Supporting Information

Photoelectrochemical cell for P-H/C-H cross-coupling with hydrogen evolution

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1. Materials and methods

^{1}H NMR spectra were recorded using a Bruker Avance DPX 400 MHz instrument with tetramethylsilane (TMS) as an internal standard. ^{13}C NMR spectra were obtained at 101 MHz and referenced to the internal solvent signals. Multiplicities are indicated, s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), coupling constants (J) are in Hertz (Hz). High-resolution mass spectra (HRMS-ESI) were recorded on a Q-TOF mass spectrometer. Commercially available reagents and solvents were used without further purification. Cyclic voltammograms were obtained on a CHI 660E potentiostat. Photoelectrochemical was carried out with blue LEDs (λ = 450 nm). The instrument for electrolysis is dual display potentiostat (DJS-292B) (made in China). The anode electrode is reticulated vitreous carbon (RVC) electrodes (500 PPI, 1.0 cm × 1.0 cm × 0.5 cm) and the cathode electrode is platinum plate electrodes (0.5 cm × 1.0 cm). The reticulated vitreous carbon electrodes can be available at Alfa Aesar (CAS No. 7440-44-0). Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were packed with 200-300 mesh silica gel in petroleum ether.

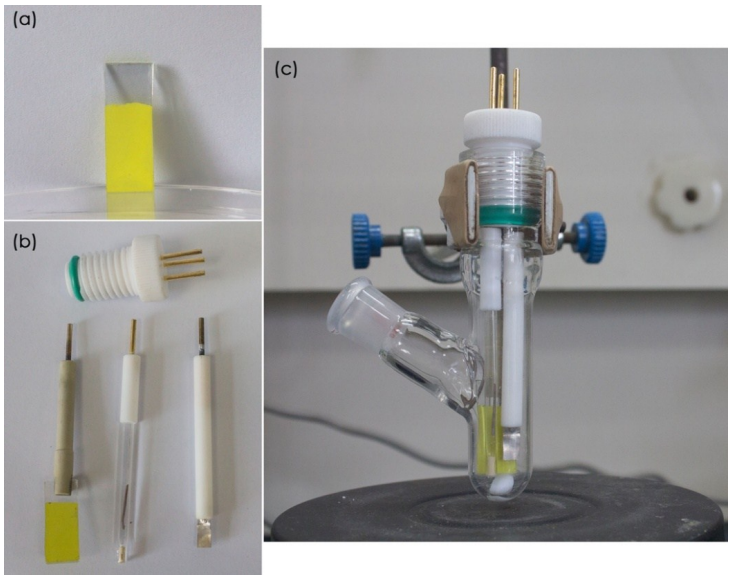


Figure S1. The picture of the home-made $BiVO_4$ electrode and the photoelectrochemical cell.

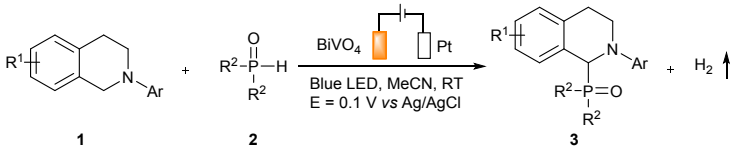
2. General experimental procedures

2.1 Preparation of the $BiVO_4$

The $BiVO_4$ electrode was prepared by electro-deposition. First, $Bi(NO_3)_3 \cdot 5H_2O$ (0.04 M) and KI (0.4 M) were dissolved in 50 mL distilled water to form a clear solution. Then, HNO_3 was added to adjust the pH to 1.7. Next, 0.23 M p-benzoquinone ethanol solution (20 mL) was added to above mixed solution and stirred vigorously for 20 min. The electrodeposition was carried out in three-electrode cell using a constant potential of -0.1 V *vs* Ag/AgCl for 10 min at room temperature. (FTO as working electrode, Pt plate as the counter electrode and Ag/AgCl as the reference electrode). After being taken out and washed with distilled water, the dried BiOI film was covered by dipping 200 μL containing vanadyl acetylacetonate (0.2 M) DMSO solution. $BiVO_4$ film was formed by heating in a muffle furnace at 450 °C for 1 hour. After being cooled down to room temperature, the electrode was soaked in 1.0 M NaOH solution for 30 min to remove excess V_2O_5. Finally, the $BiVO_4$ electrode was rinsed with distilled water.

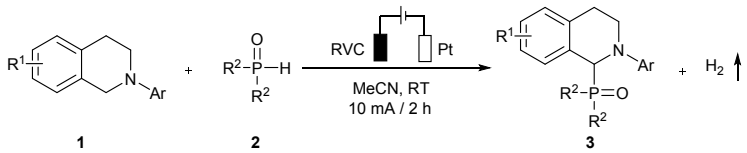
2.2 General procedure for the C-H/P-H Cross-coupling:

Method A: Photoelectrochemical



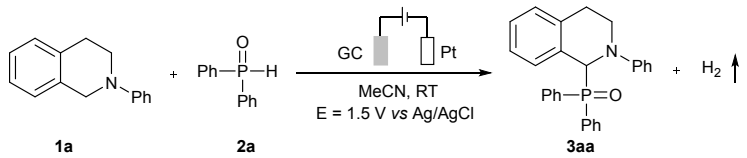
The PEC experiments were carried out in an undivided cell equipped with $BiVO_4$ as the working electrode (1.0 cm × 2.0 cm), Pt plate as the counter electrode (0.5 cm × 1.0 cm) and Ag/AgCl as the reference electrode. N-phenyl tetrahydroisoquinoline **1a** (0.2 mmol), diphenylphosphine oxide **2a** (0.2 mmol), NHPI (0.2 equiv) and 2,6-Lutidine (1.5 equiv) were dissolved in 5 mL acetonitrile with $^{n}Bu_4NBF_4$ (0.1 M) as an electrolyte. The reaction mixture was stirred for 1 minutes. Then the PEC cell was sealed using a rubber septum and flushed with nitrogen gas. After piercing the septum with a nitrogen-filled balloon to sustain nitrogen atmosphere, photoelectrochemical electrolysis was performed at a constant potential of +0.1 V *vs*. Ag/AgCl for 12 h. When the reaction was finished, the entire reaction mixture was then transferred to a silica gel column and eluted with a mixture of petrol ether and ethyl acetate to give the corresponding products.

Method B: Electrochemical



The electrolysis was carried out in an undivided cell, with a RVC anode (500 PPI, 1.0 cm × 1.0 cm × 0.5 cm) and a platinum cathode (0.5 cm × 1.0 cm). N-phenyl tetrahydroisoquinoline **1a** (0.2 mmol) and diphenylphosphine oxide **2a** (0.2 mmol) were dissolved in 5 mL acetonitrile with $^{n}Bu_4NBF_4$ (0.1 M) as an electrolyte. The reaction mixture was then stirred for 1 minutes. Then the EC cell was sealed using a rubber septum and flushed with nitrogen gas. After piercing the septum with a nitrogen-filled balloon to sustain nitrogen atmosphere, electrolysis was performed at a constant current of 10 mA for 2 h. When the reaction was finished, the entire reaction mixture was then transferred to a silica gel column and eluted with a mixture of petrol ether and ethyl acetate to give the corresponding products.

2.3 Control Experiments:



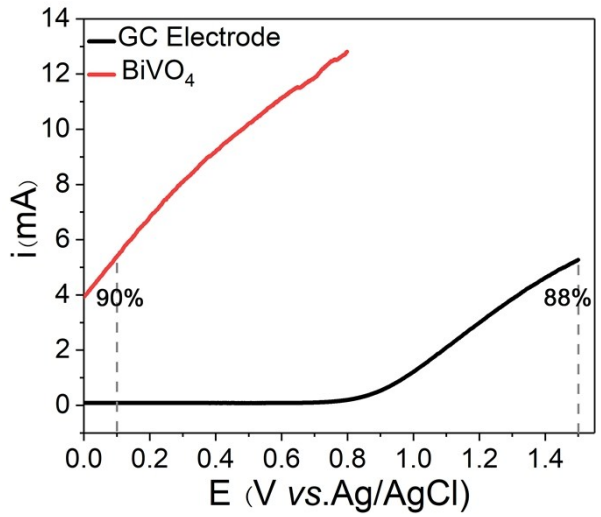


Figure S2. The comparison of PEC and EC. The NHPI-mediated P-H/C-H cross-coupling in a PEC cell (red) is cathodically shifted by 1.4 V compared to an EC cell (black).

The control experiments were carried out in an undivided cell equipped with glassy carbon electrode as the working electrode (1.0 cm × 2.0 cm × 0.3 cm), Pt plate as the counter electrode (0.5 cm × 1.0 cm) and Ag/AgCl as the reference electrode. N-phenyl tetrahydroisoquinoline **1a** (0.2 mmol), diphenylphosphine oxide **2a** (0.2 mmol), NHPI (0.2 equiv) and 2,6-Lutidine (1.5 equiv) were dissolved in 5 mL acetonitrile with $^{n}Bu_4NBF_4$ (0.1 M) as an electrolyte. The reaction mixture was stirred for 1 minutes. Then the EC cell was sealed using a rubber septum and flushed with nitrogen gas. After piercing the septum with a nitrogen-filled balloon to sustain nitrogen atmosphere, electrolysis was performed at a constant potential of +1.5 V *vs*. Ag/AgCl for 12 h. When the reaction was finished, the entire reaction mixture was then transferred to a silica gel column and eluted with a mixture of petrol ether and ethyl acetate to give the corresponding products.

3. Cyclic voltammograms experiments

The cyclic voltammograms were recorded in an electrolyte of $^{n}Bu_4NBF_4$ (0.1 M) in MeCN using a glassy carbon disk working electrode (diameter, 3 mm), a Pt wire auxiliary electrode and a Ag/AgCl reference electrode. The scan rate was 100 mV/s.

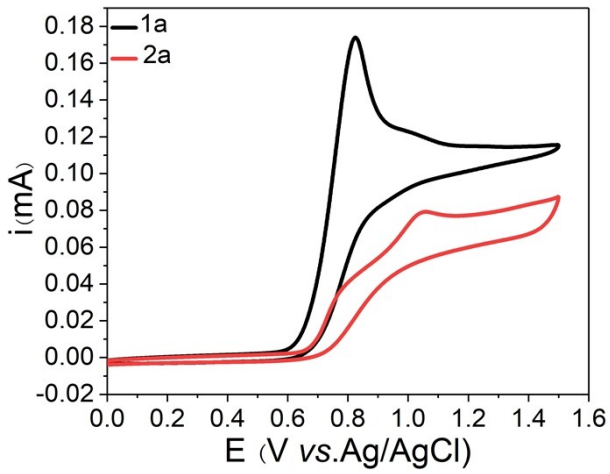


Figure S3. CVs obtained in MeCN (6 mL) with 0.1 M $^{n}Bu_4NBF_4$: black line, **1a** (5 mM); red line, **2a** (5 mM).

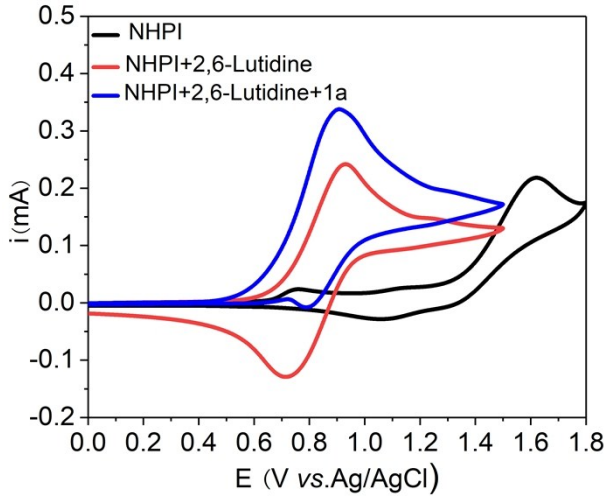
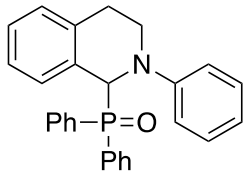


Figure S4. CVs obtained in MeCN (6 mL) with 0.1 M $^{n}Bu_4NBF_4$: 5 mM NHPI (black); 5 mM NHPI solution containing 10 mM 2,6-Lutidine (red); 5 mM NHPI solution containing 10 mM 2,6-Lutidine and 5 mM **1a** (blue).

4. Characterization data for all products

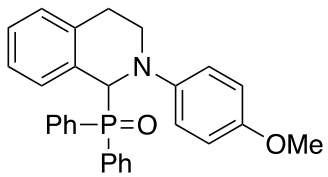


diphenyl(2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphine oxide (3aa)

Reaction was proceeded according to General Method A with 0.1 V *vs*. Ag/AgCl constant potential electrolysis. The reaction mixture was purified by silica gel column chromatography to afford the product **3aa**. (white solid, mp 197.7−200.1 °C, yield: 90%).

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3aa**. (white solid, mp 197.7−200.1 °C, yield: 85%).

^{1}H NMR (400 MHz, Chloroform-d) δ 7.85 – 7.76 (m, 2H), 7.74 – 7.66 (m, 2H), 7.59 – 7.49 (m, 1H), 7.49 – 7.39 (m, 3H), 7.35 - 7.30 (m, 2H), 7.17 – 7.04 (m, 4H), 6.93 (t, J = 7.5 Hz, 1H), 6.84 – 6.73 (m, 3H), 6.65 (d, J = 7.8 Hz, 1H), 5.56 (d, J = 10.6 Hz, 1H), 4.10 – 3.96 (m, 1H), 3.67 – 3.43 (m, 1H), 2.90 – 2.76 (m, 1H), 2.73 – 2.62 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-d) δ 150.00 (d, $J_{C\text{-}P}$ = 9.3 Hz), 136.89 (d, $J_{C\text{-}P}$ = 4.5 Hz), 132.28 (d, $J_{C\text{-}P}$ = 90.6 Hz), 132.26 (d, $J_{C\text{-}P}$ = 8.5 Hz), 131.92 (d, $J_{C\text{-}P}$ = 2.9 Hz), 131.71 (d, $J_{C\text{-}P}$ = 9.25 Hz), 131.37 (d, $J_{C\text{-}P}$ = 84.3 Hz), 131.68, 129.94, 129.24 (d, $J_{C\text{-}P}$ = 2.4 Hz), 129.13, 128.44 (d, $J_{C\text{-}P}$ = 11.1 Hz), 128.27 (d, $J_{C\text{-}P}$ = 11.38 Hz), 127.79 (d, $J_{C\text{-}P}$ = 3.6 Hz), 127.41 (d, $J_{C\text{-}P}$ = 3.4 Hz), 125.52 (d, $J_{C\text{-}P}$ = 3.0 Hz), 119.55, 116.79, 61.98 (d, $J_{C\text{-}P}$ = 79.7 Hz), 45.17, 25.63. ^{31}P NMR (162 MHz, Chloroform-d) δ 32.34. HRMS (ESI) calcd. for $C_{27}H_{24}NNaOP$ $[M+Na]^{+}$: 432.1488. Found: 432.1486.



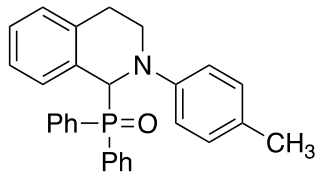
(2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)diphenylphosphine oxide (3ba)

Reaction was proceeded according to General Method A with 0.1 V *vs*. Ag/AgCl constant

potential electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ba**. (white solid, mp 175.3−176.9 °C, yield: 88%).

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3 ba**. (white solid, mp 175.3−176.9 °C, yield: 80%).

^{1}H NMR (400 MHz, Chloroform-d) δ 7.82 – 7.64 (m, 4H), 7.55 – 7.49 (m, 1H), 7.48 – 7.40 (m, 3H), 7.37 - 7.32 (m, 2H), 7.19 – 7.11 (m, 1H), 7.07 (d, J = 7.6 Hz, 1H), 6.93 (t, J = 7.5 Hz, 1H), 6.78 – 6.68 (m, 4H), 6.62 (d, J = 7.8 Hz, 1H), 5.36 (d, J = 11.8 Hz, 1H), 4.06 – 3.88 (m, 1H), 3.72 (s, 3H), 3.47 – 3.33 (m, 1H), 2.81 – 2.68 (m, 1H), 2.60 – 2.50 (m, 1H).^{13}C NMR (101 MHz, Chloroform-d) δ 154.08, 144.65 (d, $J_{C\text{-}P}$ = 10.2 Hz), 137.04 (d, $J_{C\text{-}P}$ = 4.8 Hz), 132.49 (d, $J_{C\text{-}P}$ = 96.43 Hz), 132.18 (d, $J_{C\text{-}P}$ = 8.7 Hz), 131.93 (d, $J_{C\text{-}P}$ = 92.43 Hz), 131.80 (d, $J_{C\text{-}P}$ = 9.2 Hz), 131.79 (d, $J_{C\text{-}P}$ = 2.9 Hz), 131.54 (d, $J_{C\text{-}P}$ = 3.1 Hz), 129.75, 129.36 (d, $J_{C\text{-}P}$ = 2.5 Hz), 128.39 (d, $J_{C\text{-}P}$ = 11.2 Hz), 128.22 (d, $J_{C\text{-}P}$ = 11.4 Hz), 127.80 (d, $J_{C\text{-}P}$ = 3.6 Hz), 127.23 (d, $J_{C\text{-}P}$ = 3.4 Hz), 125.46 (d, $J_{C\text{-}P}$ = 3.0 Hz), 120.38, 114.47, 62.18 (d, $J_{C\text{-}P}$ = 81.5 Hz), 55.53, 46.81, 24.82. ^{31}P NMR (162 MHz, Chloroform-d) δ 32.64. HRMS (ESI) calcd. for $C_{28}H_{26}NNaO_2P$ $[M+Na]^+$: 462.1593. Found: 462.1577



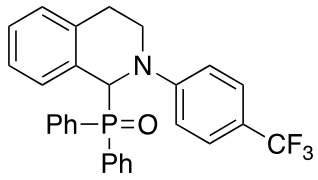
diphenyl(2-(p-tolyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphine oxide (3ca)

Reaction was proceeded according to General Method A with 0.1 V *vs*. Ag/AgCl constant potential electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ca**. (white solid, mp 184.6−186.3 °C, yield: 90%).

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ca**. (white solid, mp 184.6−186.3 °C, yield: 87%).

^{1}H NMR (400 MHz, Chloroform-d) δ 7.83 – 7.75 (m, 2H), 7.74 – 7.65 (m, 2H), 7.55 - 7.51 (m,

1H), 7.48 – 7.41 (m, 3H), 7.38 – 7.30 (m, 2H), 7.13 (t, J = 7.5 Hz, 1H), 7.06 (d, J = 7.6 Hz, 1H), 6.96 -6.90 (m, 3H), 6.71 (d, J = 8.1 Hz, 2H), 6.64 (d, J = 7.8 Hz, 1H), 5.47 (d, J = 11.4 Hz, 1H), 4.14 – 3.83 (m, 1H), 3.62 – 3.45 (m, 1H), 2.93 – 2.70 (m, 1H), 2.66 – 2.52 (m, 1H), 2.22 (s, 3H).^{13}C NMR (101 MHz, Chloroform-d) δ 148.01 (d, J_{C-P} = 9.3 Hz), 136.93 (d, J_{C-P} = 4.5 Hz), 132.43 (d, J_{C-P} = 95.8 Hz), 132.24 (d, J_{C-P} = 8.6 Hz), 131.83 (d, J_{C-P} = 3.0 Hz), 131.82 (d, J_{C-P} = 110.2 Hz), 131.75 (d, J_{C-P} = 8.8 Hz), 131.57 (d, J_{C-P} = 3.2 Hz), 129.86, 129.68, 129.31 (d, J_{C-P} = 2.5 Hz), 128.41 (d, J_{C-P} = 11.1 Hz), 128.24 (d, J_{C-P} = 11.4 Hz), 127.82 (d, J_{C-P} = 3.6 Hz), 127.28 (d, J_{C-P} = 3.4 Hz), 125.44 (d, J_{C-P} = 3.1 Hz), 117.69, 61.98 (d, J_{C-P} = 80.6 Hz), 45.71, 25.14, 20.42.^{31}P NMR (162 MHz, Chloroform-d) δ 32.45. HRMS (ESI) calcd. for $C_{28}H_{26}NNaOP$ $[M+Na]^+$: 446.1644. Found: 446.1629.



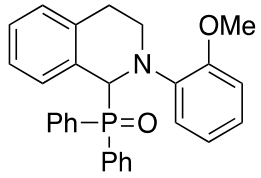
diphenyl(2-(4-(trifluoromethyl)phenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphine oxide (3da)

Reaction was proceeded according to General Method A with 0.1 V *vs*. Ag/AgCl constant potential electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3da**. (white solid, mp 204.7−206.5 °C, yield: 85%).

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3da**. (white solid, mp 204.7−206.5 °C, yield: 95%).

^{1}H NMR (400 MHz, Chloroform-d) δ 7.80 – 7.65 (m, 4H), 7.58 -7.54 (m, 1H), 7.50 – 7.41 (m, 3H), 7.37 – 7.31 (m, 4H), 7.17 (t, J = 7.5 Hz, 1H), 7.10 (d, J = 7.6 Hz, 1H), 6.96 (t, J = 7.5 Hz, 1H), 6.80 (d, J = 8.5 Hz, 2H), 6.66 (d, J = 7.8 Hz, 1H), 5.65 (d, J = 8.6 Hz, 1H), 4.12 – 4.00 (m, 1H), 3.66 – 3.55 (m, 1H), 2.96 – 2.74 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-d) δ 151.77 (d, J_{C-P} = 5.0 Hz), 136.50 (d, J_{C-P} = 4.0 Hz), 132.27 (d, J_{C-P} = 8.7 Hz), 132.21 (d, J_{C-P} = 4.0 Hz), 131.99 (d, J_{C-P} = 2.8 Hz), 131.56 (d, J_{C-P} = 9.0 Hz), 129.82, 129.04 (d, J_{C-P} = 2.4 Hz), 128.53 (d, J_{C-P} = 11.0 Hz), 128.47, 128.42 (d, J_{C-P} = 11.2 Hz), 127.85 (d, J_{C-P} = 3.0 Hz), 127.72 (d, J_{C-P} = 3.4

Hz), 126.33 (d, $J_{C\text{-}P}$ = 3.7 Hz), 125.82 (d, $J_{C\text{-}P}$ = 2.4 Hz), 123.33, 124.67 (d, $J_{C\text{-}F}$ = 271.6 Hz), 114.24, 62.23 (d, $J_{C\text{-}P}$ = 76.9 Hz), 44.29, 26.53. ^{31}P NMR (162 MHz, Chloroform-d) δ 31.83.^{19}F NMR (377 MHz, Chloroform-d) δ -61.35. HRMS (ESI) calcd. for $C_{28}H_{23}F_3NNaOP$ $[M+Na]^+$: 500.1362. Found: 500.1342.

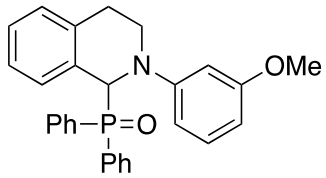


(2-(2-methoxyphenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)diphenylphosphine oxide (3ea)

Reaction was proceeded according to General Method A with 0.1 V *vs*. Ag/AgCl constant potential electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ea**. (white solid, mp 188.5−190.6 °C, yield: 91%).

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ea**. (white solid, mp 188.5−190.6 °C, yield: 84%).

^{1}H NMR (400 MHz, Chloroform-d) δ 7.89 – 7.82 (m, 2H), 7.77 – 7.70 (m, 2H), 7.52 – 7.46 (m, 1H), 7.44 – 7.35 (m, 3H), 7.33 – 7.26 (m, 2H), 7.20 – 7.13 (m, 1H), 7.07 (d, J = 7.7 Hz, 1H), 7.00 (t, J = 7.6 Hz, 1H), 6.96 – 6.85 (m, 2H), 6.79 (d, J = 8.1 Hz, 1H), 6.74 – 6.64 (m, 2H), 5.42 (d, J = 12.4 Hz, 1H), 3.73 – 3.60 (m, 4H), 3.43 – 3.32 (m, 1H), 2.77 – 2.62 (m, 1H), 2.62 – 2.50 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-d) δ 153.86, 141.16 (d, $J_{C\text{-}P}$ = 8.9 Hz), 137.61 (d, $J_{C\text{-}P}$ = 4.6 Hz), 133.70, 132.82 (d, $J_{C\text{-}P}$ = 4.6 Hz), 132.74 (d, $J_{C\text{-}P}$ = 4.6 Hz), 132.09 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.87 (d, $J_{C\text{-}P}$ = 2.7 Hz), 131.12, 129.79 (d, $J_{C\text{-}P}$ = 2.2 Hz), 128.76 (d, $J_{C\text{-}P}$ = 11.1 Hz), 128.53 (d, $J_{C\text{-}P}$ = 11.4 Hz), 128.56, 127.62 (d, $J_{C\text{-}P}$ = 3.1 Hz), 125.97 (d, $J_{C\text{-}P}$ = 2.8 Hz), 124.36, 123.47, 121.71, 113.08, 63.52 (d, $J_{C\text{-}P}$ = 80.9 Hz), 56.17, 46.32, 26.37. ^{31}P NMR (162 MHz, Chloroform-d) δ 31.82. HRMS (ESI) calcd. for $C_{28}H_{26}NNaO_2P$ $[M+Na]^+$: 462.1593. Found: 462.1574.

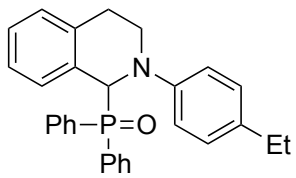


(2-(3-methoxyphenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)diphenylphosphine oxide (3fa)

Reaction was proceeded according to General Method A with 0.1 V *vs*. Ag/AgCl constant potential electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3fa**. (white solid, mp 176.1−178.5 °C, yield: 86%).

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3fa**. (white solid, mp 176.1−178.5 °C, yield: 99%).

^{1}H NMR (400 MHz, Chloroform-d) δ 7.83 – 7.76 (m, 2H), 7.74 – 7.67 (m, 2H), 7.57 – 7.50 (m, 1H), 7.48 – 7.39 (m, 3H), 7.37 – 7.28 (m, 2H), 7.17 – 7.09 (m, 1H), 7.09 – 7.00 (m, 2H), 6.92 (t, *J* = 7.5 Hz, 1H), 6.62 (d, *J* = 7.7 Hz, 1H), 6.44 – 6.39 (m, 1H), 6.35 – 6.29 (m, 2H), 5.55 (d, *J* = 10.4 Hz, 1H), 4.10 – 3.98 (m, 1H), 3.68 (s, 3H), 3.62 – 3.52 (m, 1H), 2.93 – 2.76 (m, 1H), 2.74 – 2.61 (m, 1H).^{13}C NMR (101 MHz, Chloroform-d) δ 160.71, 151.47 (d, $J_{C\text{-}P}$ = 7.5 Hz), 136.91 (d, $J_{C\text{-}P}$ = 4.2 Hz), 132.52 (d, $J_{C\text{-}P}$ = 83.0 Hz), 132.34 (d, $J_{C\text{-}P}$ = 8.3 Hz), 131.96 (d, $J_{C\text{-}P}$ = 2.9 Hz), 131.79 (d, $J_{C\text{-}P}$ = 8.7 Hz), 131.71 (d, $J_{C\text{-}P}$ = 3.2 Hz), 131.66 (d, $J_{C\text{-}P}$ = 91.2 Hz), 130.10, 129.87, 129.28 (d, $J_{C\text{-}P}$ = 2.2 Hz), 128.51 (d, $J_{C\text{-}P}$ = 11.0 Hz), 128.34 (d, $J_{C\text{-}P}$ = 11.3 Hz), 127.89 (d, $J_{C\text{-}P}$ = 3.4 Hz), 127.50 (d, $J_{C\text{-}P}$ = 3.0 Hz), 125.57 (d, $J_{C\text{-}P}$ = 2.7 Hz), 109.49, 104.44, 103.35, 62.28 (d, $J_{C\text{-}P}$ = 78.9 Hz), 55.21, 45.15, 25.91. ^{31}P NMR (162 MHz, Chloroform-d) δ 32.23. HRMS (ESI) calcd. for $C_{28}H_{26}NNaO_2P$ $[M+Na]^+$: 462.1593. Found: 462.1576.



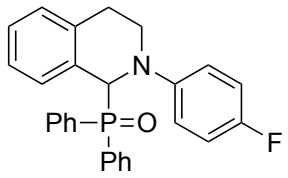
(2-(4-ethylphenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)diphenylphosphine oxide (3ga)

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ga**. (white solid, mp 156.6−158.9 °C, yield: 84%).

^{1}H NMR (400 MHz, Chloroform-d) δ 7.83 – 7.76 (m, 2H), 7.75 – 7.69 (m, 2H), 7.57 – 7.50 (m, 1H), 7.47 – 7.40 (m, 3H), 7.37 – 7.30 (m, 2H), 7.13 (t, *J* = 7.5 Hz, 1H), 7.05 (d, *J* = 7.6 Hz, 1H),

7.01 – 6.90 (m, 3H), 6.74 (d, J = 8.3 Hz, 2H), 6.65 (d, J = 7.8 Hz, 1H), 5.50 (d, J = 11.2 Hz, 1H), 4.07 – 3.94 (m, 1H), 3.59 – 3.46 (m, 1H), 2.88 – 2.75 (m, 1H), 2.66 – 2.58 (m, 1H), 2.52 (q, J = 7.6 Hz, 2H), 1.17 (t, J = 7.6 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-d) δ 148.17 (d, $J_{C\text{-}P}$ = 8.8 Hz), 136.93 (d, $J_{C\text{-}P}$ = 4.3 Hz), 135.75, 132.46 (d, $J_{C\text{-}P}$ = 95.4 Hz), 132.26 (d, $J_{C\text{-}P}$ = 8.4 Hz), 131.82 (d, $J_{C\text{-}P}$ = 2.4 Hz), 131.77 (d, $J_{C\text{-}P}$ = 8.6 Hz), 131.71 (d, $J_{C\text{-}P}$ = 90.9 Hz), 131.57 (d, $J_{C\text{-}P}$ = 2.7 Hz), 129.95, 129.29 (d, $J_{C\text{-}P}$ = 2.2 Hz), 128.45, 128.40 (d, J = 11.0 Hz), 128.24 (d, J = 11.2 Hz), 127.83 (d, $J_{C\text{-}P}$ = 3.3 Hz), 127.29 (d, $J_{C\text{-}P}$ = 2.9 Hz), 125.44 (d, $J_{C\text{-}P}$ = 2.6 Hz), 117.57, 62.13 (d, $J_{C\text{-}P}$ = 80.3 Hz), 45.59, 27.88, 25.28, 15.66. ^{31}P NMR (162 MHz, Chloroform-d) δ 32.30. HRMS (ESI) calcd. for $C_{29}H_{28}NNaOP$ $[M+Na]^+$: 460.1801. Found: 460.1785.

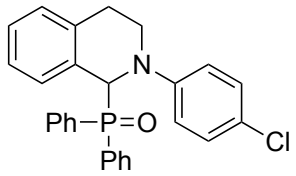


(2-(4-fluorophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)diphenylphosphine oxide (3ha)

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ha**. (white solid, mp 195.3−198.1 °C, yield: 84%).

^{1}H NMR (400 MHz, Chloroform-d) δ 7.81 – 7.65 (m, 4H), 7.56 -7.52 (m, 1H), 7.50 – 7.41 (m, 3H), 7.39 – 7.31 (m, 2H), 7.16 (t, J = 7.6 Hz, 1H), 7.08 (d, J = 7.6 Hz, 1H), 6.94 (t, J = 7.6 Hz, 1H), 6.83 (t, J = 8.5 Hz, 2H), 6.77 – 6.71 (m, 2H), 6.60 (d, J = 7.8 Hz, 1H), 5.40 (d, J = 10.9 Hz, 1H), 4.10 – 3.95 (m, 1H), 3.49 – 3.37 (m, 1H), 2.87 – 2.71 (m, 1H), 2.68 – 2.54 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-d) δ 157.15 (d, $J_{C\text{-}F}$ = 239.3 Hz), 146.83 (dd, J = 9.0, 2.8 Hz), 136.85 (d, $J_{C\text{-}P}$ = 4.5 Hz), 132.26 (d, $J_{C\text{-}P}$ = 95.9 Hz), 132.20 (d, $J_{C\text{-}P}$ = 8.7 Hz), 131.93 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.68 (d, $J_{C\text{-}P}$ = 9.0 Hz), 131.67 (d, $J_{C\text{-}P}$ = 2.7 Hz), 131.42 (d, $J_{C\text{-}P}$ = 91.2 Hz), 129.62, 129.31 (d, $J_{C\text{-}P}$ = 2.5 Hz), 128.43 (d, $J_{C\text{-}P}$ = 11.1 Hz), 128.28 (d, $J_{C\text{-}P}$ = 11.3 Hz), 127.75 (d, $J_{C\text{-}P}$ = 3.7 Hz), 127.44 (d, $J_{C\text{-}P}$ = 3.4 Hz), 125.59 (d, $J_{C\text{-}P}$ = 3.0 Hz), 119.24 (d, $J_{C\text{-}F}$ = 7.8 Hz), 115.60 (d, $J_{C\text{-}F}$ = 22.4 Hz), 62.33 (d, $J_{C\text{-}P}$ = 80.1 Hz), 46.28, 25.28. ^{31}P NMR (162 MHz, Chloroform-d) δ 32.40. ^{19}F NMR (377 MHz, Chloroform-d) δ -124.09. HRMS (ESI) calcd. for $C_{27}H_{23}FNNaOP$

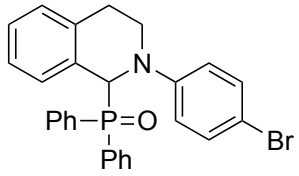
[M+Na]$^+$: 450.1394. Found: 450.1378.



(2-(4-chlorophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)diphenylphosphine oxide (3ia)

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ia**. (white solid, mp 180.2−181.8 °C, yield: 58%).

