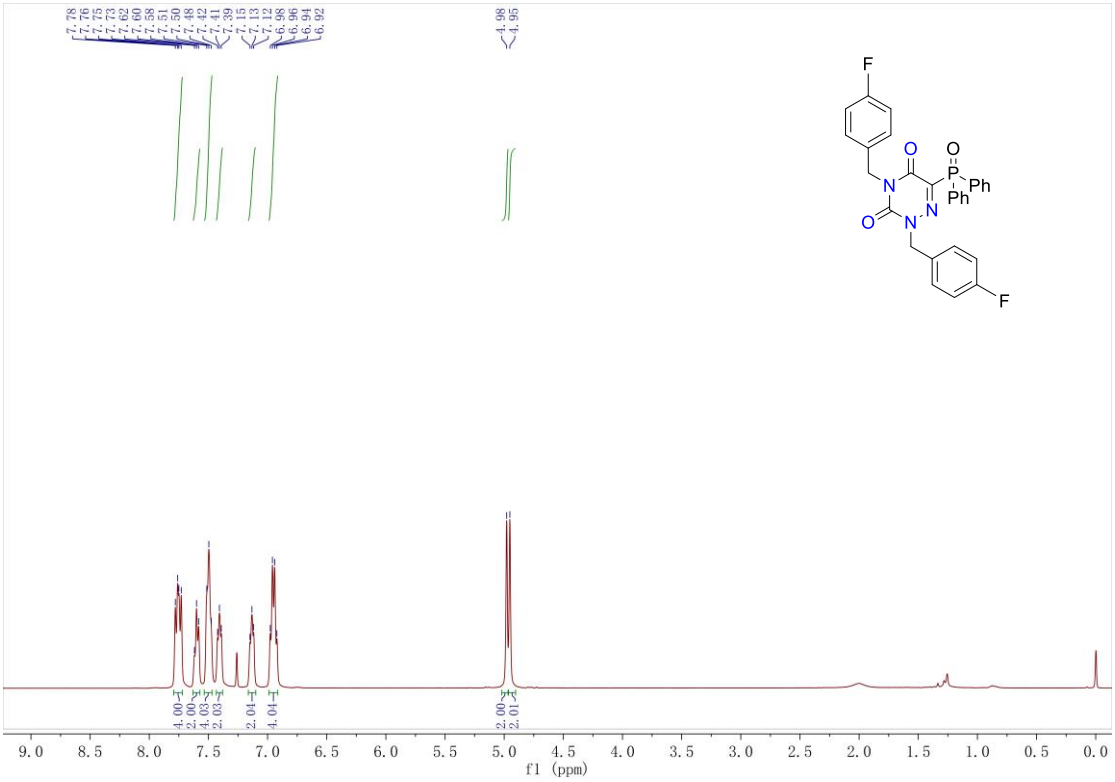
3c-^{31}P


22.86
OMe
O
P
Ph
Ph
N
O
N
N
OMe
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
−20
−30
−40
−50
−60
f1 (ppm)

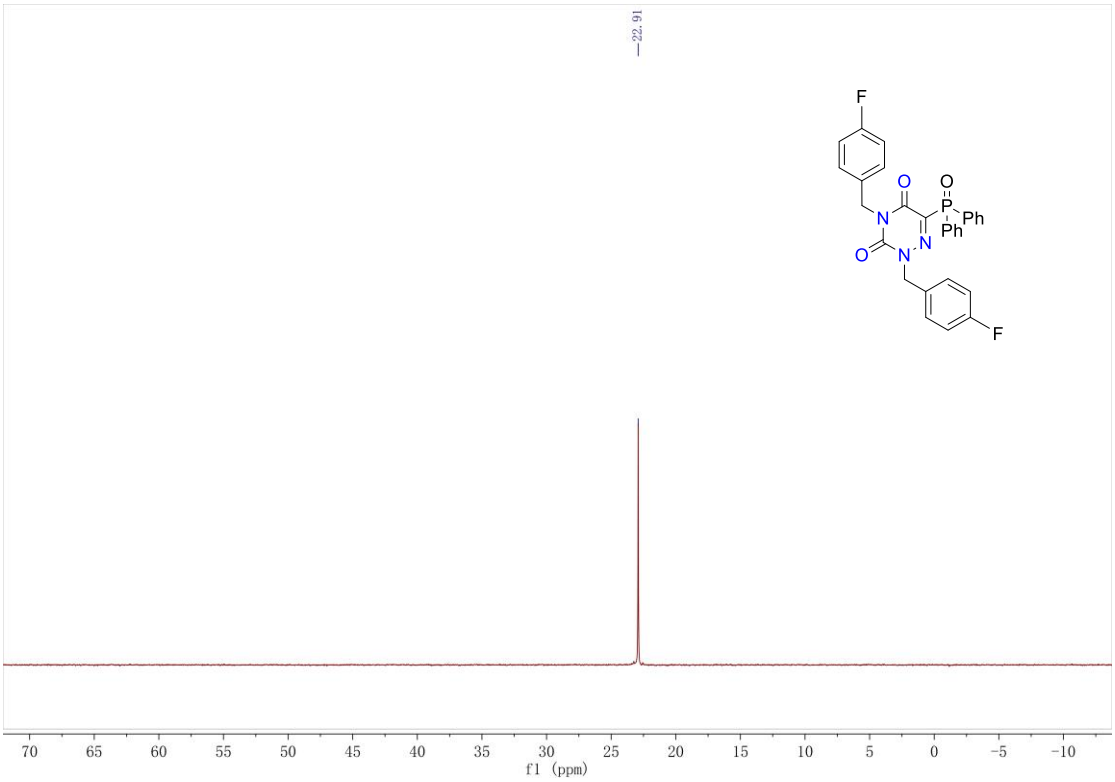
3c-^{13}C


159.69
159.43
154.77
154.58
148.00
140.76
139.50
132.34
132.31
131.81
131.71
131.17
130.58
130.27
129.16
129.06
128.33
127.11
126.60
113.98
113.80
55.29
55.21
55.16
43.57
OMe
OMe
f1 (ppm)

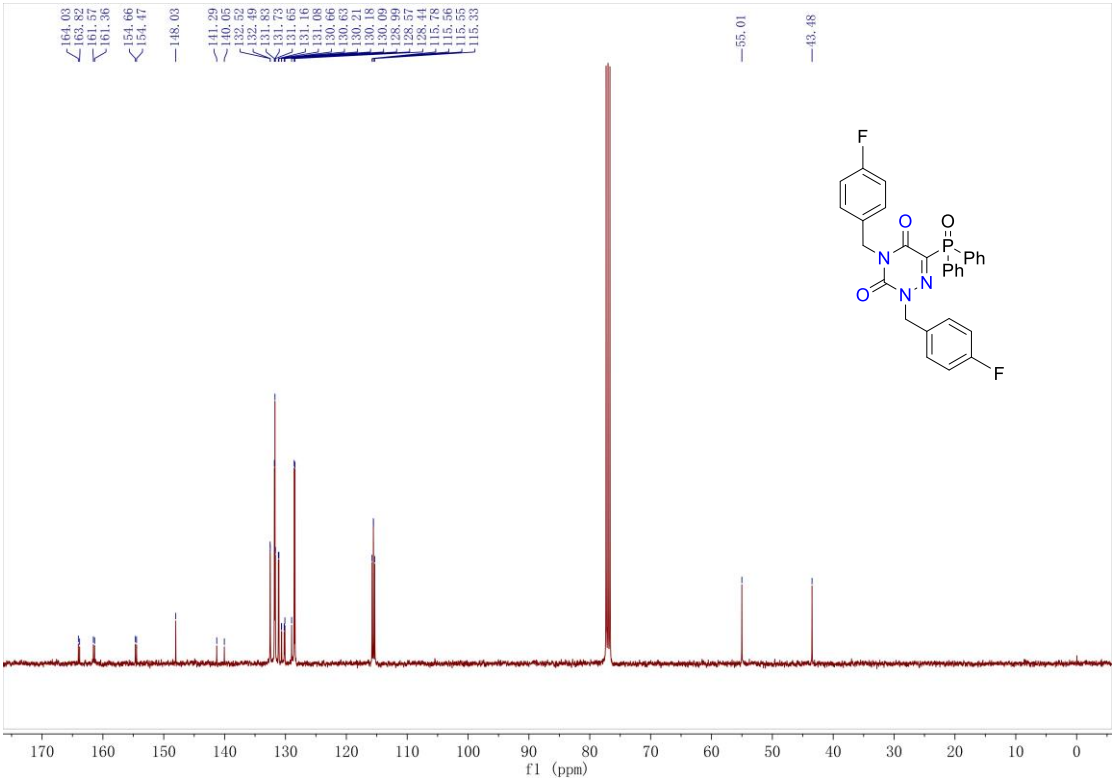
3d-^{1}H


7.78
7.76
7.75
7.73
7.62
7.60
7.58
7.51
7.50
7.48
7.42
7.41
7.39
7.15
7.13
7.12
6.98
6.96
6.94
6.92
4.98
4.95
F
F
4.00
2.00
4.03
2.03
2.04
4.04
2.00
2.01
f1 (ppm)

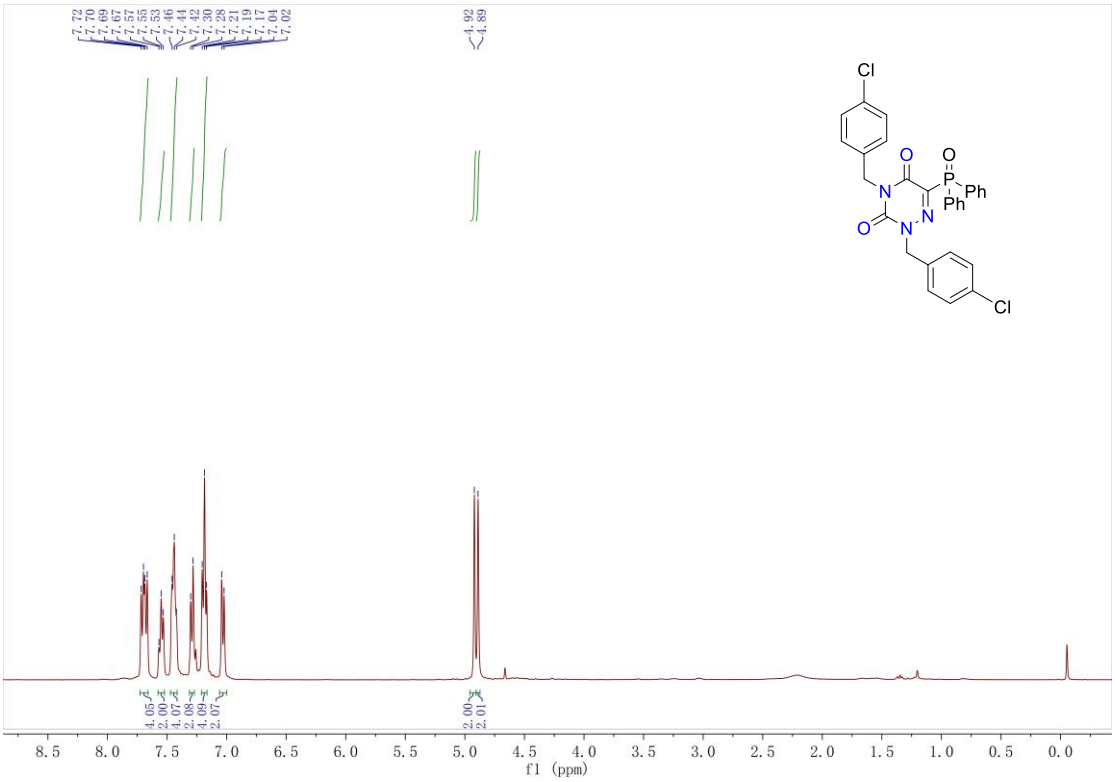
3d-^{31}P


22.91
F
O
O
P
Ph
Ph
N
N
O
N
F
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10
f1 (ppm)

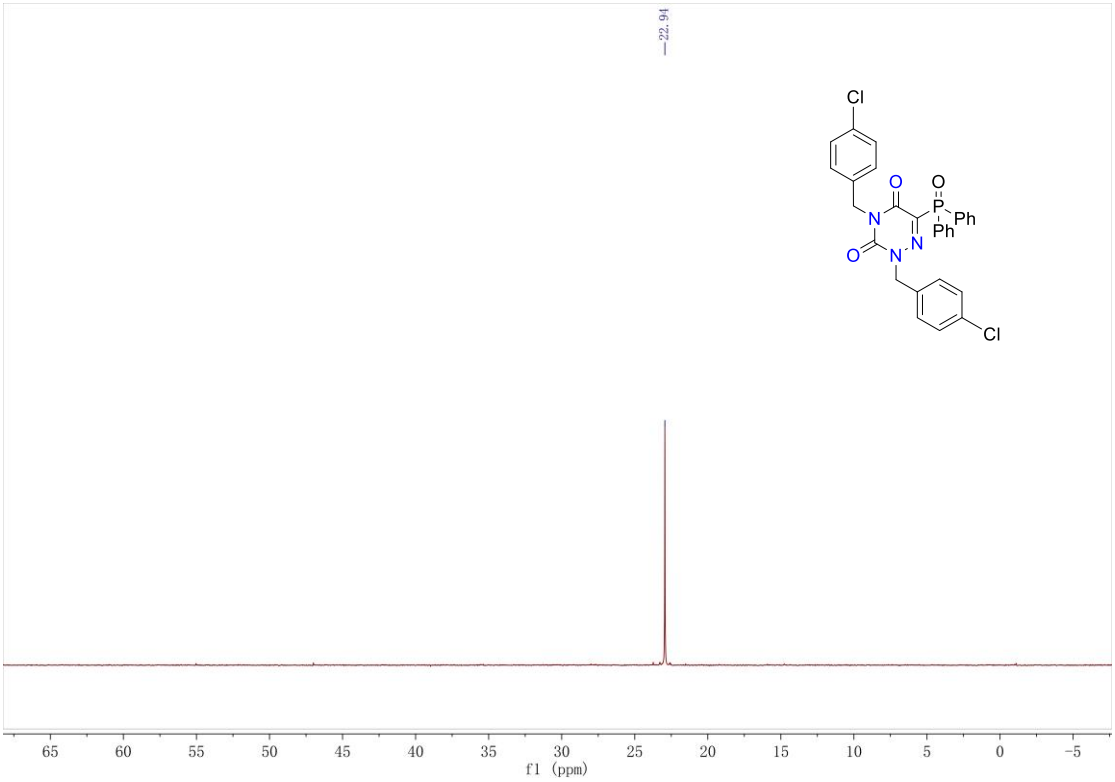
3d-^{13}C


164.03
163.82
161.57
161.36
154.66
154.47
148.03
141.29
140.65
132.52
132.49
131.83
131.73
131.65
131.16
131.08
130.66
130.63
130.21
130.18
130.09
128.99
128.57
128.44
128.18
115.56
115.55
115.33
55.01
43.48
F
O
O
P
Ph
Ph
N
N
O
N
F
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

3e-^{1}H


7.72
7.70
7.69
7.67
7.57
7.55
7.53
7.46
7.44
7.42
7.30
7.28
7.21
7.19
7.17
7.04
7.02
4.92
4.89
Cl
O
O
P
Ph
Ph
N
N
O
N
Cl
4.05
2.00
4.07
2.08
4.09
2.07
2.00
2.01
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)

3e-^{31}P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Cl
O
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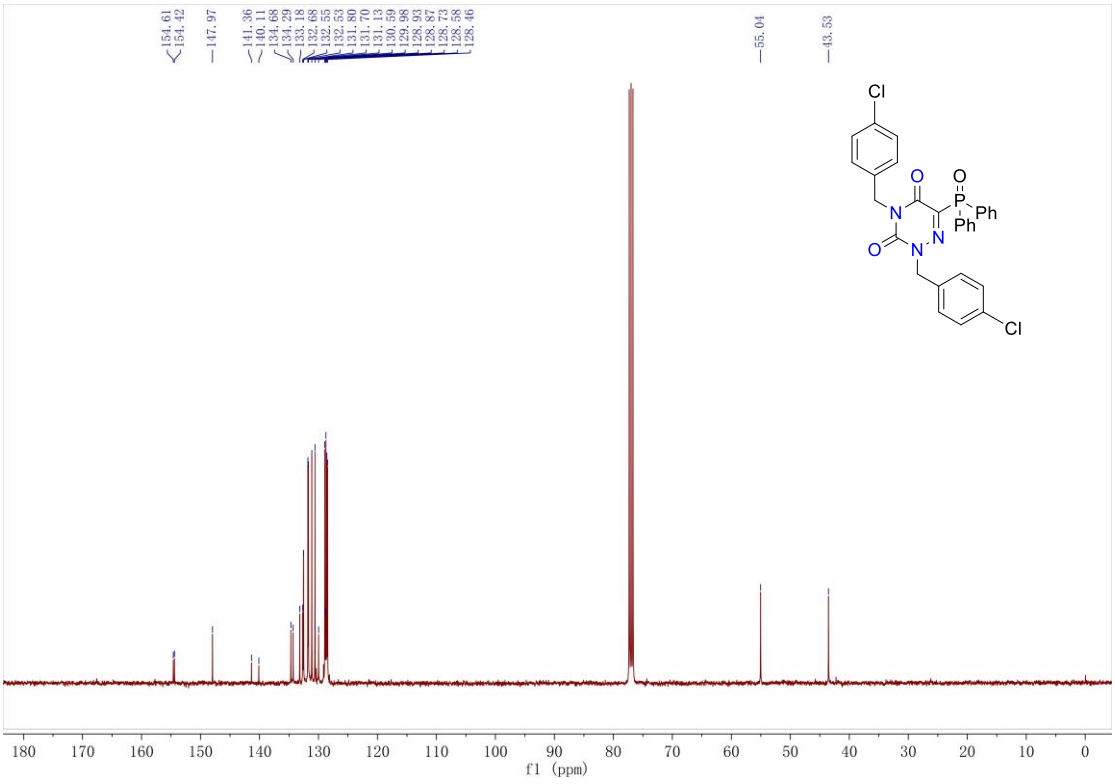
Ph
Ph
N
N
O
N
Cl
65
60
55
50
45
40
35
30
25
20
15
10
5
0
−5
f1 (ppm)

3e-^{13}C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154.42
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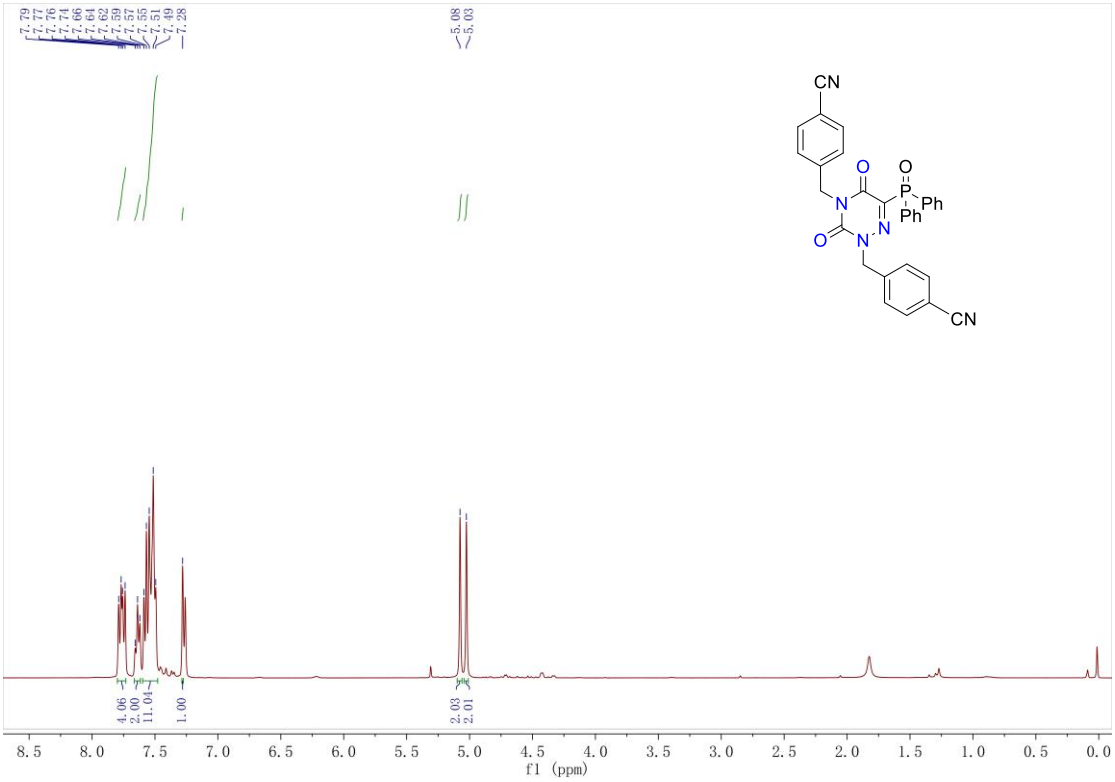
141.36
140.11
134.68
134.29
133.18
132.68
132.55
132.53
131.80
131.70
131.13
130.59
129.98
128.93
128.87
128.73
128.58
128.46
55.04
43.53
Cl
O
O
P
Ph
Ph
N
N
O
N
Cl
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

3f-^{1}H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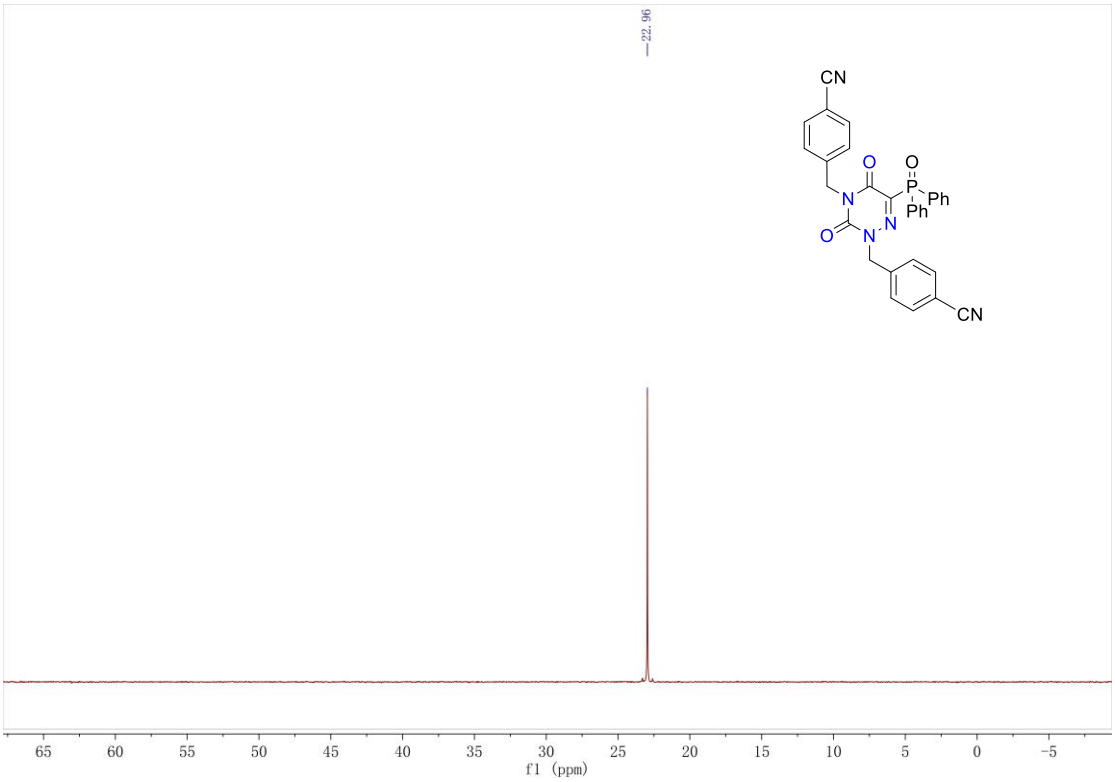
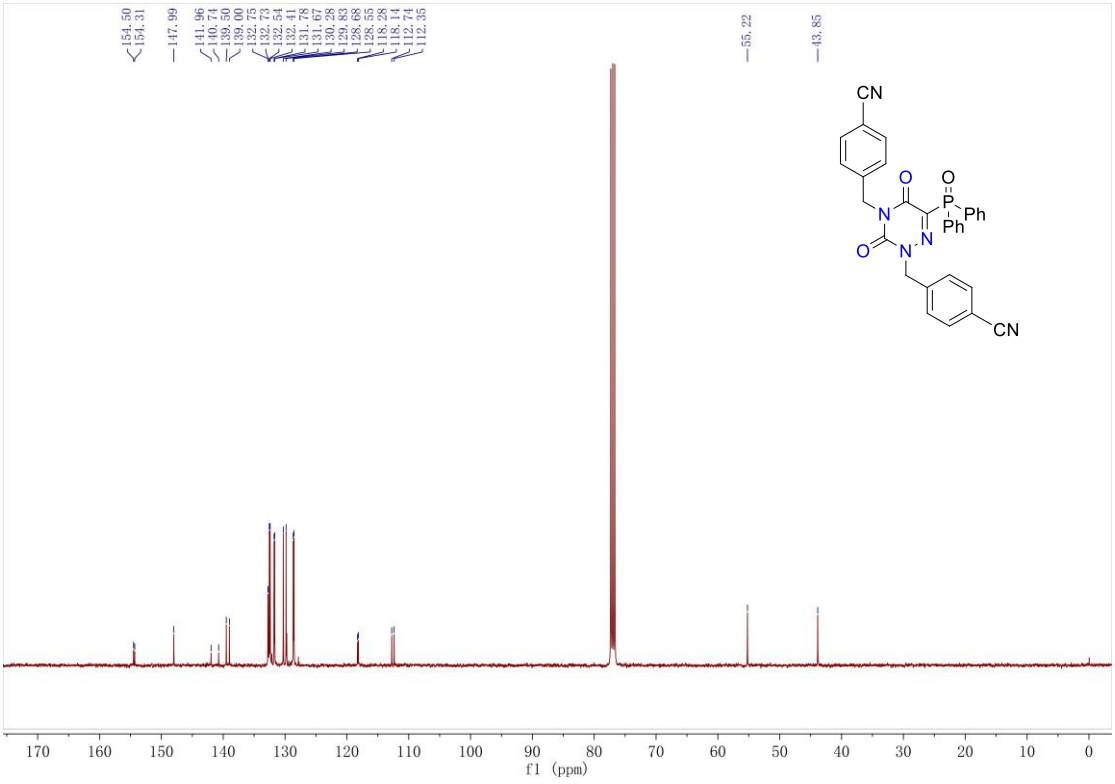
7.77
7.76
7.74
7.66
7.64
7.62
7.59
7.57
7.55
7.51
7.49
7.28
5.08
5.03
CN
O
O
P
Ph
Ph
N
N
O
N
CN
4.06
2.00
11.04
1.00
2.03
2.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

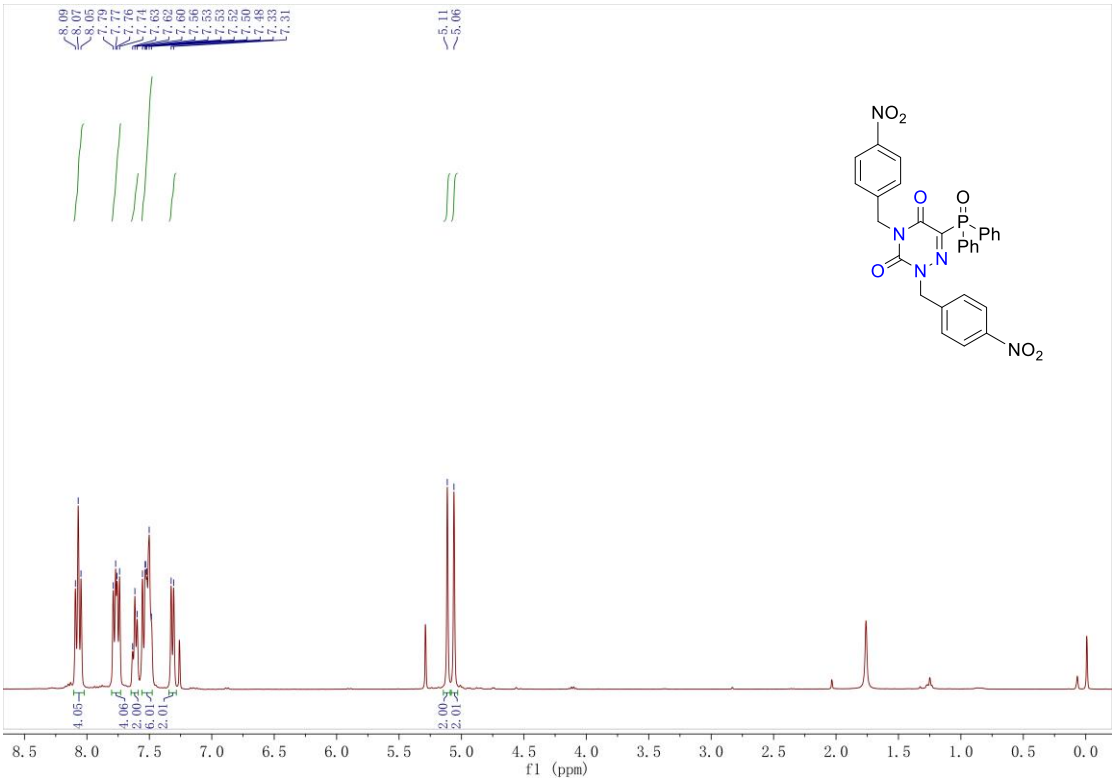
3f-^{31}P


22.96
CN
O
O
P
Ph
Ph
N
N
O
N
CN
65
60
55
50
45
40
35
30
25
20
15
10
5
0
−5
f1 (ppm)

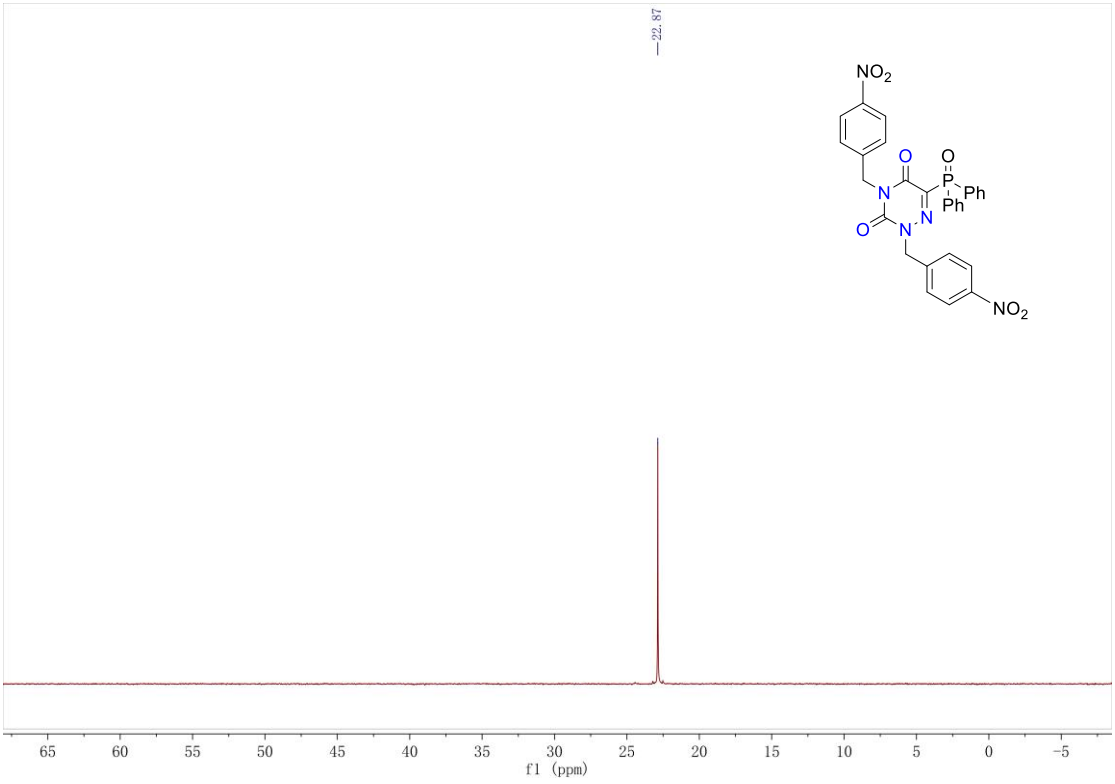
3f-^{13}C


154.50
154.31
147.99
141.96
140.74
139.63
139.00
132.75
132.73
132.54
132.41
131.78
131.67
130.28
129.83
128.68
128.55
118.28
118.14
112.74
112.35
55.22
43.85
CN
O
O
P
Ph
Ph
N
N
O
N
CN
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

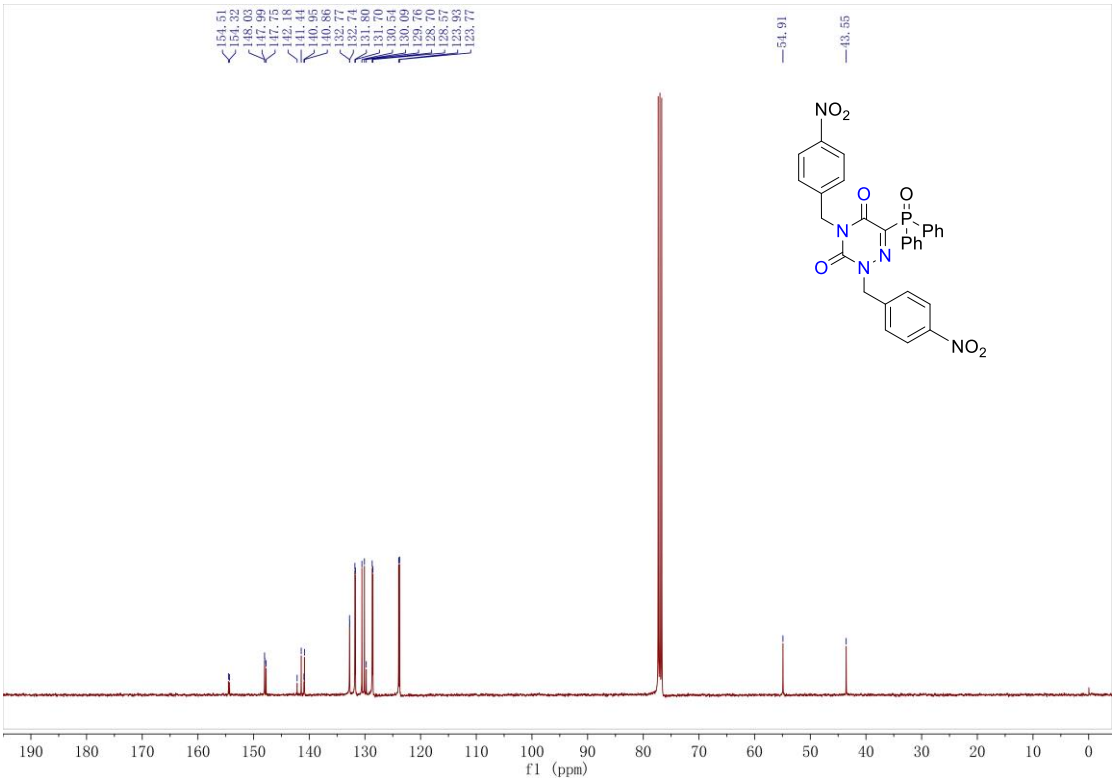
3g-^{1}H


8.09
8.07
8.05
7.79
7.77
7.76
7.74
7.63
7.62
7.60
7.56
7.53
7.52
7.50
7.48
7.33
7.31
5.11
5.06
NO2
O
O
P
Ph
Ph
N
N
O
N
NO2
4.05
4.06
2.00
6.01
2.01
2.00
2.01
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)

