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**Copper-Catalyzed Phosphorylation of $sp^2$ C − H Bonds**

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# SUPPORTING INFORMATION

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**General Considerations.** Reactions were performed without special precautions in 1-dram screw-cap vials equipped with a stir bar. For chromatography, 200-300 mesh silica gel (Qingdao, China) was employed. Preparative thin layer chromatography was performed on Analtech TLC plates (20 cm × 20 cm, 20 microns). $^{1}H$, $^{13}C$ and $^{31}P$ NMR spectra were recorded on an Bruker Avance 400 MHz instrument in deuterated chloroform $CDCl_3$ using TMS or residual solvent peak as a standard. Mass spectroscopy (ESI-MS) and High Resolution Mass spectroscopy (HRMS) data were recorded on a Finnigan $LCQ^{DECA}$ and a Bruker Daltonics Bio TOF mass spectroscopy respectively. Fluorescence emission spectra were obtained using FluoroMax-4 Spectrofluorophotometer (HORIBA Jobin Yvon) at 298 K. All solvents were dried according to the standard methods prior to use. All of the solvents were either HPLC or spectroscopic grade in the optical spectroscopic studies.

**Materials.** The following starting materials were obtained from commercial sources and were used without further purification: 8-aminoquinoline, benzoyl chloride, 4-(trifluoromethyl)benzoyl chloride, 4-nitrobenzoyl chloride, 4-methylbenzoic acid, 4-methoxybenzoic acid, 4-fluorobenzoic acid, 4-chlorobenzoic acid, 4-bromobenzoic acid, 4-iodobenzoic acid, 4-cyanobenzoic acid, monomethyl terephthalate, 3-methylbenzoic acid, 3-methoxybenzoic acid, 3-chlorobenzoic acid, 3-bromobenzoic acid, 3-iodobenzoic acid, 2-methylbenzoic acid, isonicotinic acid, thiophene-3-carboxylic acid, quinolin-8-ol, naphthalen-1-amine, pyridin-2-ylmethanol, picolinic acid, aniline, 2-(methylthio)aniline, diisopropyl phosphonate, diethyl phosphonate, dihexyl phosphonate, dibenzyl phosphonate, diphenyl phosphonate.

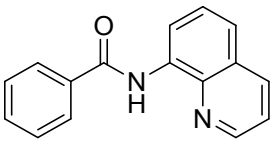
**Synthesis Of Amides.** All amides bearing 8-aminoquinoline moiety were prepared by the reaction of the corresponding acid chlorides with 8-aminoquinoline. All amides bearing 8-aminoquinoline moiety were prepared by the reaction of the corresponding acid chlorides with 8-aminoquinoline. 2-Methyl-N-phenylbenzamide (CAS 7055-03-0) was also prepared using the same procedure. 2-Methyl-N-(8-quinolinyl)benzamide (CAS 1182669-71-1), 3-methyl-N-(8-quinolinyl)benzamide (CAS 443638-87-7), N-(8-quinolinyl)-1-naphthamide (CAS 443735-56-6), 2-Methyl-N-(2-pyridinylmethyl)benzamide was prepared by previously reported procedure.[1], [2]

**General Procedure for the Preparation of Starting Amide.**

To an oven-dried 100 mL three-necked flask, 3-acetylbenzoic acid (2.5 g, 15 mmol), DMF (5 drops) and DCM (30 mL) were added under a $N_2$ atmosphere. Oxalyl chloride (1.5 mL, 18 mmol, 1.2 equiv.) was added dropwise at 0 °C. The reaction was detected by TLC, and the solvent was then removed in vacuo. The resulting acid chloride was used immediately without further purification.

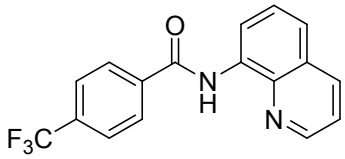
To another oven-dried 100 mL three-necked flask, 8-aminoquinoline (2.9 g, 20 mmol, 1.3 equiv.), $Et_3N$ (4.1 mL, 30 mmol, 2 equiv.) and DCM (30 mL) were added. A solution of the acid chloride in DCM (10 mL) was added dropwise to the solution at 0 °C, and the solution was then warmed to room temperature. After stirring overnight, the reaction system was quenched with sat. aq. $NaHCO_3$ (30 mL) and the organic layer was separated. The aqueous layer was extracted with DCM (2 x 15 mL). The combined organic layers were washed with 1 M HCl aq. (30 mL) and brine (30 mL), dried over $MgSO_4$ , filtered and evaporated in vacuo. The obtained crude amide was purified by column chromatography on silica gel (eluant: petroleum ether /EtOAc = 3/1) to afford the desired amide as a white solid (3.6 g, 82%).