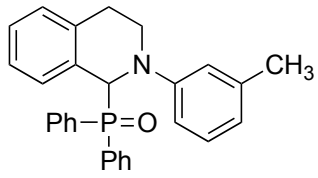
^{1}H NMR (400 MHz, Chloroform-d) δ 7.80 – 7.73 (m, 2H), 7.71 – 7.64 (m, 2H), 7.58 – 7.51 (m, 1H), 7.49 – 7.41 (m, 3H), 7.39 – 7.31 (m, 2H), 7.15 (t, J = 7.5 Hz, 1H), 7.10 – 7.03 (m, 3H), 6.94 (t, J = 7.5 Hz, 1H), 6.73 – 6.67 (m, 2H), 6.63 (d, J = 7.8 Hz, 1H), 5.48 (d, J = 10.1 Hz, 1H), 4.11 – 3.92 (m, 1H), 3.57 – 3.38 (m, 1H), 2.87 – 2.74 (m, 1H), 2.73 – 2.61 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-d) δ 148.56 (d, $J_{C\text{-}P}$ = 7.2 Hz), 136.68 (d, $J_{C\text{-}P}$ = 4.2 Hz), 132.23 (d, $J_{C\text{-}P}$ = 8.5 Hz), 132.06 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.87 (d, $J_{C\text{-}P}$ = 107.2 Hz), 131.82 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.63 (d, $J_{C\text{-}P}$ = 8.8 Hz), 130.95 (d, $J_{C\text{-}P}$ = 102.3 Hz), 129.63, 129.20 (d, $J_{C\text{-}P}$ = 2.3 Hz), 128.97, 128.49 (d, $J_{C\text{-}P}$ = 11.1 Hz), 128.35 (d, $J_{C\text{-}P}$ = 11.3 Hz), 127.76 (d, $J_{C\text{-}P}$ = 3.3 Hz), 127.60 (d, $J_{C\text{-}P}$ = 2.9 Hz), 125.67 (d, $J_{C\text{-}P}$ = 2.6 Hz), 124.30, 117.75, 62.17 (d, $J_{C\text{-}P}$ = 78.9 Hz), 45.25, 25.76. ^{31}P NMR (162 MHz, Chloroform-d) δ 32.34. HRMS (ESI) calcd. for $C_{27}H_{23}ClNNaOP$ [M+Na]$^+$: 466.1098. Found: 466.1082.



(2-(4-bromophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)diphenylphosphine oxide (3ja)

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ja**. (white solid, mp 197.6−200.4 °C, yield: 76%).

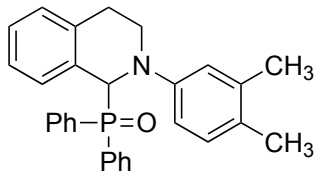
^{1}H NMR (400 MHz, Chloroform-d) δ 7.80 – 7.73 (m, 2H), 7.72 – 7.65 (m, 2H), 7.59 – 7.53 (m, 1H), 7.50 – 7.42 (m, 3H), 7.39 – 7.31 (m, 2H), 7.23 – 7.12 (m, 3H), 7.08 (d, J = 7.6 Hz, 1H), 6.94 (t, J = 7.5 Hz, 1H), 6.69 – 6.56 (m, 3H), 5.48 (d, J = 10.0 Hz, 1H), 4.09 – 3.97 (m, 1H), 3.56 – 3.45 (m, 1H), 2.88 – 2.76 (m, 1H), 2.74 – 2.66 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-d) δ 149.64 (d, J_{C-P} = 7.0 Hz), 137.27 (d, J_{C-P} = 4.2 Hz), 132.85 (d, J_{C-P} = 8.6 Hz), 132.61 (d, J_{C-P} = 2.8 Hz), 132.50, 132.38 (d, J_{C-P} = 2.8 Hz), 132.26 (d, J_{C-P} = 8.8 Hz), 130.37, 129.78 (d, J_{C-P} = 2.2 Hz), 129.08 (d, J_{C-P} = 11.0 Hz), 128.95 (d, J_{C-P} = 11.4 Hz), 128.40 (d, J_{C-P} = 3.5 Hz), 128.21 (d, J_{C-P} = 3.0 Hz), 126.26 (d, J_{C-P} = 2.6 Hz), 118.73, 112.17, 62.86 (d, J_{C-P} = 78.4 Hz), 45.75, 26.49. ^{31}P NMR (162 MHz, Chloroform-d) δ 32.16. HRMS (ESI) calcd. for $C_{27}H_{23}BrNNaOP$ $[M+Na]^+$: 510.0593. Found: 510.0578.



diphenyl(2-(m-tolyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphine oxide (3ka)

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ka**. (white solid, mp 186.2−188.7 °C, yield: 99%).

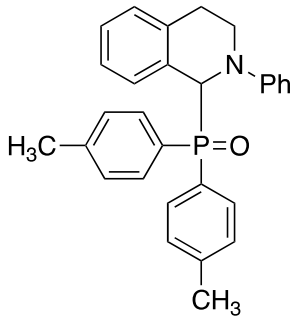
^{1}H NMR (400 MHz, Chloroform-d) δ 7.85 – 7.77 (m, 2H), 7.73 – 7.66 (m, 2H), 7.55 -7.51 (m, 1H), 7.49 – 7.39 (m, 3H), 7.38 – 7.29 (m, 2H), 7.18 – 7.10 (m, 1H), 7.09 – 6.99 (m, 2H), 6.94 (t, J = 7.5 Hz, 1H), 6.68 (d, J = 7.8 Hz, 1H), 6.63 – 6.53 (m, 3H), 5.55 (d, J = 10.6 Hz, 1H), 4.04 – 3.93 (m, 1H), 3.61 – 3.50 (m, 1H), 2.92 – 2.75 (m, 1H), 2.72 – 2.59 (m, 1H), 2.20 (s, 3H).^{13}C NMR (101 MHz, Chloroform-d) δ 150.02 (d, J_{C-P} = 7.6 Hz), 138.78, 136.90 (d, J_{C-P} = 4.2 Hz), 132.35 (d, J_{C-P} = 86.0 Hz), 132.28 (d, J_{C-P} = 8.4 Hz), 131.86 (d, J_{C-P} = 2.9 Hz), 131.74 (d, J_{C-P} = 8.8 Hz), 131.62 (d, J_{C-P} = 2.8 Hz), 130.04, 129.20 (d, J_{C-P} = 2.2 Hz), 128.90, 128.41 (d, J_{C-P} = 11.1 Hz), 128.23 (d, J_{C-P} = 11.3 Hz), 127.82 (d, J_{C-P} = 3.3 Hz), 127.36 (d, J_{C-P} = 3.0 Hz), 125.49 (d, J_{C-P} = 2.6 Hz), 120.44, 117.56, 113.82, 62.12 (d, J_{C-P} = 79.6 Hz), 45.10, 25.70, 21.75. ^{31}P NMR (162 MHz, Chloroform-d) δ 32.19. HRMS (ESI) calcd. for $C_{28}H_{26}NNaOP$ $[M+Na]^+$: 446.1644. Found: 446.1627.



(2-(3,4-dimethylphenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)diphenylphosphine oxide (3la)

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3la**. (white solid, mp 168.9−171.7 °C, yield: 90%).

^{1}H NMR (400 MHz, Chloroform-d) δ 7.85 – 7.77 (m, 2H), 7.74 – 7.66 (m, 2H), 7.55 -7.51 (m, 1H), 7.48 – 7.40 (m, 3H), 7.38 – 7.29 (m, 2H), 7.13 (t, J = 7.5 Hz, 1H), 7.05 (d, J = 7.6 Hz, 1H), 6.98 – 6.86 (m, 2H), 6.67 (d, J = 7.8 Hz, 1H), 6.61 – 6.51 (m, 2H), 5.48 (d, J = 11.4 Hz, 1H), 4.05 – 3.89 (m, 1H), 3.55 – 3.47 (m, 1H), 2.89 – 2.73 (m, 1H), 2.67 – 2.53 (m, 1H), 2.12 (s, 6H). ^{13}C NMR (101 MHz, Chloroform-d) δ 148.34 (d, $J_{C\text{-}P}$ = 8.9 Hz), 137.13, 136.92 (d, $J_{C\text{-}P}$ = 4.4 Hz), 132.89, 132.42 (d, $J_{C\text{-}P}$ = 95.5 Hz), 132.25 (d, $J_{C\text{-}P}$ = 8.3 Hz), 131.80 (d, $J_{C\text{-}P}$ = 98.3 Hz), 131.79 (d, $J_{C\text{-}P}$ = 2.7 Hz), 131.78 (d, $J_{C\text{-}P}$ = 8.8 Hz), 131.55 (d, $J_{C\text{-}P}$ = 2.8 Hz), 130.12, 129.93, 129.28 (d, $J_{C\text{-}P}$ = 2.2 Hz), 128.40 (d, $J_{C\text{-}P}$ = 11.1 Hz), 128.22 (d, $J_{C\text{-}P}$ = 11.1 Hz), 128.09, 127.85 (d, $J_{C\text{-}P}$ = 3.2 Hz), 127.25 (d, $J_{C\text{-}P}$ = 2.9 Hz), 125.41 (d, $J_{C\text{-}P}$ = 2.6 Hz), 119.18, 114.96, 62.09 (d, $J_{C\text{-}P}$ = 80.4 Hz), 45.58, 25.22, 20.18, 18.75. ^{31}P NMR (162 MHz, Chloroform-d) δ 32.44. HRMS (ESI) calcd. for $C_{29}H_{28}NNaOP$ $[M+Na]^{+}$: 460.1801. Found: 460.1785.

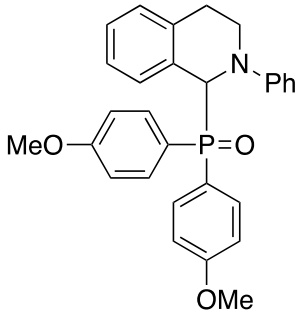


(2-Phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)di-p-tolylphosphine oxide (3ab)

Reaction was proceeded according to General Method A with 0.1 V *vs*. Ag/AgCl constant potential electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ab**. (white solid, mp 191.2−193.6 °C, yield: 85%).

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ab**. (white solid, mp 191.2−193.6 °C, yield: 76%).

^{1}H NMR (400 MHz, Chloroform-d) δ 7.71 – 7.50 (m, 4H), 7.28 – 7.21 (m, 2H), 7.17 – 7.11 (m, 5H), 7.06 (d, J = 7.6 Hz, 1H), 6.95 (t, J = 7.5 Hz, 1H), 6.84 – 6.73 (m, 3H), 6.69 (d, J = 7.8 Hz, 1H), 5.52 (d, J = 11.1 Hz, 1H), 4.08 – 3.91 (m, 1H), 3.68 – 3.49 (m, 1H), 2.93 – 2.76 (m, 1H), 2.69 – 2.57 (m, 1H), 2.41 (s, 3H), 2.33 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 150.06 (d, $J_{C\text{-}P}$ = 8.2 Hz), 142.26 (d, $J_{C\text{-}P}$ = 3.2 Hz), 141.99 (d, $J_{C\text{-}P}$ = 3.2 Hz), 136.79 (d, $J_{C\text{-}P}$ = 4.3 Hz), 132.26 (d, $J_{C\text{-}P}$ = 9.0 Hz), 131.69 (d, $J_{C\text{-}P}$ = 9.3 Hz), 130.21, 129.21, 129.17 (d, $J_{C\text{-}P}$ = 2.2 Hz), 129.09 (d, $J_{C\text{-}P}$ = 2.1 Hz), 129.07, 128.94, 127.91 (d, $J_{C\text{-}P}$ = 3.7 Hz), 127.27 (d, $J_{C\text{-}P}$ = 3.3 Hz), 125.46 (d, $J_{C\text{-}P}$ = 2.9 Hz), 119.34, 116.67, 62.05 (d, $J_{C\text{-}P}$ = 79.8 Hz), 44.96, 25.57, 21.63 (d, $J_{C\text{-}P}$ = 1.4 Hz), 21.55 (d, $J_{C\text{-}P}$ = 1.4 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ 32.70. HRMS (ESI) calcd. for $C_{29}H_{28}NNaOP$ $[M+Na]^{+}$: 460.1801. Found: 460.1782.



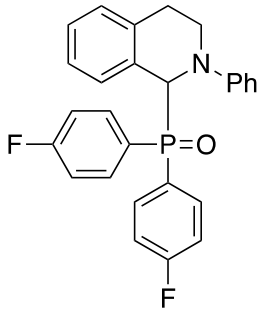
Bis(4-methoxyphenyl)(2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphine oxide (3ac)

Reaction was proceeded according to General Method A with 0.1 V *vs*. Ag/AgCl constant potential electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ac**. (white solid, mp 192.7−194.6 °C, yield: 80%).

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ac**. (white solid, mp 192.7−194.6 °C, yield: 84%).

^{1}H NMR (400 MHz, Chloroform-d) δ 7.75 – 7.64 (m, 2H), 7.63 – 7.48 (m, 2H), 7.17 – 7.09 (m, 3H), 7.06 (d, J = 7.6 Hz, 1H), 7.01 – 6.90 (m, 3H), 6.88 – 6.72 (m, 6H), 5.49 (d, J = 11.6 Hz, 1H),

3.98 – 3.88 (m, 1H), 3.84 (s, 3H), 3.78 (s, 3H), 3.63 – 3.49 (m, 1H), 2.83 (m, 1H), 2.68 – 2.58 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-d) δ 162.45 (d, J_{C-P} = 3.3 Hz), 162.23 (d, J_{C-P} = 3.2 Hz), 150.03 (d, J_{C-P} = 7.8 Hz), 136.75 (d, J_{C-P} = 4.5 Hz), 134.19 (d, J_{C-P} = 9.8 Hz), 133.55 (d, J_{C-P} = 10.4 Hz), 130.36, 129.10, 128.03 (d, J_{C-P} = 3.8 Hz), 127.29 (d, J_{C-P} = 3.5 Hz), 127.05, 125.51 (d, J_{C-P} = 3.1 Hz), 124.27 (d, J_{C-P} = 1.8 Hz), 123.78 (d, J_{C-P} = 101.5 Hz), 122.28 (d, J_{C-P} = 97.9 Hz), 116.47, 114.04, 113.92, 113.80, 62.42 (d, J_{C-P} = 80.8 Hz), 55.33, 55.26, 44.77, 25.71. ^{31}P NMR (162 MHz, Chloroform-d) δ 32.15. HRMS (ESI) calcd. for $C_{29}H_{28}NNaO_3P$ $[M+Na]^+$: 492.1699. Found: 492.1682.



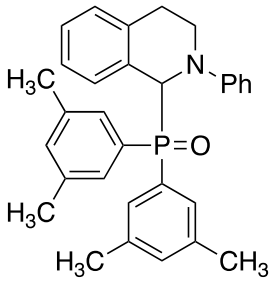
bis(4-fluorophenyl)(2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphine oxide (3ad)

Reaction was proceeded according to General Method A with 0.1 V *vs*. Ag/AgCl constant potential electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ad**. (white solid, mp 208.6−210.0 °C, yield: 72%).

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ad**. (white solid, mp 208.6−210.0 °C, yield: 78%).

^{1}H NMR (400 MHz, Chloroform-d) δ 7.84 – 7.74 (m, 2H), 7.69 – 7.58 (m, 2H), 7.21 – 7.11 (m, 5H), 7.08 (d, *J* = 7.6 Hz, 1H), 7.06 – 6.96 (m, 3H), 6.84 – 6.76 (m, 3H), 6.73 (d, *J* = 7.8 Hz, 1H), 5.50 (d, *J* = 11.0 Hz, 1H), 4.01 – 3.87 (m, 1H), 3.62 – 3.51 (m, 1H), 2.90 – 2.74 (m, 1H), 2.71 – 2.58 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-d) δ 165.22 (dd, *J* = 254.8, 3.5 Hz), 164.99 (dd, *J* = 254.5, 3.3 Hz), 149.86 (d, J_{C-P} = 7.9 Hz), 136.82 (d, J_{C-P} = 4.4 Hz), 134.73 (dd, *J* = 9.2, 9.2Hz), 134.15 (dd, *J* = 10.1, 8.7 Hz), 129.49, 129.32 (d, J_{C-P} = 2.2 Hz), 129.24, 127.95 (d, J_{C-P} = 98.4 Hz), 127.78 (d, J_{C-P} = 3.3 Hz), 127.65 (dd, *J* = 13.6, 3.0 Hz), 126.99 (d, *J* = 96.0 Hz), 125.70 (d, J_{C-P} = 2.6 Hz), 120.00, 117.00, 116.00 (dd, *J* = 21.0, 12.1 Hz), 115.67 (dd, *J* = 21.2, 12.4 Hz), 62.19 (d,

$J_{C\text{-}P}$ = 81.4 Hz), 45.29, 25.60. ^{31}P NMR (162 MHz, Chloroform-d) δ 31.05. ^{19}F NMR (377 MHz, Chloroform-d) δ-106.32, -106.62. HRMS (ESI) calcd. for $C_{27}H_{22}F_2NNaOP$ $[M+Na]^+$: 468.1299. Found: 468.1282.

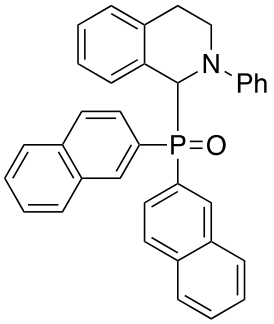


bis(3,5-dimethylphenyl)(2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphine oxide (3ae)

Reaction was proceeded according to General Method A with 0.1 V *vs*. Ag/AgCl constant potential electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ae**. (white solid, mp 179.9−182.2 °C, yield: 84%).

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ae**. (white solid, mp 179.9−182.2 °C, yield: 92%).

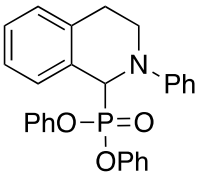
^{1}H NMR (400 MHz, Chloroform-d) δ 7.35 – 7.24 (m, 4H), 7.19 – 7.11 (m, 4H), 7.09 – 7.03 (m, 2H), 6.94 (t, J = 7.5 Hz, 1H), 6.83 (d, J = 8.2 Hz, 2H), 6.77 (t, J = 7.3 Hz, 1H), 6.61 (d, J = 7.8 Hz, 1H), 5.53 (d, J = 10.6 Hz, 1H), 4.10 – 3.96 (m, 1H), 3.63 – 3.47 (m, 1H), 2.88 – 2.74 (m, 1H), 2.70 – 2.58 (m, 1H), 2.29 (s, 6H), 2.20 (s, 6H). ^{13}C NMR (101 MHz, Chloroform-d) δ 150.19 (d, $J_{C\text{-}P}$ = 7.3 Hz), 137.98 (d, $J_{C\text{-}P}$ = 11.7 Hz), 137.76 (d, $J_{C\text{-}P}$ = 11.9 Hz), 137.02 (d, $J_{C\text{-}P}$ = 4.2 Hz), 133.48 (d, $J_{C\text{-}P}$ = 2.9 Hz), 133.27 (d, $J_{C\text{-}P}$ = 2.9 Hz), 131.97 (d, $J_{C\text{-}P}$ = 94.8 Hz), 131.21 (d, $J_{C\text{-}P}$ = 89.9 Hz), 130.25, 129.79 (d, $J_{C\text{-}P}$ = 8.5 Hz), 129.37 (d, $J_{C\text{-}P}$ = 8.8 Hz), 129.08 (d, $J_{C\text{-}P}$ = 2.3 Hz), 129.02, 127.93 (d, $J_{C\text{-}P}$ = 3.3 Hz), 127.27 (d, $J_{C\text{-}P}$ = 2.9 Hz), 125.33 (d, $J_{C\text{-}P}$ = 2.6 Hz), 119.41, 117.00, 61.69 (d, $J_{C\text{-}P}$ = 78.6 Hz), 45.21, 25.84, 21.29, 21.24. ^{31}P NMR (162 MHz, Chloroform-d) δ 33.37. HRMS (ESI) calcd. for $C_{31}H_{32}NNaOP$ $[M+Na]^+$: 488.2114. Found: 488.2097.



di(naphthalen-2-yl)(2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphine oxide (3af)

Reaction was proceeded according to General Method A with 0.1 V *vs*. Ag/AgCl constant potential electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3af**. (white solid, mp 197.4−200.1 °C, yield: 79%).

^{1}H NMR (400 MHz, Chloroform-d) δ 8.47 (d, J = 12.6 Hz, 1H), 8.35 (d, J = 12.7 Hz, 1H), 7.91 – 7.83 (m, 3H), 7.81 – 7.68 (m, 5H), 7.62 – 7.41 (m, 4H), 7.13 (t, J = 7.7 Hz, 3H), 7.06 (d, J = 7.7 Hz, 1H), 6.91 – 6.84 (m, 3H), 6.78 (t, J = 7.3 Hz, 1H), 6.69 (d, J = 7.8 Hz, 1H), 5.78 (d, J = 11.1 Hz, 1H), 4.17 – 4.03 (m, 1H), 3.67 – 3.53 (m, 1H), 2.90 – 2.75 (m, 1H), 2.68 – 2.58 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-d) δ 150.16 (d, $J_{C\text{-}P}$ = 8.5 Hz), 136.89 (d, $J_{C\text{-}P}$ = 4.6 Hz), 134.76, 134.69 (d, $J_{C\text{-}P}$ = 2.3 Hz), 134.60 (d, $J_{C\text{-}P}$ = 2.7 Hz), 133.81 (d, $J_{C\text{-}P}$ = 8.4 Hz), 132.60 (d, $J_{C\text{-}P}$ = 10.7 Hz), 132.47 (d, $J_{C\text{-}P}$ = 10.9 Hz), 129.92, 129.76 (d, $J_{C\text{-}P}$ = 89.9 Hz), 129.40 (d, $J_{C\text{-}P}$ = 2.5 Hz), 129.22, 129.03 (d, $J_{C\text{-}P}$ = 14.8 Hz), 128.29, 128.16 (d, $J_{C\text{-}P}$ = 10.8 Hz), 128.03 (d, $J_{C\text{-}P}$ = 2.5 Hz), 127.89 (d, $J_{C\text{-}P}$ = 4.4 Hz), 127.77 (d, $J_{C\text{-}P}$ = 9.7 Hz), 127.46 (d, $J_{C\text{-}P}$ = 3.3 Hz), 126.94, 126.76 (d, $J_{C\text{-}P}$ = 4.1 Hz), 126.68, 126.66 (d, $J_{C\text{-}P}$ = 4.7 Hz), 125.65 (d, $J_{C\text{-}P}$ = 3.1 Hz), 125.36 (d, $J_{C\text{-}P}$ = 91.0 Hz), 119.90, 117.34, 61.85 (d, $J_{C\text{-}P}$ = 80.1 Hz), 45.53, 25.49. ^{31}P NMR (162 MHz, Chloroform-d) δ 32.89. HRMS (ESI) calcd. for $C_{35}H_{28}NNaOP$ $[M+Na]^{+}$: 532.1801. Found: 532.1783.



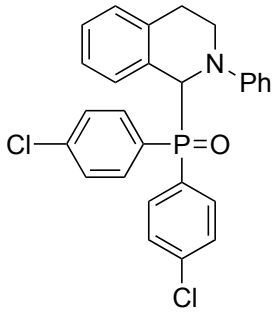
Diphenyl (2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (3ag)

Reaction was proceeded according to General Method A with 0.1 V *vs*. Ag/AgCl constant potential electrolysis . The reaction mixture was purified by silica gel column chromatography to

afford the product **3ag**. (white solid, mp 105.6−108.3 °C, yield: 75%).

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ag**. (white solid, mp 105.6−108.3 °C, yield: 78%).

^{1}H NMR (400 MHz, Chloroform-d) δ 7.53- 7.50 (m, 1H), 7.28- 7.09 (m, 10H), 7.07- 7.00 (m, 5H), 6.89 – 6.79 (m, 3H), 5.59 (d, J = 19.9 Hz, 1H), 4.14 – 3.96 (m, 1H), 3.69- 3.63 (m, 1H), 3.12 – 2.94 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-d) δ 150.82 (d, J_{C-P} = 10.6 Hz), 150.37 (d, J_{C-P} = 11.5 Hz), 149.28 (d, J_{C-P} = 7.0 Hz), 136.77 (d, J_{C-P} = 6.2 Hz), 129.64, 129.57 (d, J_{C-P} = 1.1 Hz) ,129.45, 129.28, 129.05 (d, J_{C-P} = 3.1 Hz), 128.43 (d, J_{C-P} = 5.4 Hz), 127.97 (d, J_{C-P} = 4.2 Hz), 126.24 (d, J_{C-P} = 3.4 Hz), 125.07 (d, J_{C-P} = 1.6 Hz), 124.86 (d, J_{C-P} = 1.3 Hz), 120.67 (d, J_{C-P} = 4.5 Hz), 120.41 (d, J_{C-P} = 4.4 Hz), 119.17, 115.51, 59.19 (d, J_{C-P} = 160.6 Hz), 44.03, 26.65. ^{31}P NMR (162 MHz, Chloroform-d) δ 16.37. HRMS (ESI) calcd. for $C_{27}H_{24}NNaO_3P$ $[M+Na]^+$: 464.1386. Found: 464.1371.

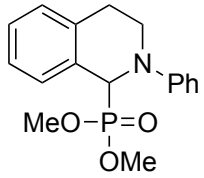


Bis(4-chlorophenyl)(2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphine oxide (3ah)

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ah**. (white solid, mp 196.2−199.4 °C, yield: 48%).

^{1}H NMR (400 MHz, Chloroform-d) δ 7.73 - 7.68 (m, 2H), 7.59 - 7.55 (m, 2H), 7.47 – 7.41 (m, 2H), 7.36 – 7.29 (m, 2H), 7.21 – 7.13 (m, 3H), 7.09 (d, J = 7.6 Hz, 1H), 7.01 (t, J = 7.5 Hz, 1H), 6.86 – 6.77 (m, 3H), 6.72 (d, J = 7.7 Hz, 1H), 5.50 (d, J = 11.0 Hz, 1H), 4.00 – 3.87 (m, 1H), 3.62 – 3.51 (m, 1H), 2.90 – 2.75 (m, 1H), 2.70 – 2.59 (m, 1H).^{13}C NMR (101 MHz, Chloroform-d) δ 149.87 (d, J_{C-P} = 8.5 Hz), 138.85 (d, J_{C-P} = 3.8 Hz), 138.52 (d, J_{C-P} = 3.7 Hz), 136.80 (d, J_{C-P} = 4.8

Hz), 133.54 (d, $J_{C\text{-}P}$ = 9.4 Hz), 133.01 (d, $J_{C\text{-}P}$ = 9.9 Hz), 130.43 (d, $J_{C\text{-}P}$ = 96.3 Hz), 129.64 (d, $J_{C\text{-}P}$ = 103.1 Hz), 129.43 (d, $J_{C\text{-}P}$ = 2.7 Hz), 129.29, 129.25, 128.93 (d, $J_{C\text{-}P}$ = 11.7 Hz), 128.72 (d, $J_{C\text{-}P}$ = 11.9 Hz), 127.70 (d, $J_{C\text{-}P}$ = 3.9 Hz), 125.76 (d, $J_{C\text{-}P}$ = 3.1 Hz), 120.22, 117.28, 61.99 (d, $J_{C\text{-}P}$ = 81.1 Hz), 45.49, 25.47. ^{31}P NMR (162 MHz, Chloroform-d) δ 31.22. HRMS (ESI) calcd. for $C_{27}H_{22}C_{l2}NNaOP$ $[M+Na]^+$: 500.0708. Found: 500.0691.

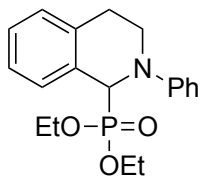


dimethyl (2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (3ai)

Reaction was proceeded according to General Method A with 0.1 V *vs*. Ag/AgCl constant potential electrolysis. The reaction mixture was purified by silica gel column chromatography to afford the product **3ai**. (white solid, mp 85.9−87.9 °C, yield: 81%).

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ai**. (white solid, mp 85.9−87.9 °C, yield: 89%).

^{1}H NMR (400 MHz, Chloroform-d) δ 7.38 – 7.32 (m, 1H), 7.29 – 7.23 (m, 2H), 7.21 – 7.12 (m, 3H), 6.97 (d, *J* = 8.3 Hz, 2H), 6.80 (t, *J* = 7.3 Hz, 1H), 5.20 (d, *J* = 19.9 Hz, 1H), 4.09 – 3.94 (m, 1H), 3.79 – 3.53 (m, 7H), 3.17 – 2.86 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-d) δ 149.27 (d, $J_{C\text{-}P}$ = 6.2 Hz), 136.44 (d, $J_{C\text{-}P}$ = 5.9 Hz), 130.43, 129.27, 128.86 (d, $J_{C\text{-}P}$ = 3.0 Hz), 127.97 (d, $J_{C\text{-}P}$ = 4.8 Hz), 127.57 (d, $J_{C\text{-}P}$ = 4.0 Hz), 126.08 (d, $J_{C\text{-}P}$ = 3.2 Hz), 118.71, 114.81, 58.78 (d, $J_{C\text{-}P}$ = 159.4 Hz), 53.95 (d, $J_{C\text{-}P}$ = 7.4 Hz), 52.97 (d, $J_{C\text{-}P}$ = 7.9 Hz), 43.59, 26.71. ^{31}P NMR (162 MHz, Chloroform-d) δ 26.01. HRMS (ESI) calcd. for $C_{17}H_{20}NNaO_3P$ $[M+Na]^+$: 340.1073. Found: 340.1060.

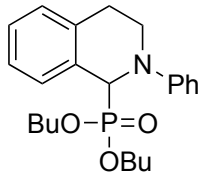


diethyl (2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (3aj)

Reaction was proceeded according to General Method A with 0.1 V *vs*. Ag/AgCl constant potential electrolysis. The reaction mixture was purified by silica gel column chromatography to afford the product **3aj**. (white solid, mp 61.4−63.8 °C, yield: 70%).

Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3aj**. (white solid, mp 61.4−63.8 °C, yield: 62%).

^{1}H NMR (400 MHz, Chloroform-d) δ 7.41 – 7.34 (m, 1H), 7.27 – 7.21 (m, 2H), 7.20 – 7.12 (m, 3H), 6.97 (d, J = 8.3 Hz, 2H), 6.79 (t, J = 7.2 Hz, 1H), 5.18 (d, J = 20.0 Hz, 1H), 4.14 – 3.93 (m, 4H), 3.93 – 3.82 (m, 1H), 3.65-3.59 (m, 1H), 3.15 – 2.91 (m, 2H), 1.24 (t, J = 7.1 Hz, 3H), 1.13 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 149.42 (d, J_{C-P} = 6.1 Hz), 136.46 (d, J_{C-P} = 5.8 Hz), 130.69, 129.14, 128.75 (d, J_{C-P} = 3.0 Hz), 128.14 (d, J_{C-P} = 5.1 Hz), 127.43 (d, J_{C-P} = 3.8 Hz), 125.87 (d, J_{C-P} = 2.9 Hz), 118.47, 114.81, 63.32 (d, J_{C-P} = 7.7 Hz), 62.34 (d, J_{C-P} = 8.1 Hz), 58.83 (d, J_{C-P} = 159.2 Hz), 43.50, 26.79, 16.47 (d, J_{C-P} = 5.6 Hz), 16.38 (d, J_{C-P} = 6.0 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ 23.79. HRMS (ESI) calcd. for $C_{19}H_{24}NNaO_3P$ $[M+Na]^+$: 368.1386. Found: 368.1373.



Dibutyl (2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (3ak)

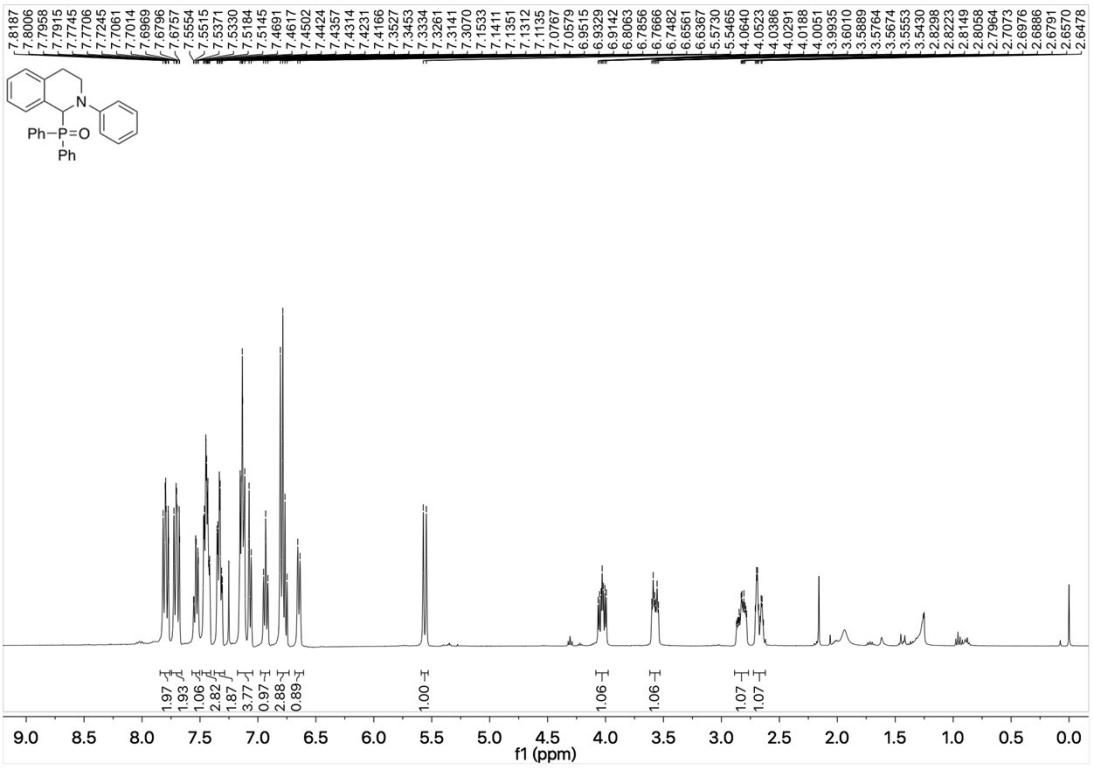
Reaction was proceeded according to General Method A with 0.1 V *vs*. Ag/AgCl constant potential electrolysis. The reaction mixture was purified by silica gel column chromatography to afford the product **3ak**. (white solid, mp 46.3−49.0 °C, yield: 64%).