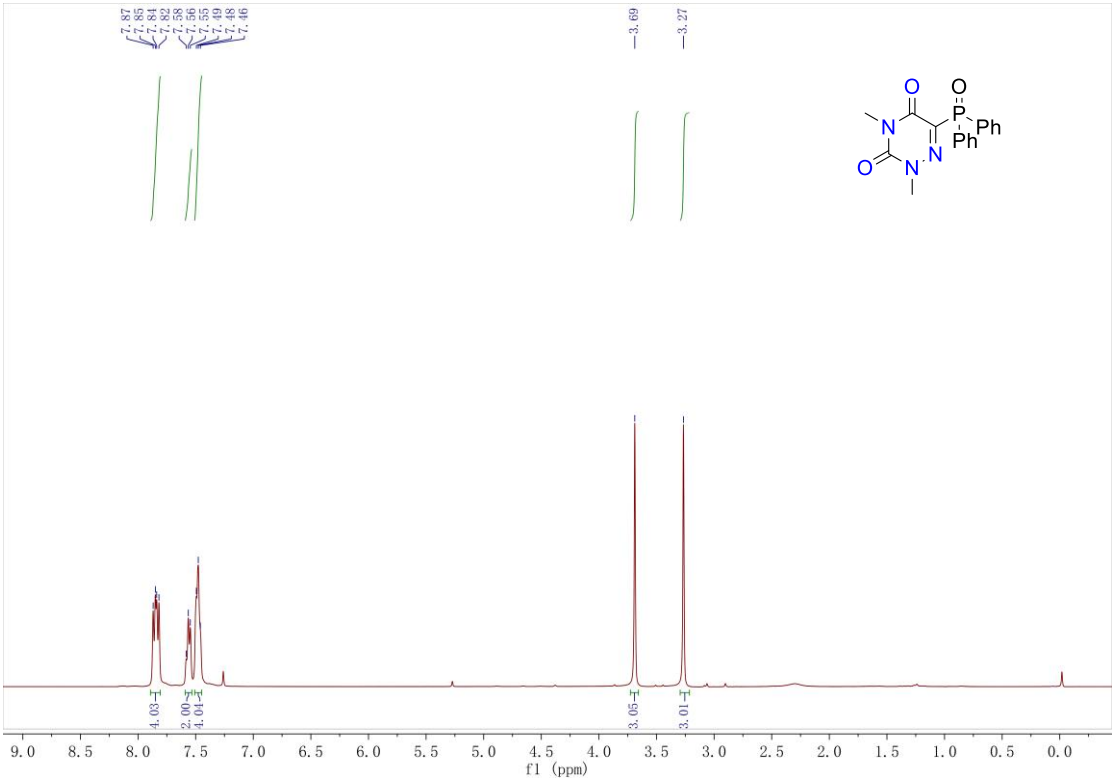
3g-^{31}P


22.87
NO2
O
O
P
Ph
Ph
N
N
O
N
NO2
65
60
55
50
45
40
35
30
25
20
15
10
5
0
−5
f1 (ppm)

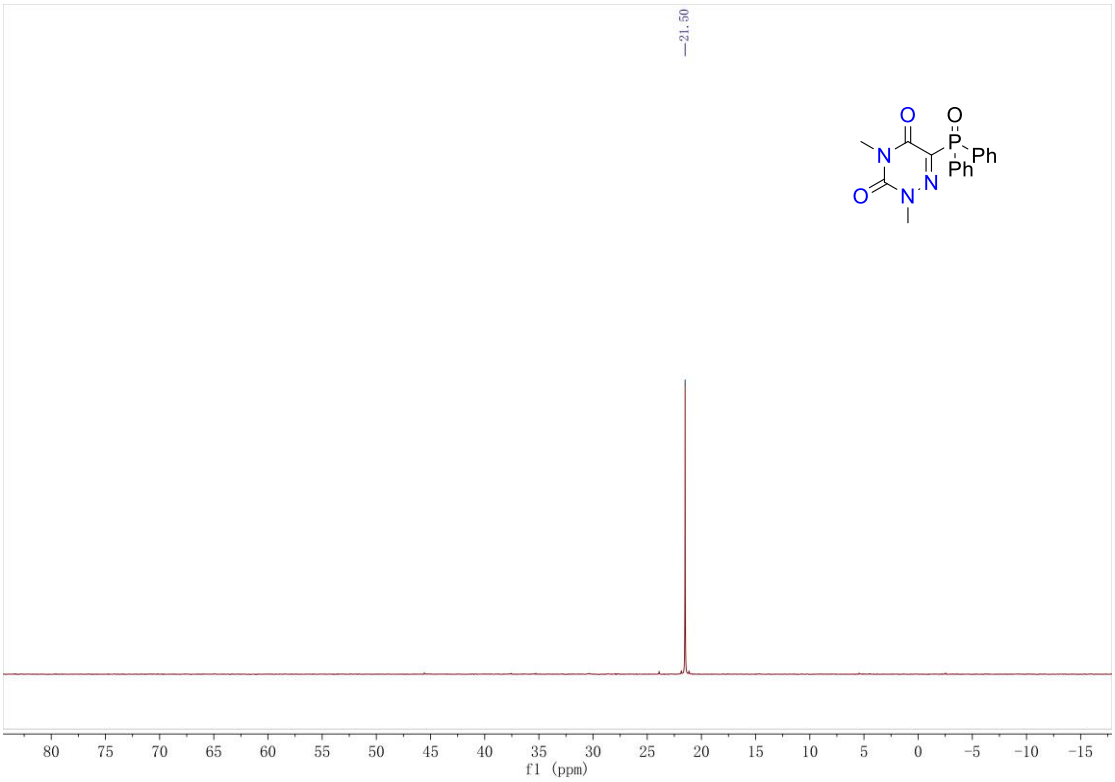
3g-^{13}C


154.51
154.32
148.03
147.99
147.75
142.18
141.44
140.95
140.86
132.77
132.74
131.80
131.70
130.54
130.09
129.76
128.70
128.57
123.93
123.77
54.91
43.55
NO2
O
O
P
Ph
Ph
N
N
O
N
N
NO2
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

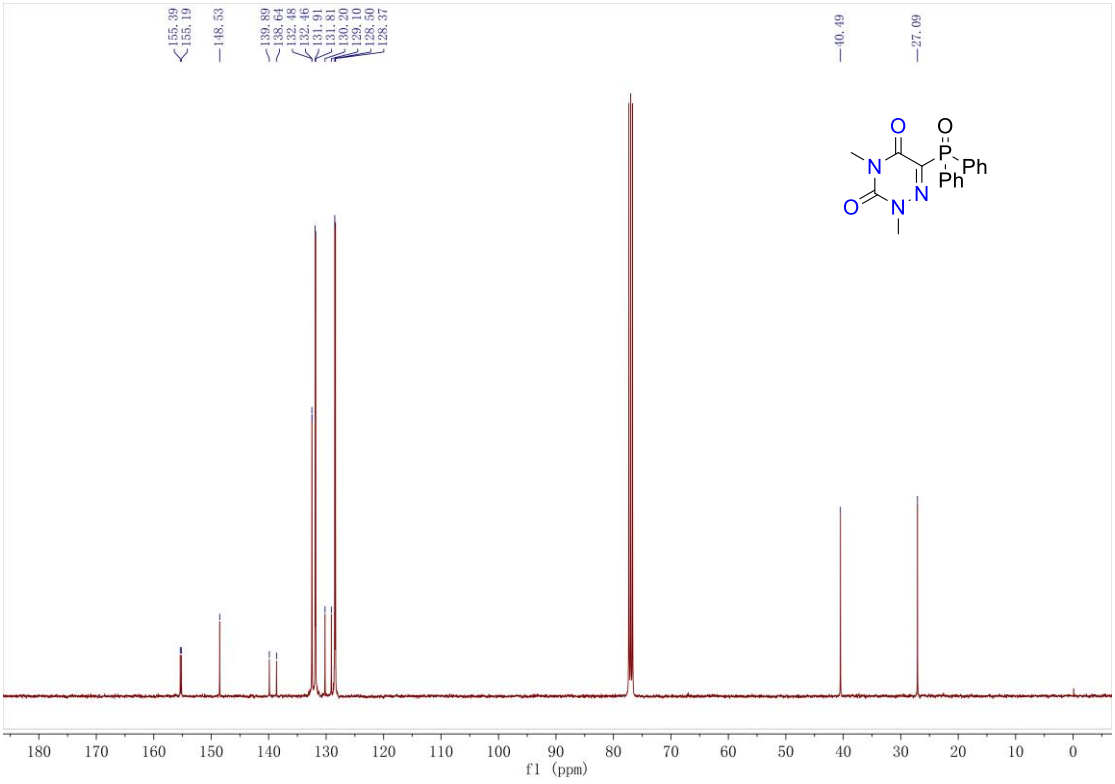
3h-^{1}H


7.87
7.85
7.84
7.82
7.58
7.56
7.55
7.49
7.48
7.46
3.69
3.27
O
O
N
P
Ph
Ph
N
O
N
4.03
2.00
4.04
3.05
3.01
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)

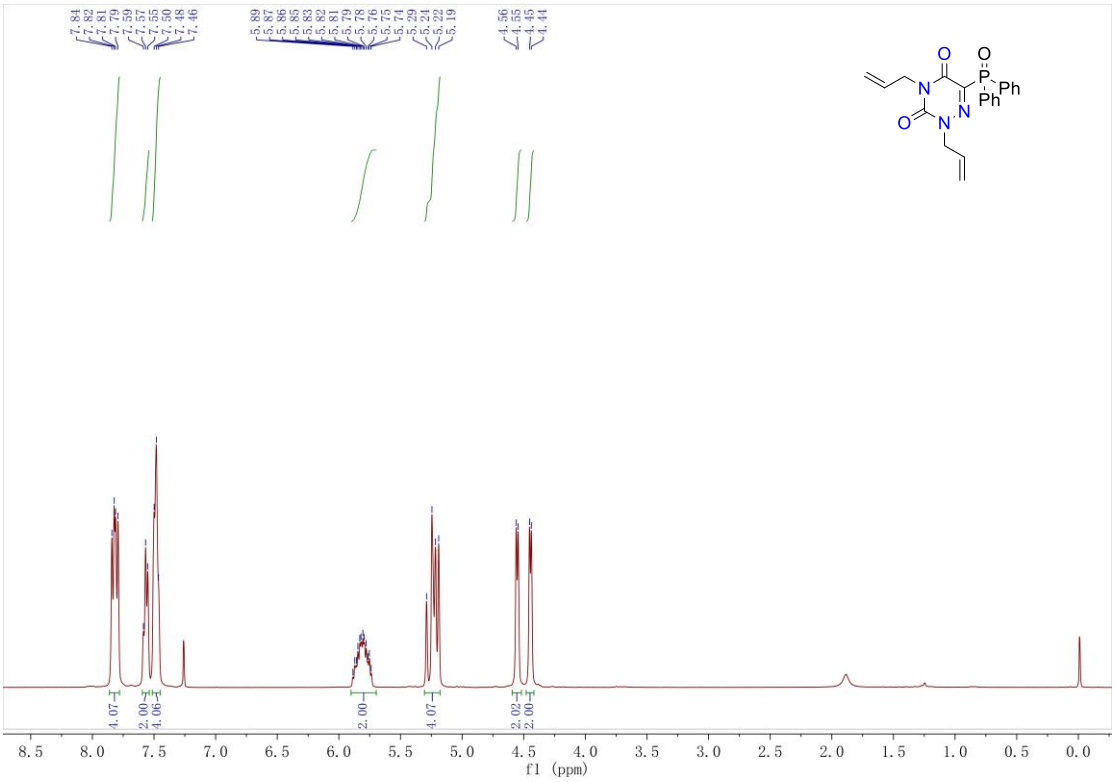
3h-^{31}P


21.50
80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5 -10 -15
f1 (ppm)

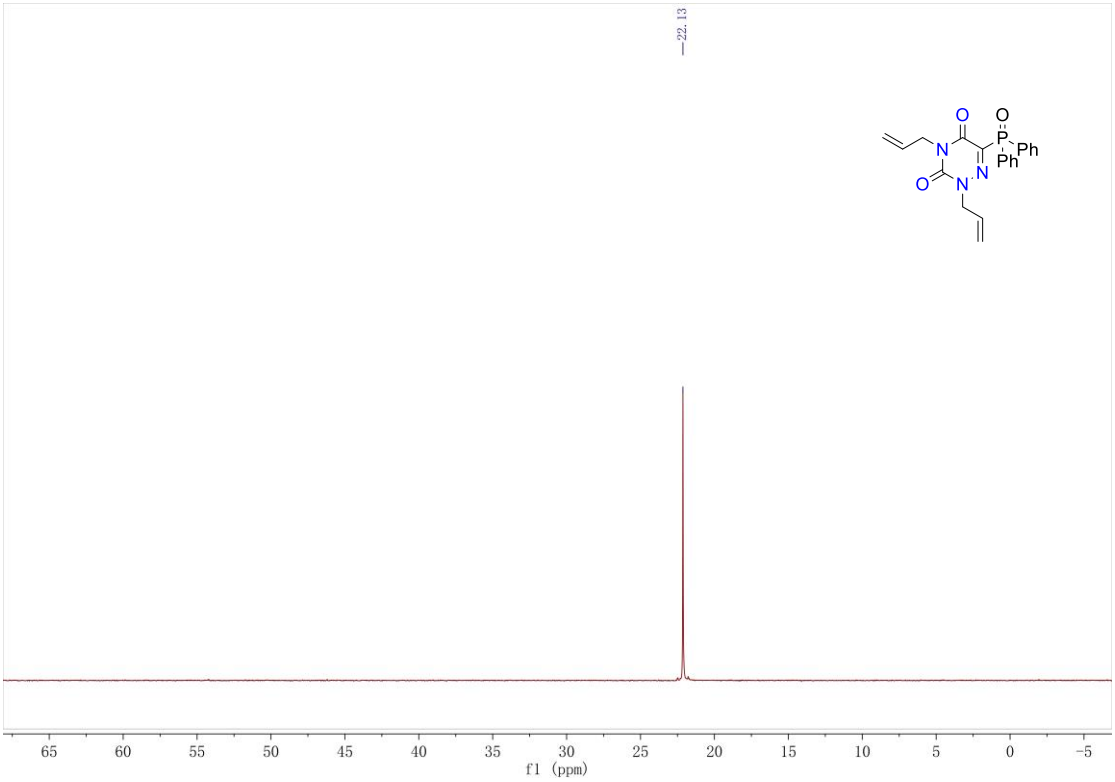
3h-^{13}C


155.39
155.19
148.53
139.89
138.64
132.48
132.46
131.91
131.81
130.20
129.10
128.50
128.37
40.49
27.09
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

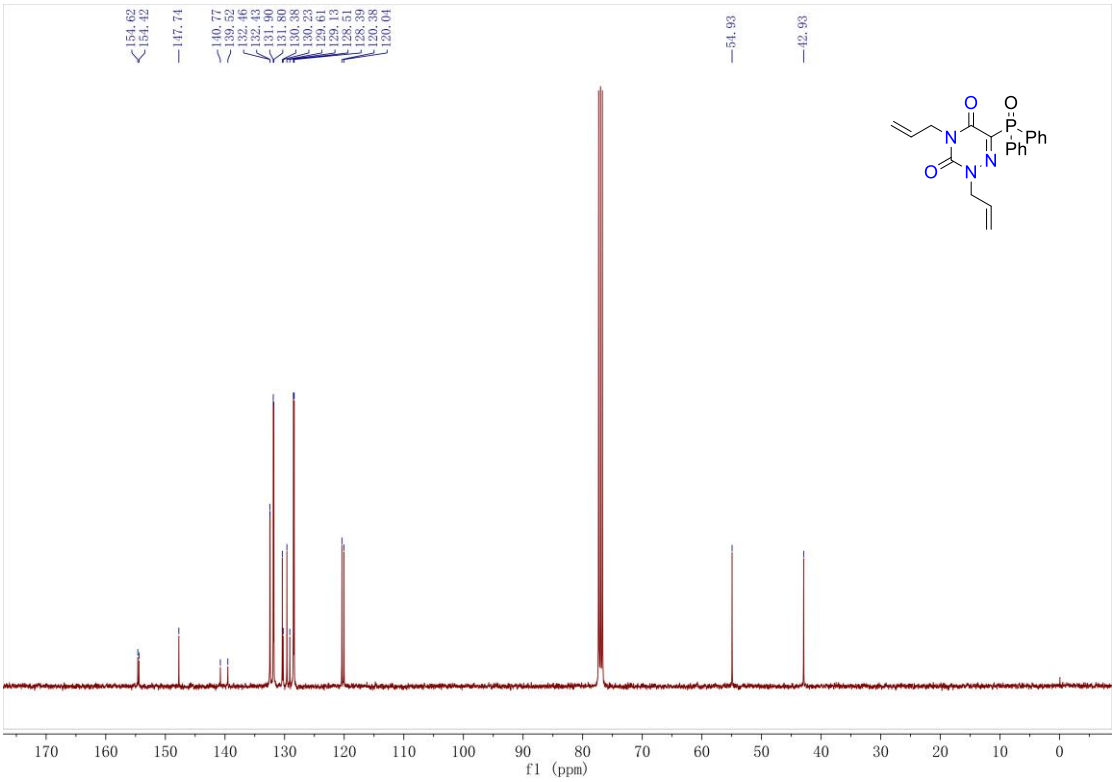
3i-^{1}H


7.84
7.82
7.81
7.79
7.59
7.57
7.55
7.50
7.48
7.46
5.89
5.87
5.86
5.85
5.83
5.82
5.81
5.79
5.78
5.76
5.75
5.74
5.29
5.24
5.22
5.19
4.56
4.55
4.45
4.44
4.07
2.00
4.06
2.00
4.07
2.02
2.00
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)

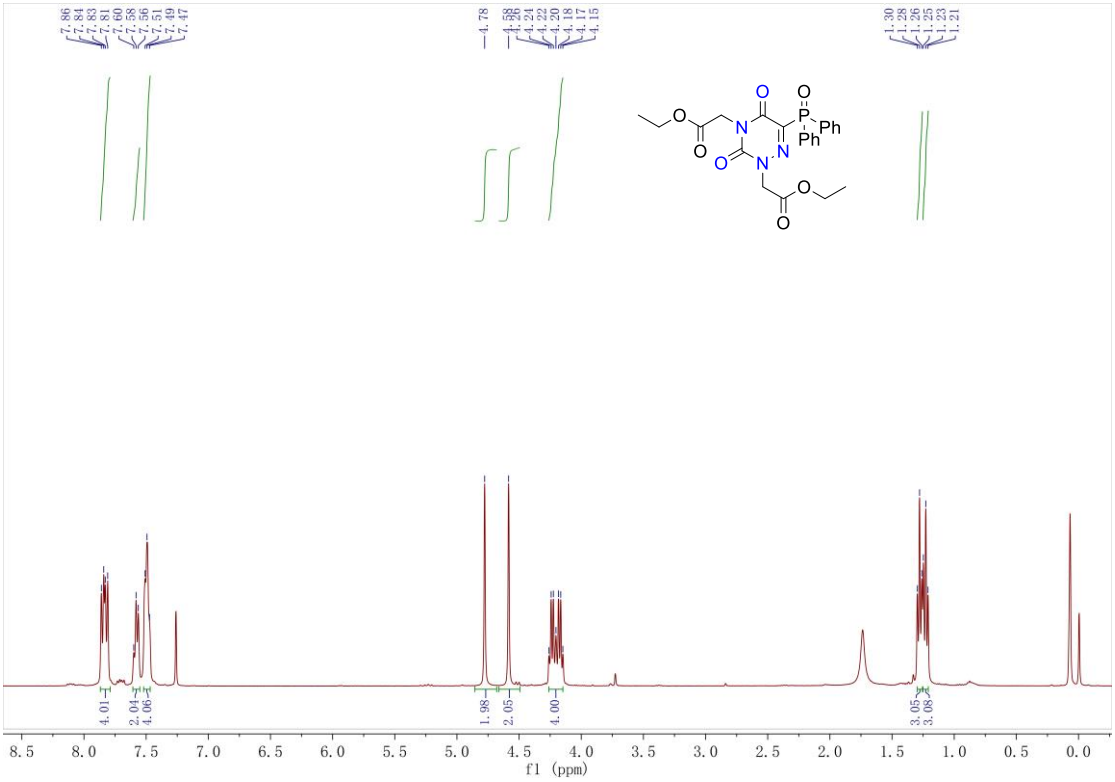
3i-^{31}P


22.13
65
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
f1 (ppm)

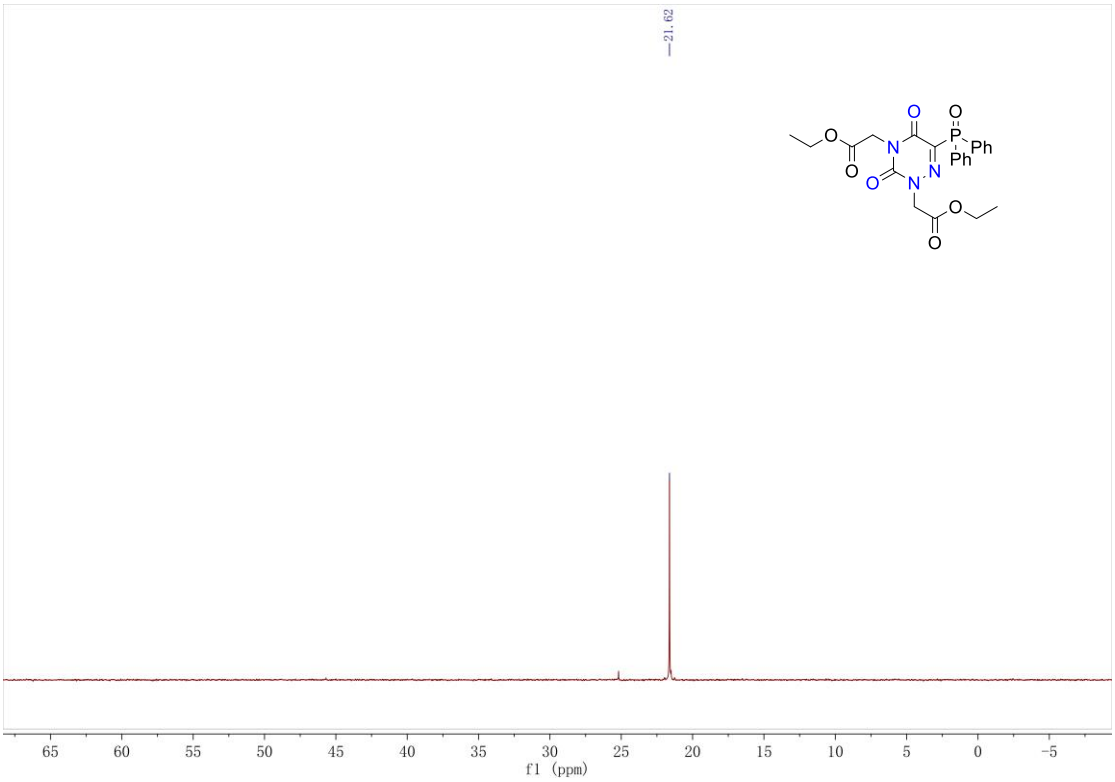
3i-^{13}C


154.62
154.42
147.74
140.77
139.52
132.46
132.43
131.90
131.80
131.38
130.23
129.61
129.13
128.51
128.39
120.38
120.04
54.93
42.93
f1 (ppm)

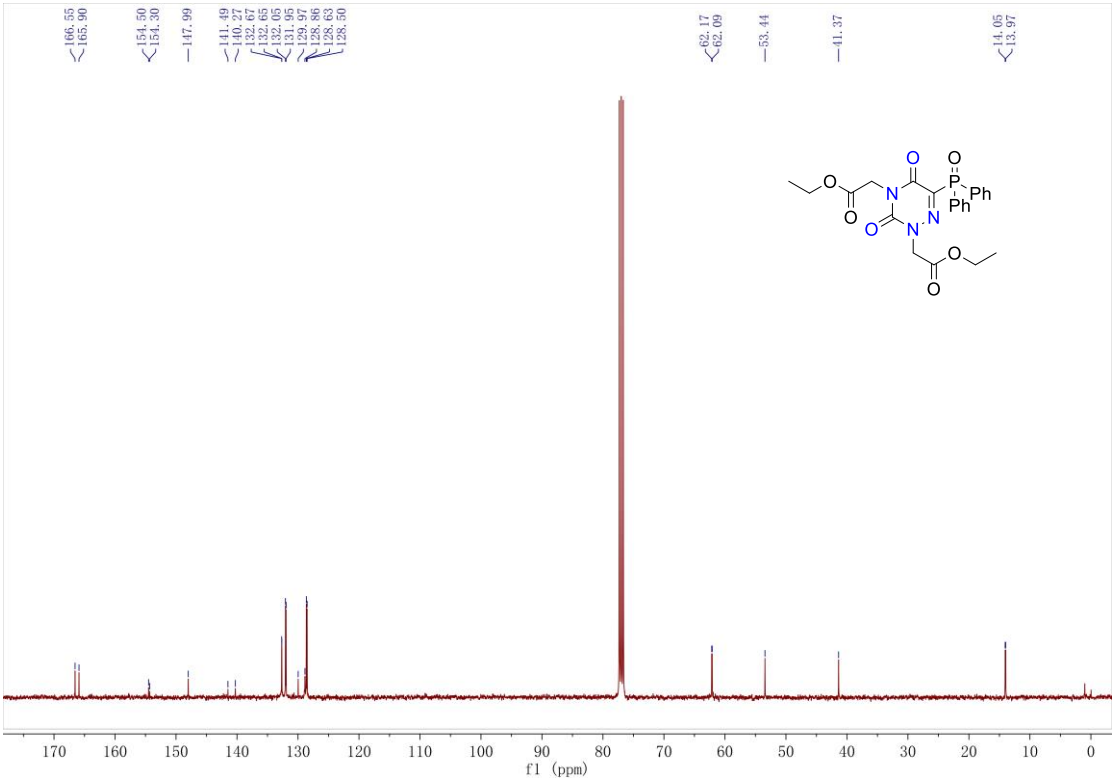
3j-^{1}H


7.86
7.84
7.83
7.81
7.60
7.58
7.56
7.51
7.49
7.47
4.78
4.58
4.38
4.24
4.22
4.20
4.18
4.17
4.15
1.30
1.28
1.26
1.25
1.23
1.21
4.01
2.04
4.06
1.98
2.05
4.00
3.05
3.08
f1 (ppm)

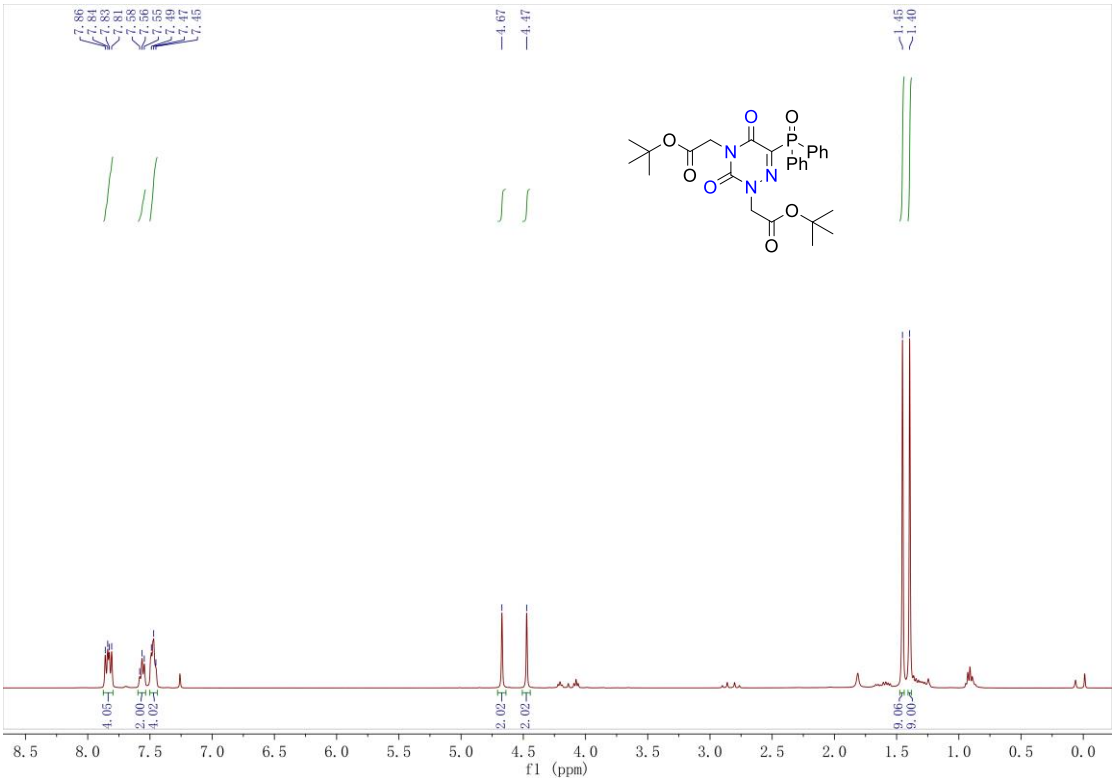
3j-^{31}P


21.62
f1 (ppm)

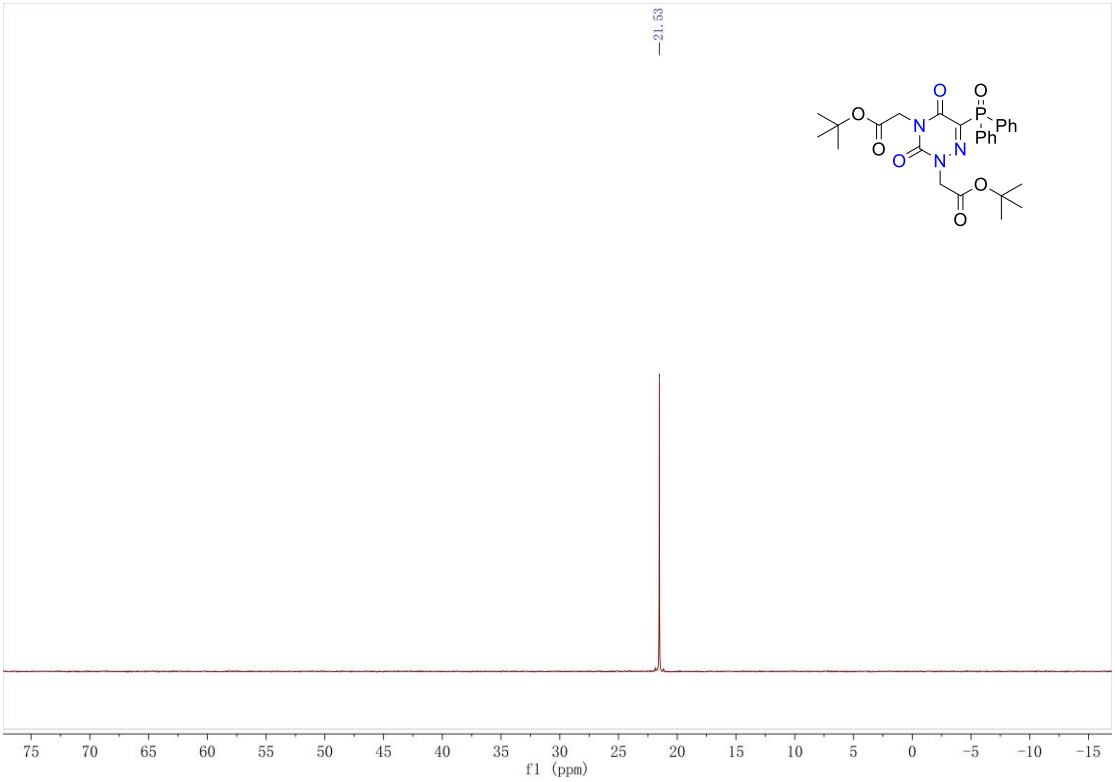
3j-^{13}C


166.55
165.90
154.50
154.30
147.99
141.49
140.27
132.67
132.65
132.05
131.95
129.97
128.86
128.63
128.50
62.17
62.09
53.44
41.37
14.05
13.97
f1 (ppm)

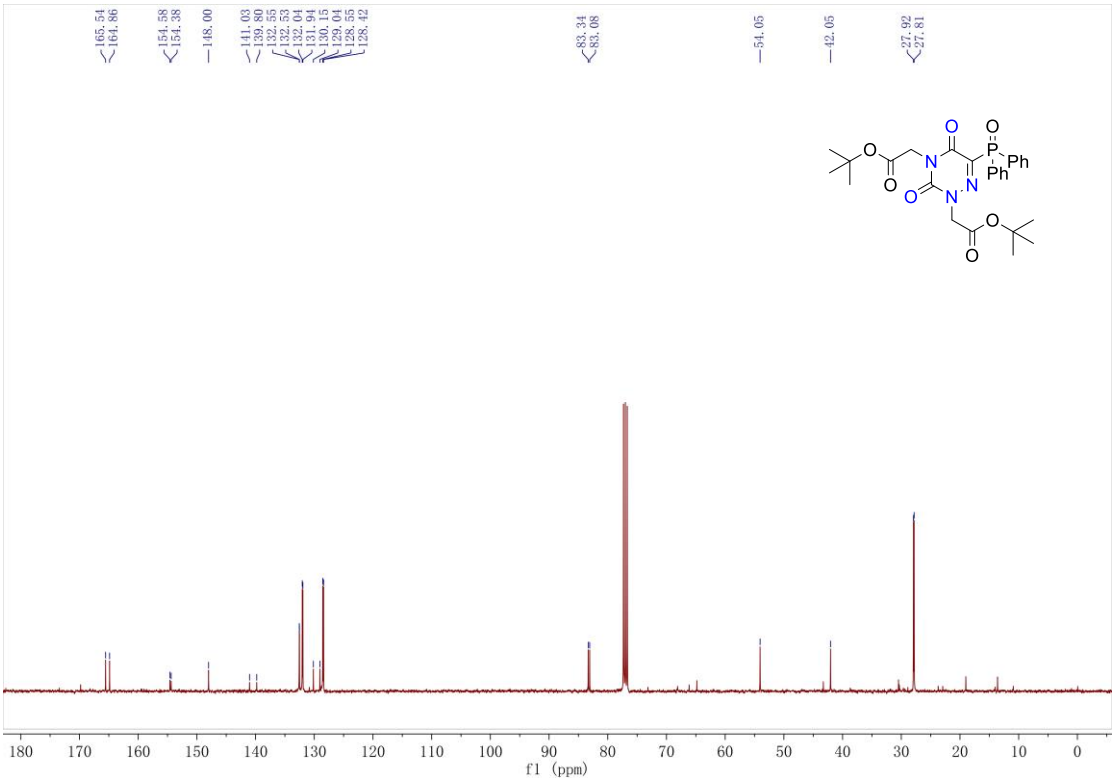
3k-^{1}H


7.86
7.84
7.83
7.81
7.58
7.56
7.55
7.49
7.47
7.45
4.67
4.47
1.45
1.40
4.05
2.00
4.02
2.02
2.02
9.06
9.00
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)

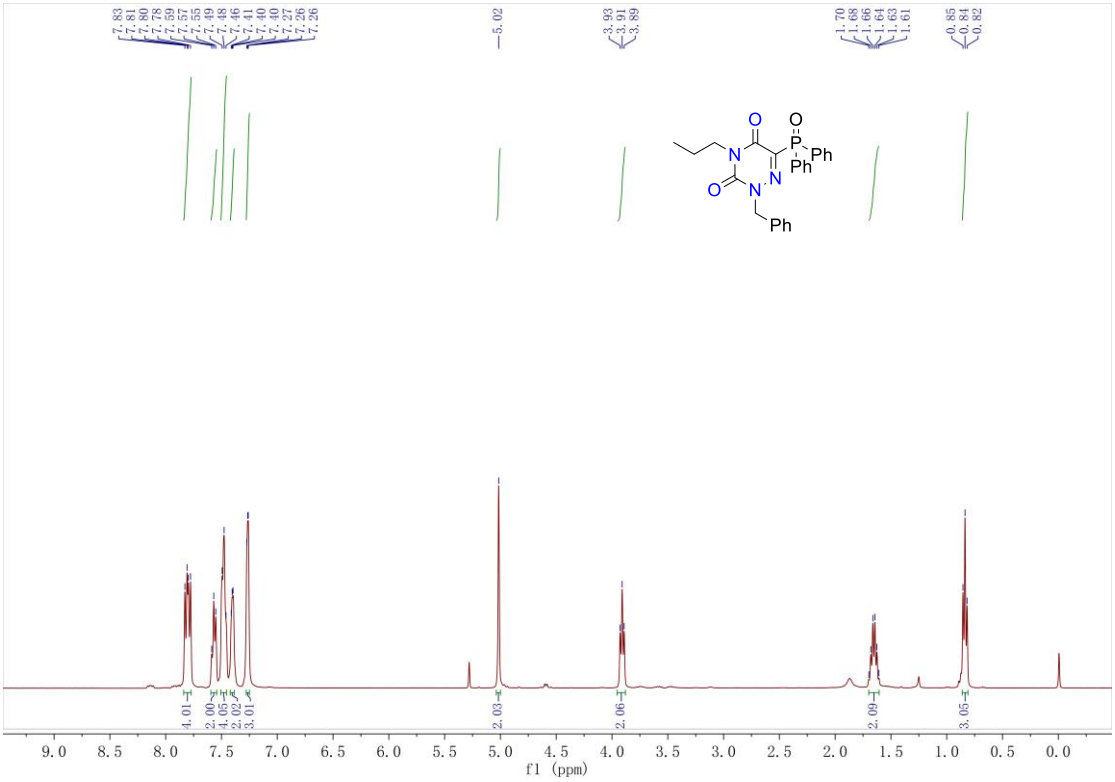
3k-^{31}P


21.53
75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5 -10 -15
f1 (ppm)

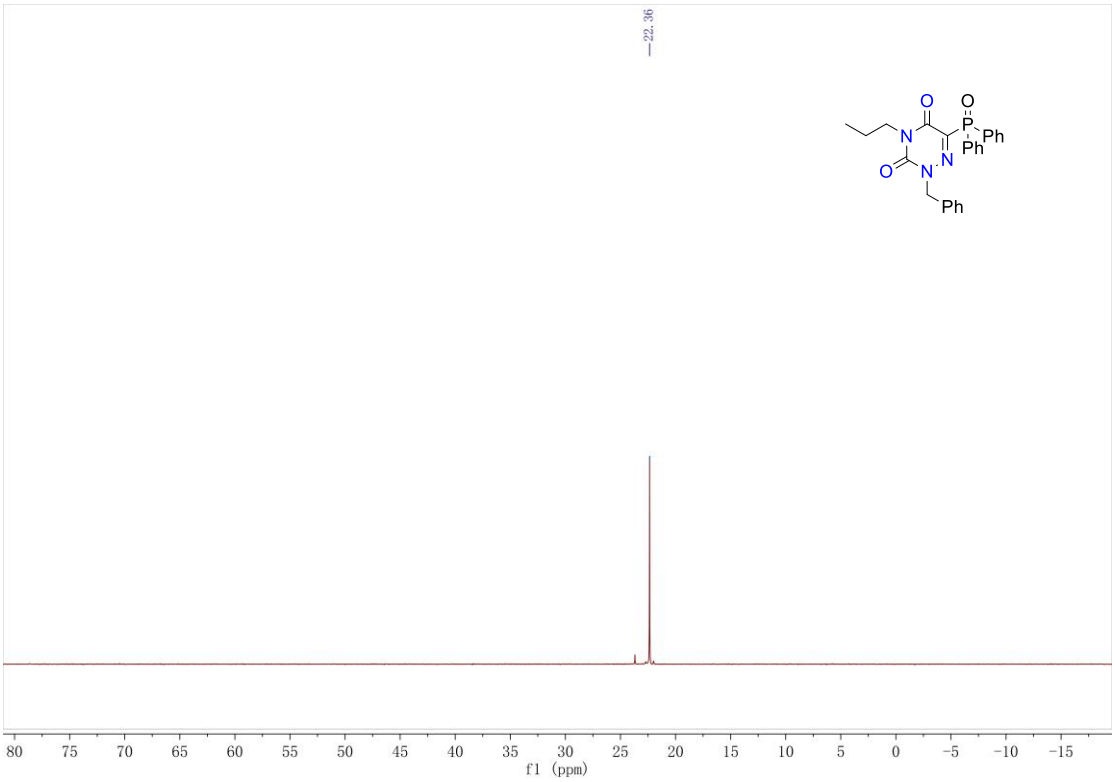
3k-^{13}C


165.54
164.86
154.58
154.38
148.00
141.03
139.80
132.55
132.53
132.04
131.94
130.15
129.04
128.55
128.42
83.34
83.08
54.05
42.05
27.92
27.81
f1 (ppm)