**N-(quinolin-8-yl)benzamide. (1a)**



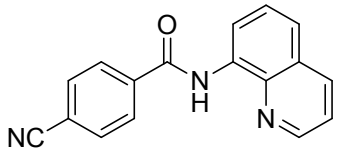

$R_f$ = 0.49 (petroleum ether /EtOAc = 5/1).White Solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.78 (s, 1H), 8.97 (d, *J* = 7.6 Hz, 1H), 8.88 (d, *J* = 4.0 Hz, 1H), 8.22 (d, *J* = 8.0 Hz, 1H), 8.12 (d, *J* = 7.6 Hz, 2H), 7.65–7.57 (m, 5H), 7.53–7.50 (m, 1H).

**N-(quinolin-8-yl)-4-(trifluoromethyl)benzamide. (1h)**



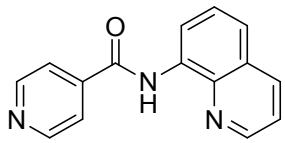

$R_f$ 0.56 (petroleum ether /EtOAc = 5/1).White Solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.83 (s, 1H), 8.95 (dd, *J* = 7.2, 1.6 Hz, 1H), 8.88 (dd, *J* = 8, 1.6 Hz, 1H), 8.26-8.21 (m, 3H), 7.85 (d, *J* = 8.4 Hz, 2H), 7.67 – 7.61 (m, 2H), 7.53 (dd, *J* = 8.4, 4.0 Hz, 1H).

**4-cyano-N-(quinolin-8-yl)benzamide. (1j)**



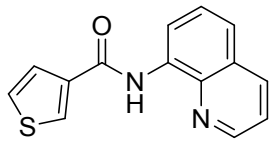
$R_f$ 0.32 (petroleum ether /EtOAc = 5/1).White Solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.82 (s, 1H), 8.94-8.88 (m, 2H), 8.26-8.20 (m, 3H), 7.88 (d, *J* = 8.4 Hz, 2H), 7.67 – 7.61 (m, 2H), 7.54 (dd, *J* = 8.4, 4.0 Hz, 1H).

**N-(quinolin-8-yl)isonicotinamide. (1r)**



$R_f$ 0.26 (petroleum ether /EtOAc = 1/1).White Solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.85 (s, 1H), 8.95-8.89 (m, 4H), 8.25 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.94 (dd, *J* = 4.4, 1.6 Hz, 2H), 7.67 – 7.61 (m, 2H), 7.54 (dd, *J* = 8.0, 4.0 Hz, 1H).

**N-(quinolin-8-yl)thiophene-3-carboxamide. (1s)**



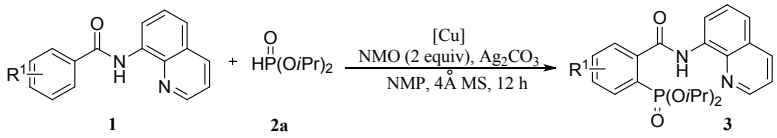
$R_f$ 0.46 (petroleum ether /EtOAc = 5/1).White Solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.58 (s, 1H), 8.92 – 8.87 (m, 2H), 8.23-8.18 (m, 2H), 7.72 (dd, *J* = 4.8, 1.2 Hz, 1H), 7.64 – 7.49 (m, 2H), 7.46(dd, *J* = 6.0, 3.2 Hz, 2H).