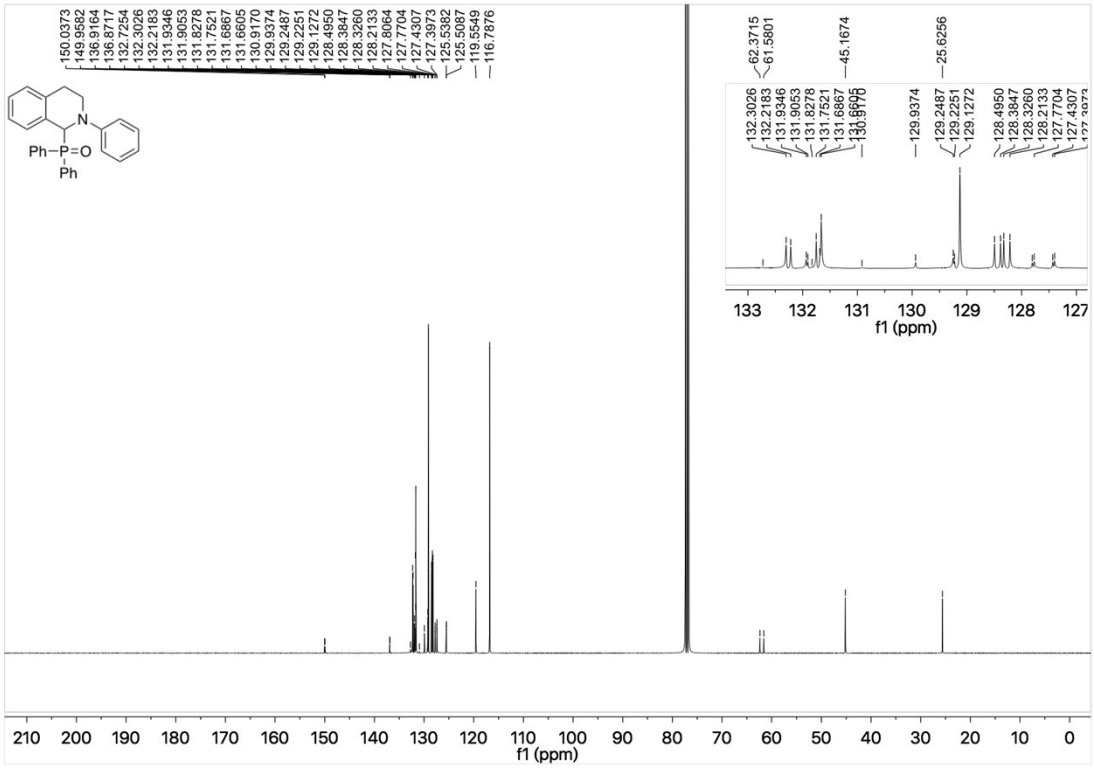
Reaction was proceeded according to General Method B with 10 mA constant current electrolysis . The reaction mixture was purified by silica gel column chromatography to afford the product **3ak**. (white solid, mp 46.3−49.0 °C, yield: 43%).

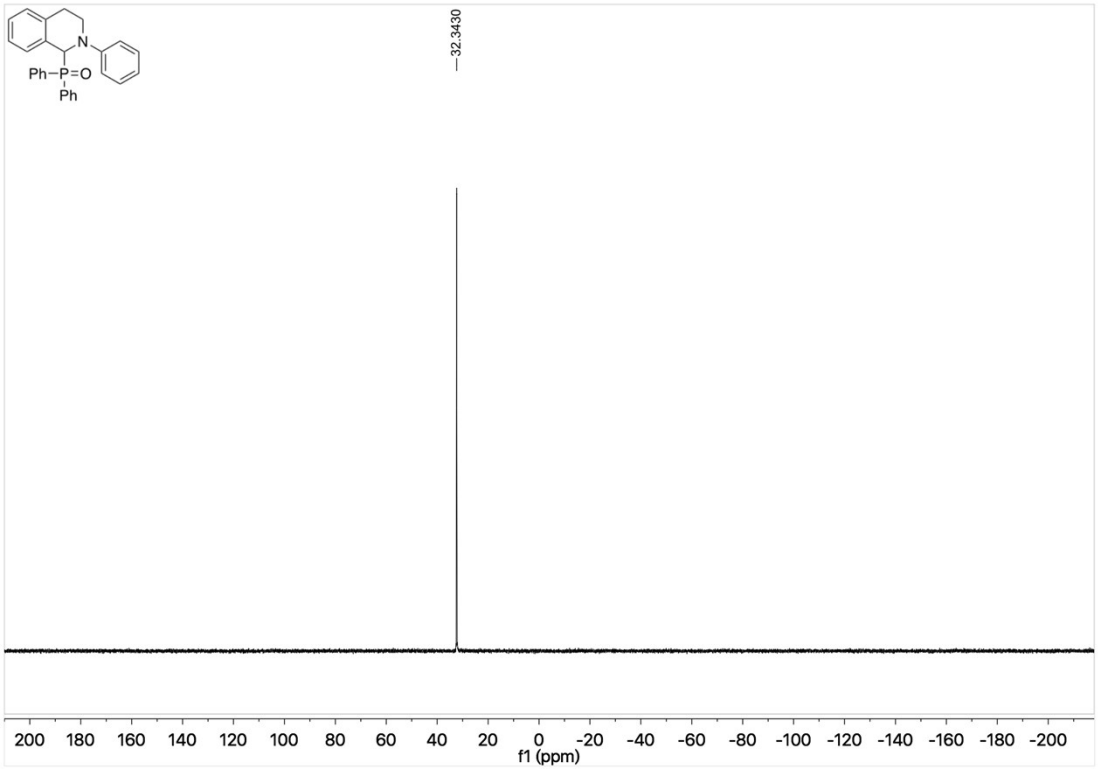
^{1}H NMR (400 MHz, Chloroform-d) δ 7.40 – 7.34 (m, 1H), 7.27 – 7.21 (m, 2H), 7.21 – 7.11 (m, 3H), 6.97 (d, J = 8.3 Hz, 2H), 6.78 (t, J = 7.2 Hz, 1H), 5.20 (d, J = 19.9 Hz, 1H), 4.09 – 3.74 (m,

5H), 3.66-3.60 (m, 1H), 3.16 – 2.89 (m, 2H), 1.62 – 1.51 (m, 2H), 1.49 – 1.40 (m, 2H), 1.39 – 1.16 (m, 4H), 0.88 (t, J = 7.3 Hz, 3H), 0.81 (t, J = 7.4 Hz, 3H).^{13}C NMR (101 MHz, Chloroform-d) δ 149.36 (d, $J_{C\text{-}P}$ = 5.8 Hz), 136.41 (d, $J_{C\text{-}P}$ = 5.9 Hz), 130.79, 129.12, 128.73 (d, $J_{C\text{-}P}$ = 3.0 Hz), 128.13 (d, $J_{C\text{-}P}$ = 5.0 Hz), 127.40 (d, $J_{C\text{-}P}$ = 3.9 Hz), 125.85 (d, $J_{C\text{-}P}$ = 3.3 Hz), 118.41, 114.77, 66.92 (d, $J_{C\text{-}P}$ = 7.8 Hz), 65.98 (d, $J_{C\text{-}P}$ = 8.5 Hz), 58.75 (d, $J_{C\text{-}P}$ = 158.5 Hz), 43.46, 32.58 (dd, $J_{C\text{-}P}$ = 6.2, 6.4 Hz), 26.84, 18.68 (d, $J_{C\text{-}P}$ = 6.6 Hz), 13.57 (d, $J_{C\text{-}P}$ = 4.5 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ 23.93. HRMS (ESI) calcd. for $C_{23}H_{32}NNaO_3P$ $[M+Na]^+$: 424.2012. Found: 424.1997.

5. ^{1}H and ^{13}C spectra of products



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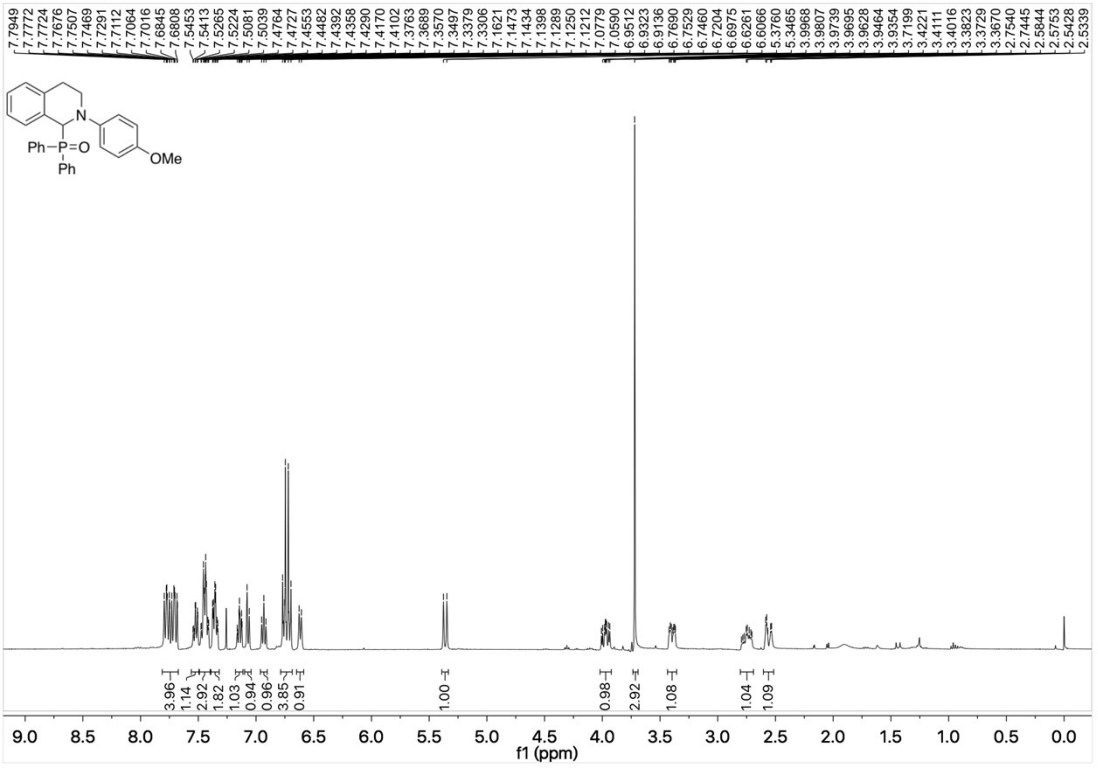
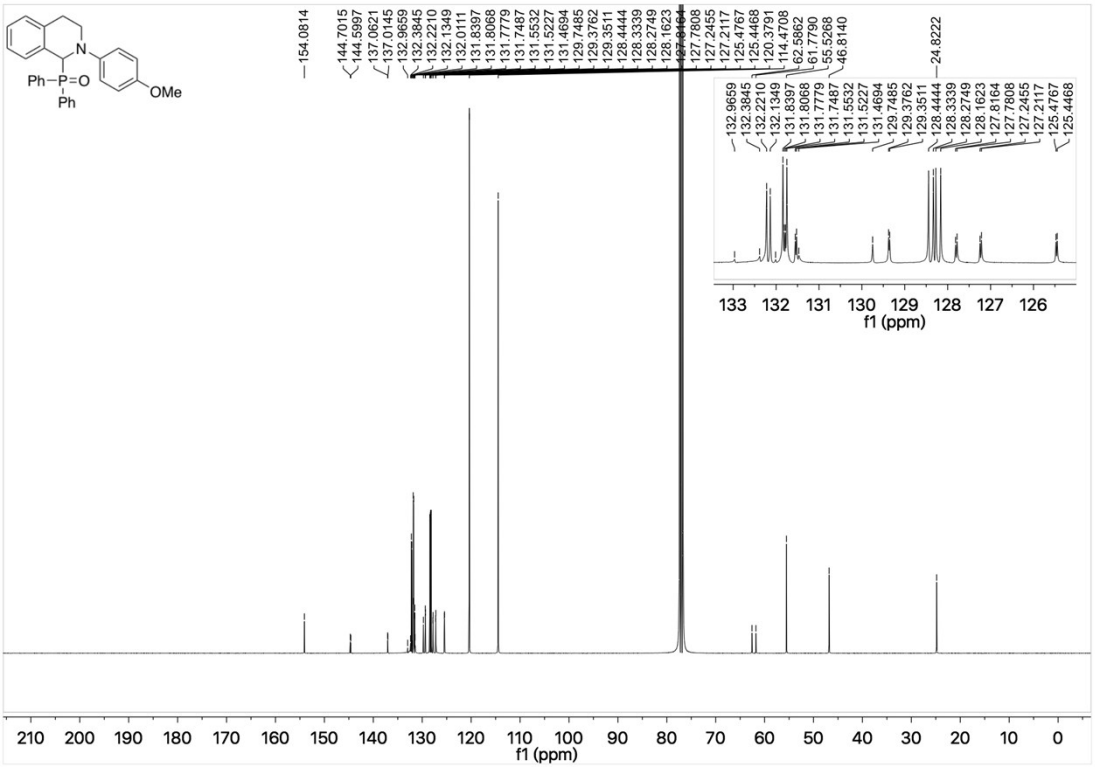
f1 (ppm)


Ph–P=O
Ph
OMe
3.96
1.14
2.92
1.82
1.03
0.94
0.96
3.85
0.91
1.00
0.98
2.92
1.08
1.04
1.09
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)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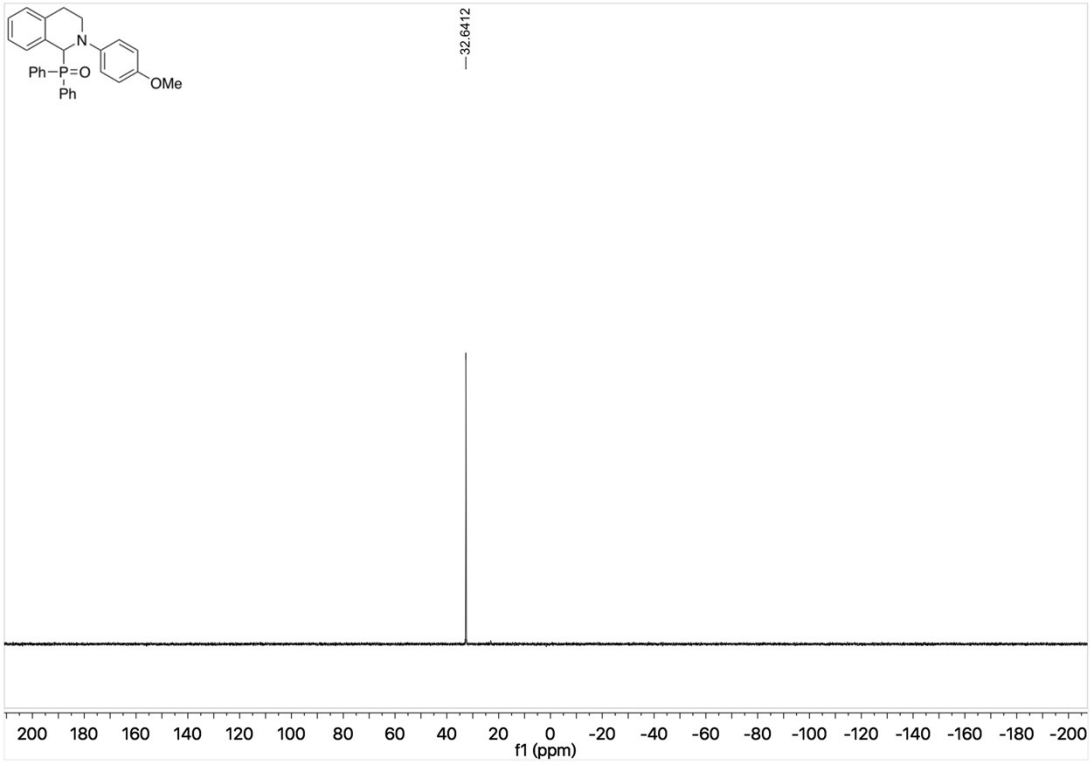
f1 (ppm)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Ph
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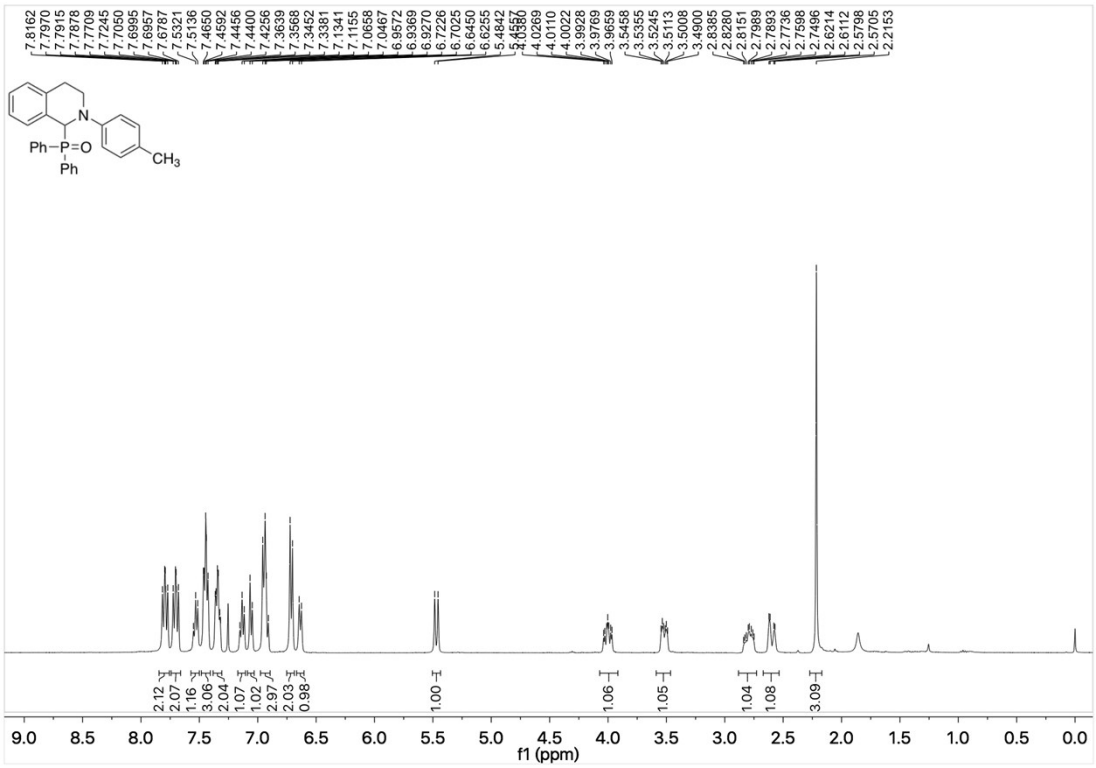
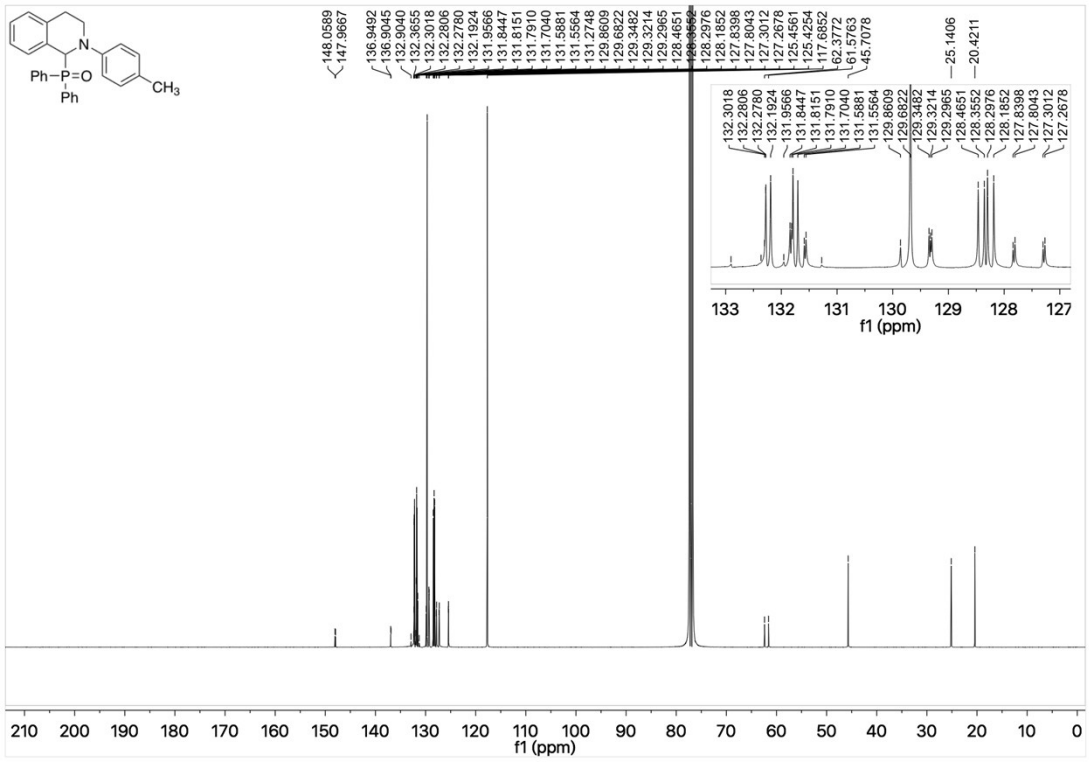
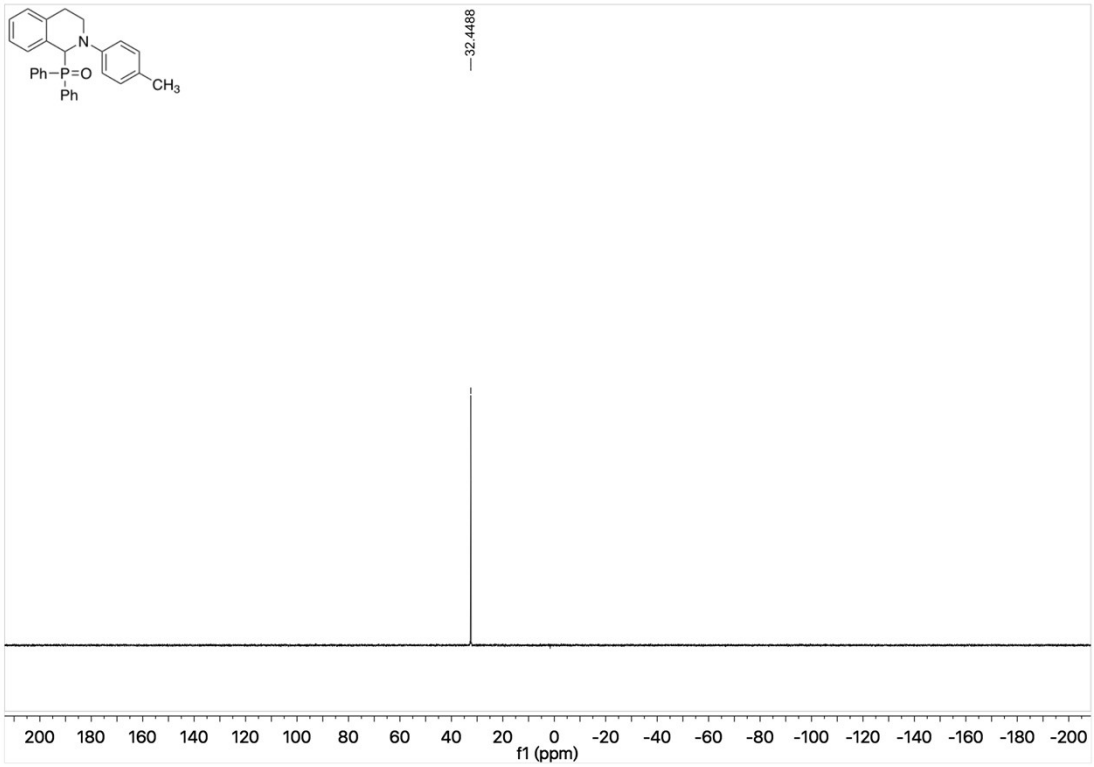
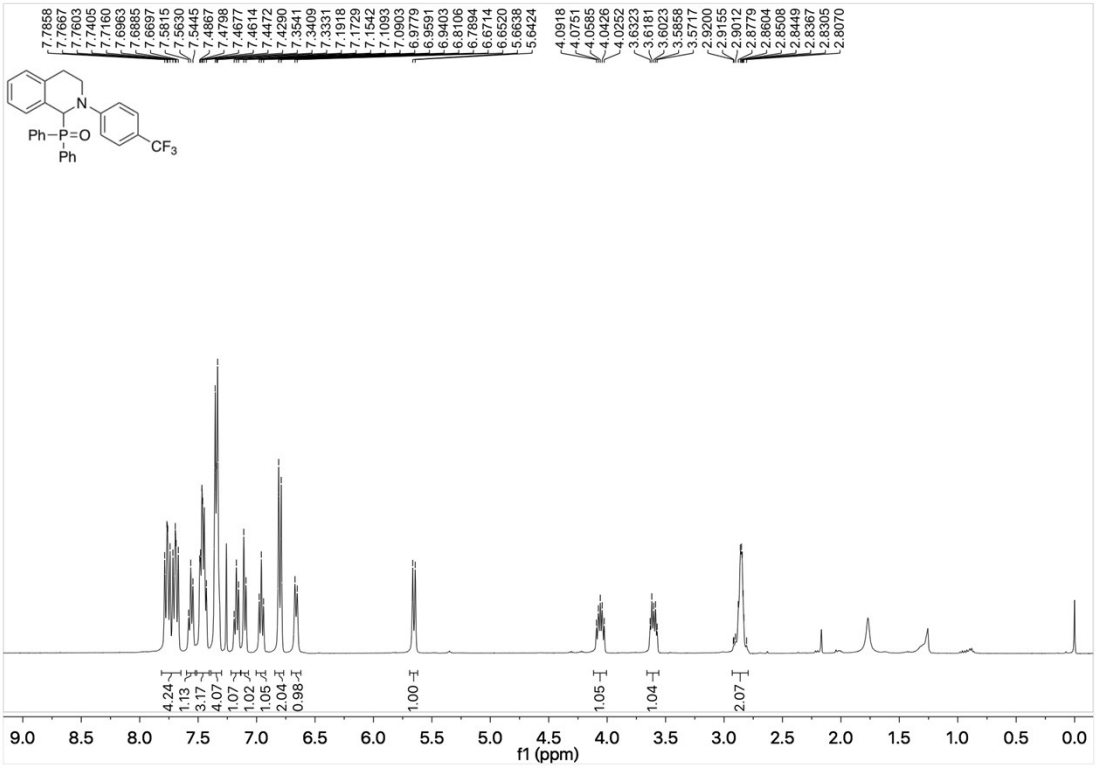
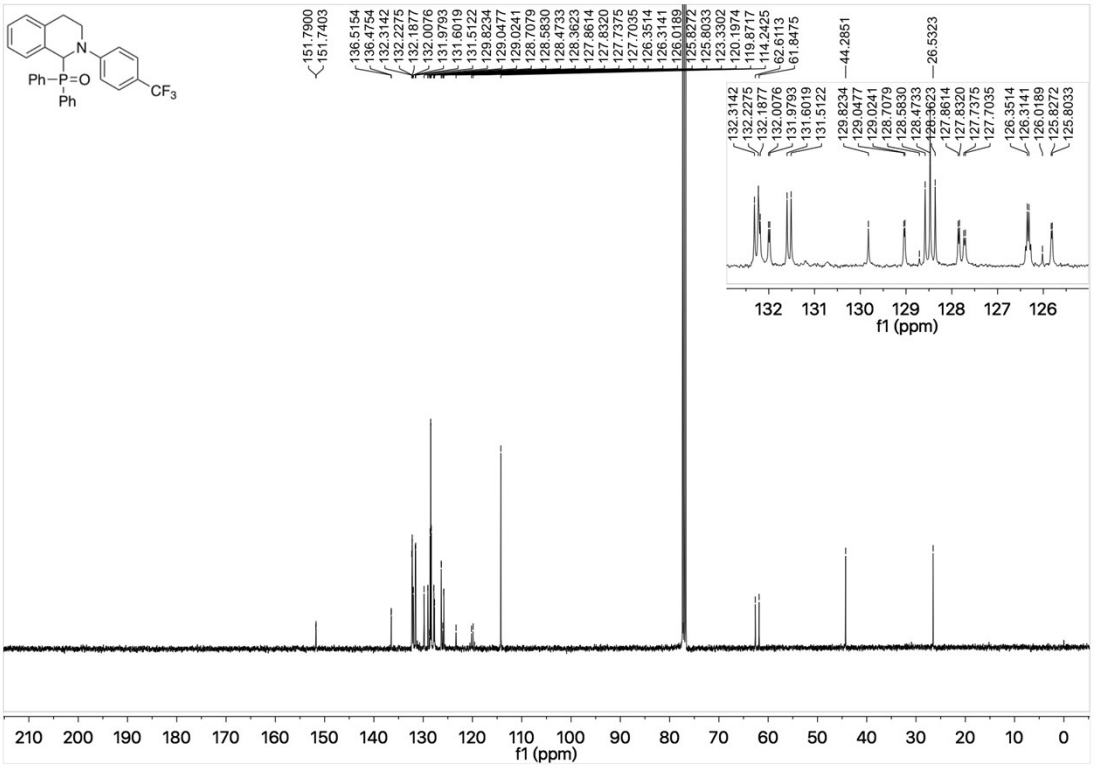
f1 (ppm)

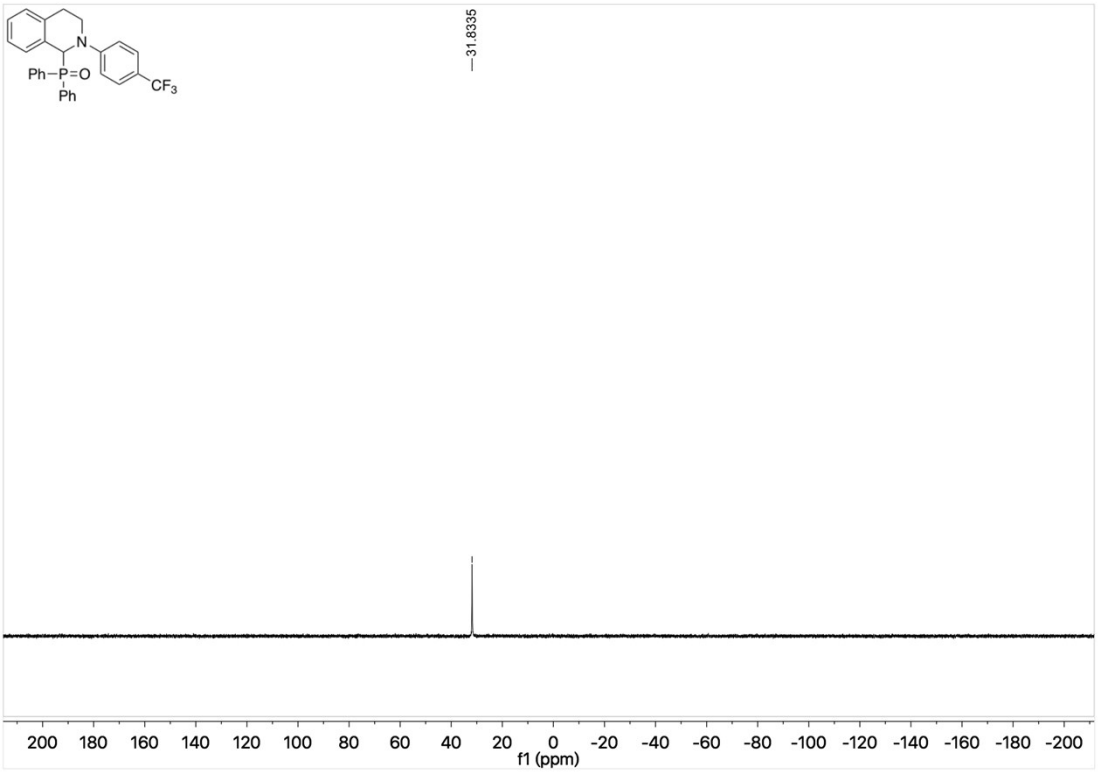

f1 (ppm)

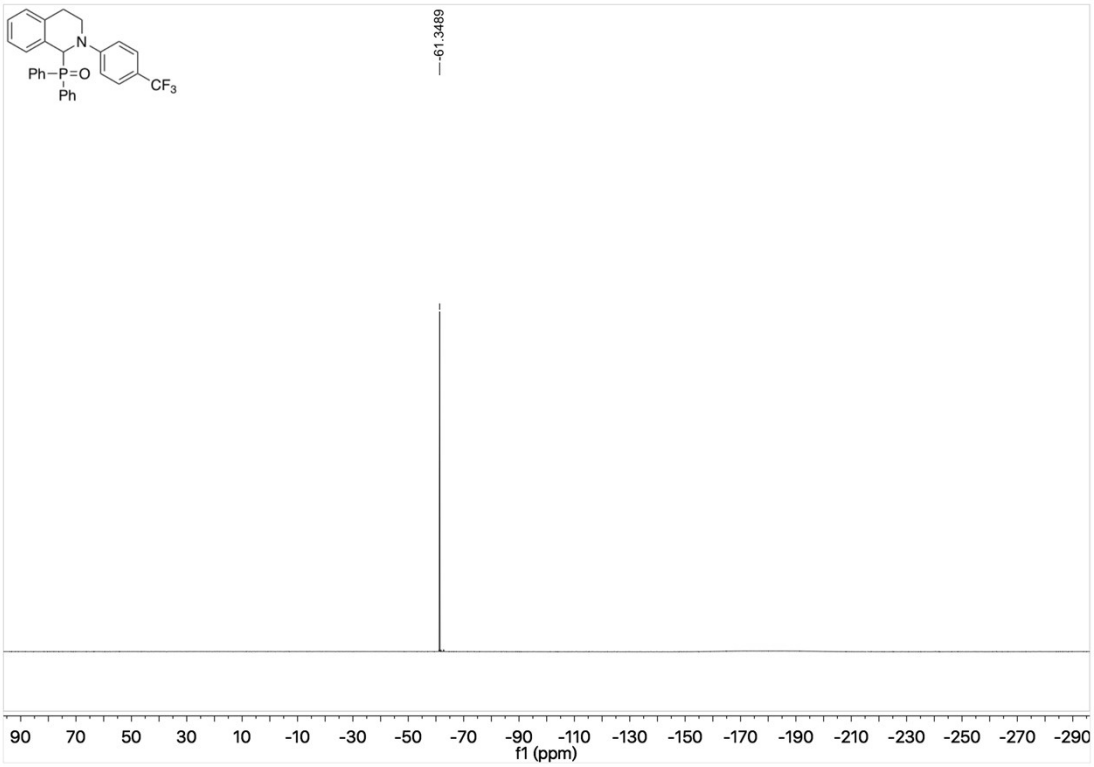

f1 (ppm)

