

**Highly Regio- and Diastereoselective Construction of Spirocyclopenteneoxindole
Phosphonates Through Phosphine-catalyzed [3+2] Annulation Reaction**

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Supporting Information

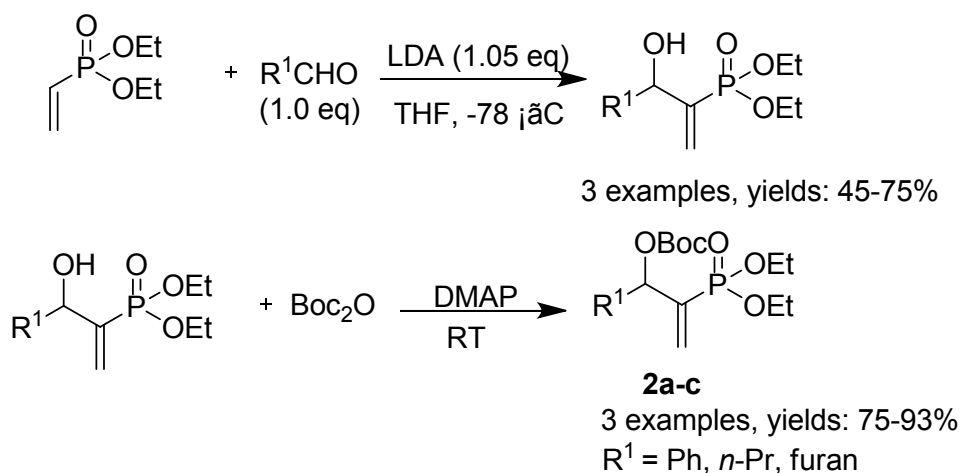
List of contents (pages)

1. General comments.....	S1
2. The synthesis pathway of MBH phosphonates 2	S1
3. General procedure for the preparation of 3	S8
4. X-ray crystal structure of 2a	S53

General Comments.

Solvents were dried and distilled prior to use according to the standard methods. Unless otherwise indicated, all materials were obtained from commercial sources, and used as purchased without dehydration. Flash column chromatography was performed on silica gel (particle size 10-40 μm , Ocean Chemical Factory of Qingdao, China). Nitrogen gas (99.999%) was purchased from Boc Gas Inc. ^1H NMR, ^{13}C NMR and ^{31}P NMR spectra were recorded in CDCl_3 at Bruker 400 MHz spectrometers, TMS served as internal standard ($\delta = 0$ ppm) for ^1H NMR and ^{13}C NMR, H_3PO_4 served as internal standard ($\delta = 0$ ppm) for ^{31}P NMR. The crystal structure was determined on a Bruker SMART 1000 CCD diffractometer. Mass spectra were recorded on a LCQ advantage spectrometer with ESI resource. HR-MS were recorded on APEXII and ZAB-HS spectrometer. Melting points were determined on a T-4 melting point apparatus (uncorrected). Optical rotations were recorded on a Perkin Elemer 241 Polarimeter.

The synthesis pathway of MBH phosphonates **2**:

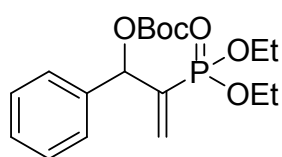


General procedure for the preparation of 2:

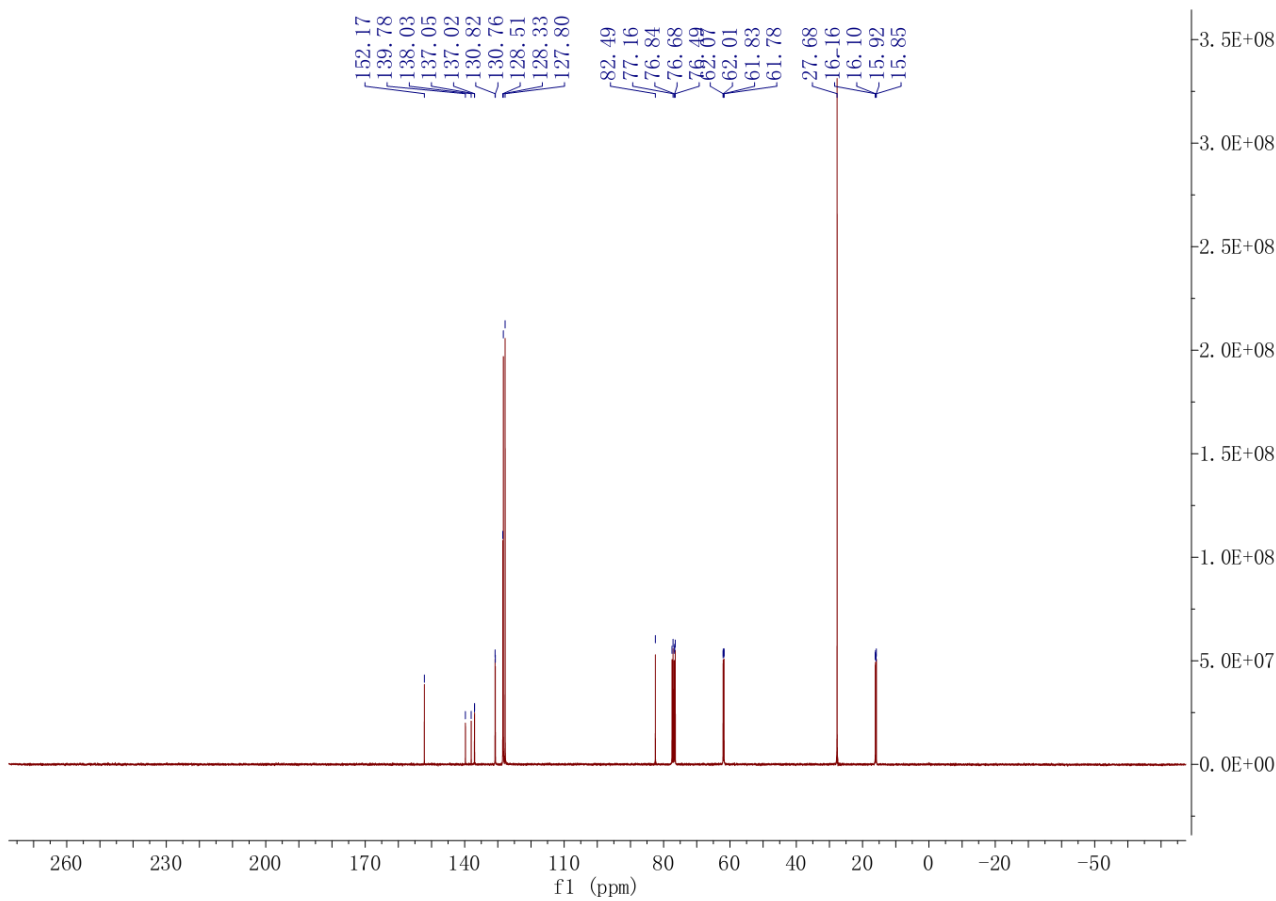
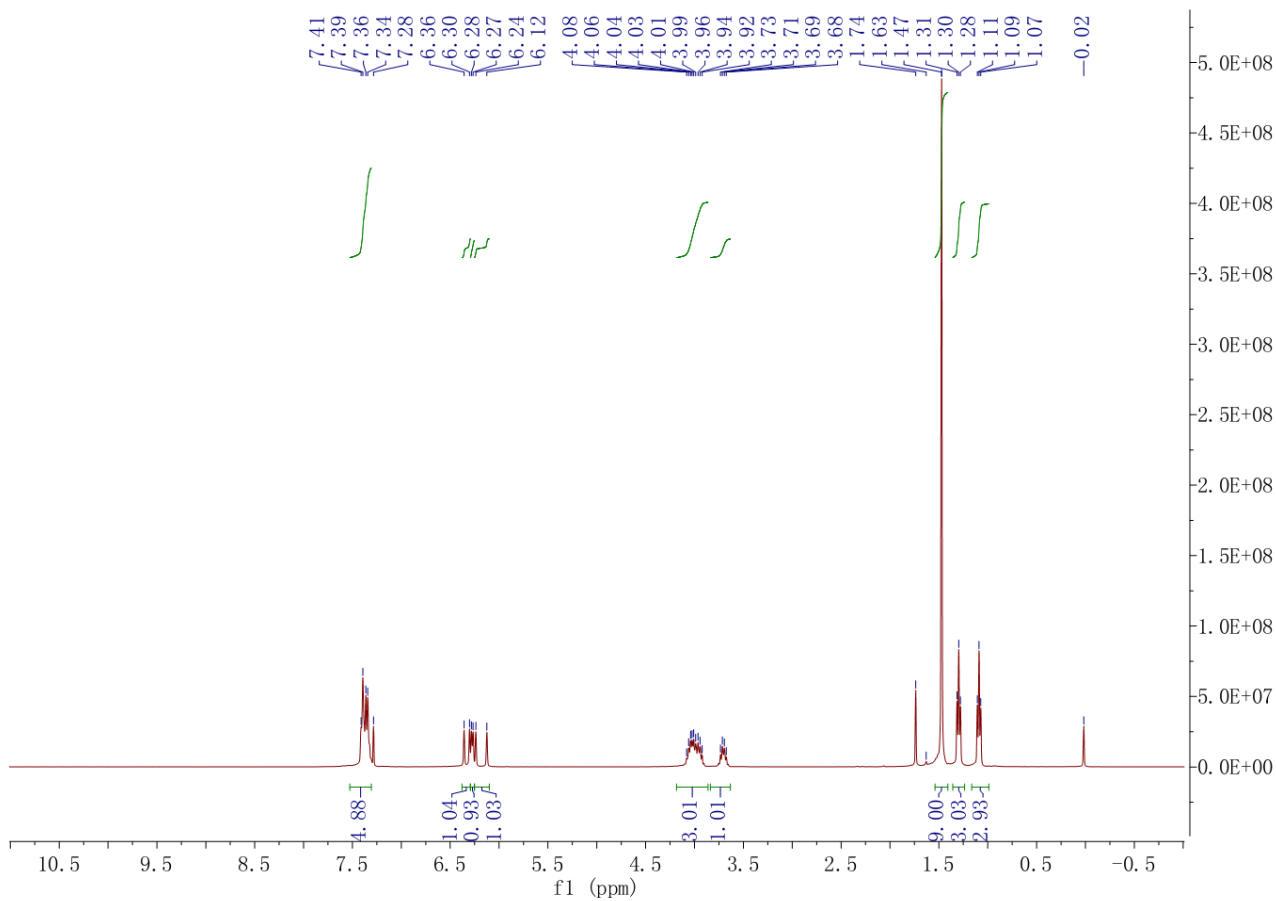
A solution of LDA (22.0 mmol) in THF (50 mL) was added to a mixture of diethyl vinylphosphonate (20.0 mmol) and aldehyde (20.0 mmol) in THF (50 mL) at -78 °C. After being stirred for 60 min at this temperature, the reaction mixture was quenched with saturated NH₄Cl (50 mL) and extracted with EtOAc (150 mL×3). Concentration and subsequent purification by silica gel column chromatography (EtOAc: hexane = 3: 2) afforded (1-hydroxy-vinyl) phosphonates in 45-75% yield.

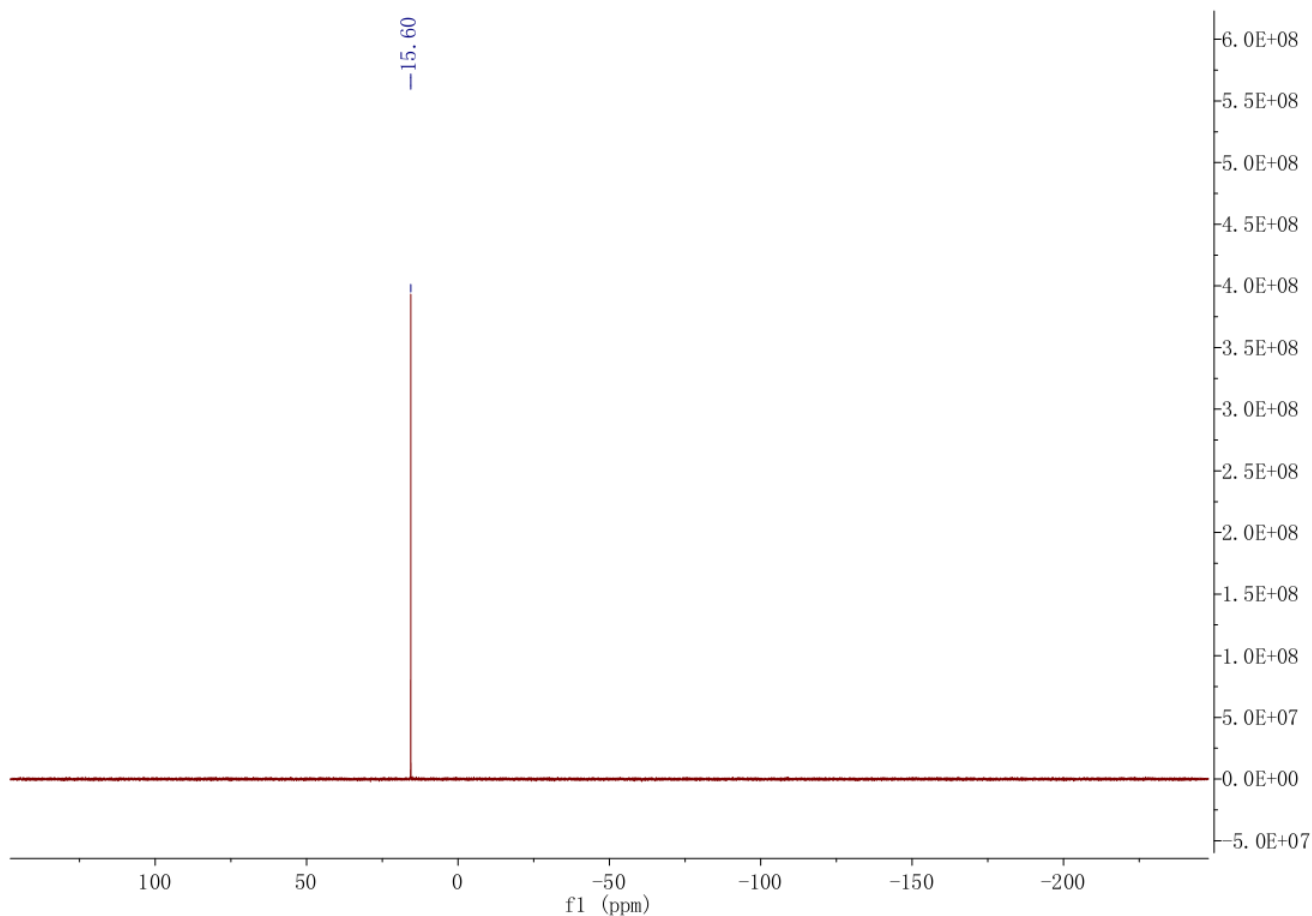
To an ice-water cooled solution of (1-hydroxy-vinyl) phosphonates (10 mmol) in dry CH₂Cl₂ (10 mL) were added Boc₂O (11 mmol) and DMAP (0.5 mmol) in dry CH₂Cl₂ (10 mL) over half an hour. The reaction mixture was stirred at room temperature overnight. To the solution was added 50 mL CH₂Cl₂ and washed with aqueous hydrochloric acid (15%, 20 mL), saturated sodium bicarbonate (20 mL), and brine (20 mL) sequentially, dried over anhydrous sodium sulfate, concentrated, and purified by column chromatography to get the product **2a-c** in 75-93% yield.

tert-Butyl (2-(diethoxyphosphoryl)-1-phenylallyl) carbonate (**2a**):

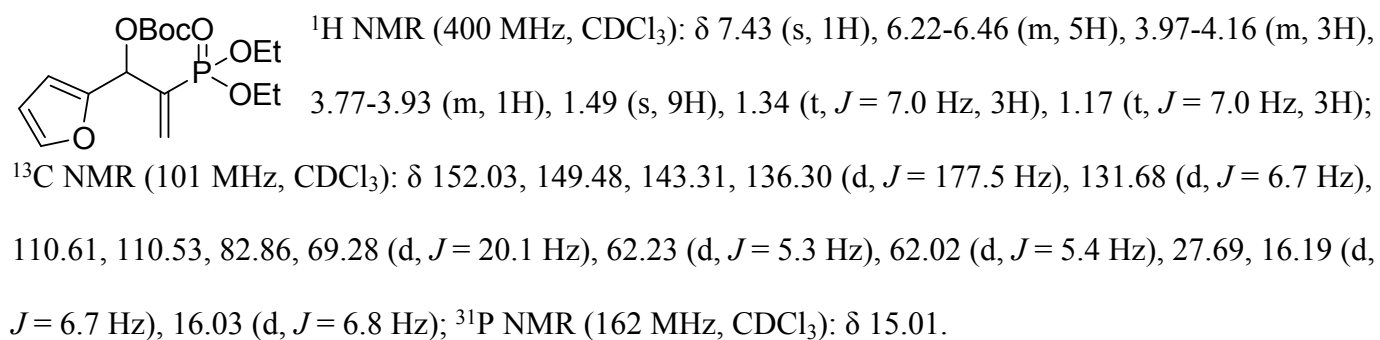


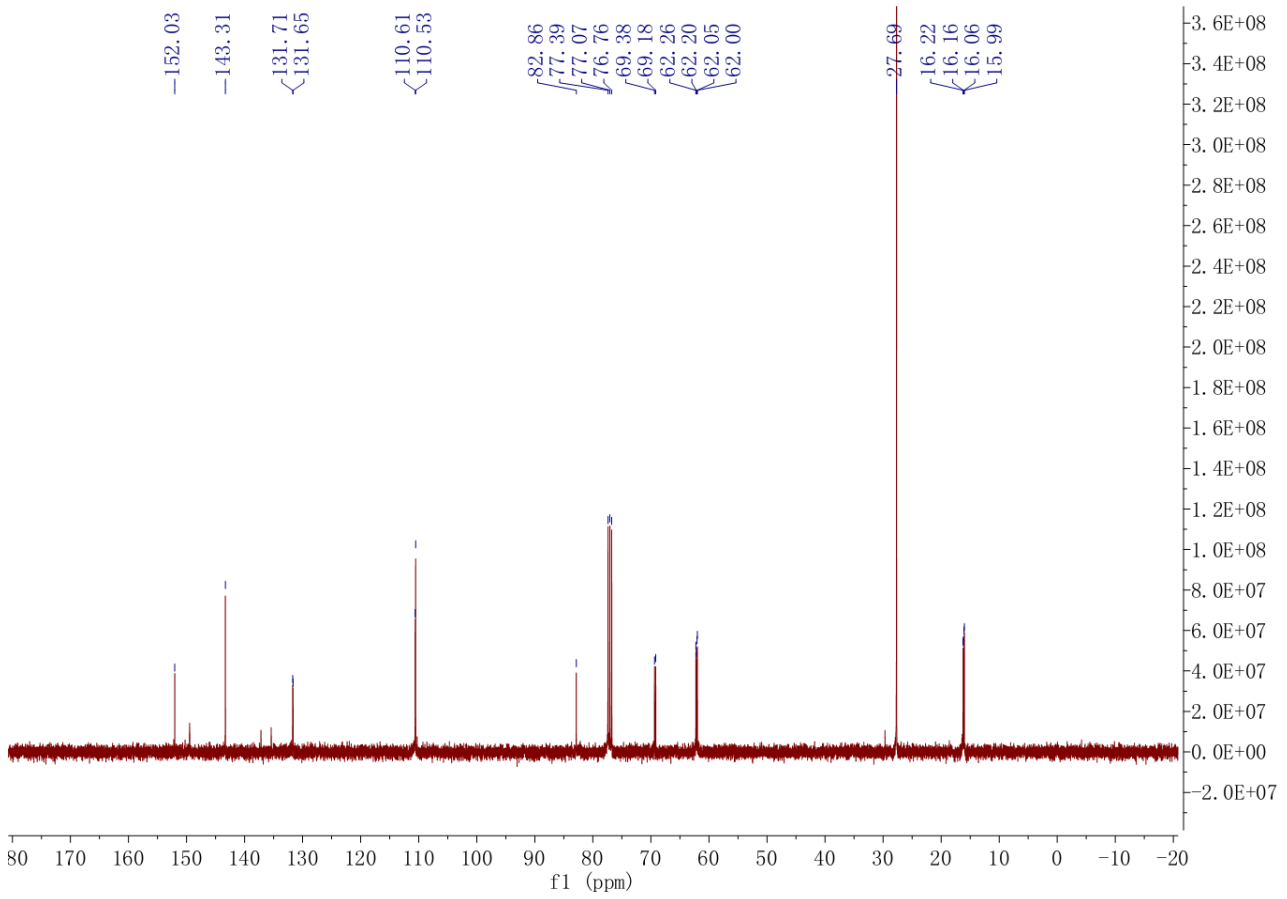
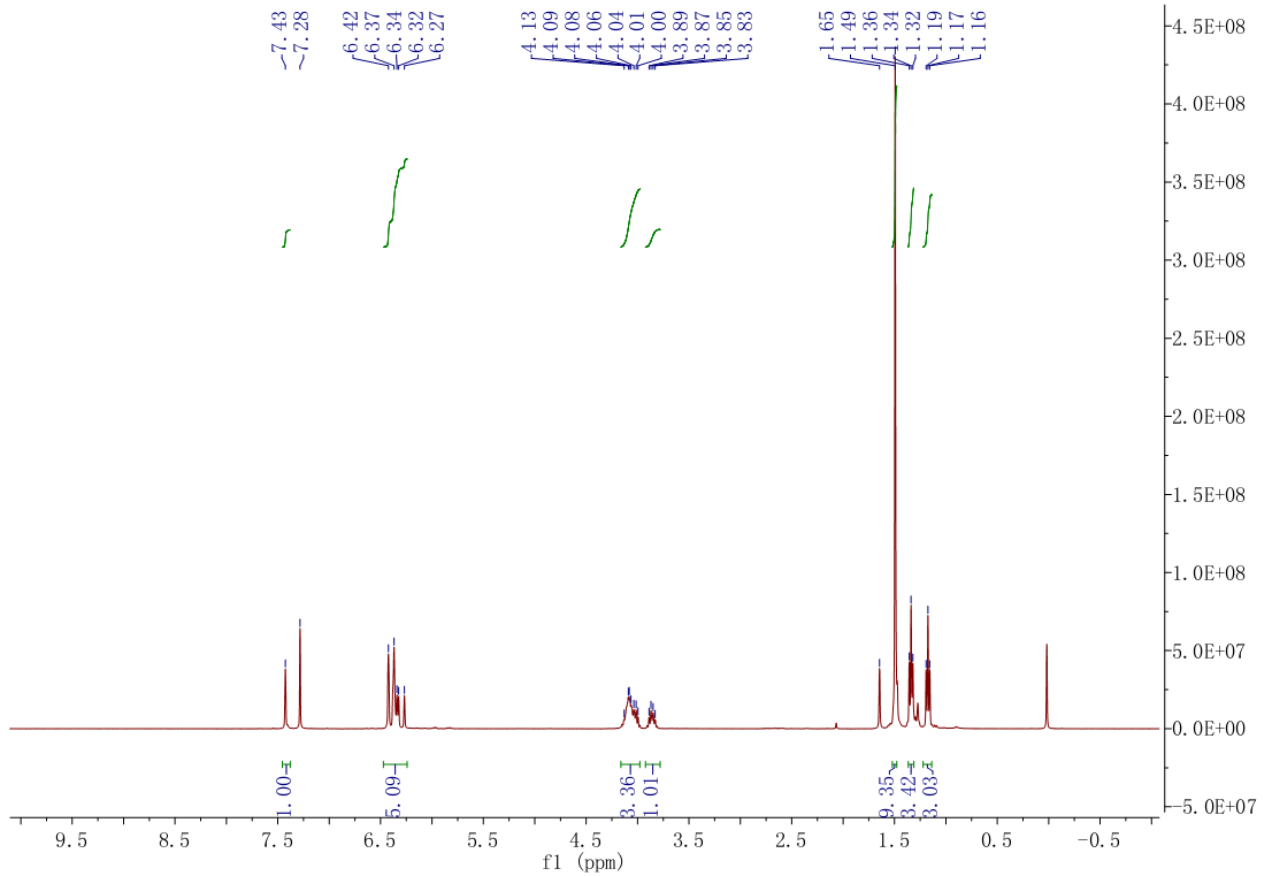
¹H NMR (400 MHz, CDCl₃): δ 7.29-7.45 (m, 5H), 6.33 (d, *J* = 22.4 Hz, 1H), 6.27 (d, *J* = 6.7 Hz, 1H), 6.18 (d, *J* = 45.2 Hz, 1H), 3.82-4.17 (m, 3H), 3.56-3.82 (m, 1H), 1.47 (s, 9H), 1.30 (t, *J* = 7.0 Hz, 3H), 1.09 (t, *J* = 7.0 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃): δ 152.17, 139.78, 138.03, 137.04 (d, *J* = 3.1 Hz), 130.79 (d, *J* = 6.8 Hz), 128.51, 128.06 (d, *J* = 52.9 Hz), 82.49, 76.76 (d, *J* = 16.5 Hz), 62.04 (d, *J* = 5.3 Hz), 61.80 (d, *J* = 5.4 Hz), 27.68, 16.13 (d, *J* = 6.6 Hz), 15.88 (d, *J* = 6.9 Hz); ³¹P NMR (162 MHz, CDCl₃): δ 15.60.

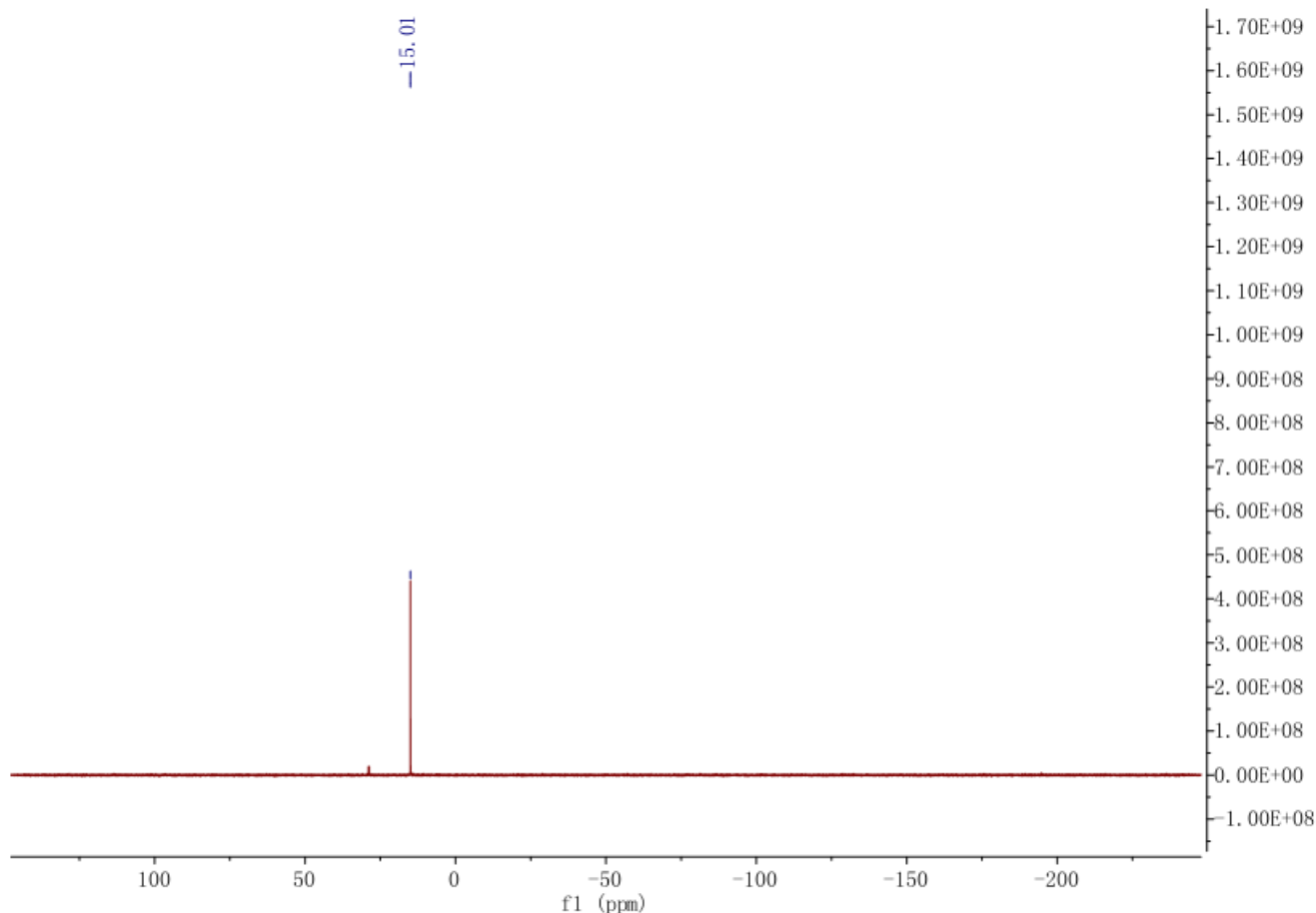




***tert*-Butyl (2-(diethoxyphosphoryl)-1-(furan-2-yl)allyl) carbonate(2b):**

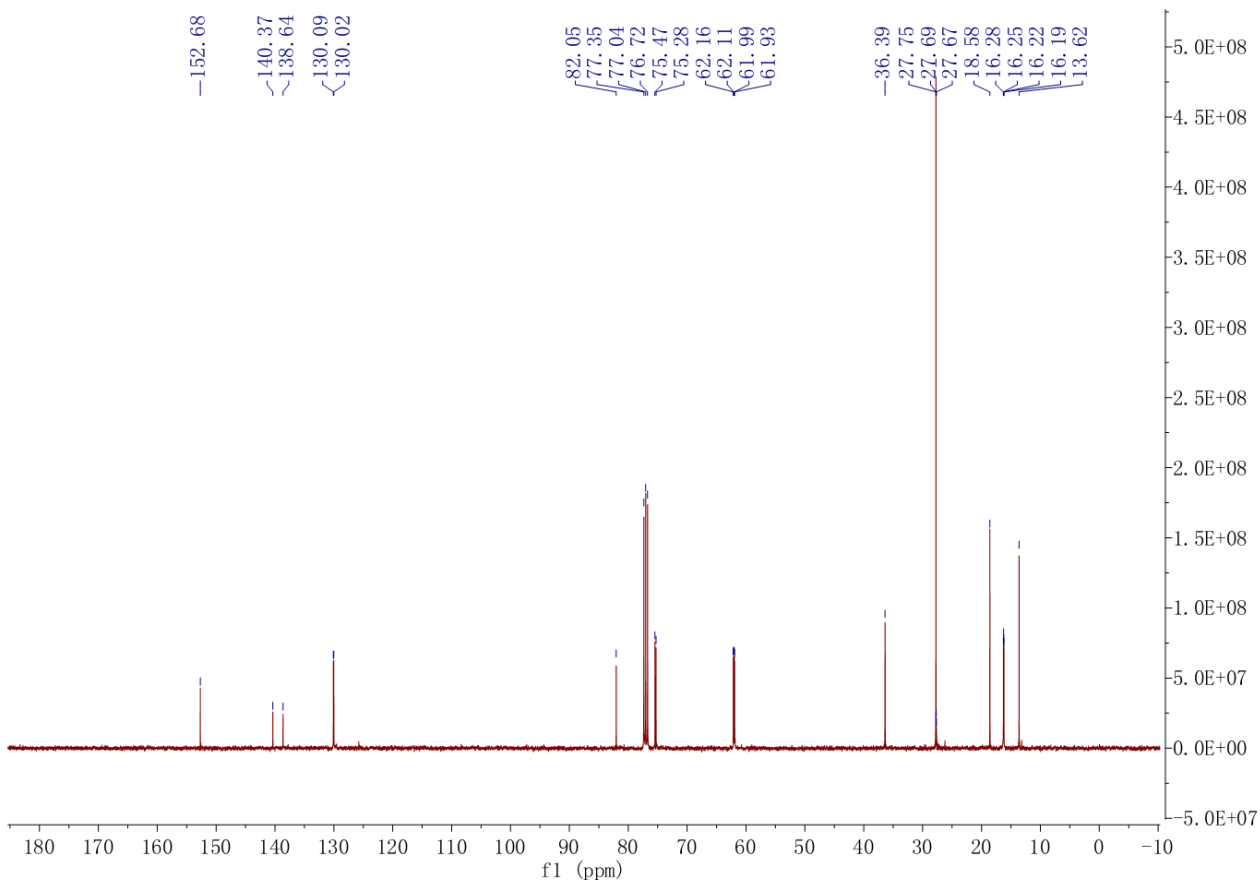
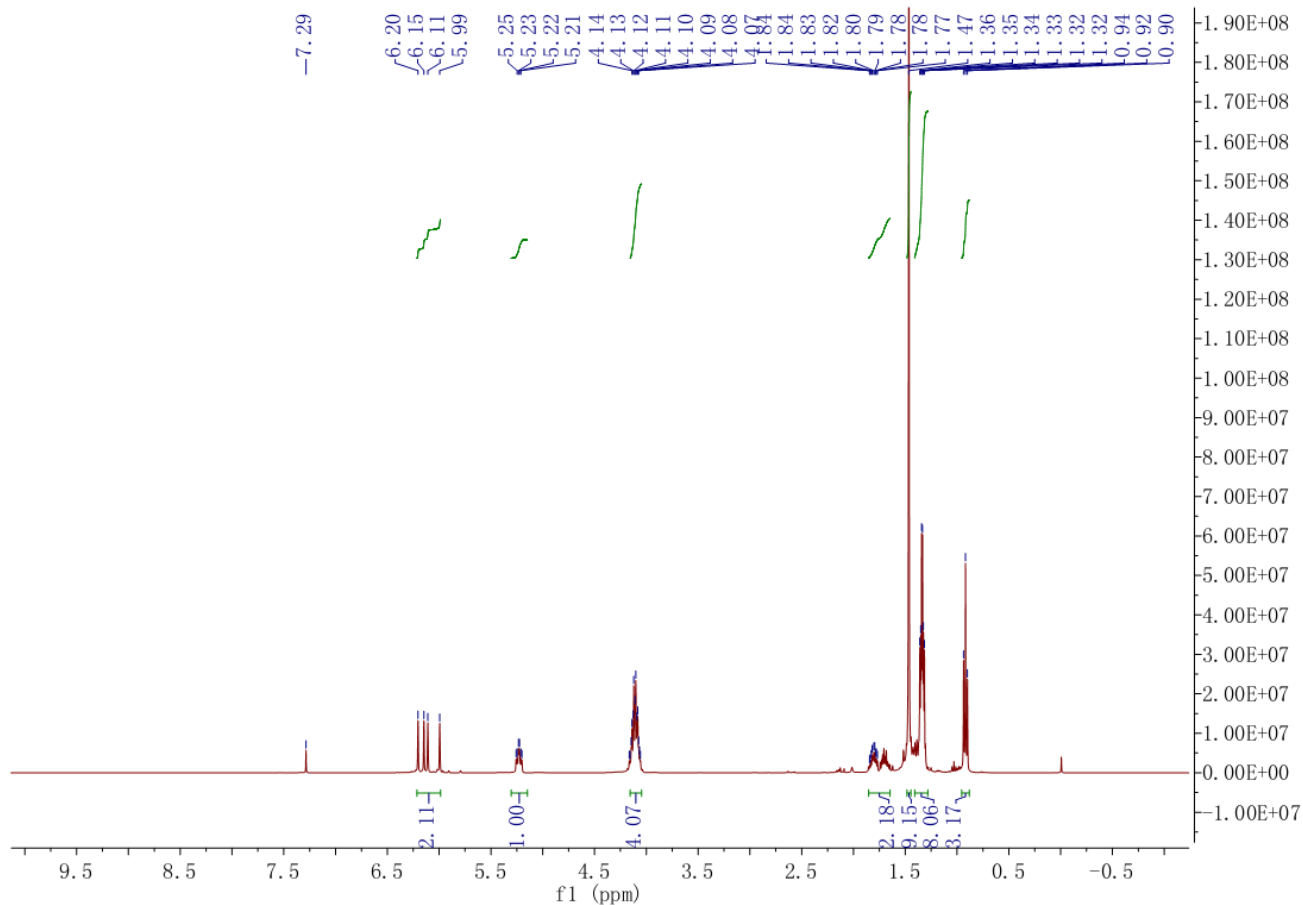


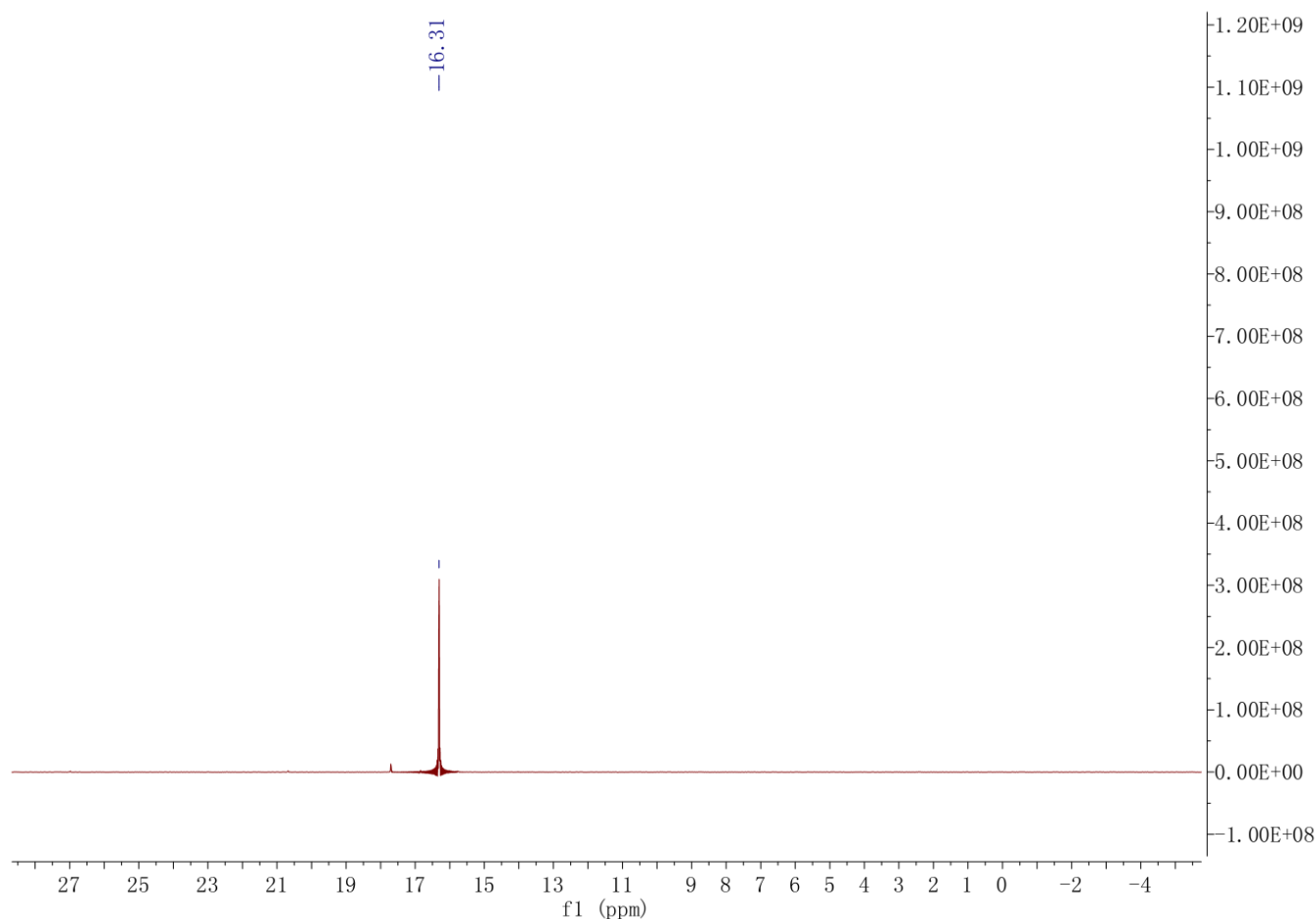




***tert*-Butyl (2-(diethoxyphosphoryl)hex-1-en-3-yl) carbonate(2c):**

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ 5.99-6.20 (m, 2H), 5.23 (td, $J = 9.1, 4.0$ Hz, 1H), 3.73-4.35 (m, 4H), 1.64-1.87 (m, 2H), 1.47 (s, 9H), 1.32-1.36 (m, 8H), 0.92 (t, $J = 7.4$ Hz, 3H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 152.68, 139.51 (d, $J = 174.6$ Hz), 130.05 (d, $J = 6.7$ Hz), 82.05, 75.37 (d, $J = 18.4$ Hz), 62.14 (d, $J = 5.4$ Hz), 61.96 (d, $J = 5.8$ Hz), 36.39, 27.75, 18.58, 16.27 (d, $J = 3.3$ Hz), 16.20 (d, $J = 3.3$ Hz), 13.62. $^{31}\text{P NMR}$ (162 MHz, CDCl_3): δ 16.31.