**Optimization Of Reaction Conditions.**

General procedure for reactions using NMO oxidant (Table S1, Table S2, Table S3 (entry 1), Table S4): a 1-dram vial equipped with a stir bar was charged with N-(quinolin-8-yl)benzamide (50 mg, 0.2 mmol, 1 equiv), catalyst (20 mol%), $Ag_2CO_3$ (1 equiv, 55 mg),Inside the glove box, NMO (N-methylmorpholine N-oxide) (47 mg, 0.4 mmol, 2 equiv) was added to the vial. Outside the glove box, solvent (0.8 mL) and diisopropyl phosphonate (66 uL, 0.4 mmol, 2 equiv) were added to the resulting mixture. The resulting mixture was heated with stirring at 55 °C for 12h. After completion, the reaction mixture was cooled down to room temperature. The mixture was washed with ethyl acetate or $CH_2Cl_2$ (2 × 25 mL), and the combined extracts were washed with $H_2O$ (15.0 mL) and brine (15.0 mL).

General procedure for reactions with other oxidant (entries 2-10, table S3): 1-dram vial equipped with a stir bar was charged with N-(quinolin-8-yl)benzamide (50 mg, 0.2 mmol, 1 equiv), catalyst (20 mol%), $Ag_2CO_3$ (1 equiv, 55 mg), solvent (0.8 mL) and diisopropyl phosphonate (66 uL, 0.4 mmol, 2 equiv). The resulting mixture was heated with stirring at 55 °C for 12h. After completion, the reaction mixture was cooled down to room temperature. The mixture was washed with ethyl acetate (2 × 25 mL), and the combined extracts were washed with $H_2O$ (15.0 mL) and brine (15.0 mL).

**Table S1** Optimization of Cu catalysts.[a]

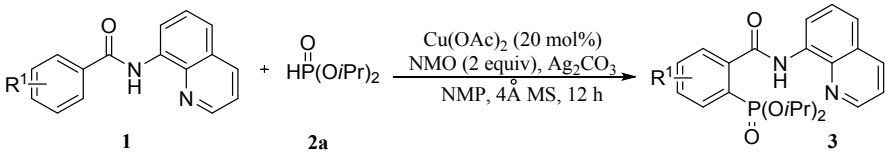



1 2a 3

| Entry | Catalyst | Yield (%)[b] |
|---|---|---|
| 1 | CuCl | 20 |
| 2 | $CuCl_2$ | 29 |
| 3 | CuCN | 10 |
| 4 | CuI | 16 |
| 5 | CuBr | 27 |
| 6 | CuAcAc | 18 |
| 7 | $Cu(NO_3)_2 \cdot 3H_2O$ | 13 |
| 8 | $Cu(OAc)_2$ | 30 |
| 9[c] | $Cu(OAc)_2$ | 21 |
| 10[d] | $Cu(OAc)_2$ | 13 |
| 11[e] | $Cu(OAc)_2$ | Trace |

[a] Reaction conditions: amide (0.2 mmol), catalyst (20 mol%), NMO (2 equiv), $Ag_2CO_3$ (2 equiv), 4Å MS (100mg), NMP (0.8 mL) and diisopropyl phosphonate (2 equiv), 120 °C, 12 h. [b] Isolated yield. [c] $Cu(OAc)_2$ (10 mol%, 3.6 mg). [d] $Cu(OAc)_2$ (50 mol%, 14.4 mg). [e] $Cu(OAc)_2$ (1 equiv, 28.8 mg).

**Table S2** Optimization of reaction temperature.[a]

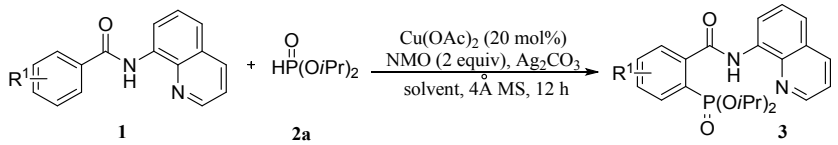



1 2a 3

| Entry | Temperature (°C) | Yield(%)[b] |
|---|---|---|
| 1 | 10 | N.R |
| 2 | 20 | Trace |
| 3 | 30 | 14 |
| 4 | 40 | 33 |
| 5 | 50 | 59 |
| 6 | 55 | 59 |
| 7 | 60 | 58 |
| 8 | 70 | 43 |
| 9 | 80 | 43 |
| 10 | 90 | 41 |
| 11 | 120 | 30 |
| 12 | 140 | 23 |
| 13[c] | 55 | 70 |

[a] Reaction conditions: amide (0.2 mmol), $Cu(OAc)_2$ (20 mol%), NMO (2 equiv), $Ag_2CO_3$ (2 equiv), 4Å MS (100mg), NMP (0.8 mL) and diisopropyl phosphonate (2 equiv), 12 h. [b] Isolated yield. [c] The mixture was stirred at 15 °C for 30 min before diisopropyl phosphonate was added. The reaction was transferred and carried out under 55 °C immediately.