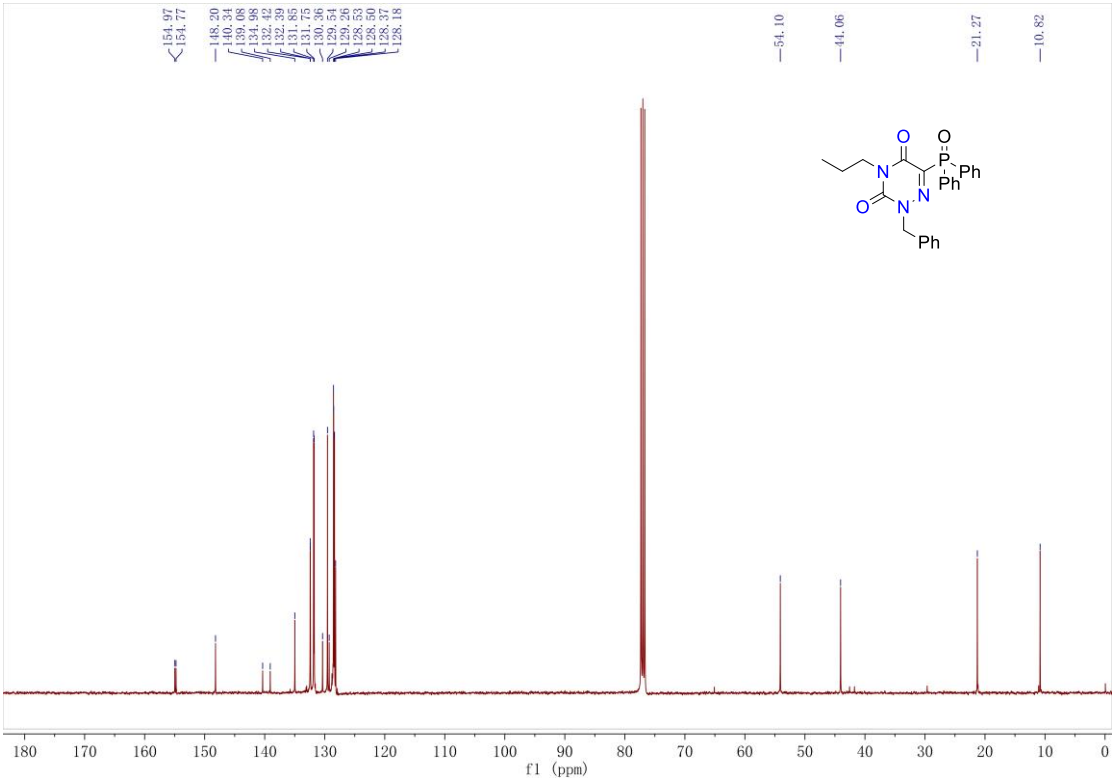
3l-^{1}H


7.83
7.81
7.80
7.78
7.59
7.57
7.55
7.49
7.48
7.46
7.41
7.40
7.40
7.27
7.26
7.26
5.02
3.93
3.91
3.89
1.70
1.68
1.66
1.64
1.63
1.61
0.85
0.84
0.82
4.01
2.00
4.05
2.02
3.01
2.03
2.06
2.09
3.05
f1 (ppm)

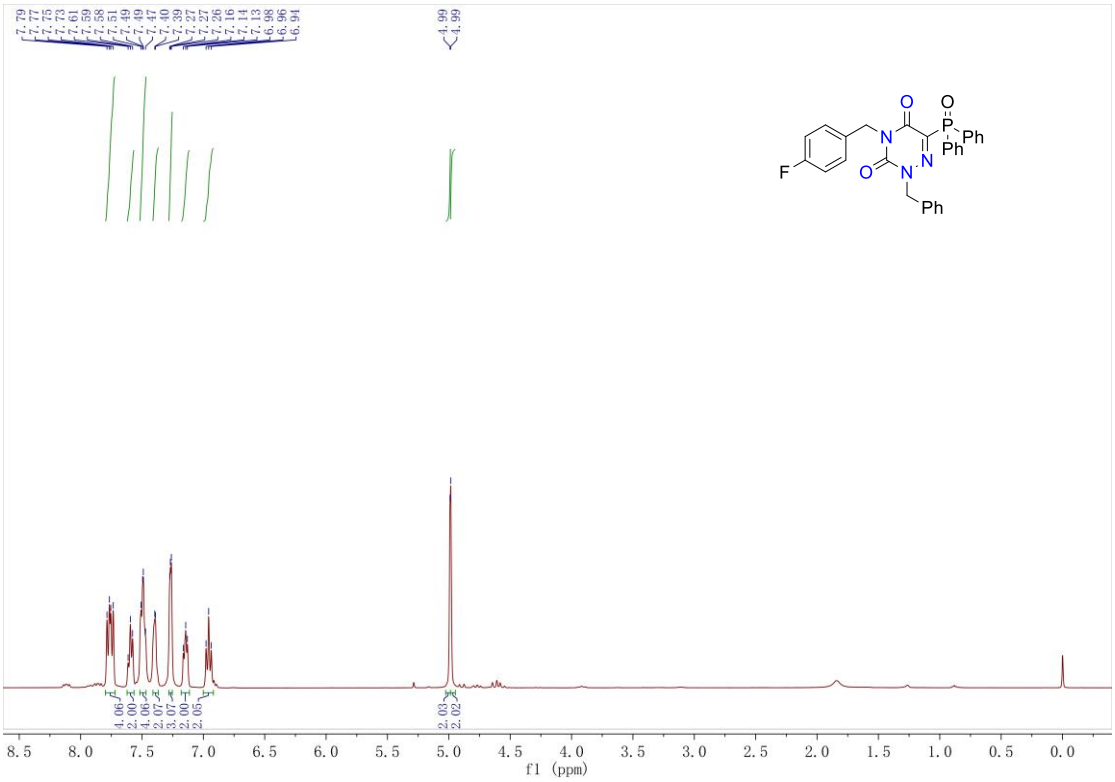
3l-^{31}P


22.36
f1 (ppm)

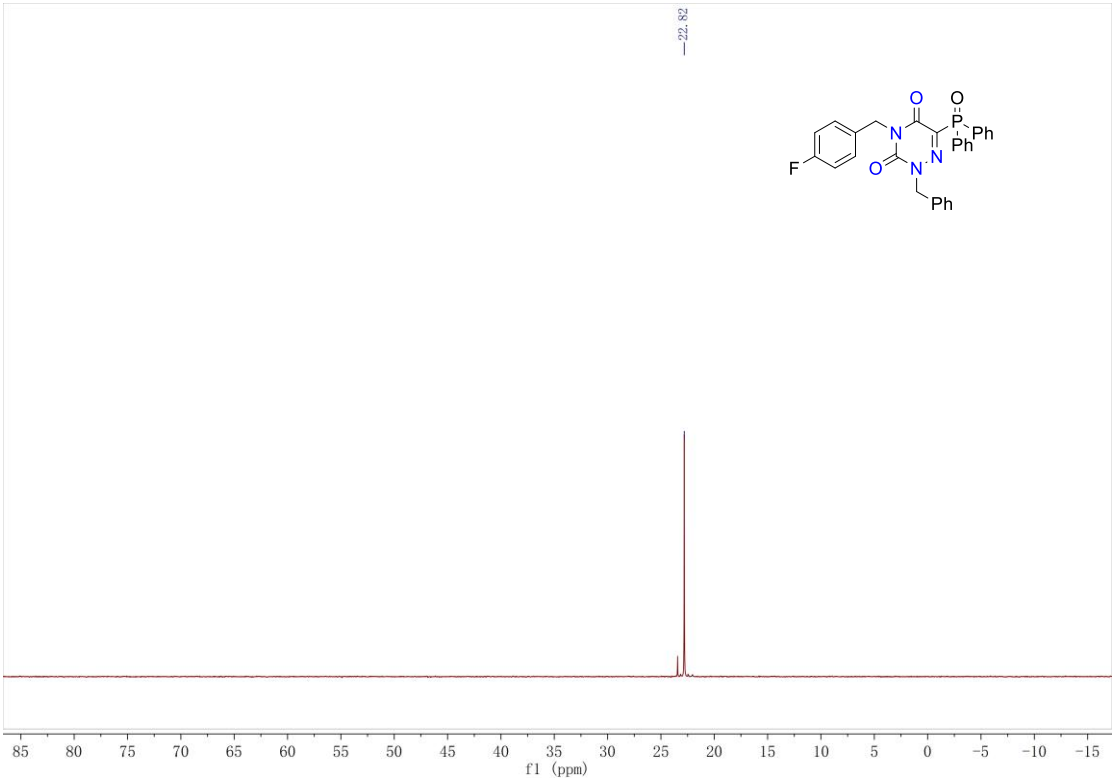
3l-^{13}C


154.97
154.77
148.20
140.34
139.08
134.98
132.42
132.39
131.85
131.75
130.36
129.54
129.26
128.53
128.50
128.37
128.18
54.10
44.06
21.27
10.82
f1 (ppm)

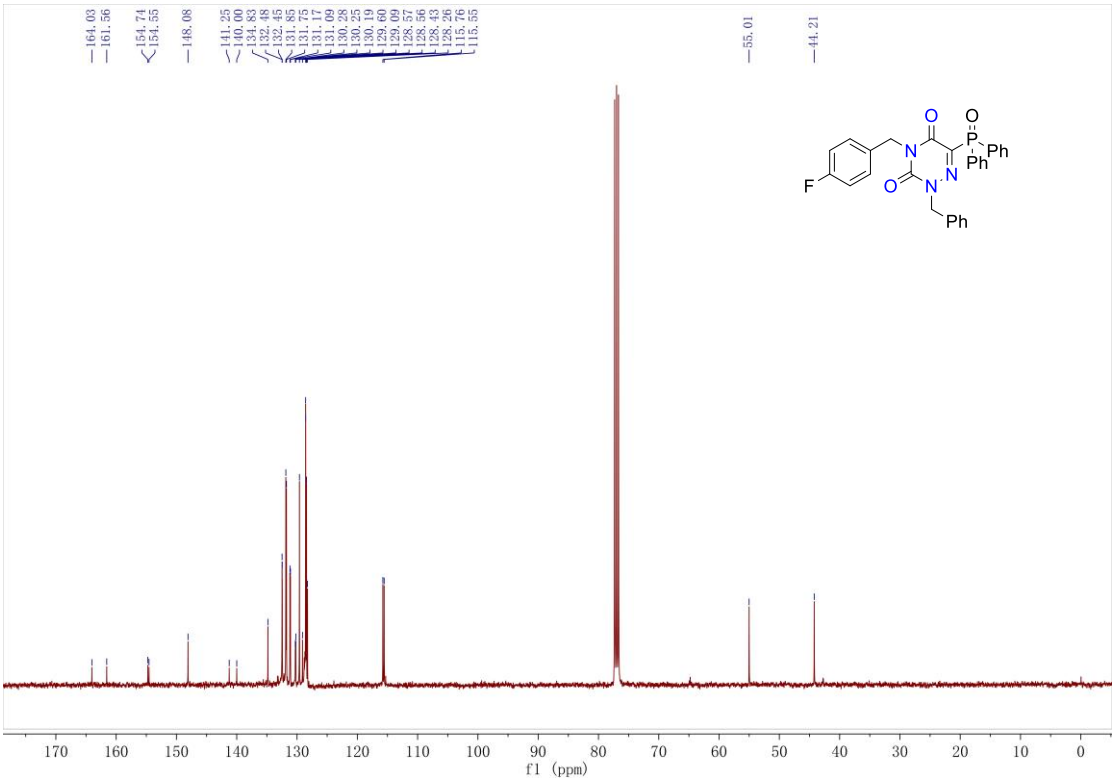
3m-^{1}H


7.79
7.77
7.75
7.73
7.61
7.59
7.58
7.51
7.49
7.49
7.47
7.40
7.39
7.27
7.27
7.26
7.16
7.14
7.13
6.98
6.96
6.94
4.99
4.99
4.06
2.00
4.06
2.07
3.07
2.00
2.05
2.03
2.02
F
O
O
P
Ph
Ph
N
N
O
N
Ph
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)

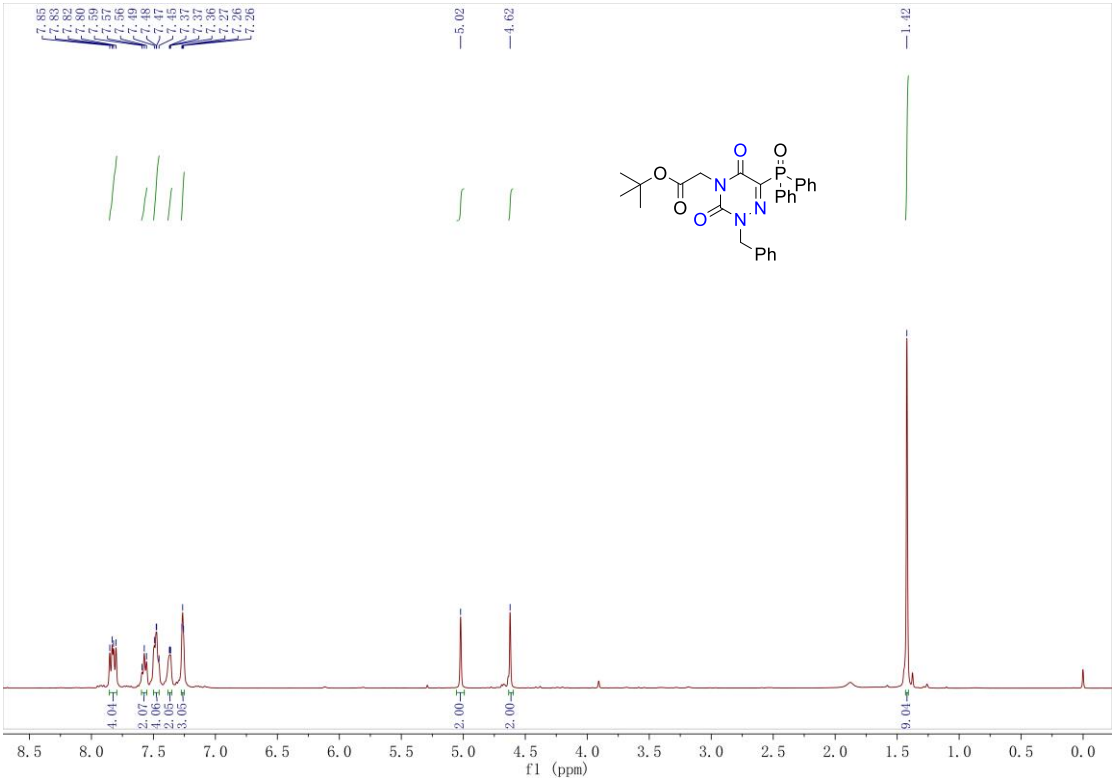
3m-^{31}P


22.82
F
O
O
P
Ph
Ph
N
N
O
N
Ph
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
−5
−10
−15
f1 (ppm)

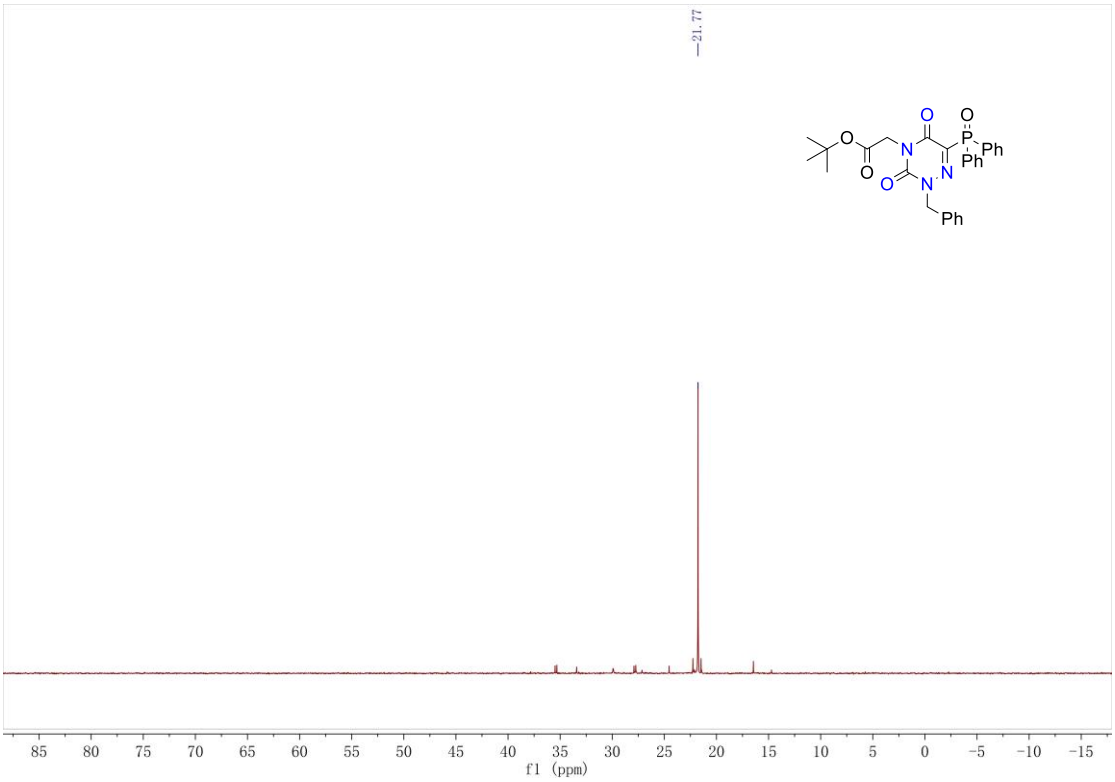
3m-^{13}C


164.03
161.56
154.74
154.55
148.08
141.25
140.66
134.83
132.48
132.45
131.85
131.75
131.17
131.09
130.28
130.25
130.19
129.60
129.09
128.57
128.55
128.43
128.26
115.76
115.55
55.01
44.21
O
O
P
Ph
Ph
N
N
O
N
F
Ph
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

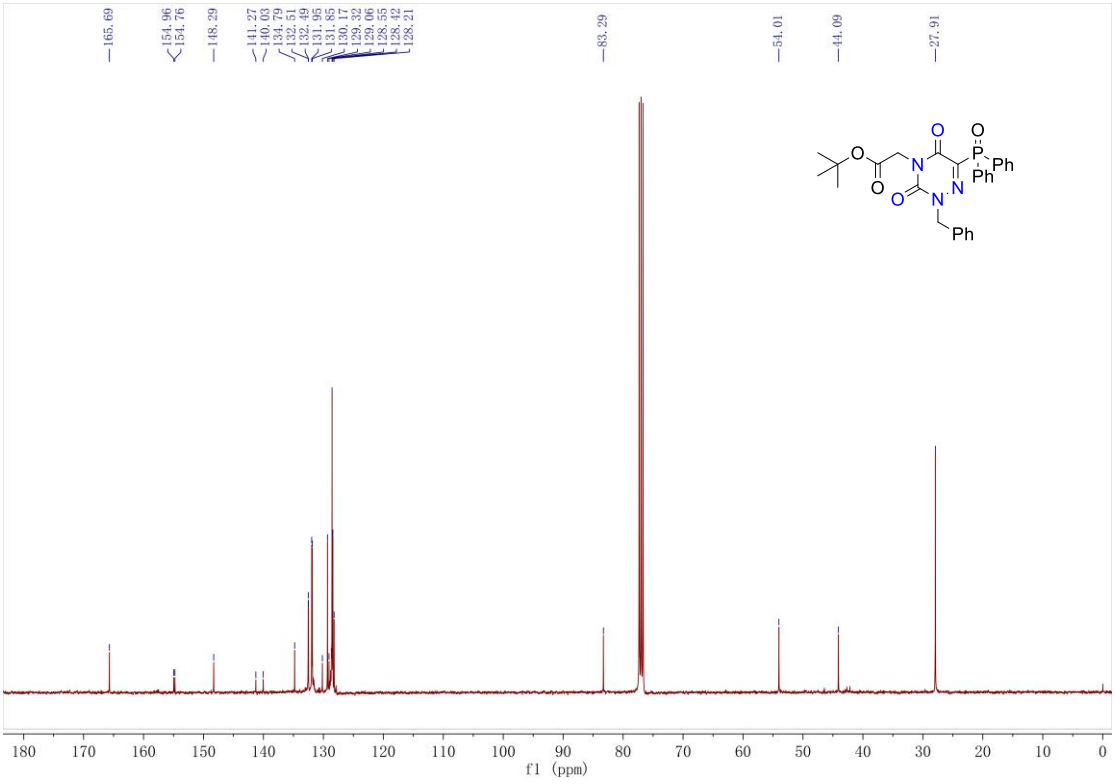
3n-^{1}H


7.85
7.83
7.82
7.80
7.59
7.57
7.56
7.49
7.48
7.47
7.45
7.37
7.37
7.36
7.27
7.26
7.26
5.02
4.62
1.42
O
O
O
P
Ph
Ph
N
N
O
N
Ph
4.04
2.07
1.06
1.05
3.05
2.00
2.00
9.04
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)

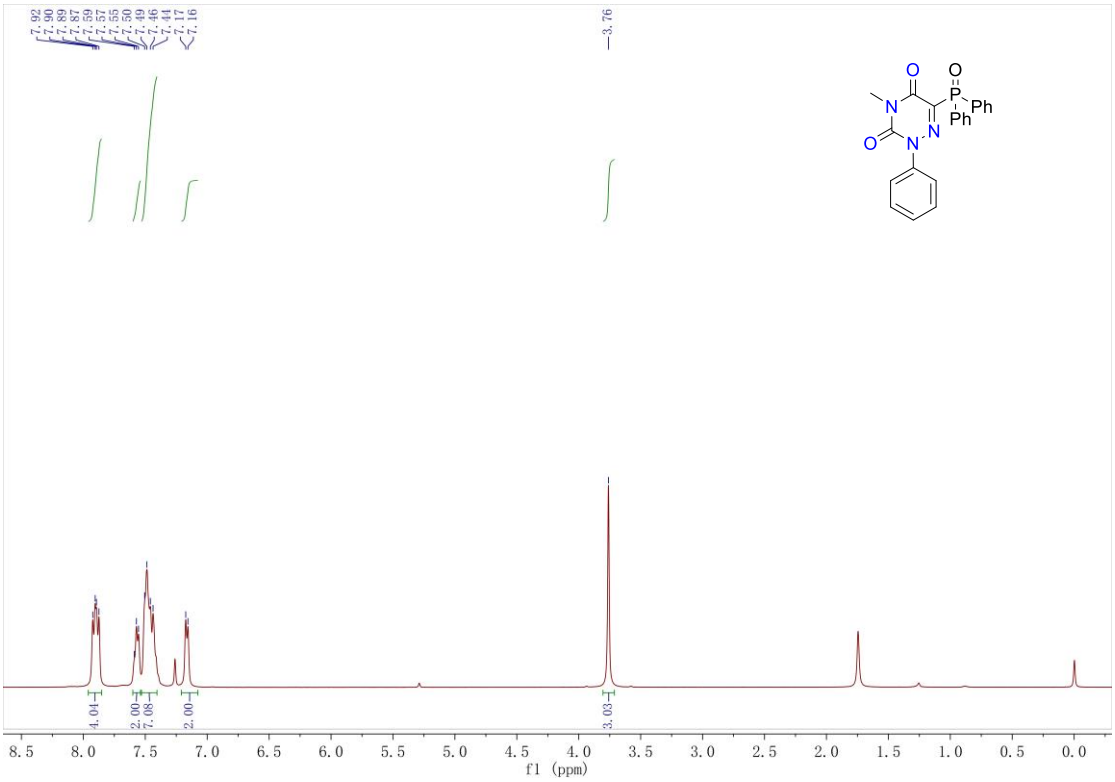
3n-^{31}P


21.77
85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5 -10 -15
f1 (ppm)

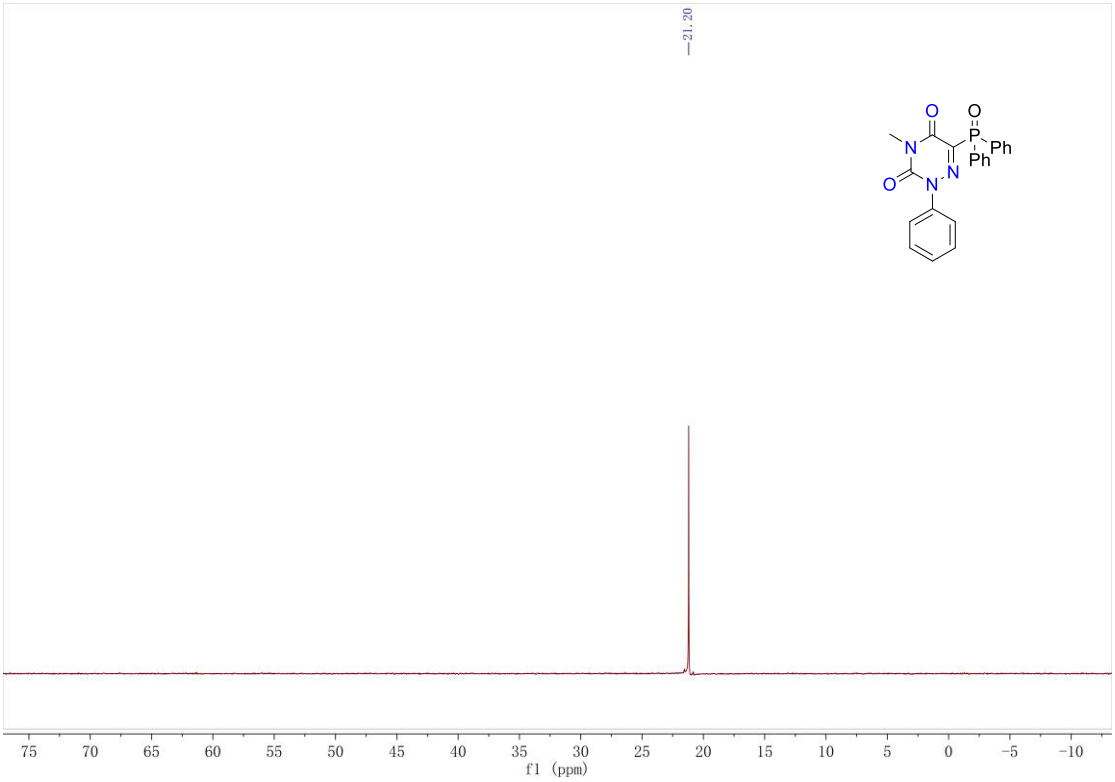
3n-^{13}C


165.69
154.96
154.76
148.29
141.27
140.03
134.79
132.51
132.49
131.95
131.85
130.17
129.32
129.06
128.55
128.42
128.21
83.29
51.01
41.09
27.91
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

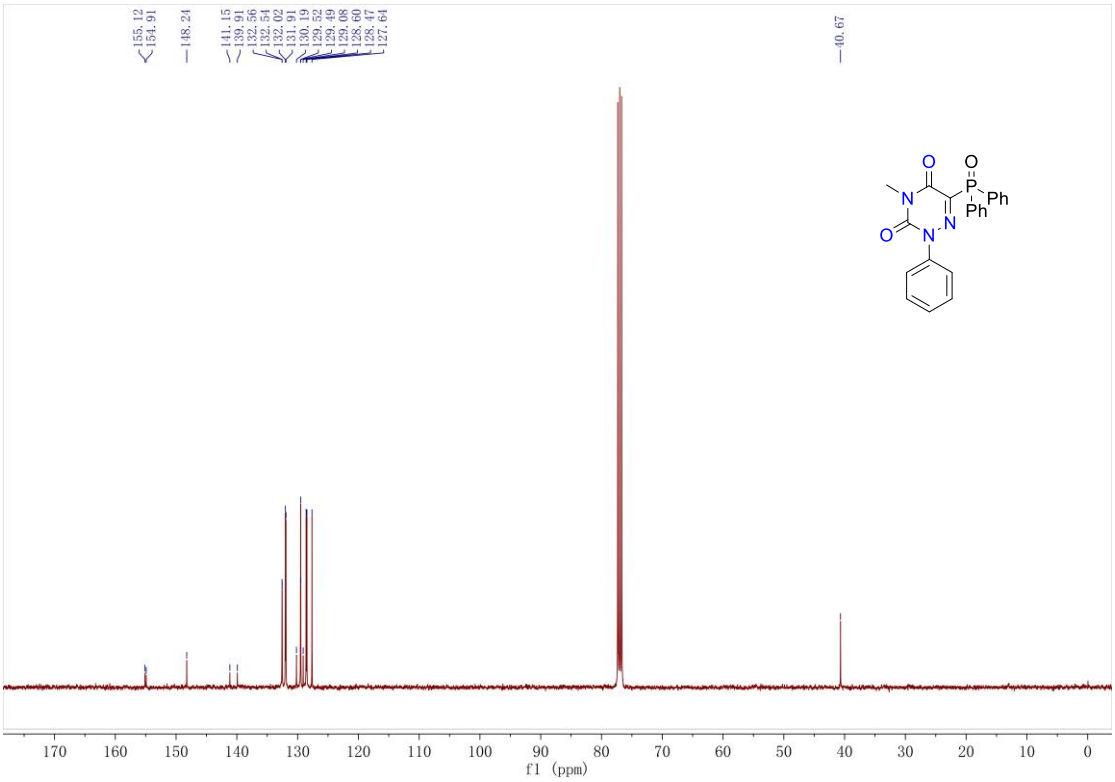
3o-^{1}H


7.92
7.90
7.89
7.87
7.59
7.57
7.55
7.50
7.49
7.46
7.44
7.17
7.16
3.76
4.04
2.00
7.08
2.00
3.03
f1 (ppm)

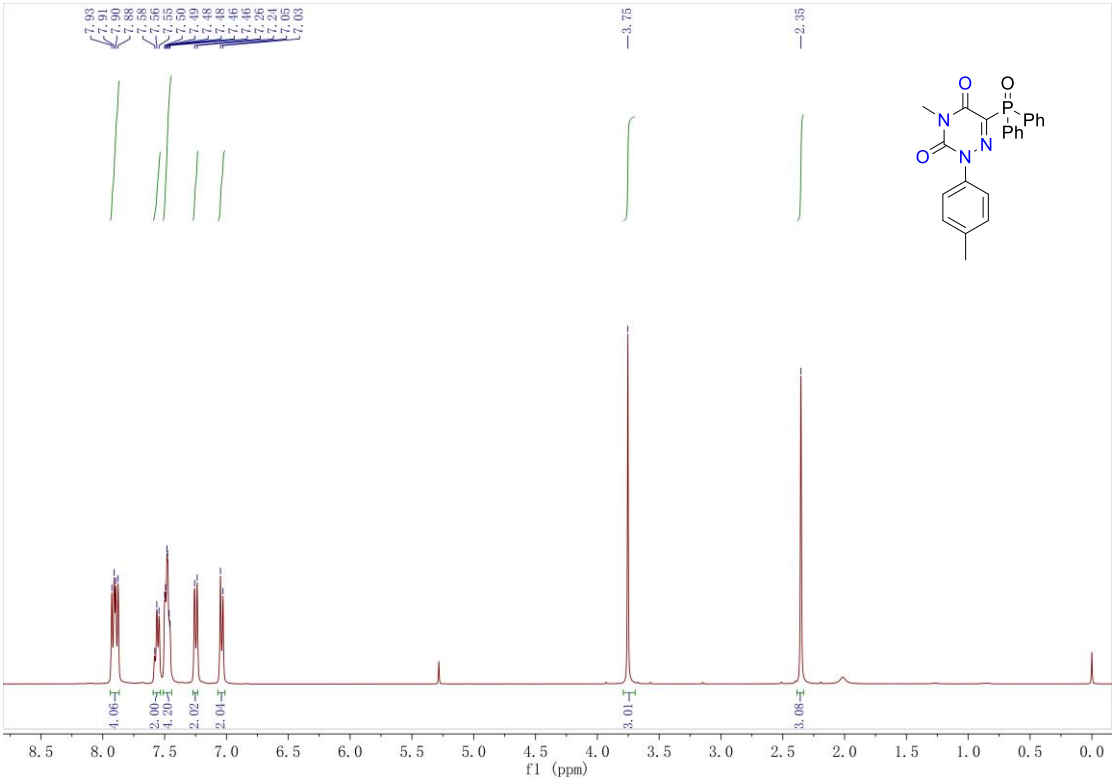
3o-^{31}P


21.20
f1 (ppm)

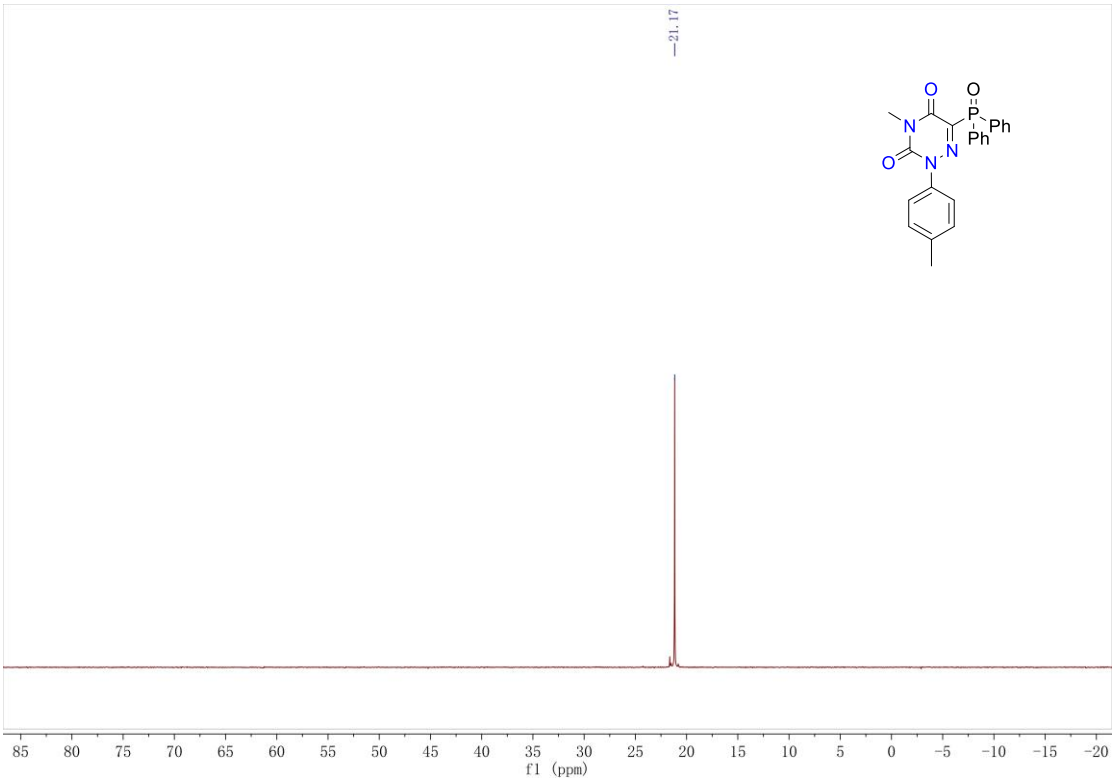
3o-^{13}C


155.12
154.91
148.24
141.15
139.91
132.56
132.54
132.02
131.91
130.19
129.52
129.49
129.08
128.60
128.47
127.64
40.67
f1 (ppm)

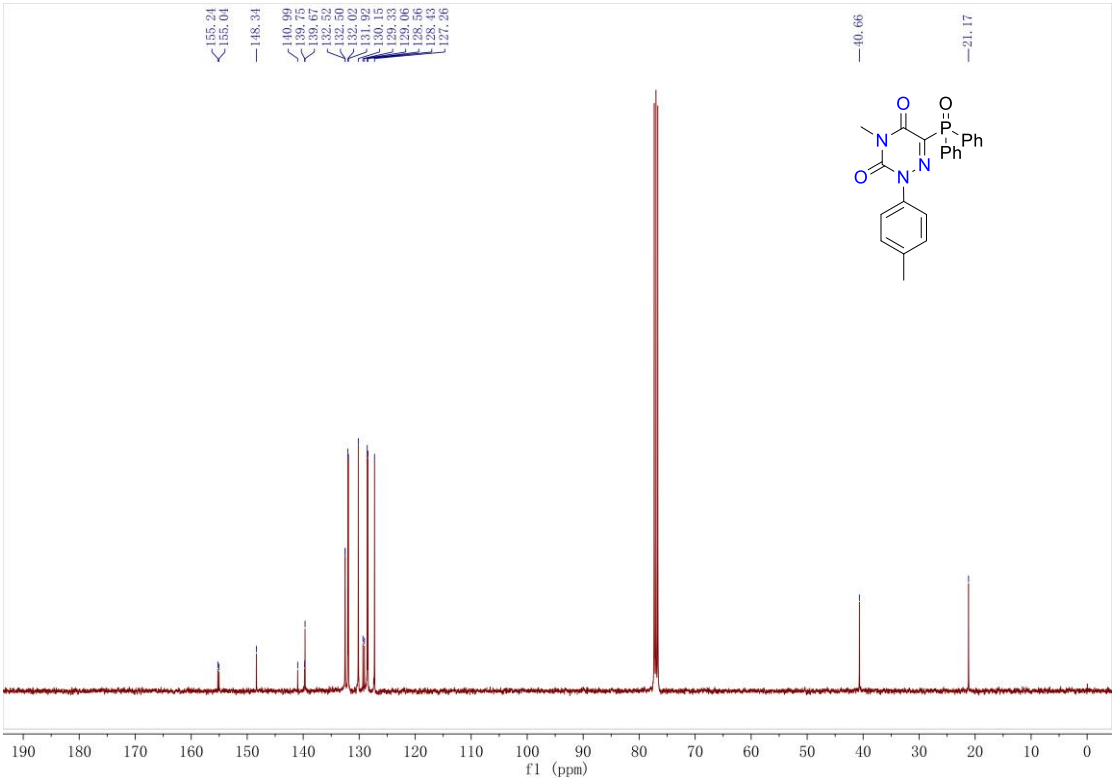
3p-^{1}H


7.93
7.91
7.90
7.88
7.58
7.56
7.55
7.50
7.49
7.48
7.46
7.46
7.26
7.24
7.05
7.03
3.75
2.35
4.06
2.00
4.20
2.02
2.04
3.01
3.08
f1 (ppm)