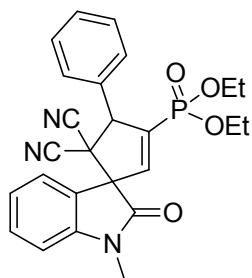


General procedure for the preparation of **3**:

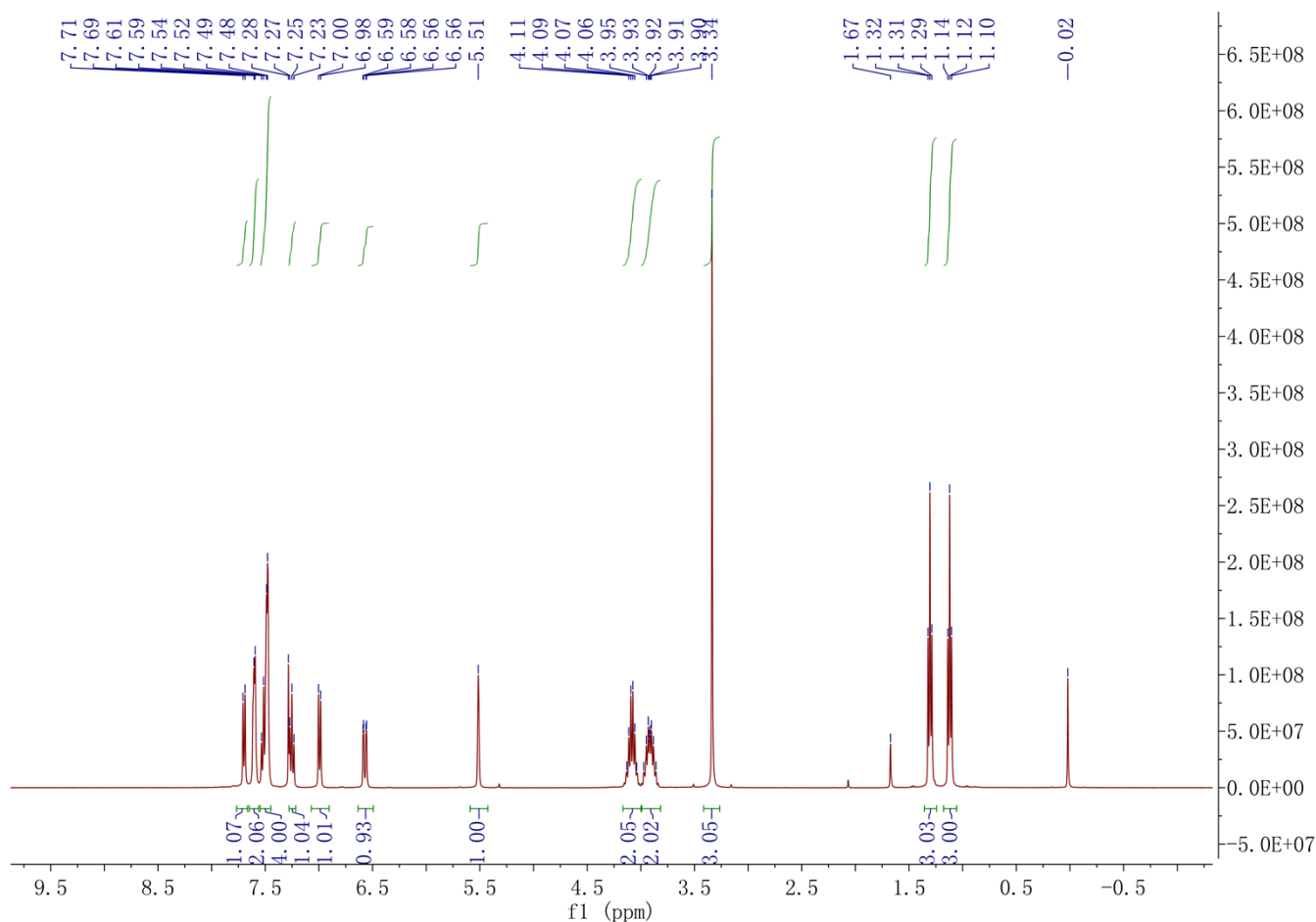
A solution of MBH phosphonates **2** (0.13 mmol), isatyridene malononitrile **1** (0.1 mmol) and catalyst Ph₂PEt (0.015 mmol) in toluene (2.0 mL) was stirred at 50°C under N₂ atmosphere. After isatyridene malononitrile **1** was completely consumed (monitored by TLC), the solvent was removed under reduced pressure and the residue was chromatographed on silica gel (elution with petroleum ether/EtOAc = 1:1) to afford product as a white solid.

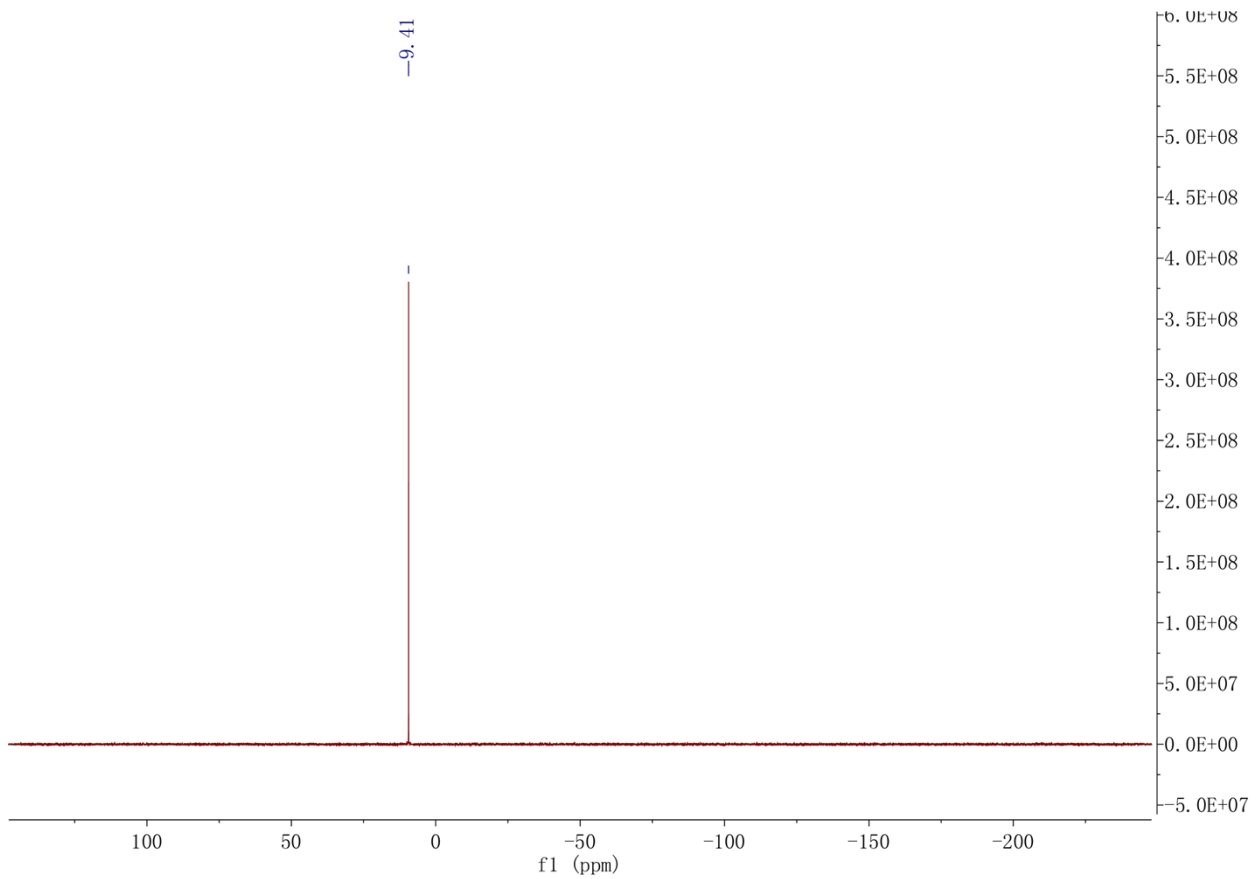
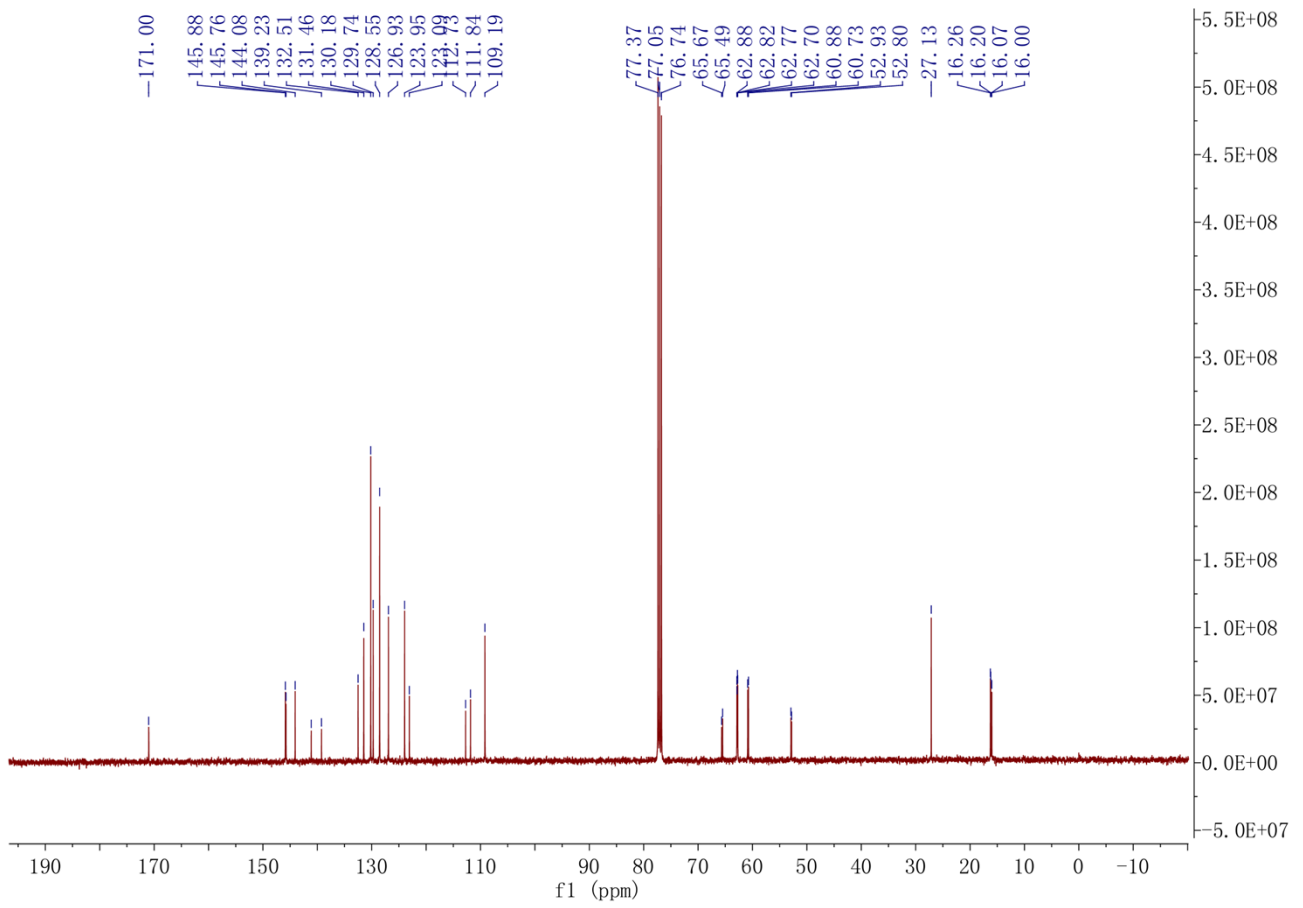
Diethyl(5,5-dicyano-1'-methyl-2'-oxo-4-phenylspiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate

(3a):

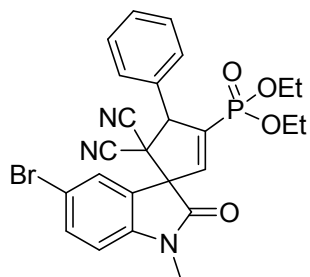


White solid; mp 233 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.70 (d, $J = 7.6$ Hz, 1H), 7.60 (d, $J = 4.5$ Hz, 2H), 7.50 (dd, $J = 16.7, 5.7$ Hz, 4H), 7.25 (t, $J = 7.7$ Hz, 1H), 6.99 (d, $J = 7.8$ Hz, 1H), 6.57 (dd, $J = 11.0, 2.2$ Hz, 1H), 5.51 (s, 1H), 4.00-4.17 (m, 2H), 3.80-4.01 (m, 2H), 3.34 (s, 3H), 1.31 (t, $J = 7.0$ Hz, 3H), 1.12 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 171.00, 145.82 (d, $J = 12.4$ Hz), 144.08, 140.18 (d, $J = 190.8$ Hz), 132.51, 131.46, 130.18, 129.74, 128.55, 126.93, 123.95, 123.09, 112.73, 111.84, 109.19, 65.58 (d, $J = 18.7$ Hz), 62.85 (d, $J = 5.9$ Hz), 62.74 (d, $J = 6.5$ Hz), 60.81 (d, $J = 15.1$ Hz), 52.87 (d, $J = 13.0$ Hz), 27.13, 16.23 (d, $J = 6.5$ Hz), 16.03 (d, $J = 6.5$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 9.41; HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{25}\text{H}_{24}\text{N}_3\text{O}_4\text{P}$: 484.1402, found: 484.1399.

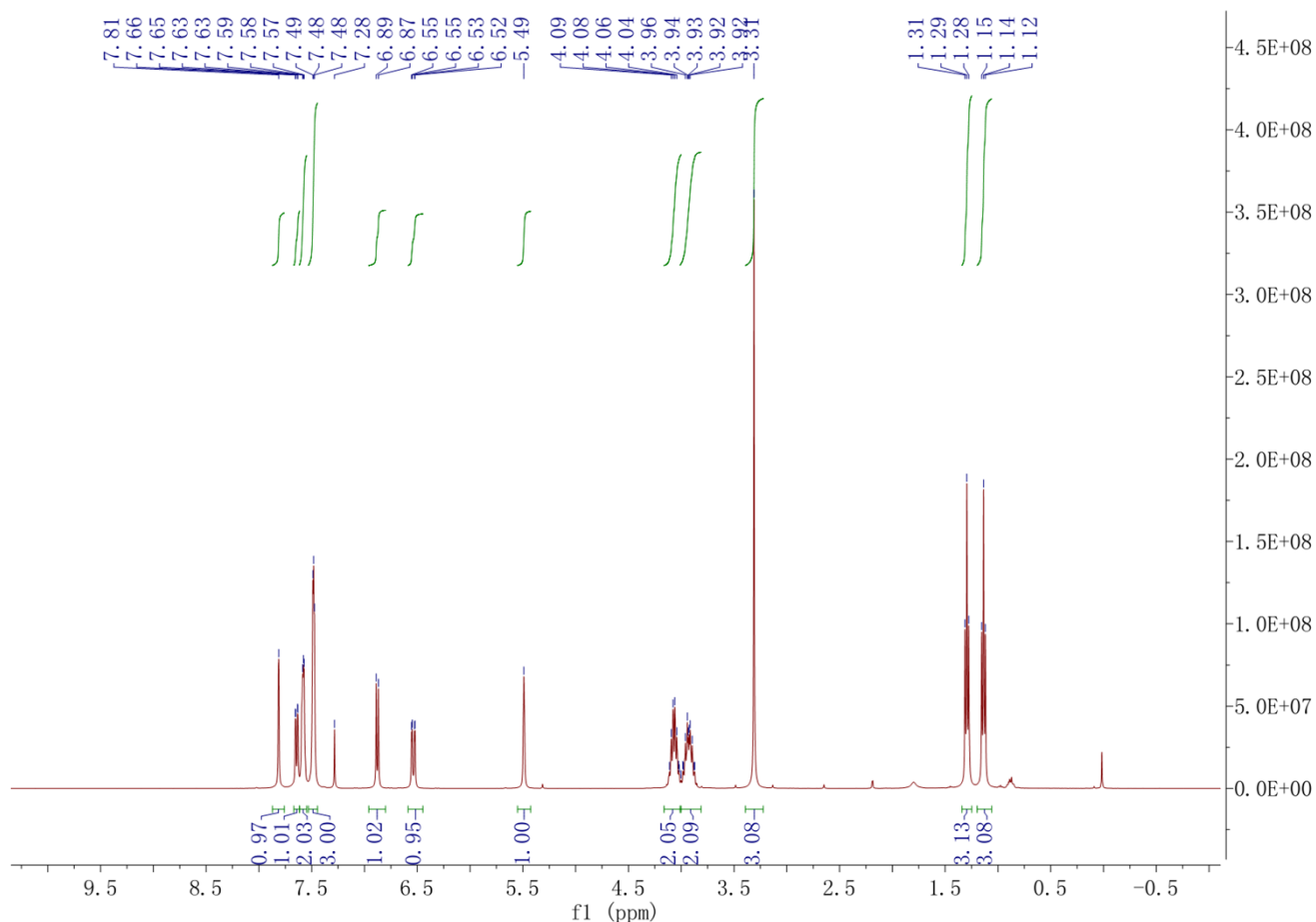


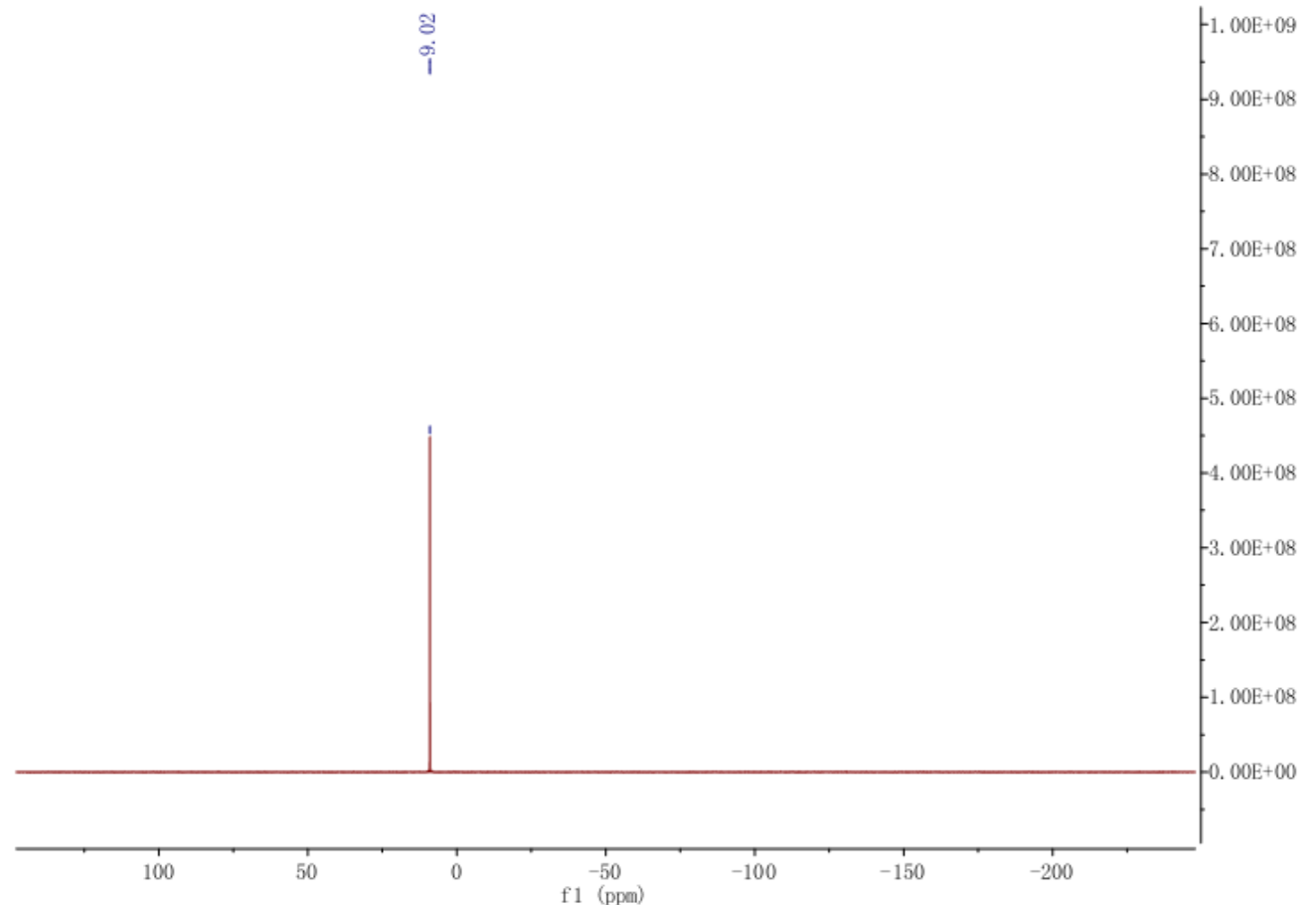
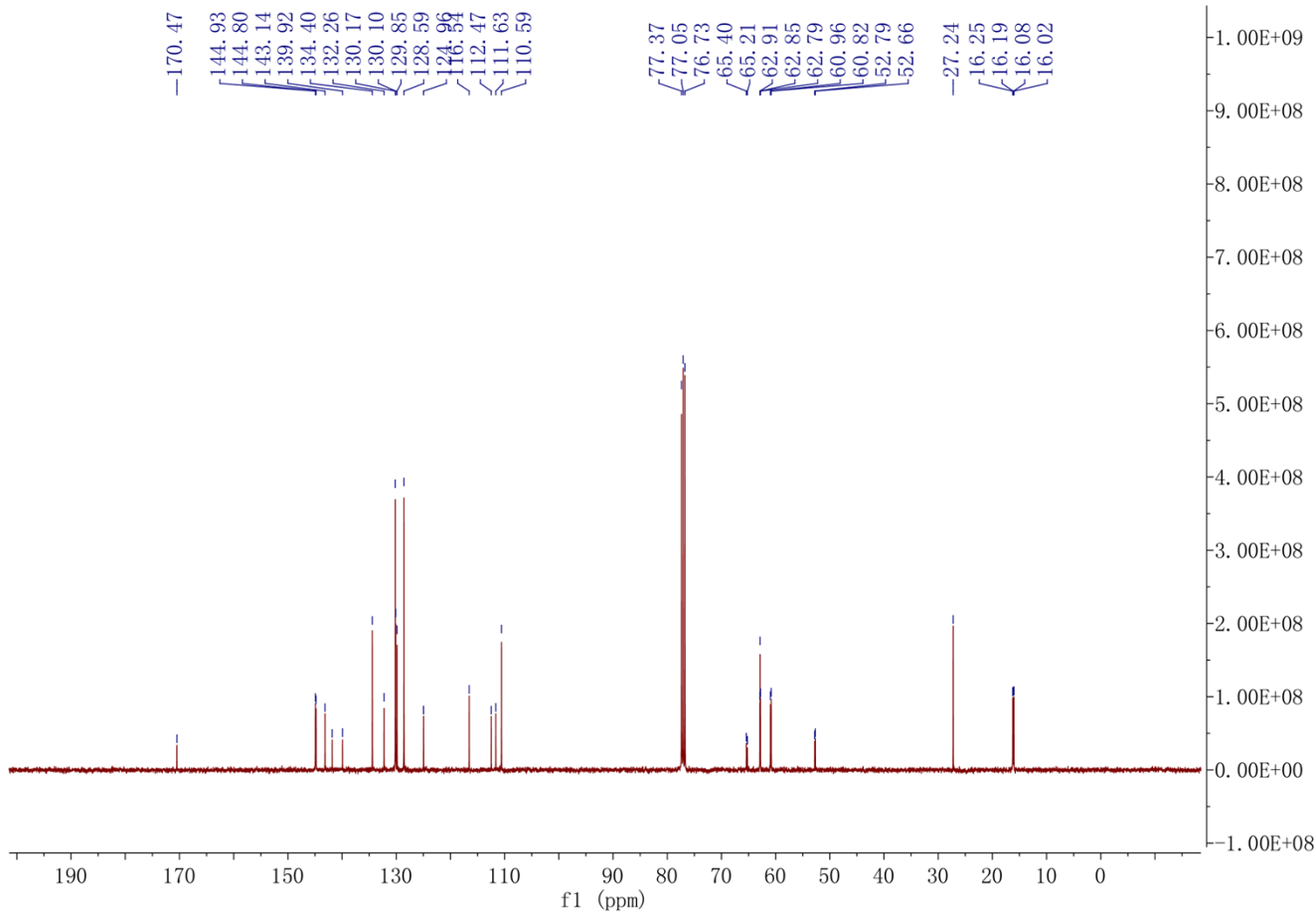


Diethyl(5'-bromo-5,5-dicyano-1'-methyl-2'-oxo-4-phenylspiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (3b):

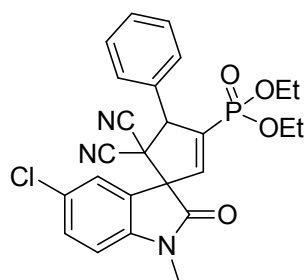


White solid; mp 185 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.81 (s, 1H), 7.64 (dt, $J = 9.8, 4.9$ Hz, 1H), 7.55-7.61 (m, 2H), 7.40-7.53 (m, 3H), 6.88 (d, $J = 8.3$ Hz, 1H), 6.54 (dd, $J = 10.9, 2.5$ Hz, 1H), 5.49 (s, 1H), 4.01-4.19 (m, 2H), 3.82-4.00 (m, 2H), 3.31 (s, 3H), 1.29 (t, $J = 7.0$ Hz, 3H), 1.14 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 170.47, 144.86 (d, $J = 12.7$ Hz), 143.14, 140.87 (d, $J = 190.6$ Hz), 134.40, 132.26, 130.17, 130.10, 129.85, 128.59, 124.96, 116.54, 112.47, 111.63, 110.59, 65.31 (d, $J = 19.2$ Hz), 62.85 (t, $J = 5.9$ Hz), 60.89 (d, $J = 14.7$ Hz), 52.73 (d, $J = 13.0$ Hz), 27.24, 16.22 (d, $J = 6.4$ Hz), 16.05 (d, $J = 6.5$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 9.02. HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{25}\text{H}_{23}\text{BrN}_3\text{O}_4\text{P}$: 562.0507, found: 562.0504.

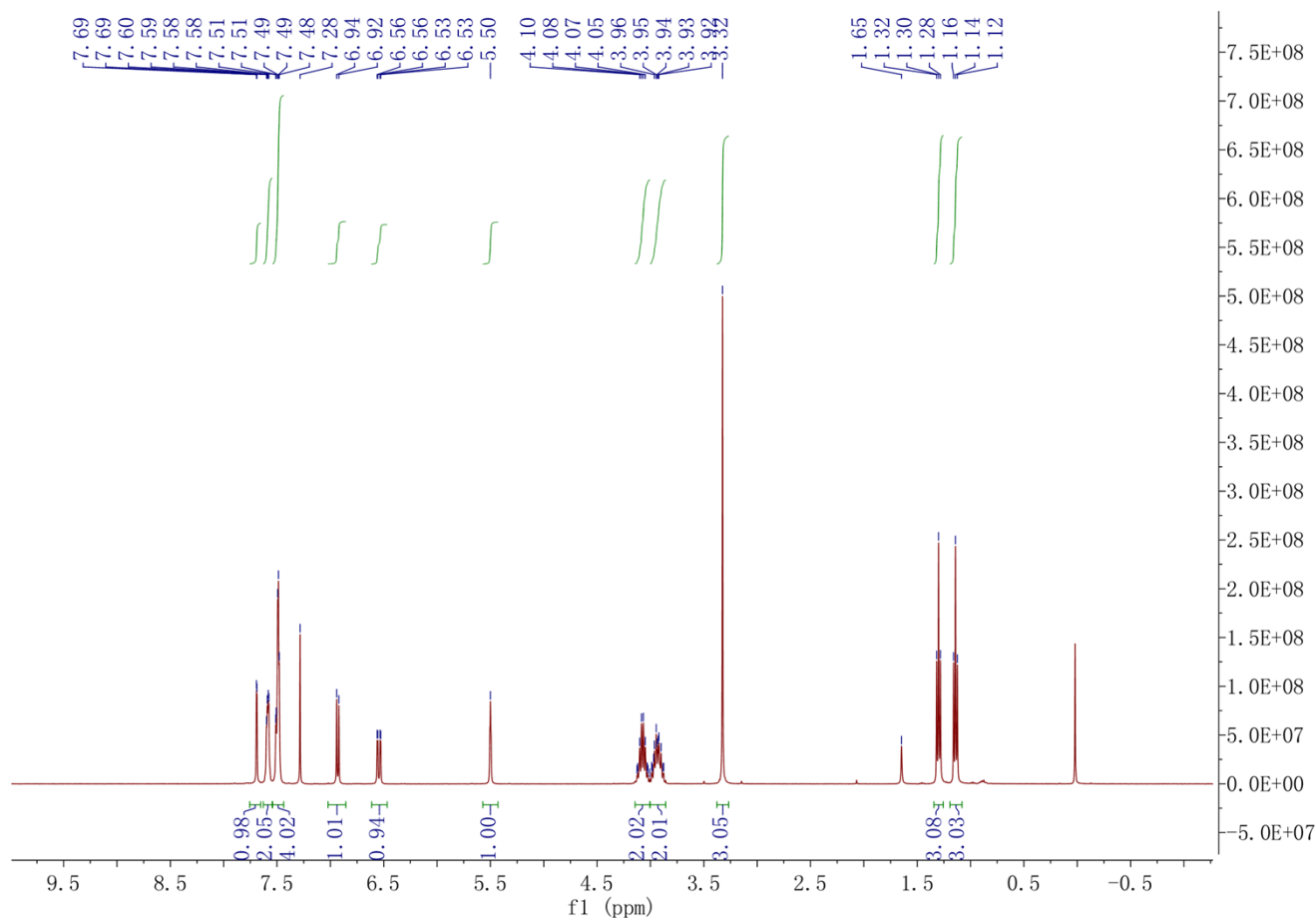


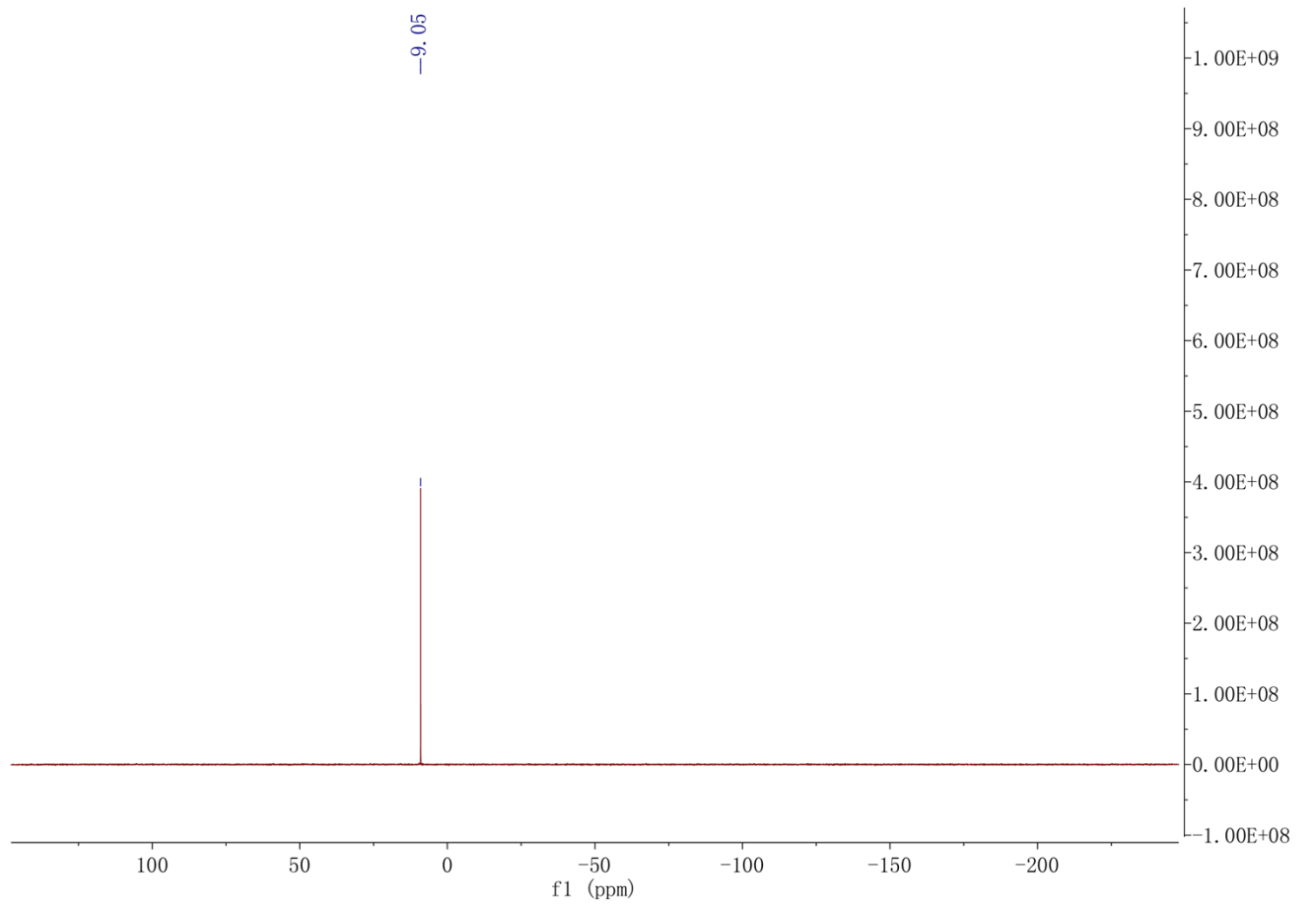
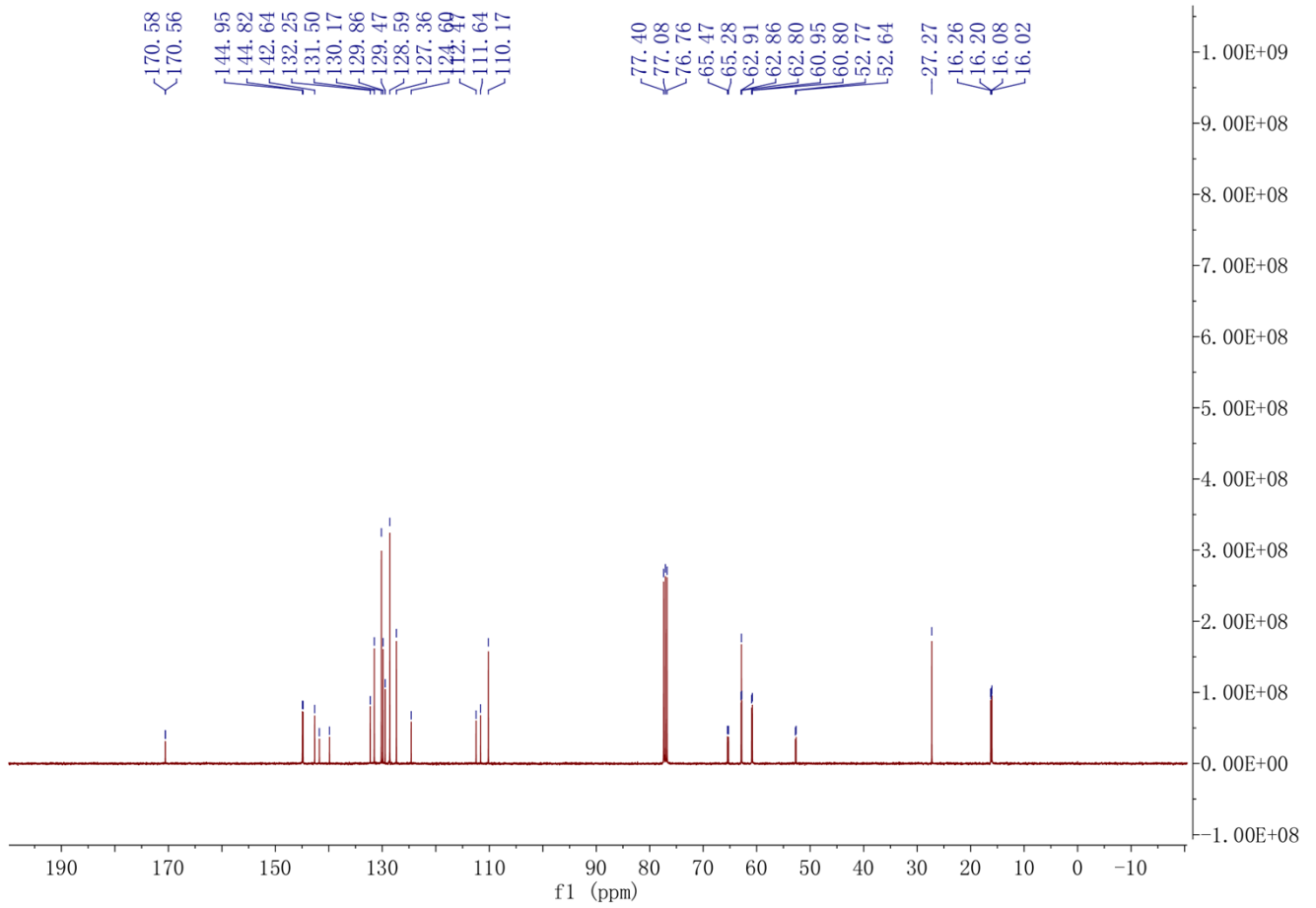


Diethyl(5'-chloro-5,5-dicyano-1'-methyl-2'-oxo-4-phenylspiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (3c):

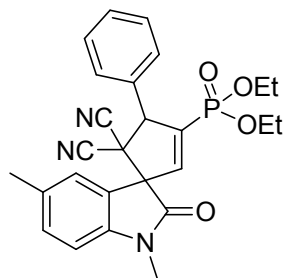


White solid; mp 225 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.69 (d, $J = 1.7$ Hz, 1H), 7.59 (dd, $J = 6.2, 2.7$ Hz, 2H), 7.44-7.53 (m, 4H), 6.93 (d, $J = 8.4$ Hz, 1H), 6.54 (dd, $J = 11.0, 2.6$ Hz, 1H), 5.50 (s, 1H), 4.02-4.18 (m, 2H), 3.85-4.01 (m, 2H), 3.32 (s, 3H), 1.30 (t, $J = 7.1$ Hz, 3H), 1.14 (t, $J = 7.1$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 170.57 (d, $J = 2.3$ Hz), 144.89 (d, $J = 12.8$ Hz), 142.64, 140.82 (d, $J = 190.6$ Hz), 132.25, 131.50, 130.17, 129.86, 129.47, 128.59, 127.36, 124.60, 112.47, 111.64, 110.17, 65.37 (d, $J = 19.0$ Hz), 62.86 (t, $J = 6.0$ Hz), 60.87 (d, $J = 14.9$ Hz), 52.70 (d, $J = 12.9$ Hz), 27.27, 16.23 (d, $J = 6.4$ Hz), 16.05 (d, $J = 6.5$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 9.05. HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{25}\text{H}_{23}\text{ClN}_3\text{O}_4\text{P}$: 518.1012, found: 518.1005.