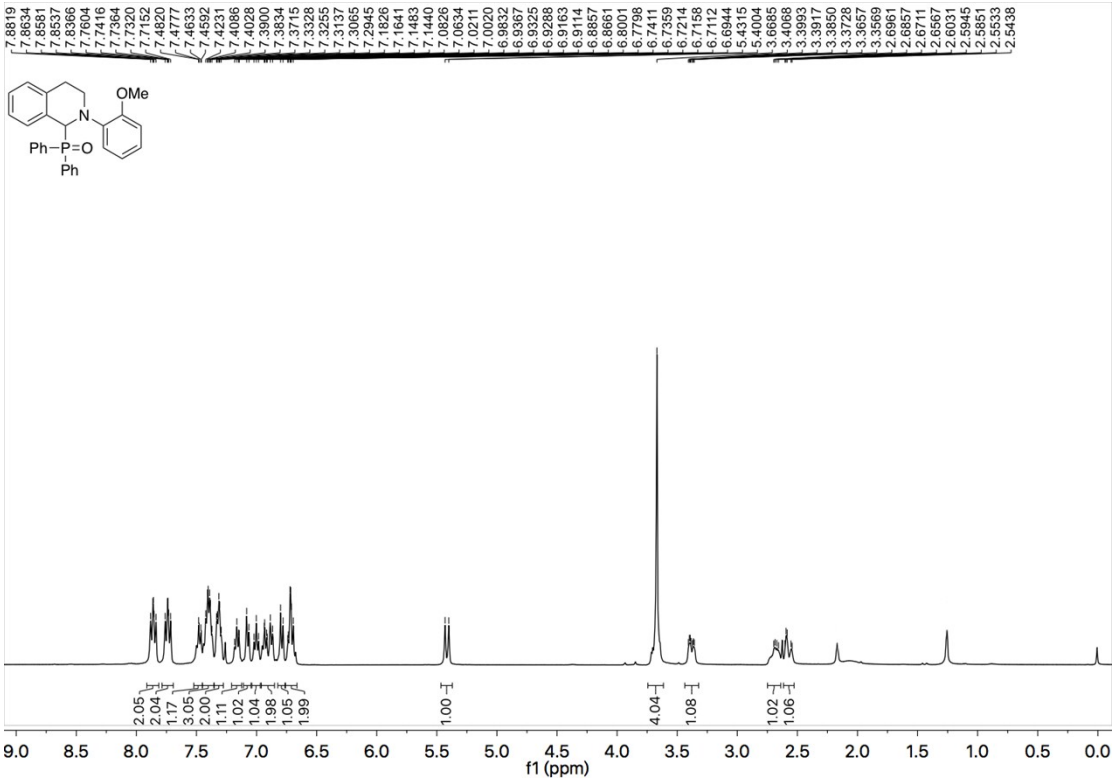

32.4488
Ph–P=O
Ph
CH3
200 180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200
f1 (ppm)

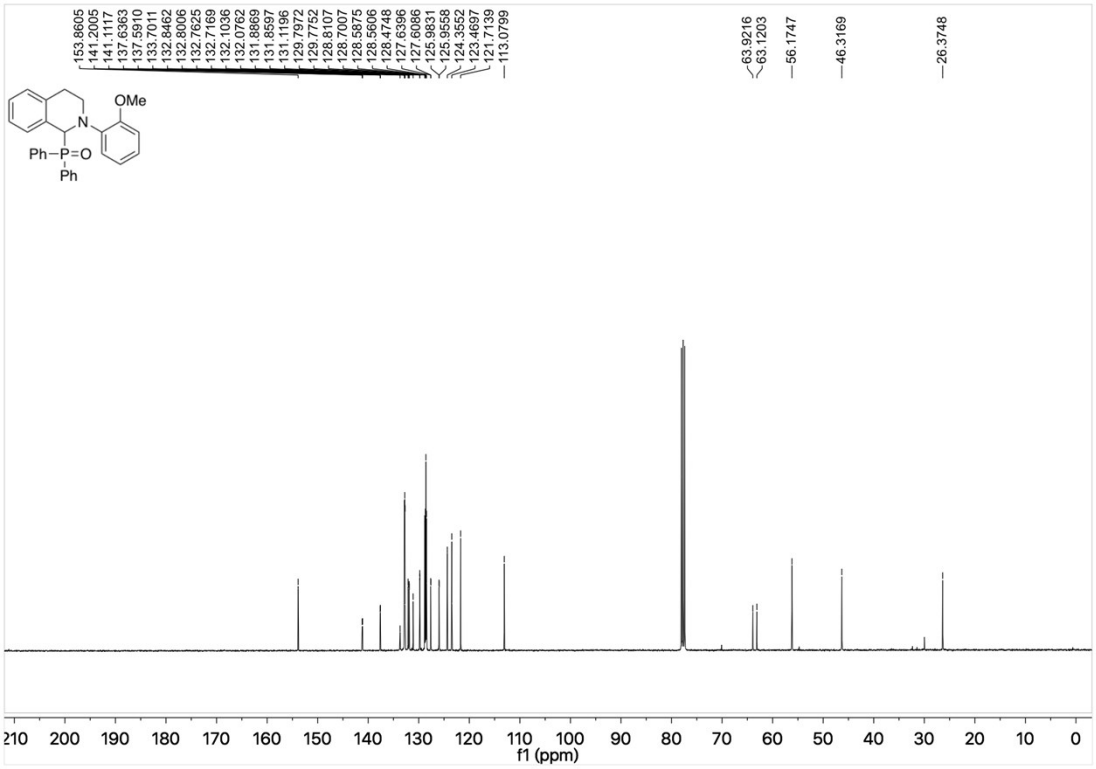

7.7858 7.7667 7.7603 7.7405 7.7160 7.6963 7.6885 7.6697 7.5815 7.5630 7.5445 7.4867 7.4798 7.4677 7.4614 7.4472 7.4290 7.3541 7.3409 7.3331 7.1918 7.1729 7.1542 7.1093 7.0903 6.9779 6.9591 6.9403 6.8106 6.7894 6.6714 6.6520 5.6638 5.6424
4.0918 4.0751 4.0585 4.0426 4.0252 3.6323 3.6181 3.6023 3.5858 3.5717
2.9200 2.9155 2.9012 2.8779 2.8604 2.8508 2.8449 2.8367 2.8305 2.8070
Ph–P=O
Ph
CF3
4.24 1.13 3.17 4.07 1.07 1.02 1.05 2.04 0.98 1.00 1.05 1.04 2.07
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)

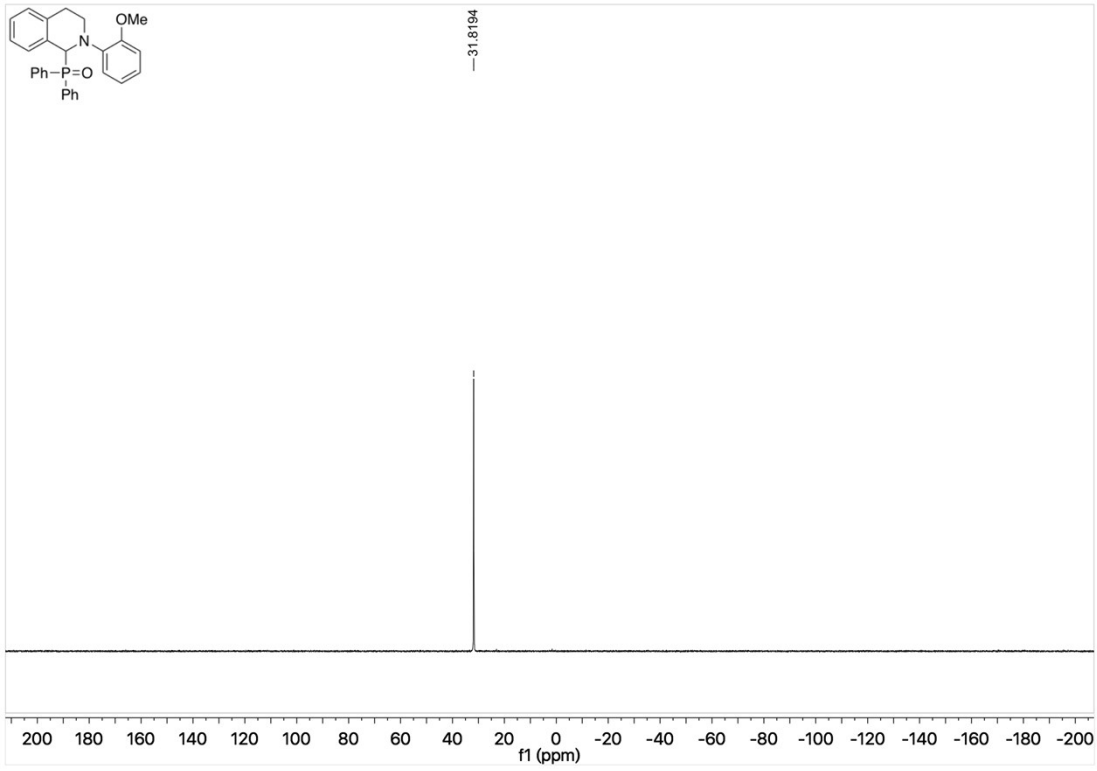

Ph–P=O
Ph
CF3
151.7900
151.7403
136.5154
136.4754
132.3142
132.2275
132.1877
132.0076
131.9793
131.6019
131.5122
129.8234
129.0477
129.0241
128.7079
128.5830
128.4733
128.3623
127.8614
127.8320
127.7375
127.7035
126.3514
126.3141
126.0189
125.8272
125.8033
123.3302
120.1974
119.8717
114.2425
62.6113
61.8475
44.2851
26.5323
f1 (ppm)

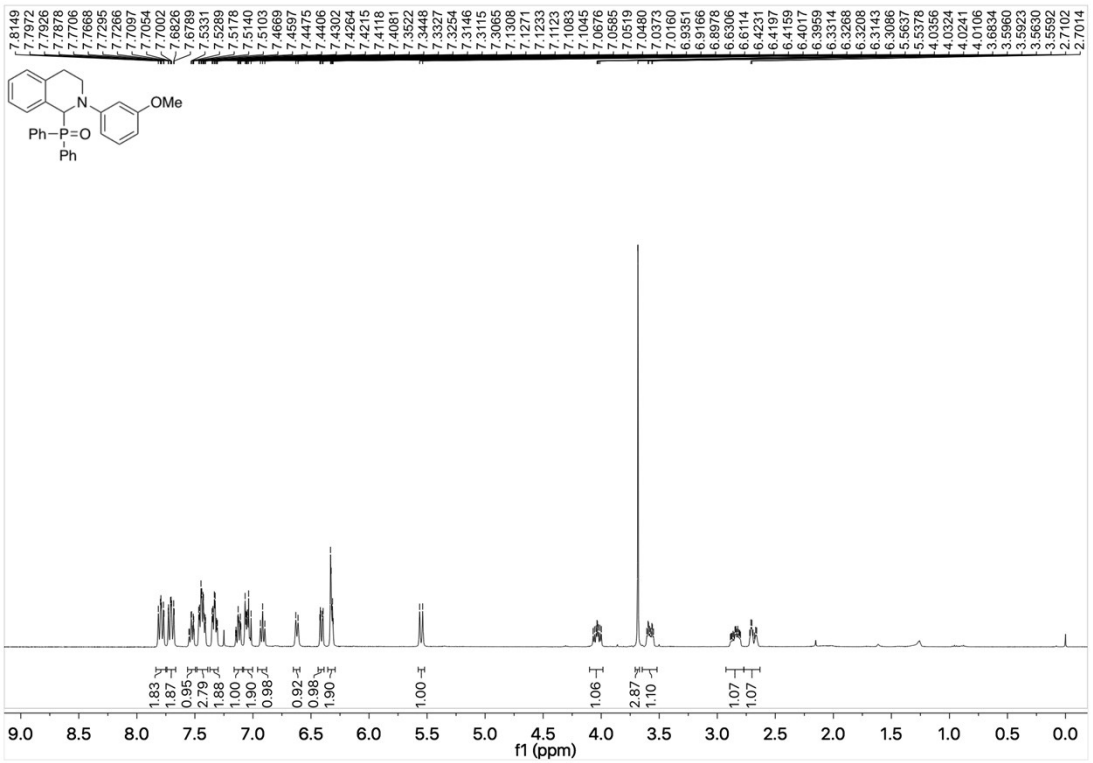

Ph–P=O
Ph
CF3
31.8335
f1 (ppm)

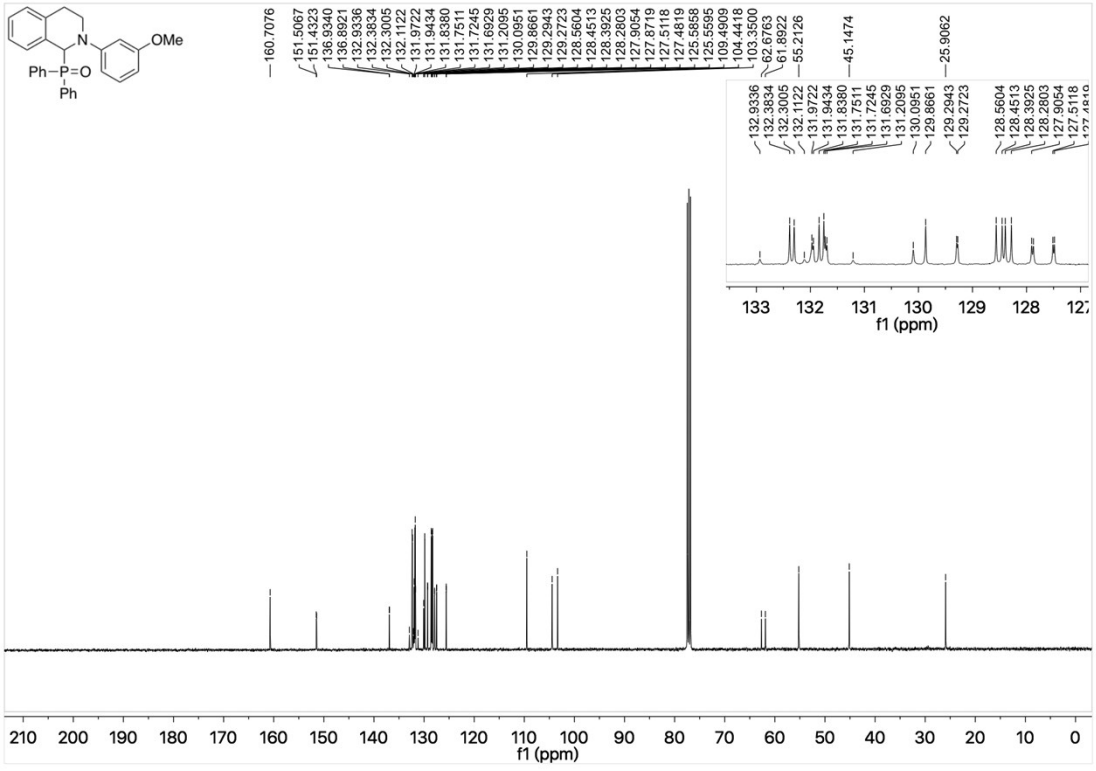

Ph–P=O
Ph
N
CF3
-61.3489
90 70 50 30 10 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230 -250 -270 -290
f1 (ppm)

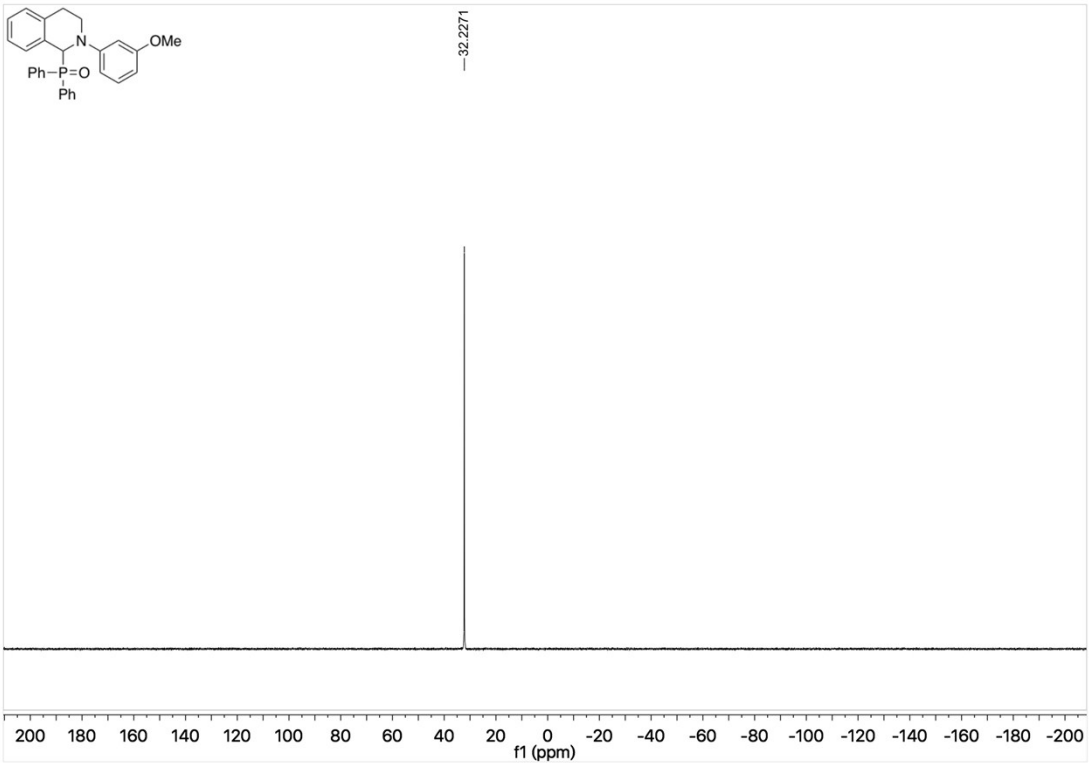

OMe
Ph–P=O
Ph
2.05 2.04 1.17 3.05 2.00 1.11 1.02 1.04 1.98 1.05 1.99
1.00
4.04
1.08
1.02 1.06
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

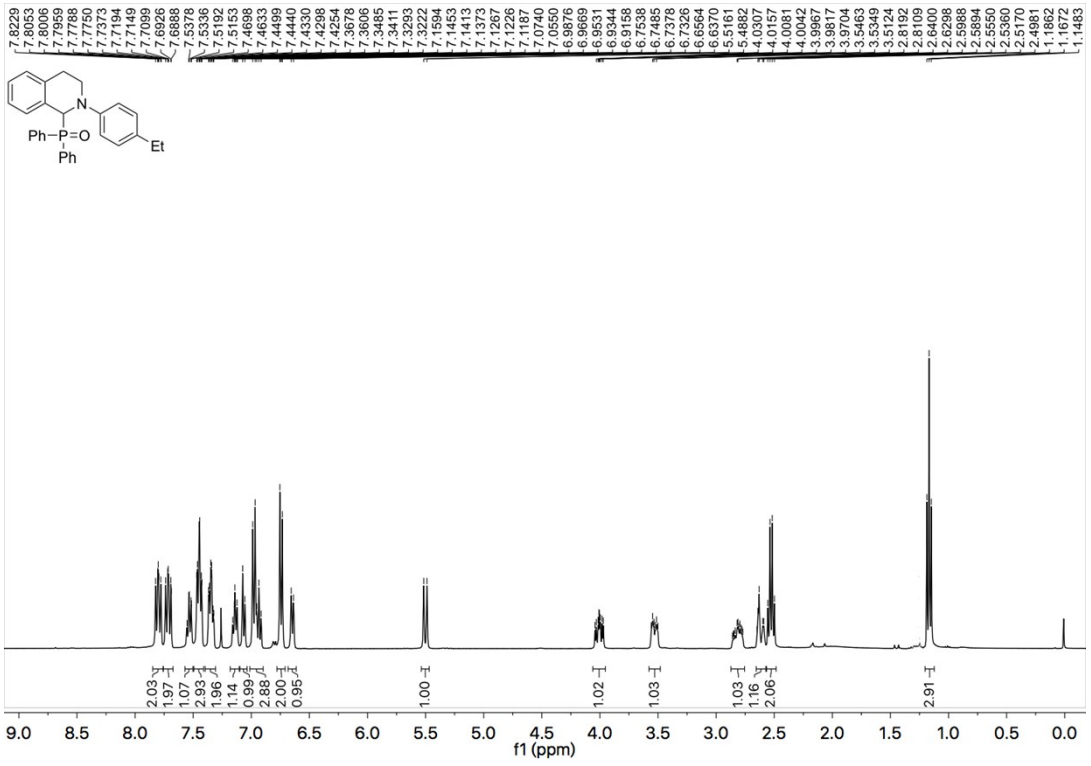

153.8605
141.2005
141.1117
137.6363
137.5910
133.7011
132.8462
132.8006
132.7625
132.7169
132.1036
132.0762
131.8869
131.8597
131.1196
129.7972
129.7752
128.8107
128.7007
128.5875
128.5606
128.4748
127.6396
127.6086
125.9831
125.9558
124.3552
123.4697
121.7139
113.0799
63.9216
63.1203
56.1747
46.3169
26.3748
OMe
N
Ph–P=O
Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

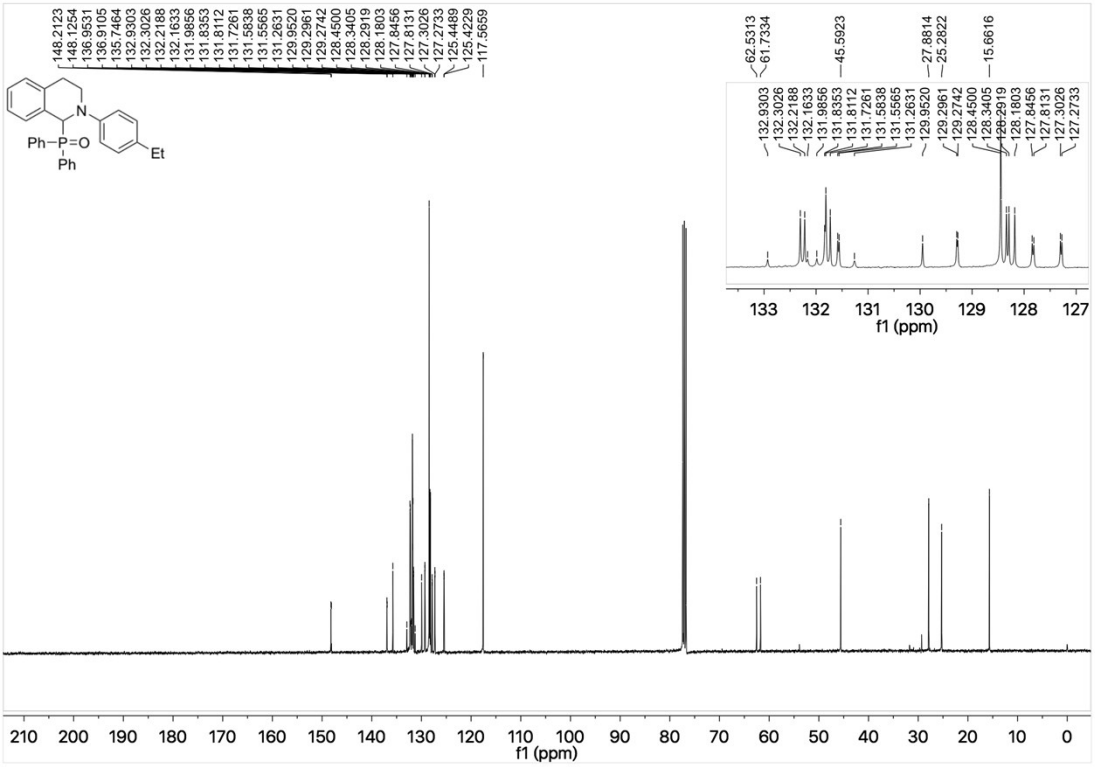

31.8194
OMe
N
Ph–P=O
Ph
200 180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200
f1 (ppm)


OMe
Ph–P=O
Ph
f1 (ppm)


OMe
Ph–P=O
Ph
f1 (ppm)


OMe
Ph–P=O
Ph
32.2271
200 180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200
f1 (ppm)


Ph–P=O
Ph
Et
2.03
1.97
1.07
2.93
1.96
1.14
0.99
2.88
2.00
0.95
1.00
1.02
1.03
1.03
1.16
2.06
2.91
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)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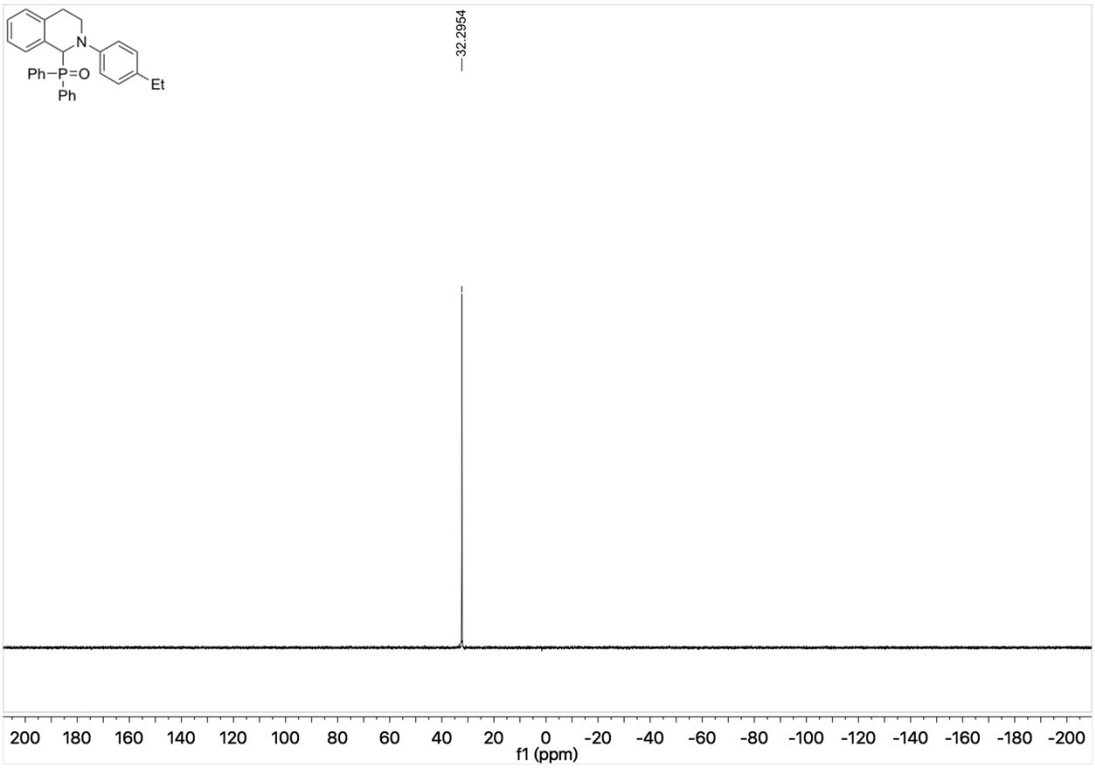
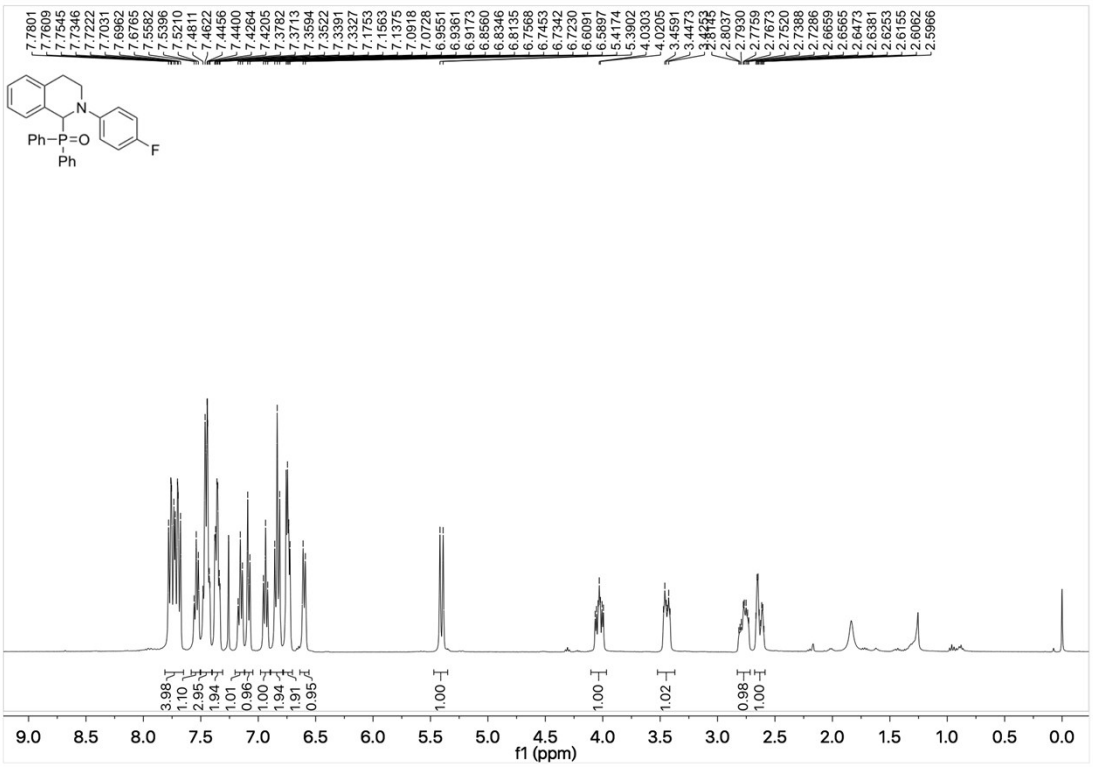
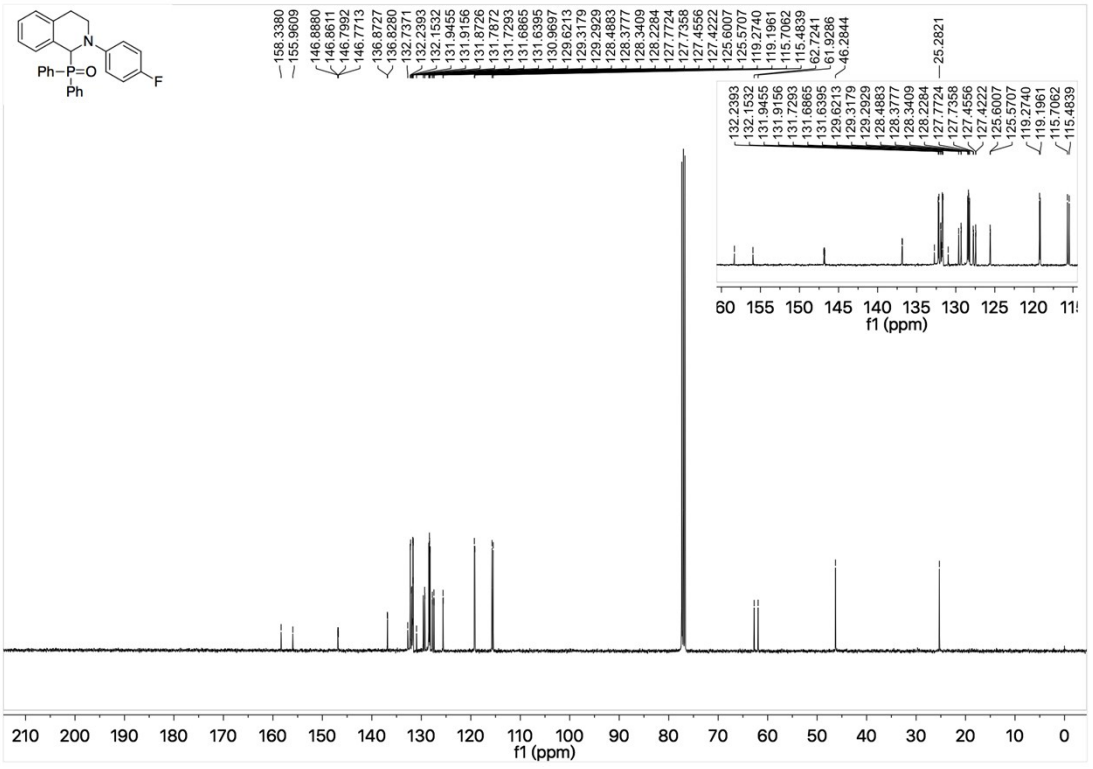
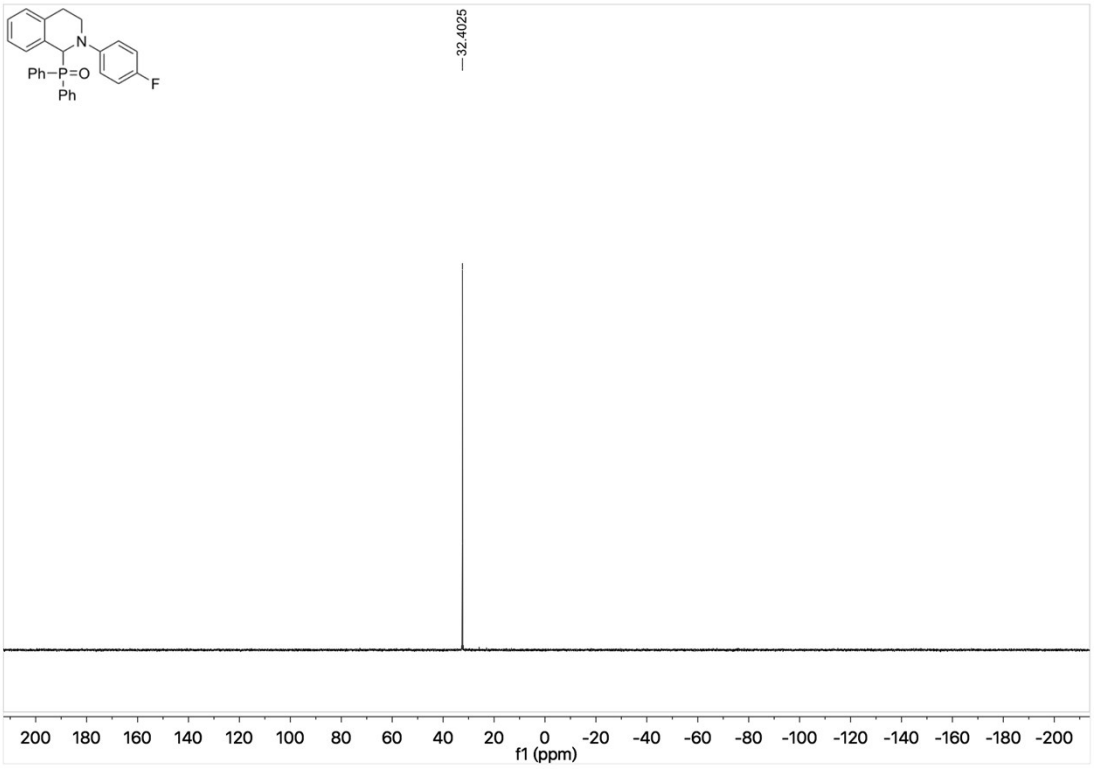
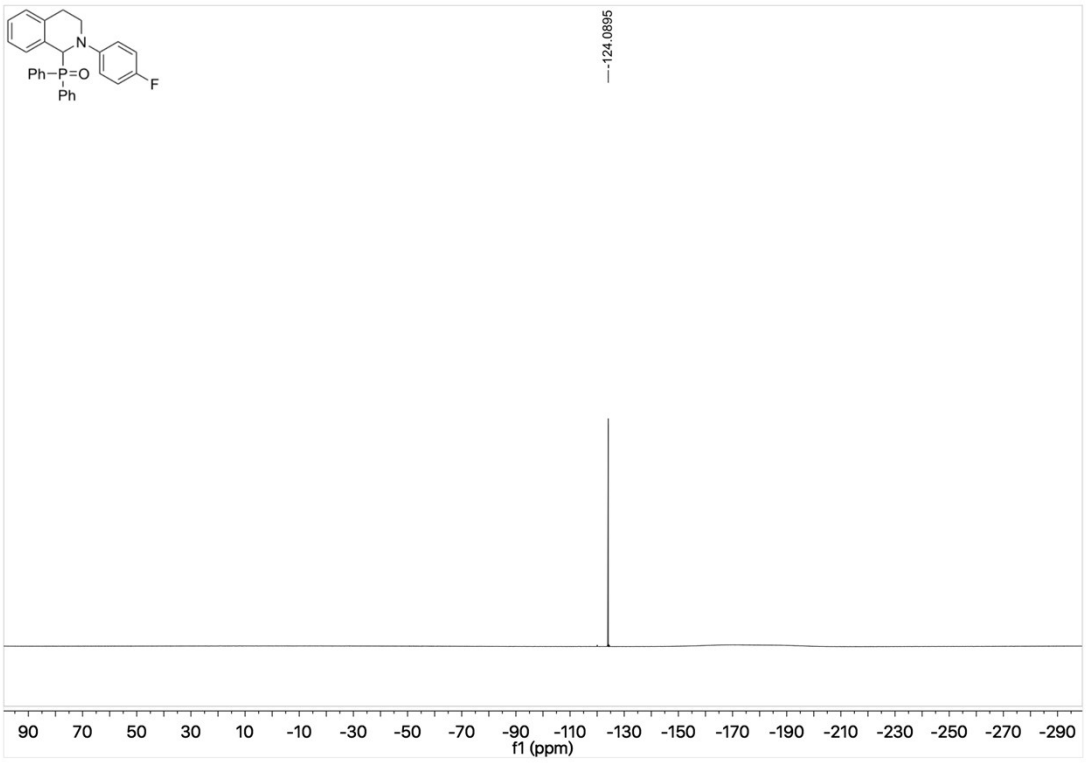
148.1254
136.9531
136.9105
135.7464
132.9303
132.3026
132.2188
132.1633
131.9856
131.8353
131.8112
131.7261
131.5838
131.5565
131.2631
129.9520
129.2961
129.2742
128.4500
128.3405
128.2919
128.1803
127.8456
127.8131
127.3026
127.2733
125.4489
125.4229
117.5659
62.5313
61.7334
45.5923
27.8814
25.2822
15.6616
Ph
P
O
Ph
N
Et
f1 (ppm)