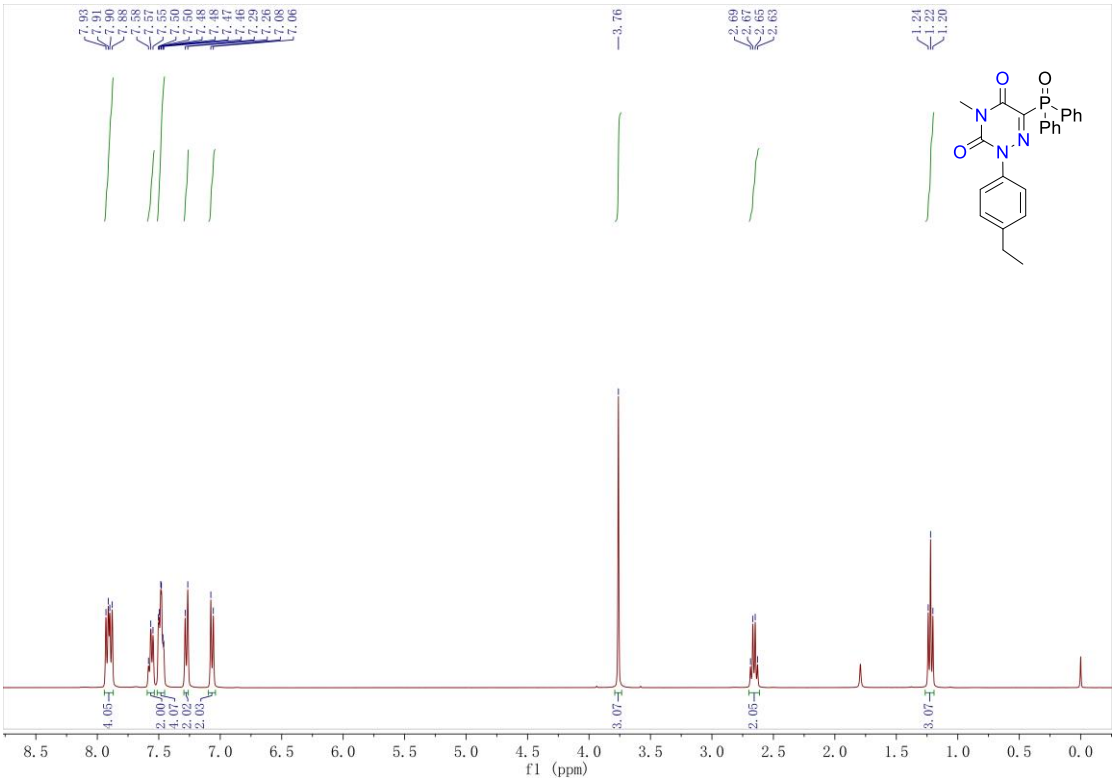
3p-^{31}P


21.17
O
O
N
P
Ph
Ph
N
O
N
85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20
f1 (ppm)

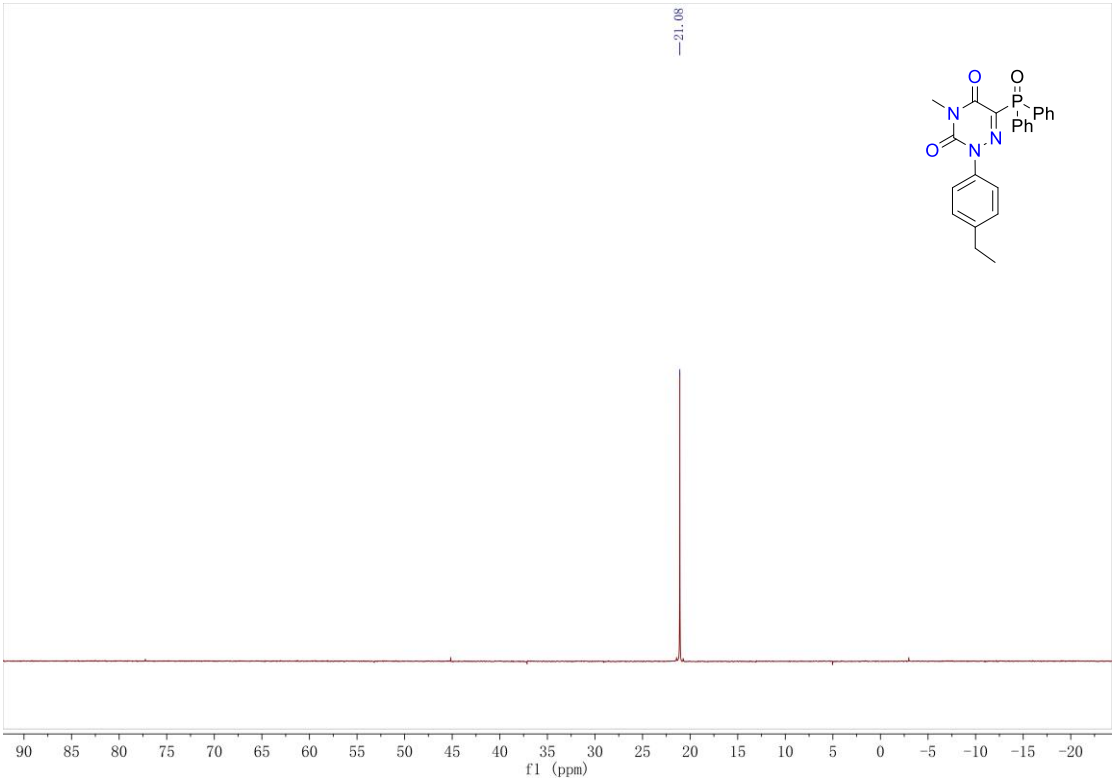
3p-^{13}C


155.24
155.04
148.34
140.99
139.75
139.67
132.52
132.50
132.02
131.92
131.15
129.33
129.06
128.56
128.43
127.26
40.66
21.17
O
O
N
P
Ph
Ph
N
O
N
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

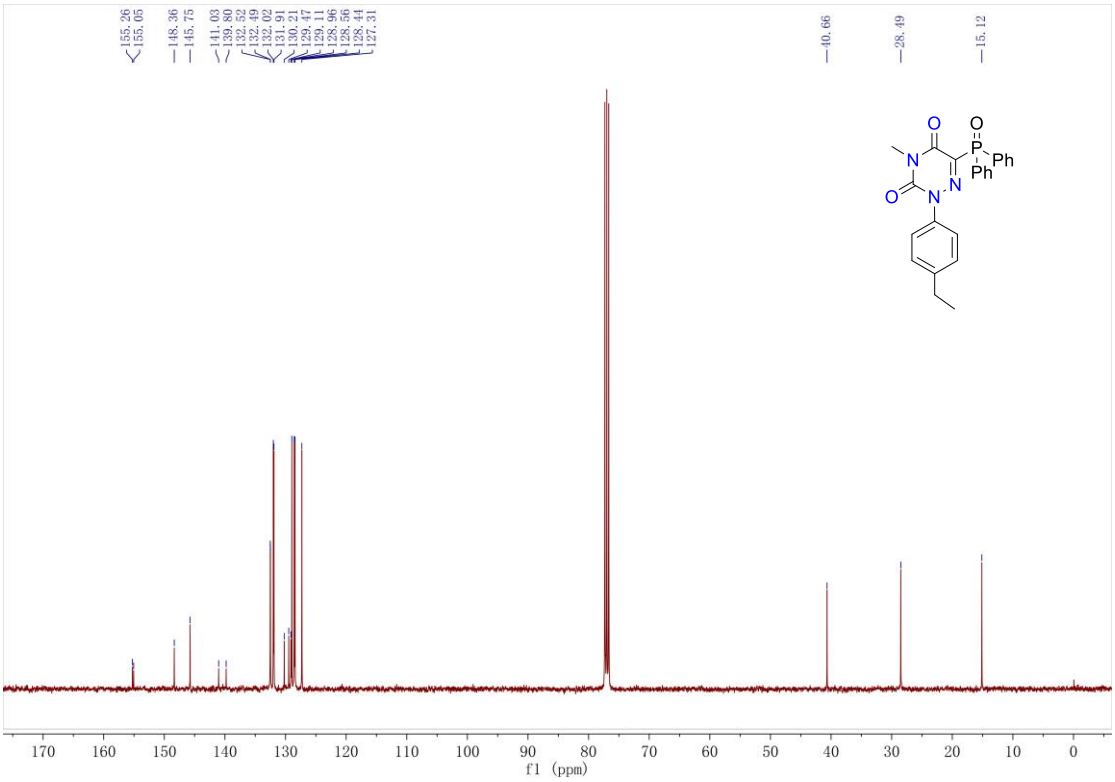
3q-^{1}H


7.93
7.91
7.90
7.88
7.58
7.57
7.55
7.50
7.50
7.48
7.47
7.46
7.29
7.08
7.06
3.76
2.69
2.67
2.65
2.63
1.24
1.22
1.20
4.05
2.00
4.07
2.02
2.03
3.07
2.05
3.07
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)

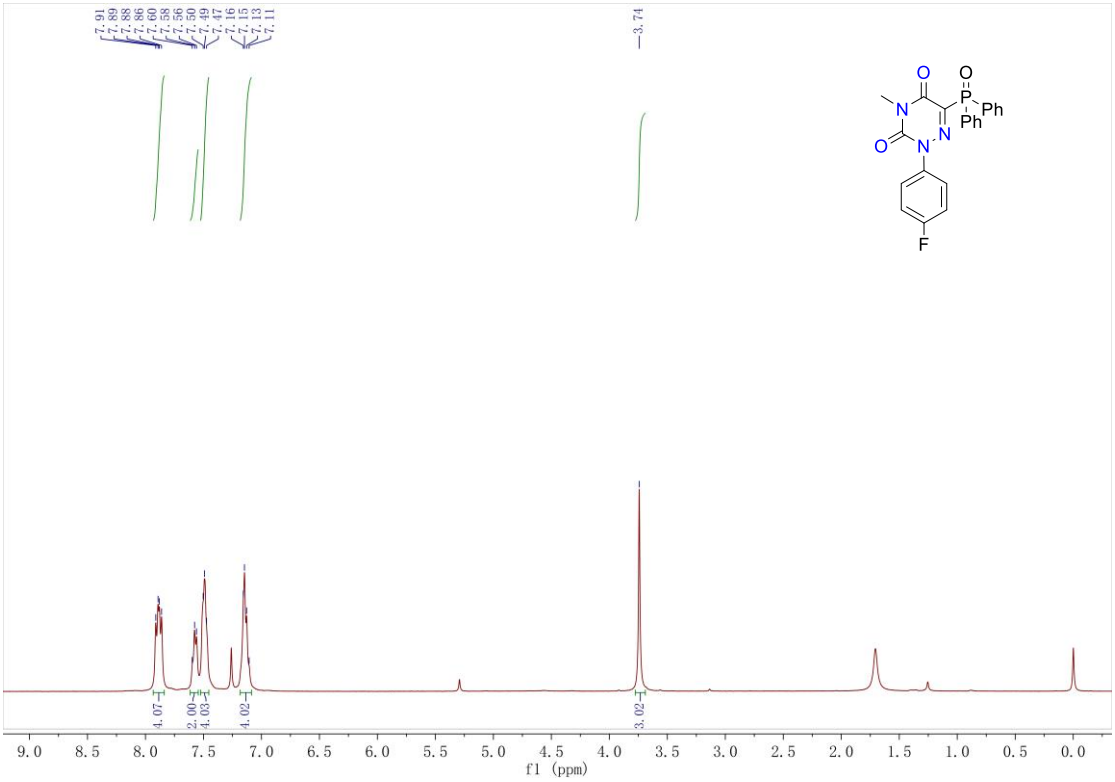
3q-^{31}P


21.08
90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20
f1 (ppm)

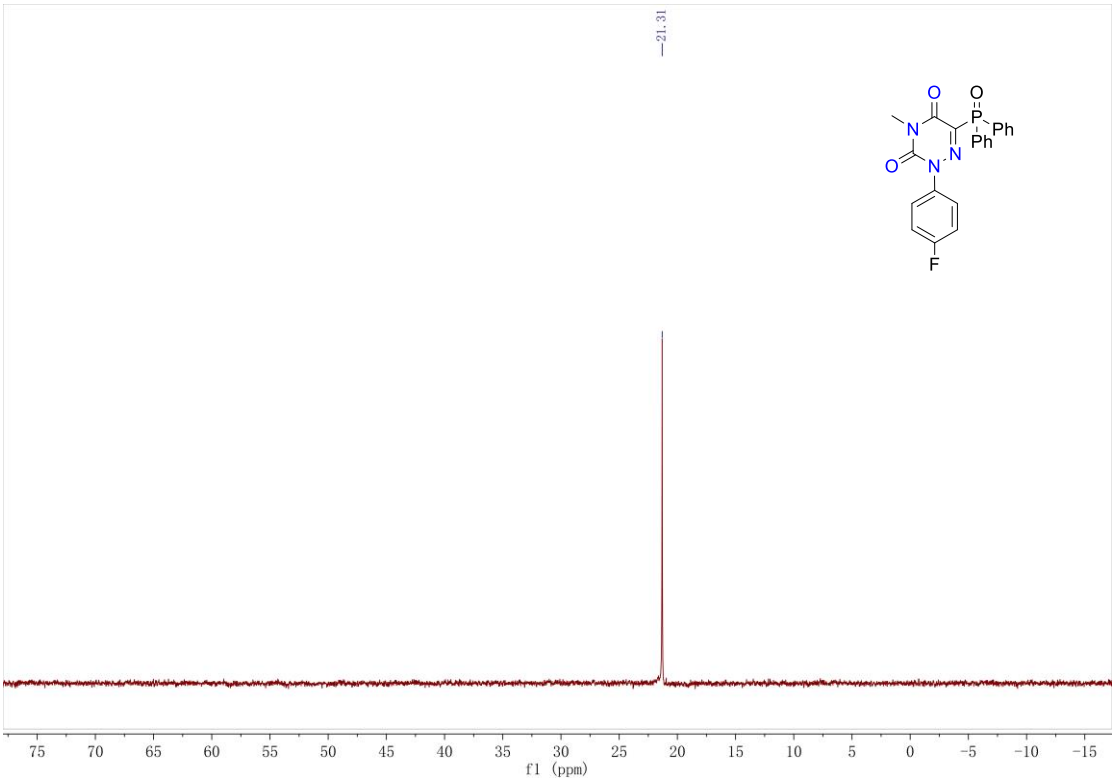
3q-^{13}C


155.26
155.05
148.36
145.75
141.03
139.80
132.52
132.49
132.02
131.91
130.21
129.47
129.11
128.96
128.56
128.44
127.31
40.66
28.49
15.12
O
O
P
Ph
Ph
N
N
O
N
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

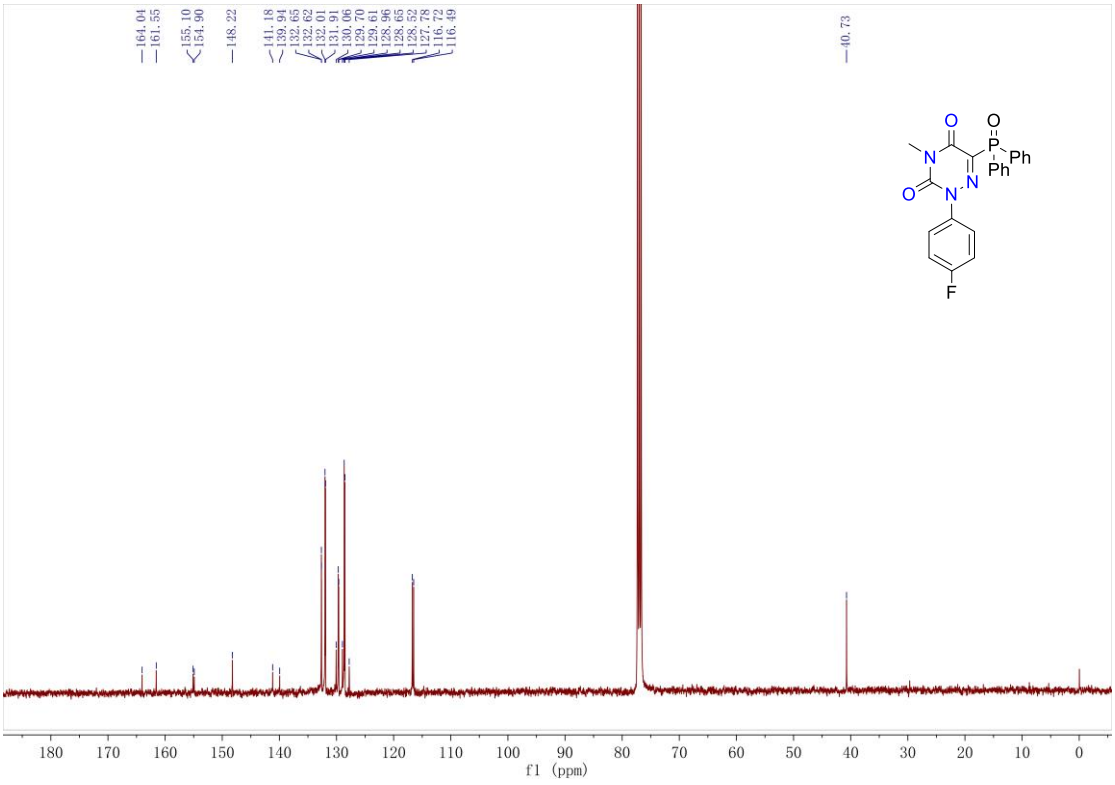
3r-^{1}H


7.91
7.89
7.88
7.86
7.60
7.58
7.56
7.50
7.49
7.47
7.16
7.15
7.13
7.11
3.74
O
O
P
Ph
Ph
N
N
O
N
F
4.07
2.00
4.03
4.02
3.02
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)

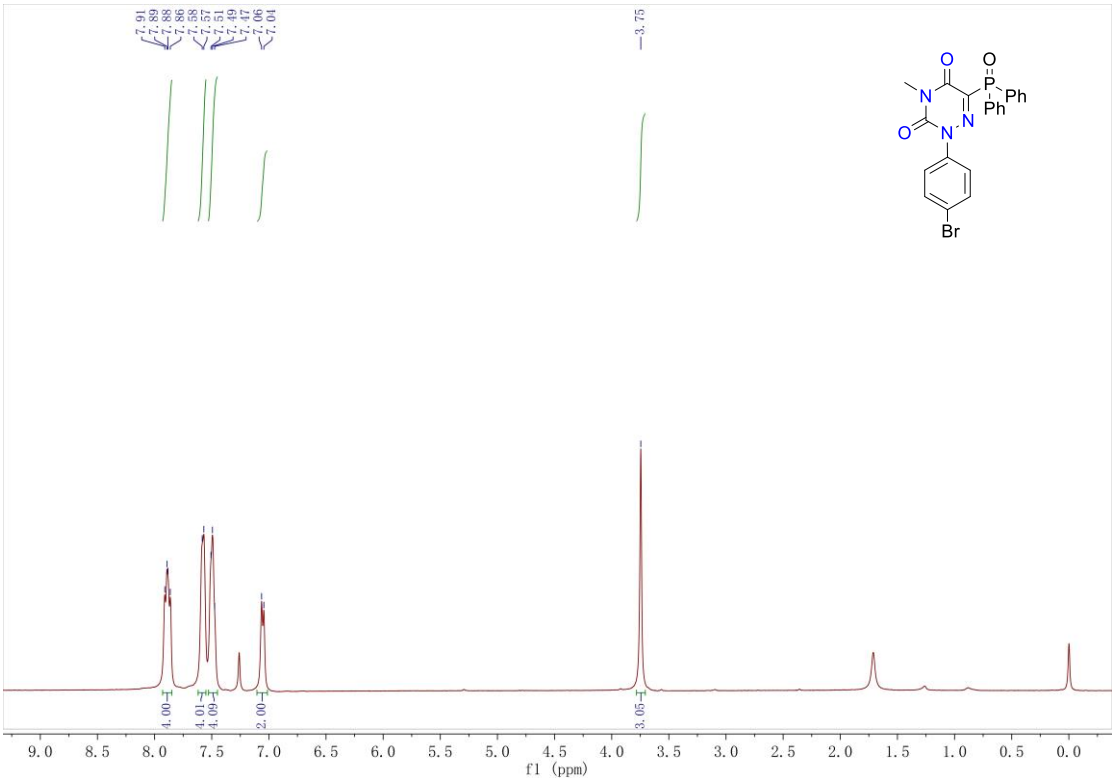
3r-^{31}P


21.31
O
O
N
P
Ph
Ph
O
N
N
F
75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 −15
f1 (ppm)

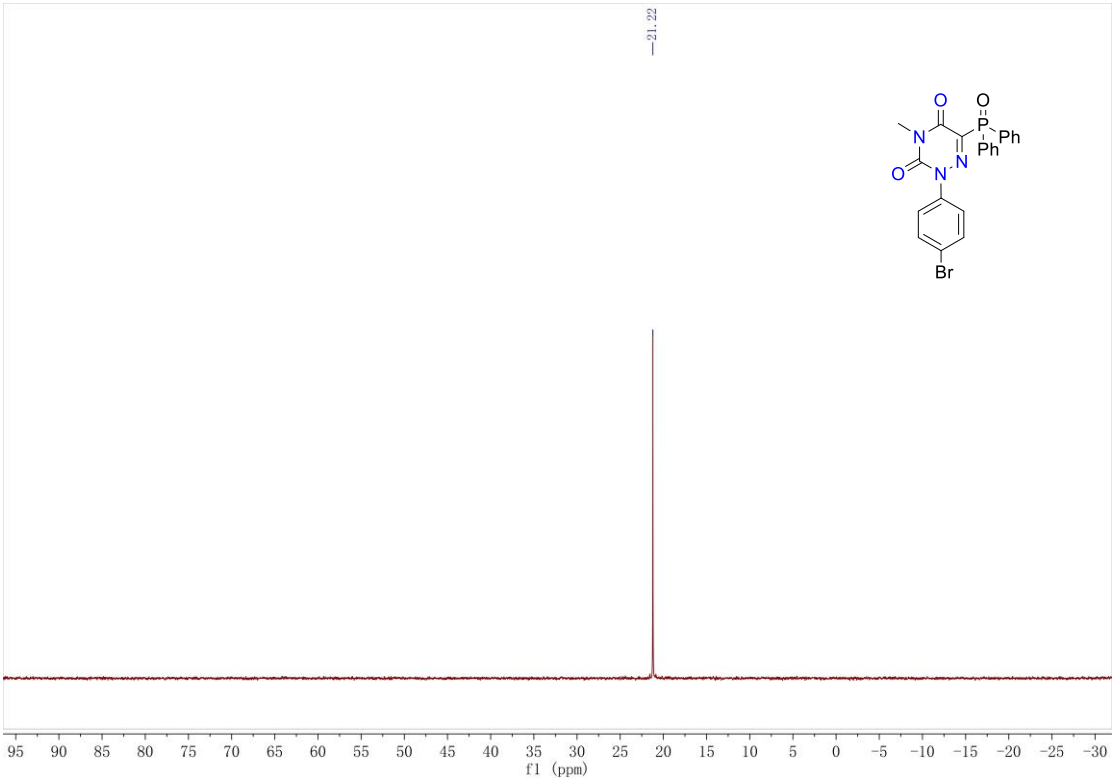
3r-^{13}C


164.04
161.55
155.10
154.90
148.22
141.18
139.94
132.65
132.62
132.01
131.91
130.06
129.70
129.61
128.96
128.65
128.52
127.78
116.72
116.49
40.73
O
O
N
P
Ph
Ph
O
N
N
F
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

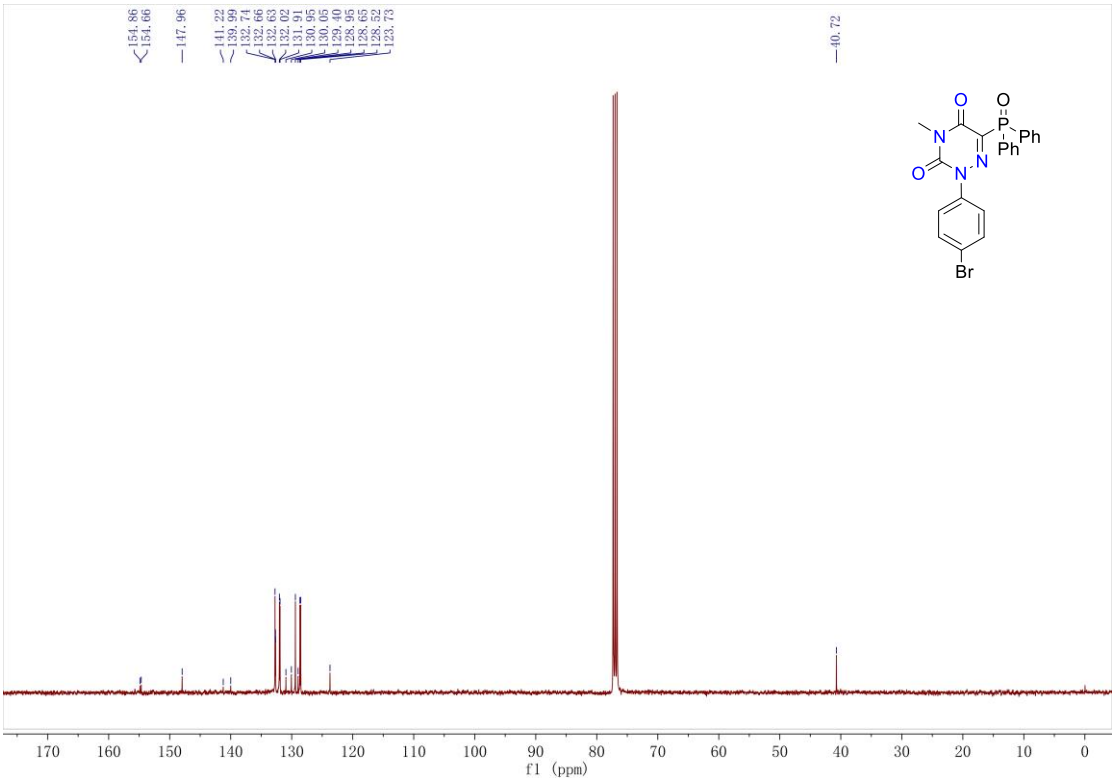
3s-^{1}H


7.91
7.89
7.88
7.86
7.58
7.57
7.51
7.49
7.47
7.06
7.04
3.75
4.00
4.01
4.09
2.00
3.05
f1 (ppm)

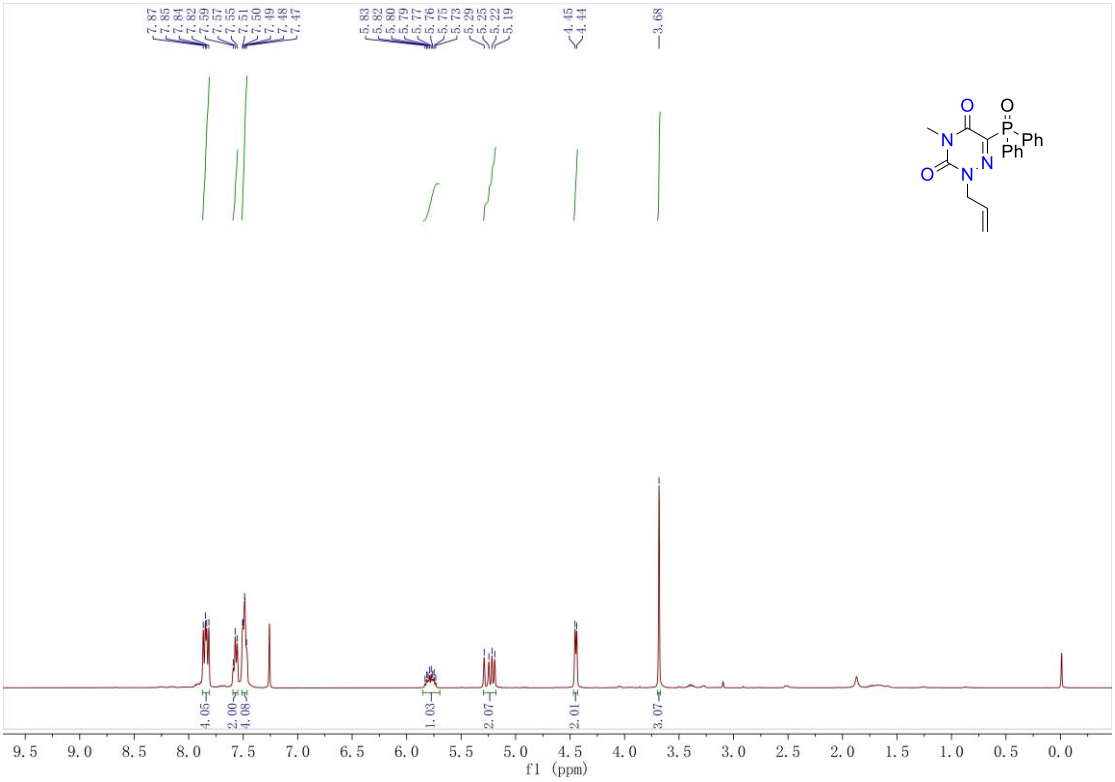
3s-^{31}P


21.22
f1 (ppm)

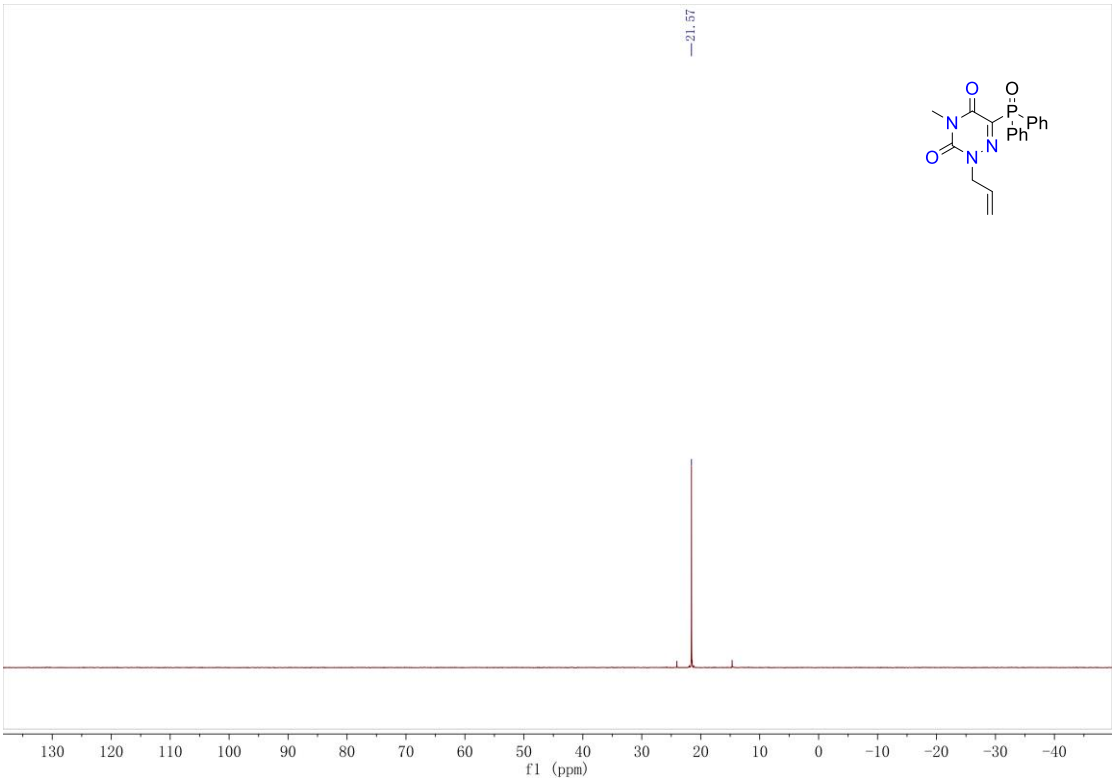
3s-^{13}C


154.86
154.66
147.96
141.22
139.99
132.74
132.66
132.63
132.02
131.91
130.95
130.65
129.40
128.95
128.65
128.52
123.73
40.72
O
O
N
P
Ph
Ph
N
O
N
Br
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

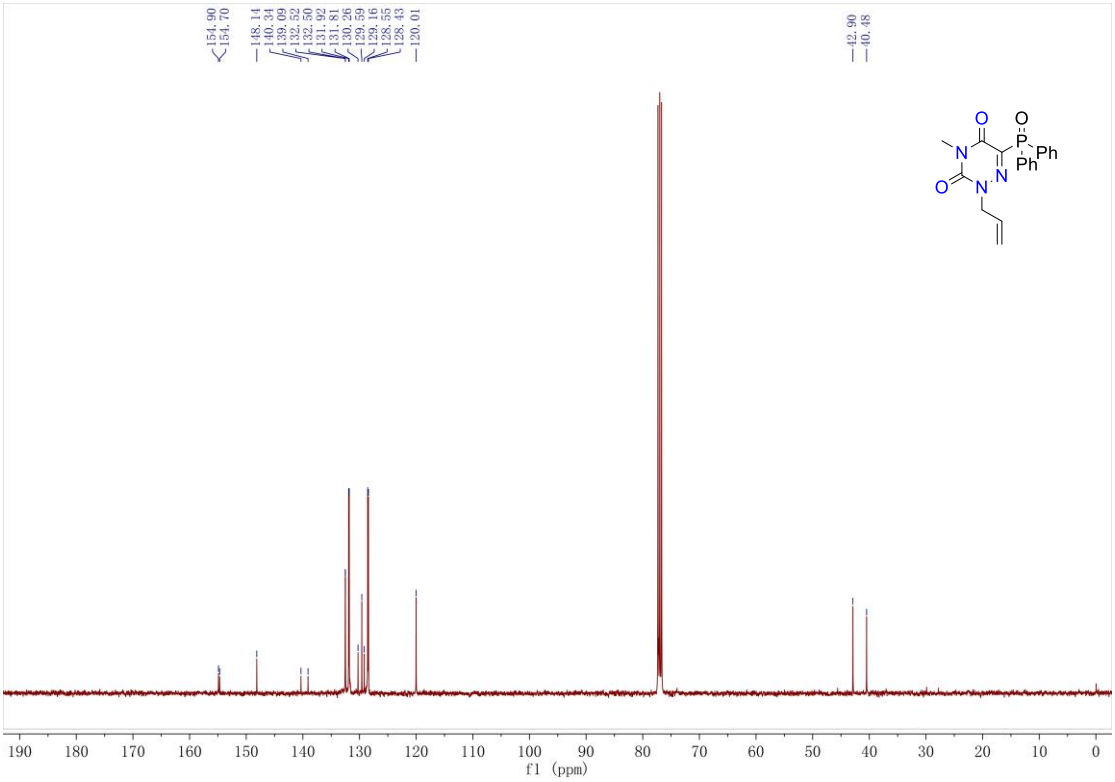
3t-^{1}H


7.87
7.85
7.84
7.82
7.59
7.57
7.55
7.51
7.50
7.49
7.48
7.47
5.83
5.82
5.80
5.79
5.77
5.76
5.75
5.73
5.29
5.25
5.22
5.19
4.45
4.44
3.68
O
O
N
P
Ph
Ph
N
O
N
4.05
2.00
4.08
1.03
2.07
2.01
3.07
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)

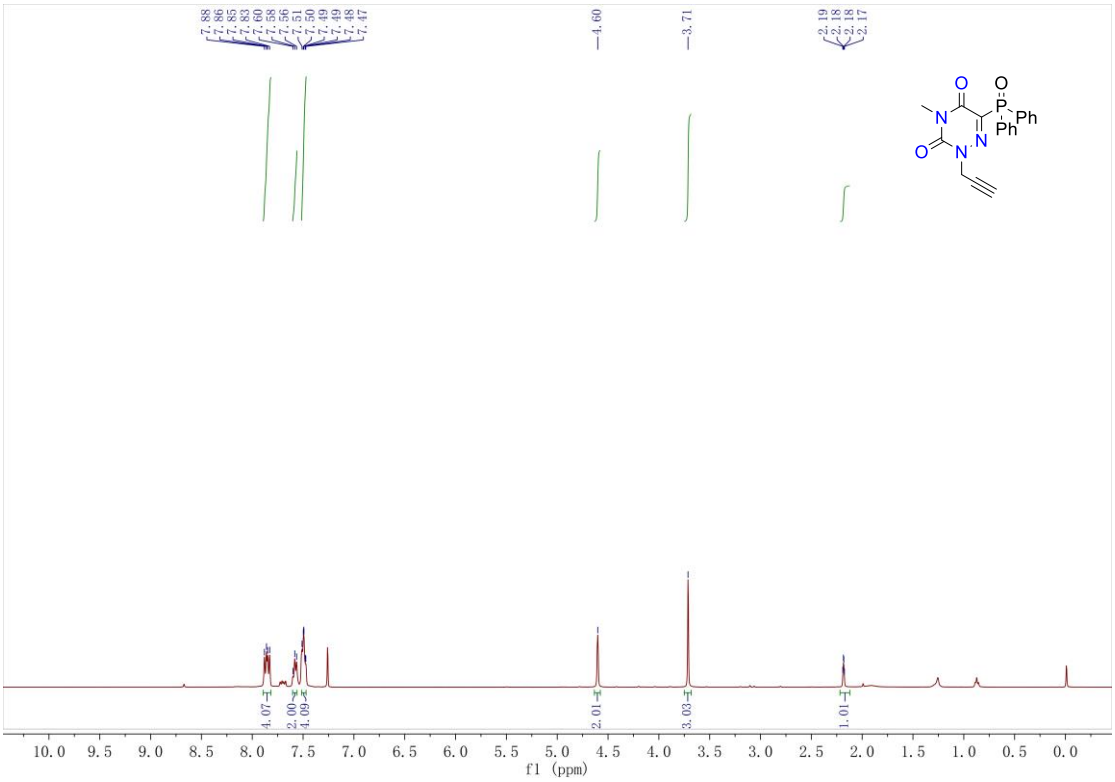
3t-^{31}P


21.57
f1 (ppm)