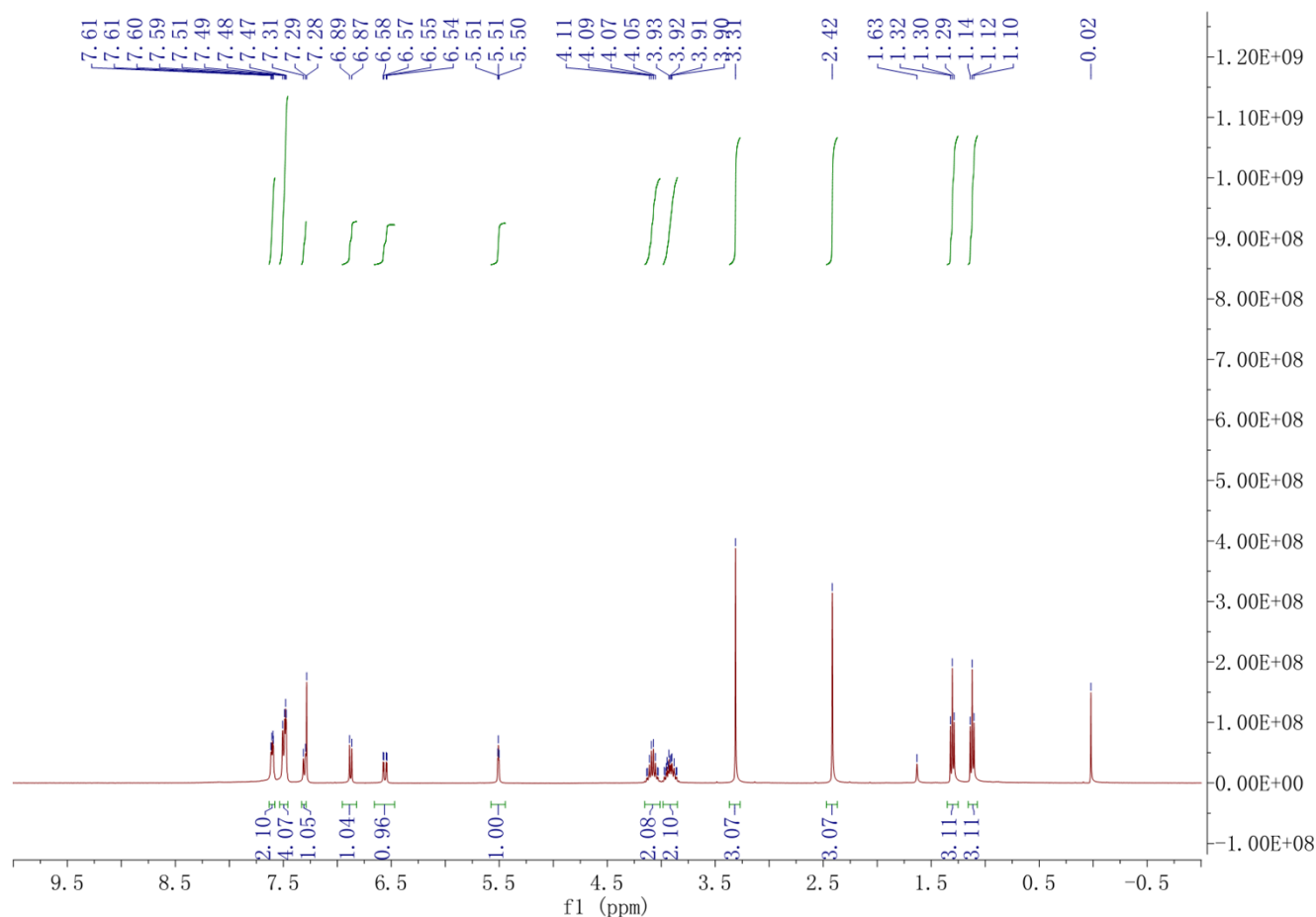


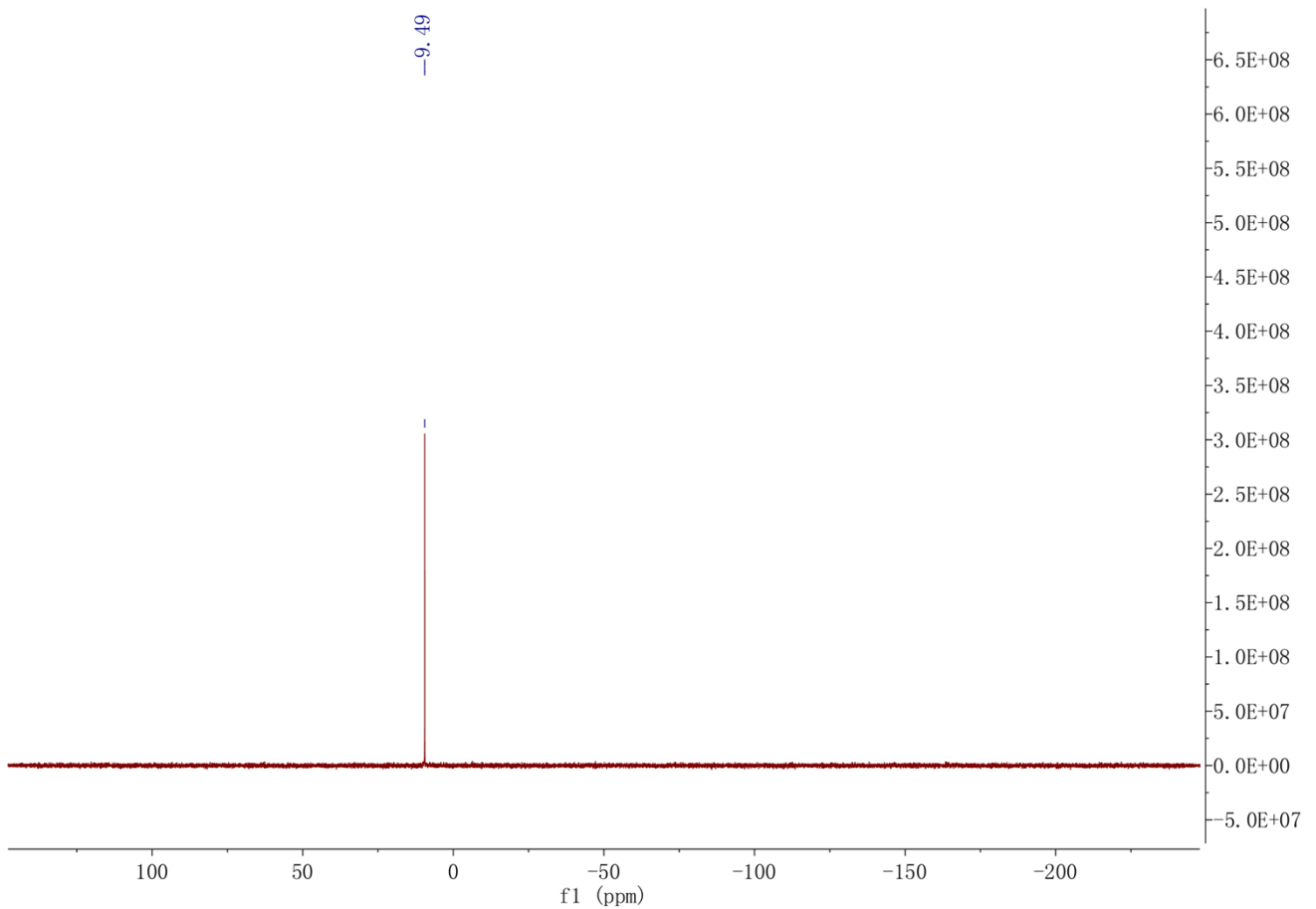
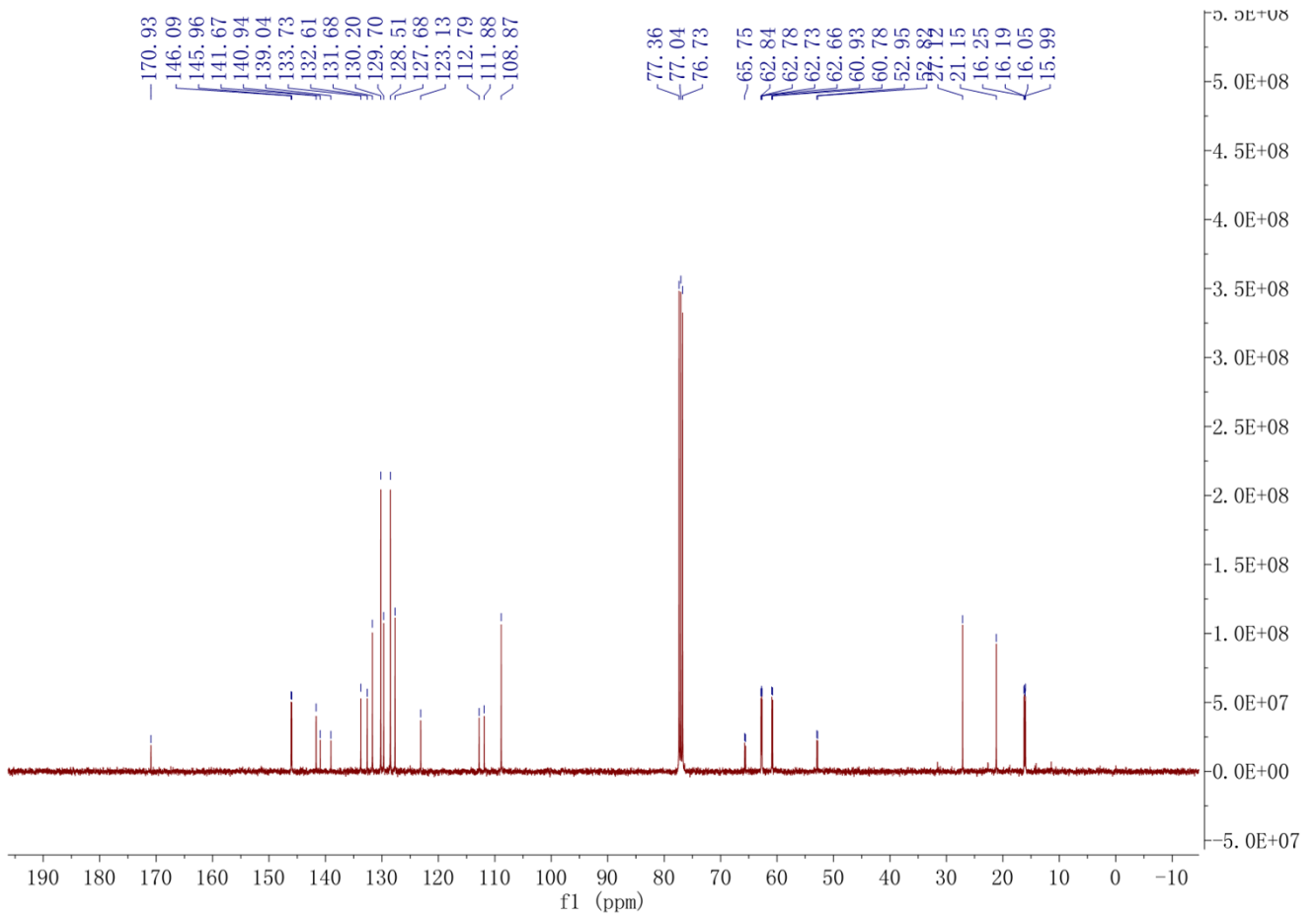
Diethyl(5,5-dicyano-1',5'-dimethyl-2'-oxo-4-phenylspiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (3d):



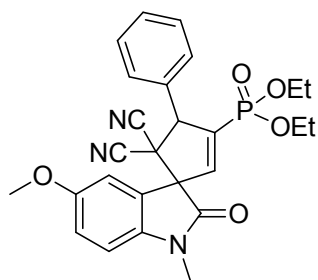
White solid; mp 195 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.60 (dd, $J = 6.5, 2.8$ Hz, 2H), 7.49 (dd, $J = 8.7, 4.7$ Hz, 4H), 7.30 (d, $J = 8.2$ Hz, 1H), 6.88 (d, $J = 8.0$ Hz, 1H), 6.56 (dd, $J = 11.0, 2.7$ Hz, 1H), 5.51 (t, $J = 2.2$ Hz, 1H), 4.01-4.18 (m, 2H), 3.82-4.00 (m, 2H), 3.31 (s, 3H), 2.42 (s, 3H), 1.30 (t, $J = 7.1$ Hz, 3H), 1.12 (t, $J =$

7.1 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 170.93, 146.02 (d, $J = 12.4$ Hz), 141.67, 139.99 (d, $J = 190.7$ Hz), 133.73, 132.61, 131.68, 130.20, 129.70, 128.51, 127.68, 123.13, 112.79, 111.88, 108.87, 65.65 (d, $J = 19.1$ Hz), 62.81 (d, $J = 6.0$ Hz), 62.69 (d, $J = 6.3$ Hz), 60.85 (d, $J = 15.0$ Hz), 52.89 (d, $J = 13.0$ Hz), 27.12, 21.15, 16.22 (d, $J = 6.4$ Hz), 16.02 (d, $J = 6.6$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 9.49. HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{26}\text{H}_{26}\text{N}_3\text{O}_4\text{P}$: 498.1559, found: 498.1561.

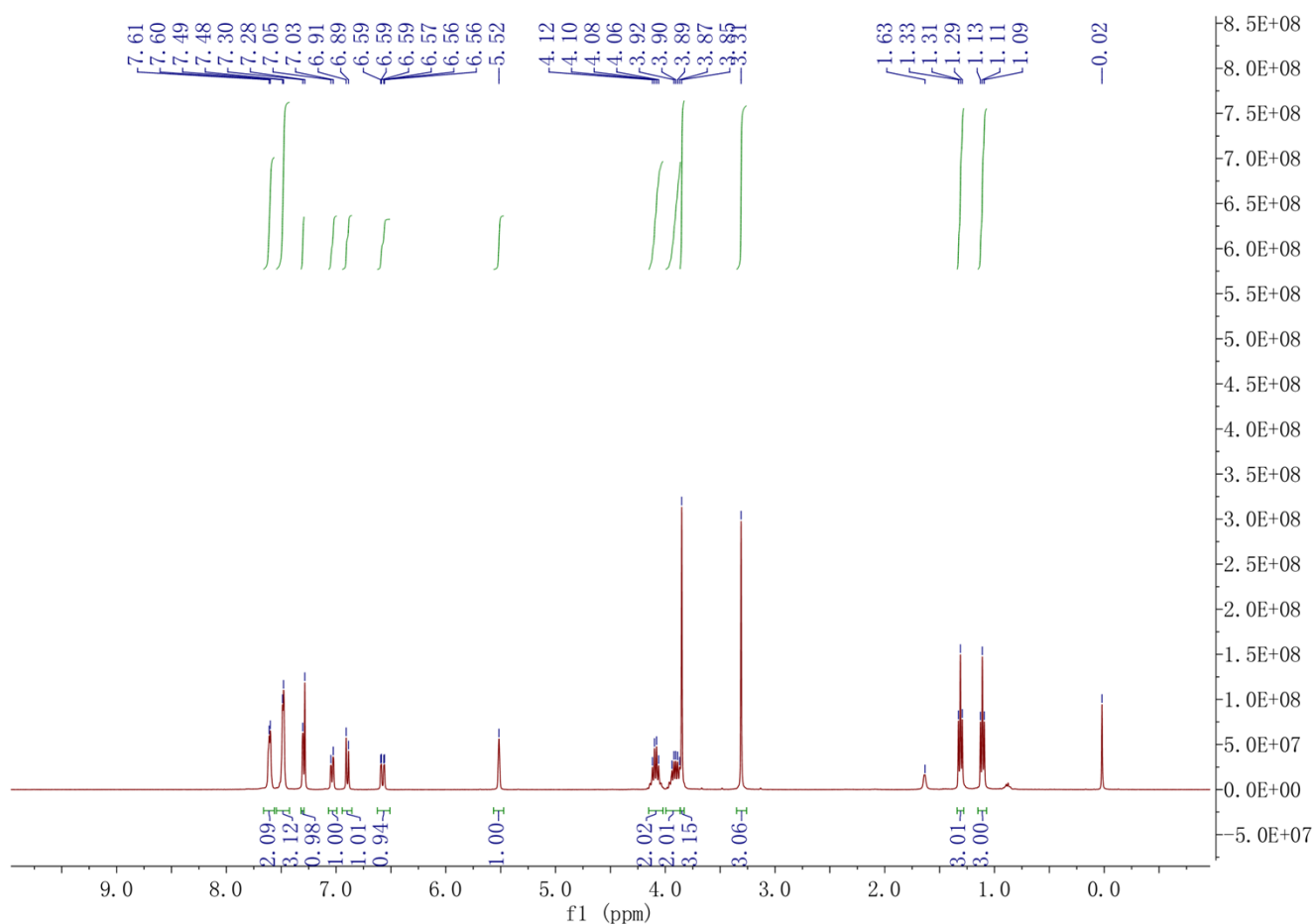


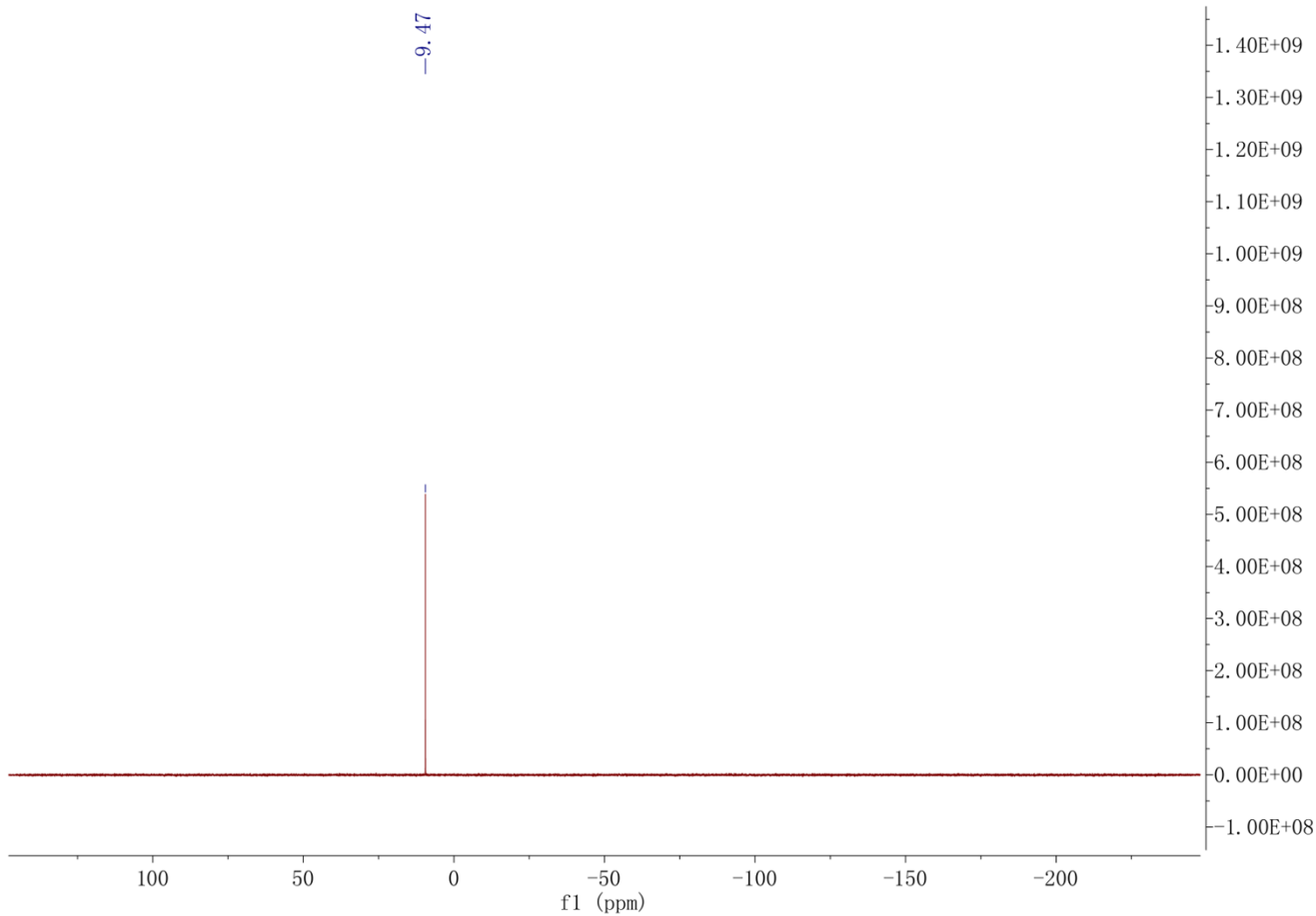
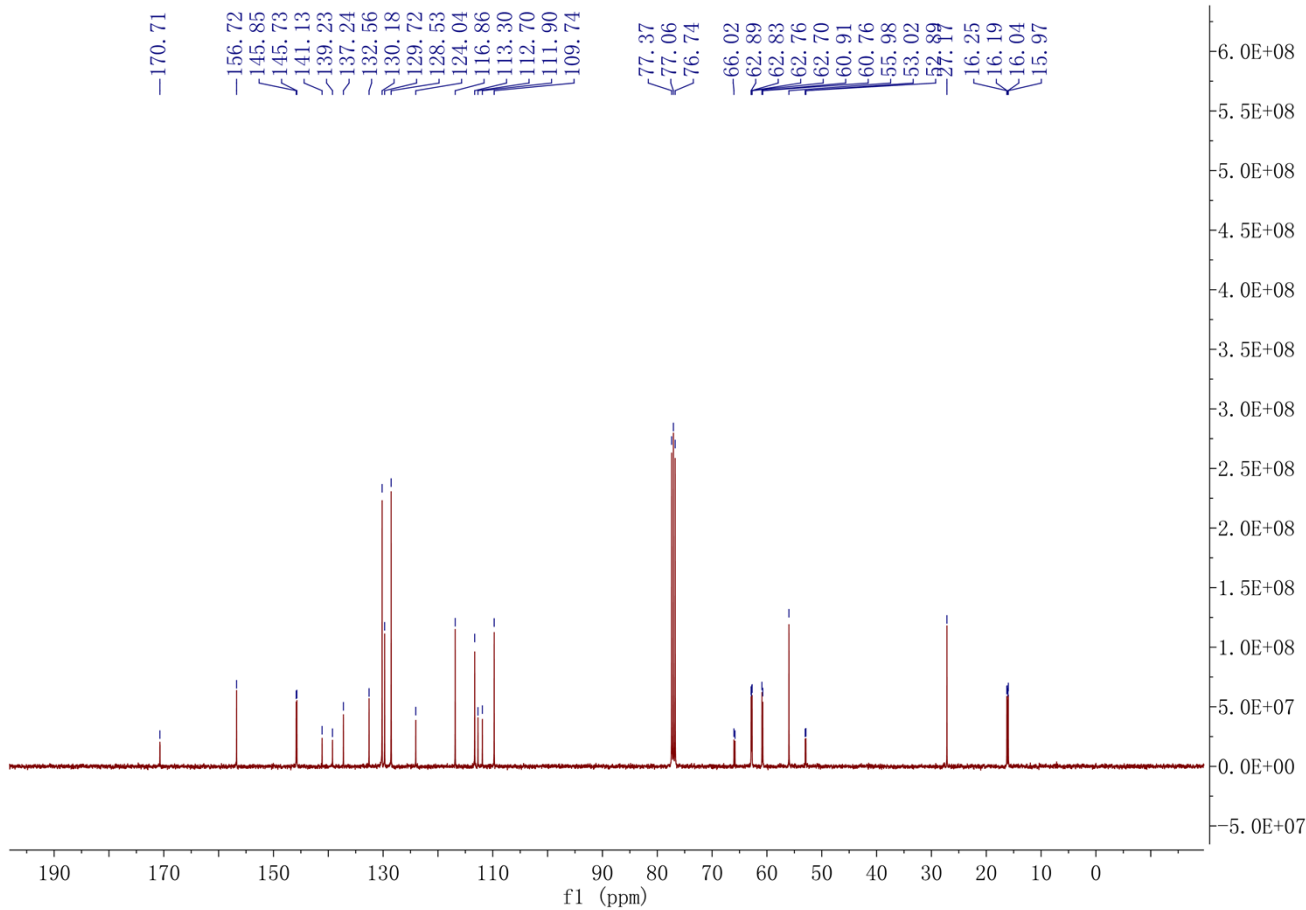


Diethyl(5,5-dicyano-5'-methoxy-1'-methyl-2'-oxo-4-phenylspiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (3e):

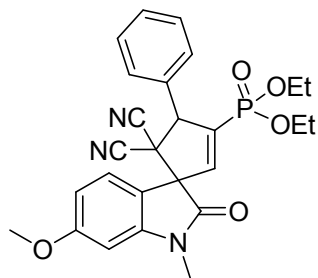


White solid; mp 205 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.60 (d, $J = 4.4$ Hz, 2H), 7.48 (d, $J = 3.8$ Hz, 3H), 7.30 (s, 1H), 7.04 (d, $J = 8.6$ Hz, 1H), 6.90 (d, $J = 8.6$ Hz, 1H), 6.53-6.63 (m, 1H), 5.52 (s, 1H), 4.03-4.15 (m, 2H), 3.88-3.97 (m, 2H), 3.85 (s, 3H), 3.31 (s, 3H), 1.31 (t, $J = 7.0$ Hz, 3H), 1.11 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 170.71, 156.72, 145.79 (d, $J = 12.7$ Hz), 140.18 (d, $J = 190.9$ Hz), 137.24, 132.56, 130.18, 129.72, 128.53, 124.04, 116.86, 113.30, 112.70, 111.90, 109.74, 65.92 (d, $J = 19.1$ Hz), 62.86 (d, $J = 5.9$ Hz), 62.73 (d, $J = 6.4$ Hz), 60.83 (d, $J = 14.9$ Hz), 55.98, 52.95 (d, $J = 13.0$ Hz), 27.17, 16.22 (d, $J = 6.4$ Hz), 16.01 (d, $J = 6.5$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 9.47. HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{26}\text{H}_{26}\text{N}_3\text{O}_5\text{P}$: 514.1508, found: 514.1503.

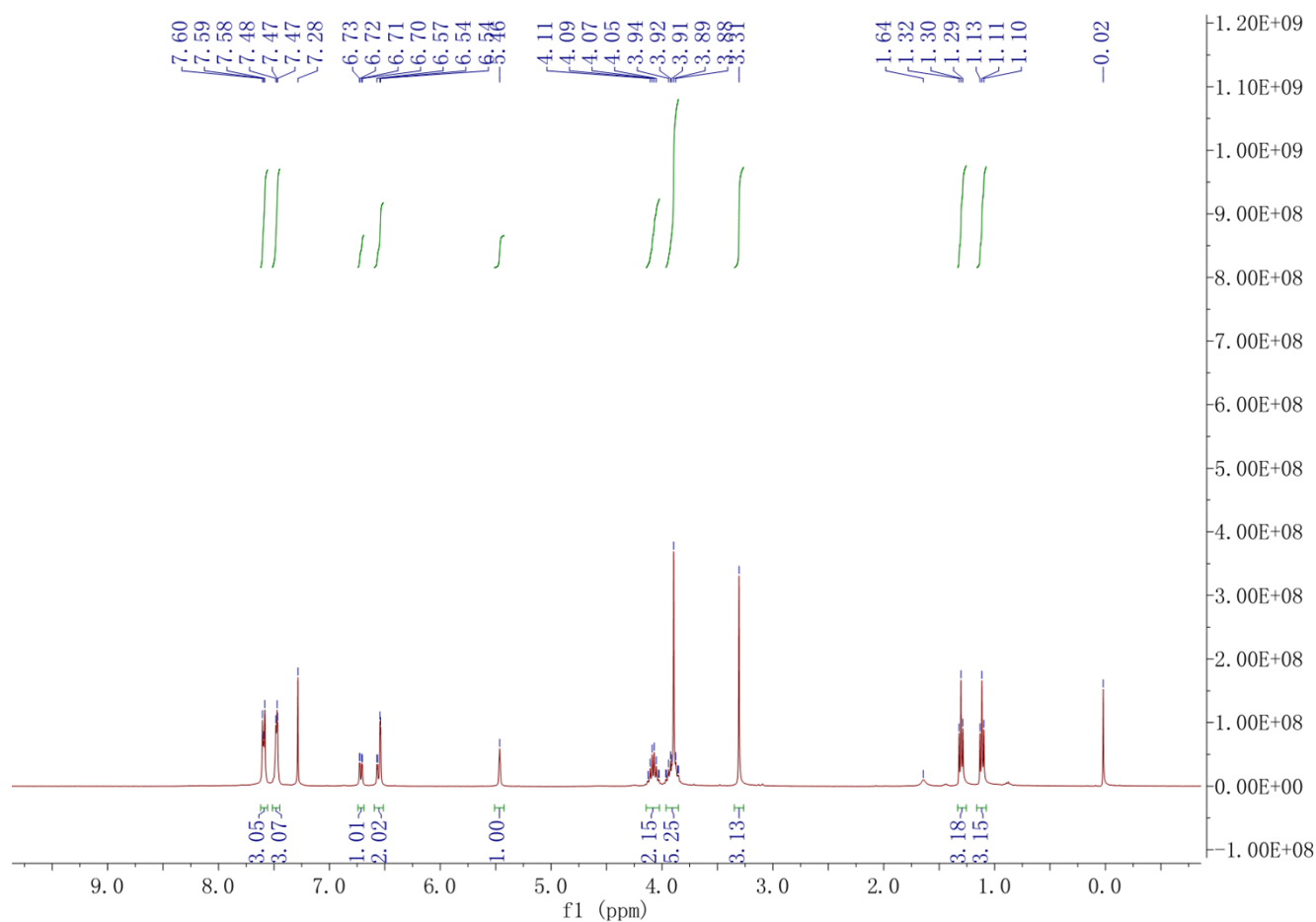


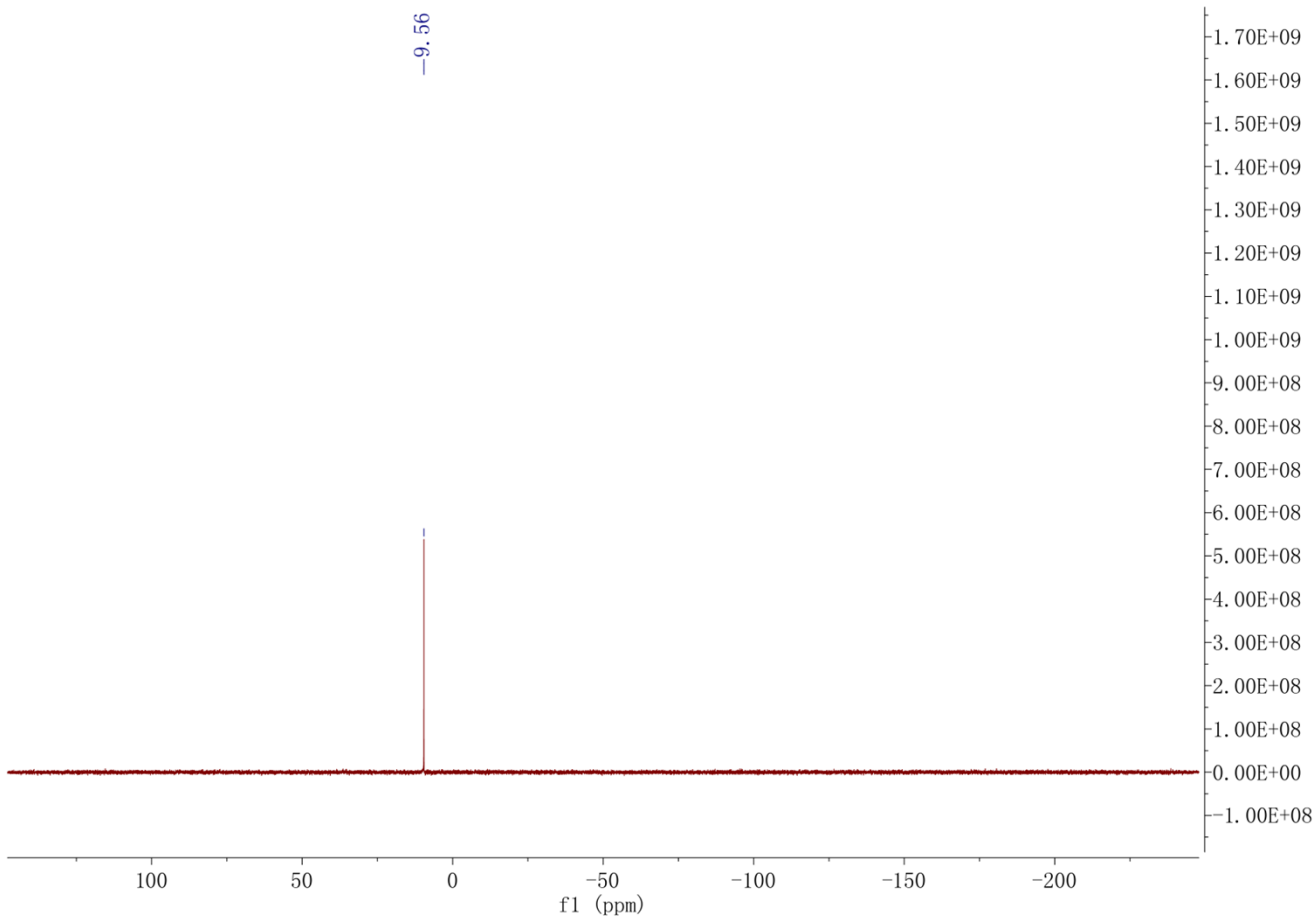
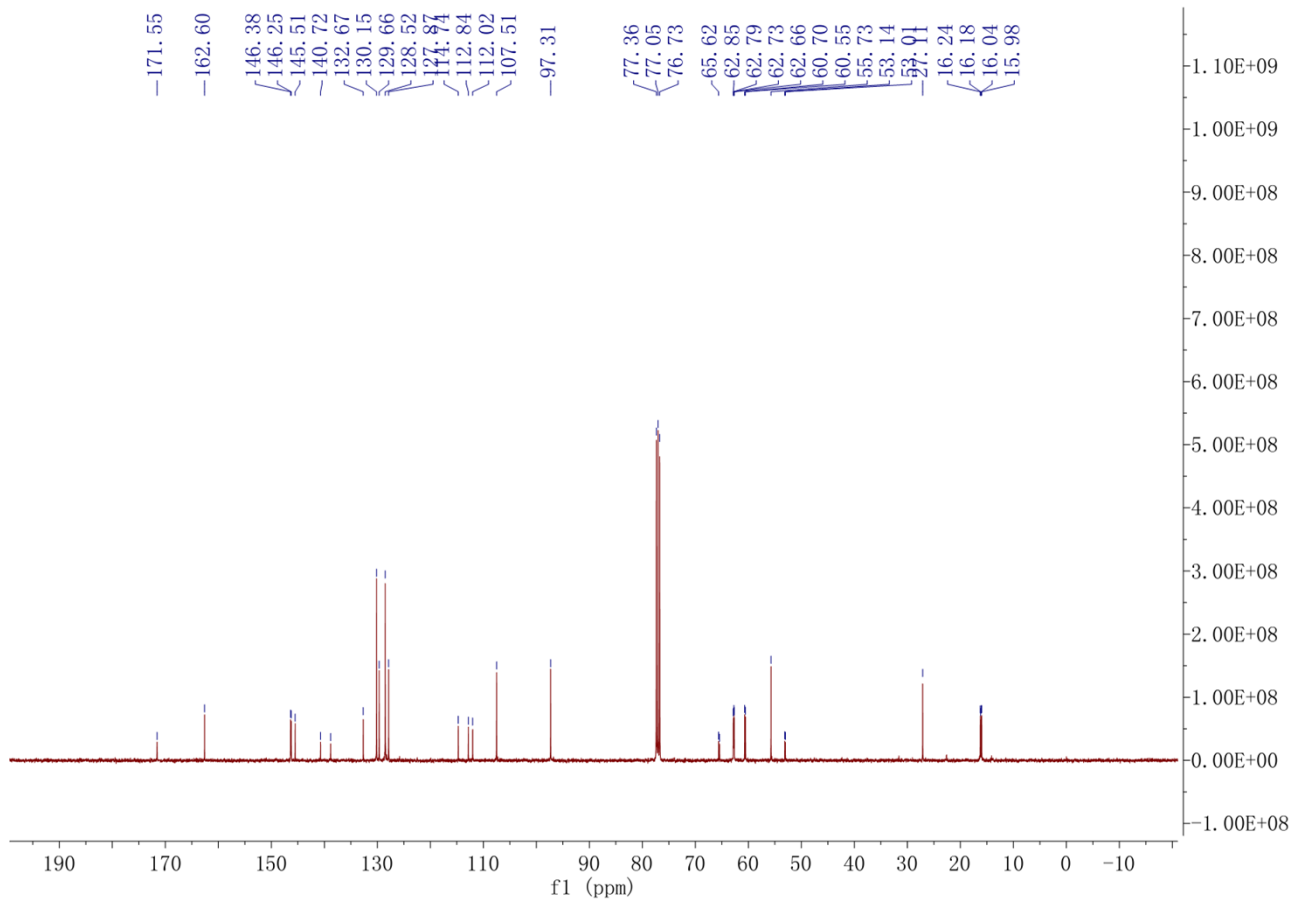


Diethyl(5,5-dicyano-6'-methoxy-1'-methyl-2'-oxo-4-phenylspiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (3f):

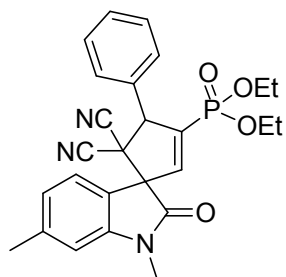


White solid; mp 155 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.55-7.64 (m, 3H), 7.42-7.52 (m, 3H), 6.72 (dd, $J = 8.4, 2.1$ Hz, 1H), 6.55 (dd, $J = 11.0, 2.4$ Hz, 2H), 5.46 (s, 1H), 4.02-4.16 (m, 2H), 3.83-3.97 (m, 5H), 3.31 (s, 3H), 1.30 (t, $J = 7.0$ Hz, 3H), 1.11 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 171.55, 162.60, 146.32 (d, $J = 12.4$ Hz), 145.51, 139.77 (d, $J = 191.1$ Hz), 132.67, 130.15, 129.66, 128.52, 127.87, 114.74, 112.84, 112.02, 107.51, 97.31, 65.52 (d, $J = 19.3$ Hz), 62.82 (d, $J = 5.8$ Hz), 62.70 (d, $J = 6.3$ Hz), 60.62 (d, $J = 15.0$ Hz), 55.73, 53.08 (d, $J = 12.7$ Hz), 27.11, 16.21 (d, $J = 6.4$ Hz), 16.01 (d, $J = 6.5$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 9.56. HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{26}\text{H}_{26}\text{N}_3\text{O}_5\text{P}$: 514.1508, found: 514.1508.