**Table S3** Optimization of solvents.[a]



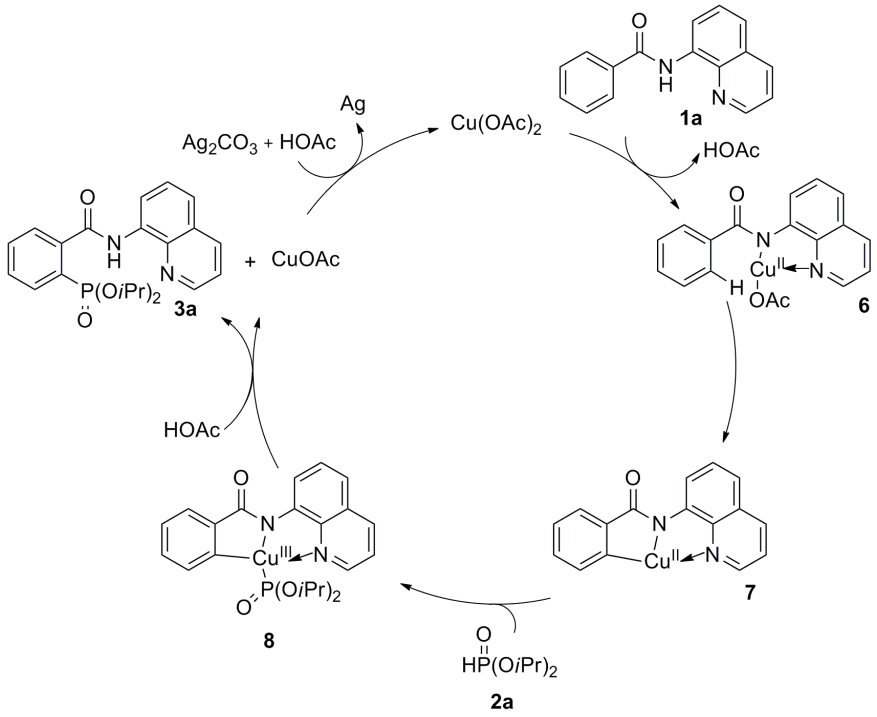

**1** **2a** **3**

| Entry | Solvent (0.8 mL) | Yield (%)[b] |
|---|---|---|
| 1 | $H_2O$ | N.R |
| 2 | EA | 50 |
| 3 | $CHCl_3$ | 41 |
| 4 | THF | 57 |
| 5 | dimethyl sulfoxide (DMSO) | 78 |
| 6 | $CH_3CN$ | 56 |
| 7 | 1,2-dichloroethane (DCE) | 26 |
| 8 | N,N-dimethylformamide (DMF) | 71 |
| 9 | N,N-dimethylacetamide (DMA) | 66 |
| 10 | toluene | 26 |
| 11 | benzene | 39 |
| 12 | 1,4-dioxane | 47 |
| 13 | *tert*-Butanol | 39 |

[a] Reaction conditions: amide (0.2 mmol), $Cu(OAc)_2$ (20 mol%), NMO (2 equiv), $Ag_2CO_3$ (2 equiv), 4Å MS (100mg), solvent (0.8 mL) and diisopropyl phosphonate (2 equiv), 55 °C, 12 h. The mixture was stirred at 15 °C for 30 min before diisopropyl phosphonate was added. The reaction was transferred and carried out under 55 °C immediately. [b] Isolated yield.

**General Procedure For $Cu(OAc)_2$-Catalyzed Phosphorylation.** A 1-dram vial equipped with a stir bar was charged with N-(quinolin-8-yl)benzamide (50 mg, 0.2 mmol, 1 equiv), $Cu(OAc)_2$ (20 mol%), $Ag_2CO_3$ (1 equiv, 55 mg),Inside the glove box, NMO (N-methylmorpholine N-oxide) (47 mg, 0.4 mmol, 2 equiv) was added to the vial. Outside the glove box, DMSO (0.8 mL) were added to the resulting mixture. The mixture was stirred at 15 °C for 30 min before diisopropyl phosphonate (66 uL, 0.4 mmol, 2 equiv) was added. The reaction was transferred and carried out under 55 °C immediately. The vial was heated with stirring at 55 °C for 12h. After completion, the reaction mixture was cooled down to room temperature. The mixture was washed with $CH_2Cl_2$ (2 × 25 mL), and the combined extracts were washed with $H_2O$ (15.0 mL) and brine (15.0 mL). Purification by column chromatography or preparative thin layer chromatography provided the desired product.