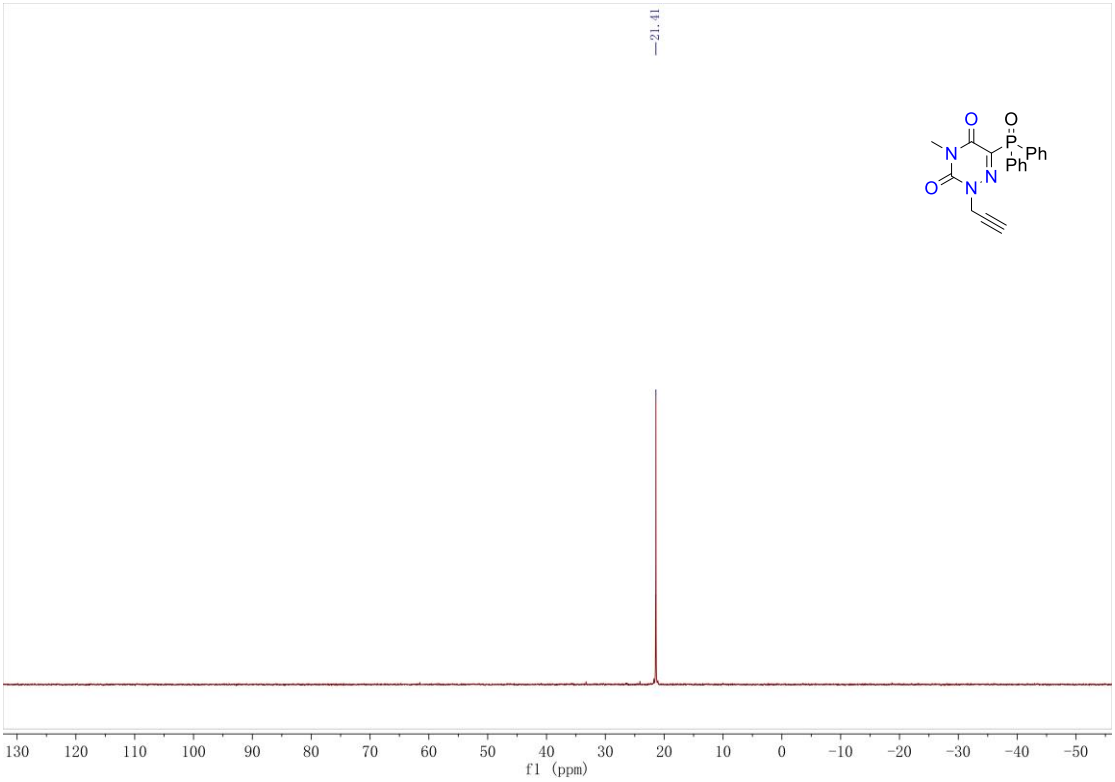
3t-^{13}C


154.90
154.70
148.14
140.34
139.69
132.52
132.50
131.92
131.81
130.26
129.59
129.16
128.55
128.43
120.01
42.90
40.48
f1 (ppm)

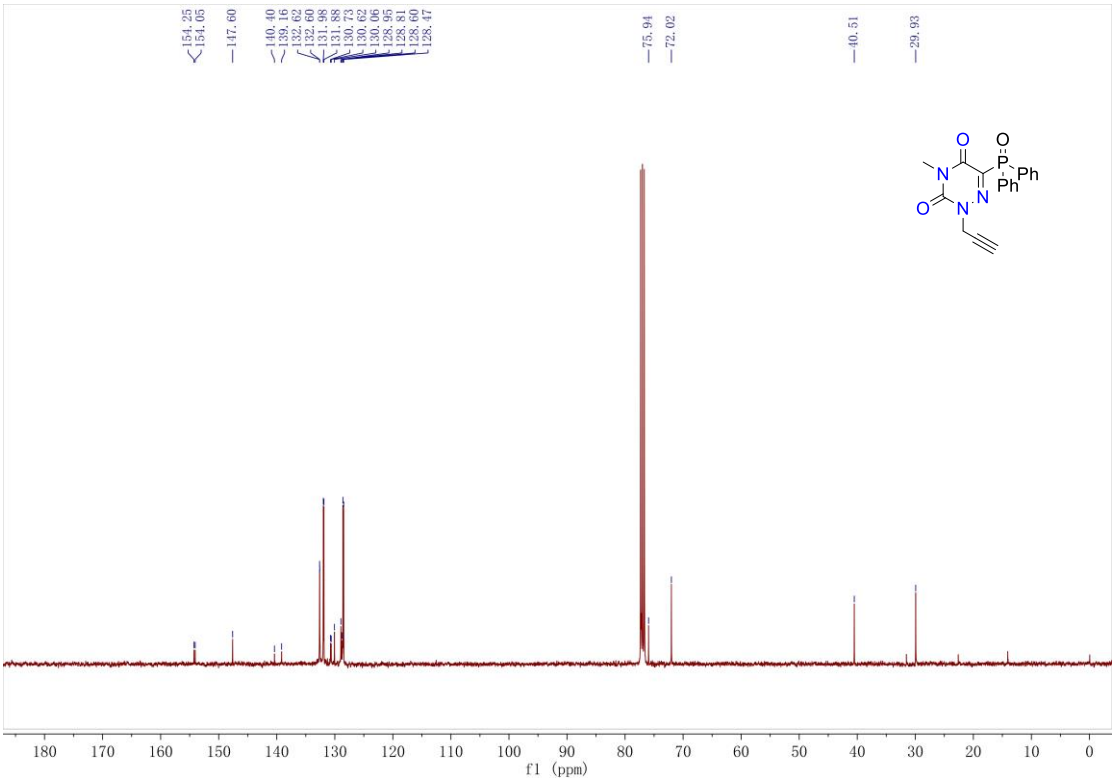
3u-^{1}H


7.88
7.86
7.85
7.83
7.60
7.58
7.56
7.51
7.50
7.49
7.48
7.47
4.60
3.71
2.19
2.18
2.18
2.17
4.07
2.00
4.09
2.01
3.03
1.01
f1 (ppm)

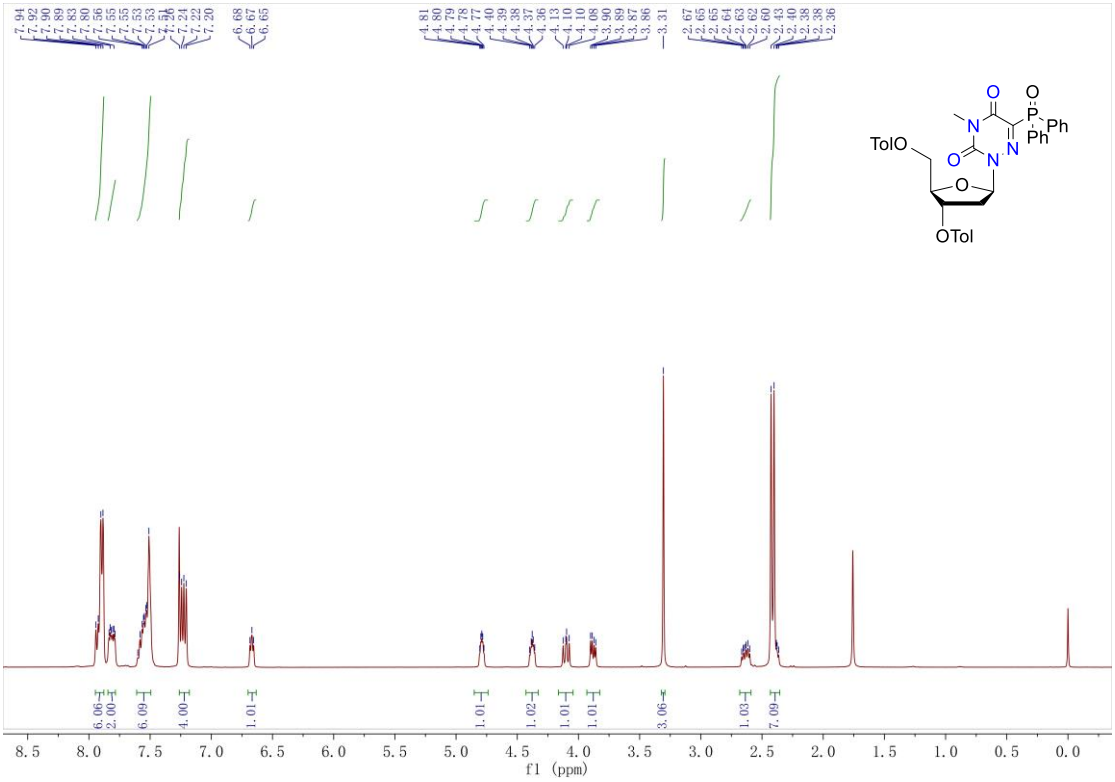
3u-^{31}P


21.41
f1 (ppm)

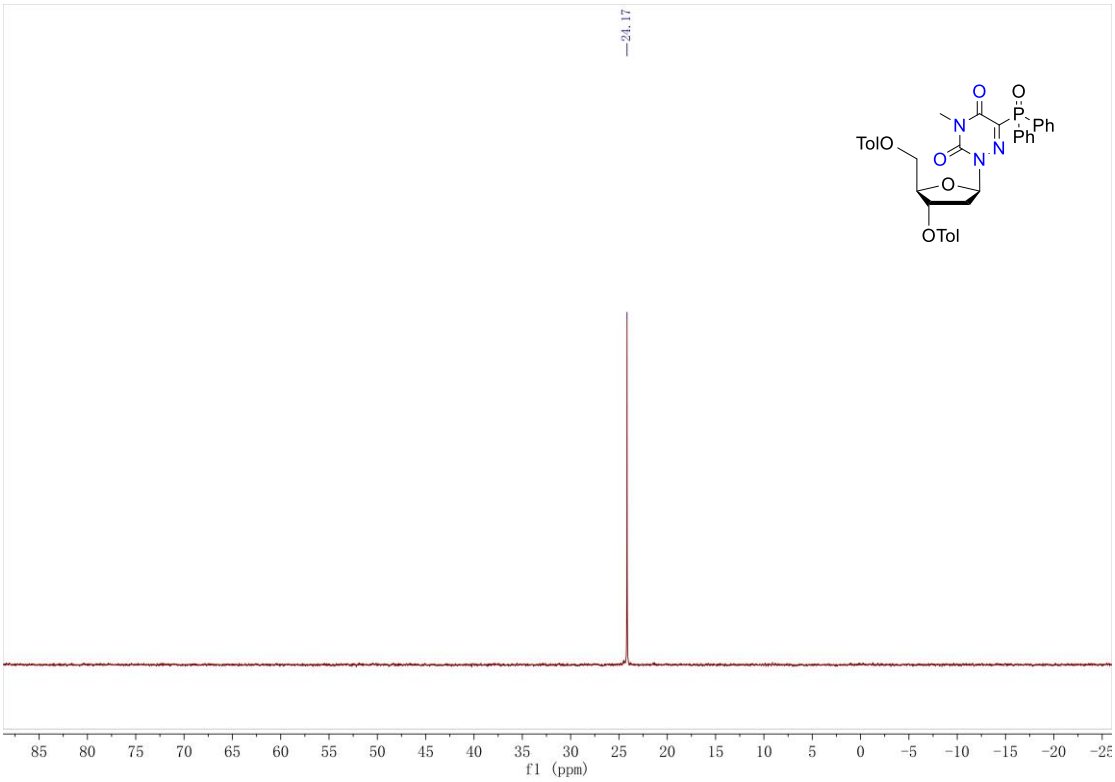
3u-^{13}C


154.25
154.05
147.60
140.40
139.16
132.62
132.60
131.98
131.88
130.73
130.62
130.06
128.95
128.81
128.60
128.47
75.94
72.02
40.51
29.93
O
O
P
Ph
Ph
N
N
O
N
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

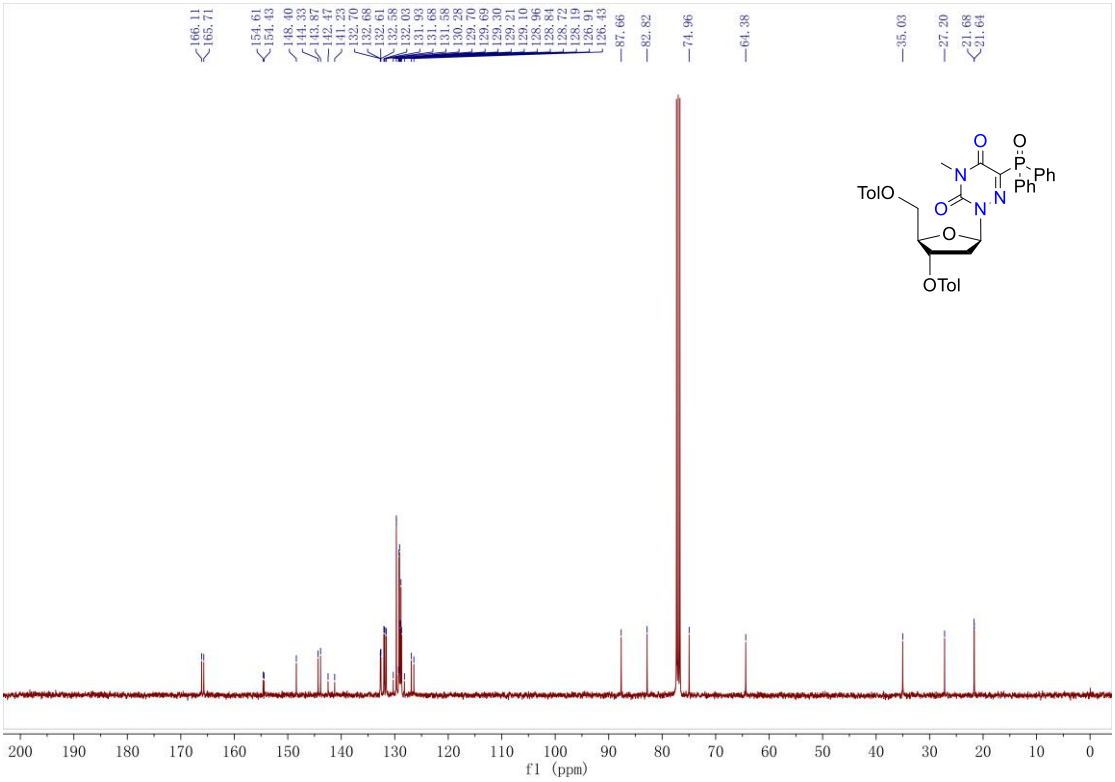
3v-^{1}H


7.94
7.92
7.90
7.89
7.83
7.80
7.56
7.55
7.55
7.53
7.53
7.24
7.22
7.20
6.68
6.67
6.65
4.81
4.80
4.79
4.78
4.77
4.40
4.39
4.38
4.37
4.36
4.13
4.10
4.10
4.08
3.90
3.89
3.87
3.86
3.31
2.67
2.65
2.65
2.64
2.63
2.62
2.60
2.43
2.40
2.38
2.38
2.36
TolO
O
O
P
Ph
Ph
N
N
O
N
O
OTol
6.06
2.00
6.09
4.00
1.01
1.01
1.02
1.01
1.01
3.06
1.03
7.09
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)

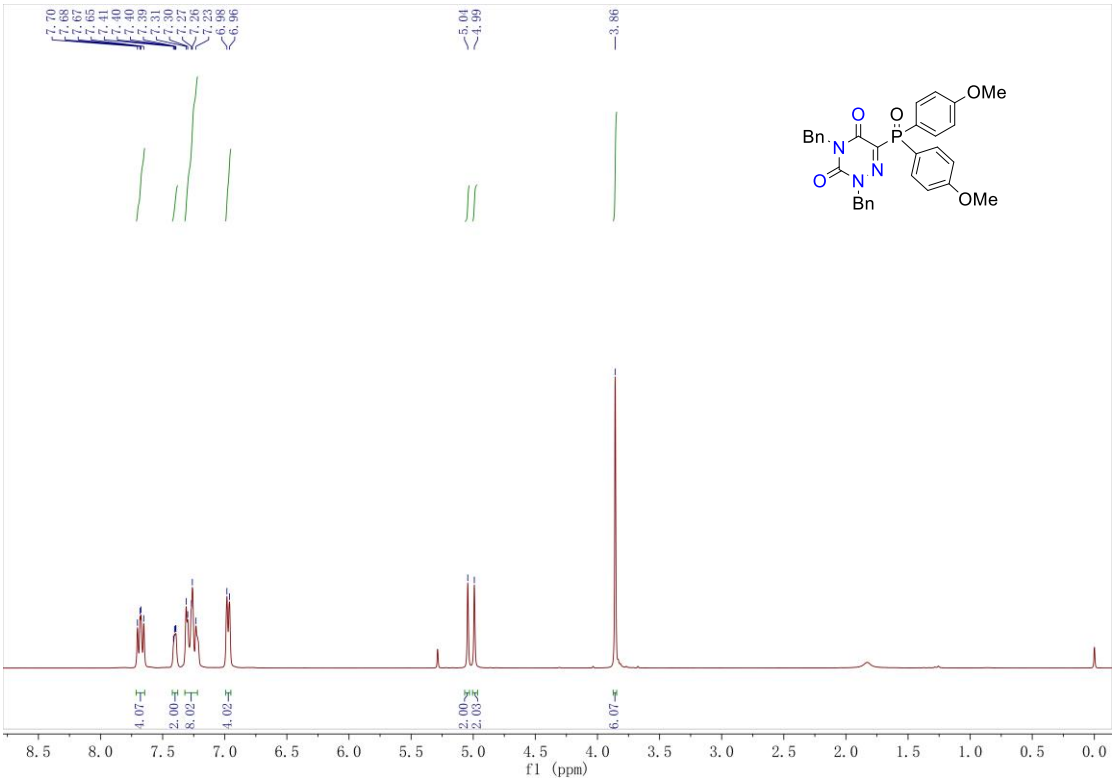
3v-^{31}P


24.17
TolO
OTol
f1 (ppm)

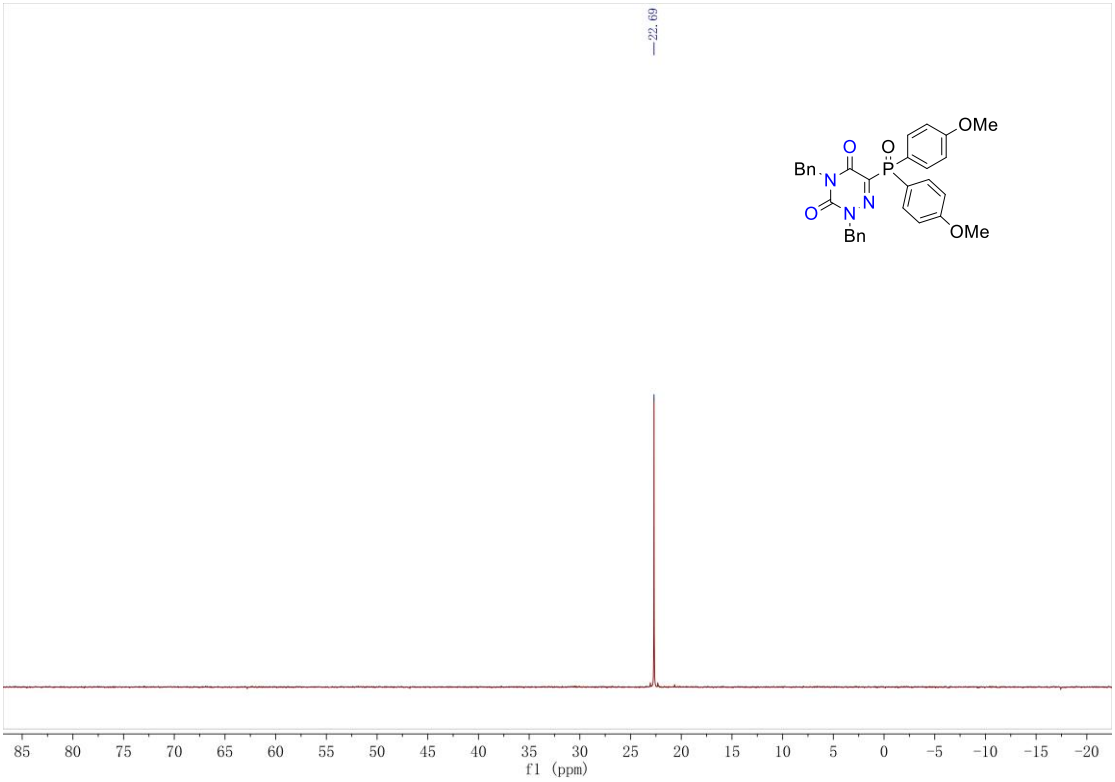
3v-^{13}C


166.11
165.71
154.61
154.43
148.40
144.33
143.87
142.47
141.23
132.70
132.68
132.61
132.58
132.03
131.93
131.68
131.58
130.28
129.70
129.69
129.30
129.21
129.10
128.96
128.84
128.72
128.19
126.91
126.43
87.66
82.82
74.96
64.38
35.03
27.20
21.68
21.64
TolO
OTol
f1 (ppm)

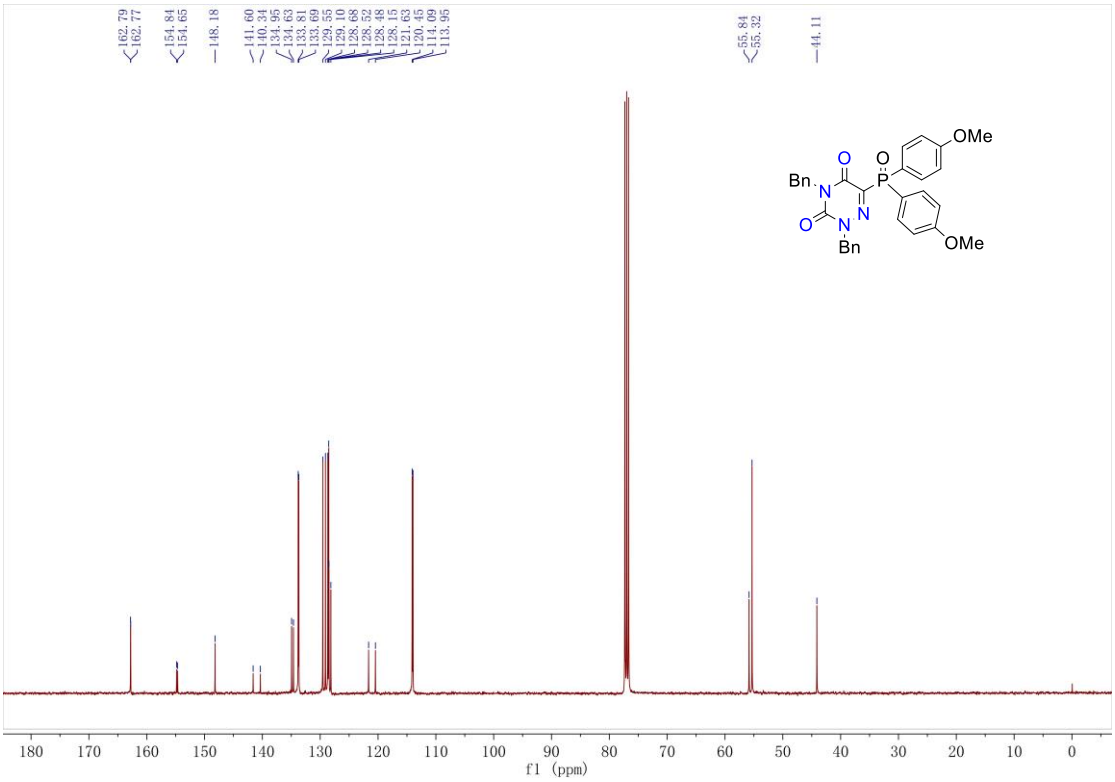
3w-^{1}H


7.70
7.68
7.67
7.65
7.41
7.40
7.40
7.39
7.31
7.30
7.27
7.26
7.23
6.98
6.96
5.04
4.99
3.86
OMe
O
O
P
Bn
N
N
O
N
N
Bn
OMe
4.07
2.00
8.02
4.02
2.00
2.03
6.07
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)

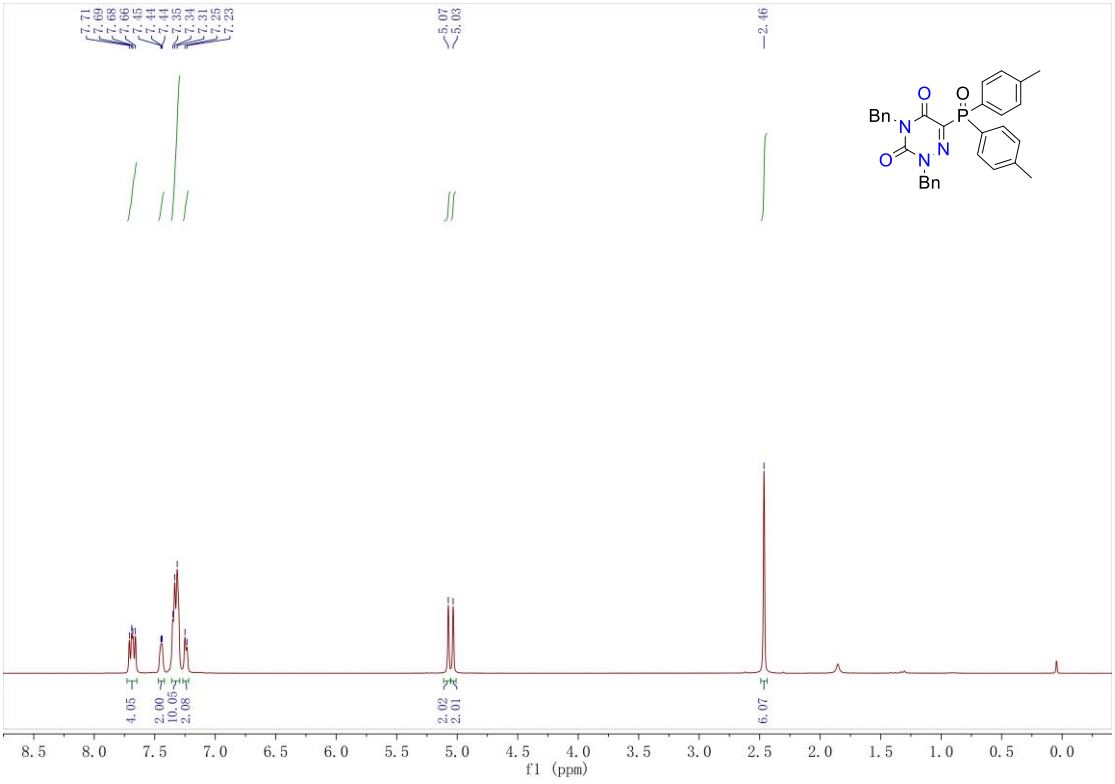
3w-^{31}P


22.69
OMe
O
O
P
Bn
N
N
O
N
N
Bn
OMe
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
−5
−10
−15
−20
f1 (ppm)

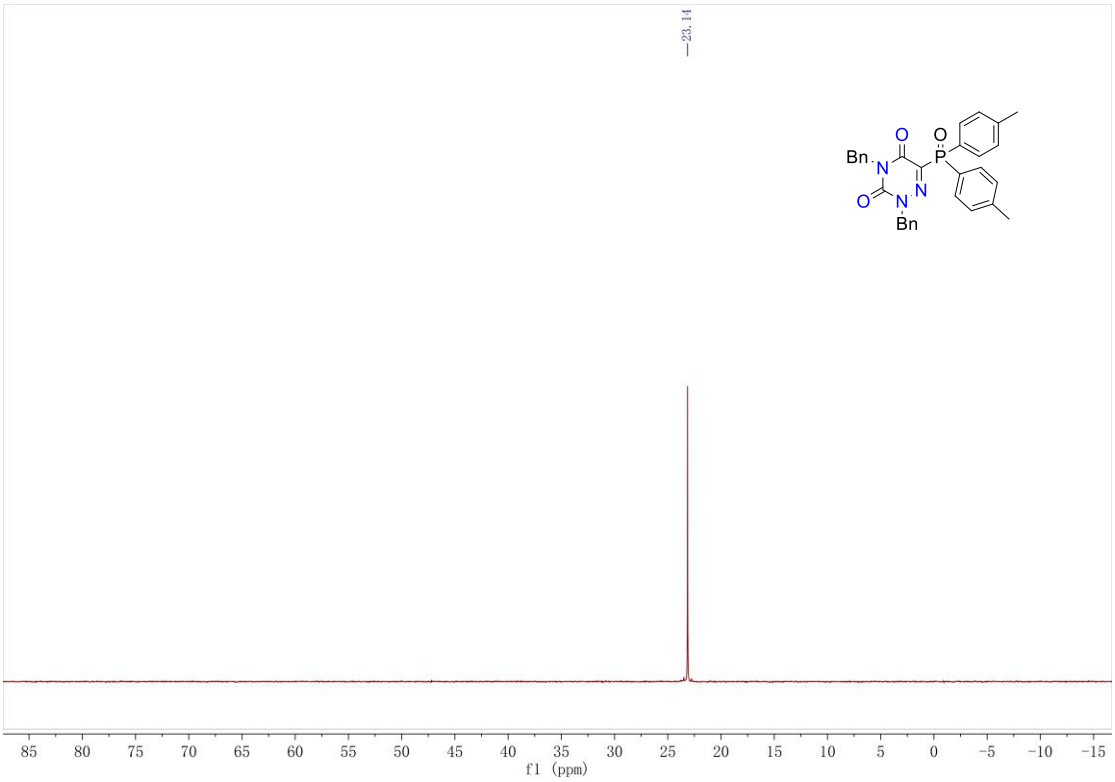
3w-^{13}C


162.79
162.77
154.84
154.65
148.18
141.60
140.34
134.95
134.63
133.81
133.69
129.55
129.10
128.68
128.52
128.48
128.15
121.63
120.45
114.09
113.95
55.84
55.32
44.11
OMe
OMe
Bn
Bn
f1 (ppm)

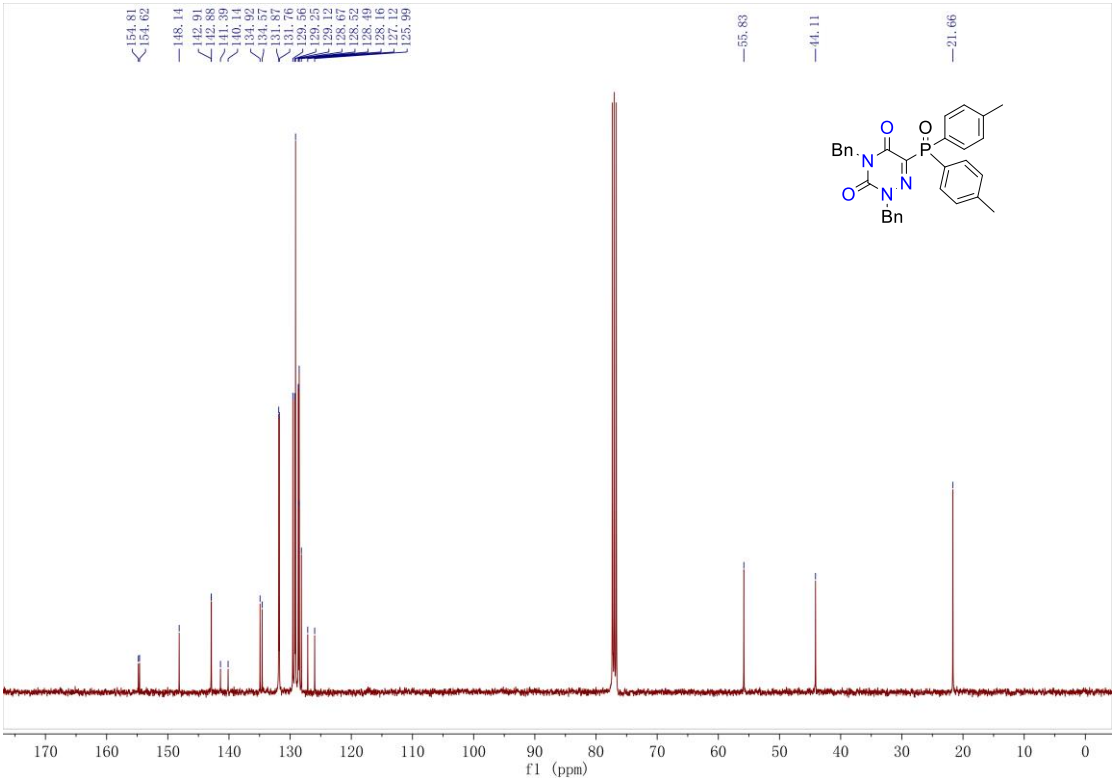
3x-^{1}H


7.71
7.69
7.68
7.66
7.45
7.44
7.44
7.35
7.34
7.31
7.25
7.23
5.07
5.03
2.46
Bn
Bn
4.05
2.00
10.05
2.08
2.02
2.01
6.07
f1 (ppm)

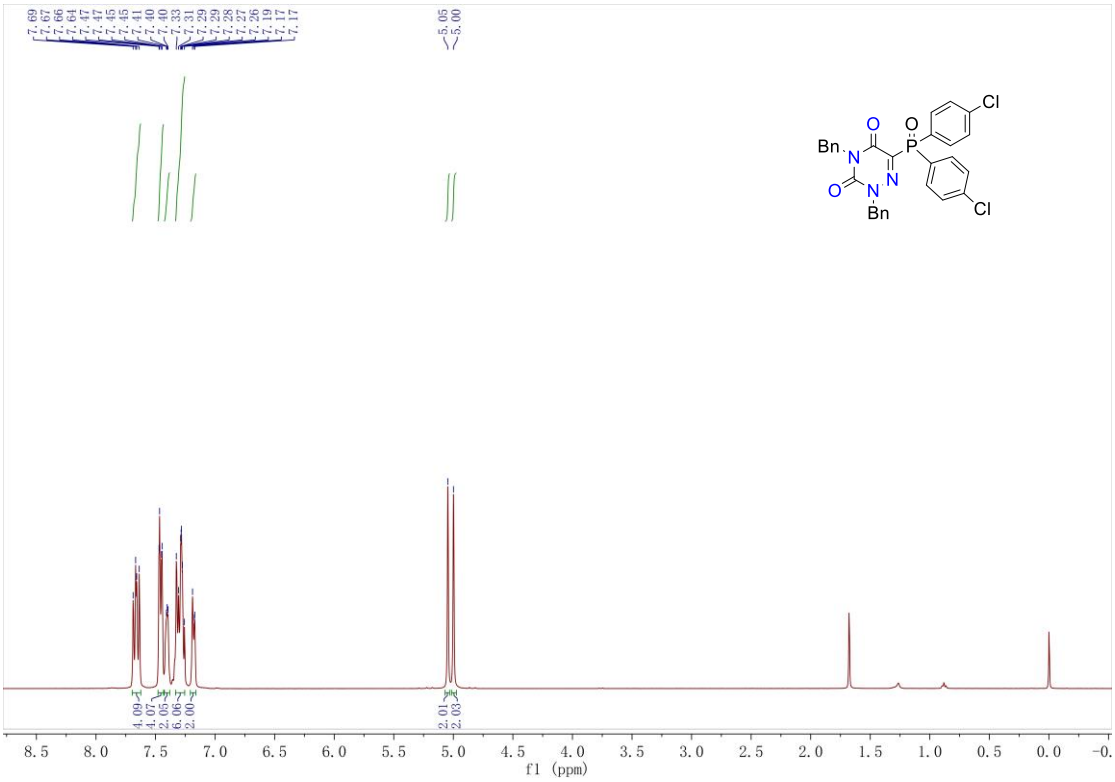
3x-^{31}P


23.14
Bn
O
P
N
O
N
Bn
85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 −15
f1 (ppm)

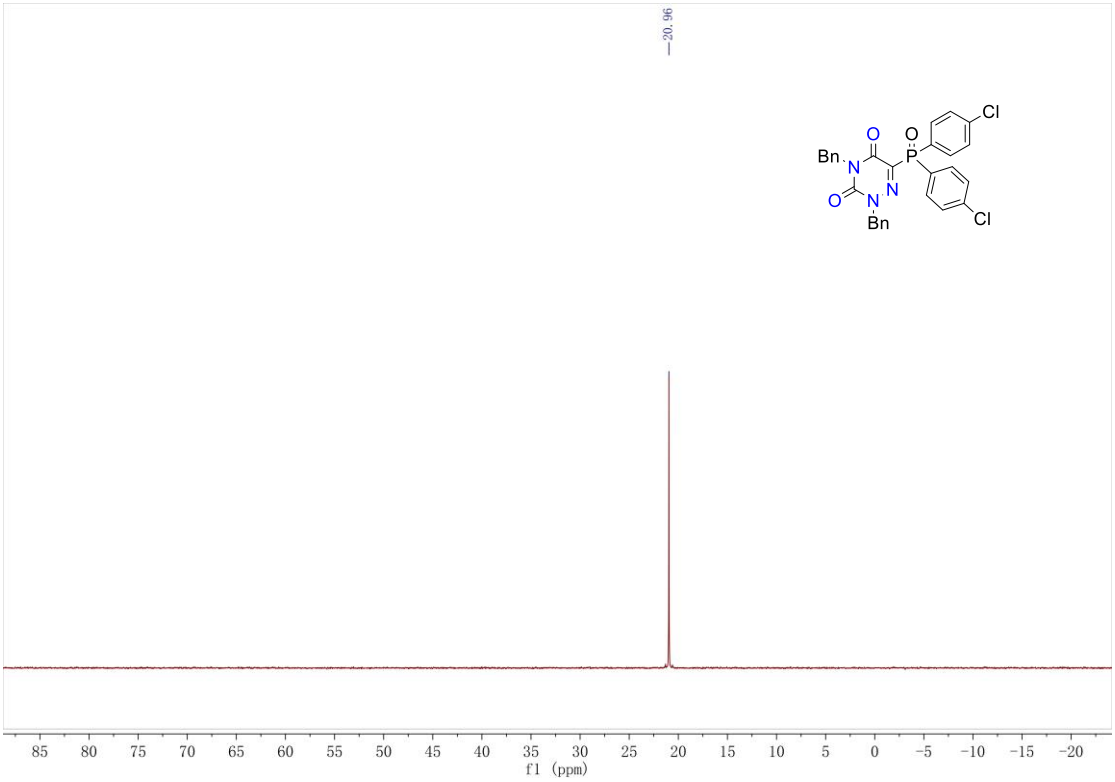
3x-^{13}C


154.81
154.62
148.14
142.91
142.88
141.39
140.14
134.92
134.57
131.87
131.76
129.56
129.25
129.12
128.67
128.52
128.49
128.16
127.12
125.99
55.83
44.11
21.66
Bn
O
P
N
O
N
Bn
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