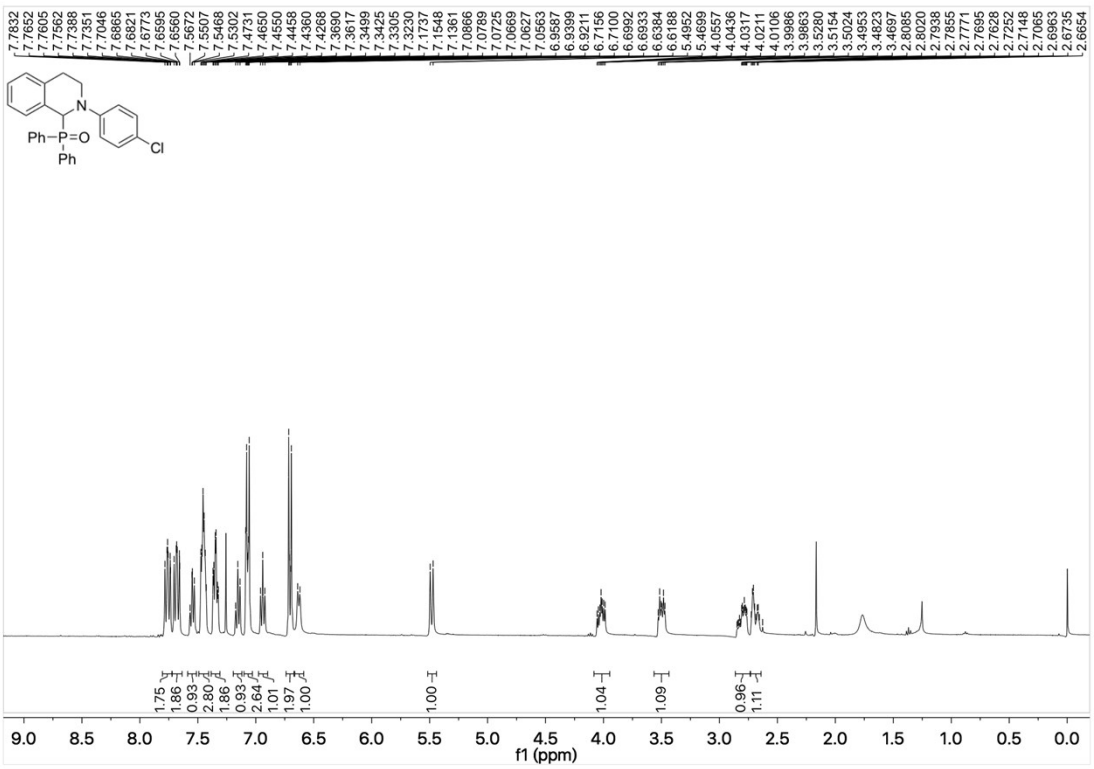

32.2954
Ph
P
O
Ph
N
Et
f1 (ppm)

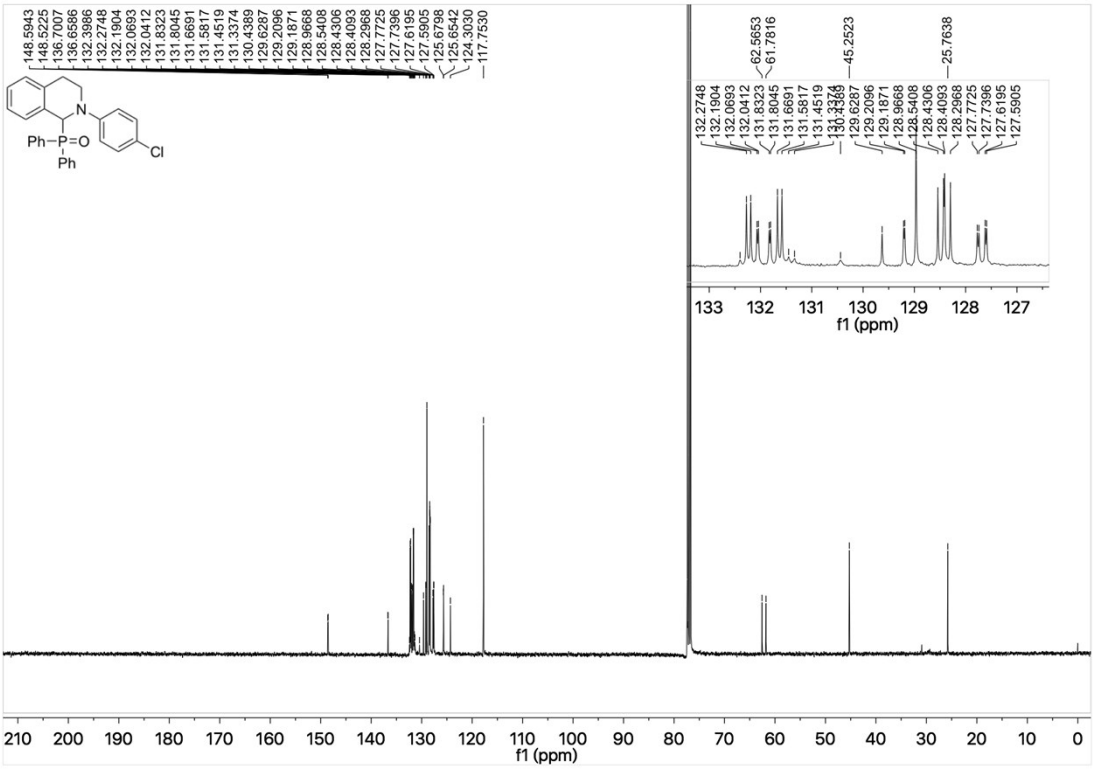

Ph–P=O
Ph
F
f1 (ppm)

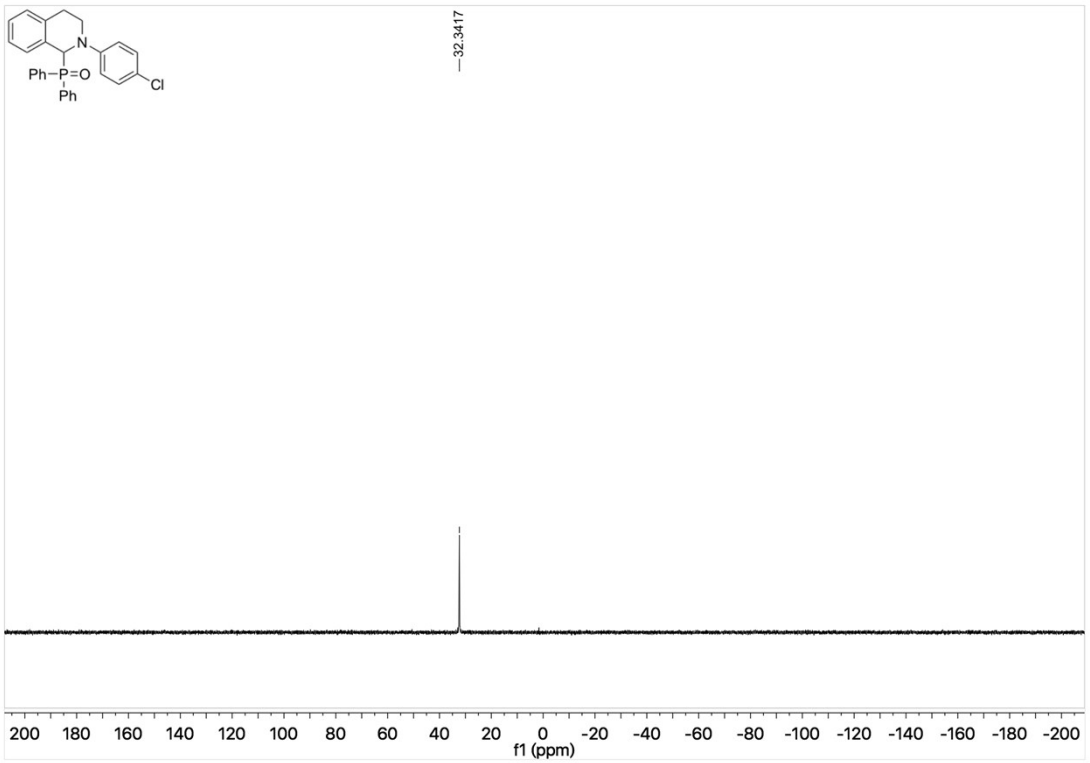

Ph–P=O
Ph
F
f1 (ppm)

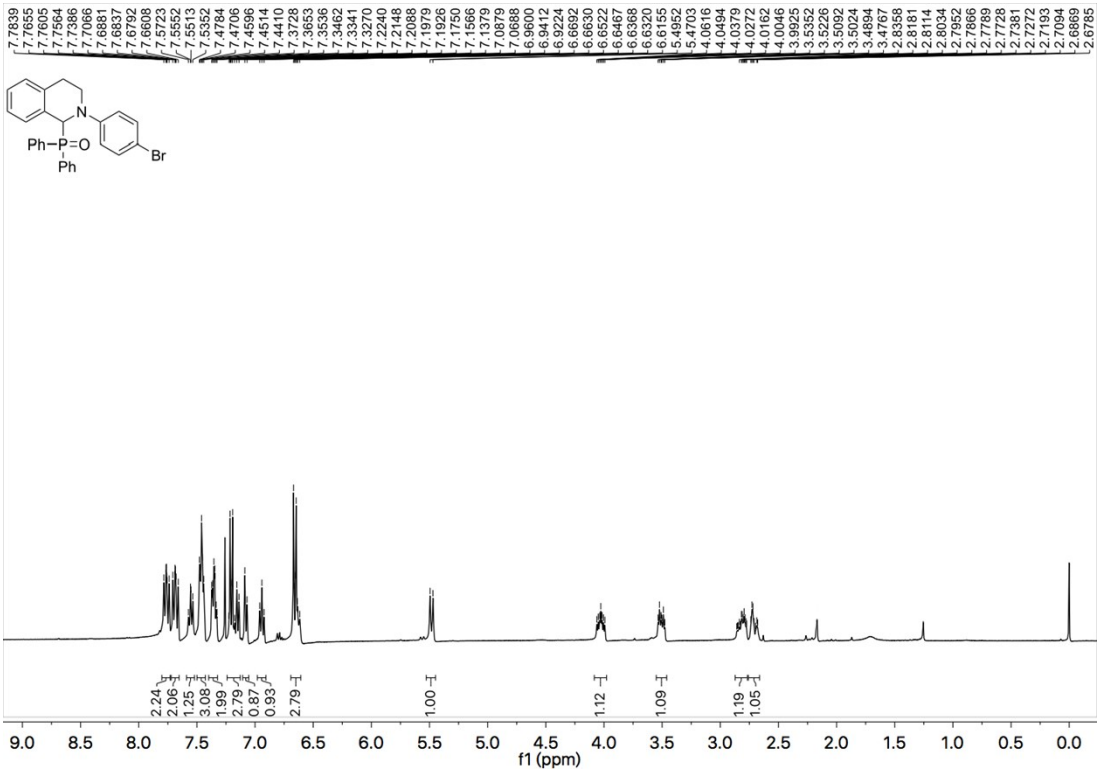

Ph–P=O
Ph
N
F
32.4025
200 180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200
f1 (ppm)

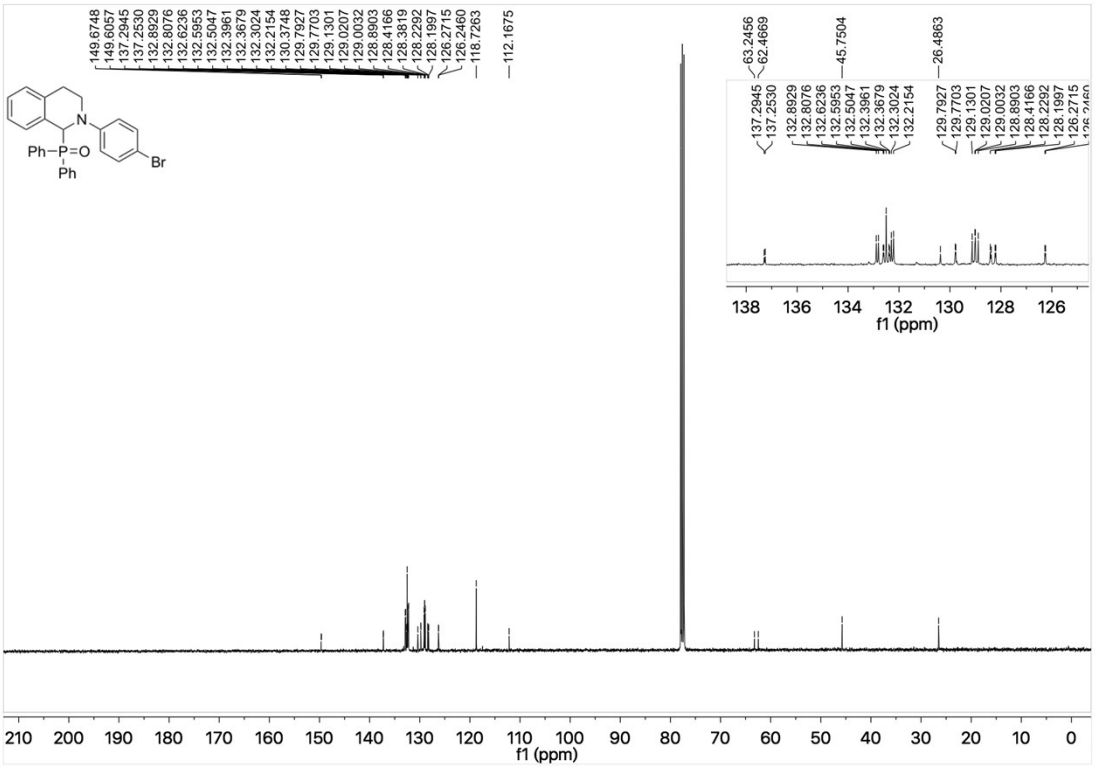

Ph–P=O
Ph
N
F
-124.0895
90 70 50 30 10 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230 -250 -270 -290
f1 (ppm)

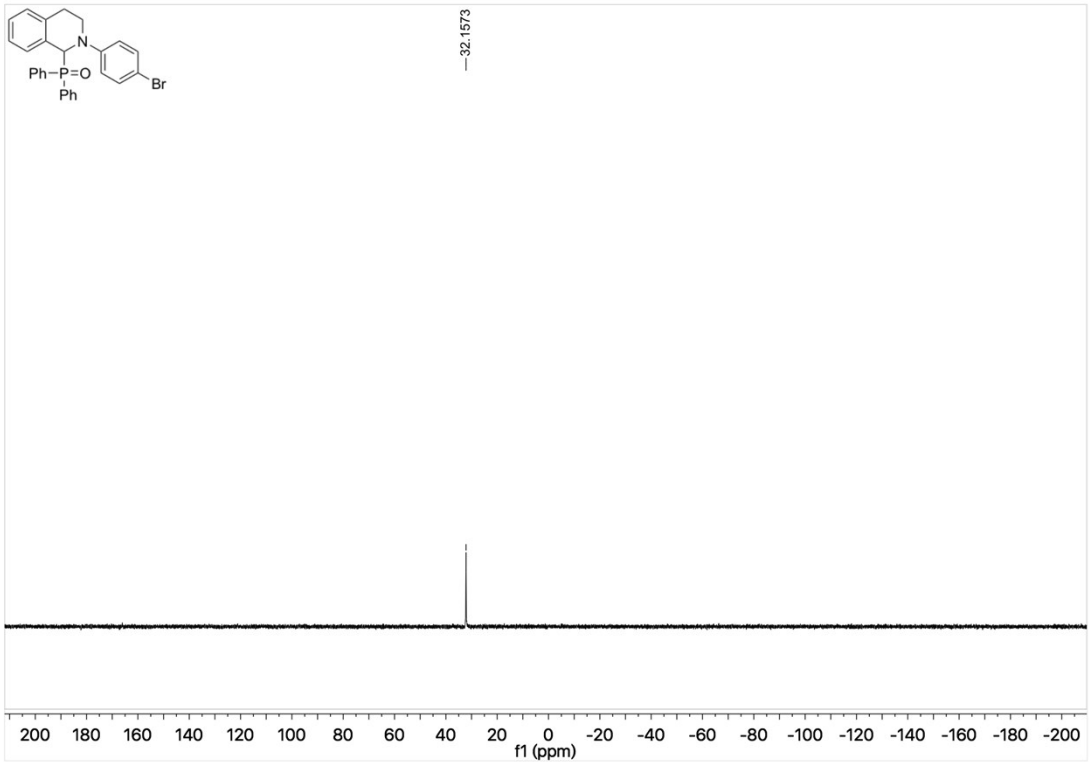

Ph–P=O
Ph
N
Cl
f1 (ppm)

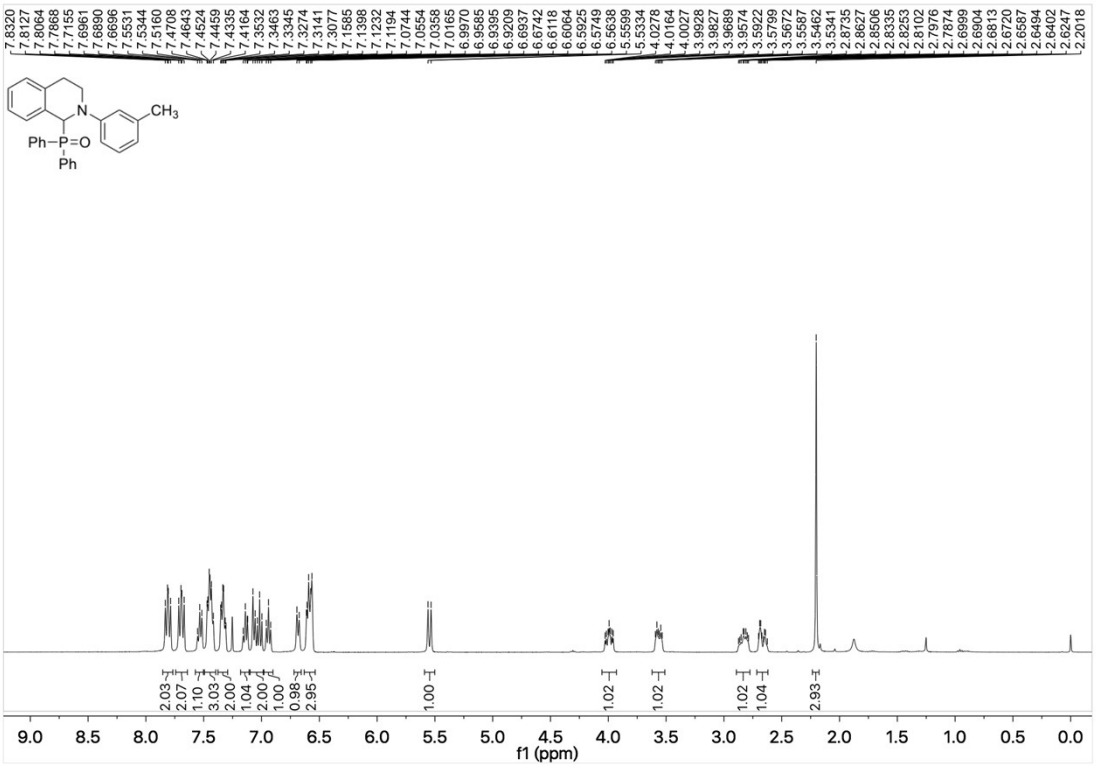

Ph–P=O
Ph
N
Cl
f1 (ppm)

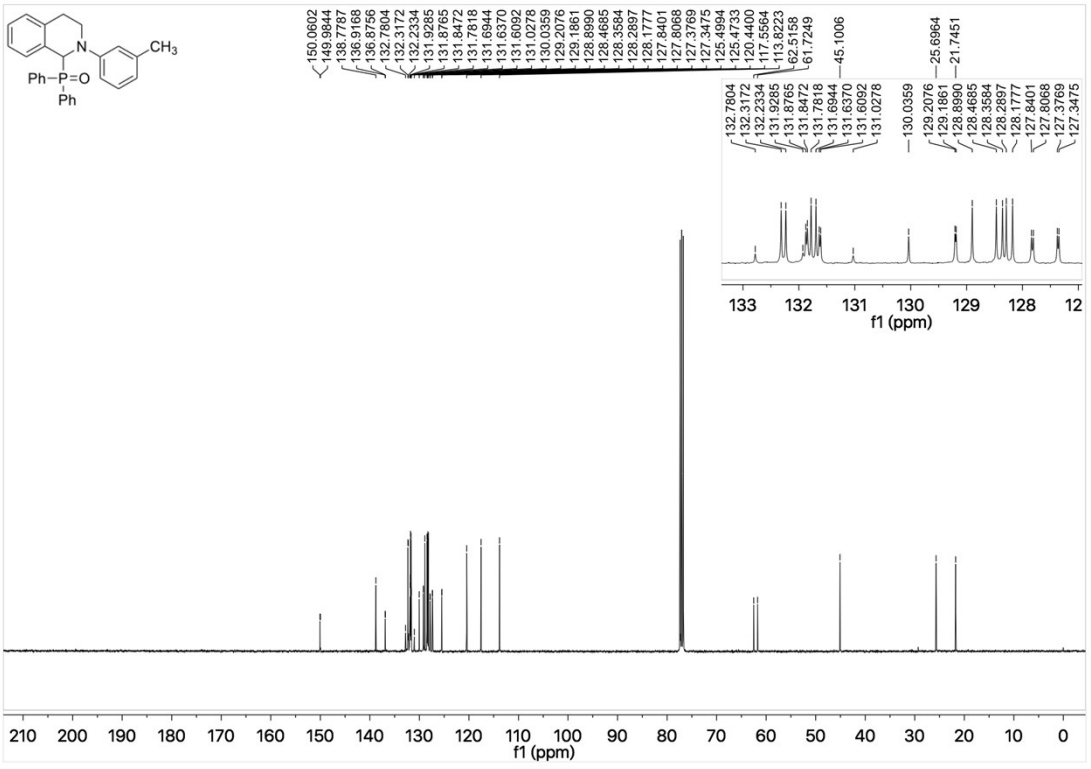

Ph–P=O
Ph
N
Cl
32.3417
200 180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200
f1 (ppm)

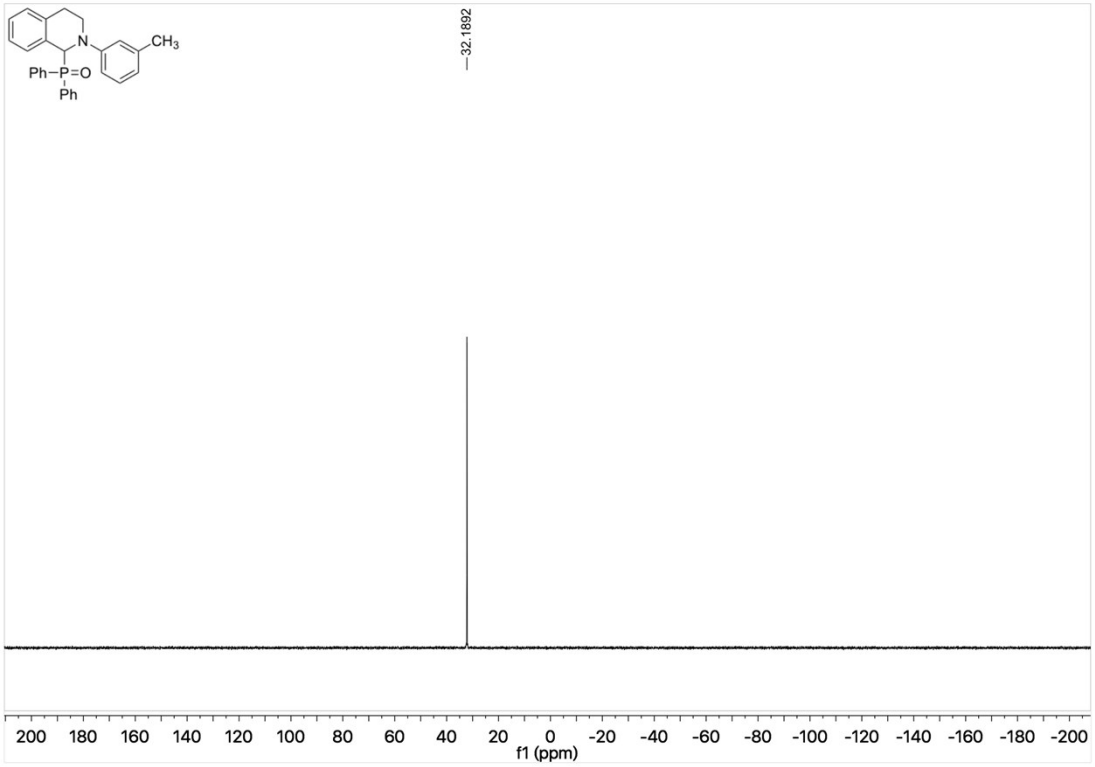

Ph–P=O
Ph
N
Br
2.24
2.06
1.25
3.08
1.99
2.79
0.87
0.93
2.79
1.00
1.12
1.09
1.19
1.05
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

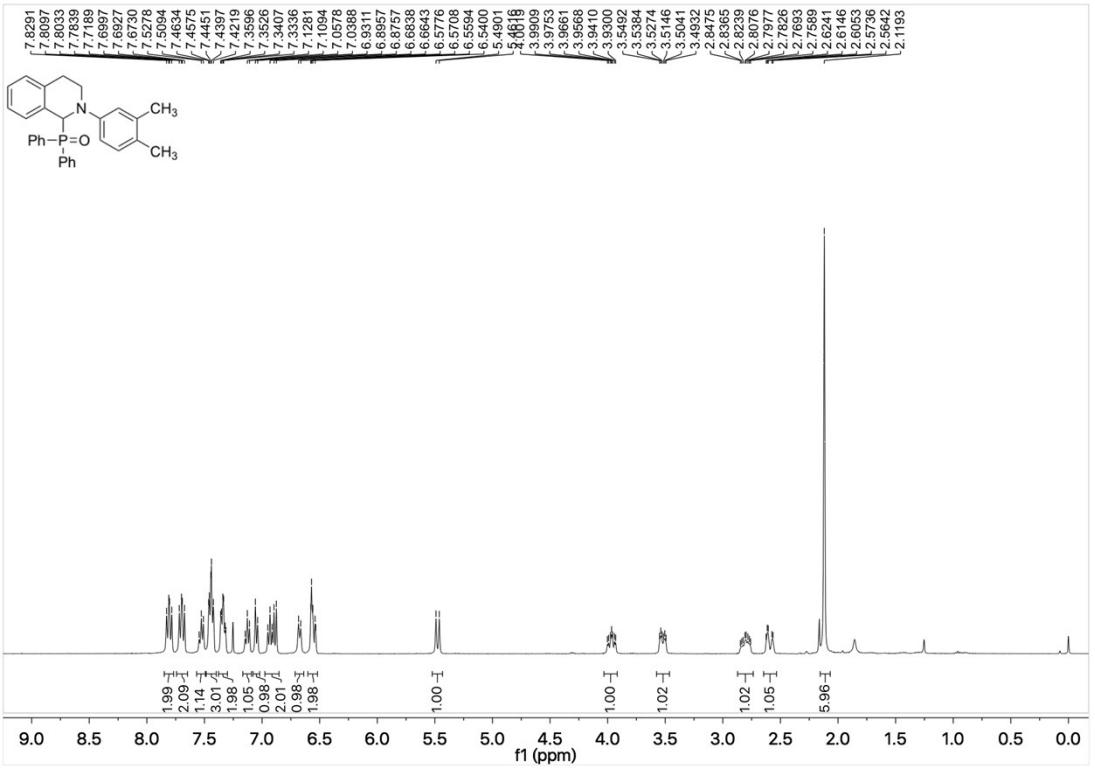

149.6748
149.6057
137.2945
137.2530
132.8929
132.8076
132.6236
132.5953
132.5047
132.3961
132.3679
132.3024
132.2154
130.3748
129.7927
129.7703
129.1301
129.0207
129.0032
128.8903
128.4166
128.3819
128.2292
128.1997
126.2715
126.2460
118.7263
112.1675
63.2456
62.4669
45.7504
26.4863
Ph–P=O
Ph
N
Br
f1 (ppm)

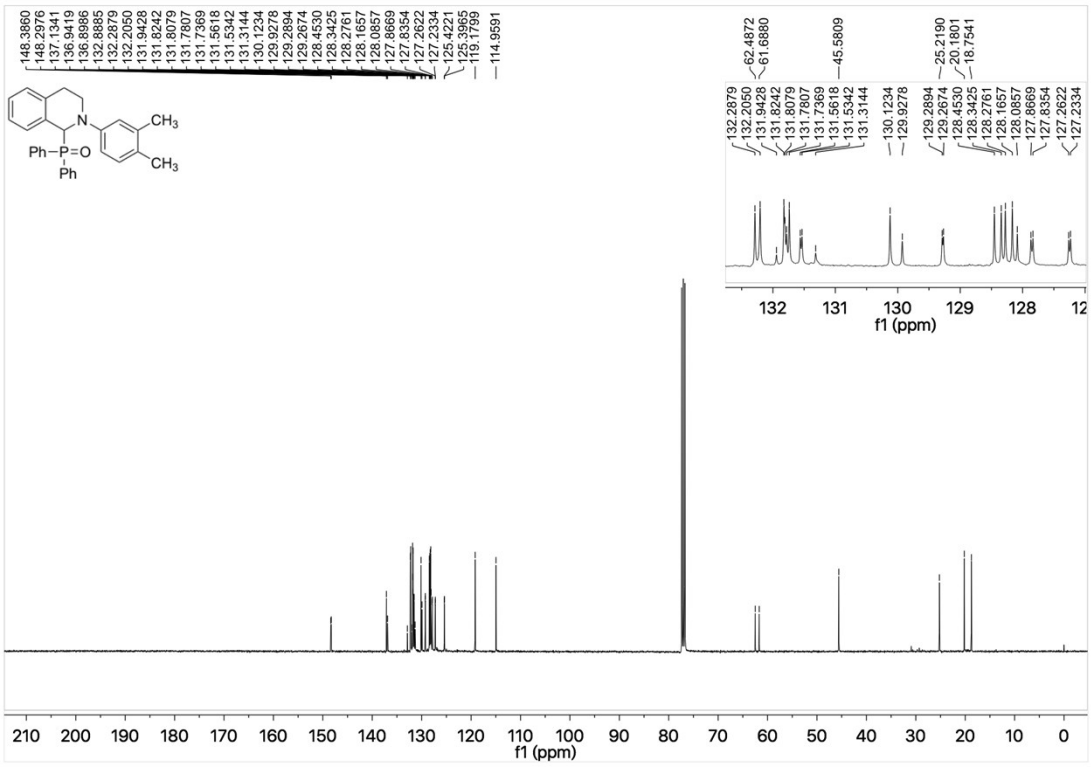

32.1573
Ph–P=O
Ph
N
Br
f1 (ppm)


f1 (ppm)


f1 (ppm)