Diethyl(5,5-dicyano-1',6'-dimethyl-2'-oxo-4-phenylspiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (3g):



White solid; mp 181 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.59 (d, $J = 4.0$ Hz, 2H),

7.49 (dd, $J = 10.7, 5.7$ Hz, 4H), 7.23 (d, $J = 7.7$ Hz, 1H), 7.12 (t, $J = 7.6$ Hz, 1H),

6.55 (d, $J = 11.0$ Hz, 1H), 5.50 (s, 1H), 4.00-4.15 (m, 2H), 3.83-3.98 (m, 2H),

3.60 (s, 3H), 2.64 (s, 3H), 1.30 (t, $J = 7.0$ Hz, 3H), 1.12 (t, $J = 7.0$ Hz, 3H); ^{13}C

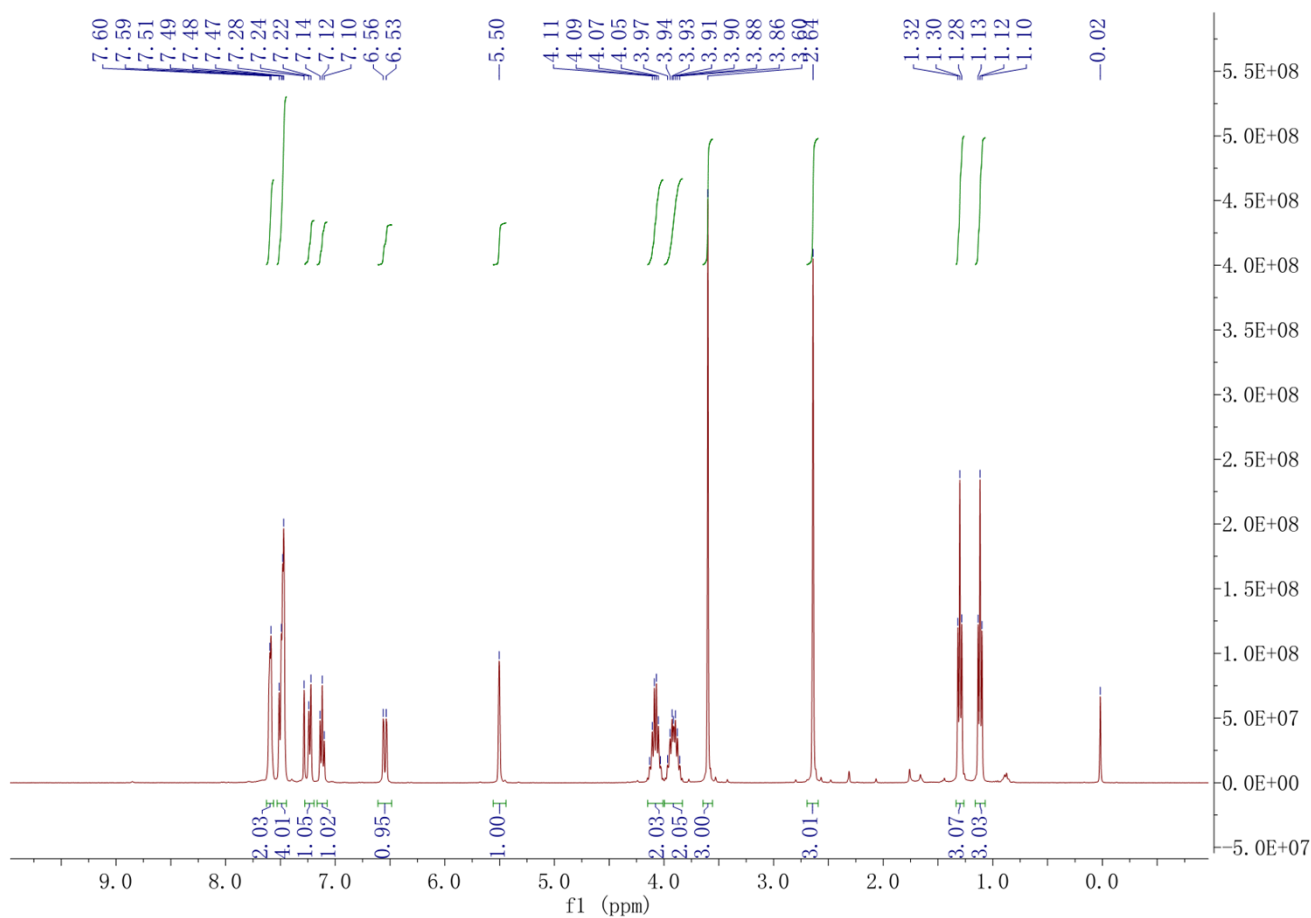
NMR (101 MHz, CDCl_3): δ 171.77 (d, $J = 2.4$ Hz), 146.05 (d, $J = 12.6$ Hz), 141.79, 139.94 (d, $J = 190.8$

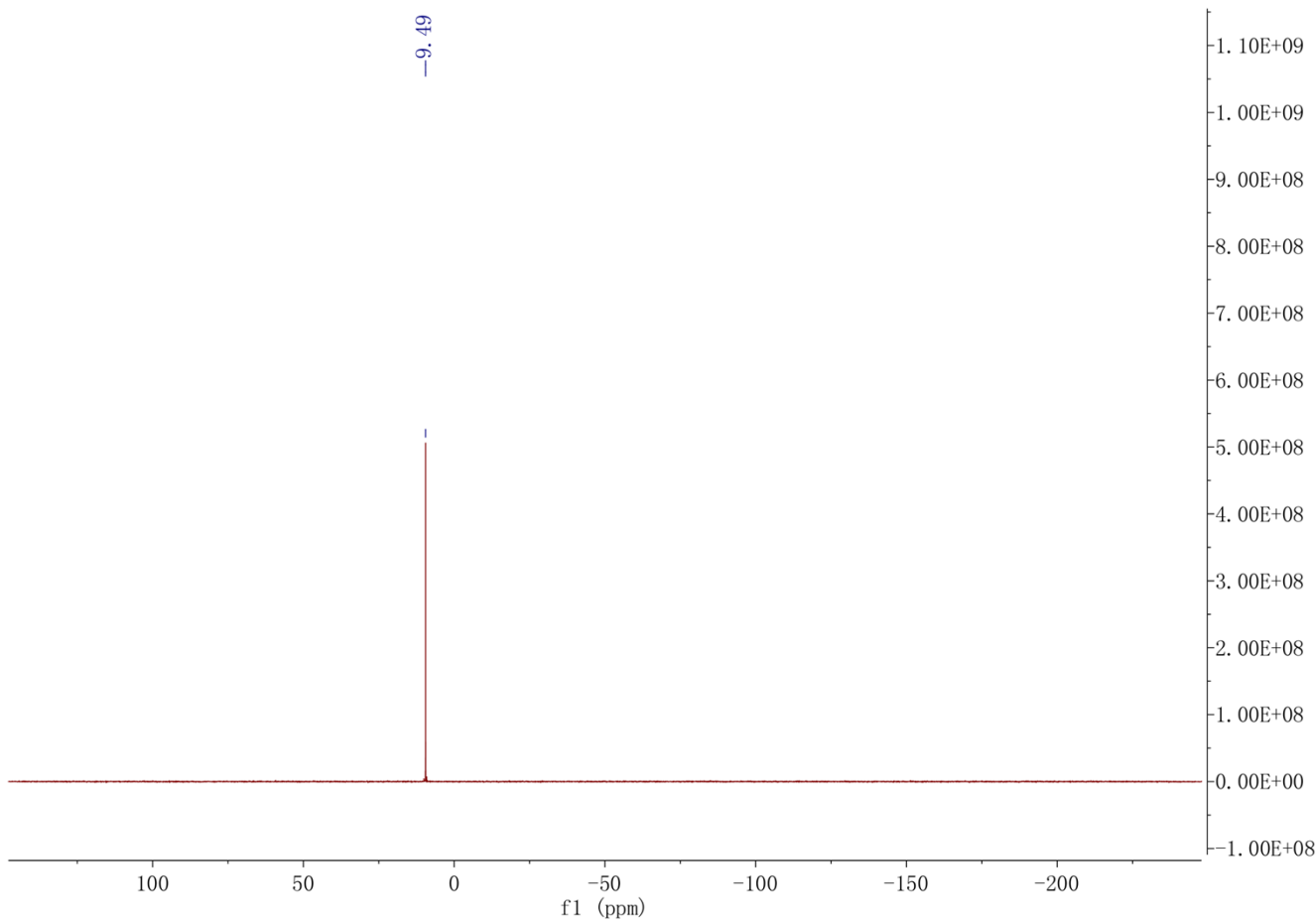
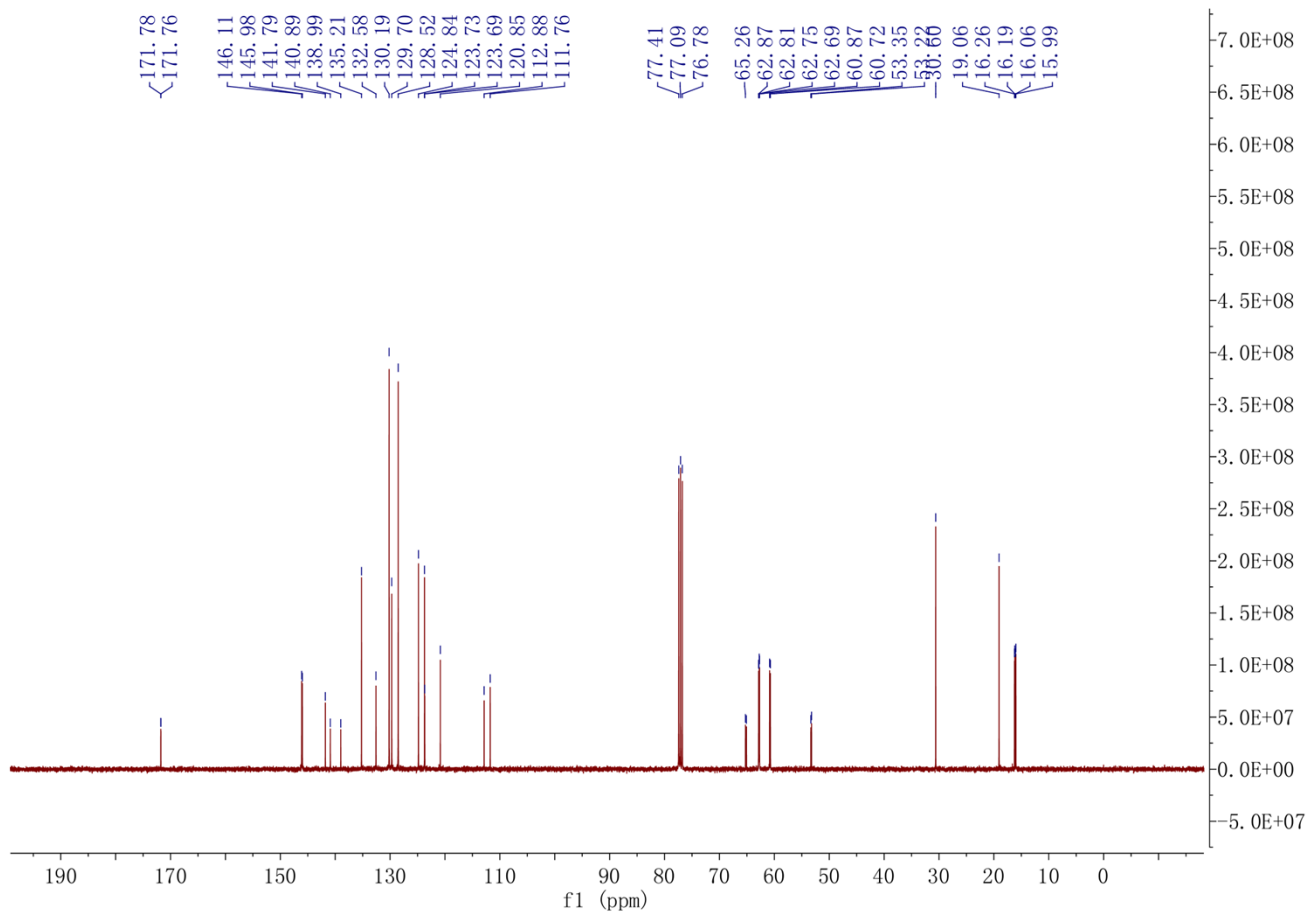
Hz), 135.21, 132.58, 130.19, 129.70, 128.52, 124.84, 123.73, 123.69, 120.85, 112.88, 111.76, 65.17 (d, J

$= 18.9$ Hz), 62.84 (d, $J = 6.0$ Hz), 62.72 (d, $J = 6.3$ Hz), 60.79 (d, $J = 15.0$ Hz), 53.28 (d, $J = 12.8$ Hz),

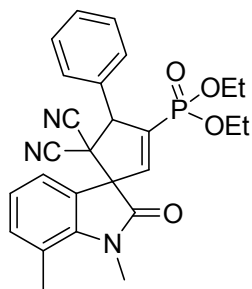
30.60, 19.06, 16.22 (d, $J = 6.4$ Hz), 16.03 (d, $J = 6.5$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 9.49. HRMS

calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{26}\text{H}_{26}\text{N}_3\text{O}_4\text{P}$: 498.1559, found: 498.1558.

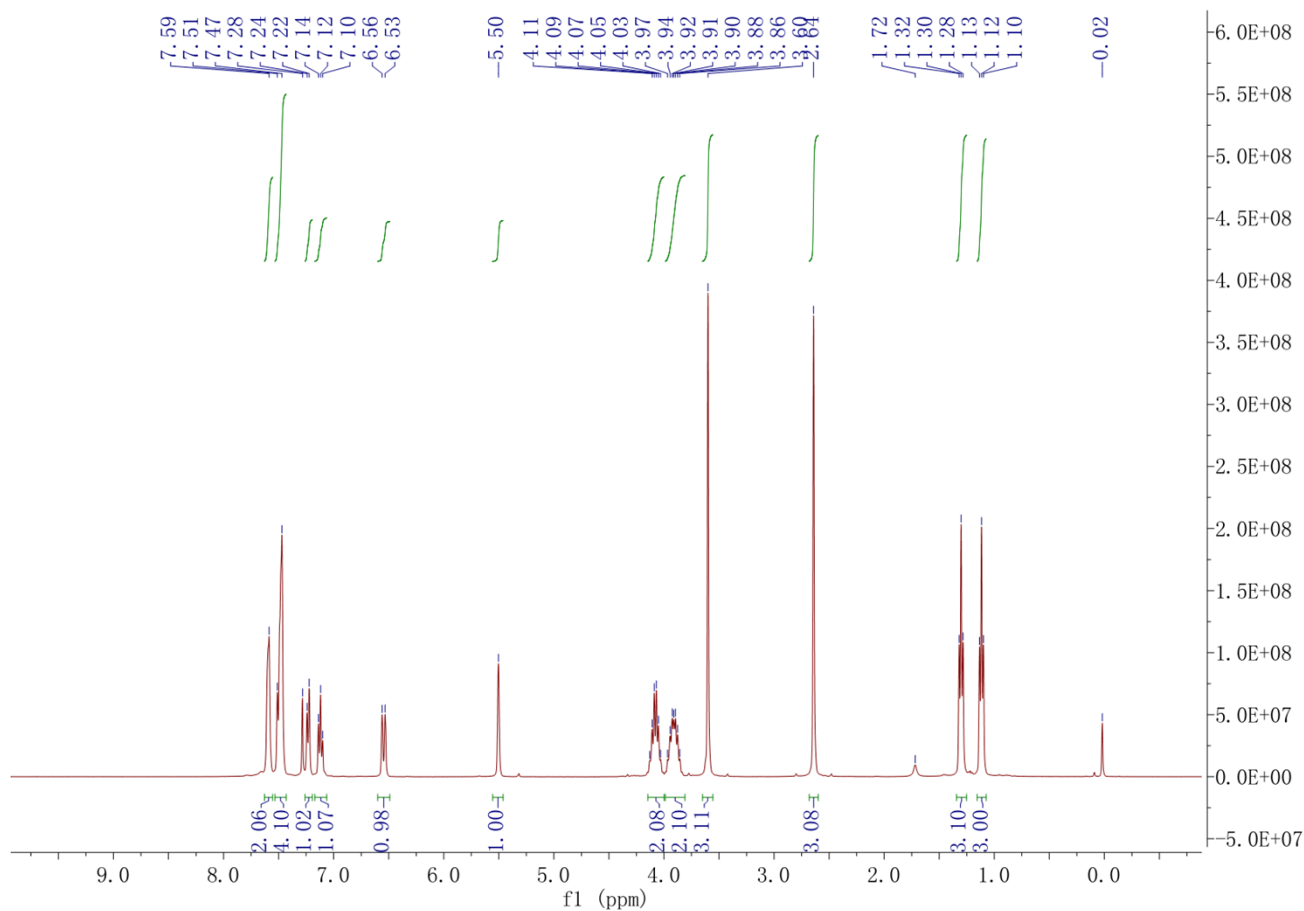


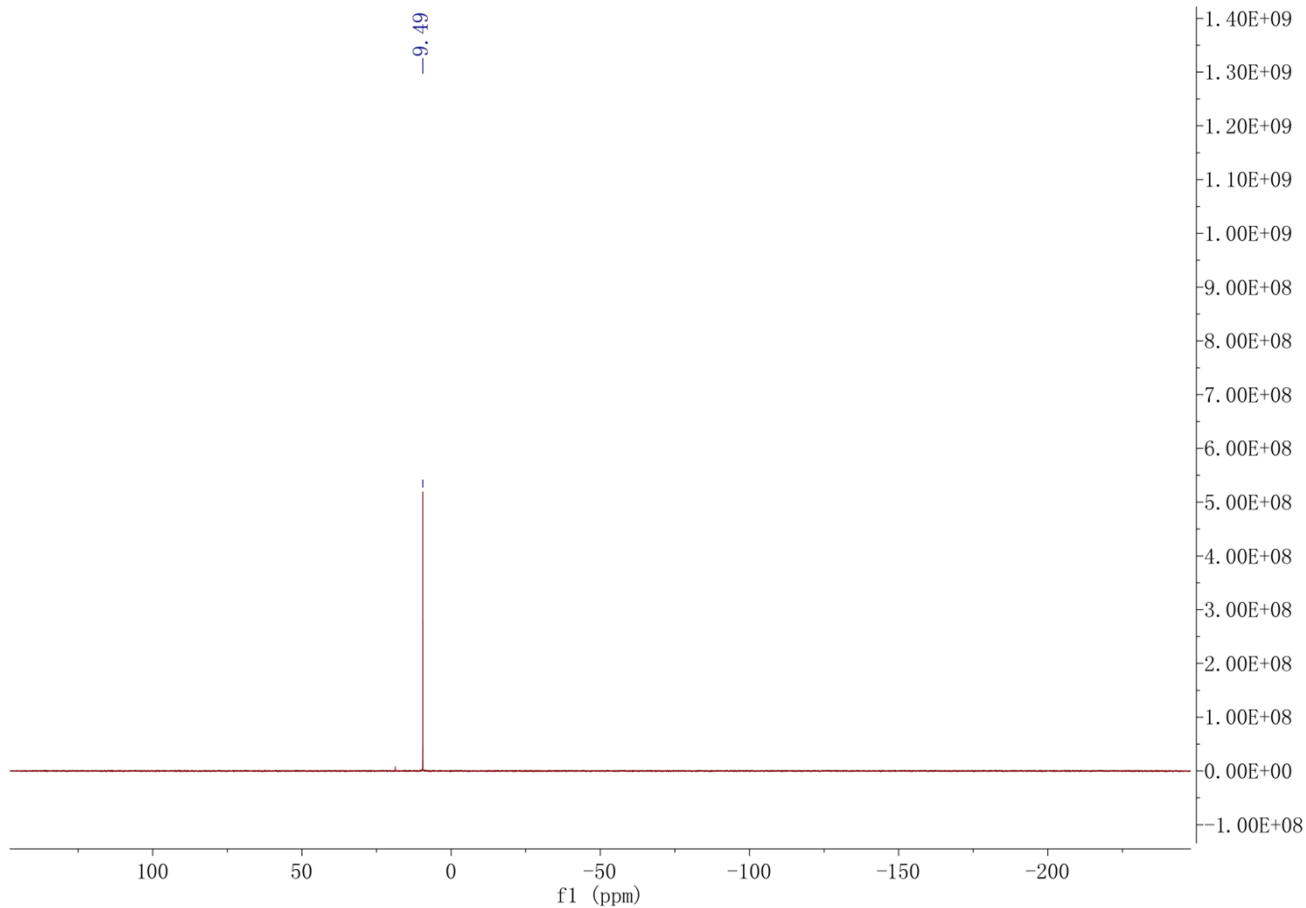
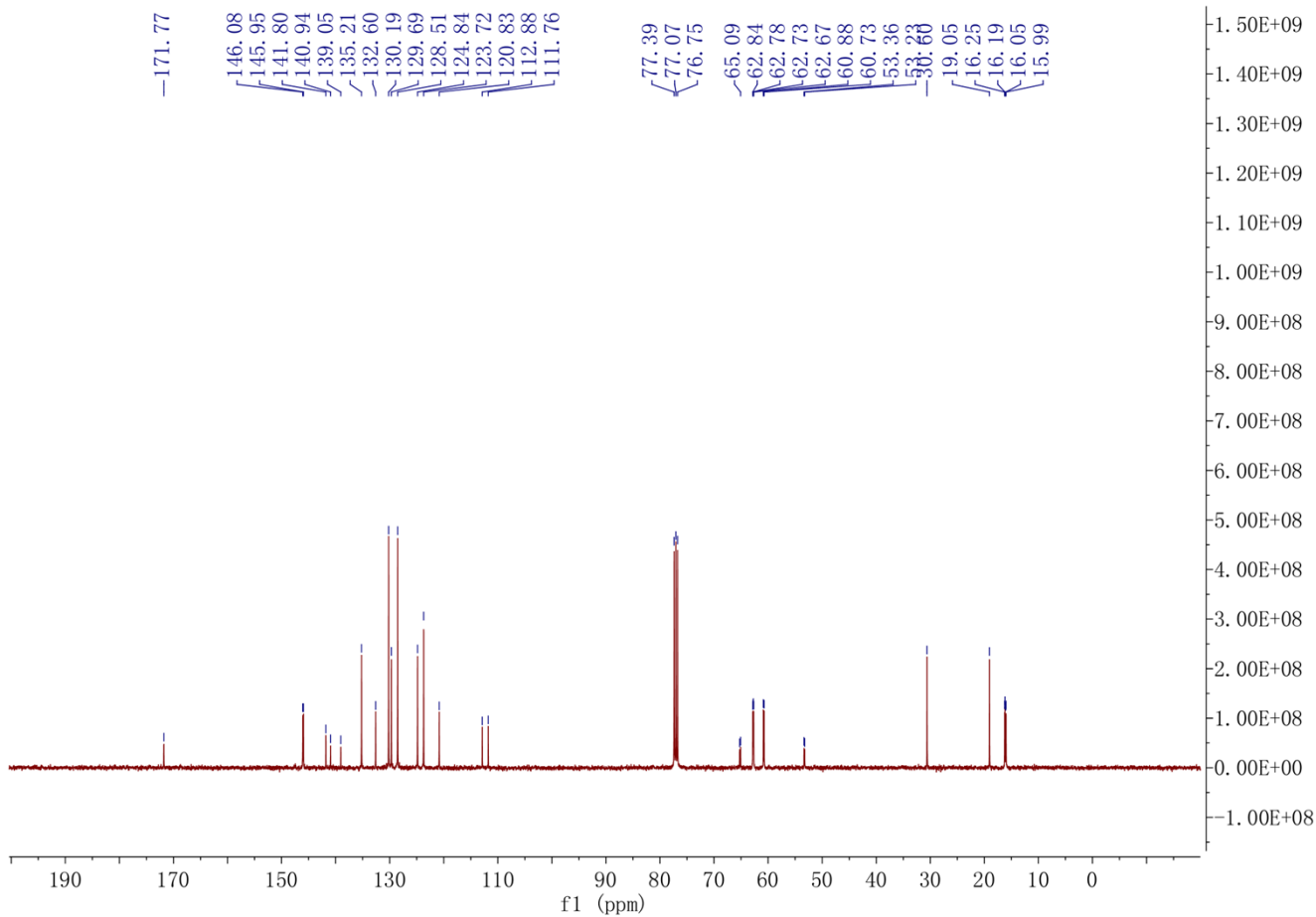


Diethyl(5,5-dicyano-1',7'-dimethyl-2'-oxo-4-phenylspiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (3h):

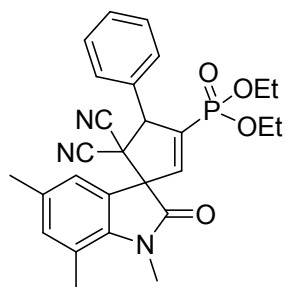


White solid; mp 183 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.56 (d, $J = 19.9$ Hz, 2H), 7.49 (d, $J = 16.5$ Hz, 4H), 7.23 (d, $J = 7.6$ Hz, 1H), 7.12 (t, $J = 7.6$ Hz, 1H), 6.55 (d, $J = 11.0$ Hz, 1H), 5.50 (s, 1H), 4.02-4.17 (m, 2H), 3.82-3.99 (m, 2H), 3.60 (s, 3H), 2.64 (s, 3H), 1.30 (t, $J = 7.0$ Hz, 3H), 1.12 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 171.77, 146.01 (d, $J = 12.5$ Hz), 141.80, 140.00 (d, $J = 190.7$ Hz), 135.21, 132.60, 130.19, 129.69, 128.51, 124.84, 123.72, 120.83, 112.88, 111.76, 65.18 (d, $J = 18.8$ Hz), 62.81 (d, $J = 6.1$ Hz), 62.70 (d, $J = 6.2$ Hz), 60.81 (d, $J = 15.0$ Hz), 53.30 (d, $J = 12.8$ Hz), 30.60, 19.05, 16.22 (d, $J = 6.3$ Hz), 16.02 (d, $J = 6.4$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 9.49. HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{26}\text{H}_{26}\text{N}_3\text{O}_4\text{P}$: 498.1559, found: 498.1555.

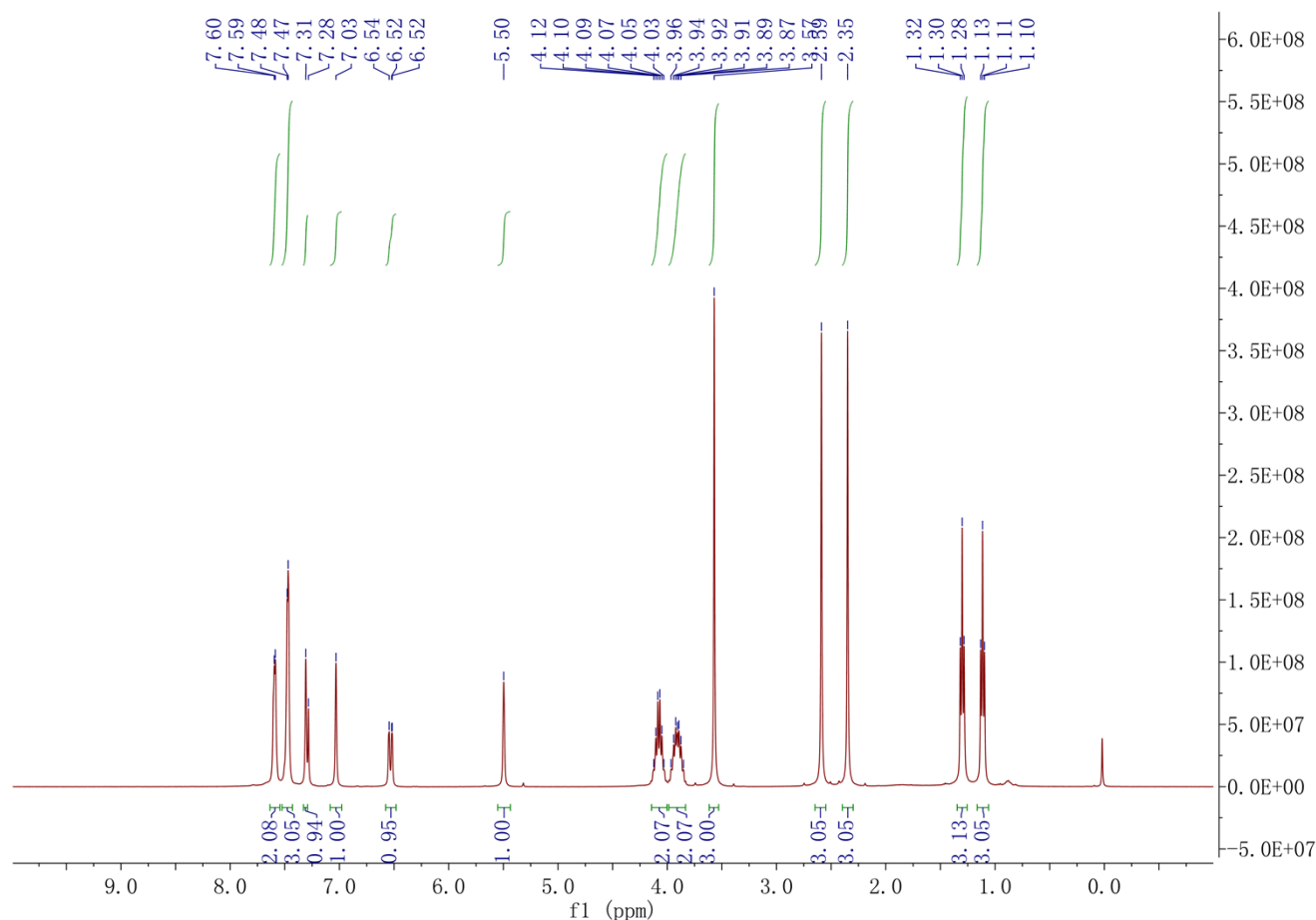


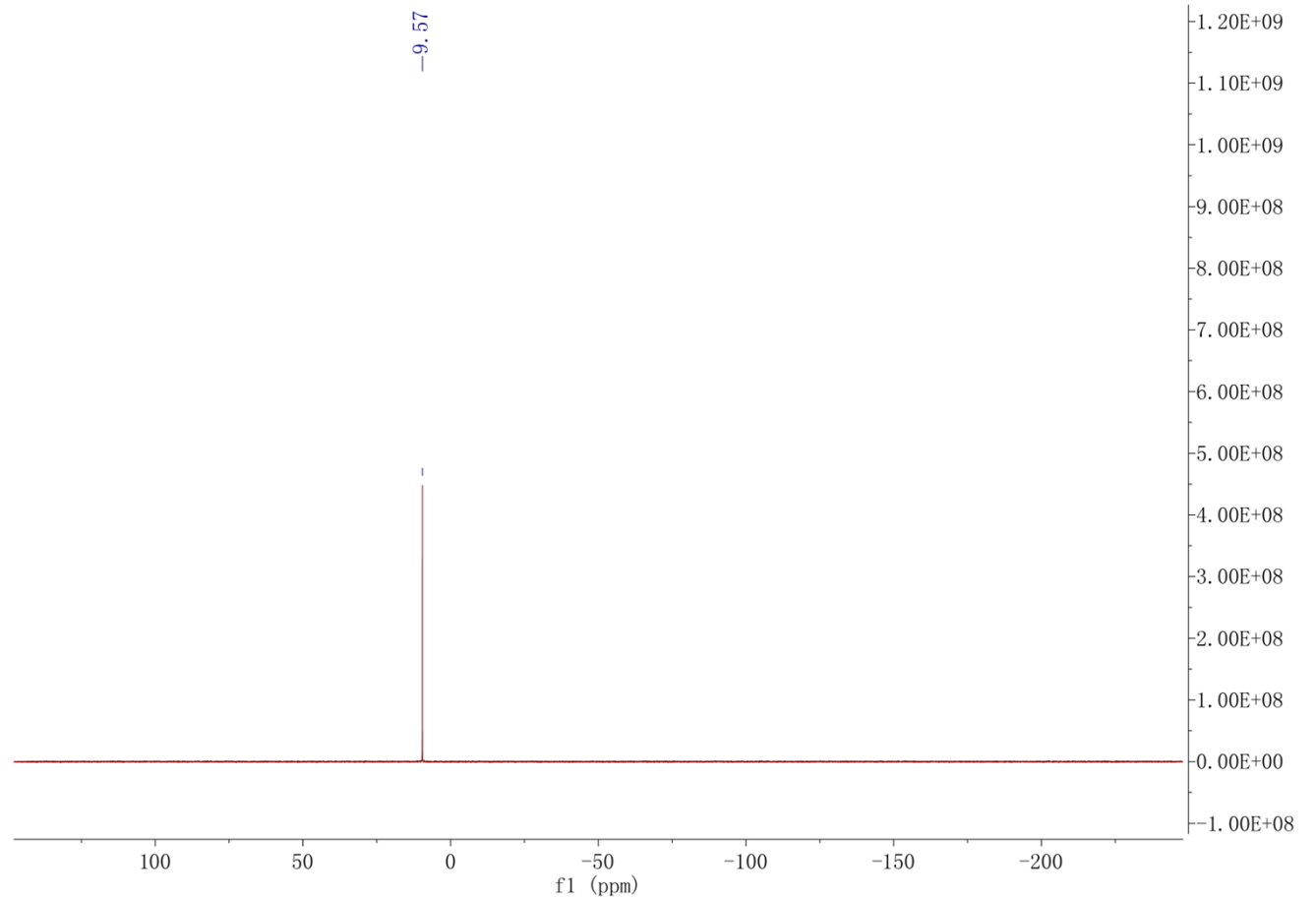
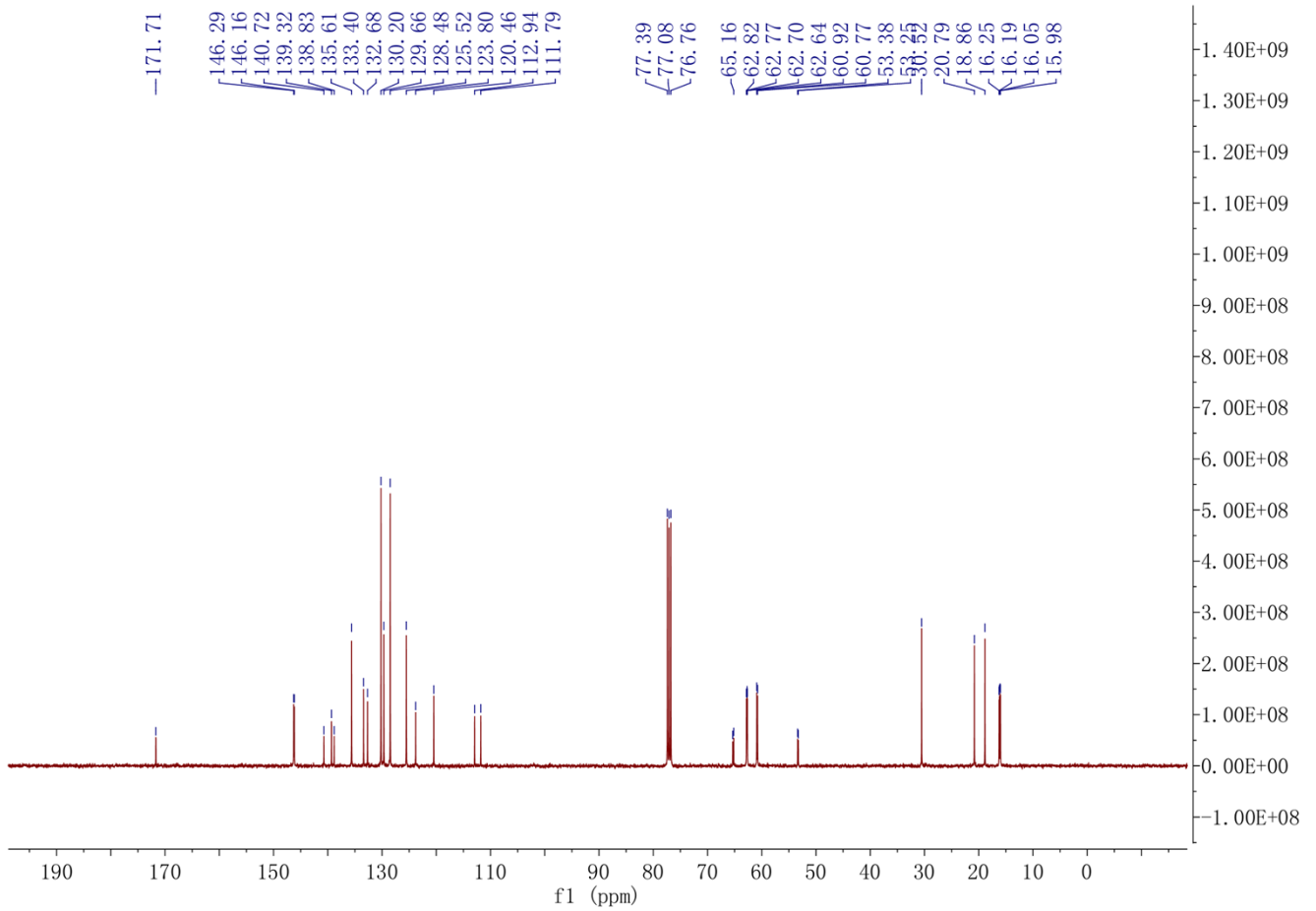


Diethyl(5,5-dicyano-1',5',7'-trimethyl-2'-oxo-4-phenylspiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (3i):



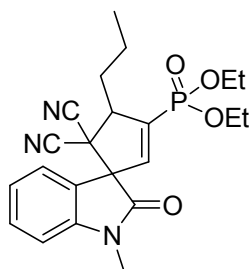
White solid; mp 183 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.59 (d, $J = 3.8$ Hz, 2H), 7.47 (d, $J = 3.3$ Hz, 3H), 7.31 (s, 1H), 7.03 (s, 1H), 6.40-6.62 (m, 1H), 5.50 (s, 1H), 4.01-4.17 (m, 2H), 3.81-4.00 (m, 2H), 3.57 (s, 3H), 2.59 (s, 3H), 2.35 (s, 3H), 1.30 (t, $J = 7.0$ Hz, 3H), 1.11 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 171.71, 146.23 (d, $J = 12.6$ Hz), 139.77 (d, $J = 190.7$ Hz), 139.32, 135.61, 133.40, 132.68, 130.20, 129.66, 128.48, 125.52, 123.80, 120.46, 112.94, 111.79, 65.26 (d, $J = 19.2$ Hz), 62.79 (d, $J = 5.9$ Hz), 62.67 (d, $J = 6.3$ Hz), 60.84 (d, $J = 15.0$ Hz), 53.31 (d, $J = 13.0$ Hz), 30.52, 20.79, 18.86, 16.22 (d, $J = 6.4$ Hz), 16.02 (d, $J = 6.5$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 9.57. HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{27}\text{H}_{28}\text{N}_3\text{O}_4\text{P}$: 512.1715, found: 512.1707.





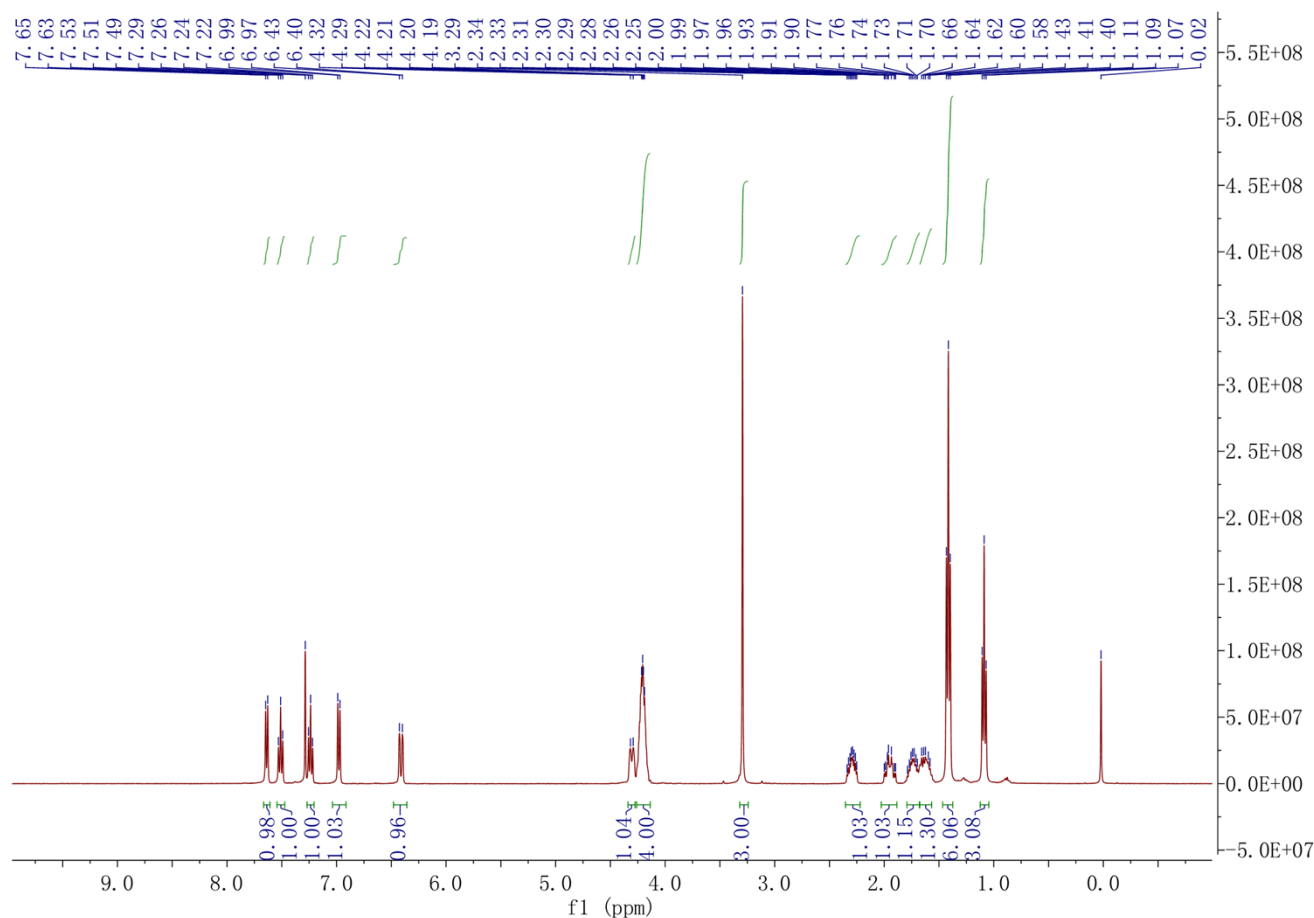
Diethyl(5,5-dicyano-1'-methyl-2'-oxo-4-propylspiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate

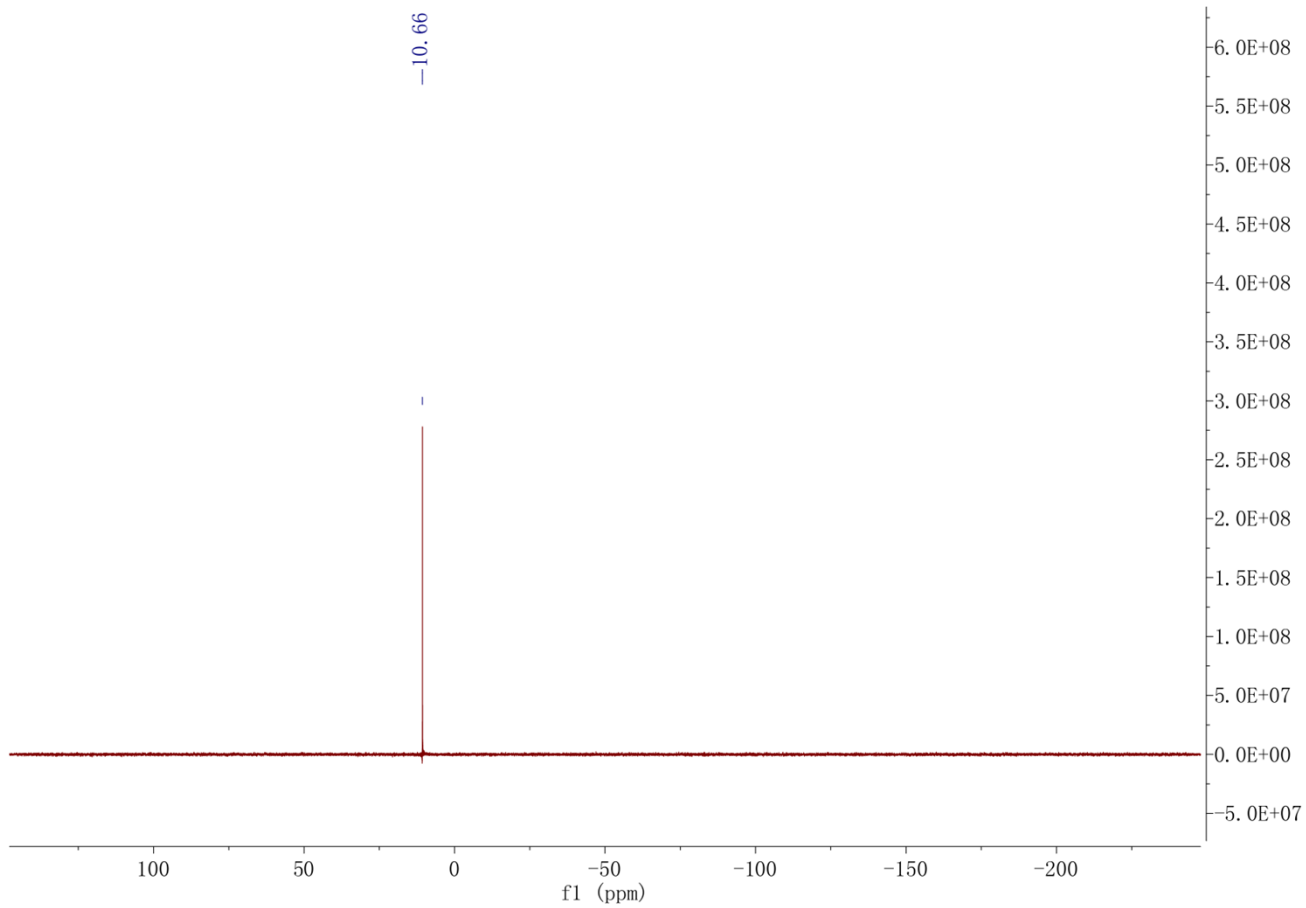
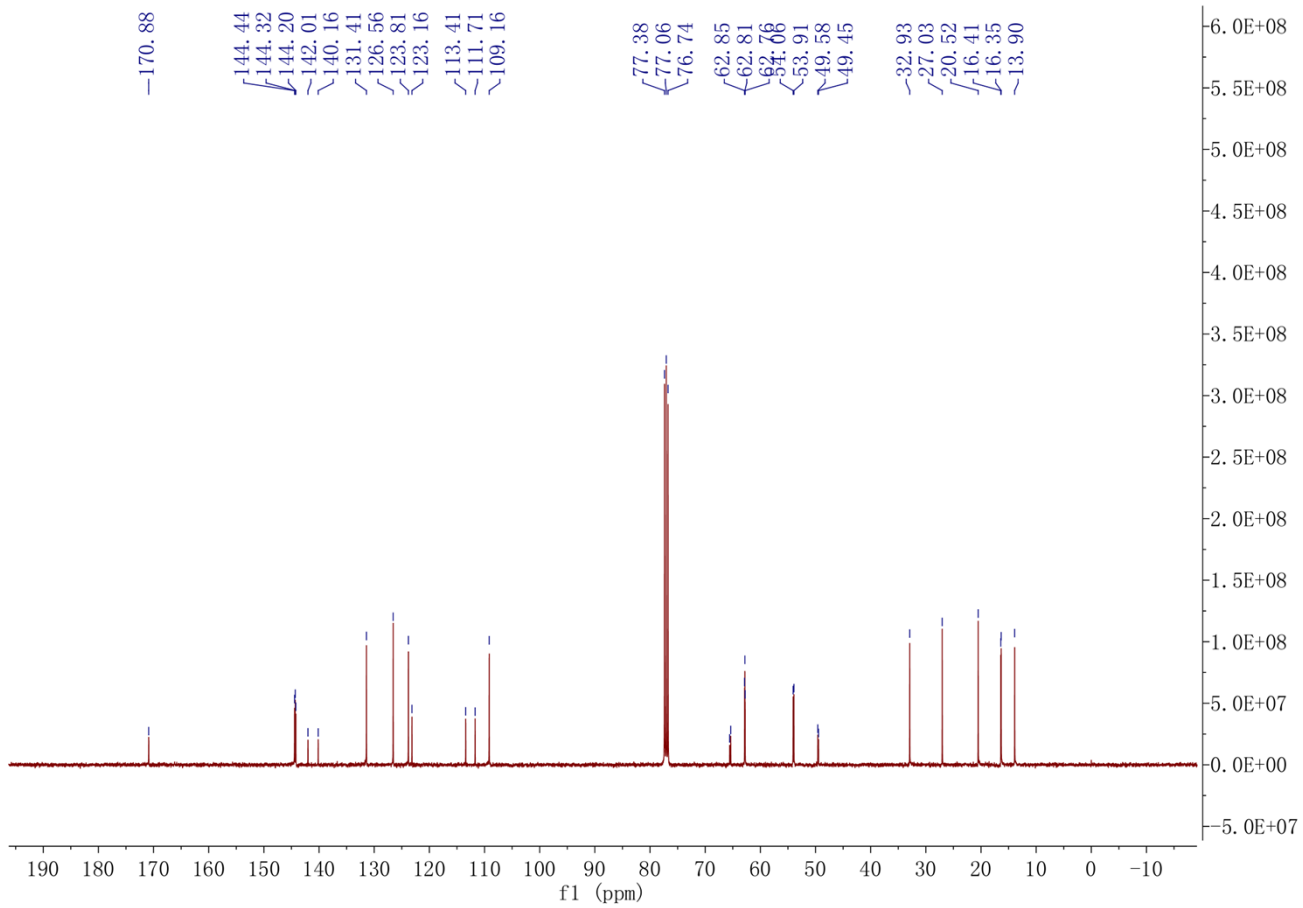
(3j):



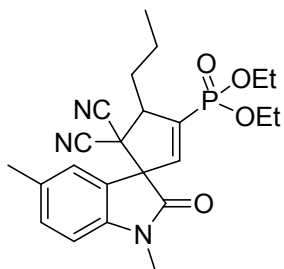
White solid; mp 150 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.64 (d, $J = 7.6$ Hz, 1H), 7.51 (t, $J = 7.8$ Hz, 1H), 7.24 (t, $J = 7.6$ Hz, 1H), 6.98 (d, $J = 7.8$ Hz, 1H), 6.41 (d, $J = 11.1$ Hz, 1H), 4.30 (d, $J = 10.1$ Hz, 1H), 4.20 (dd, $J = 7.0, 4.1$ Hz, 4H), 3.29 (s, 3H), 2.29 (ddd, $J = 20.2, 10.6, 5.1$ Hz, 1H), 1.87-2.03 (m, 1H), 1.69-1.80 (m, 1H),

1.57-1.69 (m, 1H), 1.41 (t, $J = 7.0$ Hz, 6H), 1.09 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 170.88, 144.44, 144.26 (d, $J = 11.1$ Hz), 141.08 (d, $J = 186.4$ Hz), 131.41, 126.56, 123.81, 123.16, 113.41, 111.71, 109.16, 65.47 (d, $J = 19.2$ Hz), 62.83 (d, $J = 4.3$ Hz), 62.78 (d, $J = 4.8$ Hz), 53.99 (d, $J = 15.2$ Hz), 49.52 (d, $J = 13.3$ Hz), 32.93, 27.03, 20.52, 16.38 (d, $J = 6.2$ Hz), 13.90; ^{31}P NMR (162 MHz, CDCl_3): δ 10.66. HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{22}\text{H}_{26}\text{N}_3\text{O}_4\text{P}$: 450.1559, found: 450.1554.

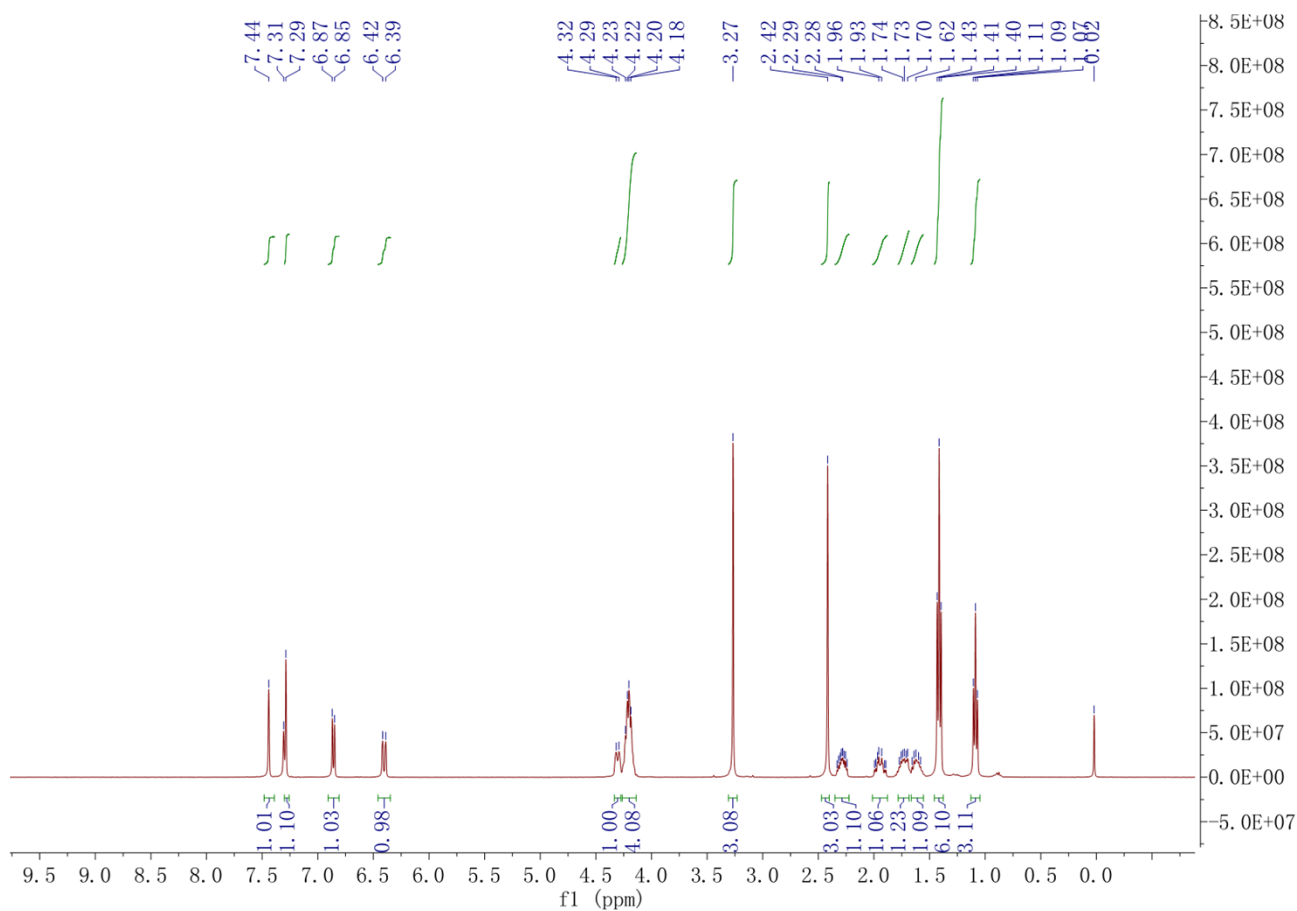


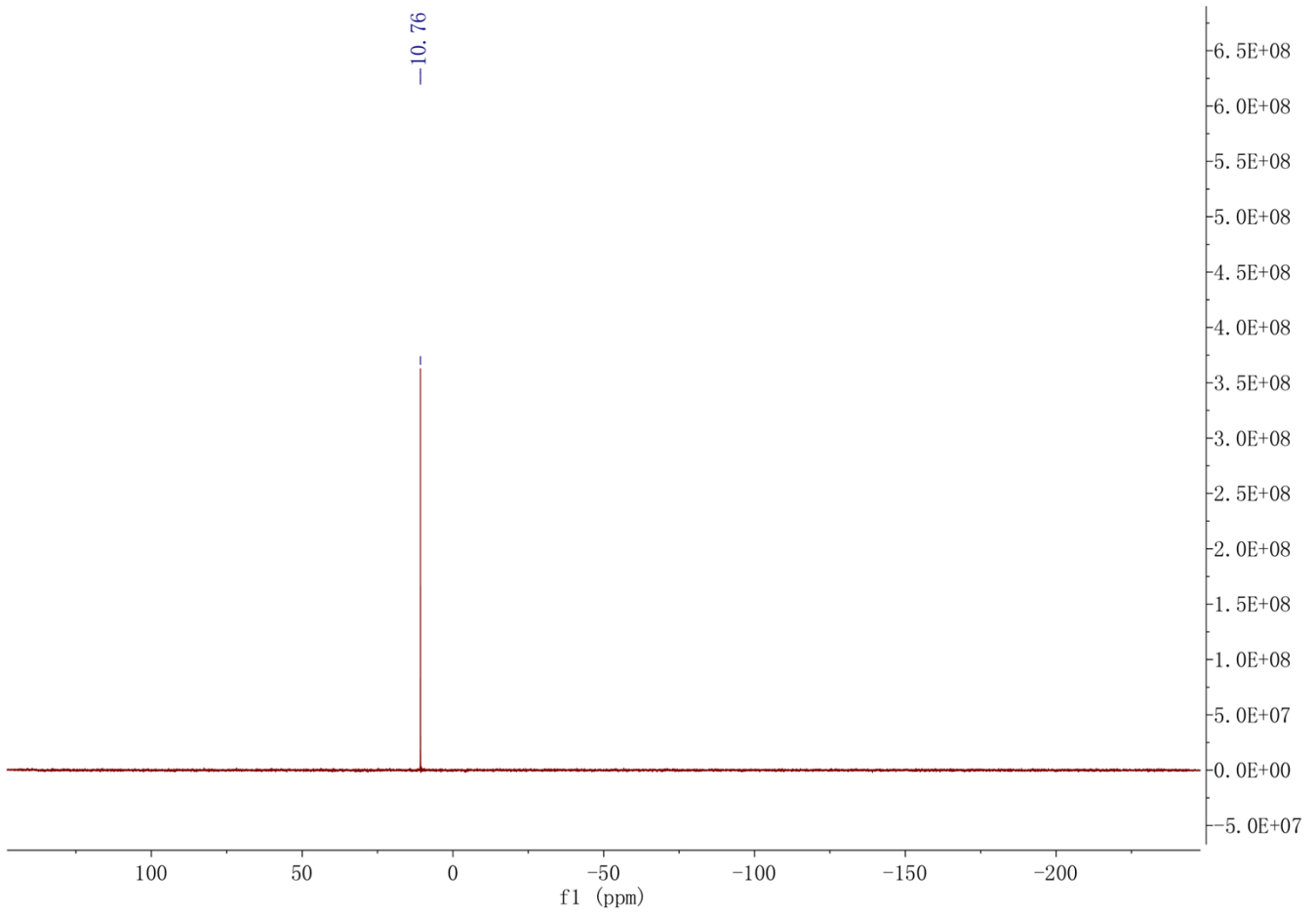
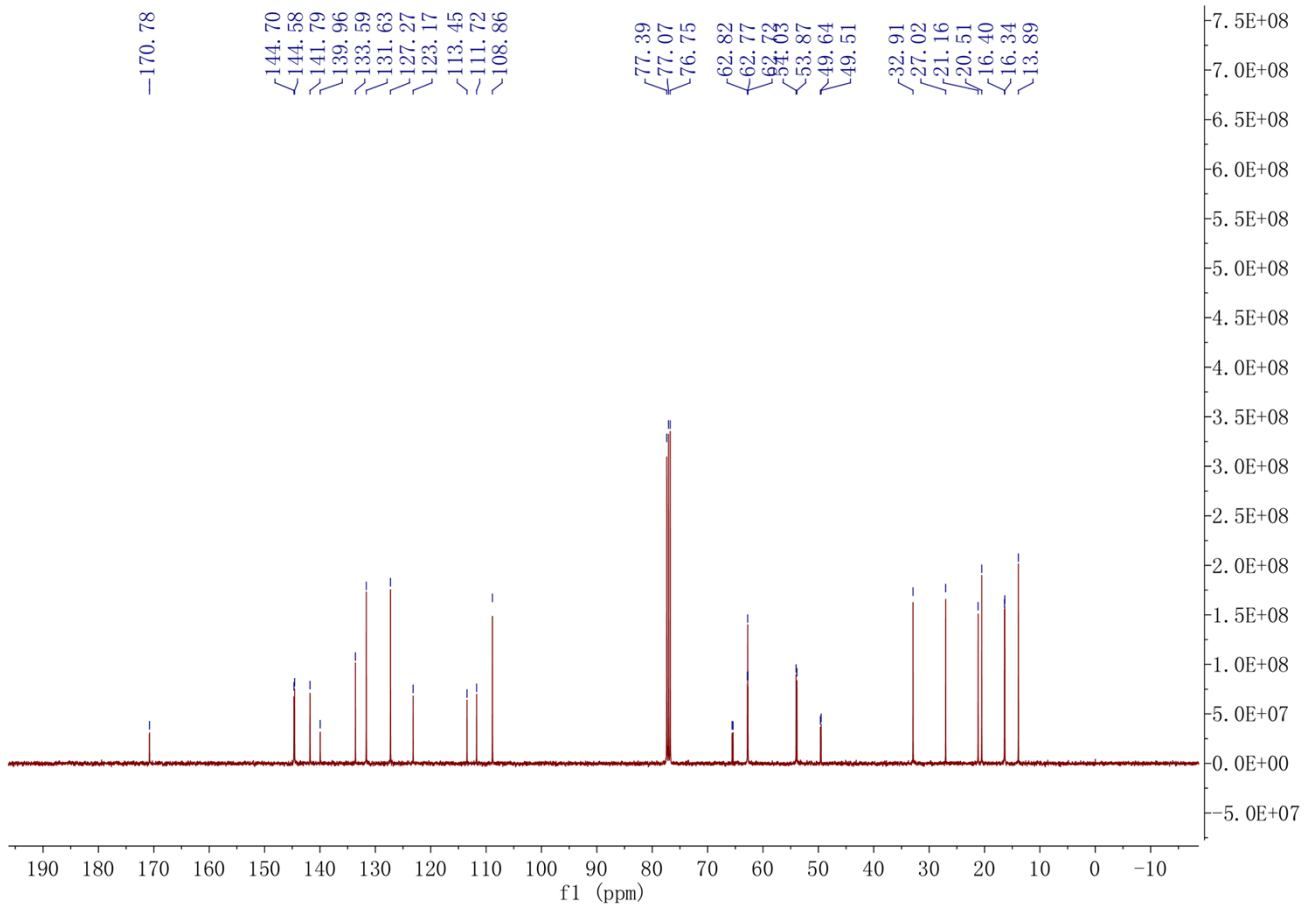


Diethyl(5,5-dicyano-1',5'-dimethyl-2'-oxo-4-propylspiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (3k):

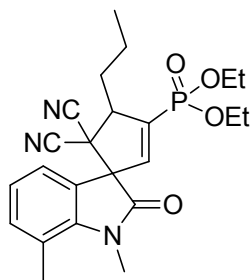


White solid; mp 153 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.44 (s, 1H), 7.29 (s, 1H), 6.86 (d, $J = 8.0$ Hz, 1H), 6.40 (d, $J = 11.1$ Hz, 1H), 4.30 (d, $J = 9.6$ Hz, 1H), 4.21 (dd, $J = 12.5, 6.8$ Hz, 4H), 3.27 (s, 3H), 2.42 (s, 3H), 2.29 (ddd, $J = 15.4, 10.9, 5.3$ Hz, 1H), 1.88-2.01 (m, 1H), 1.68-1.79 (m, 1H), 1.56-1.66 (m, 1H), 1.41 (t, $J = 7.0$ Hz, 6H), 1.09 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 170.78, 144.64 (d, $J = 12.7$ Hz), 141.79, 139.96, 133.59, 131.63, 127.27, 123.17, 113.45, 111.72, 108.86, 65.49 (d, $J = 19.2$ Hz), 62.77 (t, $J = 4.8$ Hz), 53.95 (d, $J = 15.5$ Hz), 49.57 (d, $J = 13.0$ Hz), 32.91, 27.02, 21.16, 20.51, 16.37 (d, $J = 6.3$ Hz), 13.89; ^{31}P NMR (162 MHz, CDCl_3): δ 10.76. HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{23}\text{H}_{28}\text{N}_3\text{O}_4\text{P}$: 464.1715, found: 464.1710.



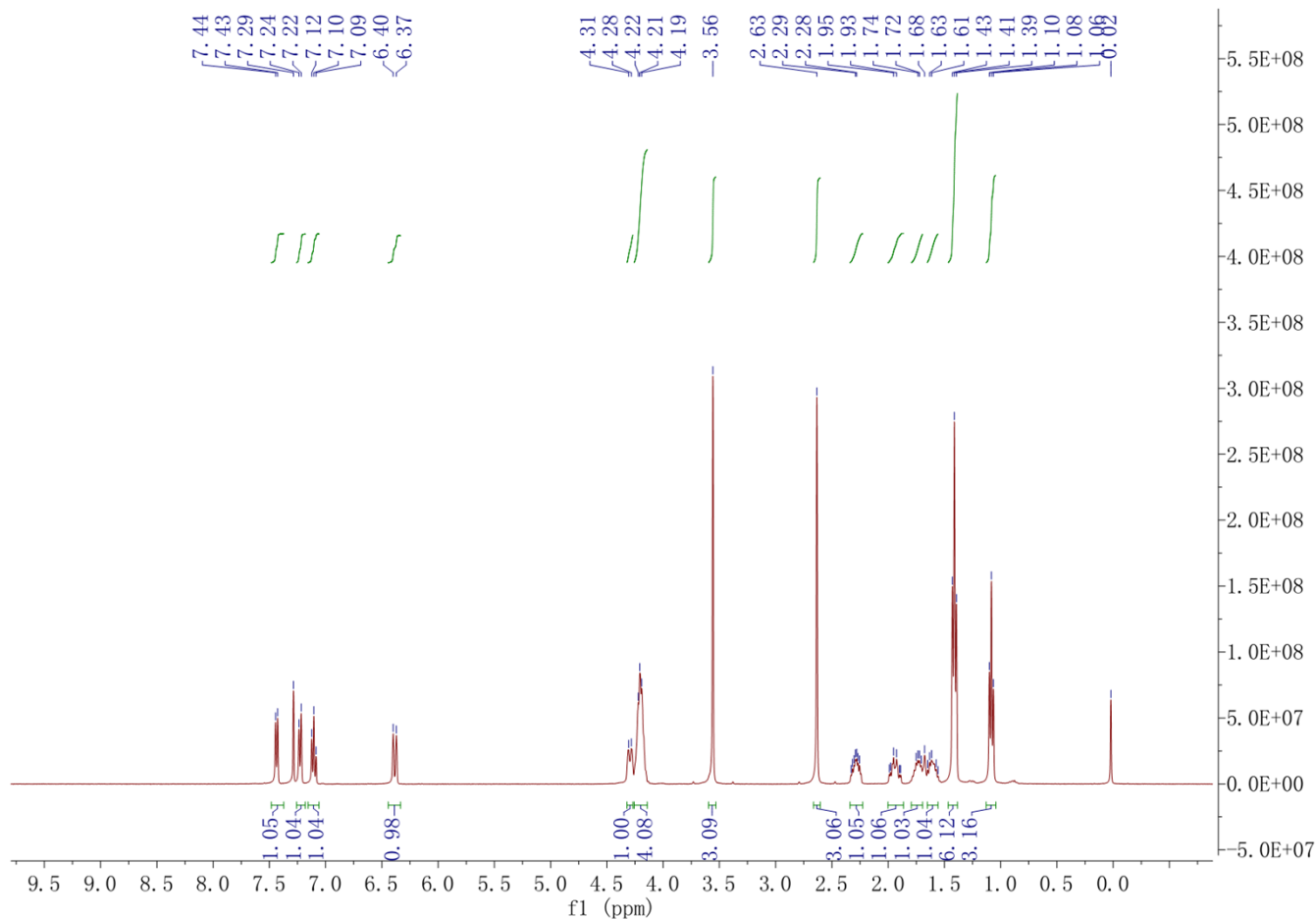


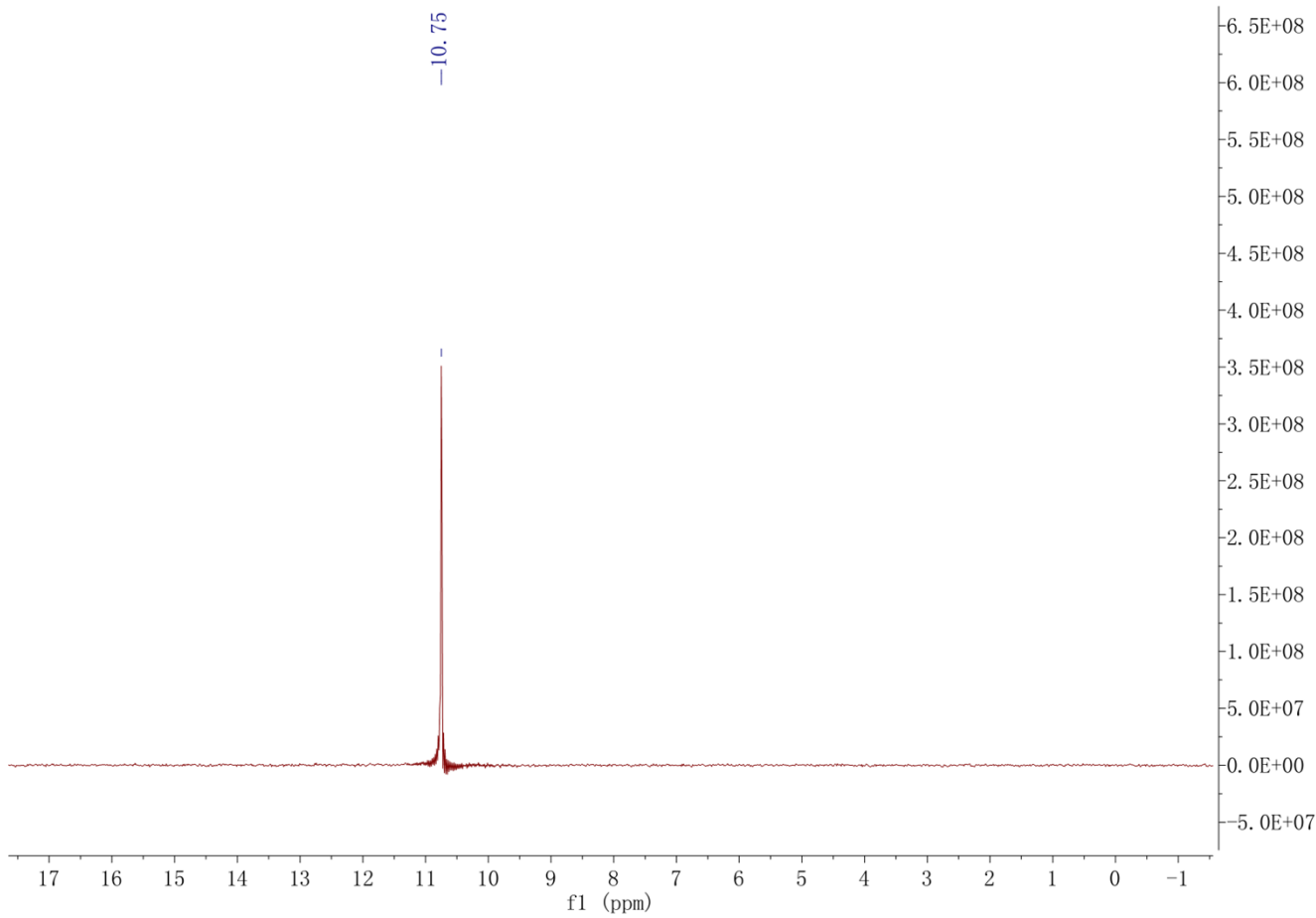
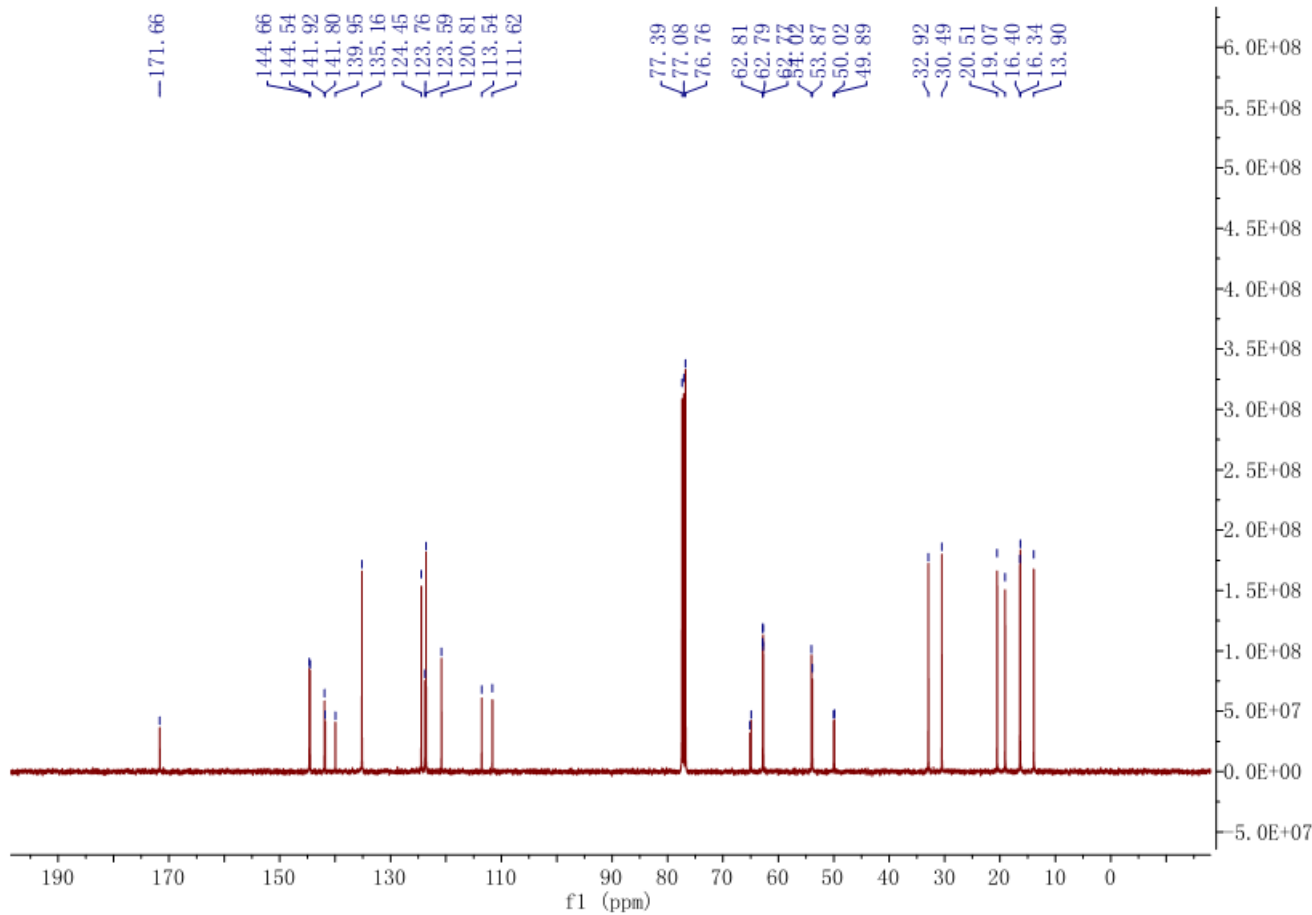
Diethyl(5,5-dicyano-1',7'-dimethyl-2'-oxo-4-propylspiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (31):



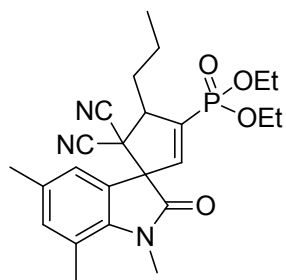
White solid; mp 151 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.44 (d, *J* = 7.5 Hz, 1H), 7.23 (d, *J* = 7.7 Hz, 1H), 7.10 (t, *J* = 7.7 Hz, 1H), 6.38 (d, *J* = 11.1 Hz, 1H), 4.29 (d, *J* = 9.6 Hz, 1H), 4.15-4.26 (m, 4H), 3.56 (s, 3H), 2.63 (s, 3H), 2.22-2.35 (m, 1H), 1.94 (td, *J* = 14.8, 4.3 Hz, 1H), 1.73 (dd, *J* = 11.8, 6.3 Hz, 1H), 1.59 (dd, *J* = 22.9,

6.9 Hz, 1H); ¹³C NMR (101 MHz, CDCl₃): δ 171.66, 144.60 (d, *J* = 12.6 Hz), 141.92, 140.87 (d, *J* = 186.6 Hz), 135.16, 124.45, 123.76, 123.59, 120.81, 113.54, 111.62, 65.02 (d, *J* = 19.5 Hz), 62.80 (d, *J* = 2.4 Hz), 62.75 (d, *J* = 3.7 Hz), 53.95 (d, *J* = 15.7 Hz), 49.96 (d, *J* = 12.7 Hz), 32.92, 30.49, 20.51, 19.07, 16.37 (d, *J* = 6.2 Hz), 13.90; ³¹P NMR (162 MHz, CDCl₃): δ 10.75; HRMS calculated [M+Na]⁺ for C₂₃H₂₈N₃O₄P: 464.1715, found: 464.1715.

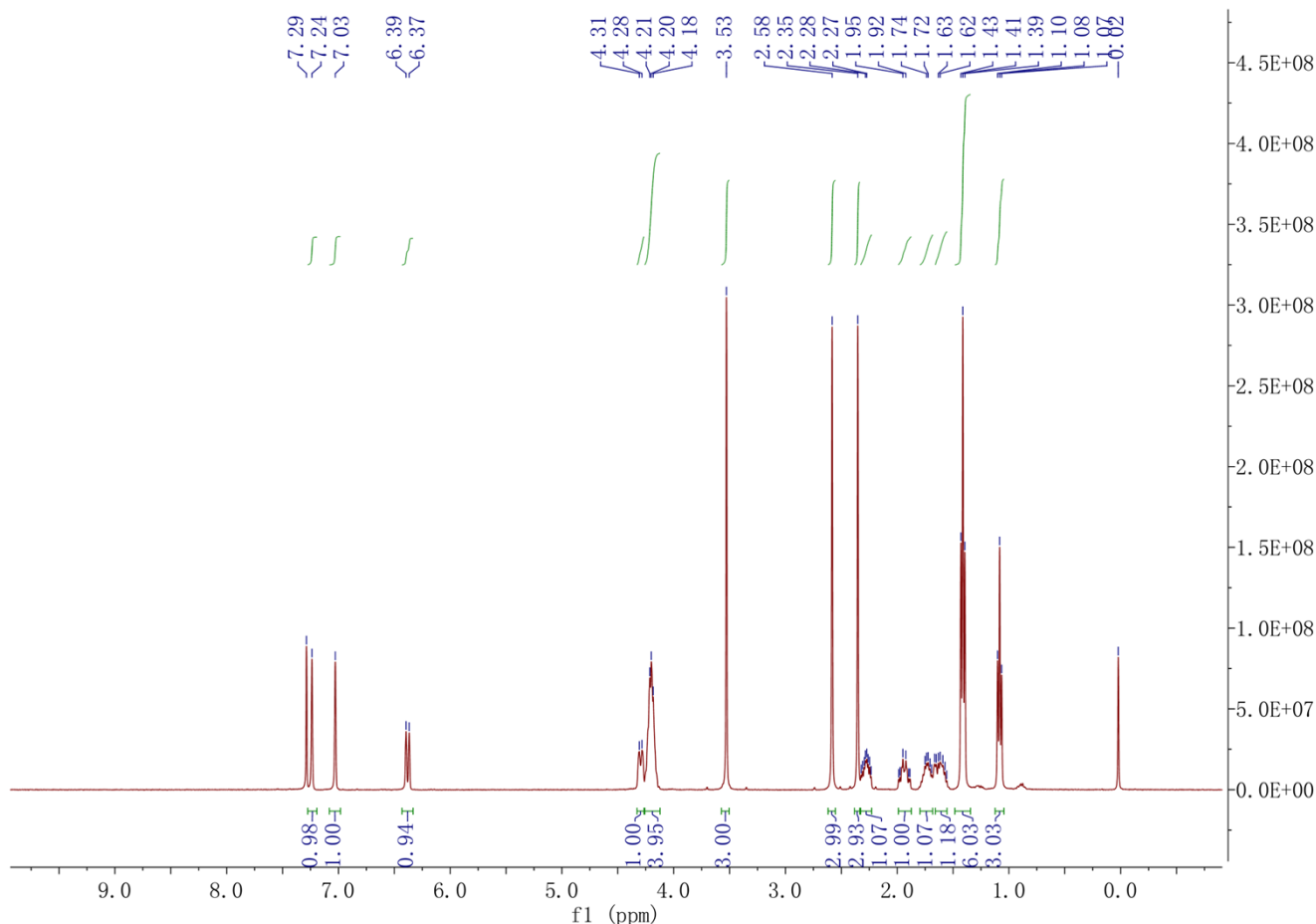


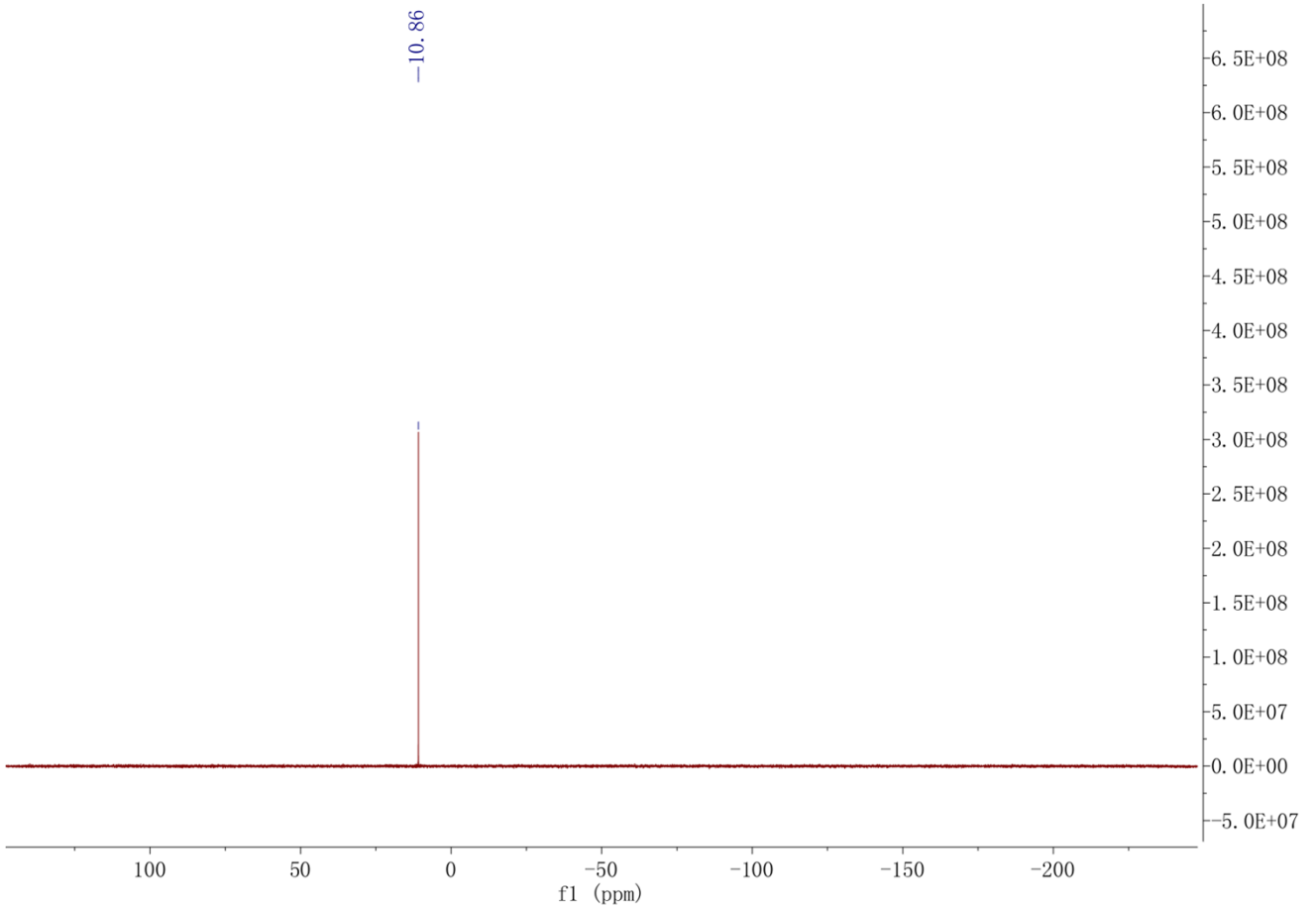
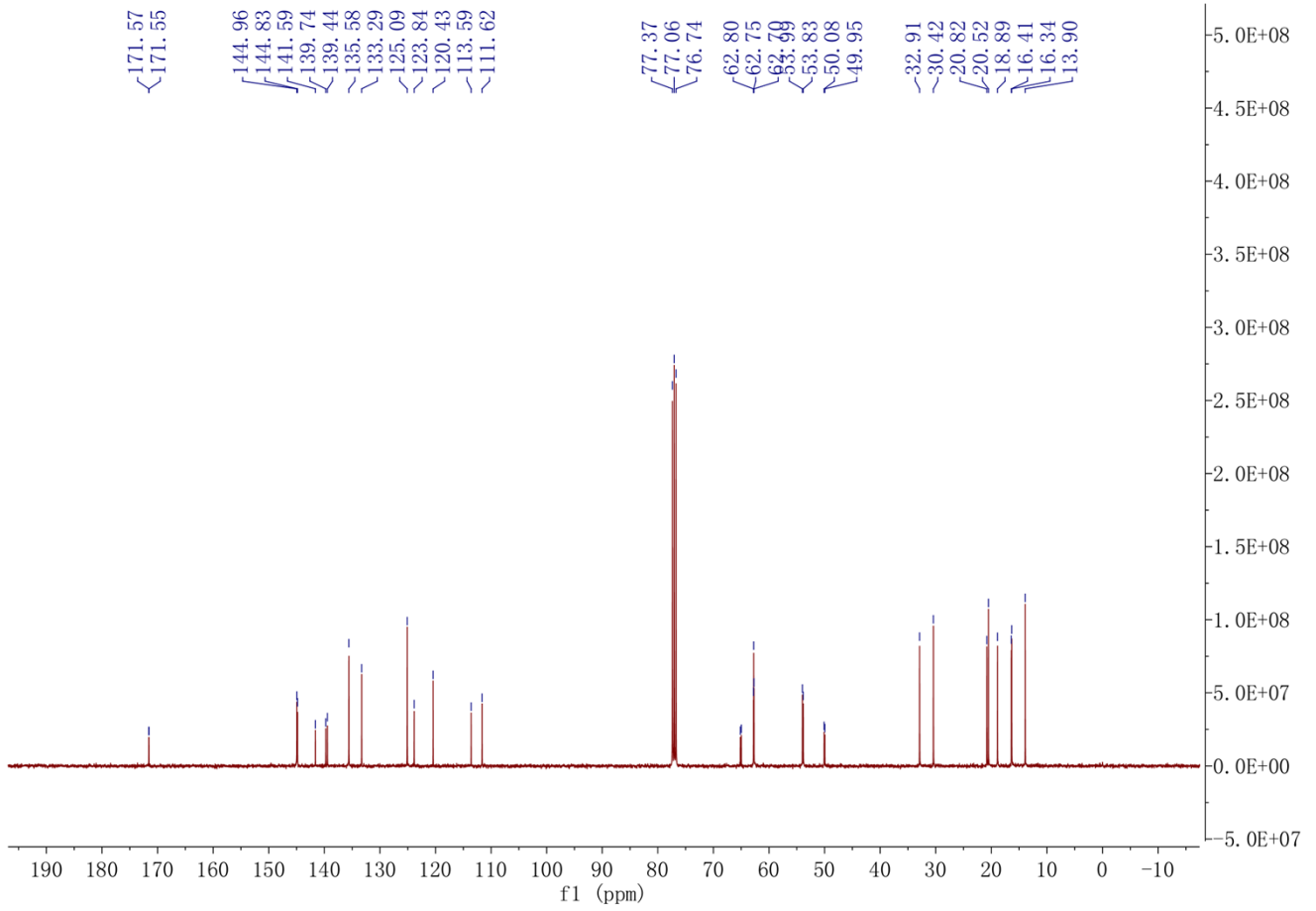


Diethyl(5,5-dicyano-1',5',7'-trimethyl-2'-oxo-4-propylspiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (3m):

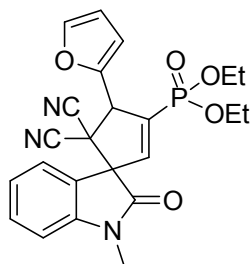


White solid; mp 151 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.24 (s, 1H), 7.03 (s, 1H), 6.38 (d, $J = 11.2$ Hz, 1H), 4.30 (d, $J = 9.7$ Hz, 1H), 4.13-4.26 (m, 4H), 3.53 (s, 3H), 2.58 (s, 3H), 2.35 (s, 3H), 2.28 (ddd, $J = 15.3, 10.8, 5.1$ Hz, 1H), 1.94 (td, $J = 14.9, 4.6$ Hz, 1H), 1.69-1.80 (m, 1H), 1.60 (td, $J = 13.9, 7.0$ Hz, 1H), 1.41 (t, $J = 7.0$ Hz, 6H), 1.08 (t, $J = 7.1$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 171.56 (d, $J = 2.2$ Hz), 144.89 (d, $J = 12.9$ Hz), 140.67 (d, $J = 186.6$ Hz), 139.44, 135.58, 133.29, 125.09, 123.84, 120.43, 113.59, 111.62, 65.07 (d, $J = 19.1$ Hz), 62.75 (t, $J = 4.8$ Hz), 53.91 (d, $J = 15.4$ Hz), 50.01 (d, $J = 13.3$ Hz), 32.91, 30.42, 20.82, 20.52, 18.89, 16.37 (d, $J = 6.3$ Hz), 13.90; ^{31}P NMR (162 MHz, CDCl_3): δ 10.86. HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{24}\text{H}_{30}\text{N}_3\text{O}_4\text{P}$: 478.1872, found: 478.1869.