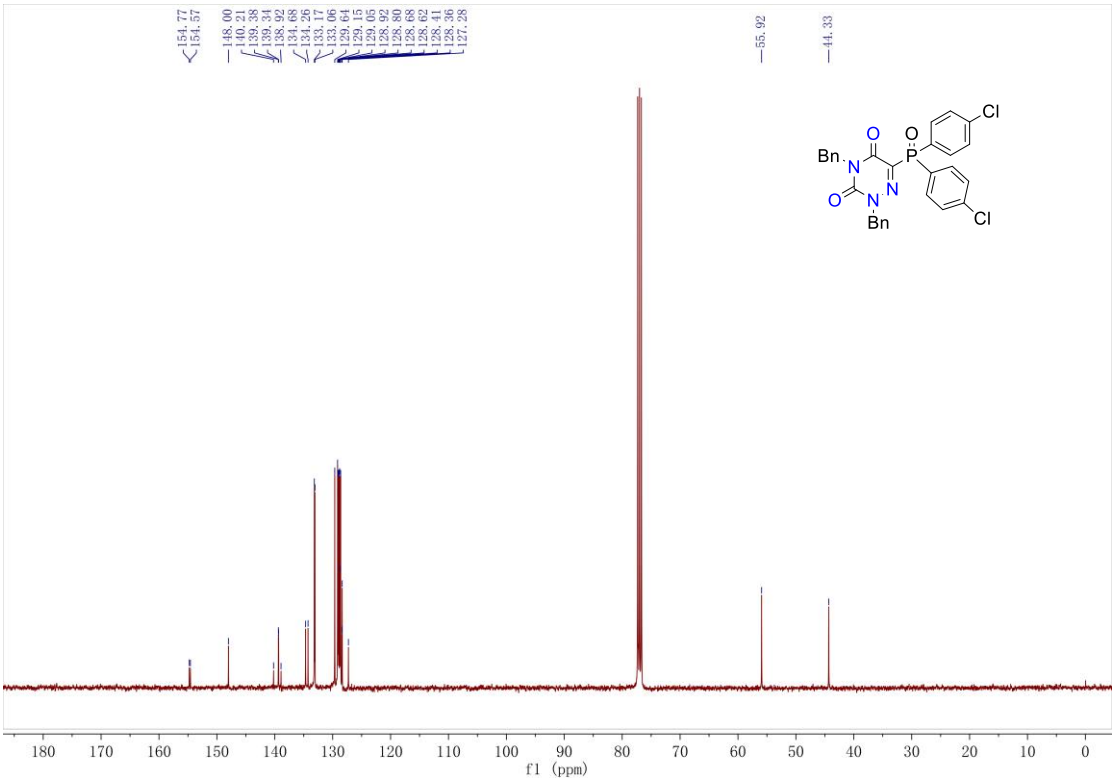
3y-^{1}H


7.69
7.67
7.66
7.64
7.47
7.45
7.41
7.40
7.33
7.31
7.29
7.28
7.27
7.26
7.19
7.17
5.05
5.00
Cl
O
O
Bn
N
P
O
N
N
Bn
Cl
4.09
4.07
2.05
6.06
2.00
2.01
2.03
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)

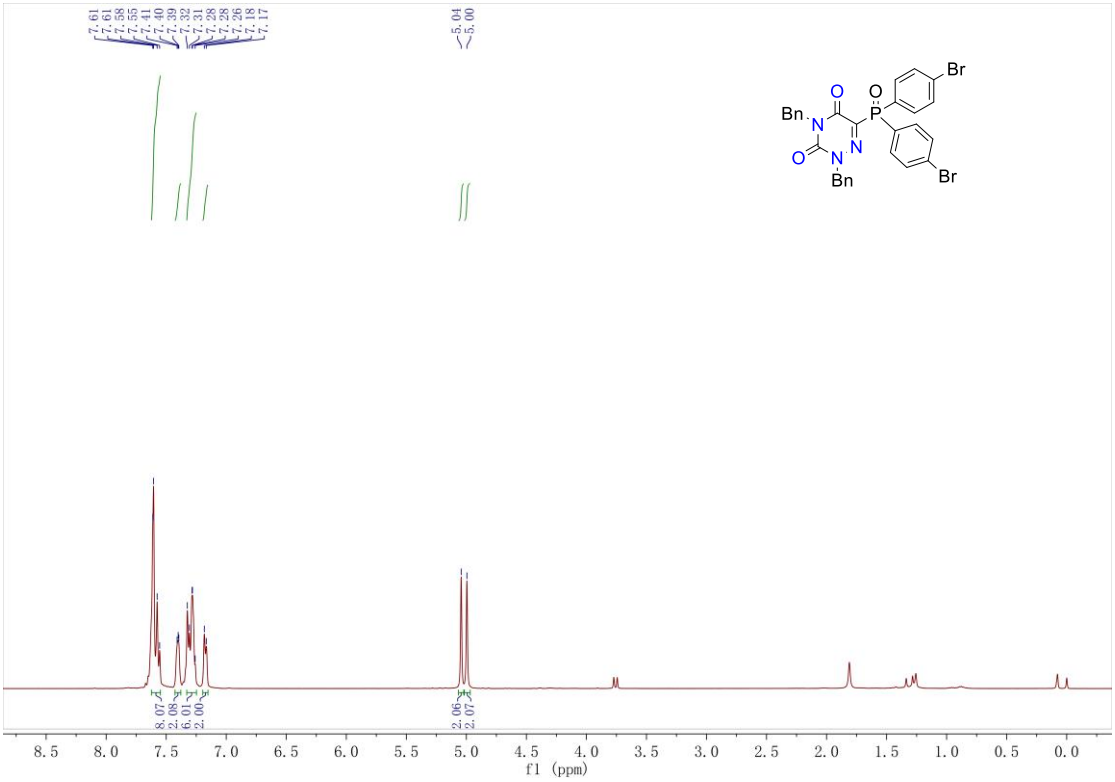
3y-^{31}P


20.96
Cl
O
O
Bn
N
P
O
N
N
Bn
Cl
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
−5
−10
−15
−20
f1 (ppm)

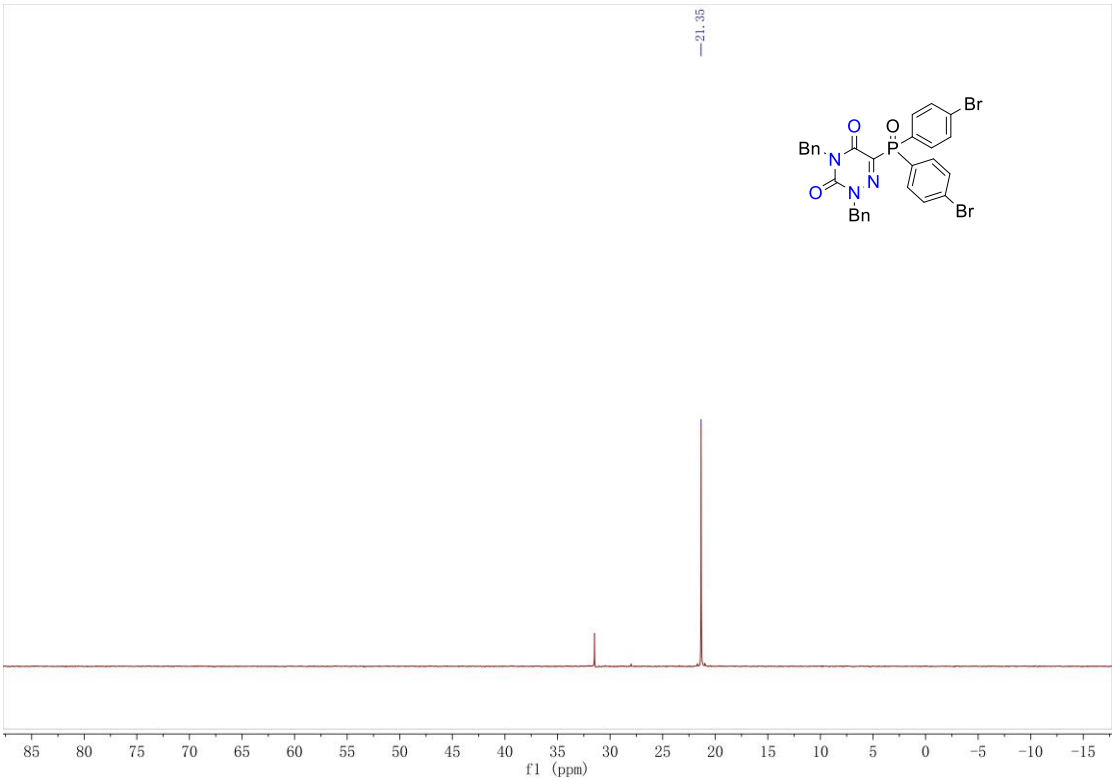
3y-^{13}C


154.77
154.57
148.00
140.21
140.08
139.34
138.92
134.68
134.26
133.17
133.06
129.64
129.15
129.05
128.92
128.80
128.68
128.62
128.41
128.36
127.28
55.92
44.33
Cl
Cl
Bn
Bn
f1 (ppm)

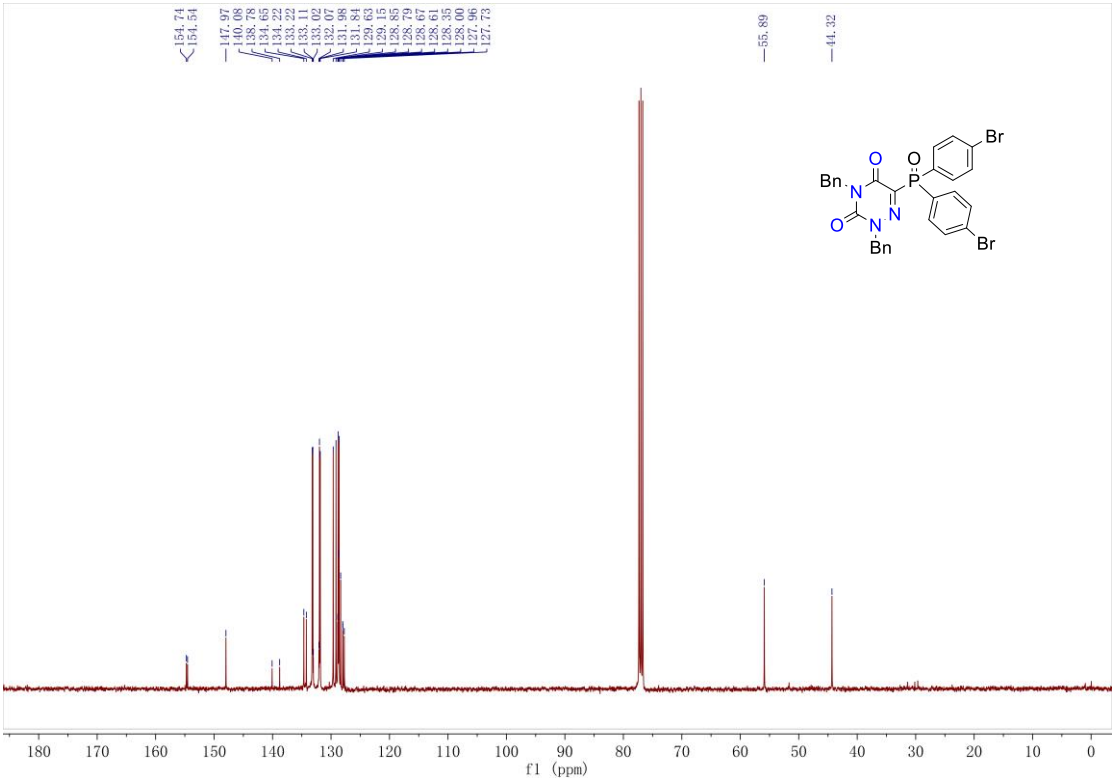
3z-^{1}H


7.61
7.61
7.58
7.55
7.41
7.40
7.39
7.32
7.31
7.28
7.26
7.18
7.17
5.04
5.00
Br
Br
Bn
Bn
8.07
2.08
6.01
2.00
2.06
2.07
f1 (ppm)

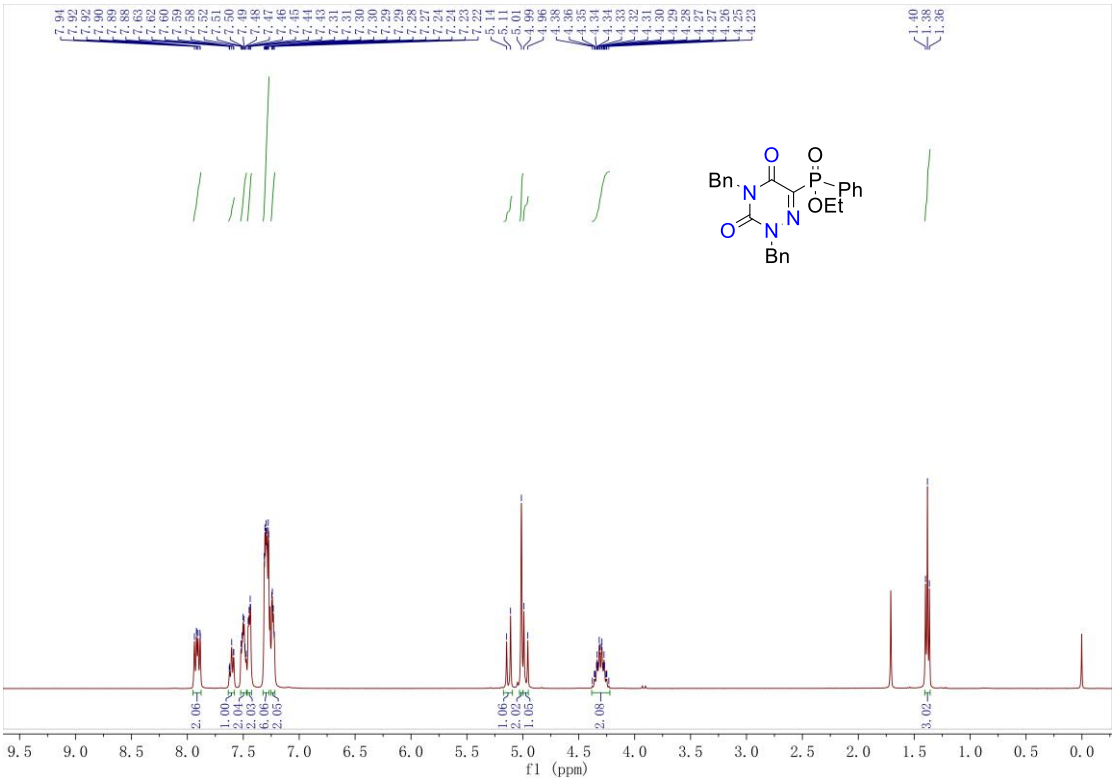
3z-^{31}P


21.35
Br
O
O
Bn
N
P
N
O
N
Bn
Br
85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5 -10 -15
f1 (ppm)

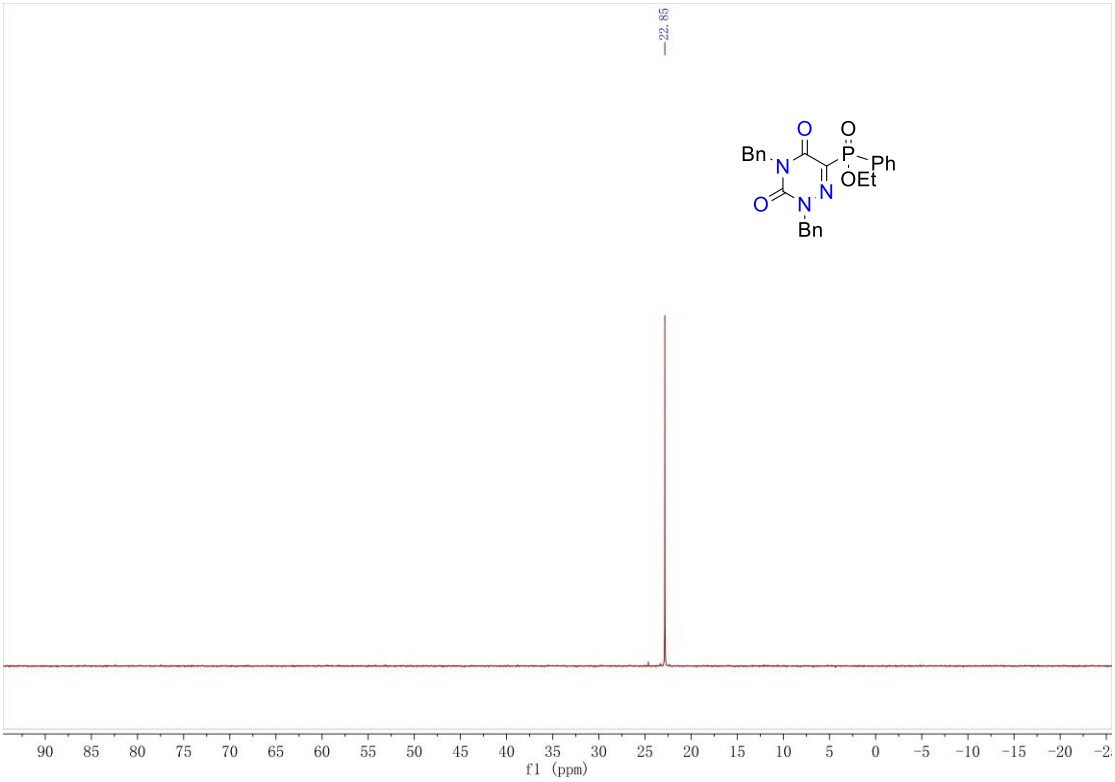
3z-^{13}C


154.74
154.54
147.97
140.08
138.78
134.65
134.22
133.22
133.11
133.02
132.07
131.98
131.84
129.63
129.15
128.85
128.79
128.67
128.61
128.35
128.00
127.96
127.73
55.89
44.32
Br
O
O
Bn
N
P
N
O
N
Bn
Br
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

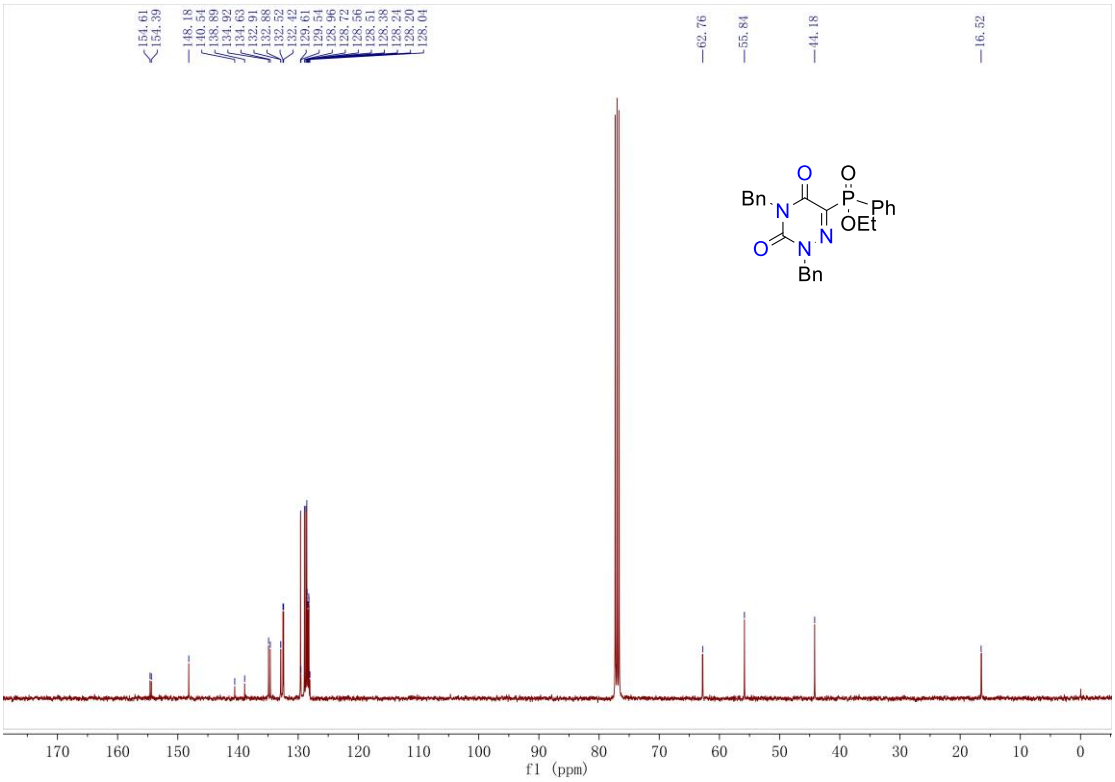
3aa-^{1}H



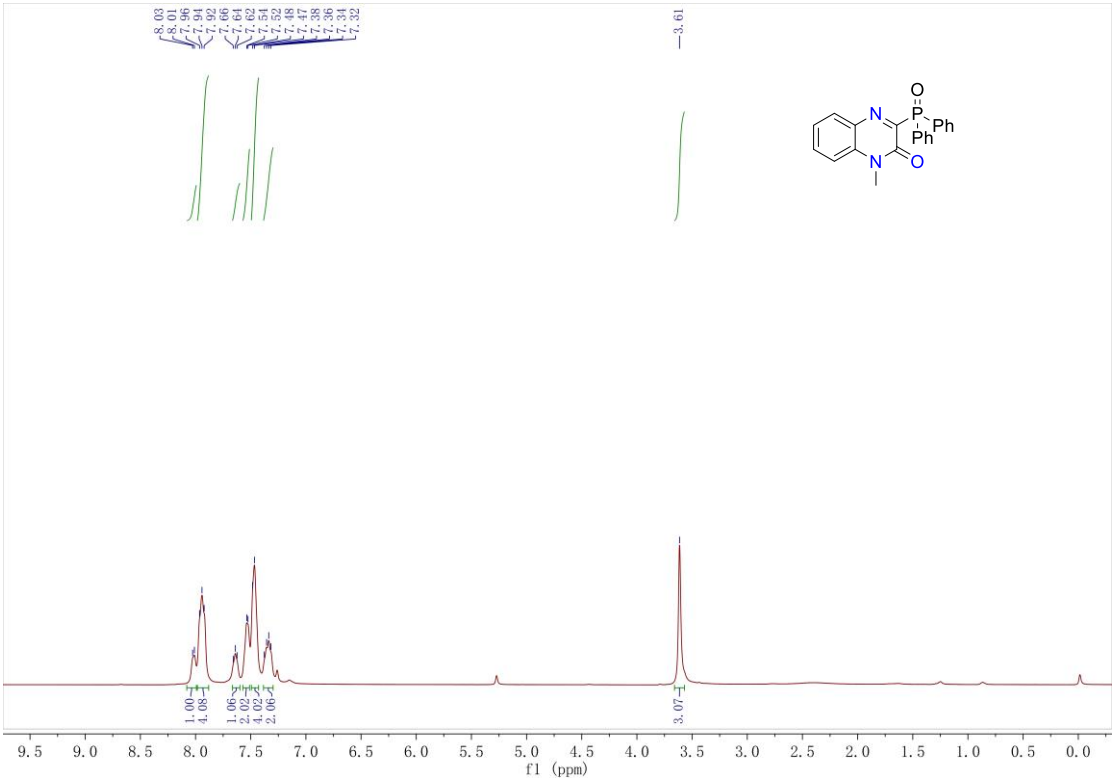
3aa-^{31}P



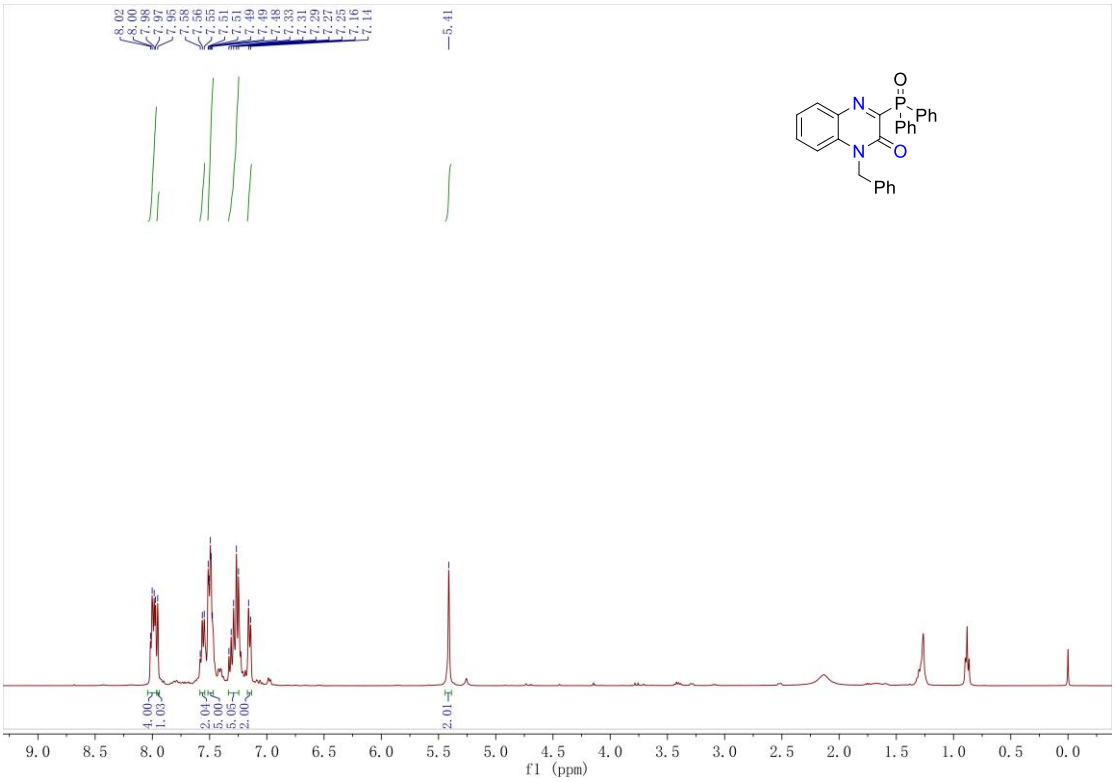
3aa-^{13}C



3ab-^{1}H



3ac-^{1}H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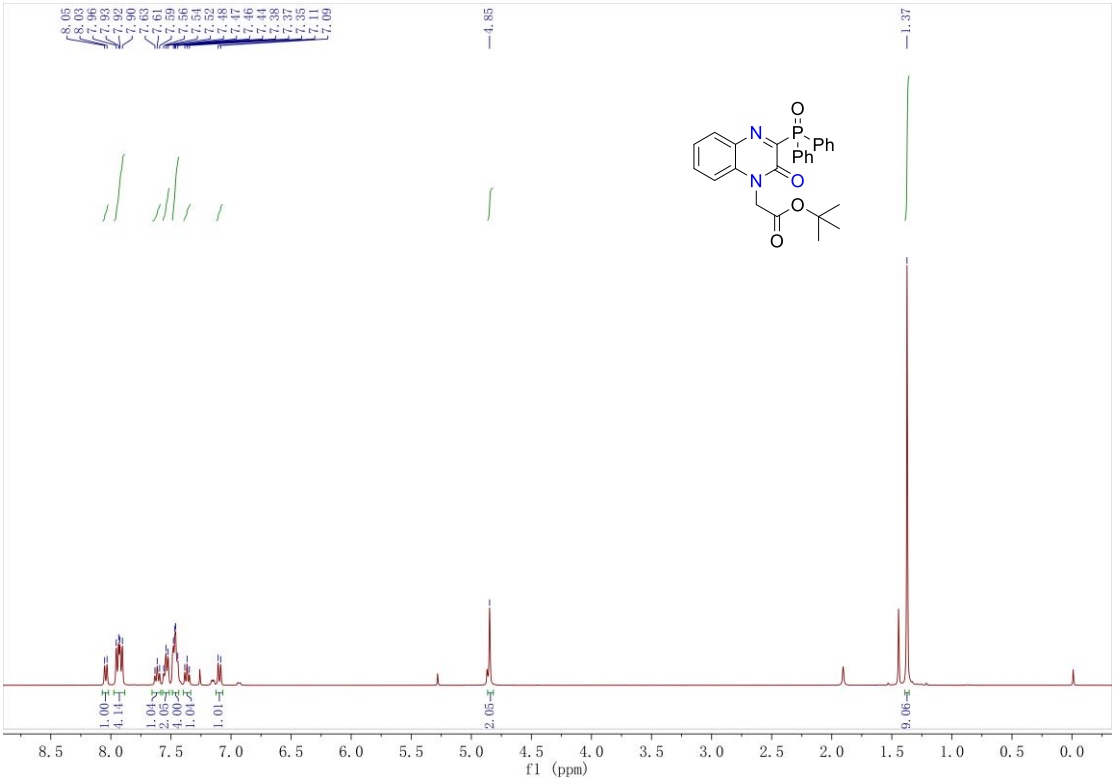
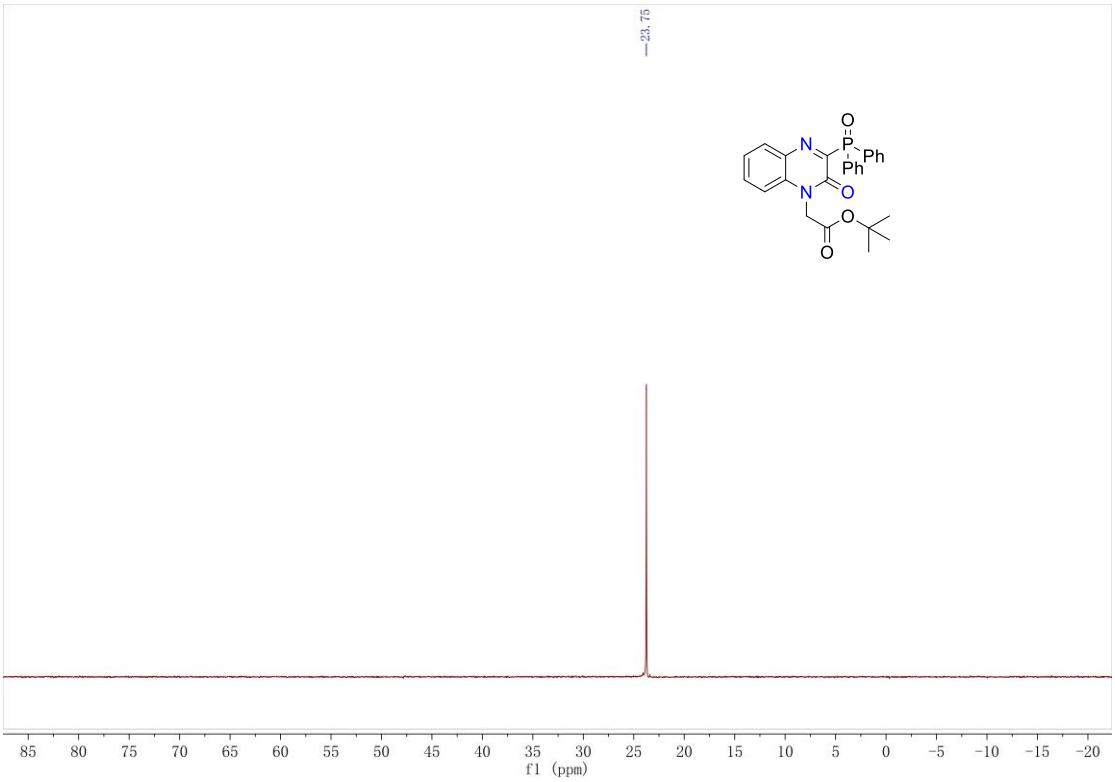
8.00
7.98
7.97
7.95
7.58
7.56
7.55
7.51
7.49
7.48
7.33
7.31
7.29
7.27
7.25
7.16
7.14
5.41
O
P
Ph
Ph
N
N
O
Ph
4.00
1.03
2.04
5.00
5.05
2.00
2.01
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

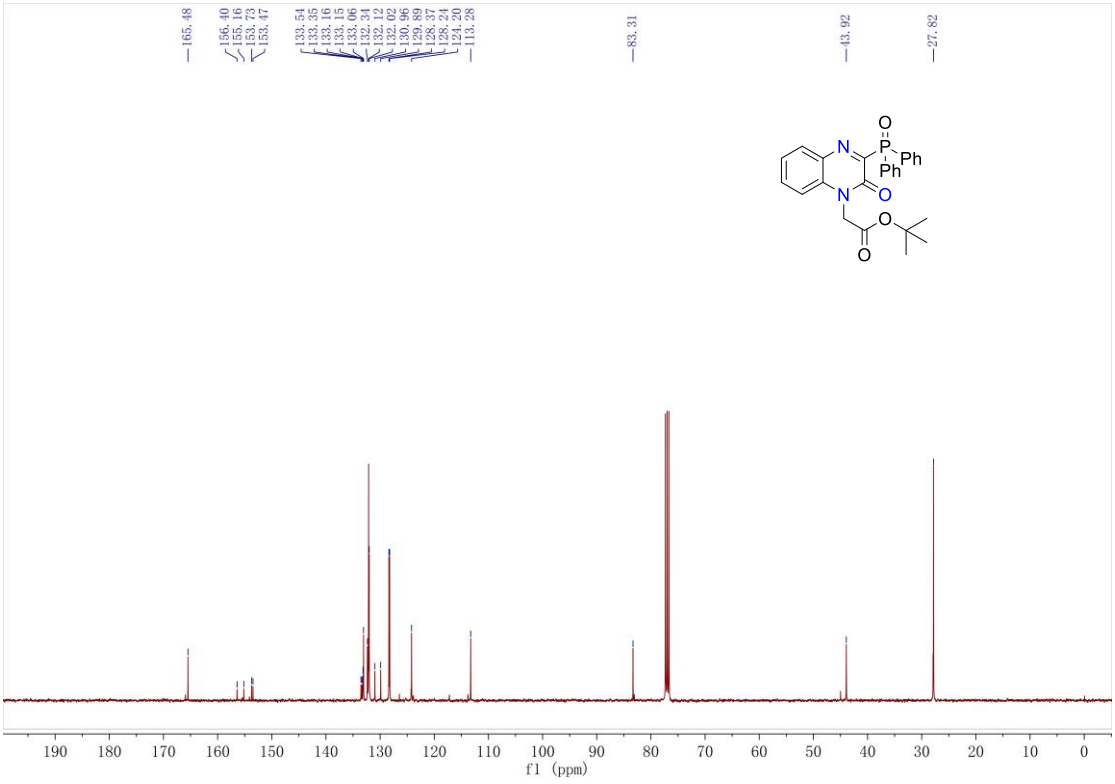
3ad-^{1}H


8.05
8.03
7.96
7.93
7.92
7.90
7.63
7.61
7.59
7.56
7.54
7.52
7.48
7.47
7.46
7.44
7.38
7.37
7.35
7.11
7.09
4.85
1.37
O
P
Ph
Ph
N
N
O
O
O
1.00
4.14
1.04
2.05
4.00
1.04
1.01
2.05
9.06
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)