32.1892
Ph–P=O
Ph
CH3
200 180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200
f1 (ppm)


7.8291
7.8097
7.8033
7.7839
7.7189
7.6997
7.6927
7.6730
7.5278
7.5094
7.4634
7.4575
7.4451
7.4397
7.4219
7.3596
7.3526
7.3407
7.3336
7.1281
7.1094
7.0578
7.0388
6.9311
6.8957
6.8757
6.6838
6.6643
6.5776
6.5708
6.5594
6.5400
5.4901
3.9909
3.9753
3.9661
3.9568
3.9410
3.9300
3.5492
3.5384
3.5274
3.5146
3.5041
3.4932
2.8475
2.8365
2.8239
2.8076
2.7977
2.7826
2.7693
2.7589
2.6241
2.6146
2.6053
2.5736
2.5642
2.1193
Ph–P=O
Ph
CH3
CH3
1.99
2.09
1.14
3.01
1.98
1.05
0.98
2.01
0.98
1.98
1.00
1.00
1.02
1.02
1.05
5.96
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)


148.3860
148.2976
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136.9419
136.8986
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131.9428
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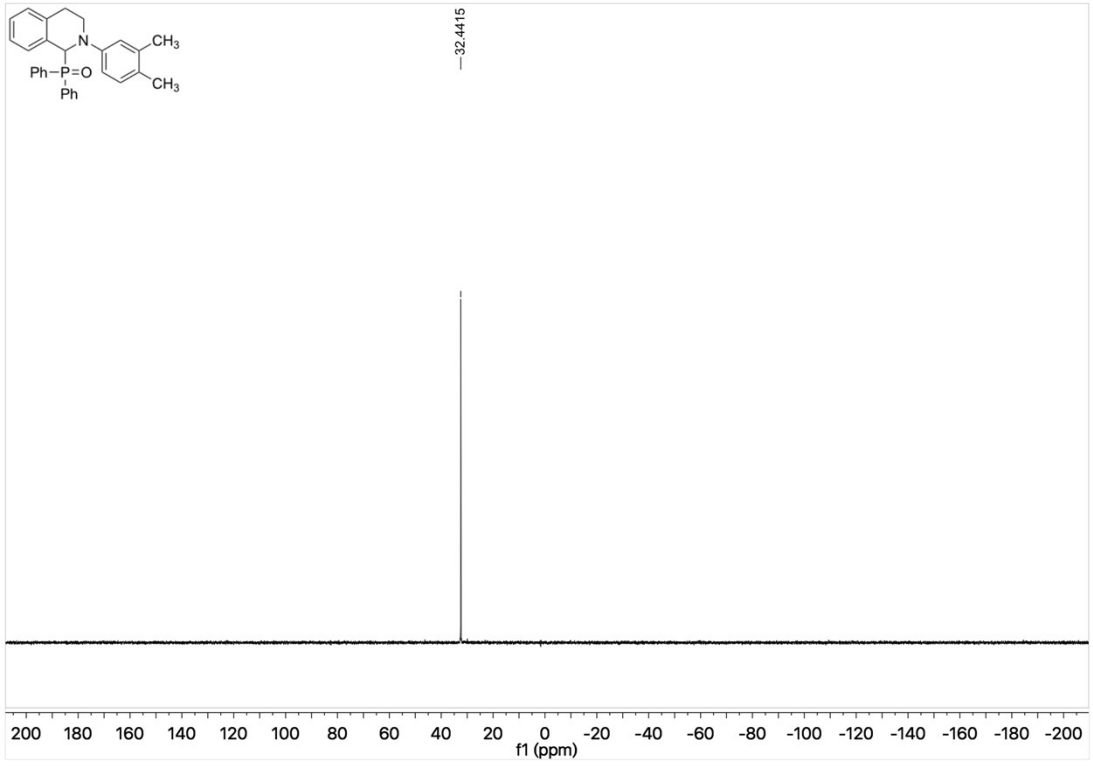
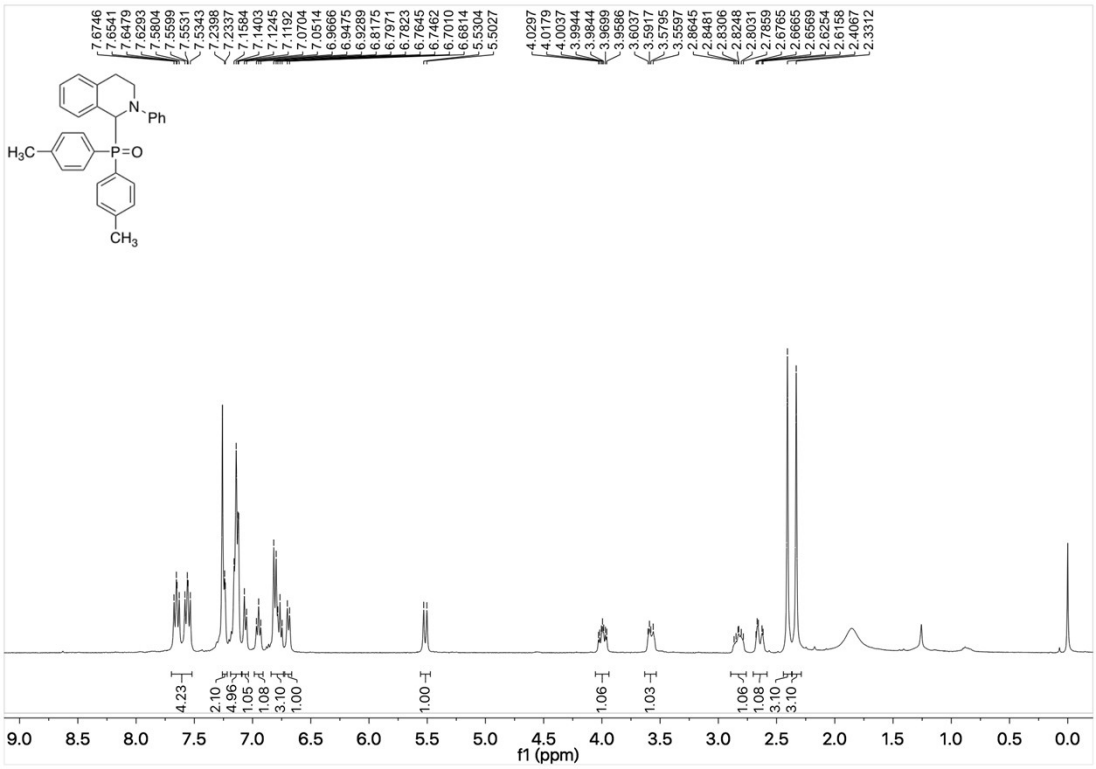
131.7807
131.7369
131.5618
131.5342
131.3144
130.1234
129.9278
129.2894
129.2674
128.4530
128.3425
128.2761
128.1657
128.0857
127.8669
127.8354
127.2622
127.2334
125.4221
125.3965
119.1799
114.9591
62.4872
61.6880
45.5809
25.2190
20.1801
18.7541
Ph–P=O
Ph
CH3
CH3
f1 (ppm)
f1 (ppm)


32.4415
Ph–P=O
Ph
CH3
CH3
f1 (ppm)


7.6746
7.6541
7.6479
7.6293
7.5804
7.5599
7.5531
7.5343
7.2398
7.2337
7.1584
7.1403
7.1245
7.1192
7.0704
7.0514
6.9666
6.9475
6.9289
6.8175
6.7971
6.7823
6.7645
6.7462
6.7010
6.6814
5.5304
5.5027
4.0297
4.0179
4.0037
3.9944
3.9844
3.9699
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3.5917
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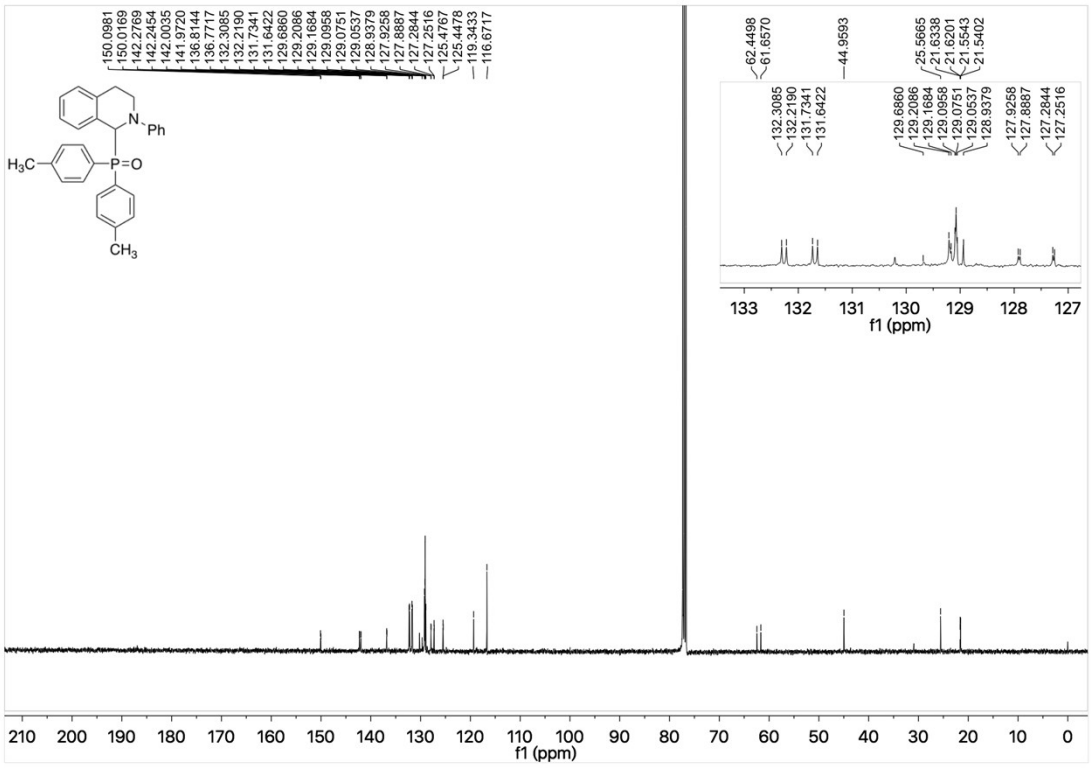
2.8248
2.8031
2.7859
2.6765
2.6665
2.6569
2.6254
2.6158
2.4067
2.3312
N
Ph
P=O
H3C
CH3
4.23
2.10
4.96
1.05
1.08
3.10
1.00
1.00
1.06
1.03
1.06
1.08
3.10
3.10
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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142.0035
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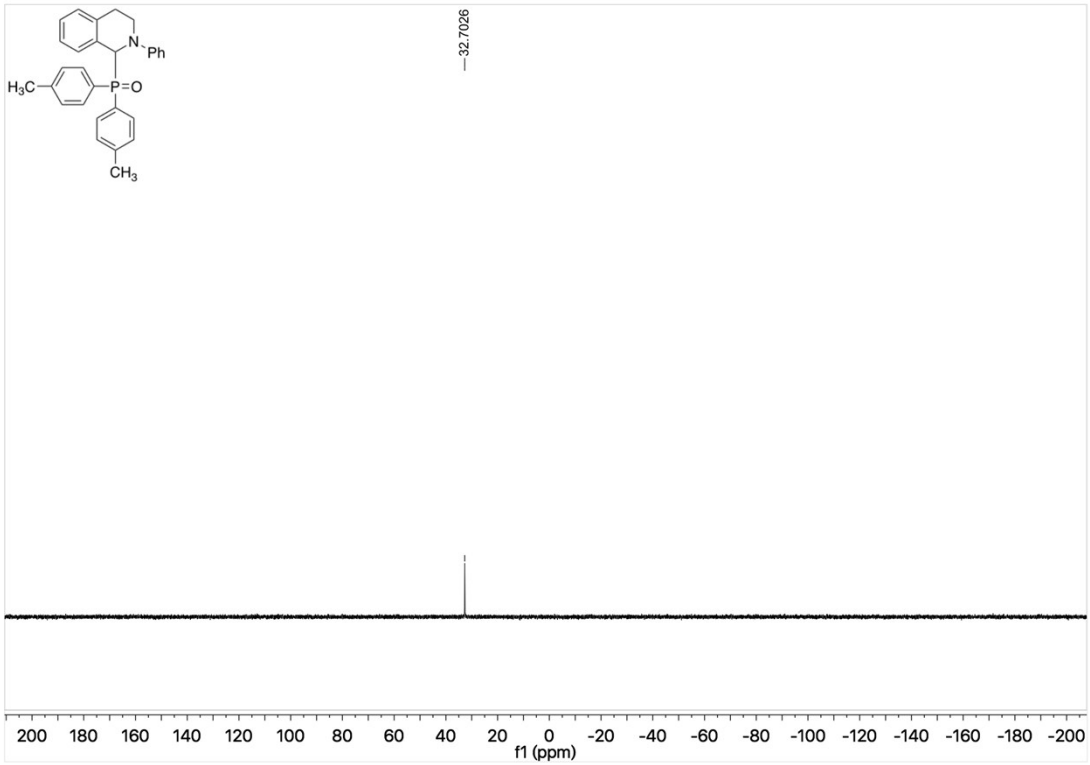
136.8144
136.7717
132.3085
132.2190
131.7341
131.6422
129.6860
129.2086
129.1684
129.0958
129.0751
129.0537
128.9379
127.9258
127.8887
127.2844
127.2516
125.4767
125.4478
119.3433
116.6717
62.4498
61.6570
44.9593
25.5665
21.6338
21.6201
21.5543
21.5402
N
Ph
P=O
H3C
CH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
133 132 131 130 129 128 127
f1 (ppm)


32.7026
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Ph
H3C
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CH3
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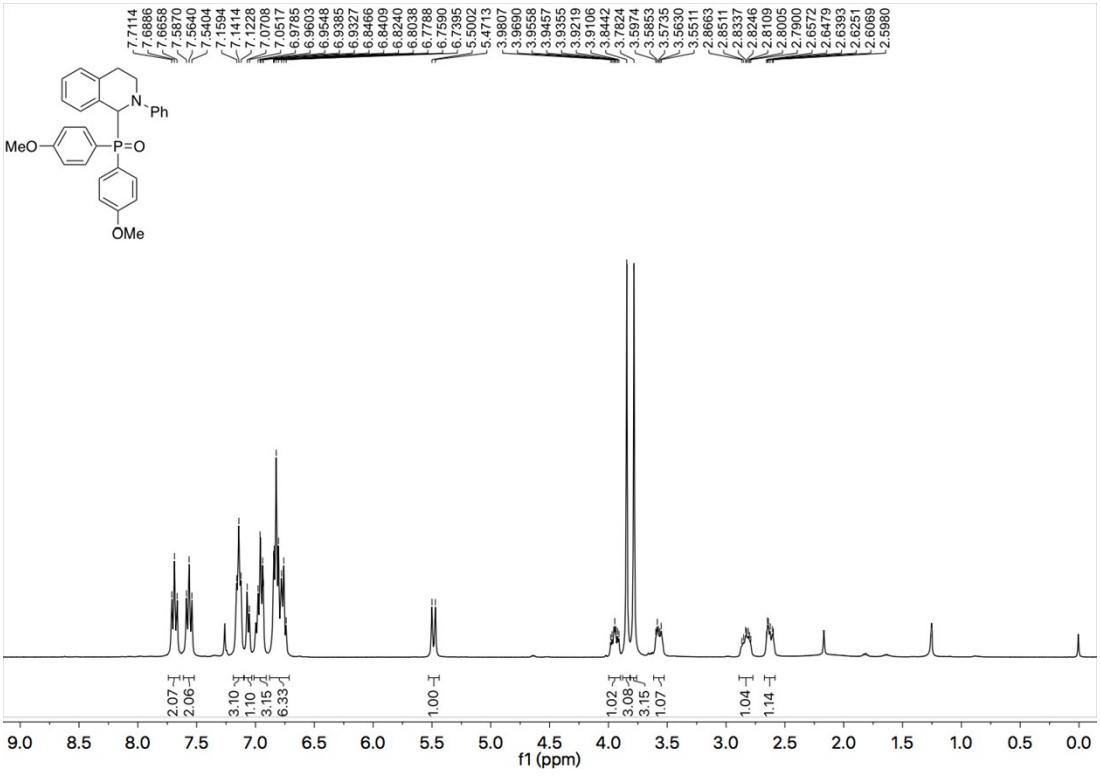
f1 (ppm)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7.1594
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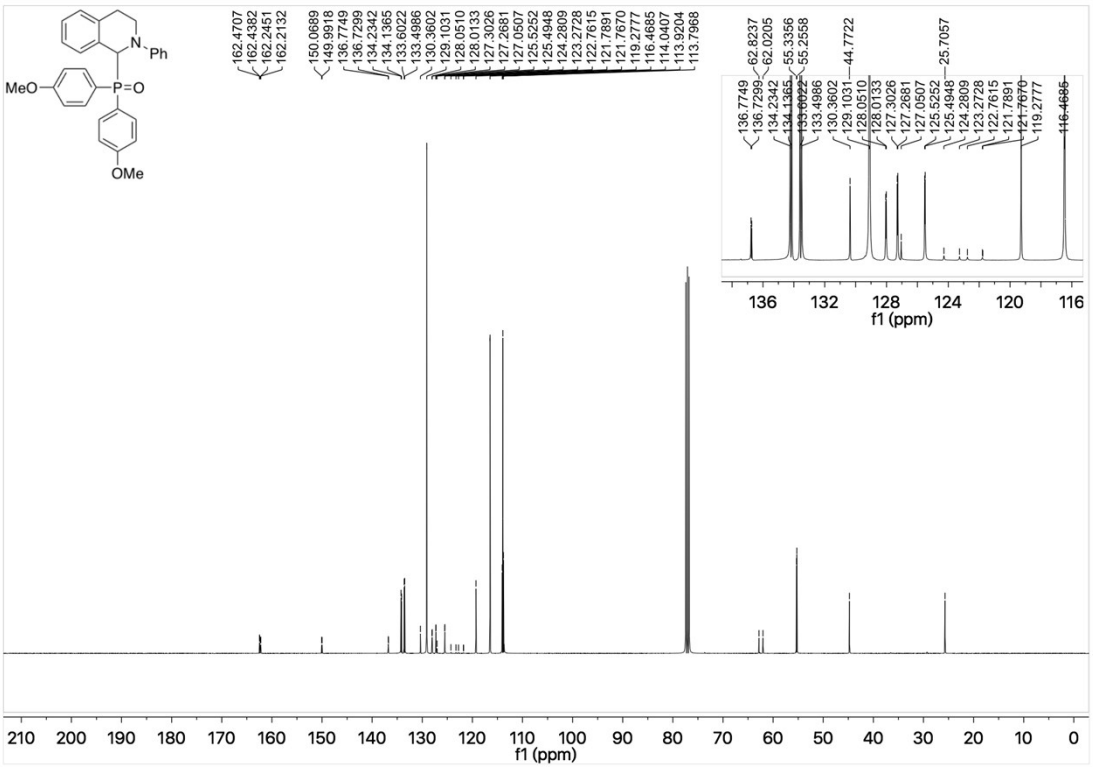
7.1228
7.0708
7.0517
6.9785
6.9603
6.9548
6.9385
6.9327
6.8466
6.8409
6.8240
6.8038
6.7788
6.7590
6.7395
5.4713
5.5002
3.9807
3.9690
3.9558
3.9457
3.9355
3.9219
3.9106
3.8442
3.7824
3.5974
3.5853
3.5735
3.5630
3.5511
2.8663
2.8511
2.8337
2.8246
2.8109
2.8005
2.7900
2.6572
2.6479
2.6393
2.6251
2.6069
2.5980
N
Ph
MeO
P=O
OMe
2.07
2.06
3.10
1.10
3.15
6.33
1.00
1.02
3.08
3.15
1.07
1.04
1.14
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)


MeO
P=O
N
Ph
OMe
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162.2451
162.2132
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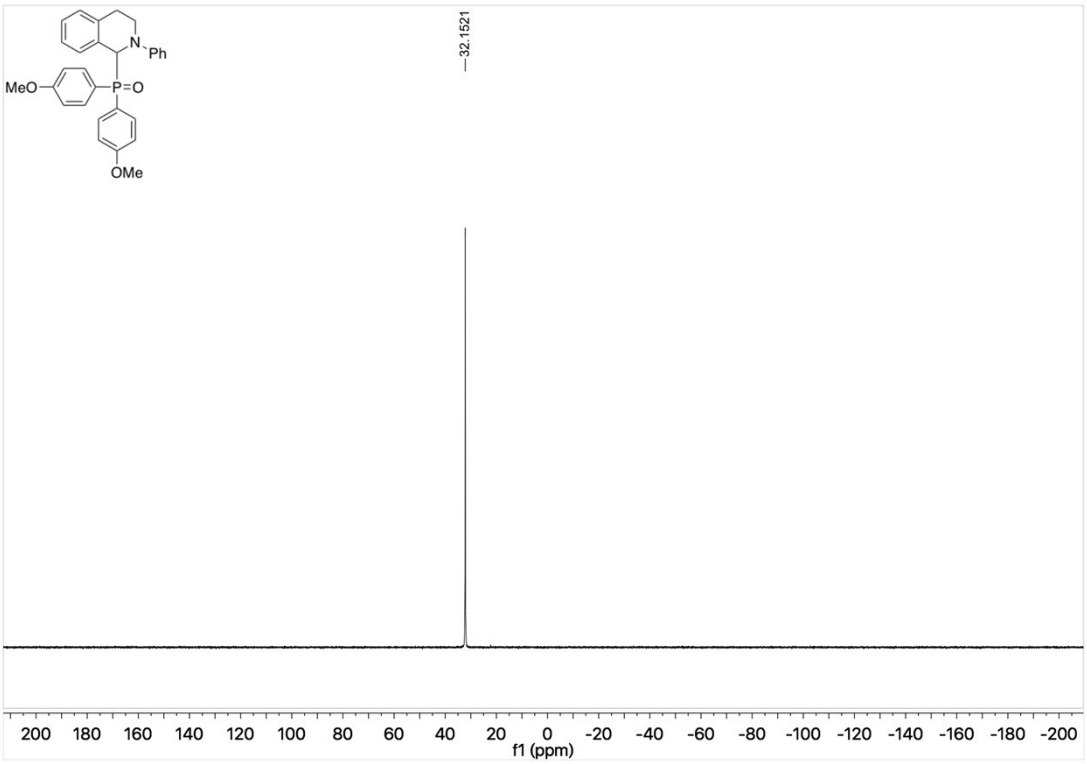
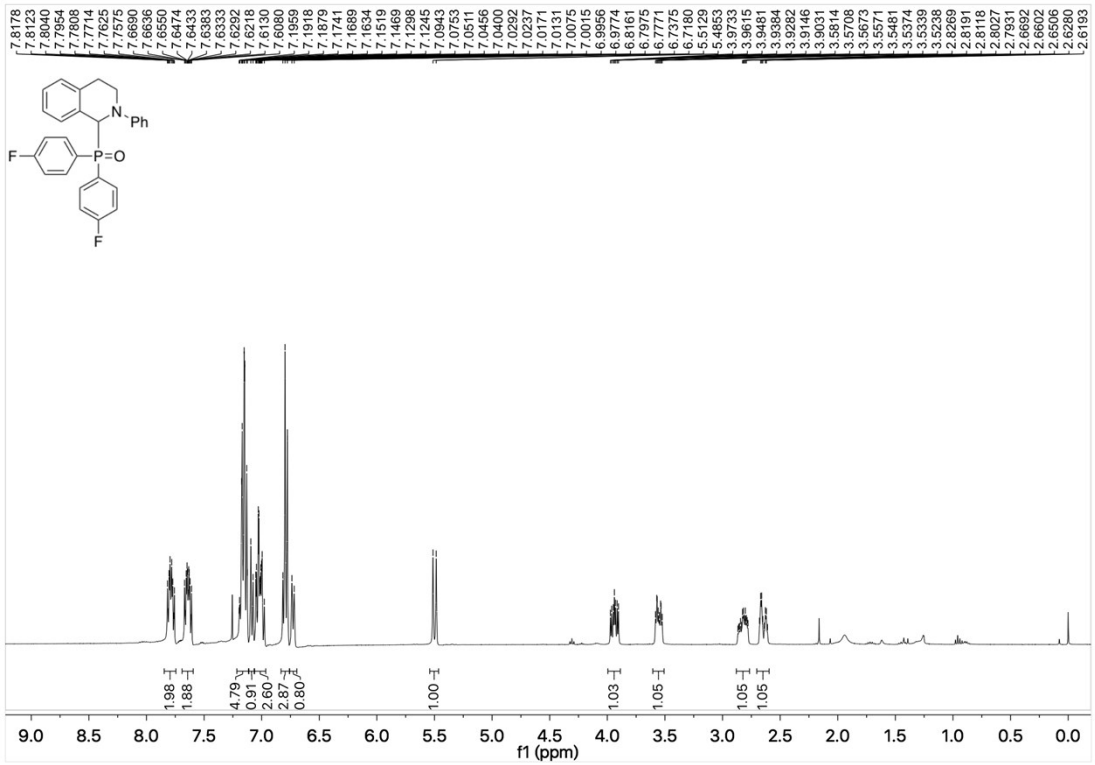
149.9918
136.7749
136.7299
134.2342
134.1365
133.6022
133.4986
130.3602
129.1031
128.0510
128.0133
127.3026
127.2681
127.0507
125.5252
125.4948
124.2809
123.2728
122.7615
121.7891
121.7670
119.2777
116.4685
114.0407
113.9204
113.7968
62.8237
62.0205
55.3356
55.2558
44.7722
25.7057
f1 (ppm)


MeO
P=O
N
Ph
OMe
32.1521
f1 (ppm)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7.7625
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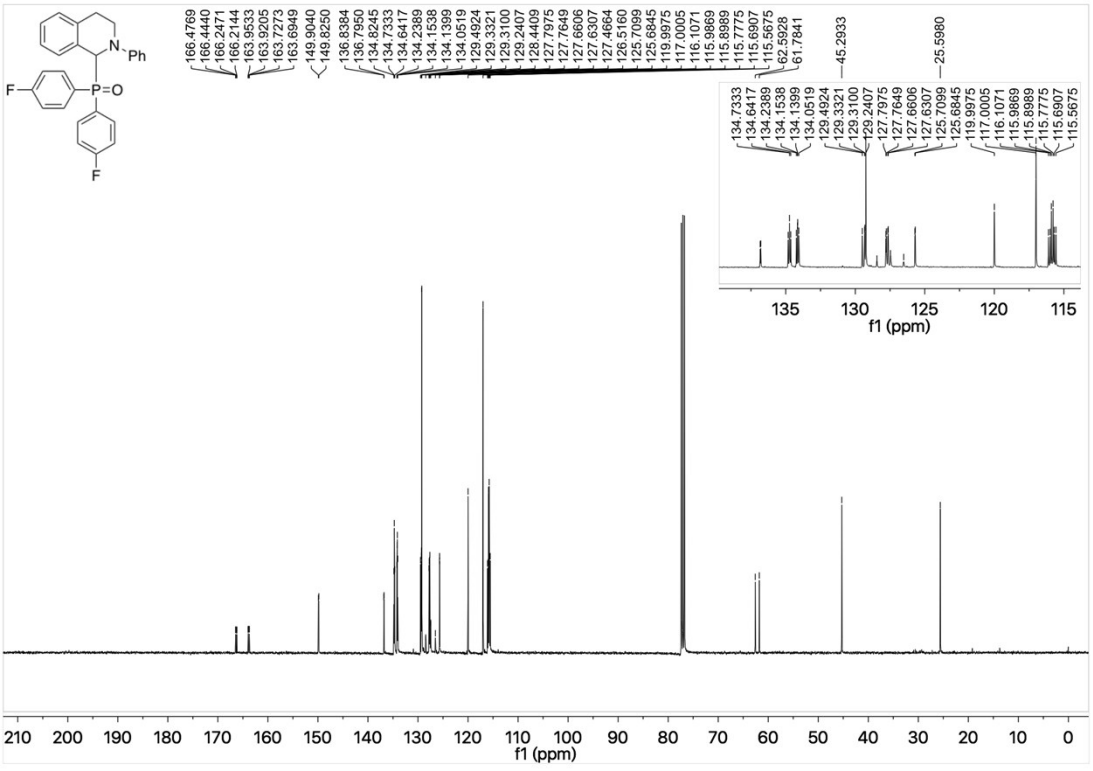
7.7490
7.7436
7.7350
7.6550
7.6474
7.6433
7.6383
7.6333
7.6292
7.6218
7.6130
7.6080
7.1959
7.1918
7.1879
7.1741
7.1689
7.1634
7.1519
7.1469
7.1298
7.1245
7.0943
7.0753
7.0511
7.0456
7.0400
7.0292
7.0237
7.0171
7.0131
7.0075
7.0015
6.9956
6.9774
6.8161
6.7975
6.7771
6.7375
6.7180
5.5129
5.4853
3.9733
3.9615
3.9481
3.9384
3.9282
3.9146
3.9031
3.5814
3.5708
3.5673
3.5571
3.5481
3.5374
3.5339
3.5238
3.5238
2.8269
2.8191
2.8118
2.8027
2.7931
2.6692
2.6602
2.6506
2.6280
2.6193
N
Ph
F
P=O
F
1.98
1.88
4.79
0.91
2.60
2.87
0.80
1.00
1.03
1.05
1.05
1.05
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)