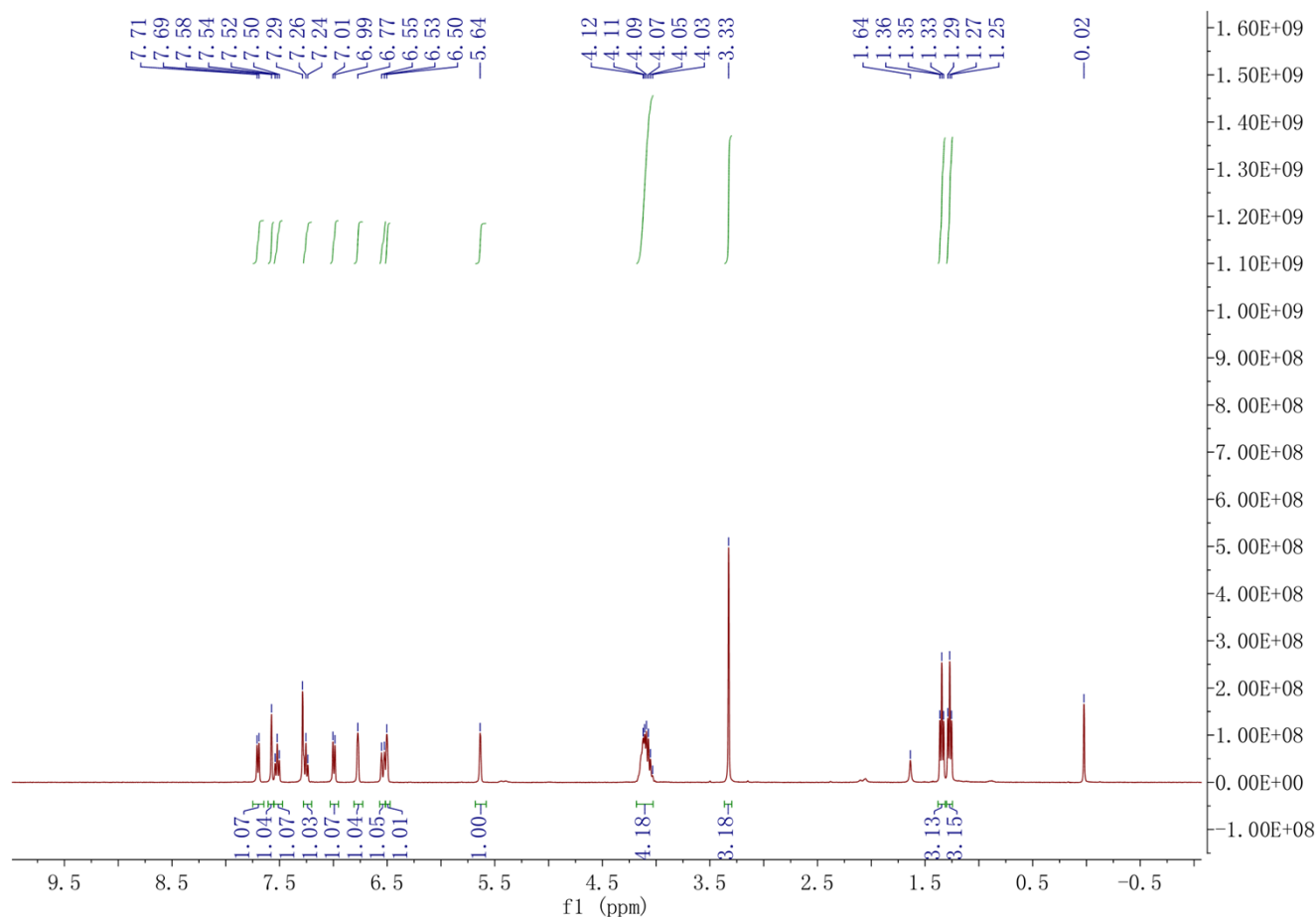


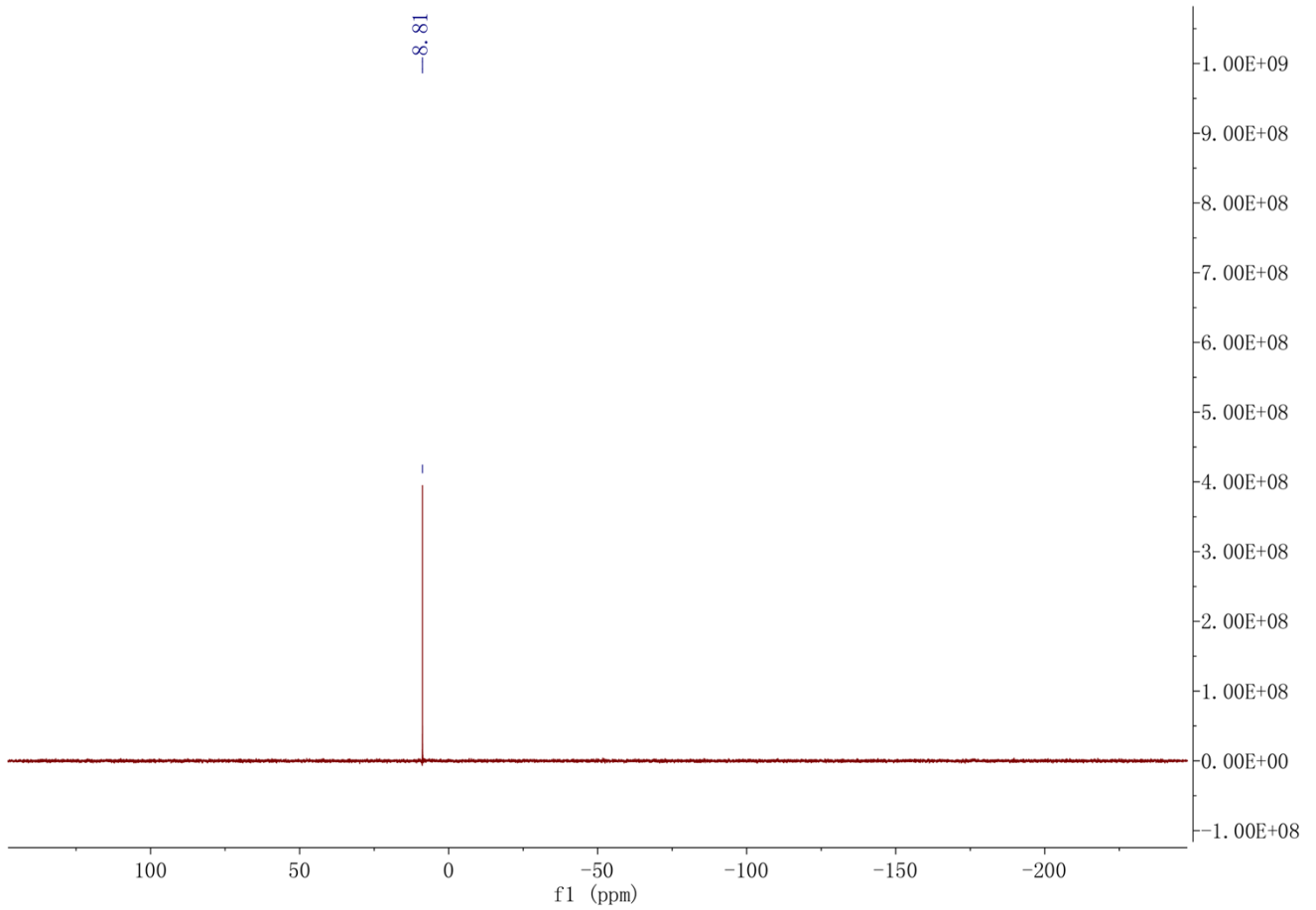
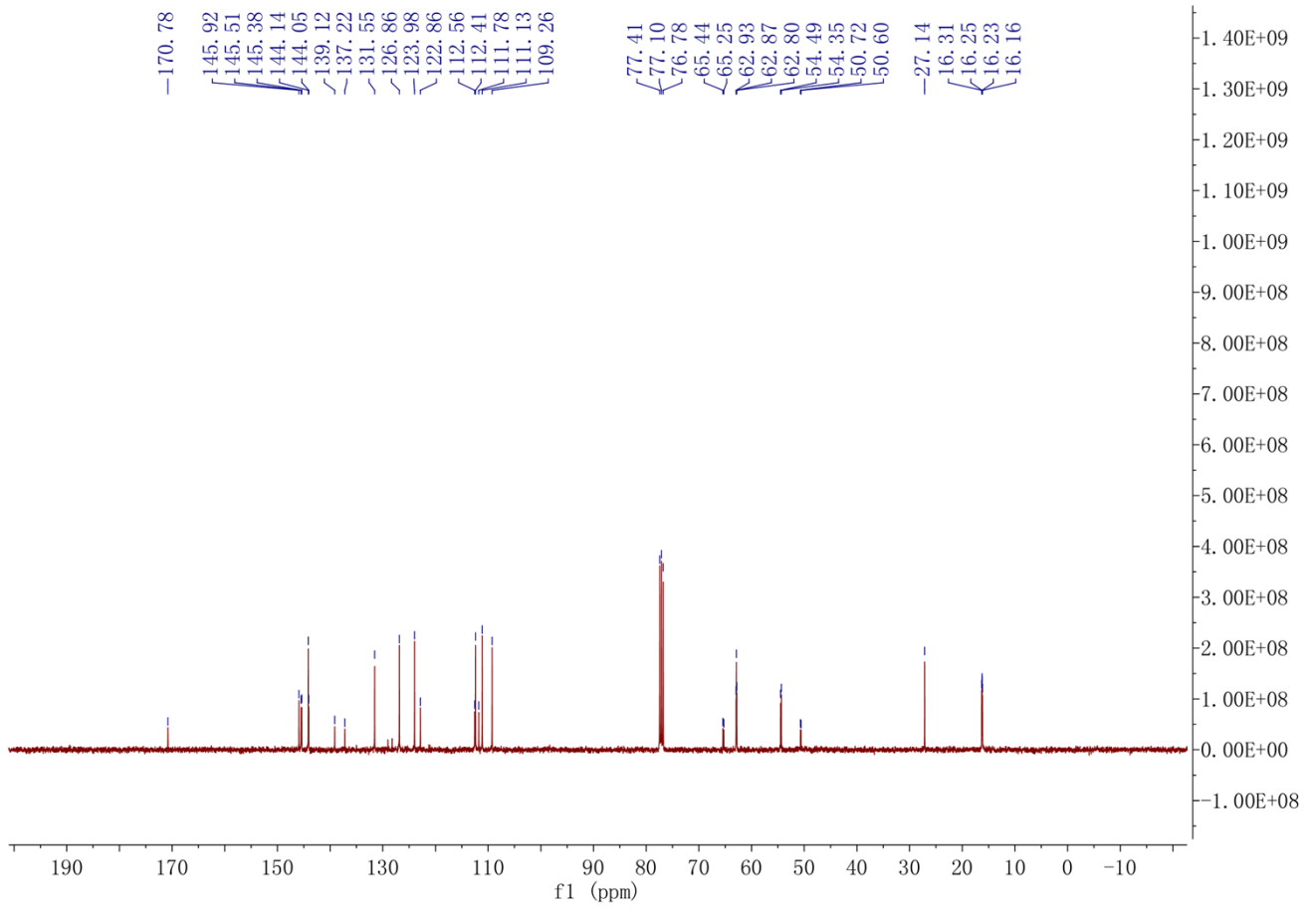


Diethyl(5,5-dicyano-4-(furan-2-yl)-1'-methyl-2'-oxospiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (3n):

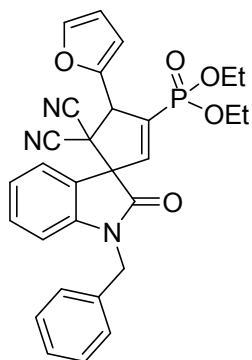


White solid; mp 188 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.70 (d, $J = 7.5$ Hz, 1H), 7.58 (s, 1H), 7.52 (t, $J = 7.7$ Hz, 1H), 7.26 (t, $J = 8.0$ Hz, 1H), 7.00 (d, $J = 7.8$ Hz, 1H), 6.77 (s, 1H), 6.54 (d, $J = 10.8$ Hz, 1H), 6.50 (s, 1H), 5.64 (s, 1H), 4.01-4.20 (m, 4H), 3.33 (s, 3H), 1.35 (t, $J = 6.9$ Hz, 3H), 1.27 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 170.78, 145.92, 145.45 (d, $J = 12.7$ Hz), 144.14, 144.05, 138.17 (d, $J = 191.6$ Hz), 131.55, 126.86, 123.98, 122.86, 112.56, 112.41, 111.78, 111.13, 109.26, 65.34 (d, $J = 18.7$ Hz), 62.87 (t, $J = 6.3$ Hz), 54.42 (d, $J = 14.1$ Hz), 50.66 (d, $J = 12.2$ Hz), 27.14, 16.28 (d, $J = 6.2$ Hz), 16.19 (d, $J = 7.2$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 8.81; HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{23}\text{H}_{22}\text{N}_3\text{O}_5\text{P}$: 474.1195, found: 474.1192.

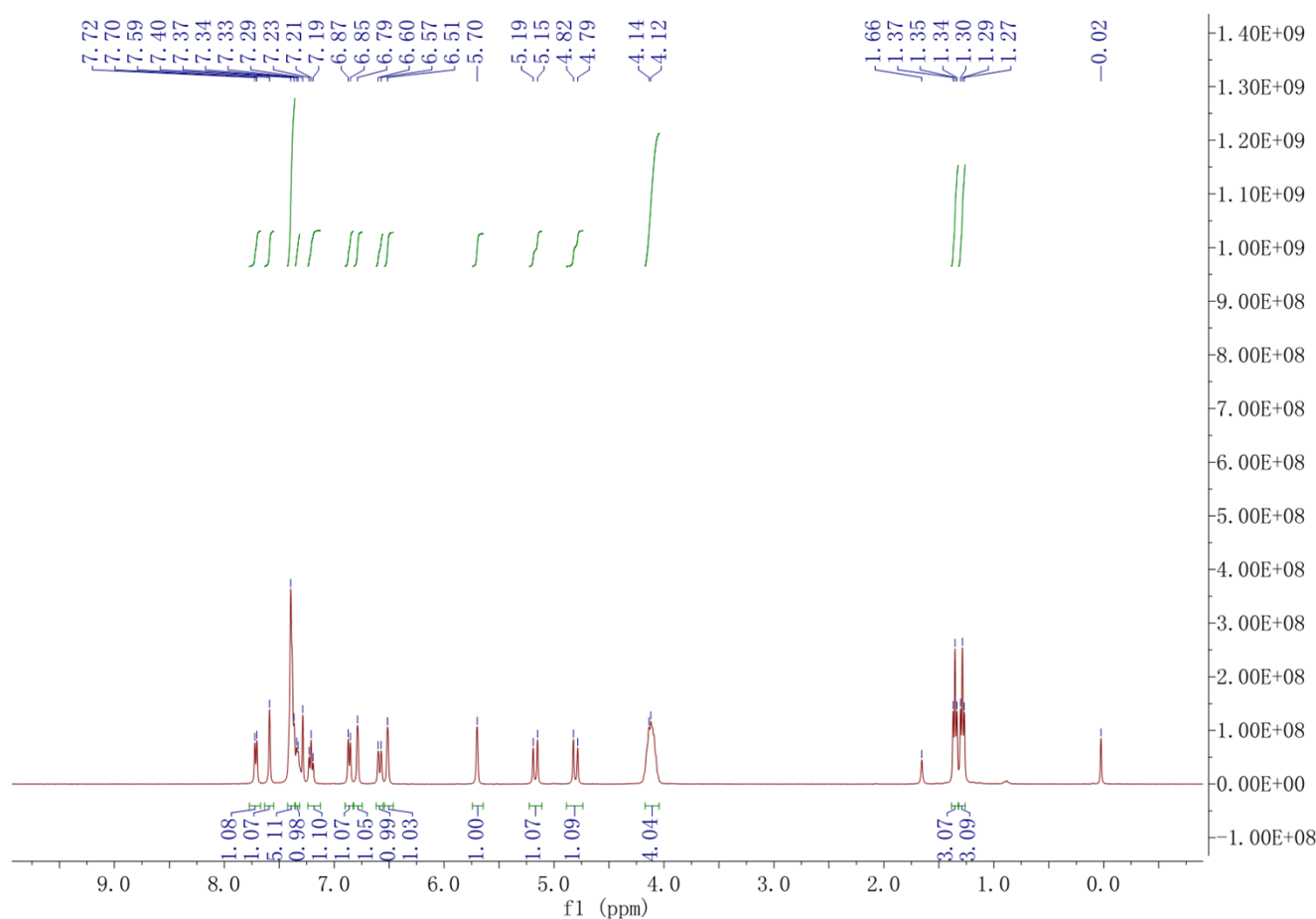


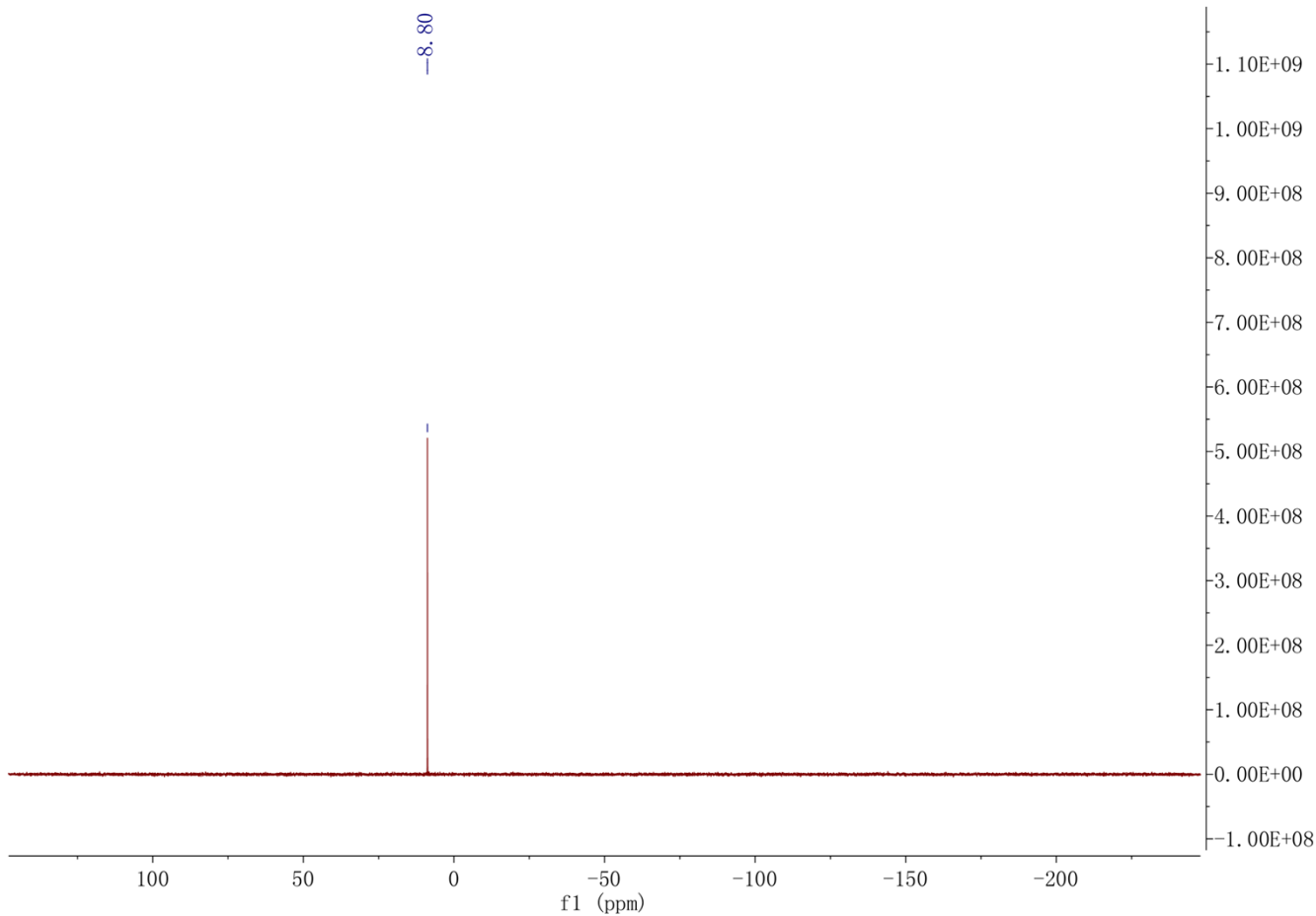
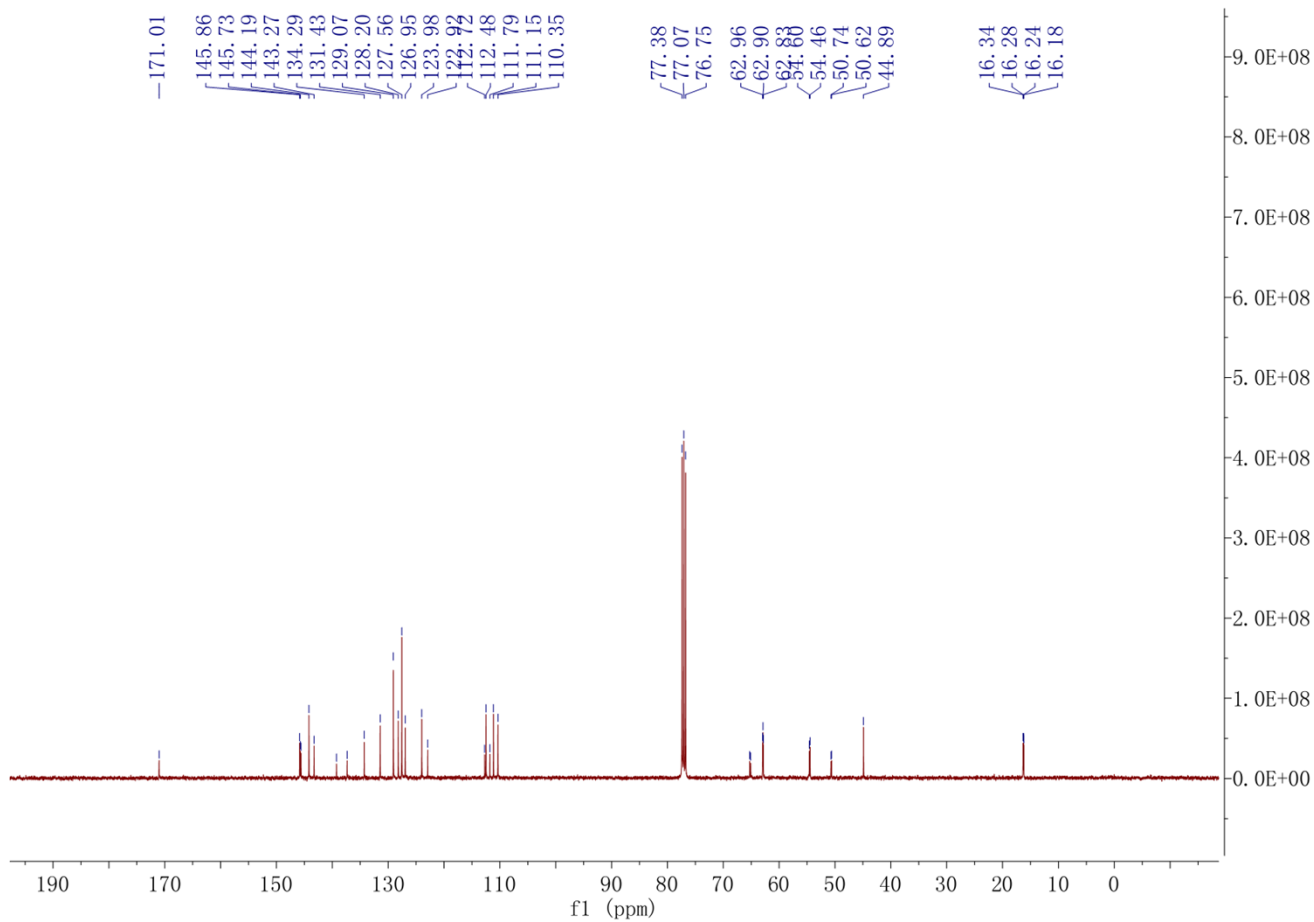


Diethyl(1'-benzyl-5,5-dicyano-4-(furan-2-yl)-2'-oxospiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (3o):

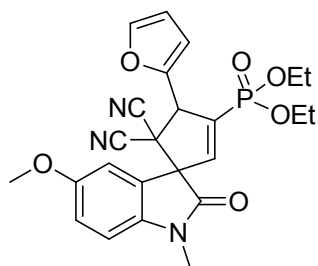


White solid; mp 155 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.71 (d, $J = 7.4$ Hz, 1H), 7.59 (s, 1H), 7.38 (d, $J = 12.0$ Hz, 5H), 7.34 (d, $J = 5.9$ Hz, 1H), 7.21 (t, $J = 7.4$ Hz, 1H), 6.86 (d, $J = 7.8$ Hz, 1H), 6.79 (s, 1H), 6.59 (d, $J = 10.9$ Hz, 1H), 6.51 (s, 1H), 5.70 (s, 1H), 5.17 (d, $J = 15.6$ Hz, 1H), 4.80 (d, $J = 15.5$ Hz, 1H), 4.13 (d, $J = 6.9$ Hz, 4H), 1.35 (t, $J = 6.9$ Hz, 3H), 1.29 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 171.01, 145.86, 145.67 (d, $J = 12.4$ Hz), 144.19, 143.27, 138.30 (d, $J = 191.5$ Hz), 134.29, 131.43, 129.07, 128.20, 127.56, 126.95, 123.98, 122.92, 112.72, 112.48, 111.79, 111.15, 110.35, 65.19 (d, $J = 19.0$ Hz), 62.93 (d, $J = 6.6$ Hz), 62.87 (d, $J = 6.4$ Hz), 54.53 (d, $J = 13.9$ Hz), 50.68 (d, $J = 12.1$ Hz), 44.89, 16.31 (d, $J = 6.6$ Hz), 16.21 (d, $J = 6.6$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 8.80; HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{29}\text{H}_{26}\text{N}_3\text{O}_5\text{P}$: 550.1508, found: 550.1505.

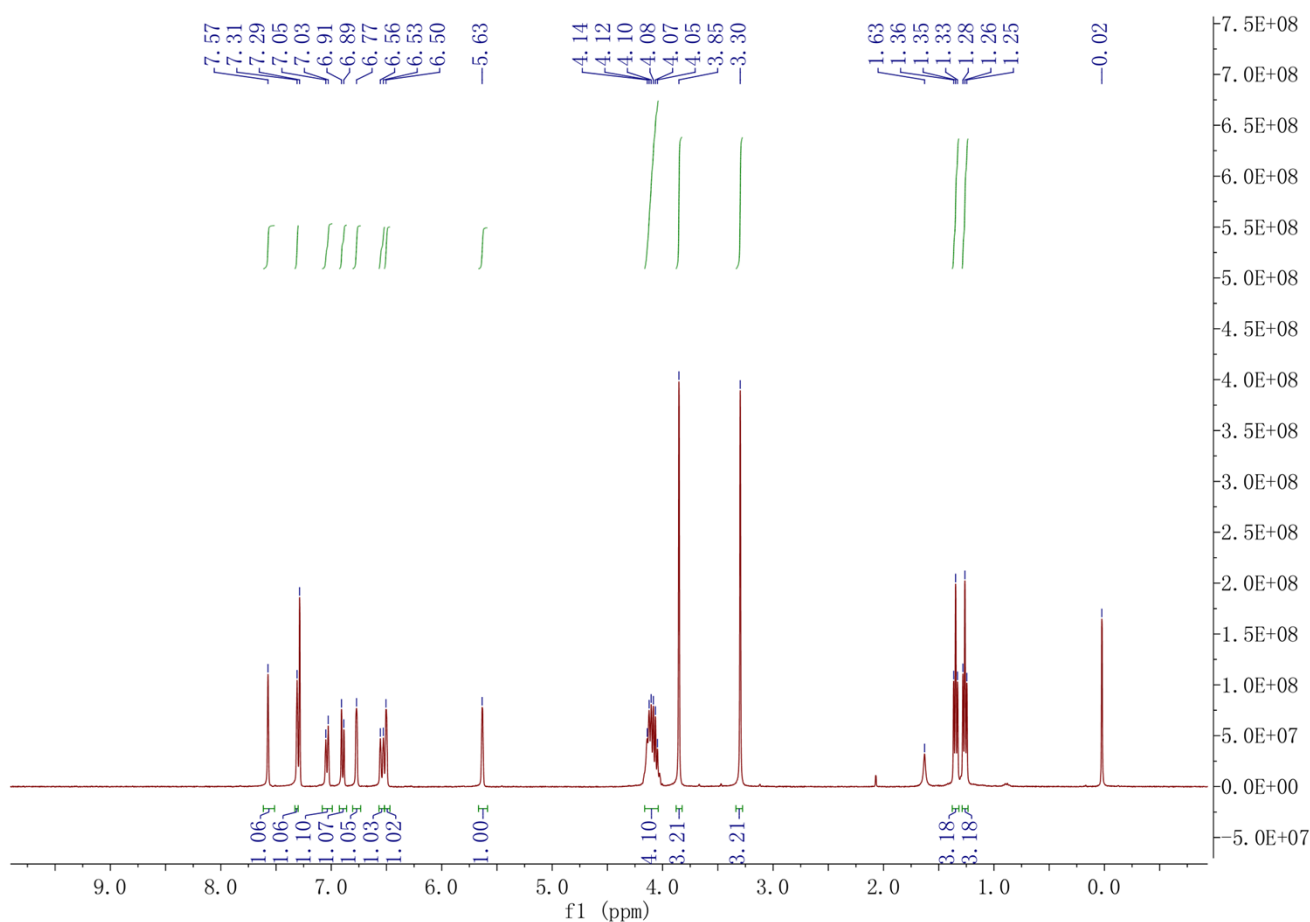


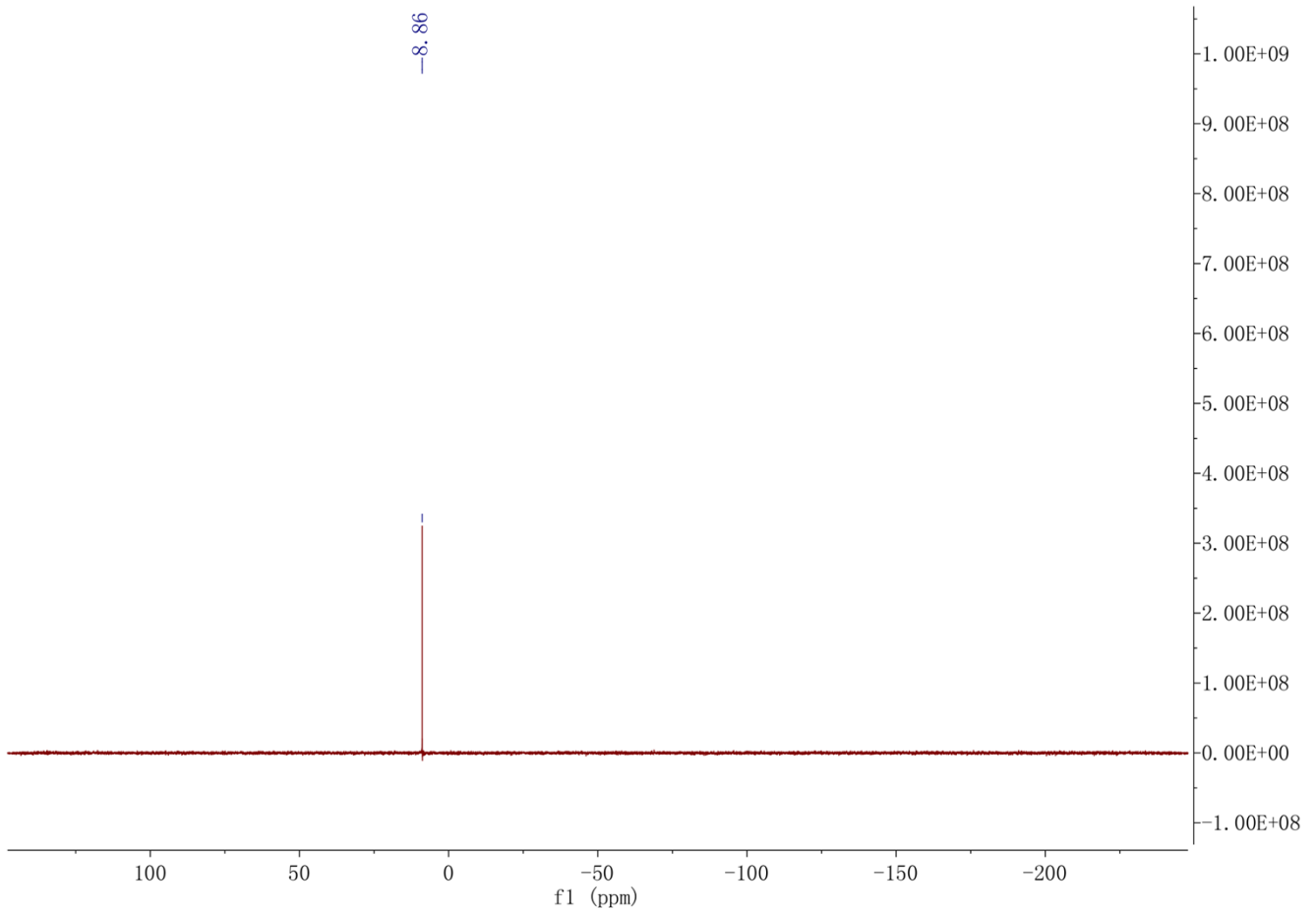
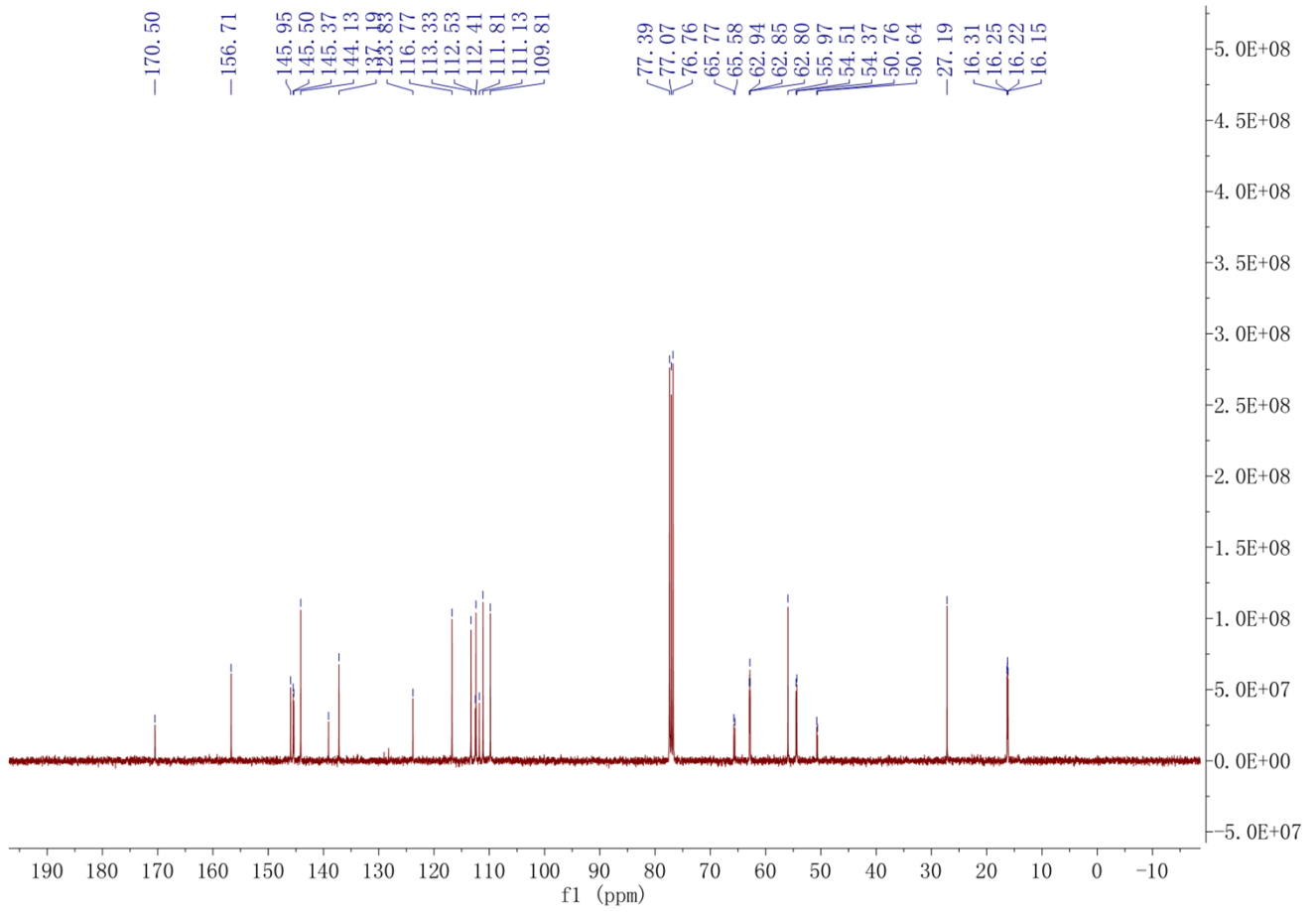


Diethyl(5,5-dicyano-4-(furan-2-yl)-5'-methoxy-1'-methyl-2'-oxospiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (3p):

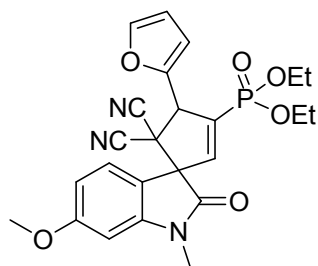


White solid; mp 175 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.57 (s, 1H), 7.31 (s, 1H), 7.04 (d, $J = 8.6$ Hz, 1H), 6.90 (d, $J = 8.5$ Hz, 1H), 6.77 (s, 1H), 6.54 (d, $J = 10.9$ Hz, 1H), 6.50 (s, 1H), 5.63 (s, 1H), 4.02-4.16 (m, 4H), 3.85 (s, 3H), 3.30 (s, 3H), 1.35 (t, $J = 6.9$ Hz, 3H), 1.26 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 170.50, 156.71, 145.95, 145.44 (d, $J = 12.6$ Hz), 144.13, 139.10, 137.19, 123.83, 116.77, 113.33, 112.53, 112.41, 111.81, 111.13, 109.81, 65.68 (d, $J = 18.6$ Hz), 62.91 (d, $J = 5.4$ Hz), 62.83 (d, $J = 5.7$ Hz), 55.97, 54.44 (d, $J = 14.0$ Hz), 50.70 (d, $J = 11.9$ Hz), 27.19, 16.28 (d, $J = 6.3$ Hz), 16.18 (d, $J = 6.6$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 8.86; HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{24}\text{H}_{24}\text{N}_3\text{O}_6\text{P}$: 504.1300, found: 504.1299.