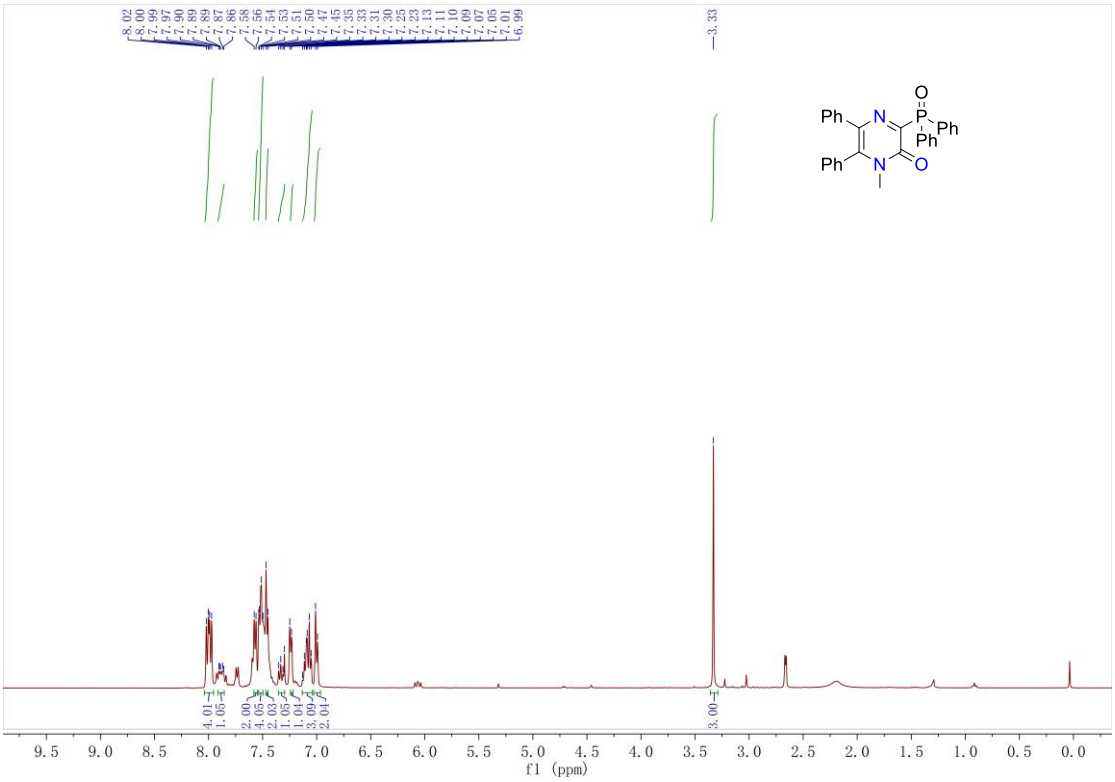
3ad-^{31}P



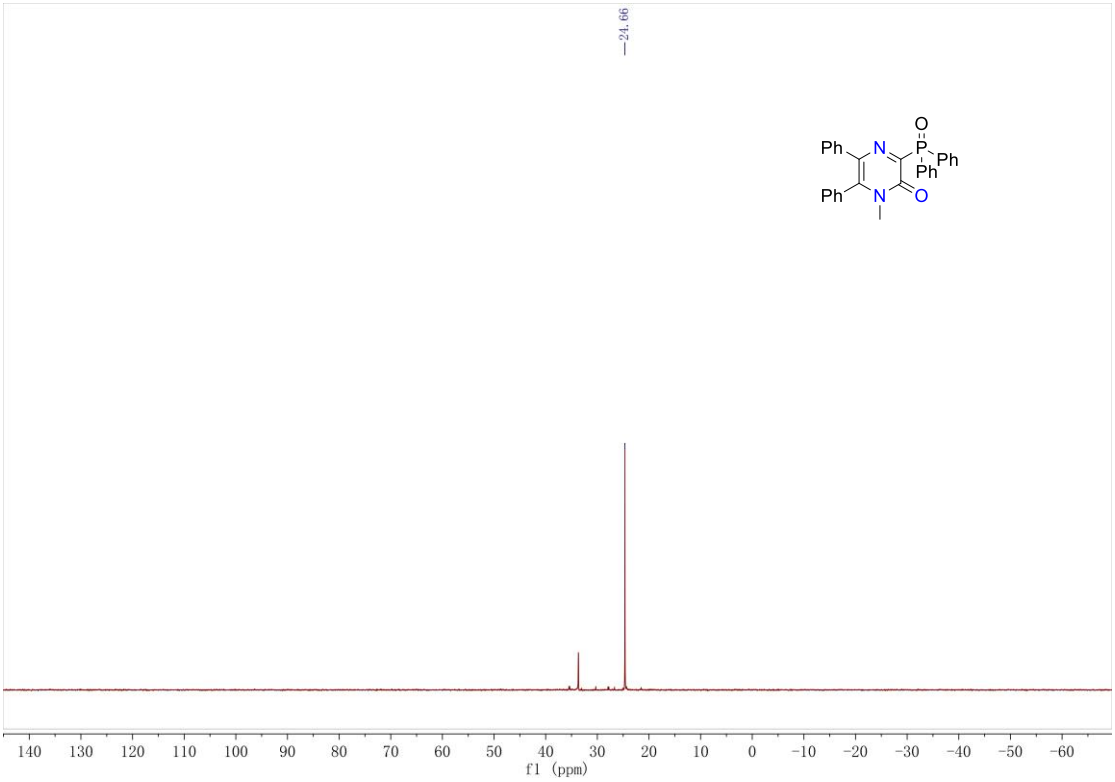
3ad-^{13}C



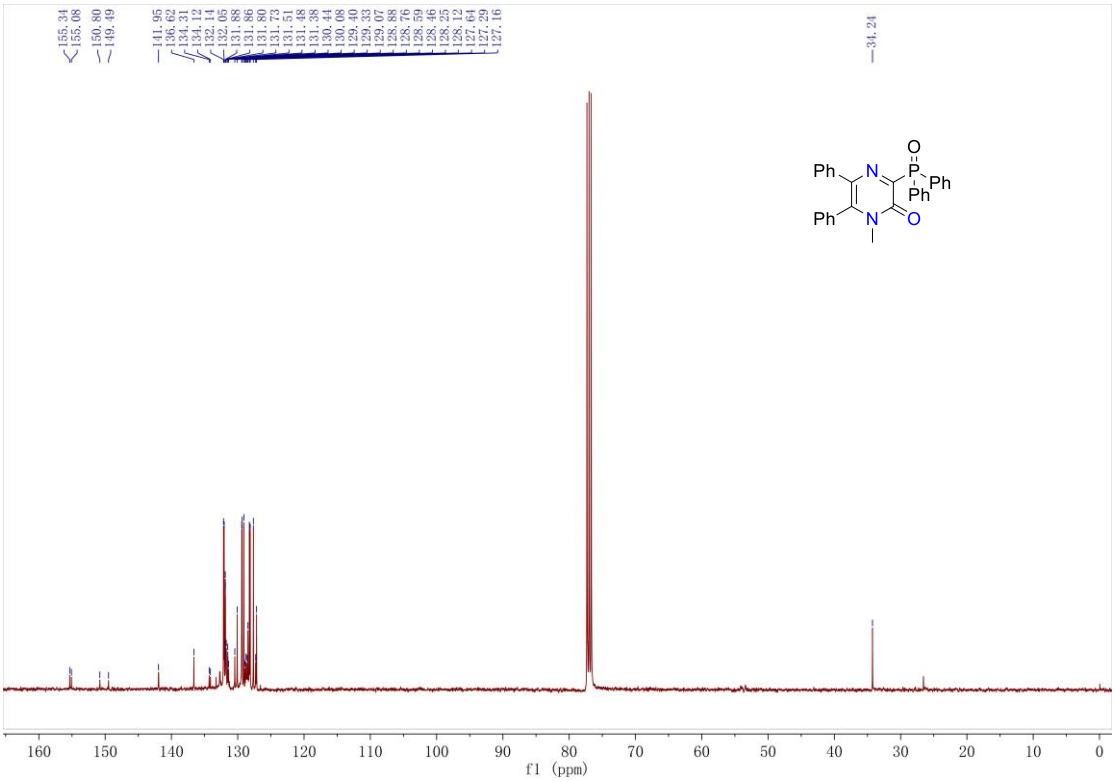
3ae-^{1}H



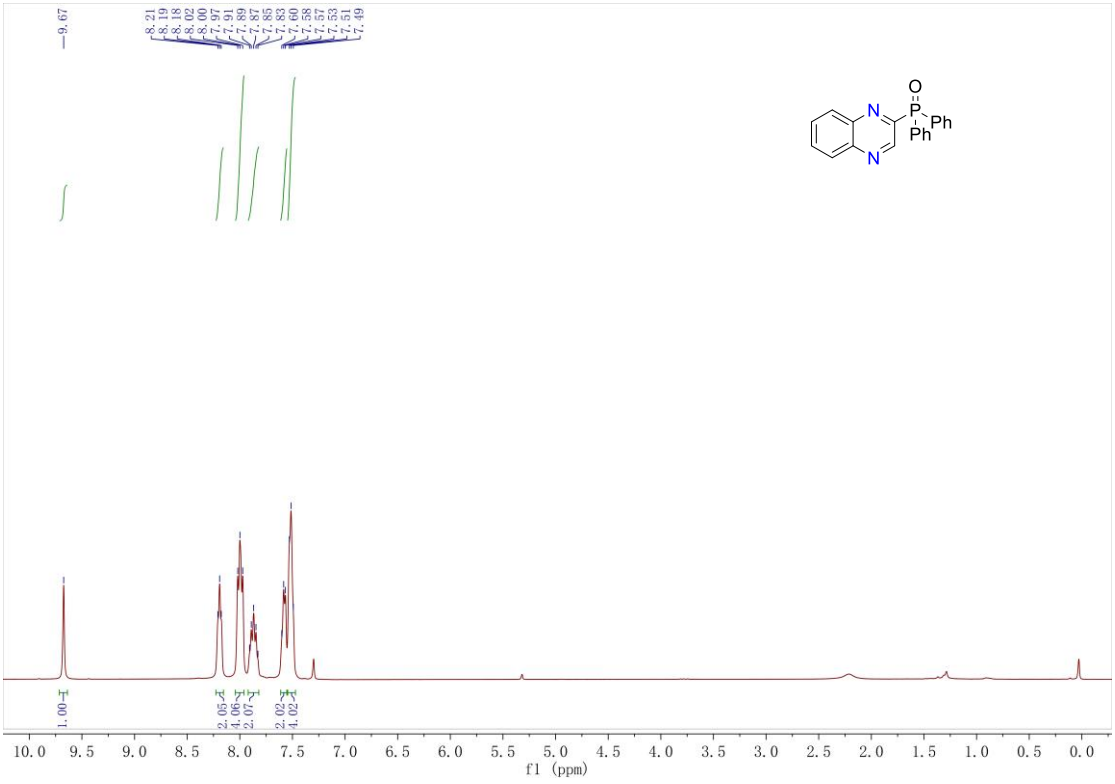
3ae-^{31}P



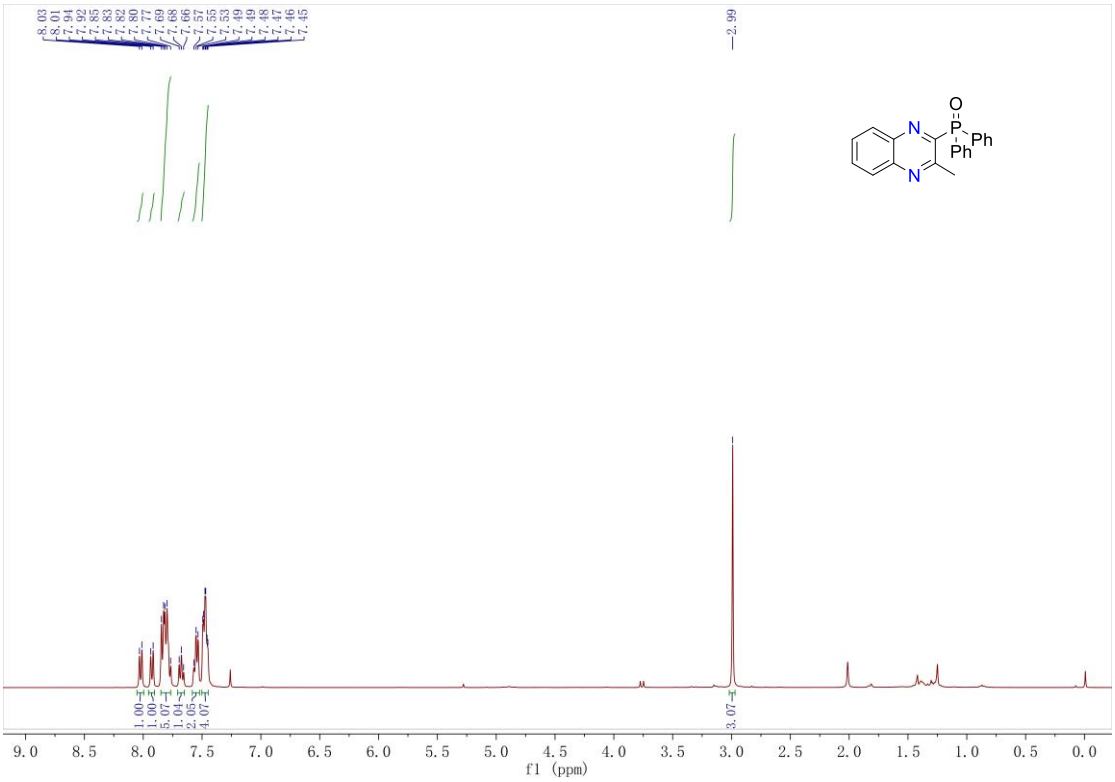
3ae-^{13}C



3af-^{1}H



3ag-^{1}H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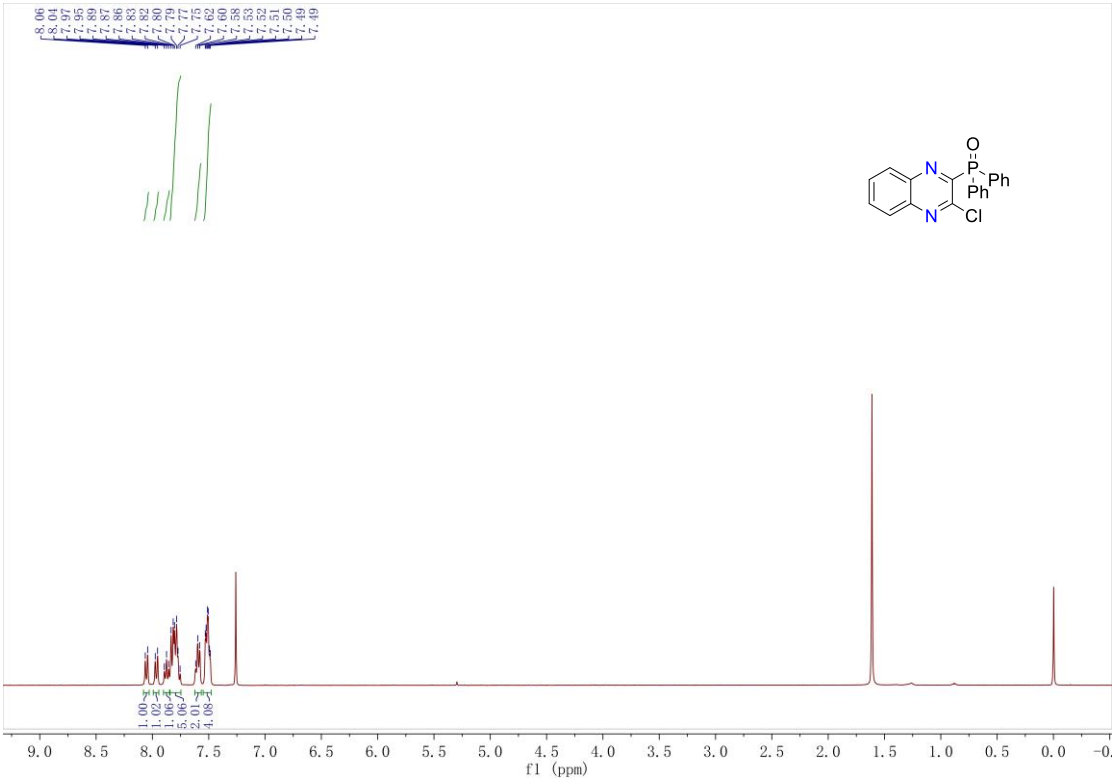
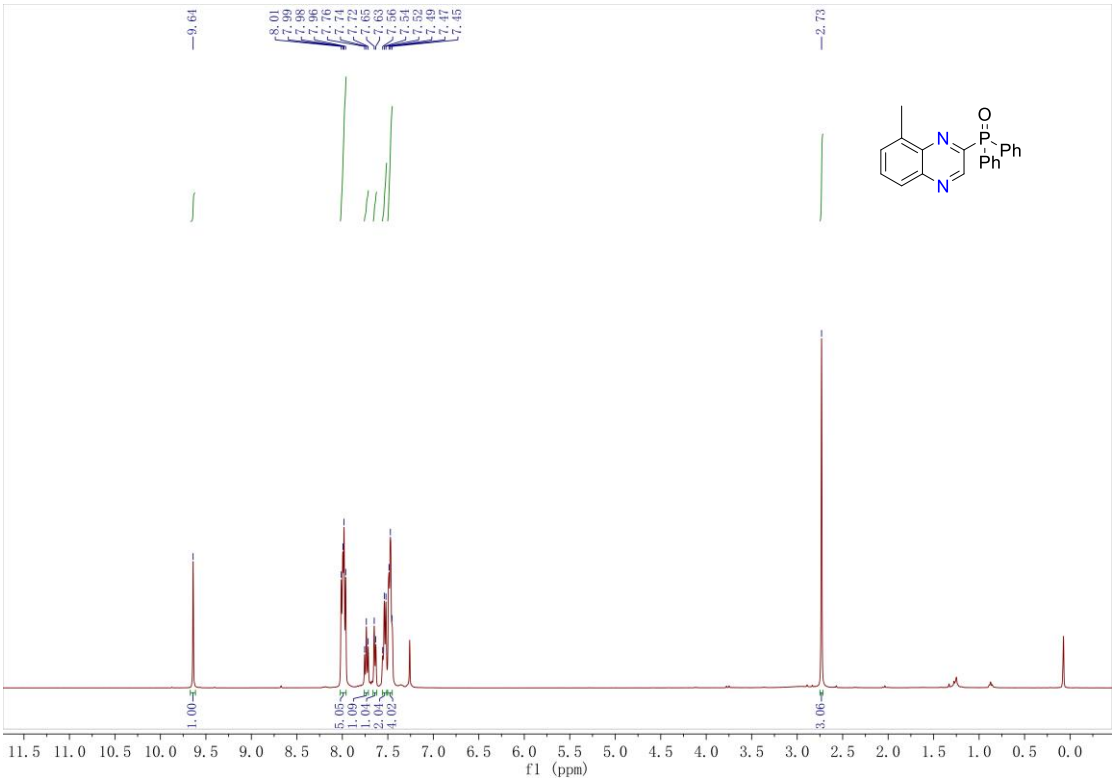
8.01
7.94
7.92
7.85
7.83
7.82
7.80
7.77
7.69
7.68
7.66
7.57
7.55
7.53
7.49
7.49
7.48
7.47
7.46
7.45
2.99
O
P
Ph
Ph
N
N
1.00
1.00
5.07
1.04
2.05
4.07
3.07
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

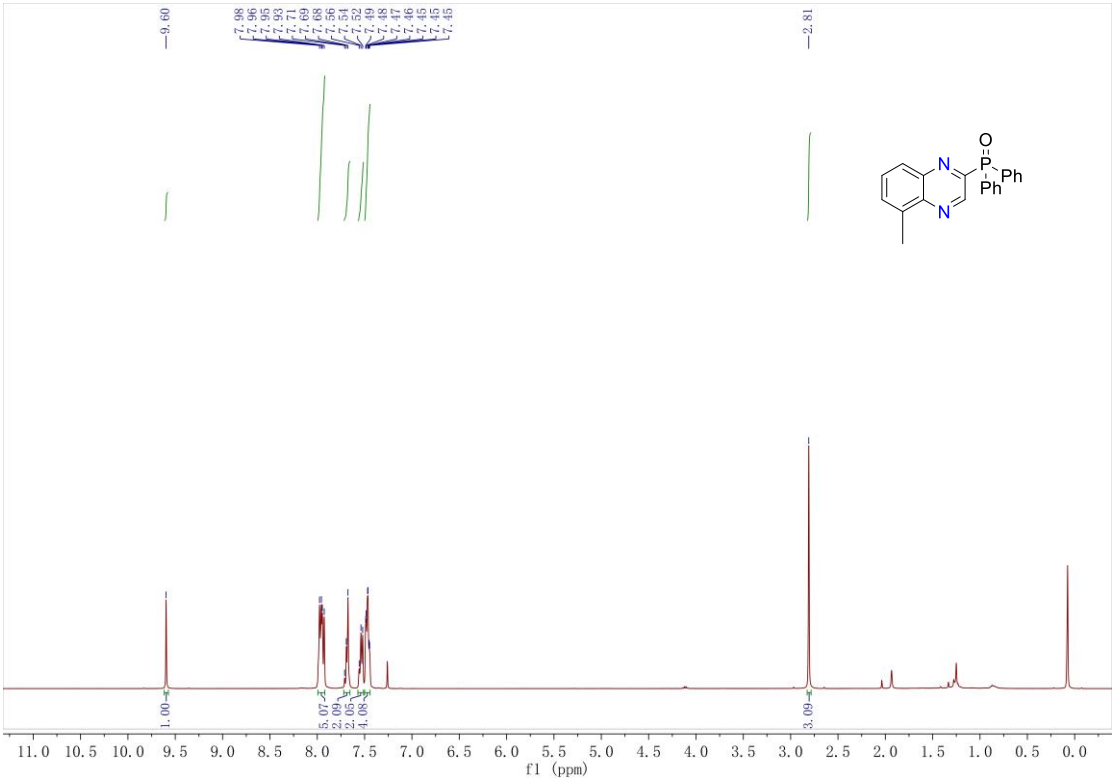
3ah-^{1}H


8.06
8.04
7.97
7.95
7.89
7.87
7.86
7.83
7.82
7.80
7.79
7.77
7.75
7.62
7.60
7.58
7.53
7.52
7.51
7.50
7.49
7.49
O
P
Ph
Ph
N
N
Cl
1.00
1.22
1.06
4.14
5.06
2.01
4.08
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.
f1 (ppm)

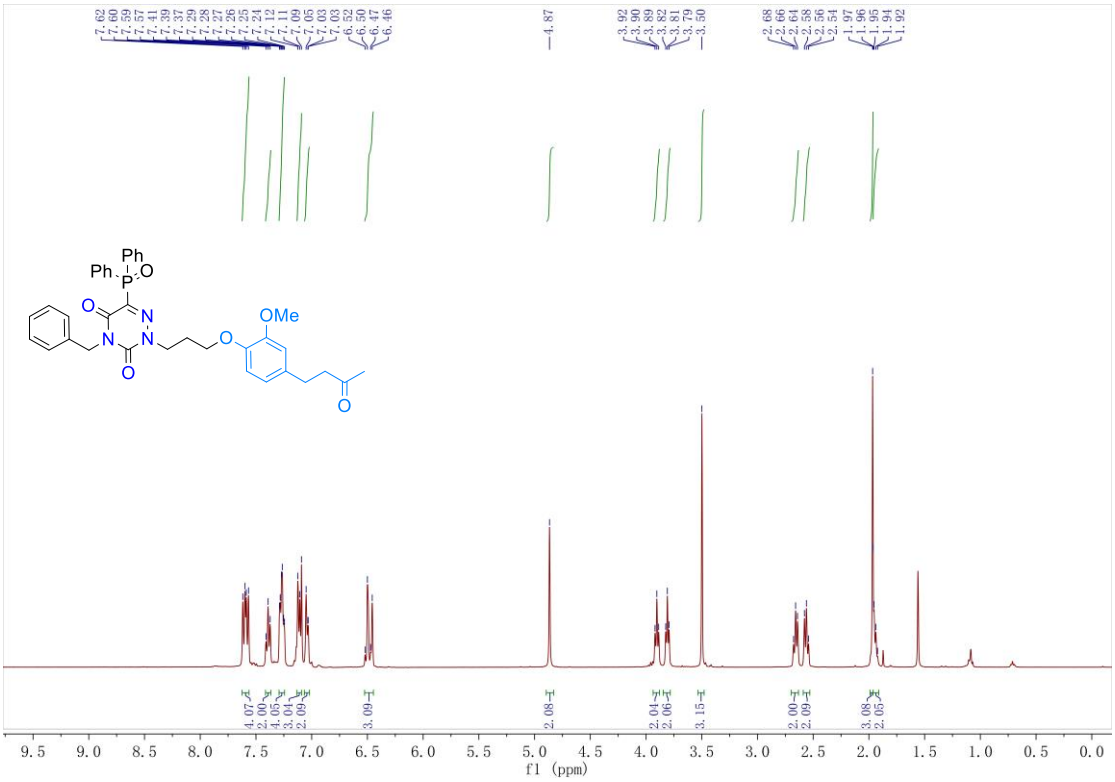
3ai-^{1}H


9.64
8.01
7.99
7.98
7.96
7.76
7.74
7.72
7.65
7.63
7.56
7.54
7.52
7.49
7.47
7.45
2.73
O
P
Ph
Ph
N
N
1.00
5.05
1.09
1.04
2.01
4.02
3.06
11.5 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
f1 (ppm)

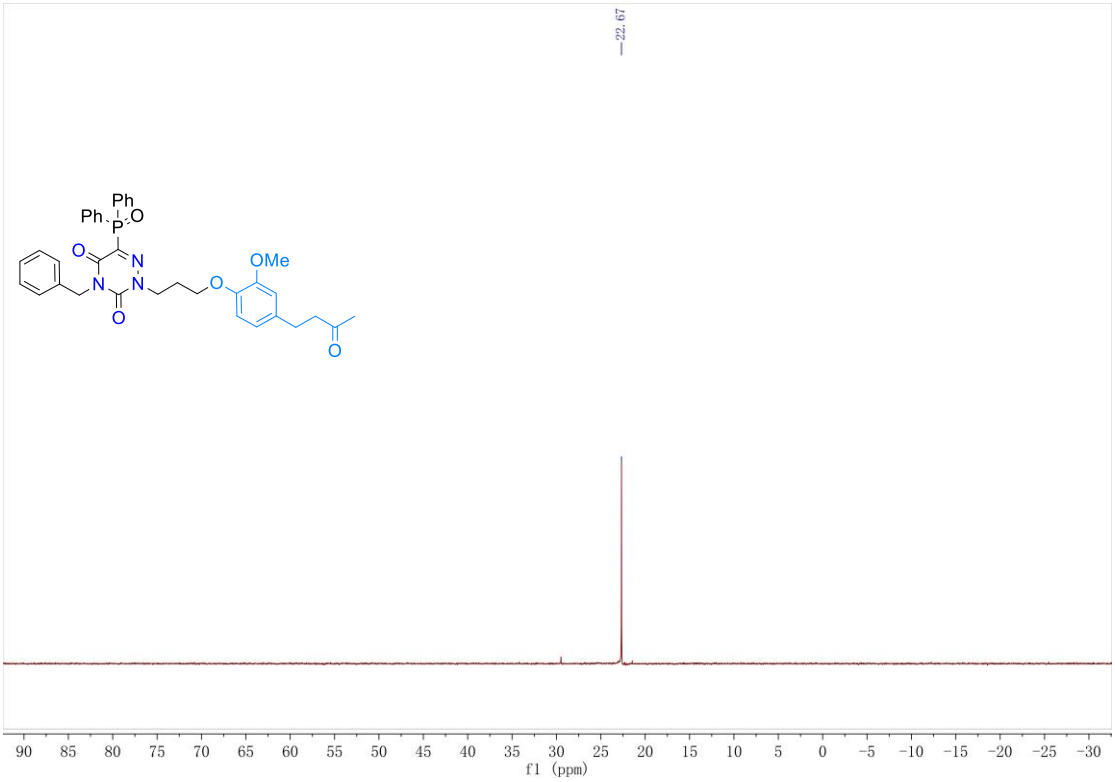
3ai' -^{1}H


9.60
7.98
7.96
7.95
7.93
7.71
7.69
7.68
7.56
7.54
7.52
7.49
7.48
7.47
7.46
7.45
7.45
7.45
2.81
O
P
Ph
Ph
N
N
1.00
5.07
2.09
2.05
4.08
3.09
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
f1 (ppm)