N
Ph
F
P=O
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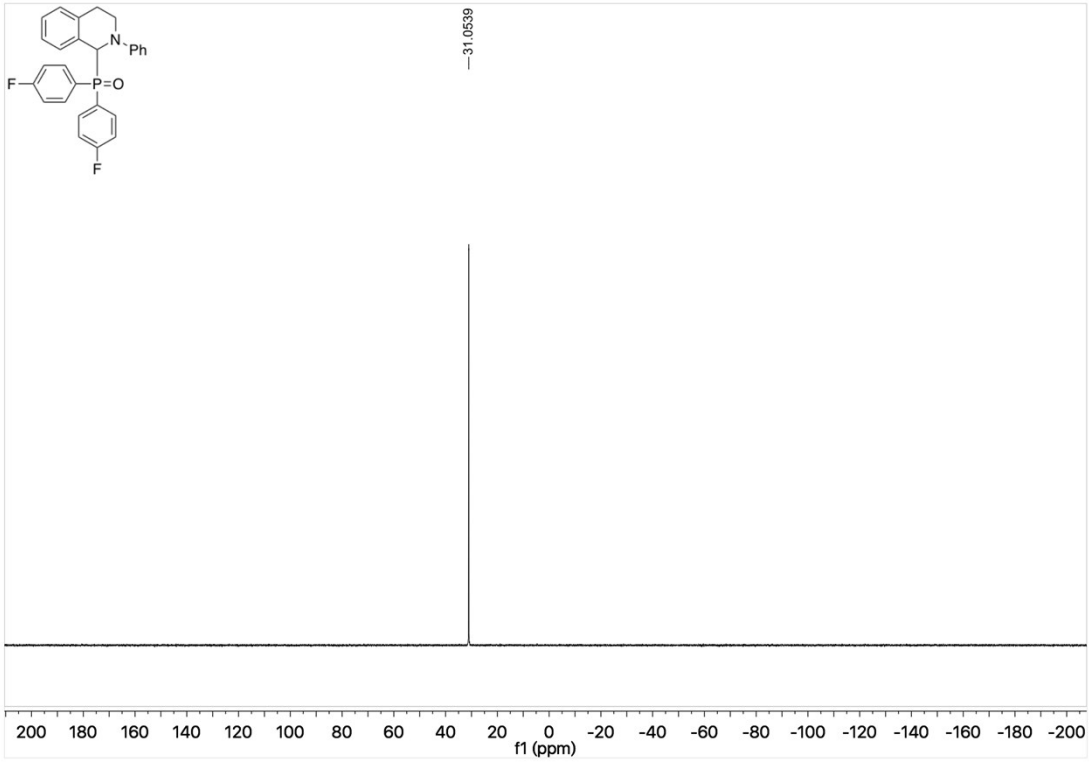
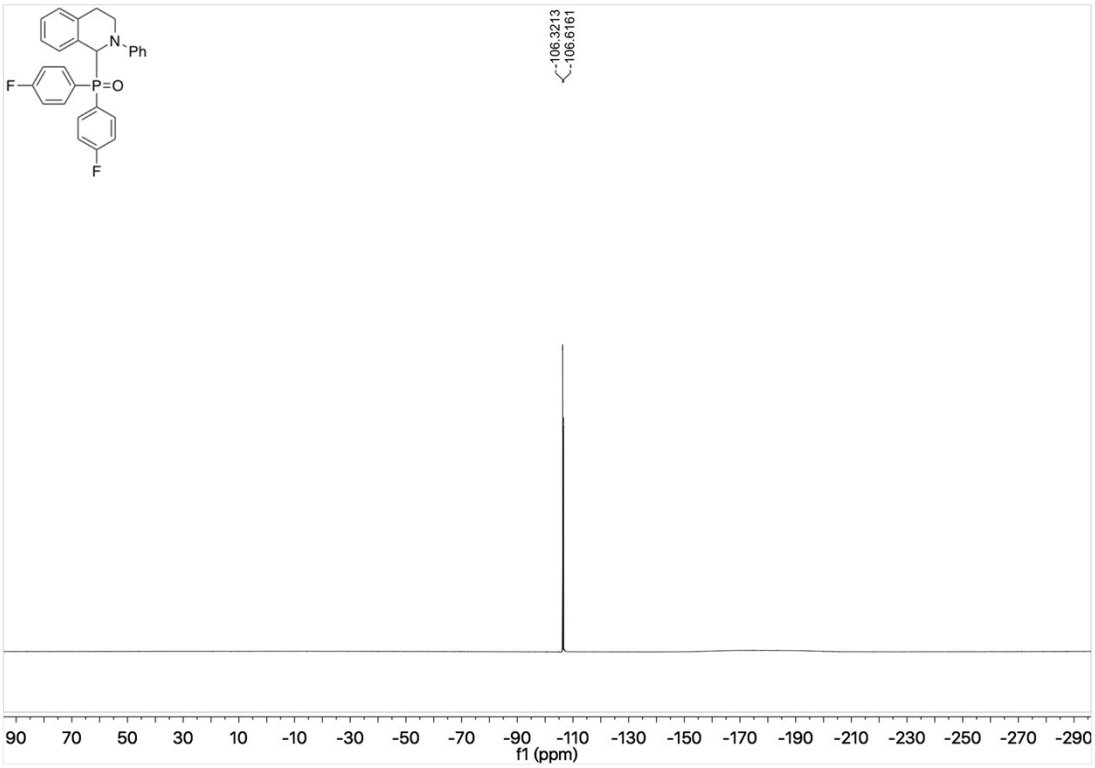
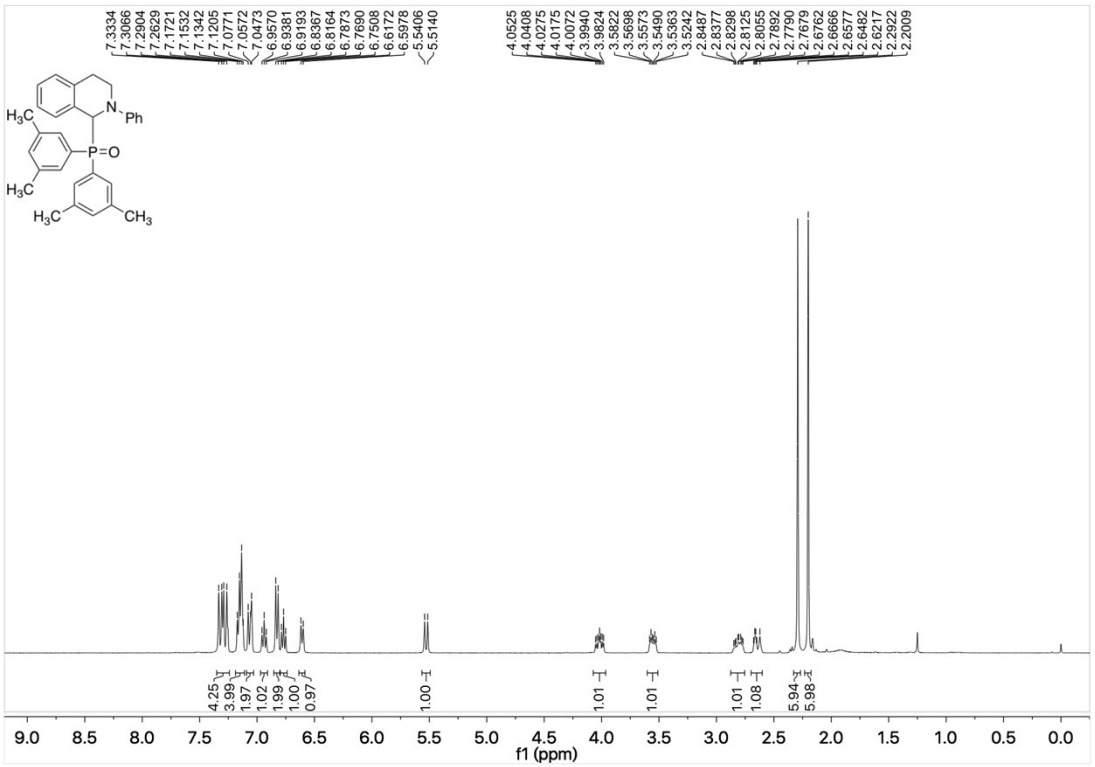
166.4440
166.2471
166.2144
163.9533
163.9205
163.7273
163.6949
149.9040
149.8250
136.8384
136.7950
134.8245
134.7333
134.6417
134.2389
134.1538
134.1399
134.0519
129.4924
129.3321
129.3100
129.2407
128.4409
127.7975
127.7649
127.6606
127.6307
127.4664
126.5160
125.7099
125.6845
119.9975
117.0005
116.1071
115.9869
115.8989
115.7775
115.6907
115.5675
62.5928
61.7841
45.2933
25.5980
134.7333
134.6417
134.2389
134.1538
134.1399
134.0519
129.4924
129.3321
129.3100
129.2407
127.7975
127.7649
127.6606
127.6307
125.7099
125.6845
119.9975
117.0005
116.1071
115.9869
115.8989
115.7775
115.6907
115.5675
135 130 125 120 115
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


31.0539
N
Ph
F
P=O
F
200 180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200
f1 (ppm)


106.3213
106.6161
N
Ph
F
P=O
F
90 70 50 30 10 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230 -250 -270 -290
f1 (ppm)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7.1721
7.1532
7.1342
7.1205
7.0771
7.0572
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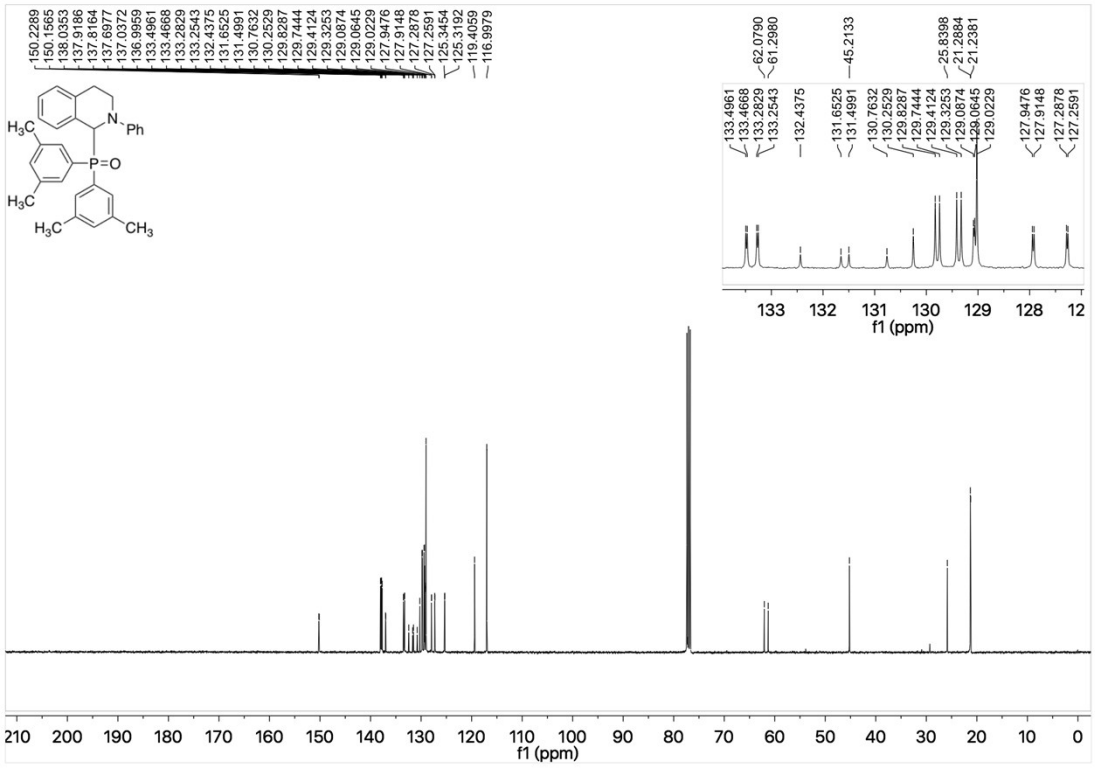
6.9570
6.9381
6.9193
6.8367
6.8164
6.7873
6.7690
6.7508
6.6172
6.5978
5.5406
5.5140
4.0525
4.0408
4.0275
4.0175
4.0072
3.9940
3.9824
3.5822
3.5698
3.5573
3.5490
3.5363
3.5242
2.8487
2.8377
2.8298
2.8125
2.8055
2.7892
2.7790
2.7679
2.6762
2.6666
2.6577
2.6482
2.6217
2.2922
2.2009
Ph
P=O
4.25
3.99
1.97
1.02
1.99
1.00
0.97
1.00
1.01
1.01
1.01
1.08
5.94
5.98
f1 (ppm)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150.1565
138.0353
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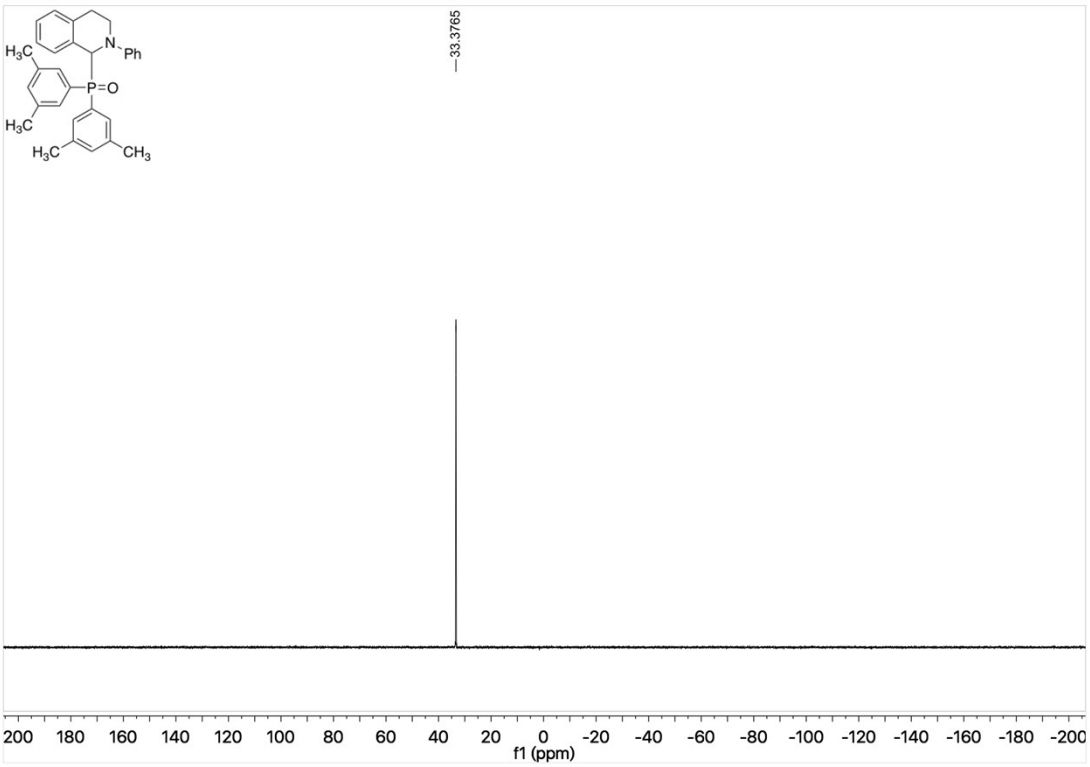
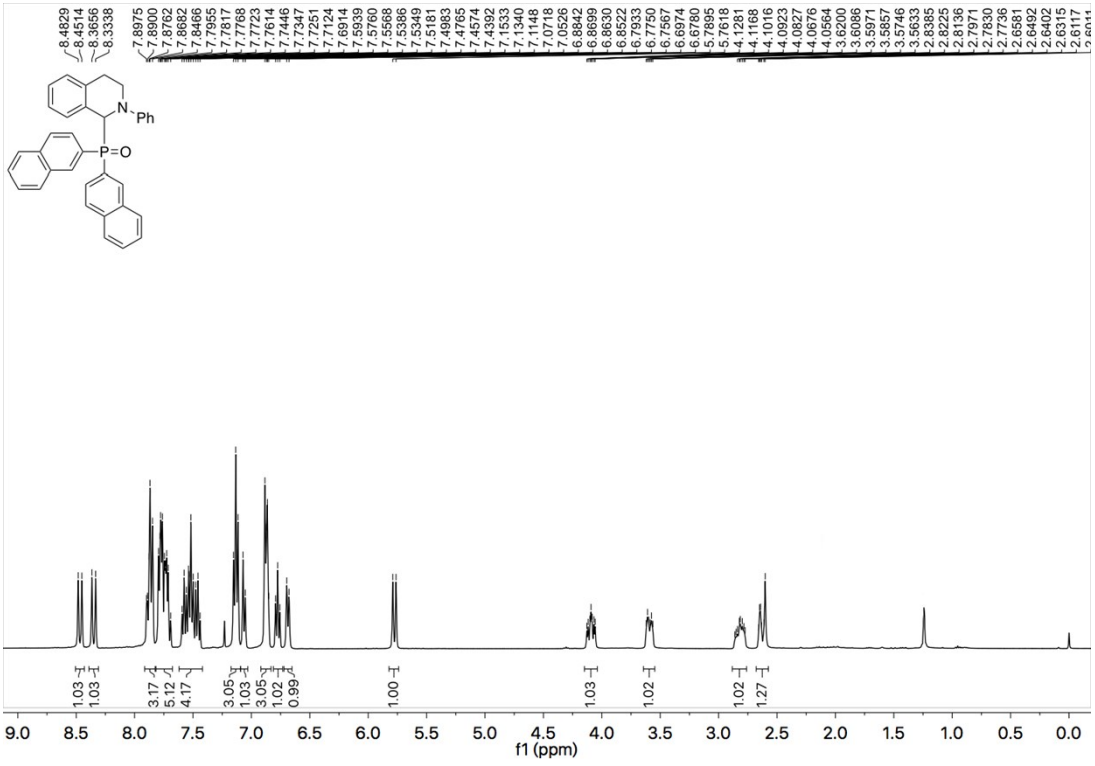
137.8164
137.6977
137.0372
136.9959
133.4961
133.4668
133.2829
133.2543
132.4375
131.6525
131.4991
130.7632
130.2529
129.8287
129.7444
129.4124
129.3253
129.0874
129.0645
129.0229
127.9476
127.9148
127.2878
127.2591
125.3454
125.3192
119.4059
116.9979
62.0790
61.2980
45.2133
25.8398
21.2884
21.2381
Ph
P=O
f1 (ppm)


33.3765
N
Ph
P=O
H3C
H3C
H3C
CH3
200 180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200
f1 (ppm)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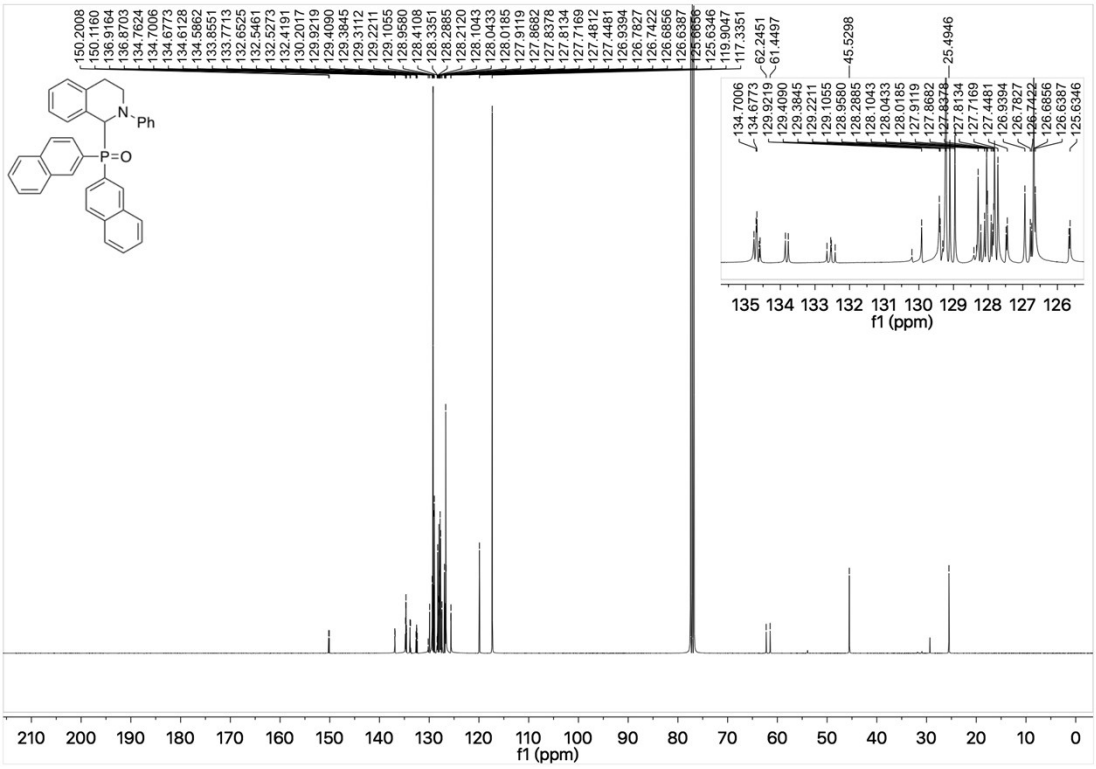
7.8466
7.7955
7.7817
7.7768
7.7723
7.7614
7.7446
7.7347
7.7251
7.7124
7.6914
7.5939
7.5760
7.5568
7.5386
7.5349
7.5181
7.4983
7.4765
7.4574
7.4392
7.1533
7.1340
7.1148
7.0718
7.0526
6.8842
6.8699
6.8630
6.8522
6.7933
6.7750
6.7567
6.6974
6.6780
5.7895
5.7618
4.1281
4.1168
4.1016
4.0923
4.0827
4.0676
4.0564
3.6200
3.6086
3.5971
3.5857
3.5746
3.5633
2.8385
2.8225
2.8136
2.7971
2.7830
2.7736
2.6581
2.6492
2.6402
2.6315
2.6117
N
Ph
P=O
1.03
1.03
3.17
5.12
4.17
3.05
1.03
3.05
1.02
0.99
1.00
1.03
1.02
1.02
1.27
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)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132.5273
132.4191
130.2017
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129.4090
129.3845
129.3112
129.2211
129.1055
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128.4108
128.3351
128.2885
128.2120
128.1043
128.0433
128.0185
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127.8378
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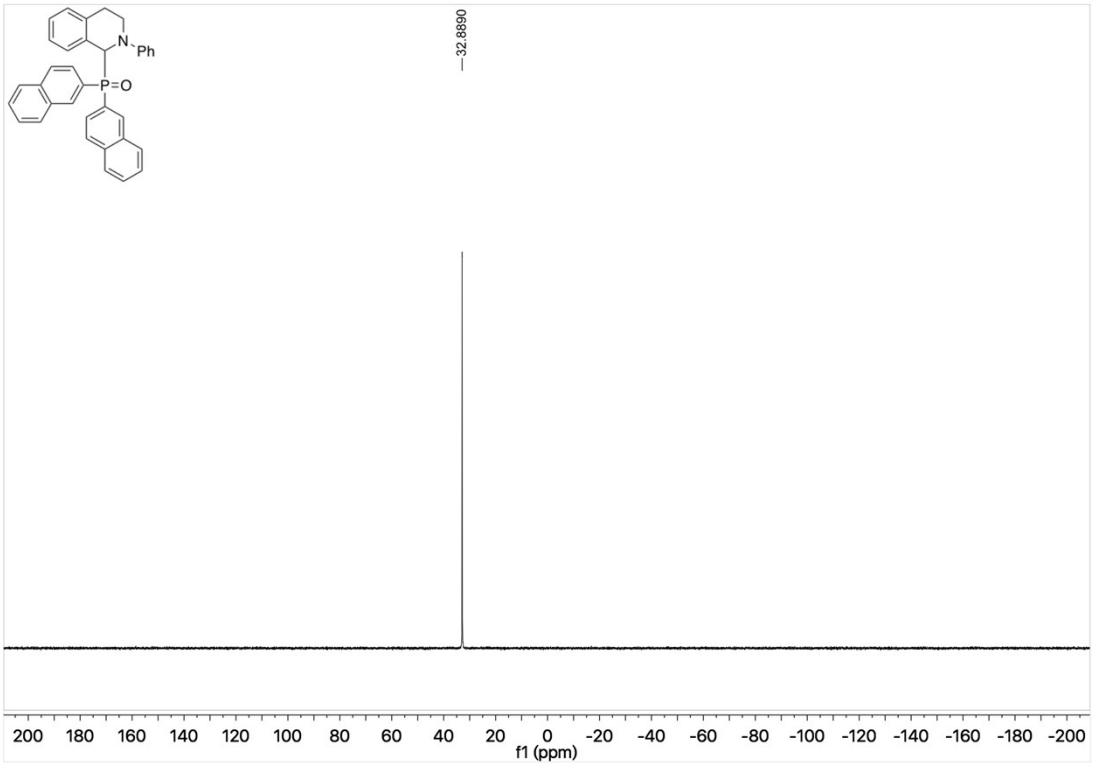
127.7169
127.4812
127.4481
126.9394
126.7827
126.7422
126.6856
126.6387
125.6346
119.9047
117.3351
62.2451
61.4497
45.5298
25.4946
N
Ph
P=O
135 134 133 132 131 130 129 128 127 126
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


32.8890
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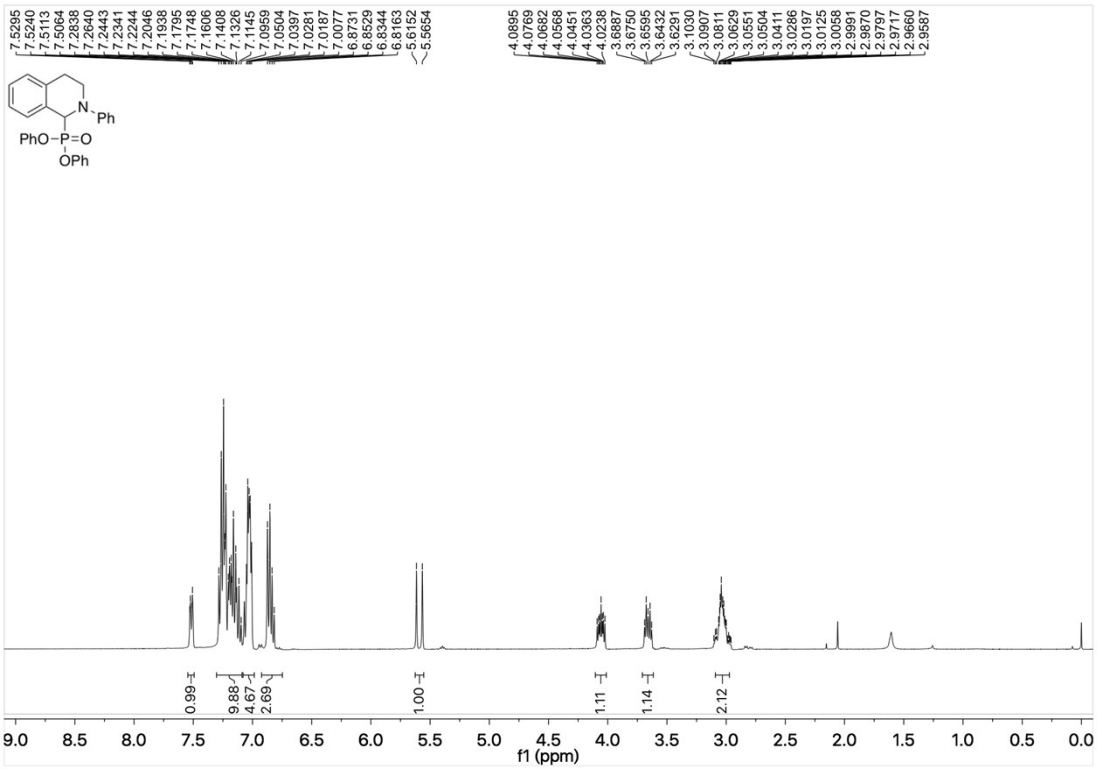
Ph
P=O
200 180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200
f1 (ppm)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7.2244
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7.1938
7.1795
7.1748
7.1606
7.1408
7.1326
7.1145
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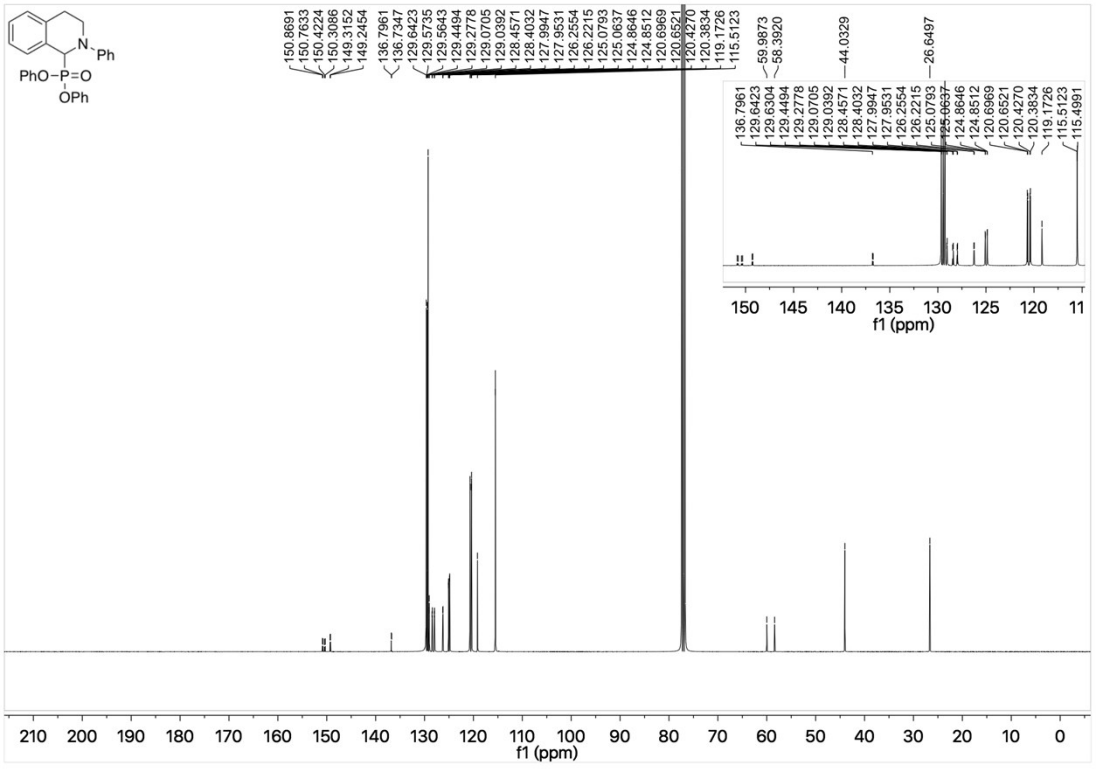
7.0504
7.0397
7.0281
7.0187
7.0077
6.8731
6.8529
6.8344
5.8163
5.6152
5.5654
4.0895
4.0769
4.0682
4.0568
4.0451
4.0363
4.0238
3.6887
3.6750
3.6595
3.6432
3.6291
3.1030
3.0907
3.0811
3.0629
3.0551
3.0504
3.0411
3.0286
3.0197
3.0125
3.0058
2.9991
2.9870
2.9797
2.9717
2.9660
2.9587
N
Ph
PhO–P=O
OPh
0.99
9.88
4.67
2.69
1.00
1.11
1.14
2.12
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)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Ph
PhO–P=O
OPh
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149.2454
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136.7347
129.6423
129.5735
129.5643
129.4494
129.2778
129.0705
129.0392
128.4571
128.4032
127.9947
127.9531
126.2554
126.2215
125.0793
125.0637
124.8646
124.8512
120.6969
120.6521
120.4270
120.3834
119.1726
115.5123
59.9873
58.3920
44.0329
26.6497
136.7961
129.6423
129.6304
129.4494
129.2778
129.0705
129.0392
128.4571
128.4032
127.9947
127.9531
126.2554
126.2215
125.0793
125.0637
124.8646
124.8512
120.6969
120.6521
120.4270
120.3834
119.1726
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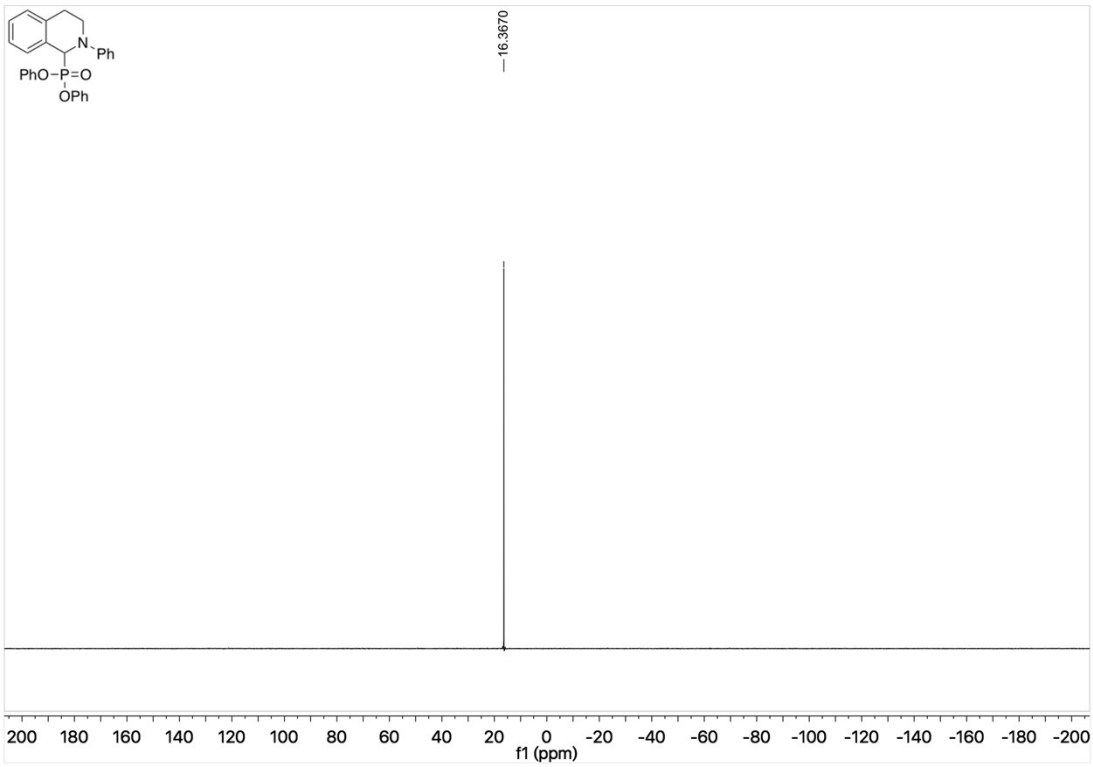
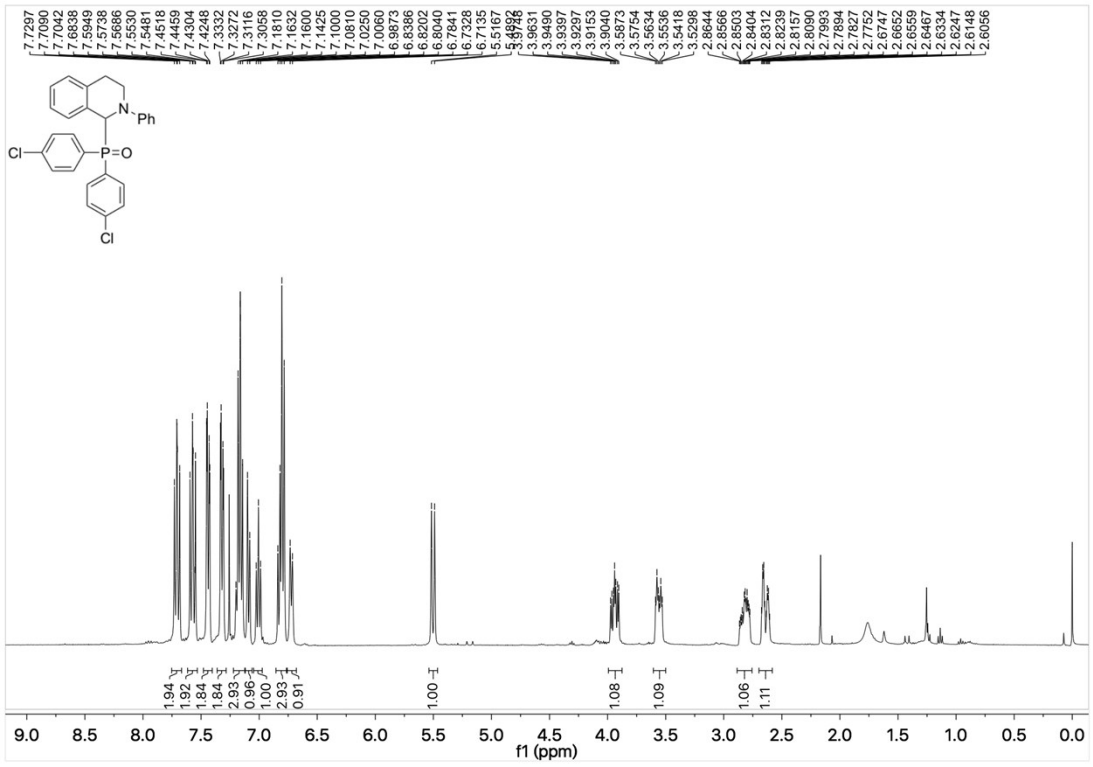
115.4991
150 145 140 135 130 125 120 11
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