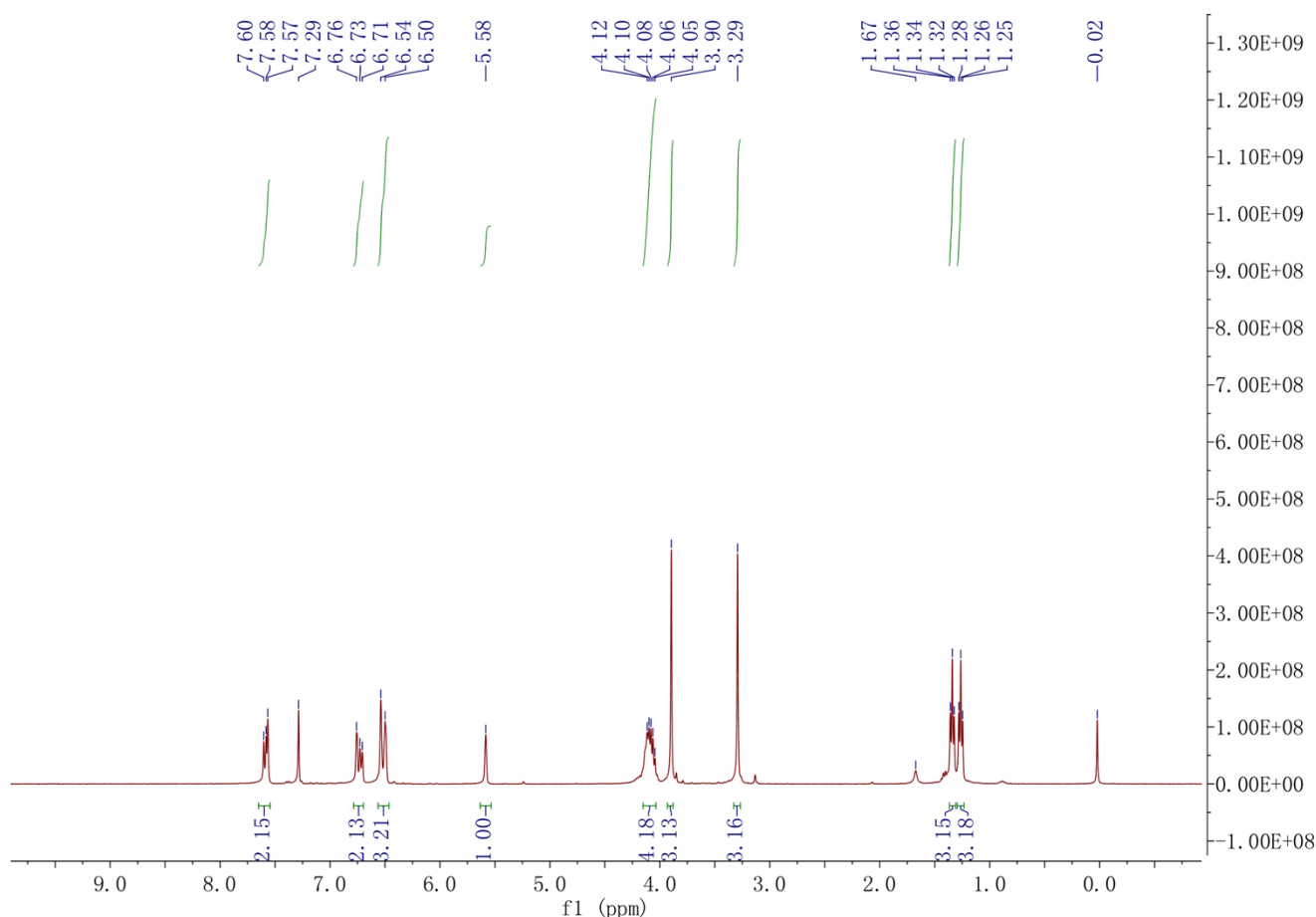


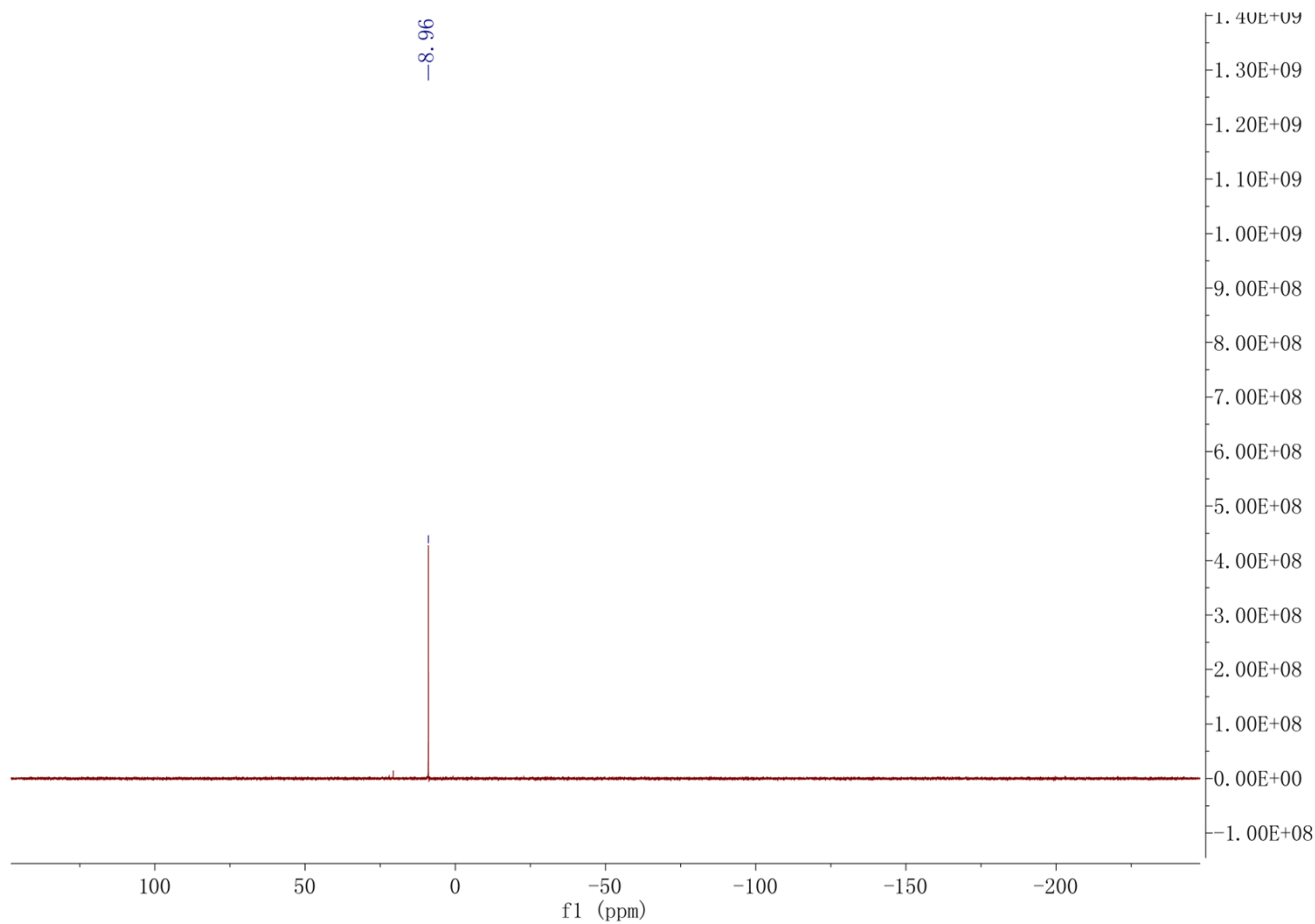
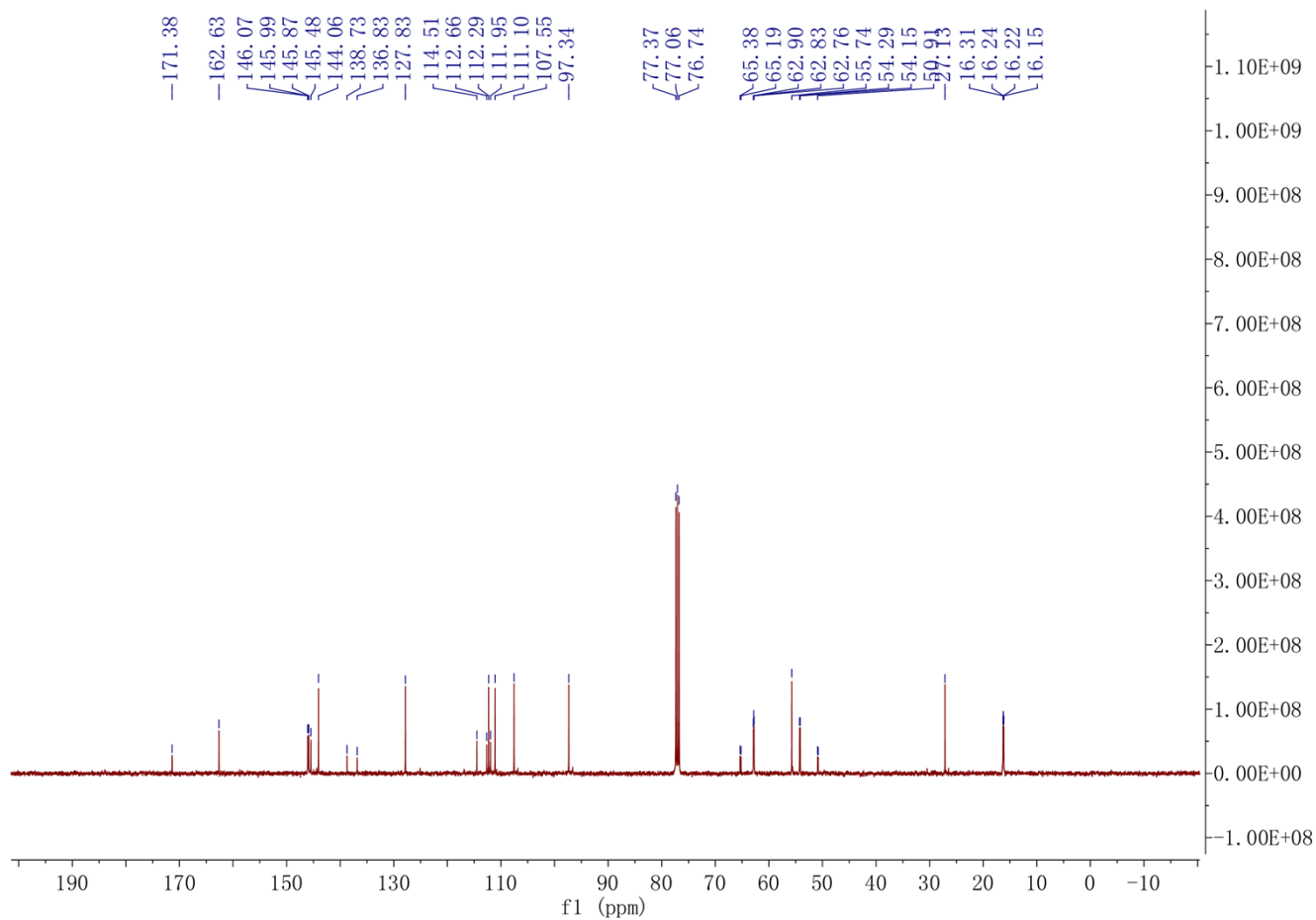


Diethyl(5,5-dicyano-4-(furan-2-yl)-6'-methoxy-1'-methyl-2'-oxospiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (3q):

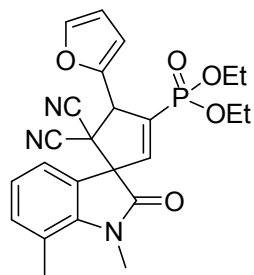


White solid; mp 153 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.49-7.65 (m, 2H), 6.67-6.80 (m, 2H), 6.52 (d, $J = 15.5$ Hz, 3H), 5.58 (s, 1H), 4.03-4.16 (m, 4H), 3.90 (s, 3H), 3.29 (s, 3H), 1.34 (t, $J = 7.0$ Hz, 3H), 1.26 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 171.38, 162.63, 146.07, 145.93 (d, $J = 12.3$ Hz), 145.48, 144.06, 137.78 (d, $J = 191.4$ Hz), 127.83, 114.51, 112.66, 112.29, 111.95, 111.10, 107.55, 97.34, 65.29 (d, $J = 18.8$ Hz), 62.86 (d, $J = 6.8$ Hz), 62.79 (d, $J = 7.1$ Hz), 55.74, 54.22 (d, $J = 14.1$ Hz), 50.85 (d, $J = 11.9$ Hz), 27.13, 16.28 (d, $J = 6.6$ Hz), 16.18 (d, $J = 6.8$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 8.96; HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{24}\text{H}_{24}\text{N}_3\text{O}_6\text{P}$: 504.1300, found: 504.1299.

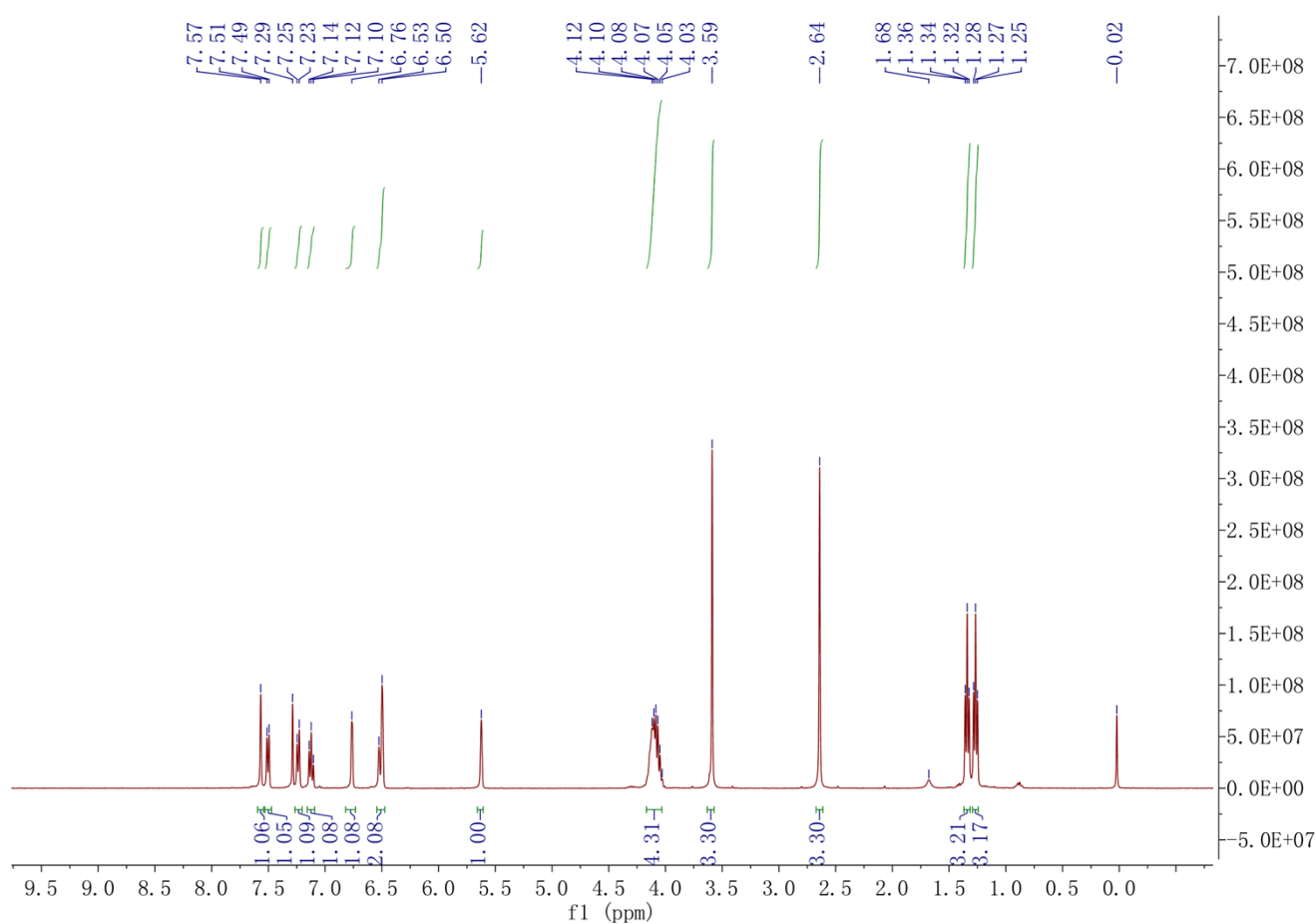


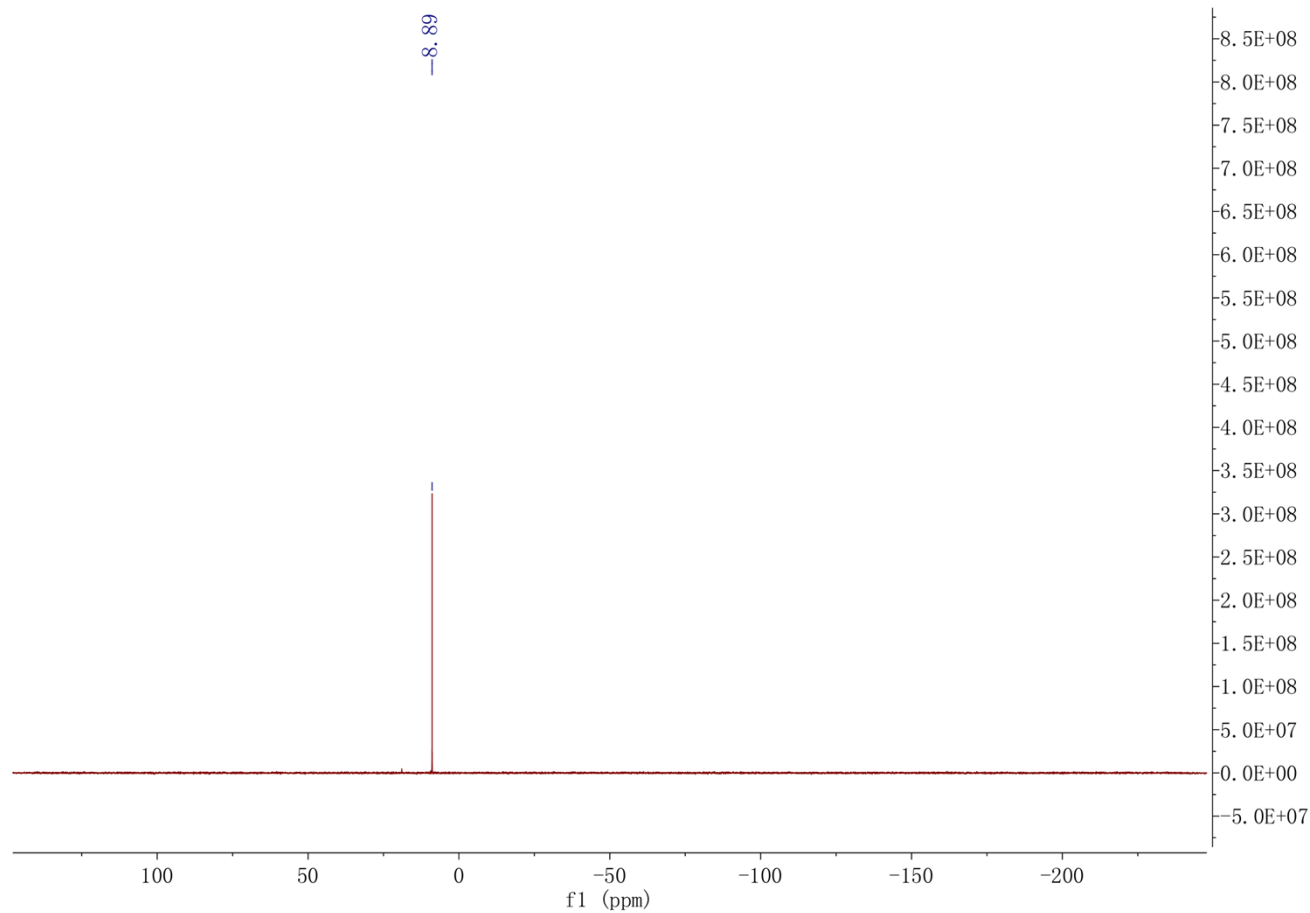
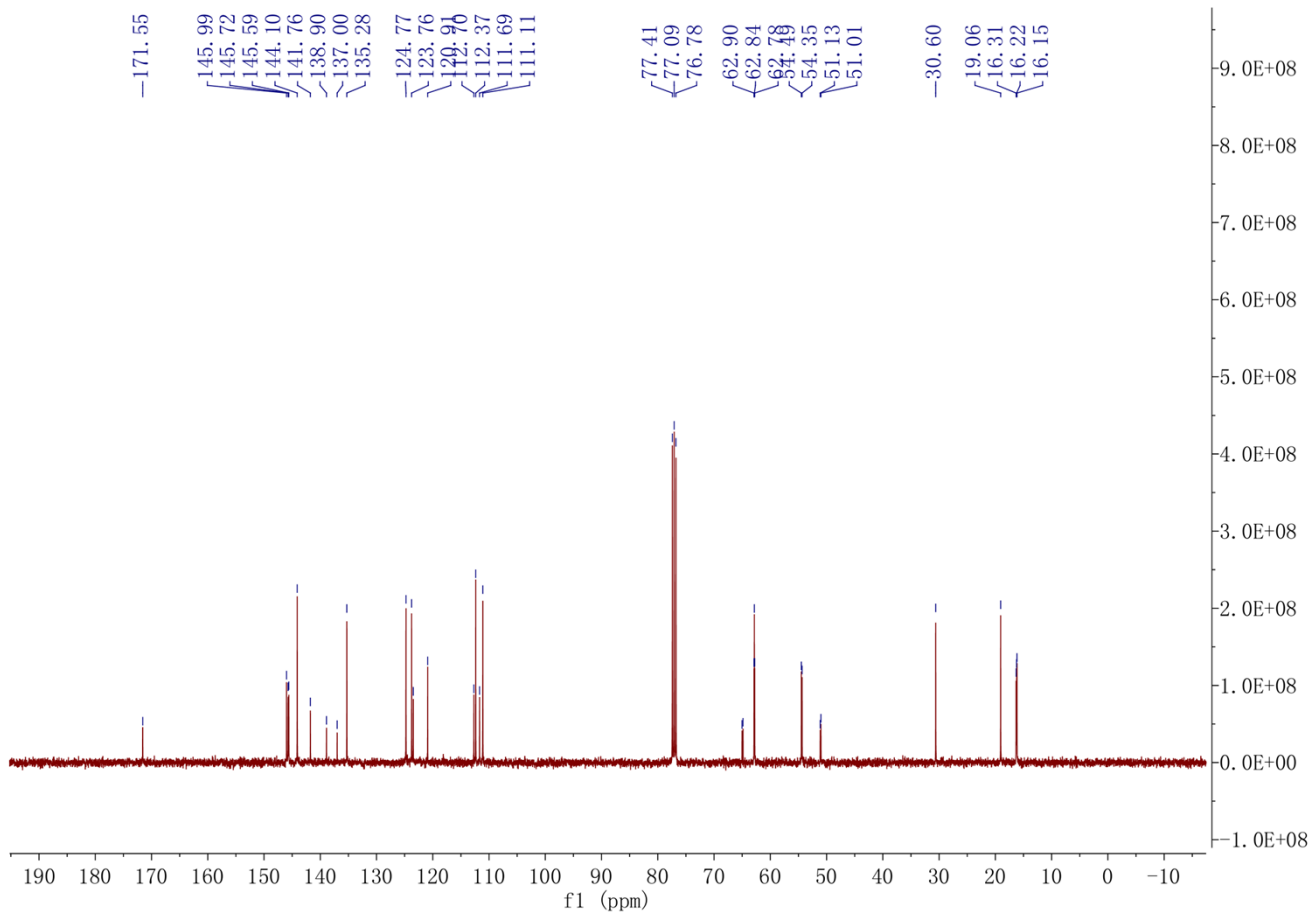


Diethyl(5,5-dicyano-4-(furan-2-yl)-1',7'-dimethyl-2'-oxospiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (3r):

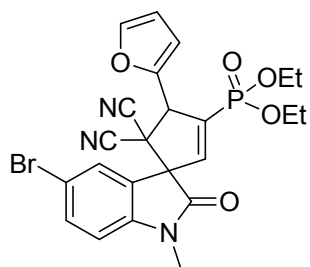


White solid; mp 163 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.57 (s, 1H), 7.50 (d, J = 7.5 Hz, 1H), 7.24 (d, J = 7.7 Hz, 1H), 7.12 (t, J = 7.6 Hz, 1H), 6.76 (s, 1H), 6.51 (d, J = 11.3 Hz, 2H), 5.62 (s, 1H), 4.03-4.16 (m, 4H), 3.59 (s, 3H), 2.64 (s, 3H), 1.34 (t, J = 7.0 Hz, 3H), 1.27 (t, J = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 171.55, 146.00, 145.65 (d, J = 12.5 Hz), 144.10, 141.76, 137.95 (d, J = 191.3 Hz), 135.28, 124.77, 123.76, 123.47, 120.91, 112.70, 112.37, 111.69, 111.11, 64.94 (d, J = 18.7 Hz), 62.84 (t, J = 6.2 Hz), 54.42 (d, J = 13.9 Hz), 51.07 (d, J = 12.4 Hz), 30.60, 19.06, 16.28 (d, J = 6.8 Hz), 16.19 (d, J = 6.6 Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 8.89; HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{24}\text{H}_{24}\text{N}_3\text{O}_5\text{P}$: 488.1351, found:488.1353.

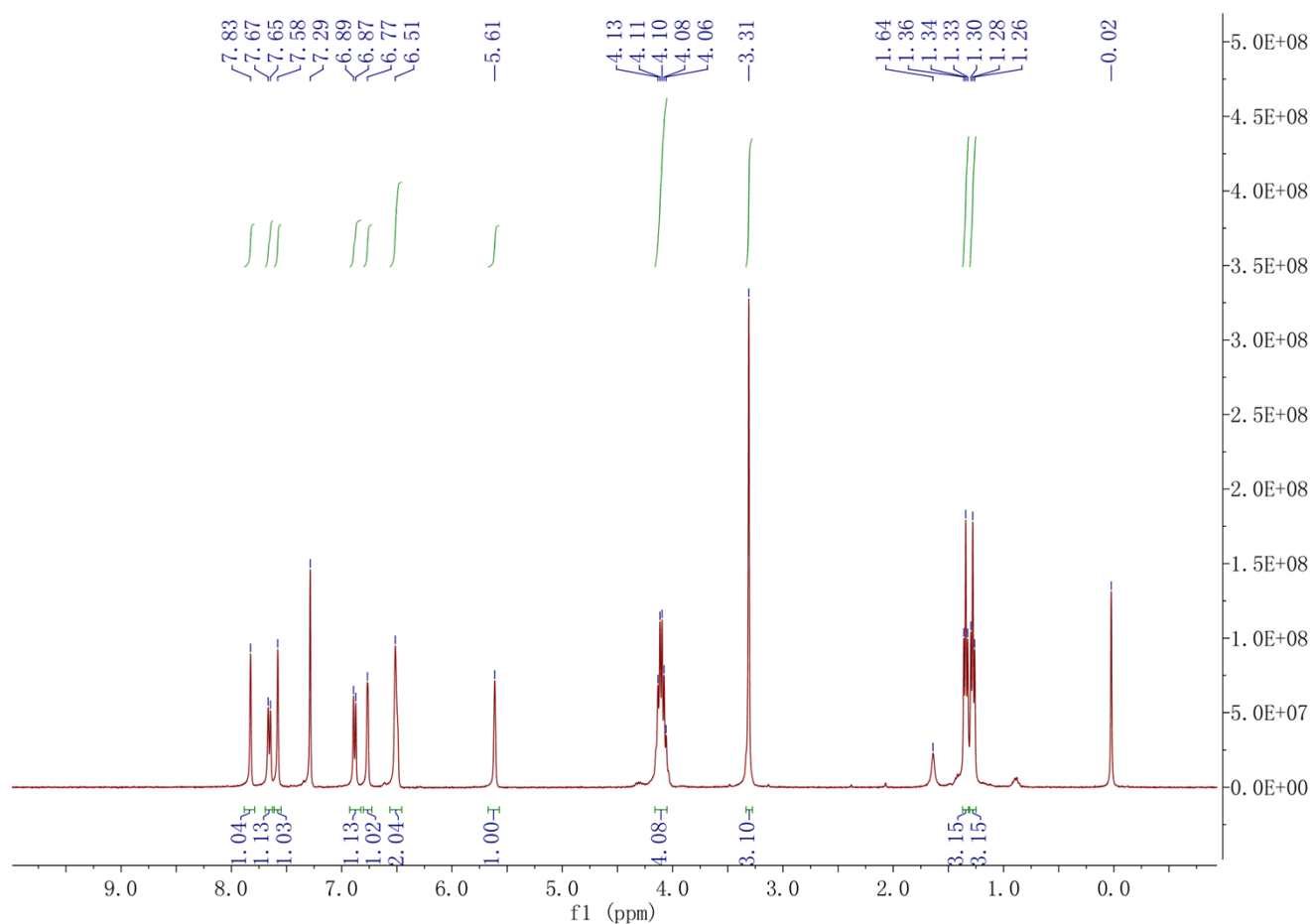


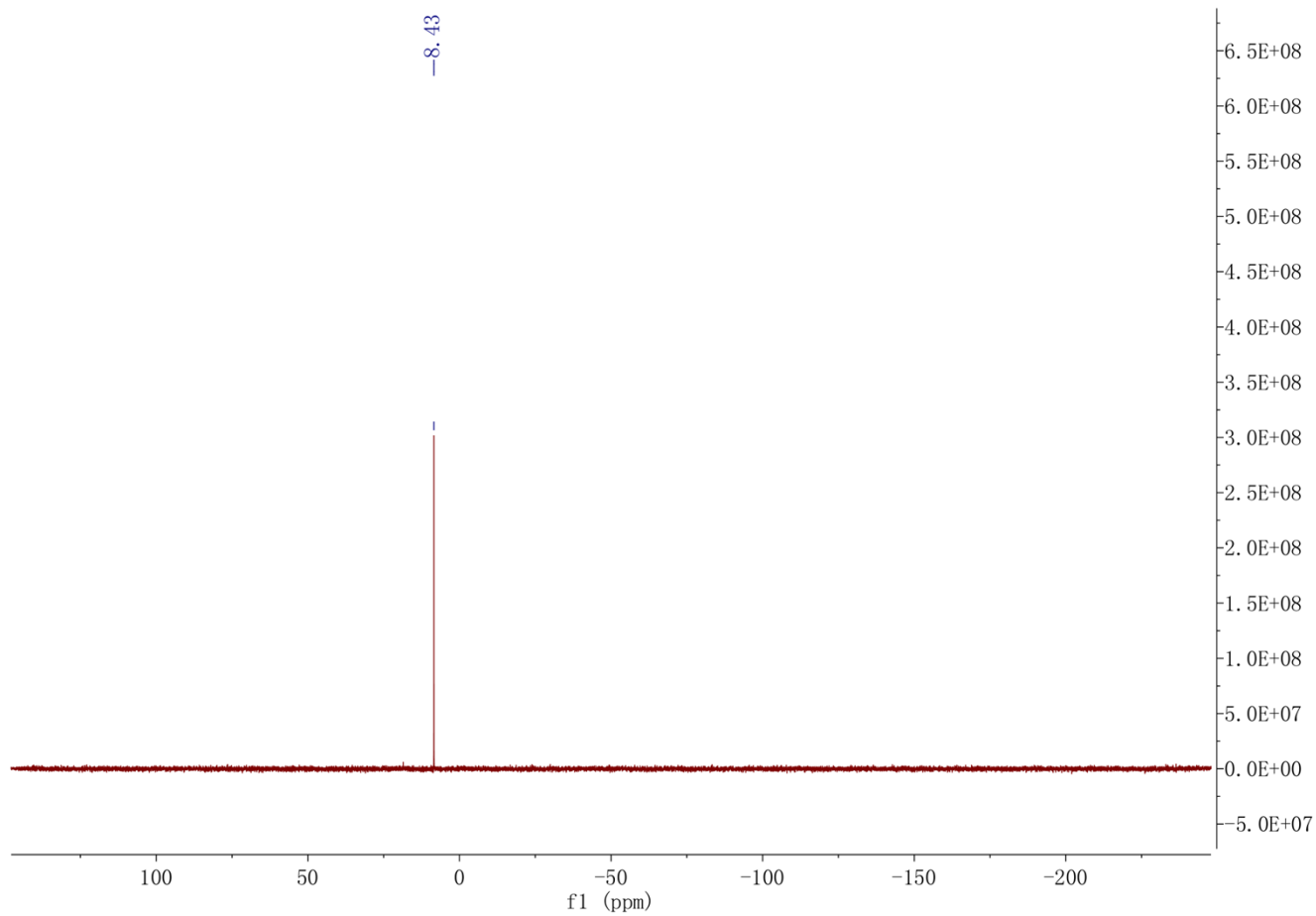
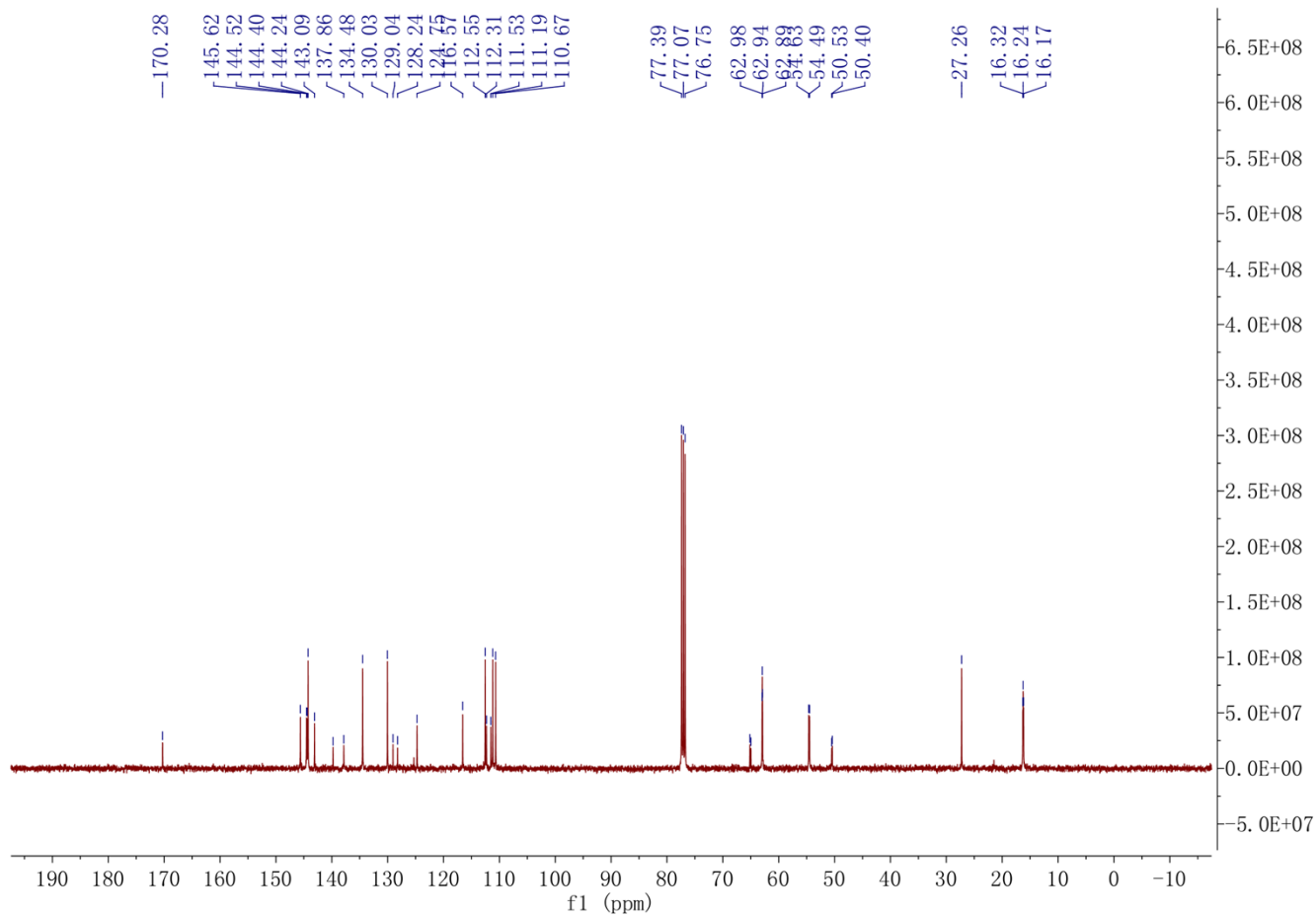


Diethyl(5'-bromo-5,5-dicyano-4-(furan-2-yl)-1'-methyl-2'-oxospiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (3s):

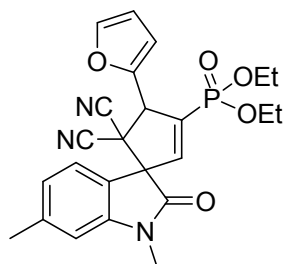


White solid; mp 193 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.83 (s, 1H), 7.66 (d, $J = 8.2$ Hz, 1H), 7.58 (s, 1H), 6.88 (d, $J = 8.3$ Hz, 1H), 6.77 (s, 1H), 6.51 (s, 2H), 5.61 (s, 1H), 4.10 (dt, $J = 14.8, 7.4$ Hz, 4H), 3.31 (s, 3H), 1.34 (t, $J = 7.0$ Hz, 3H), 1.28 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 170.28, 145.62, 144.46 (d, $J = 12.7$ Hz), 144.24, 143.09, 138.81 (d, $J = 191.6$ Hz), 134.48, 130.03, 128.64 (d, $J = 81.4$ Hz), 124.75, 116.57, 112.55, 111.92 (d, $J = 78.6$ Hz), 111.19, 110.67, 65.07 (d, $J = 18.9$ Hz), 62.96 (d, $J = 4.0$ Hz), 62.91 (d, $J = 4.4$ Hz), 54.56 (d, $J = 13.9$ Hz), 50.46 (d, $J = 12.4$ Hz), 27.26, 16.28 (d, $J = 7.7$ Hz), 16.21 (d, $J = 7.7$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 8.43; HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{23}\text{H}_{21}\text{BrN}_3\text{O}_5\text{P}$: 552.0300, found:552.0293.