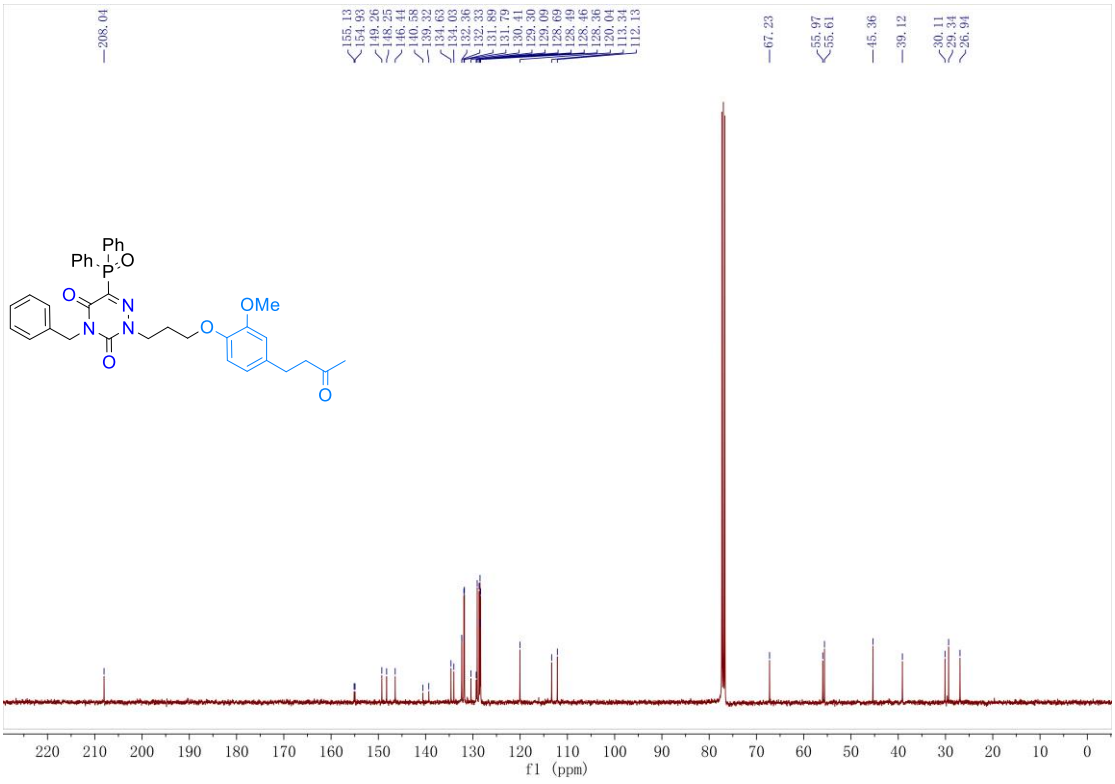
3ak-^{1}H



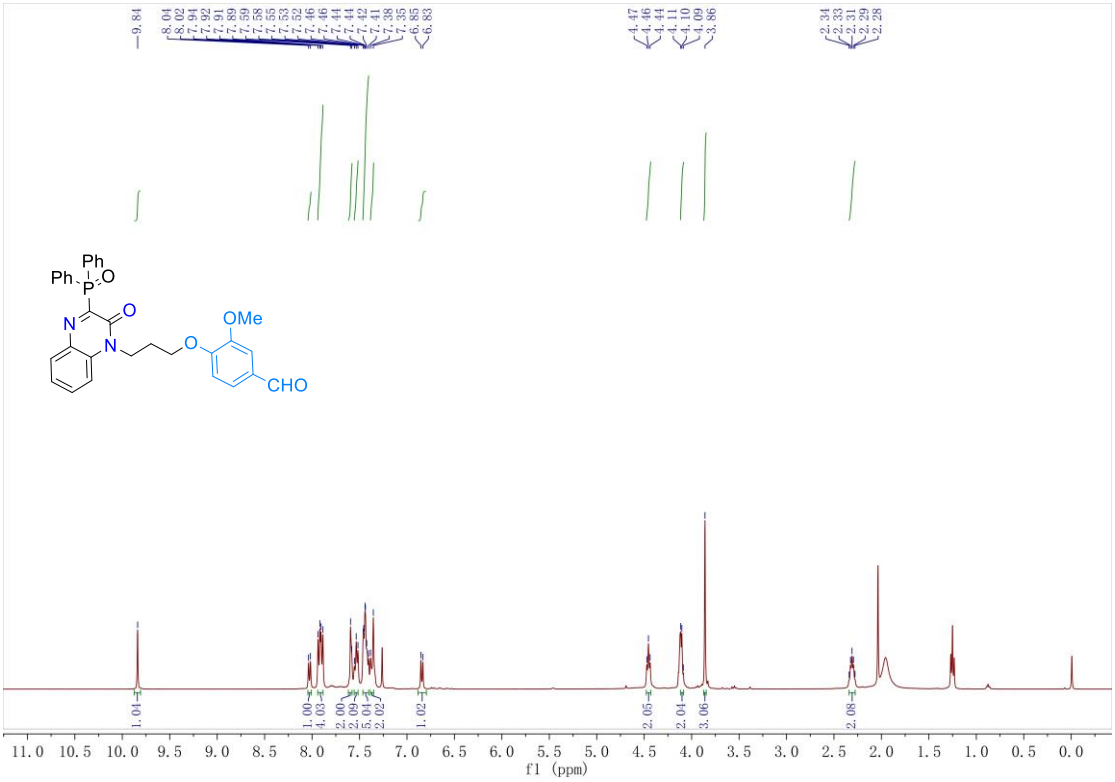
3ak-^{31}P



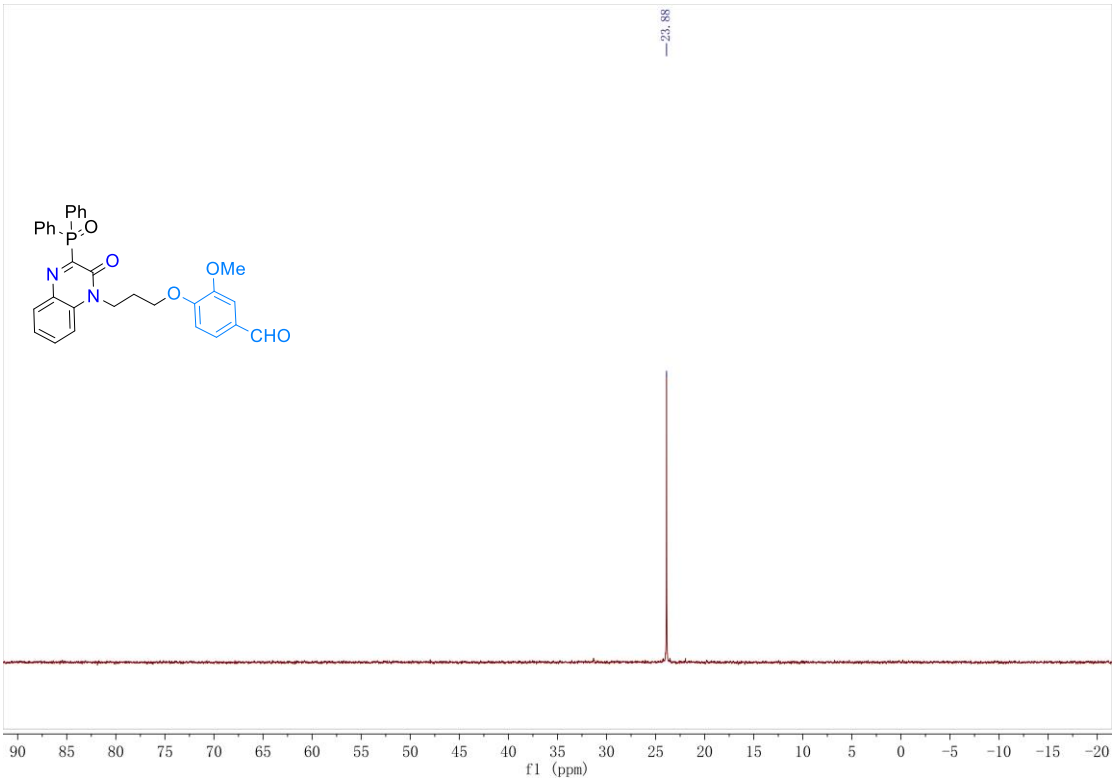
3ak-^{13}C



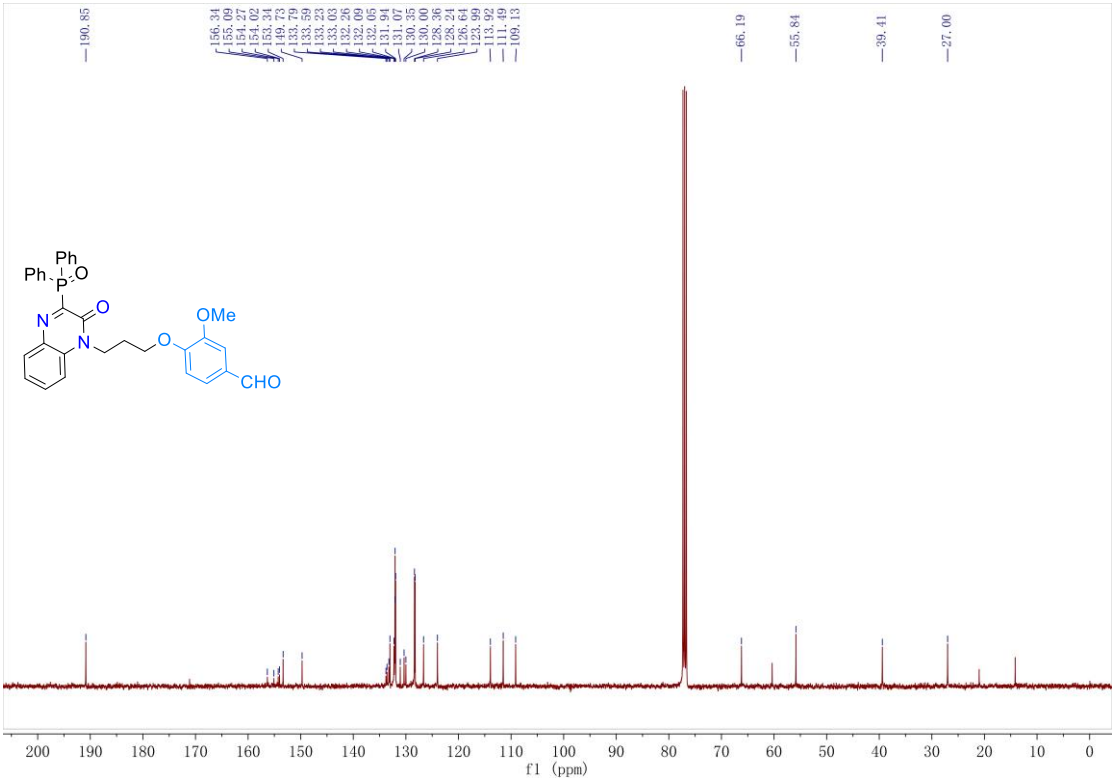
3al-^{1}H



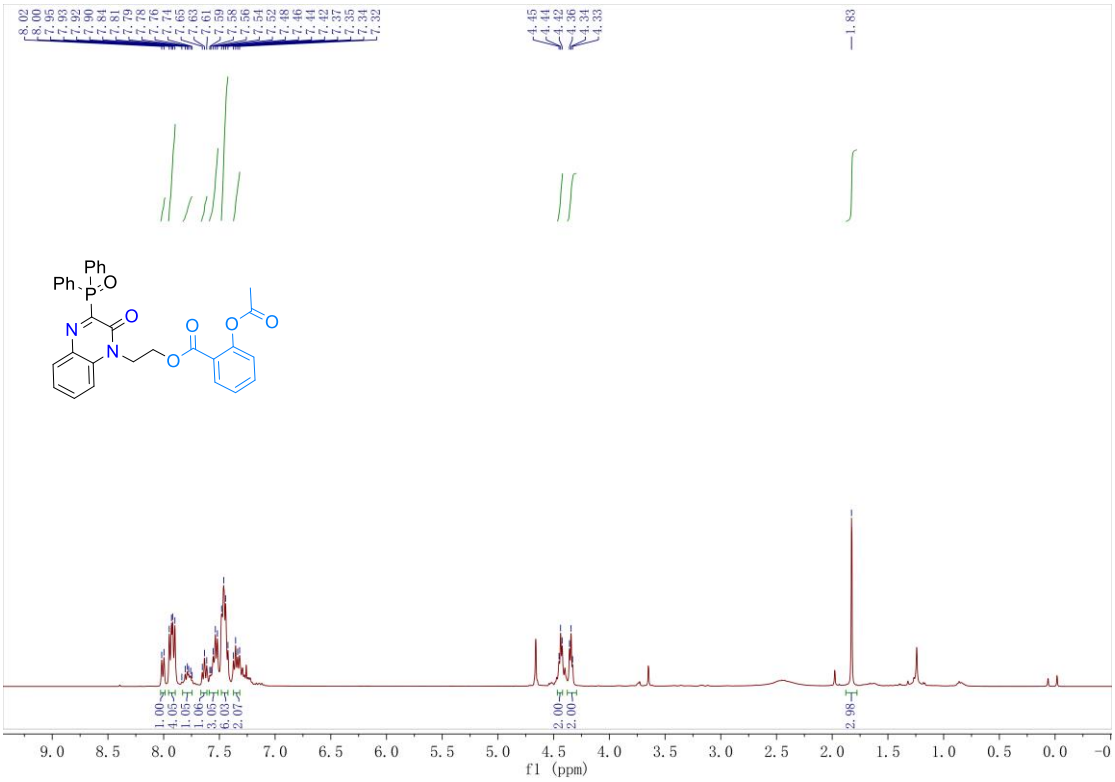
3al-^{31}P



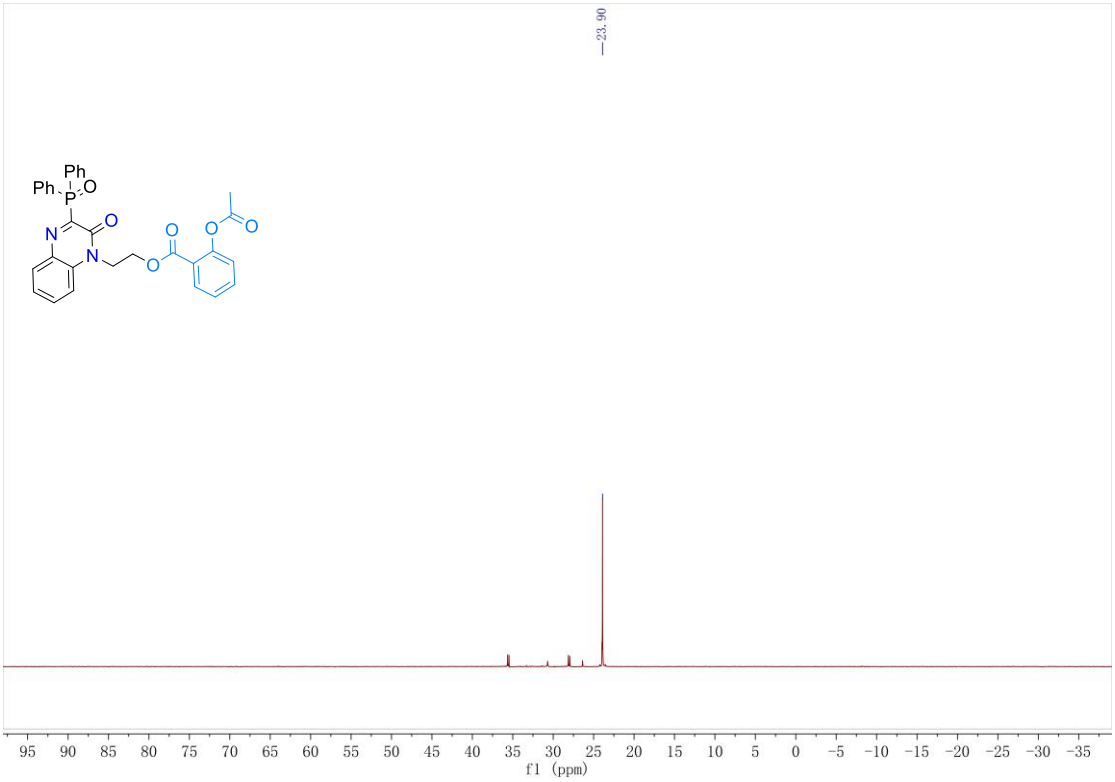
3al-^{13}C



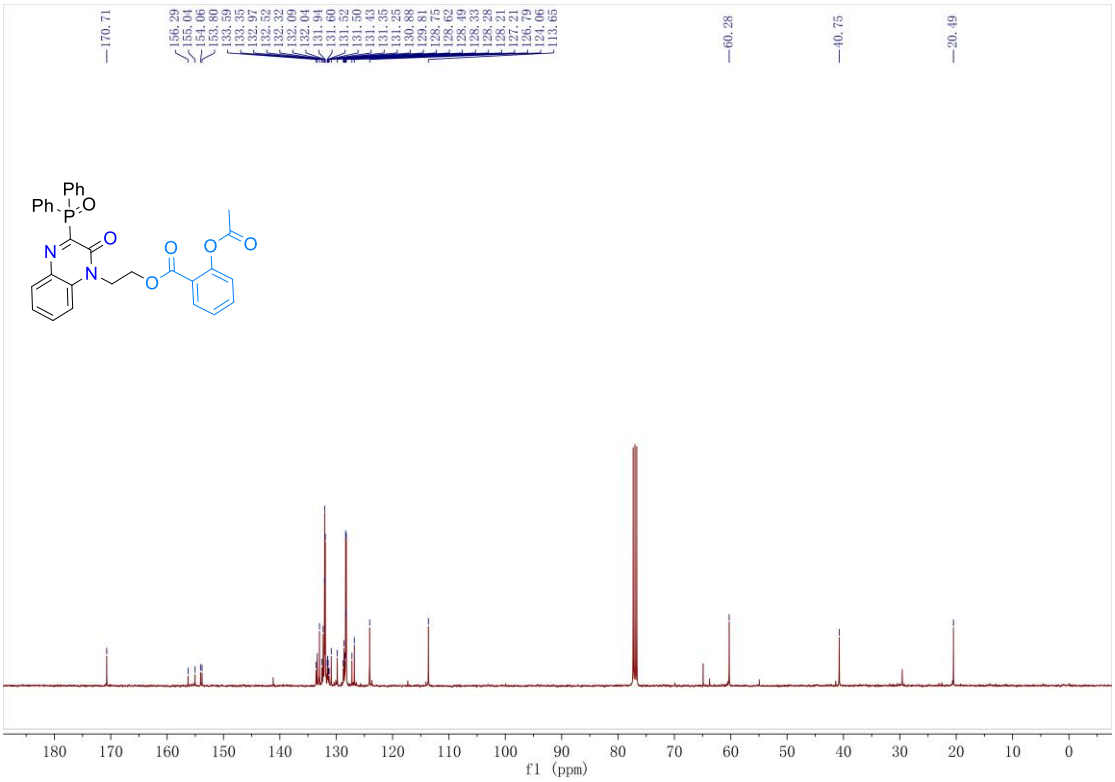
3am-^{1}H



3am-^{31}P



3am-^{13}C



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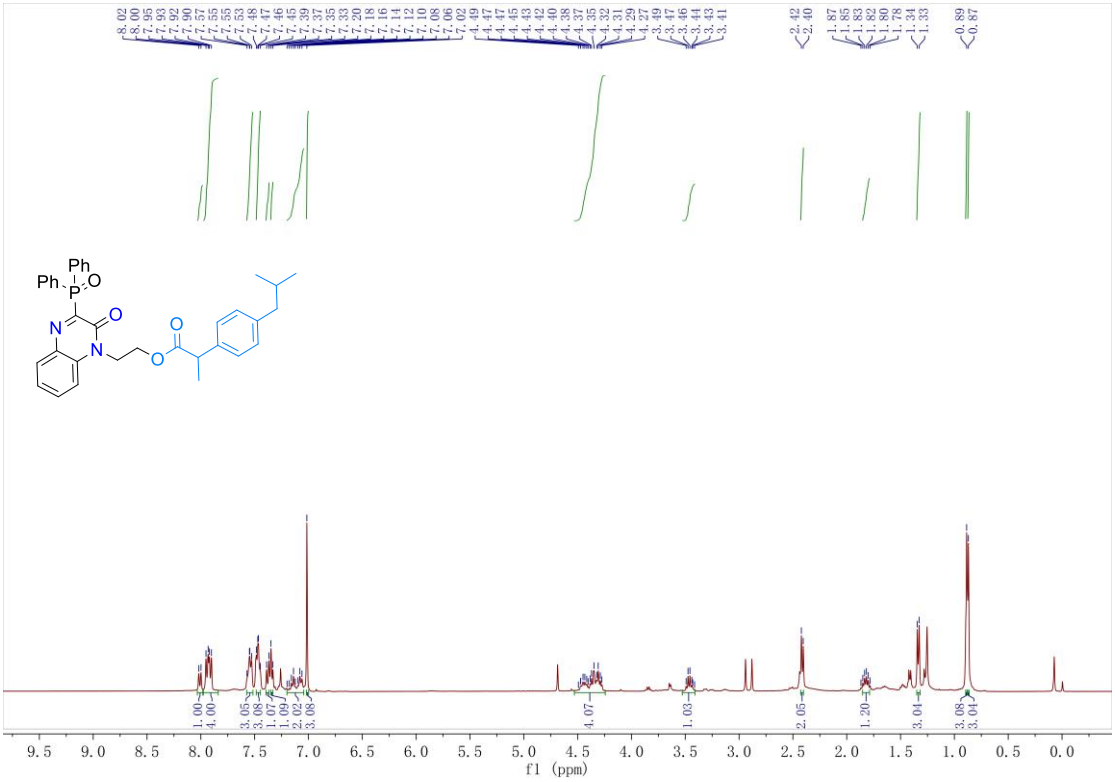
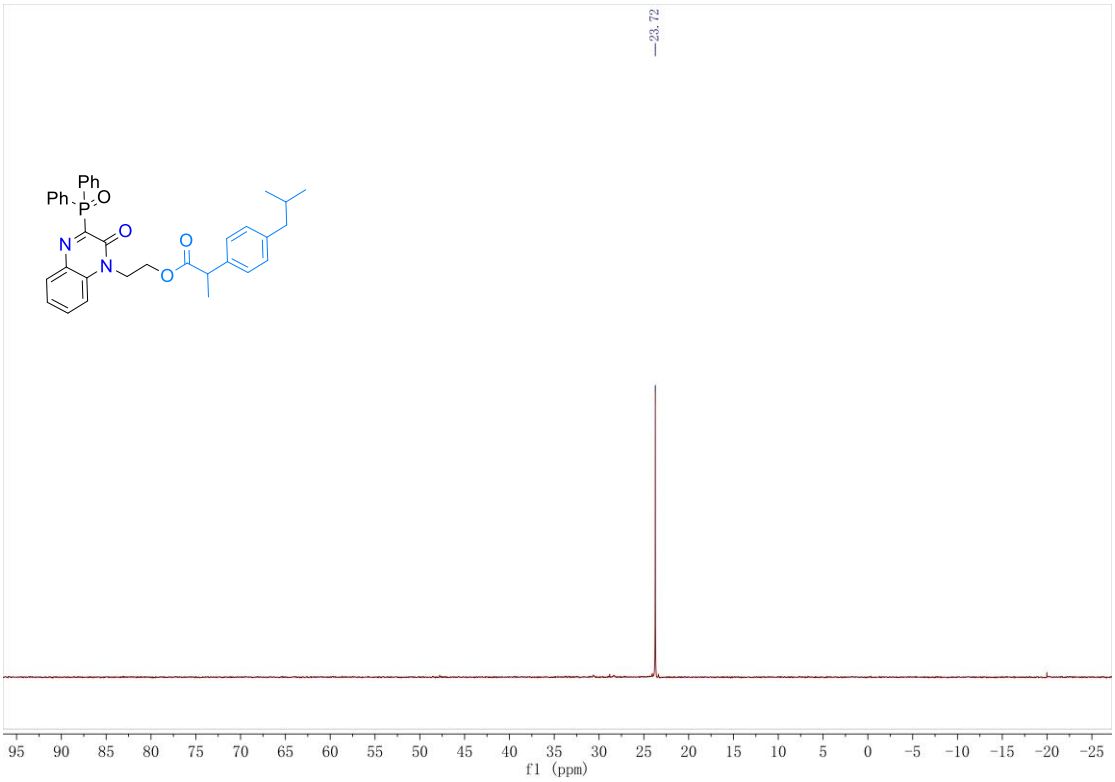
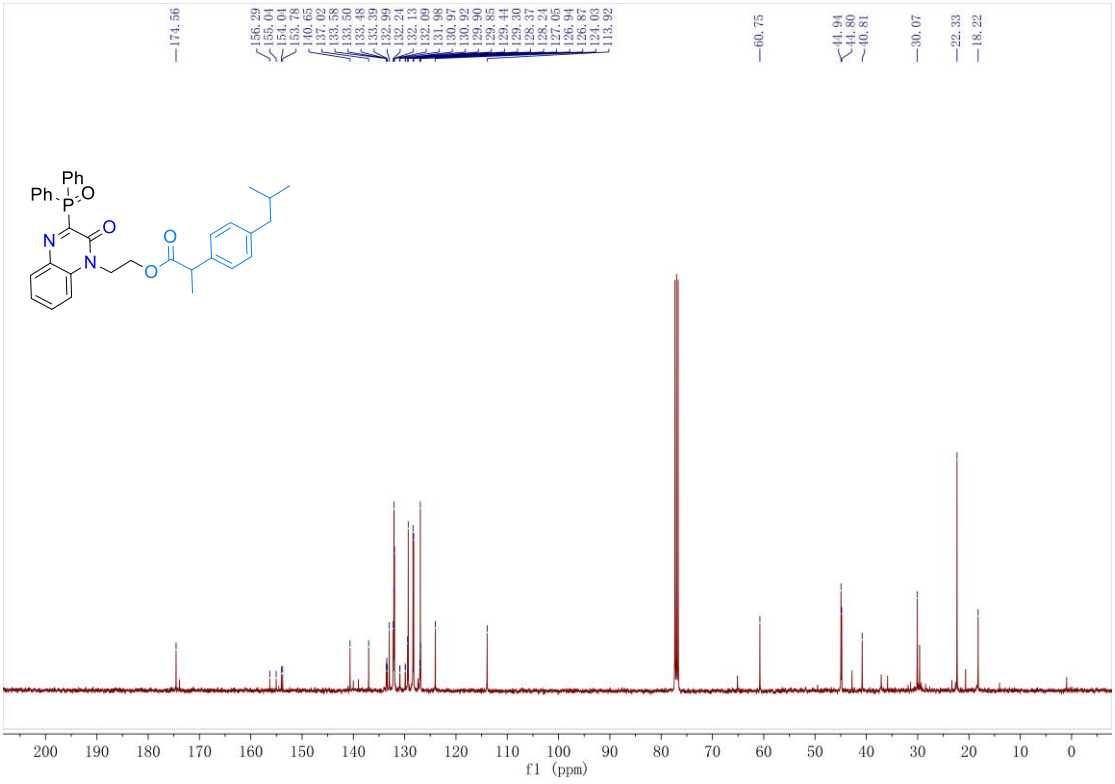


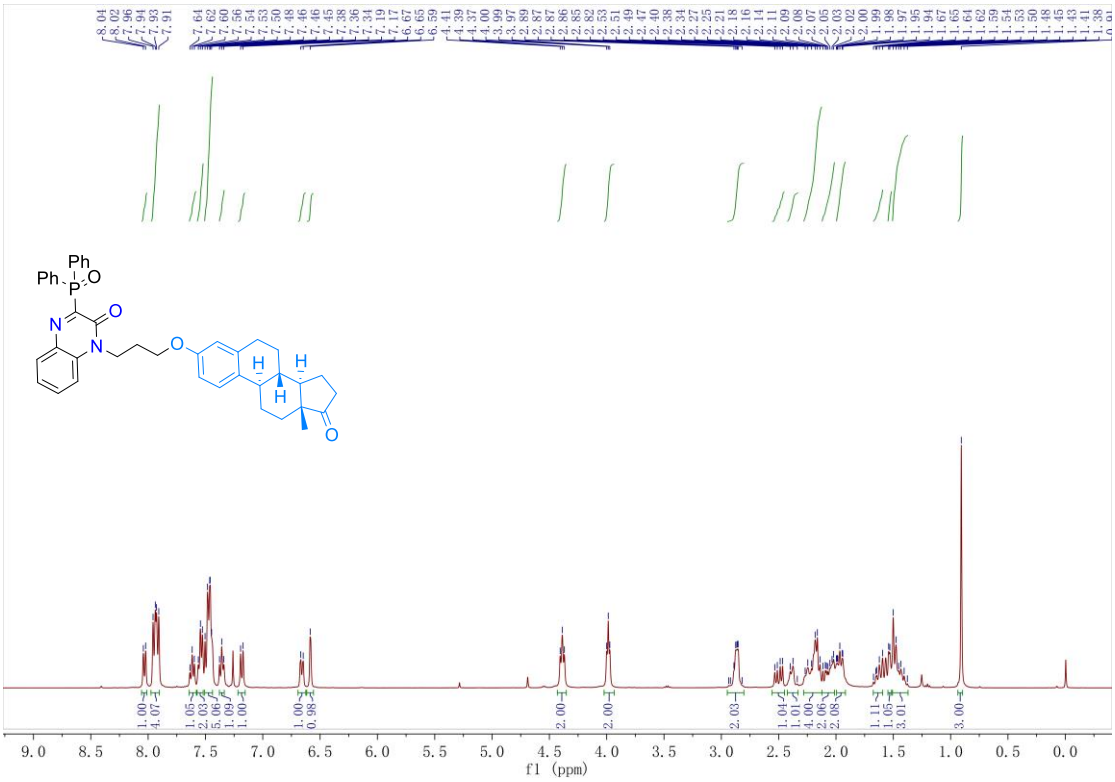
3an-^{31}P



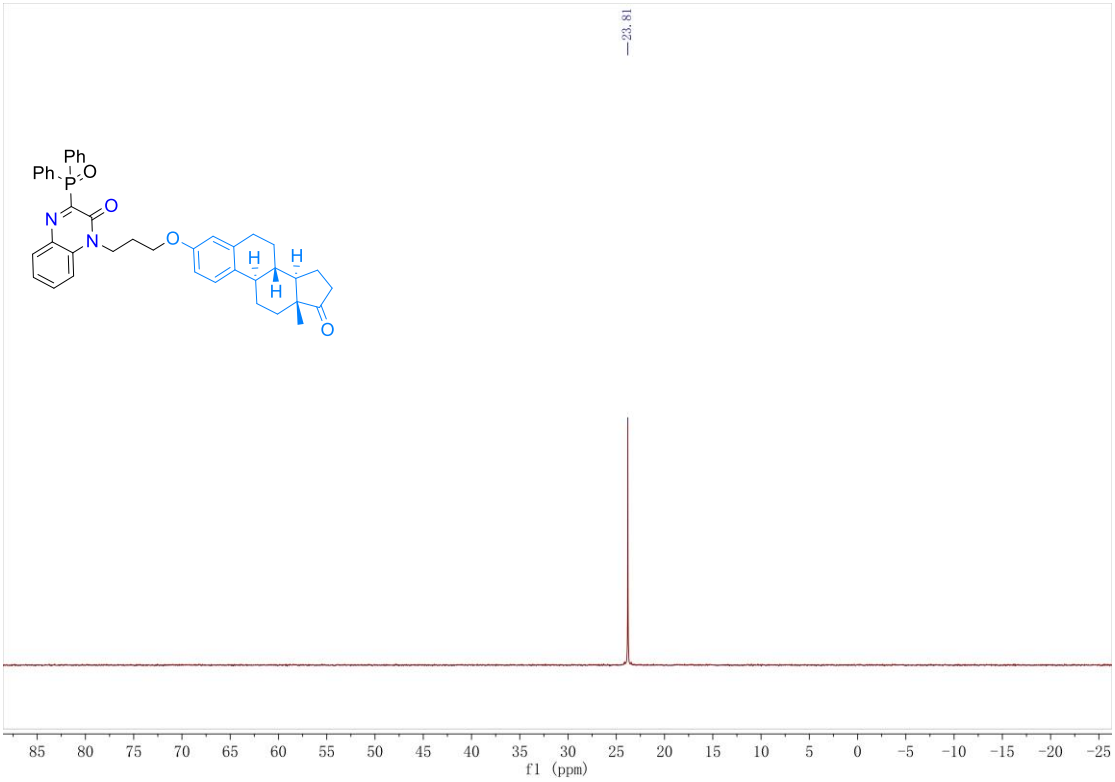
3an-^{13}C



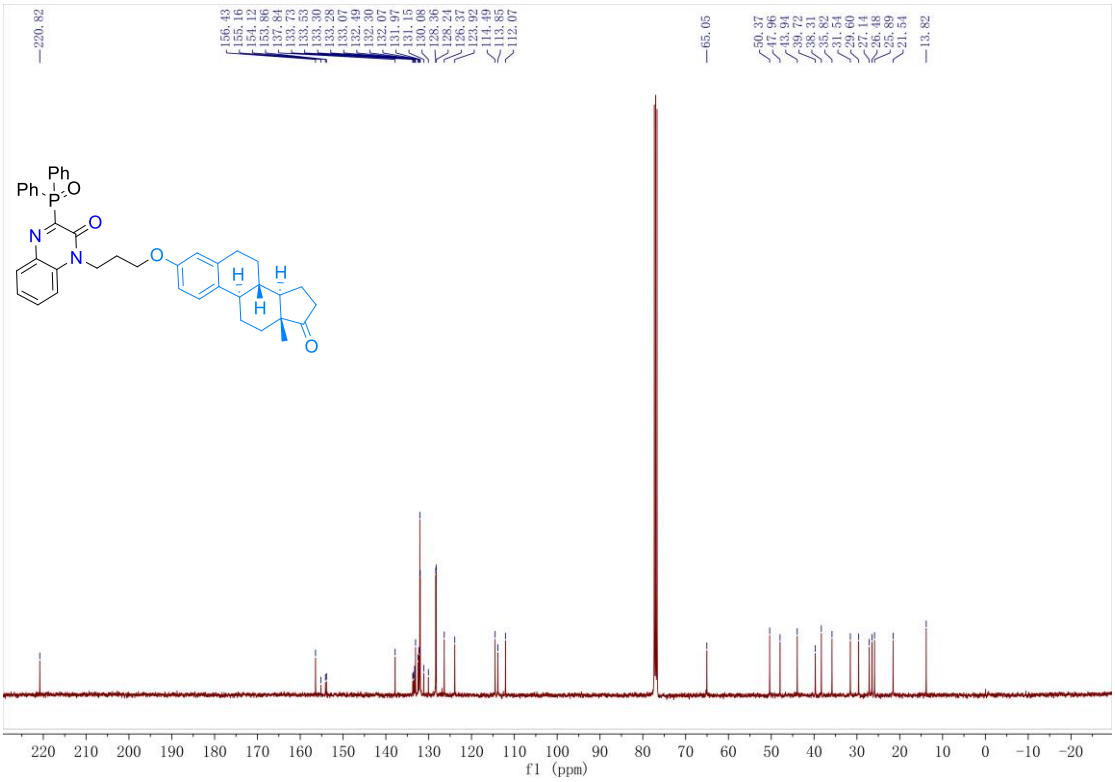
3ao-^{1}H



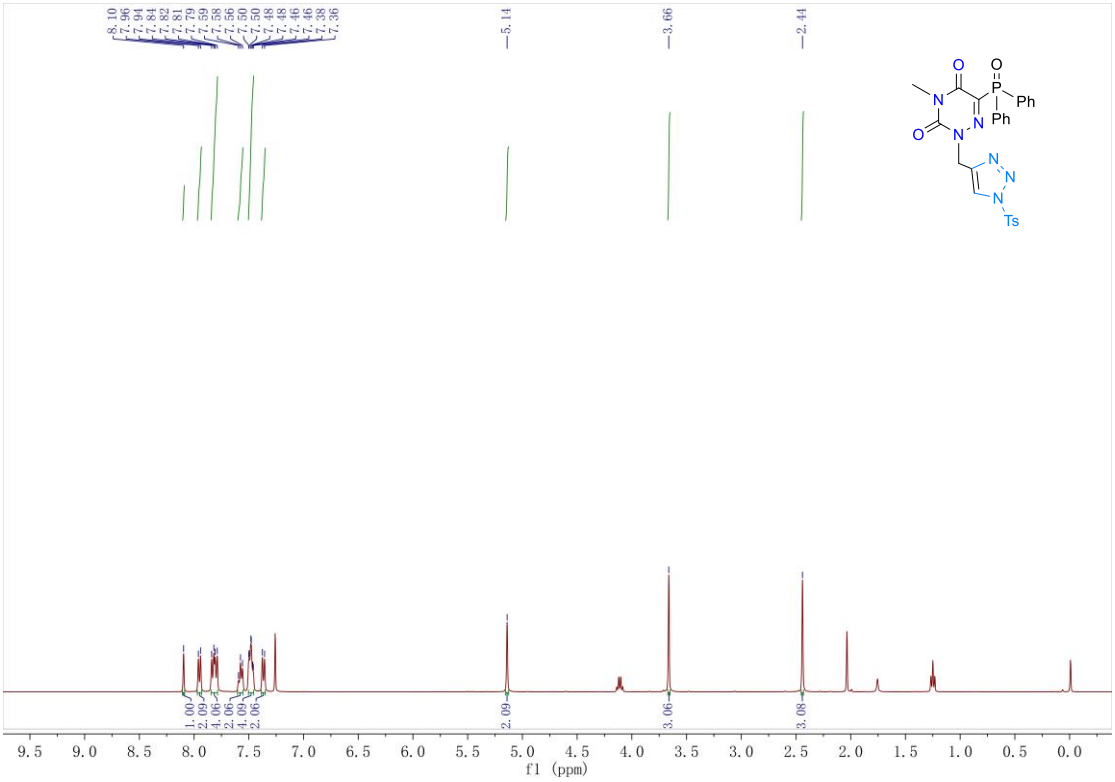
3ao-^{31}P



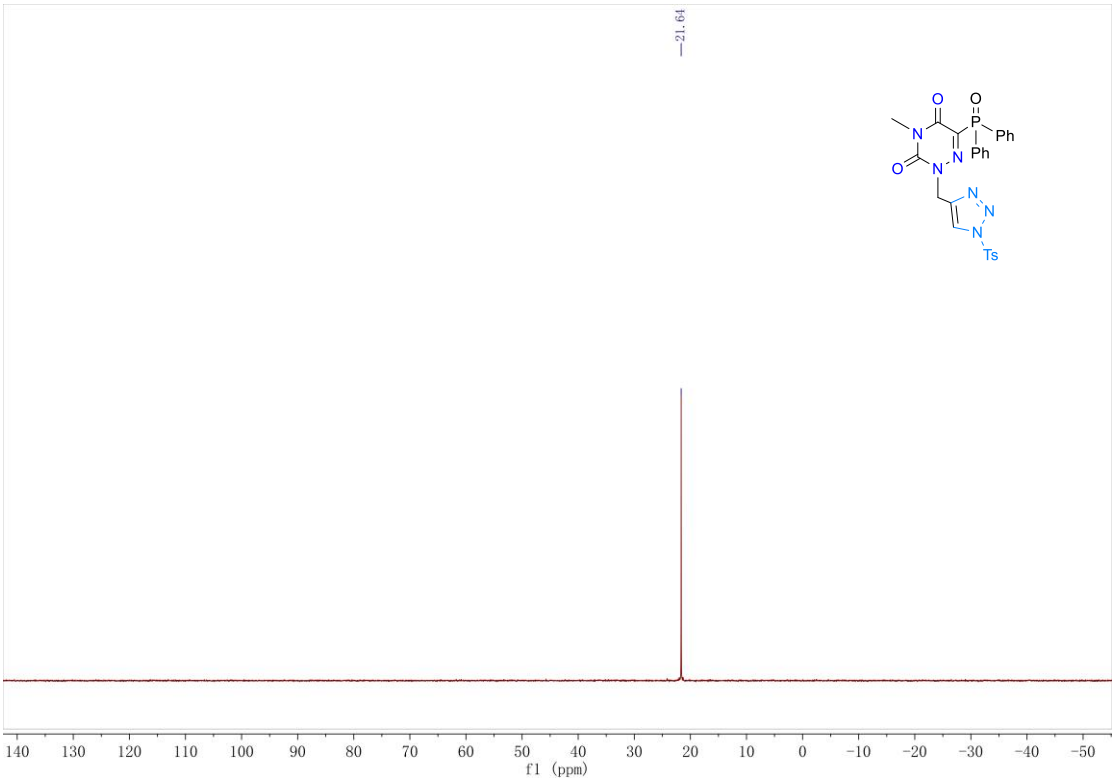
3ao-^{13}C


220.82
156.43
155.16
154.12
153.86
137.84
133.73
133.53
133.30
133.28
133.07
132.49
132.30
132.07
131.97
131.15
130.08
128.36
128.24
126.37
123.92
114.49
113.85
112.07
65.05
50.37
47.96
47.94
39.72
38.31
35.82
31.54
29.60
27.14
26.48
25.89
21.54
13.82
Ph
Ph
P
O
N
O
N
O
H
H
H
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20
f1 (ppm)

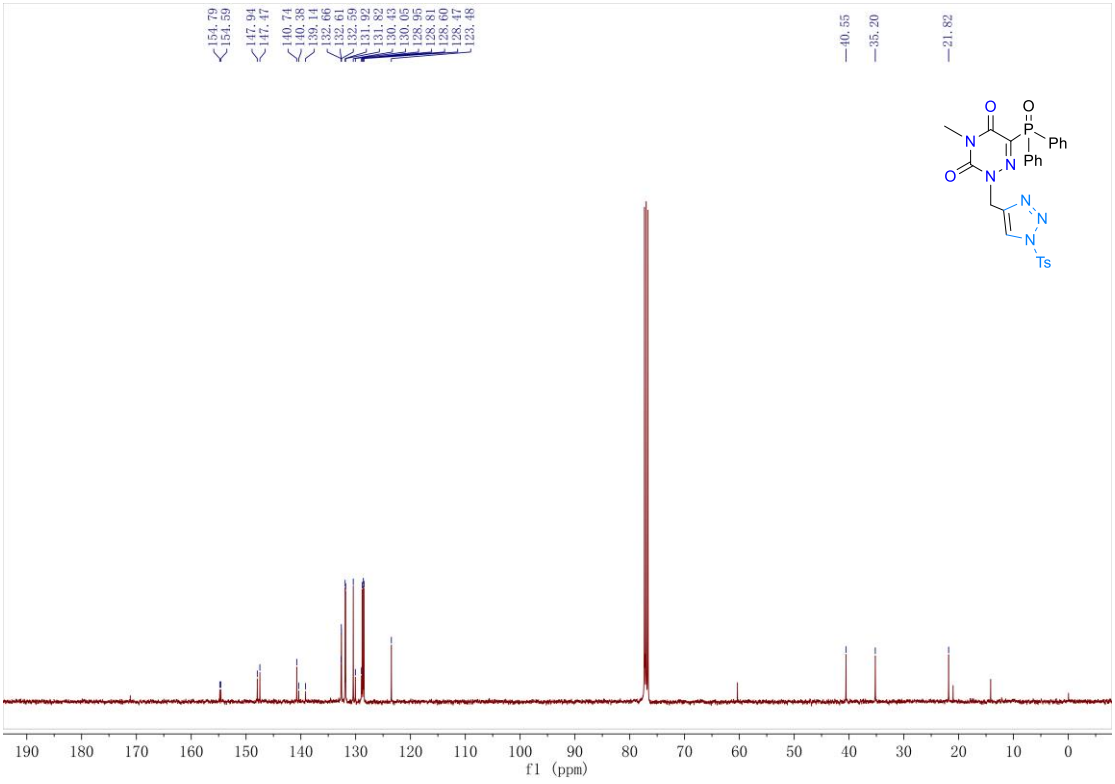
4-^{1}H


8.10
7.96
7.94
7.84
7.82
7.81
7.79
7.59
7.58
7.56
7.50
7.50
7.48
7.48
7.46
7.46
7.38
7.36
5.14
3.66
2.44
O
O
P
Ph
Ph
N
N
O
N
N
N
Ts
1.00
2.09
4.06
2.06
4.09
2.06
2.09
3.06
3.08
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)

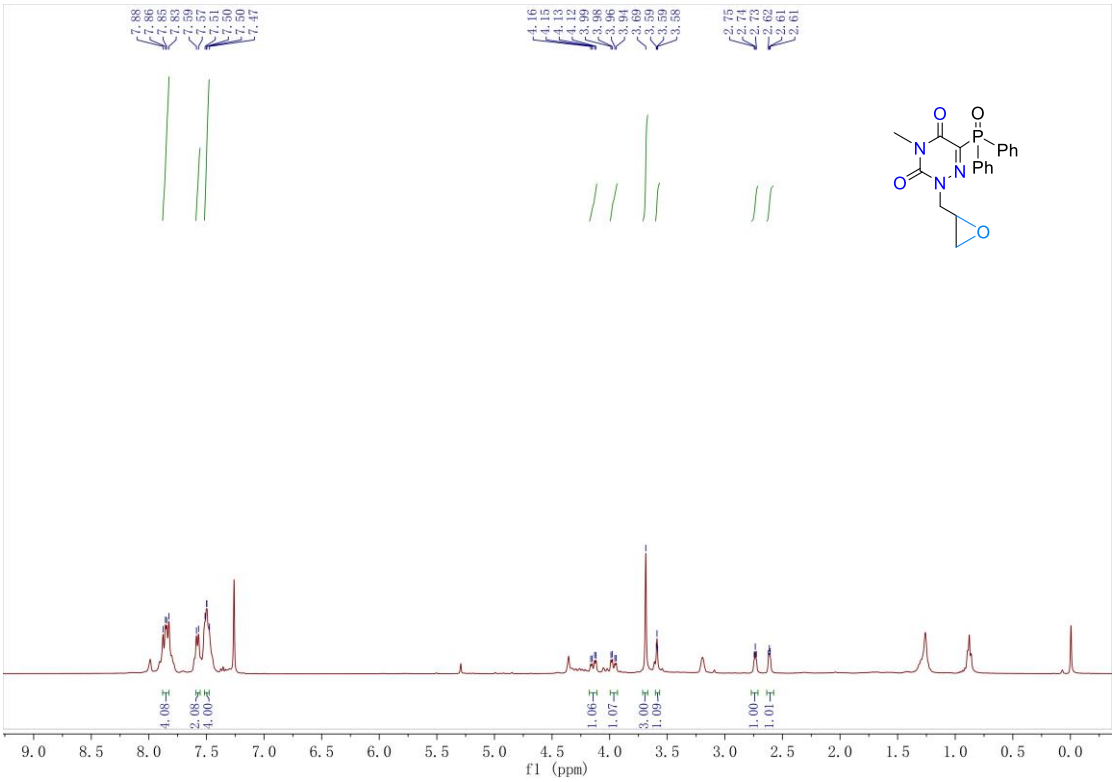
4-^{31}P


21.64
f1 (ppm)

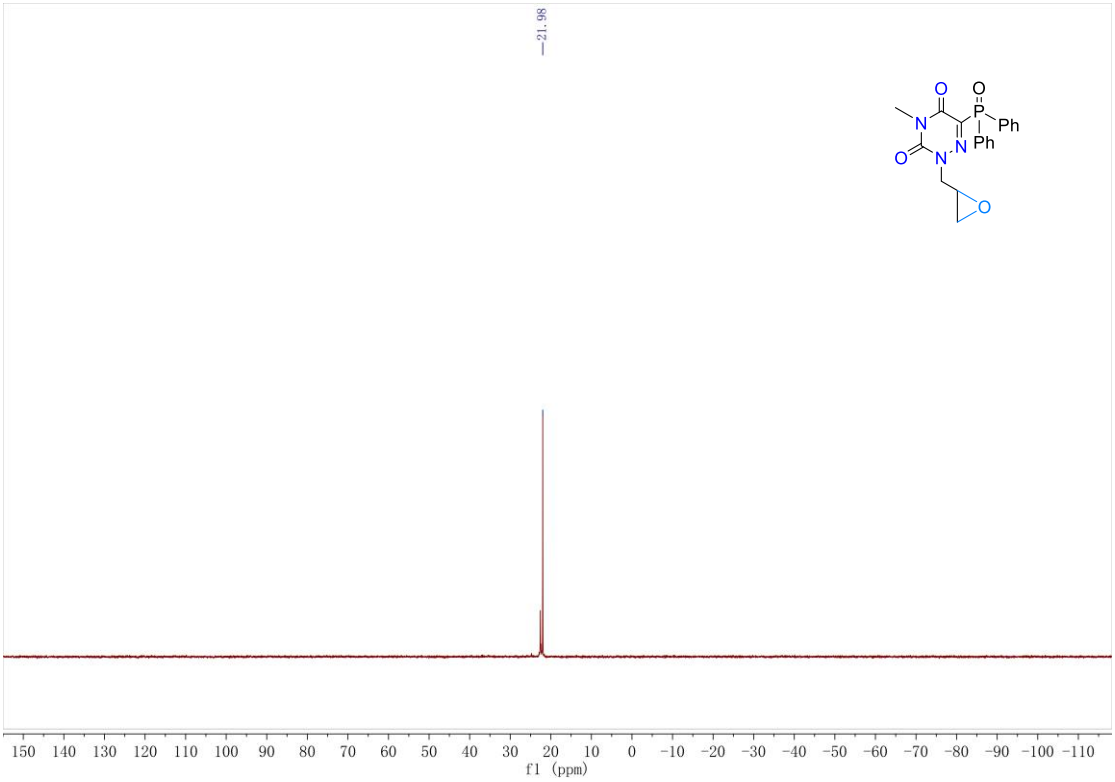
4-^{13}C


154.79
154.59
147.94
147.47
140.74
140.38
139.14
132.66
132.61
132.59
131.92
131.82
130.43
130.05
128.95
128.81
128.60
128.47
123.48
40.55
35.20
21.82
f1 (ppm)

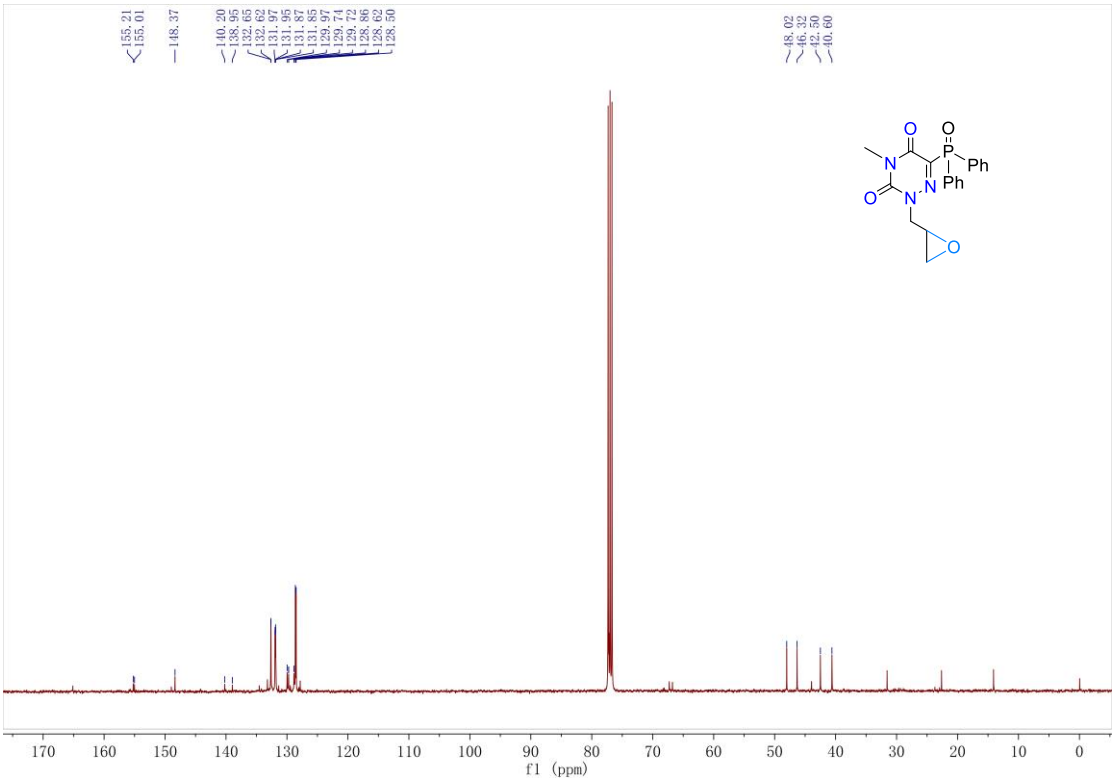
5-^{1}H


7.88
7.86
7.85
7.83
7.59
7.57
7.51
7.50
7.50
7.47
4.16
4.15
4.13
4.12
3.99
3.98
3.96
3.94
3.69
3.59
3.59
3.58
2.75
2.74
2.73
2.62
2.61
2.61
O
O
P
Ph
Ph
N
N
O
N
O
4.08
2.08
4.00
1.06
1.07
3.00
1.09
1.00
1.01
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)

5-^{31}P


21.98
O
O
P
Ph
Ph
N
N
O
N
O
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110
f1 (ppm)

5-^{13}C



VIII. References

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