16.3670
PhO–P=O
OPh
N
Ph
200 180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200
f1 (ppm)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7.3116
7.3058
7.1810
7.1632
7.1600
7.1425
7.1000
7.0810
7.0250
7.0060
6.9873
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6.7135
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3.9631
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3.9397
3.9297
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3.9040
3.5873
3.5754
3.5634
3.5536
3.5418
3.5298
2.8644
2.8566
2.8503
2.8404
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2.7894
2.7827
2.7752
2.6747
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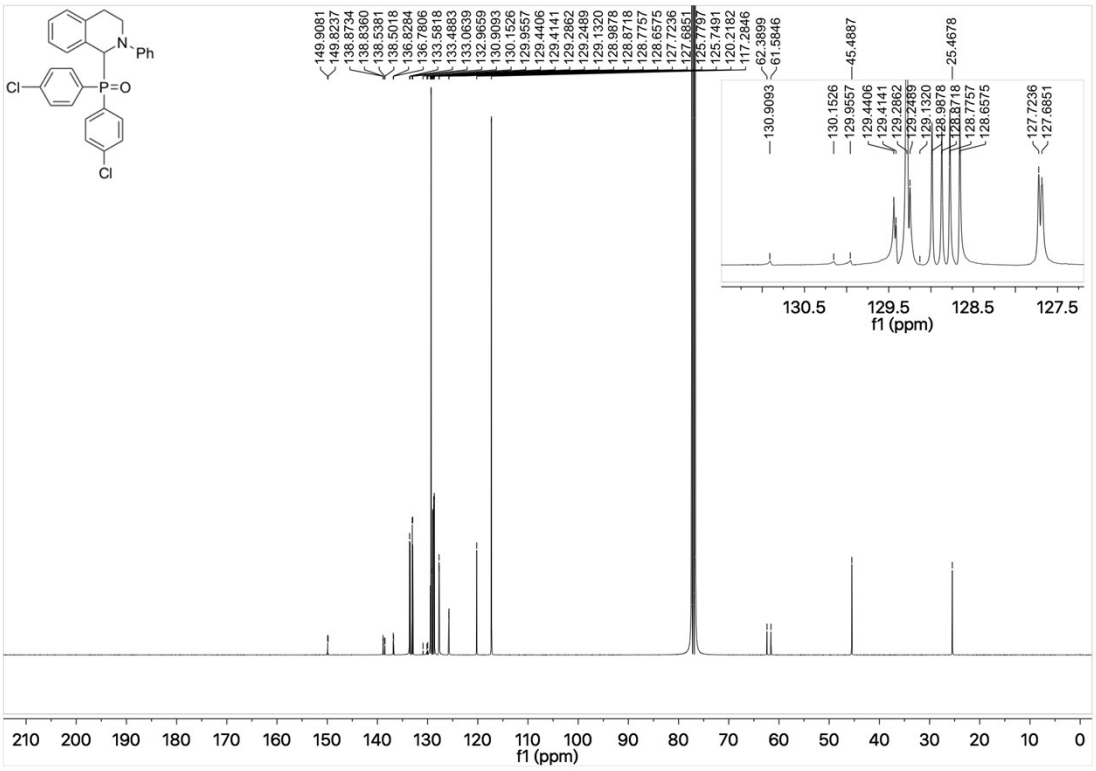
2.6559
2.6467
2.6334
2.6247
2.6148
2.6056
N
Ph
Cl
P=O
Cl
1.94
1.92
1.84
1.84
2.93
0.96
1.00
2.93
0.91
1.00
1.08
1.09
1.06
1.11
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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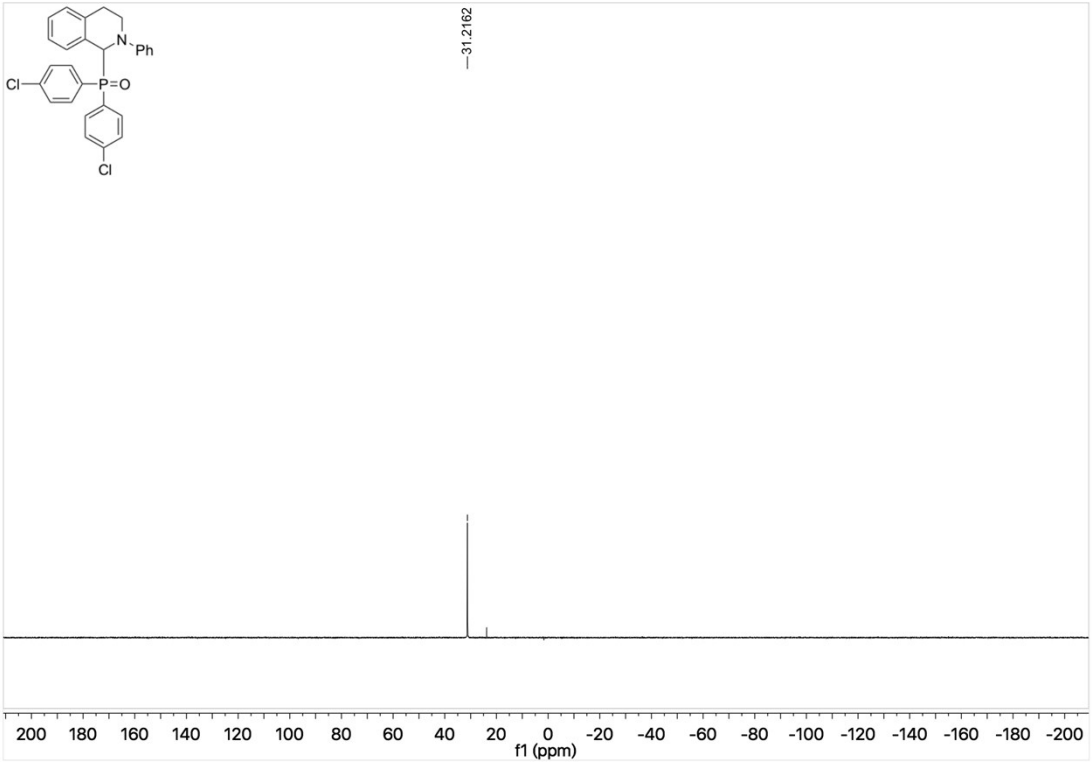
138.8734
138.8360
138.5381
138.5018
136.8284
136.7806
133.5818
133.4883
133.0639
132.9659
130.9093
130.1526
129.9557
129.4406
129.4141
129.2862
129.2485
129.1320
128.9878
128.8718
128.7757
128.6575
127.7236
127.6851
125.7797
125.7491
120.2182
117.2846
62.3899
61.5846
45.4887
25.4678
N
Ph
Cl
P=O
Cl
130.5
129.5
128.5
127.5
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


31.2162
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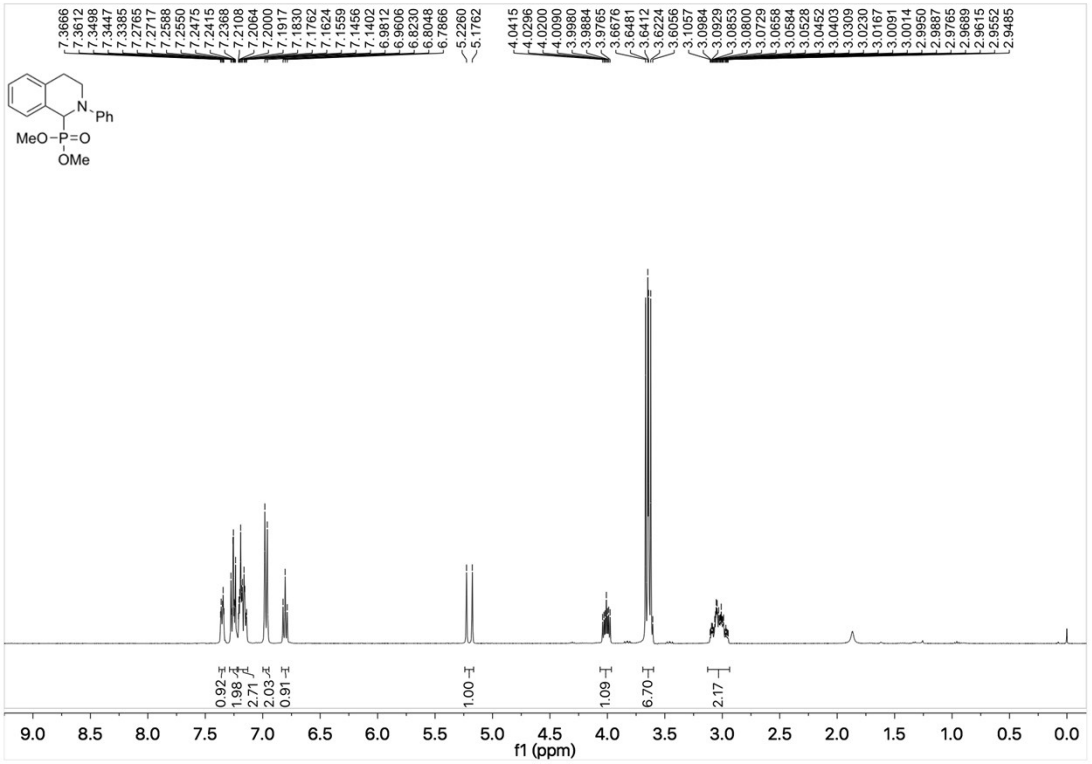
Ph
Cl
P=O
Cl
200 180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200
f1 (ppm)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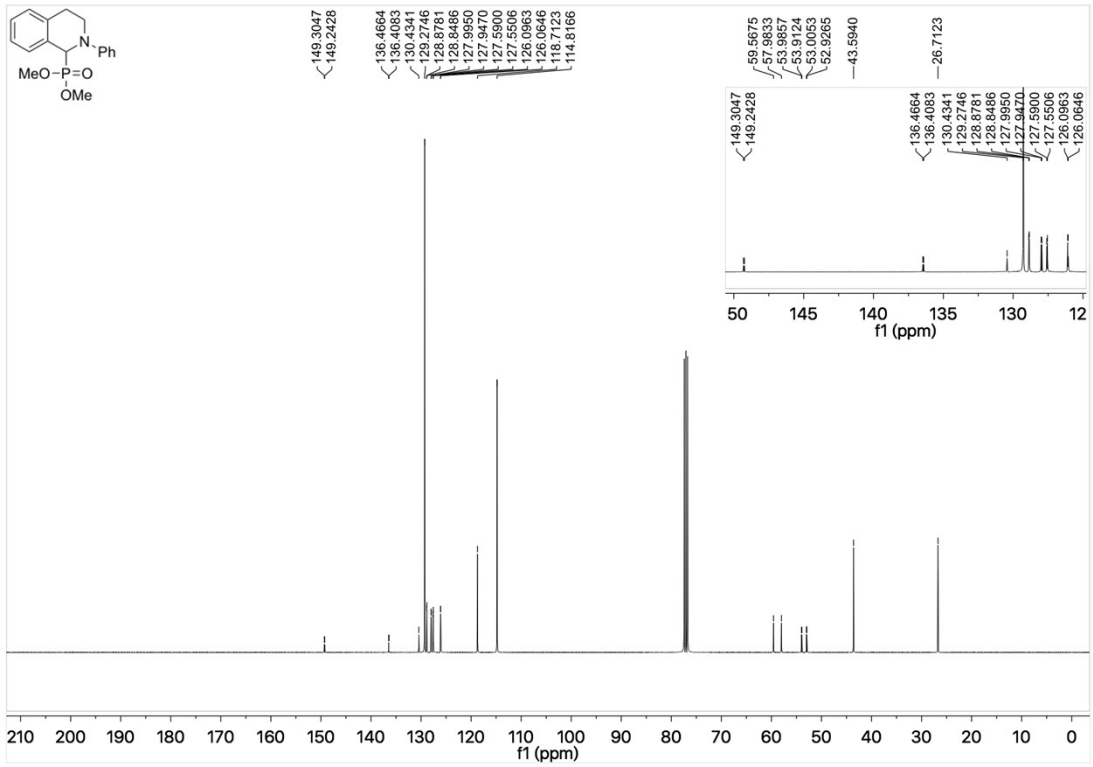
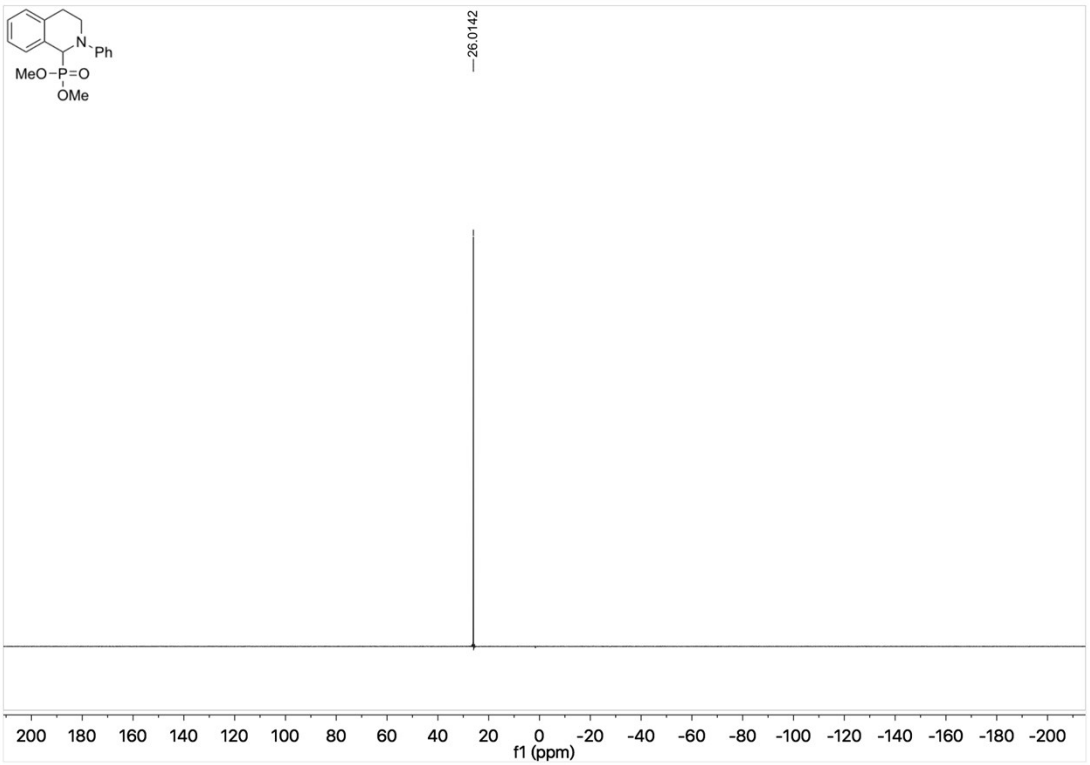
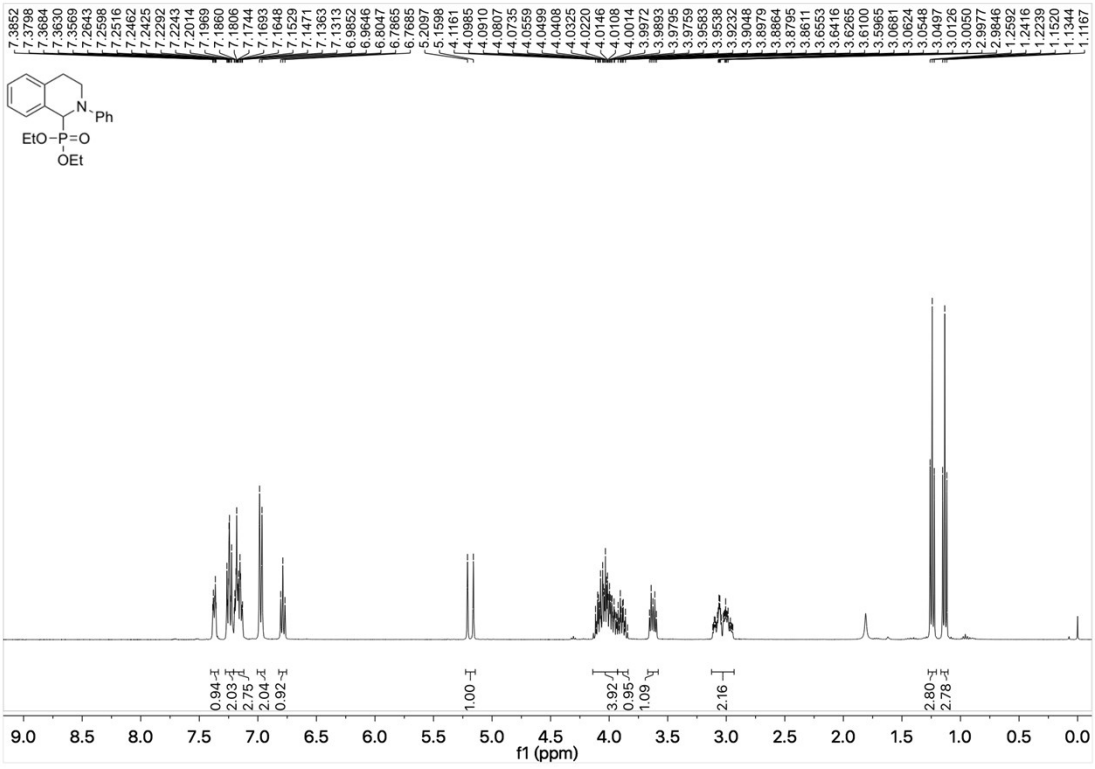
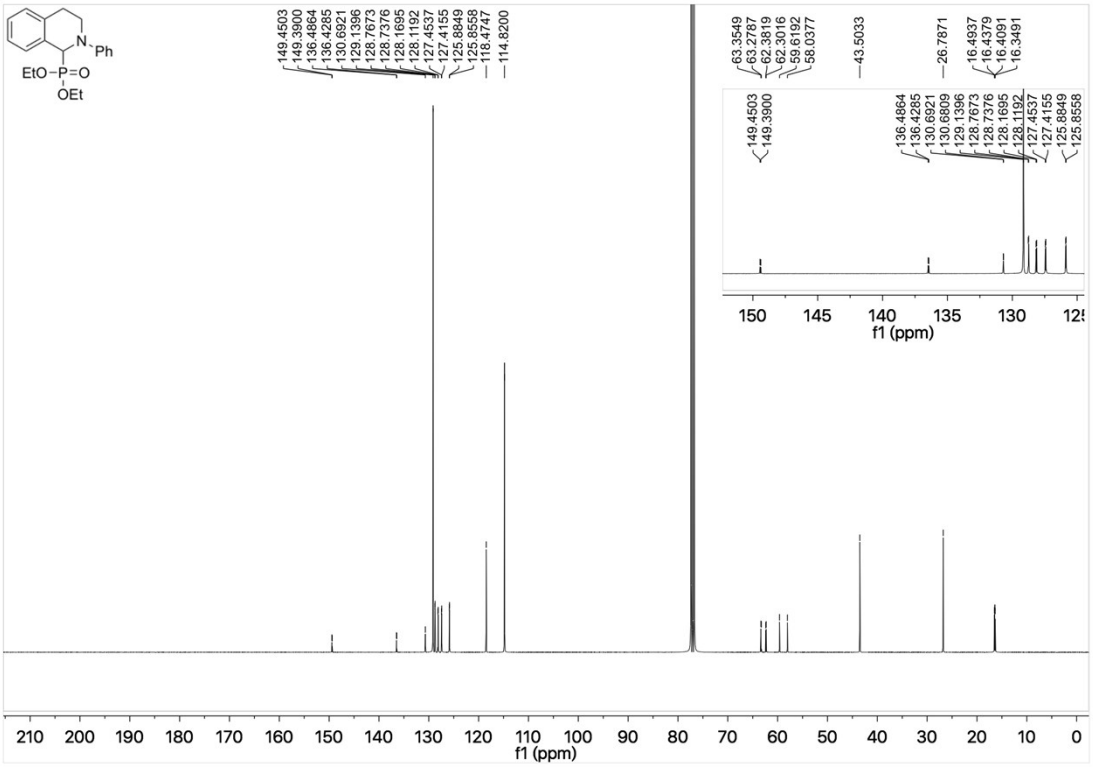
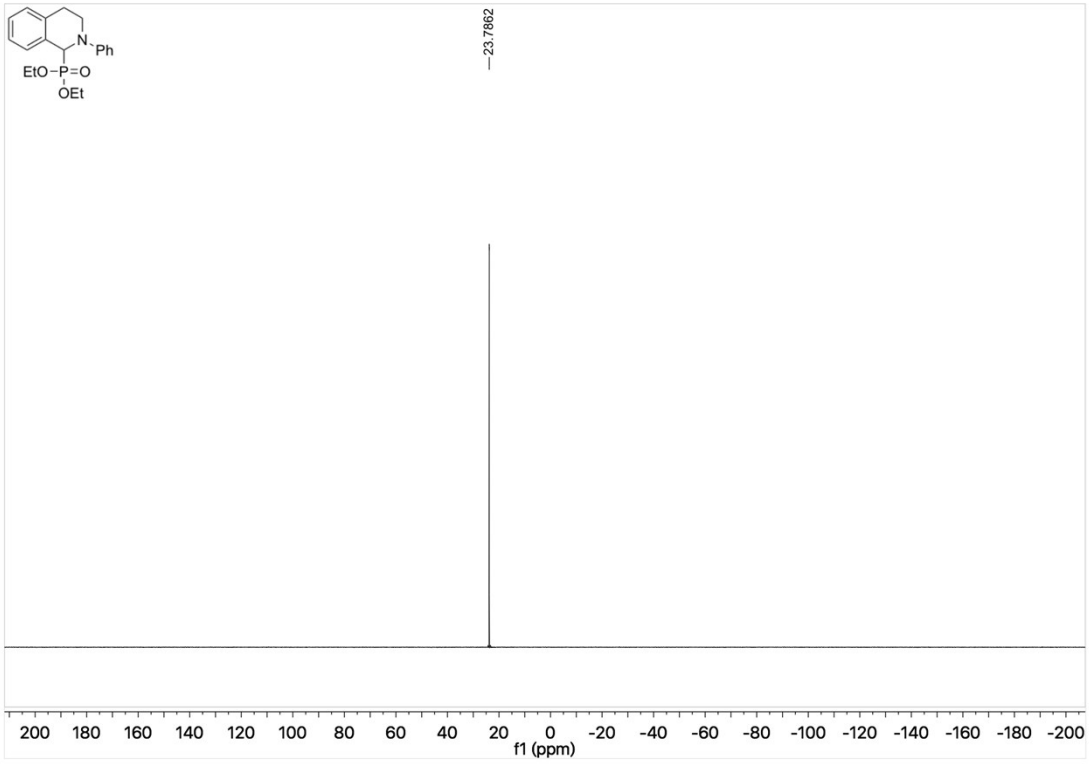
7.3612
7.3498
7.3447
7.3385
7.2765
7.2717
7.2588
7.2550
7.2475
7.2415
7.2368
7.2108
7.2064
7.2000
7.1917
7.1830
7.1762
7.1624
7.1559
7.1456
7.1402
6.9812
6.9606
6.8230
6.8048
6.7866
5.2260
5.1762
4.0415
4.0296
4.0200
4.0090
3.9980
3.9884
3.9765
3.6676
3.6481
3.6412
3.6224
3.6056
3.1057
3.0984
3.0929
3.0853
3.0800
3.0729
3.0658
3.0584
3.0528
3.0452
3.0403
3.0309
3.0230
3.0167
3.0091
3.0014
2.9950
2.9887
2.9765
2.9689
2.9615
2.9552
2.9485
N
Ph
MeO–P=O
OMe
0.92
1.98
2.71
2.03
0.91
1.00
1.09
6.70
2.17
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)

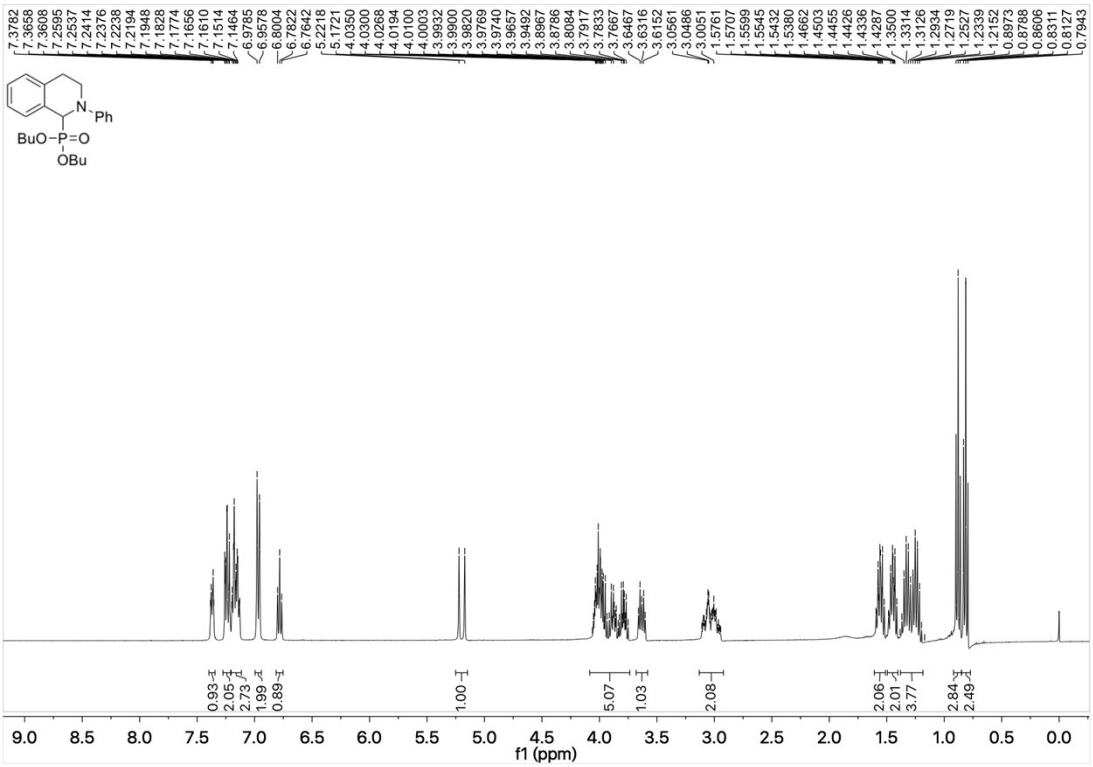

N
Ph
MeO–P=O
OMe
149.3047
149.2428
136.4664
136.4083
130.4341
129.2746
128.8781
128.8486
127.9950
127.9470
127.5900
127.5506
126.0963
126.0646
118.7123
114.8166
59.5675
57.9833
53.9857
53.9124
53.0053
52.9265
43.5940
26.7123
50 145 140 135 130 12
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

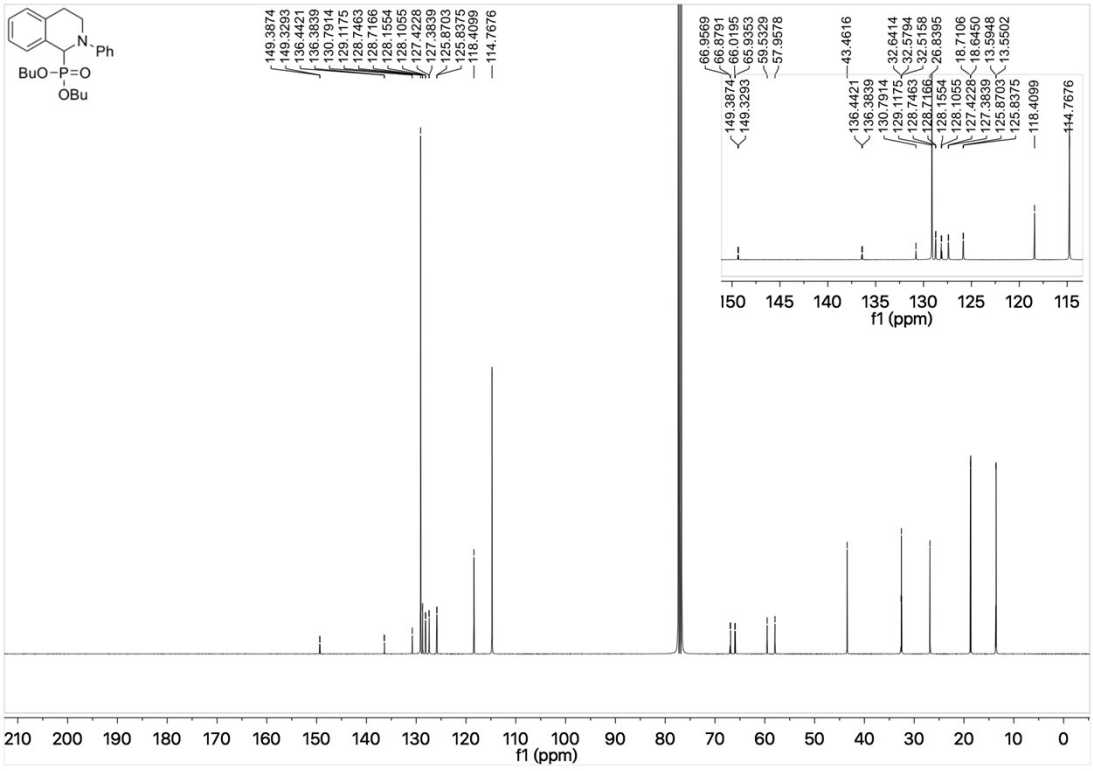

26.0142
MeO–P=O
OMe
Ph
200 180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200
f1 (ppm)

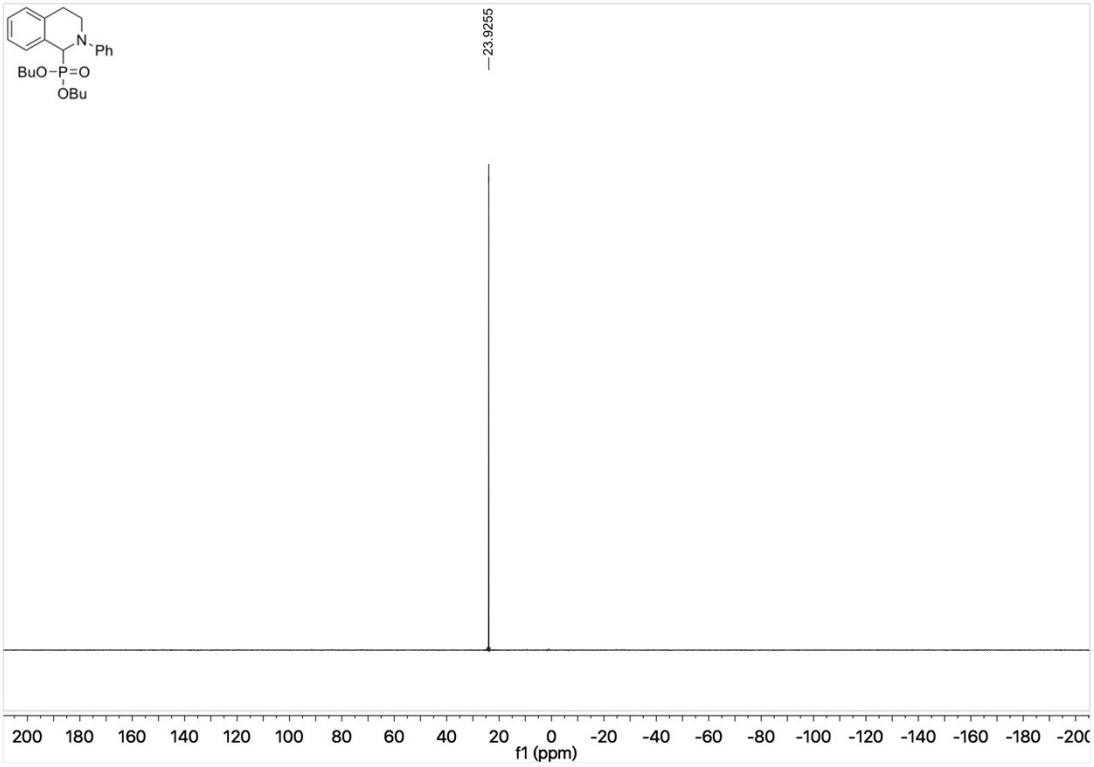

EtO–P=O
OEt
Ph
0.94
2.03
2.75
2.04
0.92
1.00
3.92
0.95
1.09
2.16
2.80
2.78
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


149.4503
149.3900
136.4864
136.4285
130.6921
129.1396
128.7673
128.7376
128.1695
128.1192
127.4537
127.4155
125.8849
125.8558
118.4747
114.8200
63.3549
63.2787
62.3819
62.3016
59.6192
58.0377
43.5033
26.7871
16.4937
16.4379
16.4091
16.3491
EtO–P=O
OEt
Ph
f1 (ppm)


23.7862
EtO–P=O
OEt
Ph
f1 (ppm)


7.3782
7.3658
7.3608
7.2595
7.2537
7.2414
7.2376
7.2238
7.2194
7.1948
7.1828
7.1774
7.1656
7.1610
7.1514
7.1464
6.9785
6.9578
6.8004
6.7822
6.7642
5.2218
5.1721
4.0350
4.0300
4.0268
4.0194
4.0100
4.0003
3.9932
3.9900
3.9820
3.9769
3.9740
3.9657
3.9492
3.8967
3.8786
3.8084
3.7917
3.7833
3.7667
3.6467
3.6316
3.6152
3.0561
3.0486
3.0051
1.5761
1.5707
1.5599
1.5545
1.5432
1.5380
1.4662
1.4503
1.4455
1.4426
1.4336
1.4287
1.3500
1.3314
1.3126
1.2934
1.2719
1.2527
1.2339
1.2152
0.8973
0.8788
0.8606
0.8311
0.8127
0.7943
Ph
BuO–P=O
OBu
0.93
2.05
2.73
1.99
0.89
1.00
5.07
1.03
2.08
2.06
2.01
3.77
2.84
2.49
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)


Ph
BuO–P=O
OBu
149.3874
149.3293
136.4421
136.3839
130.7914
129.1175
128.7463
128.7166
128.1554
128.1055
127.4228
127.3839
125.8703
125.8375
118.4099
114.7676
66.9569
66.8791
66.0195
65.9353
59.5329
57.9578
43.4616
32.6414
32.5794
32.5158
26.8395
18.7106
18.6450
13.5948
13.5502
150 145 140 135 130 125 120 115
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


23.9255
N
Ph
BuO–P=O
OBu
200 180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200
f1 (ppm)