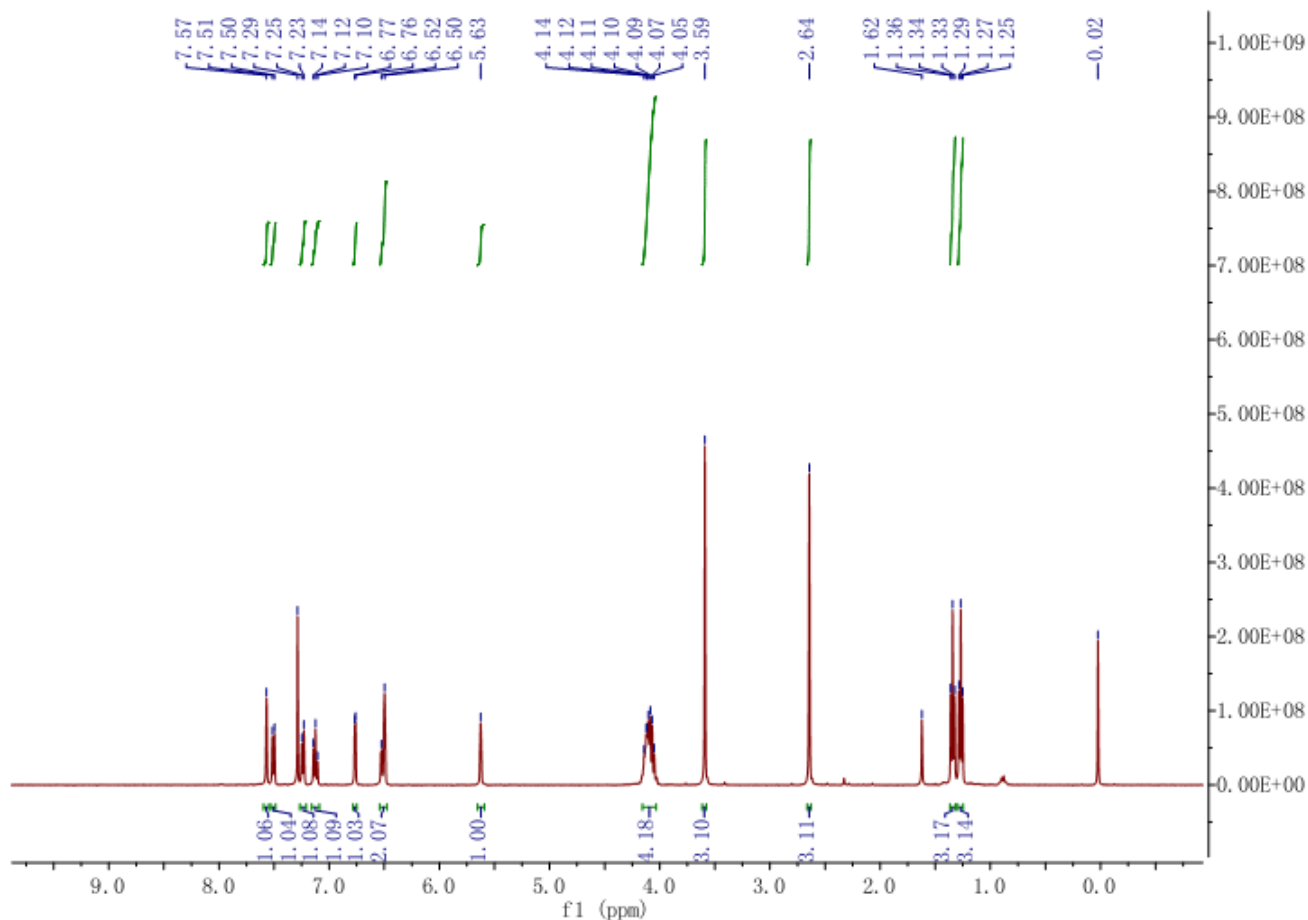


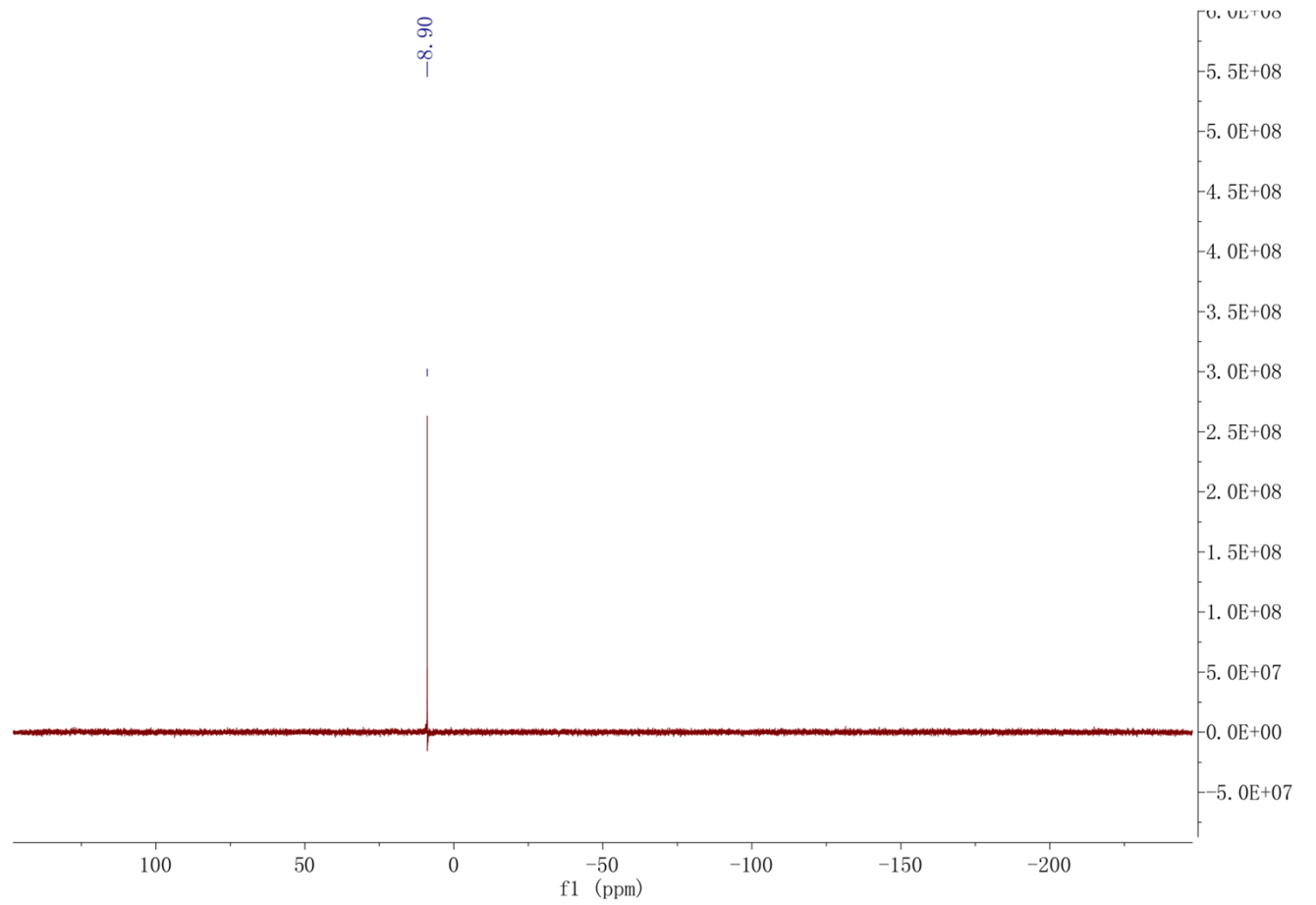
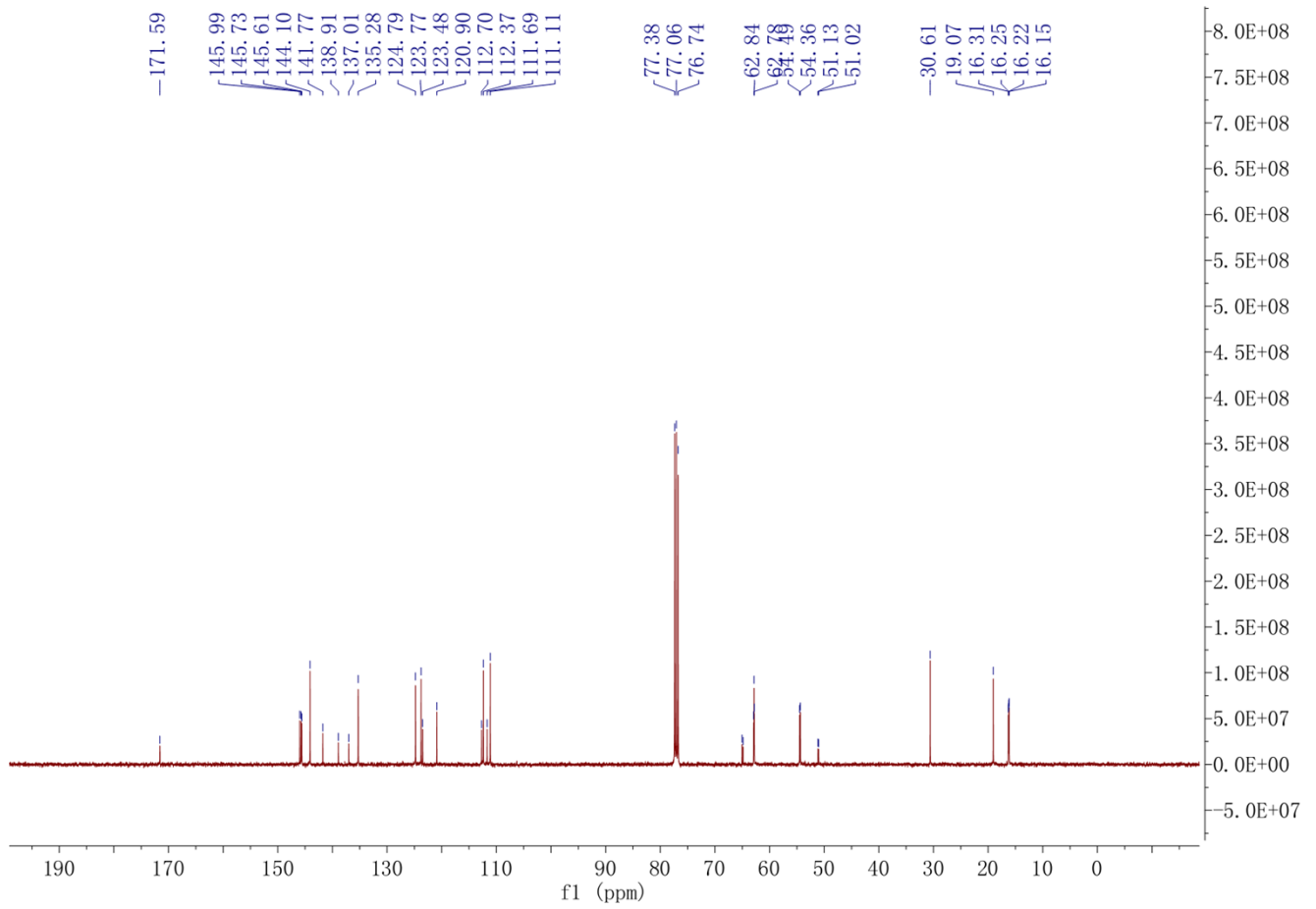


Diethyl(5,5-dicyano-4-(furan-2-yl)-1',6'-dimethyl-2'-oxospiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (3t):

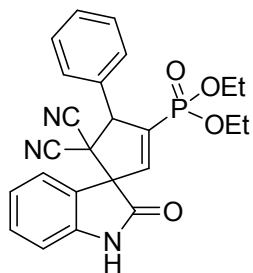


White solid; mp 181 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.57 (s, 1H), 7.51 (d, $J = 7.5$ Hz, 1H), 7.24 (d, $J = 7.7$ Hz, 1H), 7.12 (t, $J = 7.7$ Hz, 1H), 6.76 (d, $J = 3.0$ Hz, 1H), 6.51 (d, $J = 10.9$ Hz, 2H), 5.63 (s, 1H), 4.02-4.15 (m, 4H), 3.59 (s, 3H), 2.64 (s, 3H), 1.34 (t, $J = 7.0$ Hz, 3H), 1.27 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 171.59, 145.99, 145.67 (d, $J = 12.4$ Hz), 144.10, 141.77, 137.96 (d, $J = 191.5$ Hz), 135.28, 124.79, 123.77, 123.48, 120.90, 112.70, 112.37, 111.69, 111.11, 64.95 (d, $J = 18.6$ Hz), 62.84 (t, $J = 6.3$ Hz), 54.43 (d, $J = 13.9$ Hz), 51.07 (d, $J = 11.7$ Hz), 30.61, 19.07, 16.28 (d, $J = 6.6$ Hz), 16.19 (d, $J = 6.6$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 8.90; HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{24}\text{H}_{24}\text{N}_3\text{O}_5\text{P}$: 488.1351, found: 488.1353.

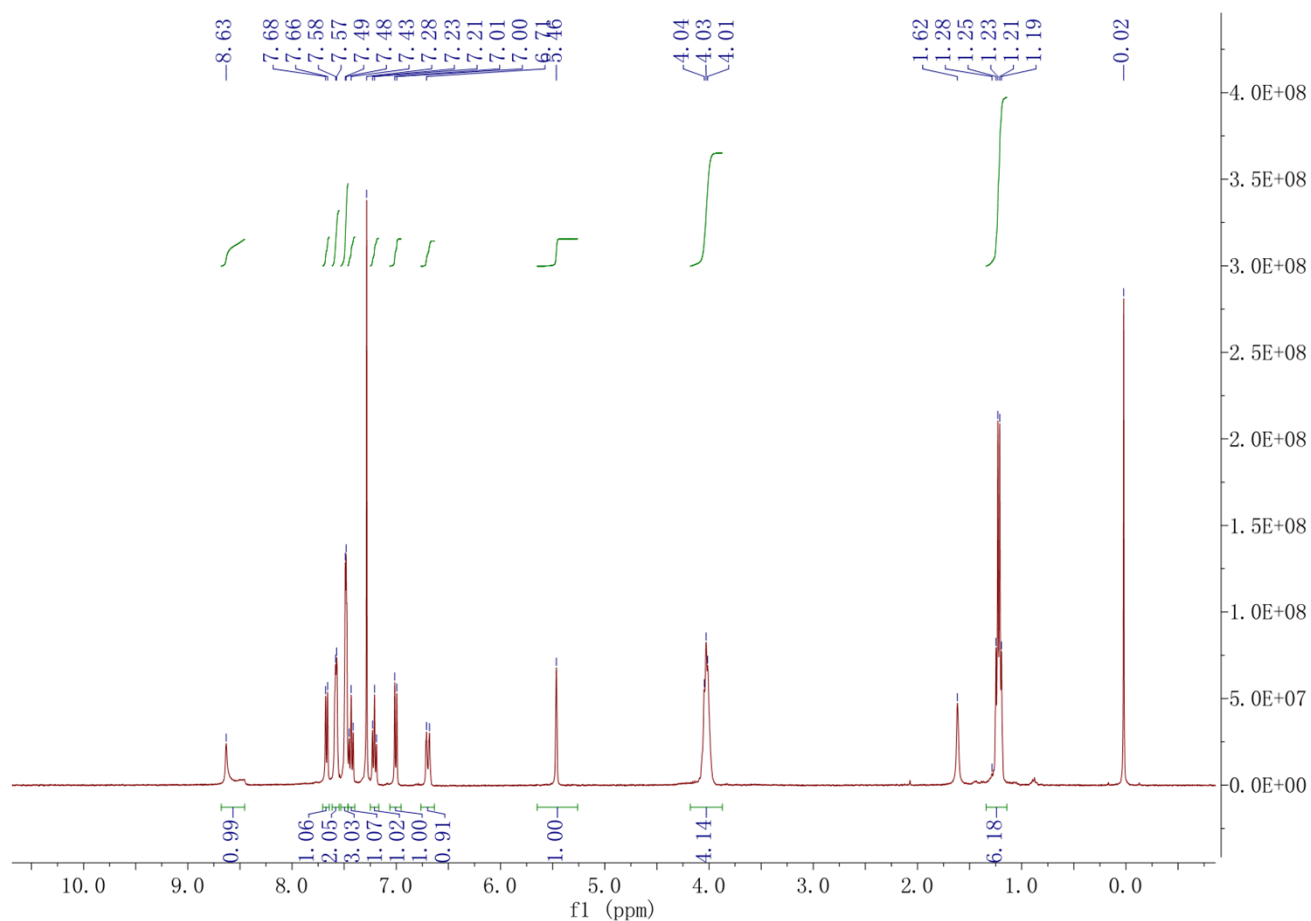


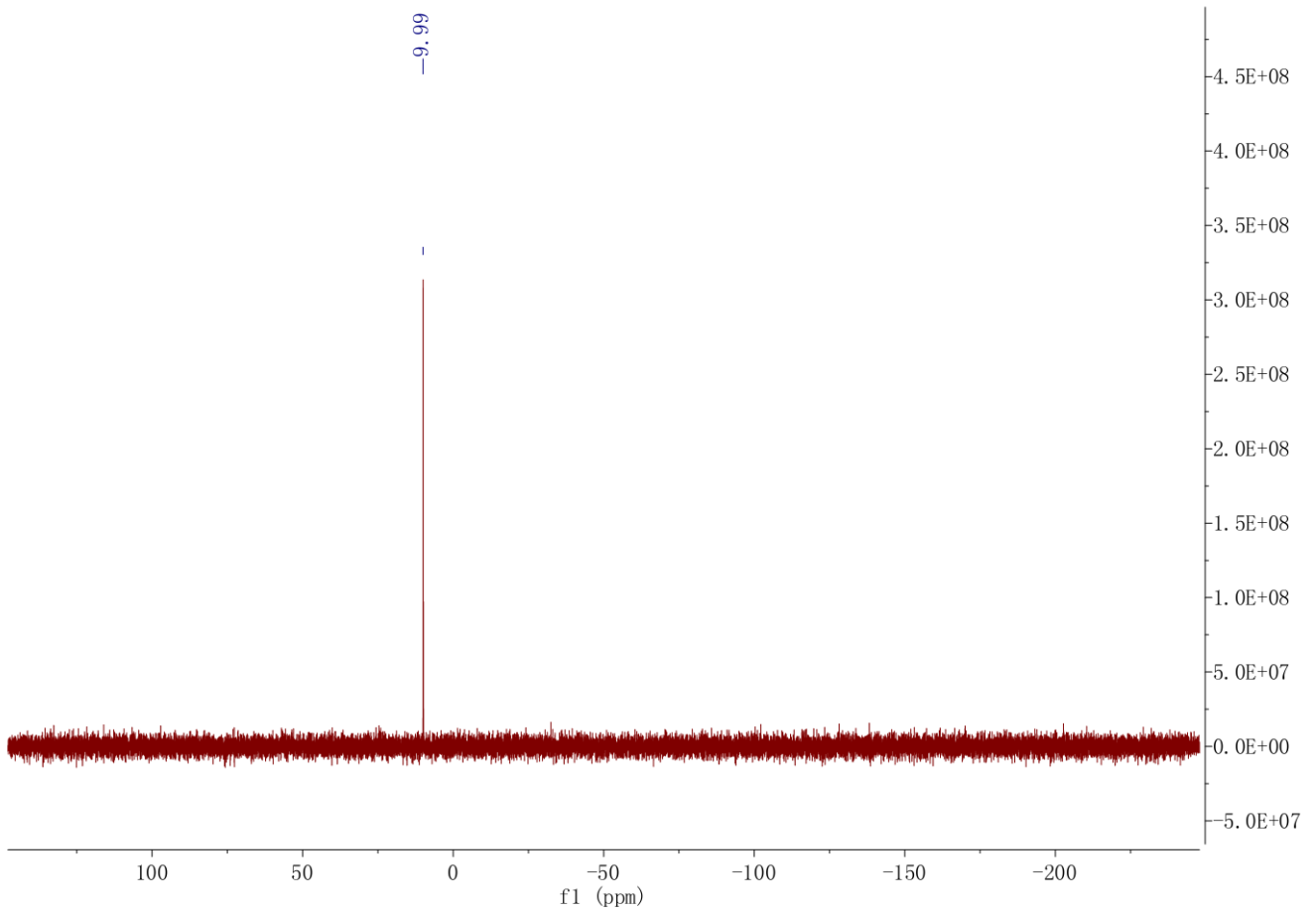
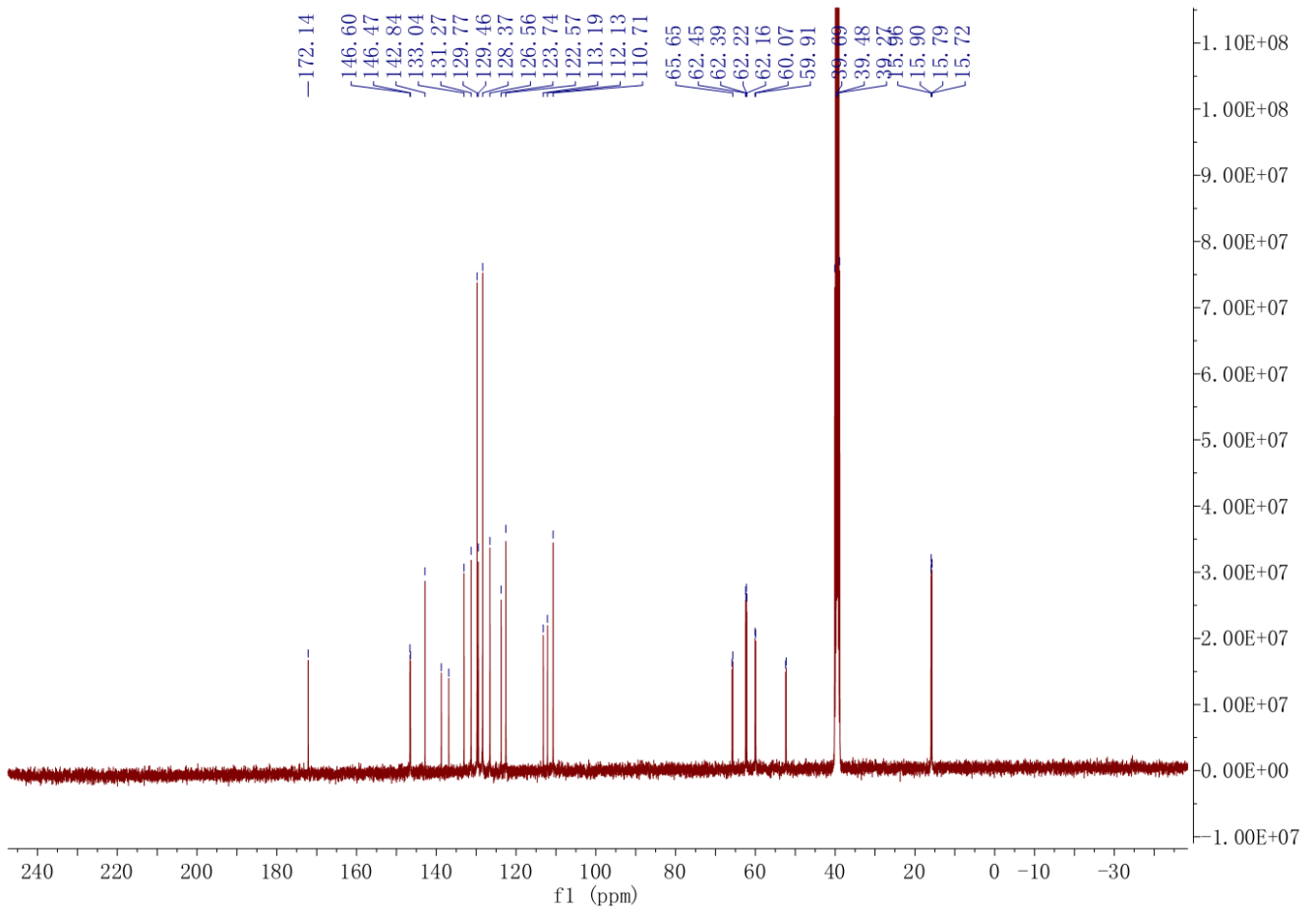


Diethyl(5,5-dicyano-2'-oxo-4-phenylspiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate (3u):



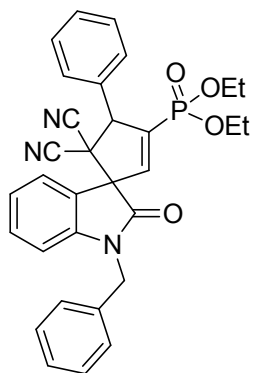
White solid; mp 253 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.63 (s, 1H), 7.67 (d, $J = 7.6$ Hz, 1H), 7.58 (d, $J = 3.8$ Hz, 2H), 7.49 (d, $J = 3.2$ Hz, 3H), 7.43 (t, $J = 7.8$ Hz, 1H), 7.21 (t, $J = 7.6$ Hz, 1H), 7.21 (t, $J = 7.6$ Hz, 1H), 7.01 (d, $J = 7.8$ Hz, 1H), 6.70 (d, $J = 11.2$ Hz, 1H), 5.46 (s, 1H), 3.97-4.08 (m, 4H), 1.22 (q, $J = 6.9$ Hz, 6H); ^{13}C NMR (101 MHz, DMSO): δ 172.14, 146.54 (d, $J = 12.8$ Hz), 142.84, 138.75, 136.88, 133.04, 131.27, 129.46, 129.07 (d, $J = 140.7$ Hz), 126.64, 123.74, 122.57, 113.19, 112.13, 110.71, 65.75 (d, $J = 19.1$ Hz), 62.42 (d, $J = 6.0$ Hz), 62.19 (d, $J = 5.9$ Hz), 59.99 (d, $J = 16.0$ Hz), 52.35 (d, $J = 12.8$ Hz), 15.93 (d, $J = 6.3$ Hz), 15.75 (d, $J = 6.2$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 9.99; HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{24}\text{H}_{22}\text{N}_3\text{O}_4\text{P}$: 470.1246, found: 470.1237.



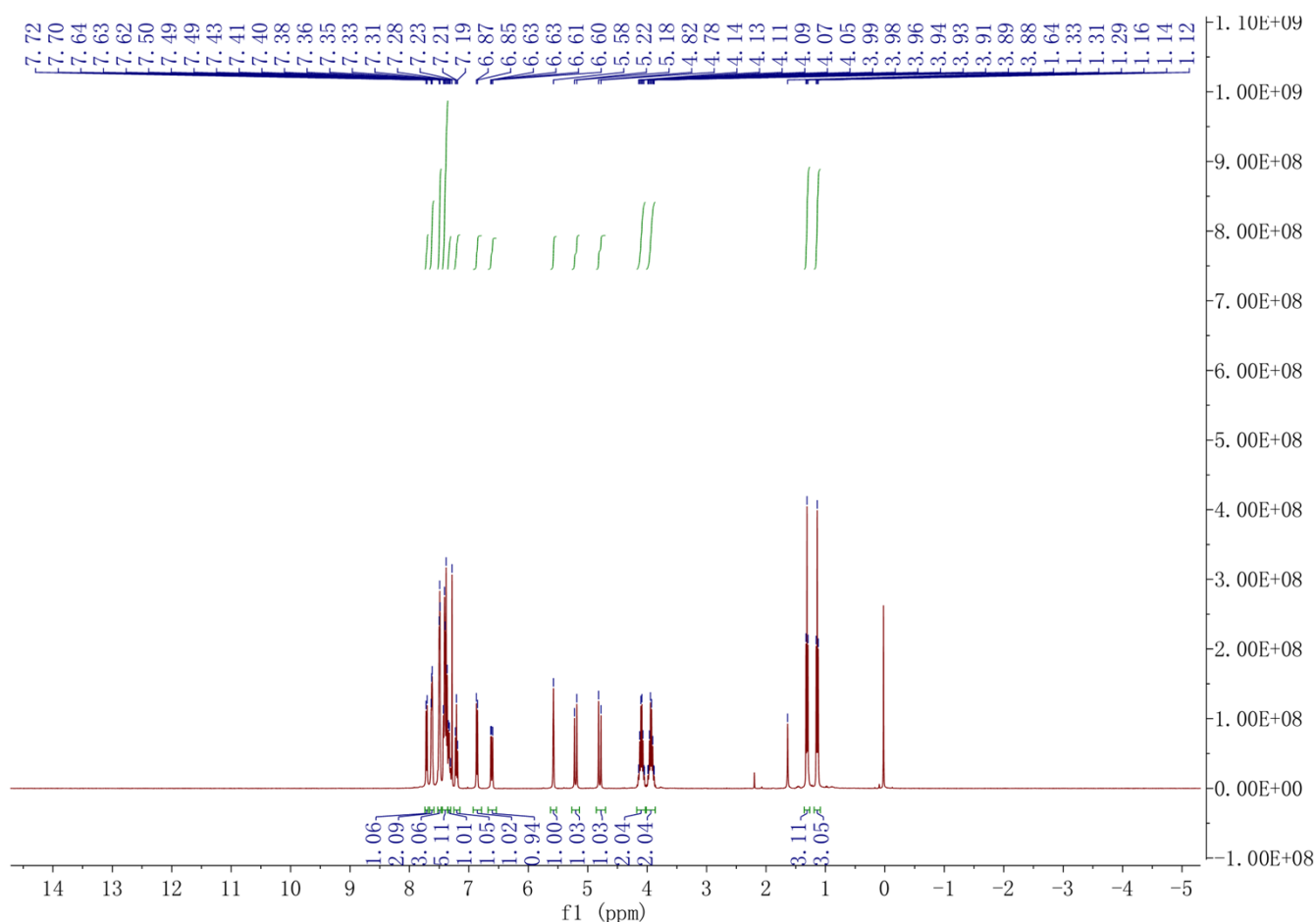


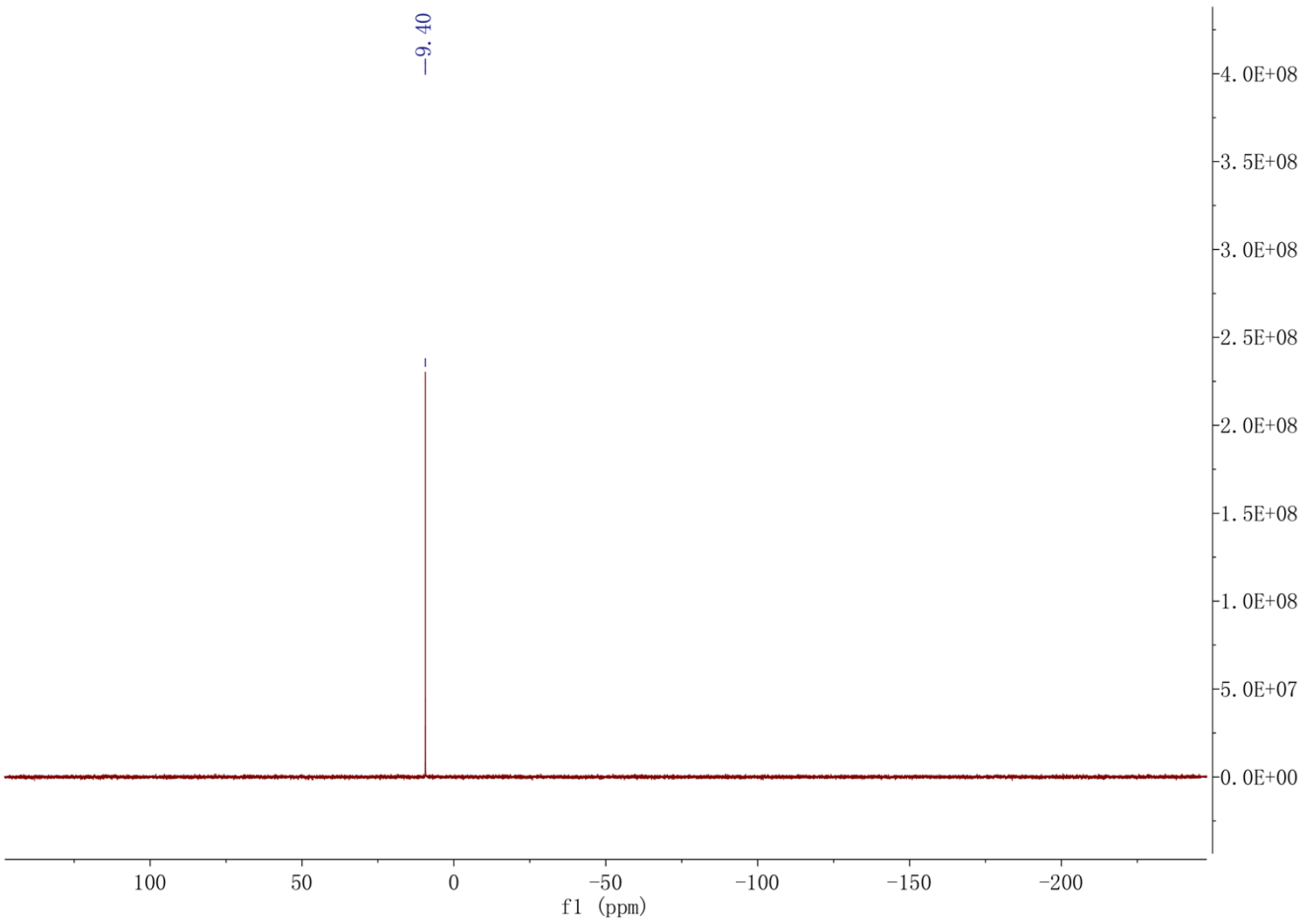
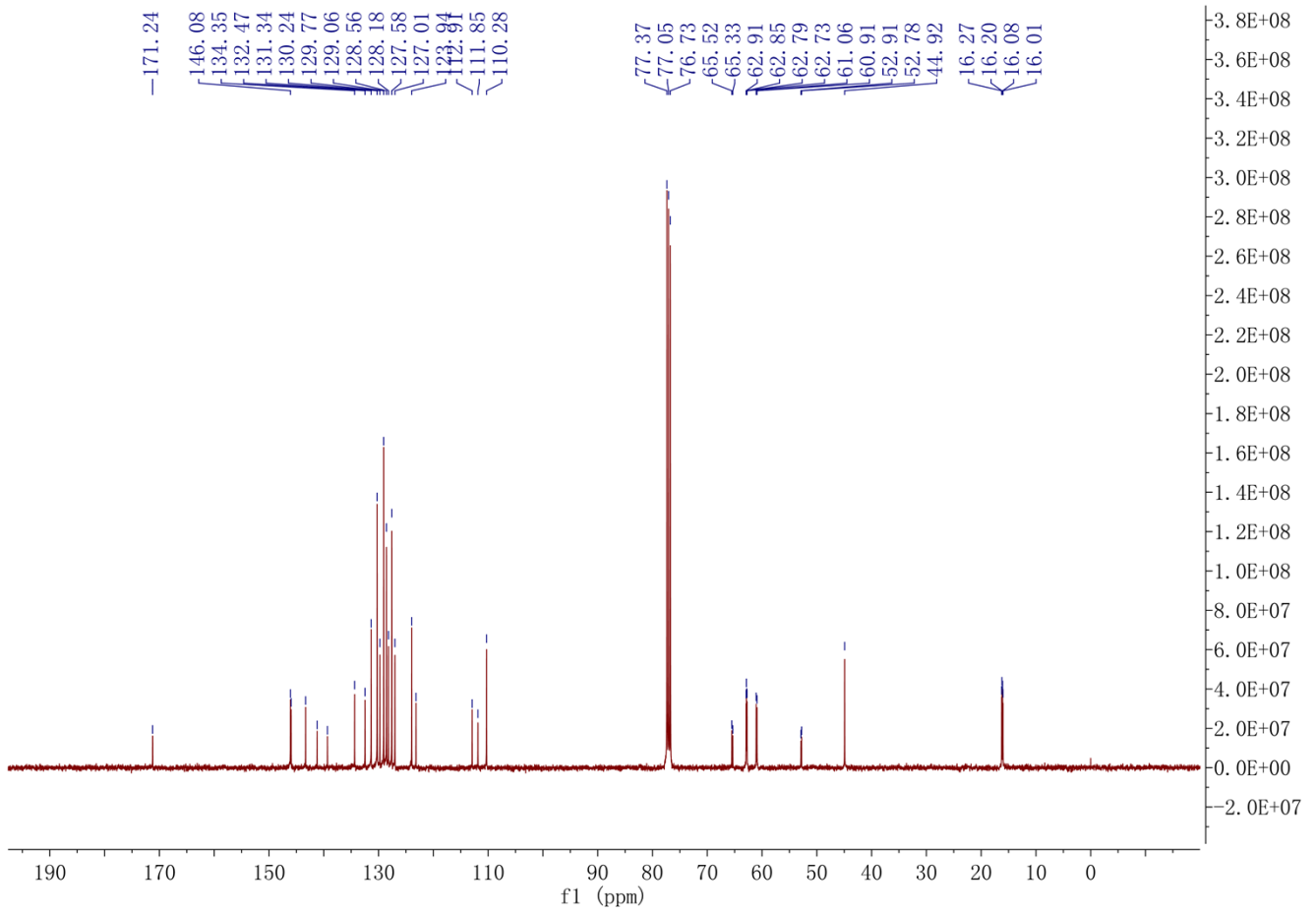
Diethyl(1'-benzyl-5,5-dicyano-2'-oxo-4-phenylspiro[cyclopent[2]ene-1,3'-indolin]-3-yl)phosphonate

(3v):



White solid; mp 168 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.71 (d, $J = 7.6$ Hz, 1H), 7.60-7.65 (m, 2H), 7.46-7.52 (m, 3H), 7.40 (dt, $J = 15.3, 7.5$ Hz, 5H), 7.31-7.35 (m, 1H), 7.21 (t, $J = 7.7$ Hz, 1H), 6.86 (d, $J = 7.9$ Hz, 1H), 6.62 (dd, $J = 11.0, 2.6$ Hz, 1H), 5.58 (s, 1H), 5.20 (d, $J = 15.6$ Hz, 1H), 4.80 (d, $J = 15.6$ Hz, 1H), 4.20 – 4.02 (m, 2H), 4.01 – 3.85 (m, 2H), 1.31 (t, $J = 7.1$ Hz, 3H), 1.14 (t, $J = 7.1$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3): δ 171.24, 146.02 (d, $J = 12.6$ Hz), 143.31, 140.26 (d, $J = 190.6$ Hz), 134.35, 132.47, 131.34, 130.24, 129.77, 129.06, 128.56, 128.18, 127.58, 127.01, 123.94, 123.16, 112.91, 111.85, 110.28, 65.43 (d, $J = 18.8$ Hz), 62.88 (d, $J = 5.9$ Hz), 62.76 (d, $J = 6.3$ Hz), 60.99 (d, $J = 14.8$ Hz), 52.84 (d, $J = 13.2$ Hz), 44.92, 16.24 (d, $J = 6.3$ Hz), 16.04 (d, $J = 6.8$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 9.40; HRMS calculated $[\text{M}+\text{Na}]^+$ for $\text{C}_{31}\text{H}_{28}\text{N}_3\text{O}_4\text{P}$: 560.1715, found: 560.1714.





Single Crystal X-Ray Analysis **3a** (CCDC 993220 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge by contacting The Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033; E-mail: deposit@ccdc.cam.ac.uk.)

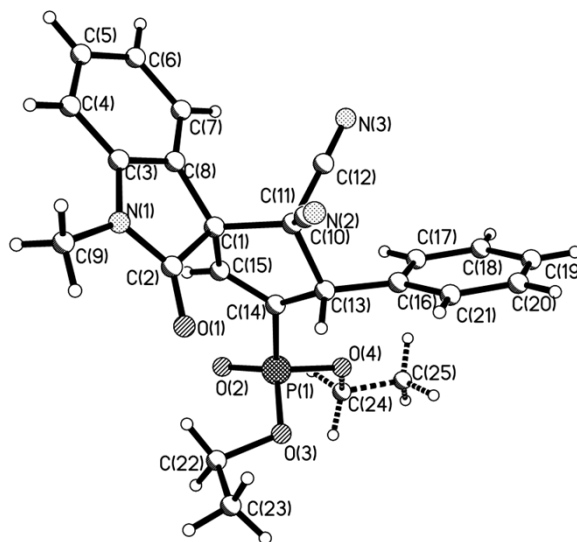


Table 1. Crystal data and structure refinement for shelxl.

Identification code	shelxl	
Empirical formula	C ₂₅ H ₂₄ N ₃ O ₄ P	
Formula weight	461.44	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system, space group	Monoclinic, P2 ₁ /c	
Unit cell dimensions	a = 11.598(2) Å	alpha = 90 deg.
	b = 12.442(3) Å	beta = 122.44(2) deg.
	c = 19.597(6) Å	gamma = 90 deg.
Volume	2386.6(10) Å ³	
Z, Calculated density	4, 1.284 Mg/m ³	
Absorption coefficient	0.151 mm ⁻¹	
F(000)	968	
Crystal size	0.30 x 0.14 x 0.12 mm	

Theta range for data collection	2.05 to 25.02 deg.
Limiting indices	-13<=h<=13, -14<=k<=14, -23<=l<=23
Reflections collected / unique	19606 / 4210 [R(int) = 0.0603]
Completeness to theta = 25.02	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9821 and 0.9561
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	4210 / 128 / 337
Goodness-of-fit on F ²	0.961
Final R indices [I>2sigma(I)]	R1 = 0.0530, wR2 = 0.1311
R indices (all data)	R1 = 0.0812, wR2 = 0.1498
Extinction coefficient	0.035(3)
Largest diff. peak and hole	0.270 and -0.227 e.A ⁻³