**Proposed Reaction Mechanism.**

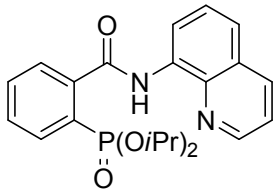



**Scheme S1** Proposed reaction mechanism.

The catalytic cycle was proposed in Scheme S1. Coordination of the amide **1a** to the copper center gives the copper complex **6** followed by ligand exchange with the concomitant generation of HOAc, which undergoes cyclometalation to give the complex **7**. The oxidative addition of diisopropyl phosphonate gives **8**, which experiences reductive elimination and protonation to give the phosphorylation product **3a** and CuOAc. Then, copper (II) was regenerated upon the oxidation of $Ag_2CO_3$.

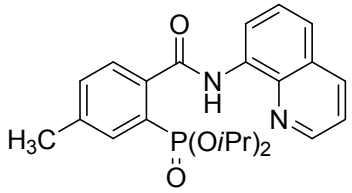
**Characterization Data for Products**

**diisopropyl (2-(quinolin-8-ylcarbamoyl)phenyl)phosphonate. (3a)**



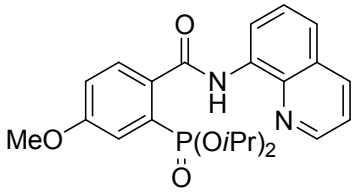
$R_f$ = 0.37 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.18 (s, 1H), 8.97 (dd, *J* = 7.4, 1.2 Hz, 1H), 8.80 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.20 (dd, *J* = 8.2, 1.6 Hz, 1H), 8.15 - 8.09 (m, 1H), 7.78 - 7.70 (m, 1H), 7.69 - 7.64 (m, 1H), 7.63 - 7.53 (m, 3H), 7.48 - 7.45 (m, 1H), 4.80 - 4.75 (m, 2H), 1.26 (d, *J* = 6.0 Hz, 6H), 1.18 (d, *J* = 6.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 167.47 (d, *J* = 4.5 Hz), 148.2, 140.85 (d, *J* = 9.3 Hz), 138.75, 136.3, 134.75, 133.47 (d, *J* = 9.3), 132.28 (d, *J* = 2.8 Hz), 129.49 (d, *J* = 14.1 Hz), 128.60 (d, *J* = 12.9 Hz), 128.30, 128.03, 127.40, 126.43, 121.97, 121.61, 117.22, 71.52 (d, J = 6.0 Hz), 23.82 (d, *J* = 4.3 Hz) 23.75 (d, *J* = 4.8 Hz). $^{31}$P NMR (162 MHz, $CDCl_3$) δ 14.39. HRMS (ESI+): Calculated for $C_{22}H_{25}N_2O_4P$ $[M+H]^+$ 413.1630, Found 413.1629.

**diisopropyl (5-methyl-2-(quinolin-8-ylcarbamoyl)phenyl)phosphonate. (3b)**



$R_f$ = 0.43 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.14 (s, 1H), 8.93 (d, *J* = 7.4 Hz, 1H), 8.81 - 8.71 (m, 1H), 8.18 (d, *J* = 8.0 Hz, 1H), 7.92 (d, *J* = 14.8 Hz, 1H), 7.64 - 7.55 (m, 3H), 7.46 - 7.44 (m, 2H), 4.79 - 4.72 (m, 2H), 2.48 (s, 3H), 1.25 (d, *J* = 6.0 Hz, 6H), 1.15 (d, *J* = 6.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 167.61 (d, *J* = 4.3 Hz), 148.15, 139.74 (d, *J* = 14.1 Hz), 138.72, 138.06 (d, *J* = 9.3 Hz), 136.24, 134.77, 134.00 (d, *J* = 9.54 Hz), 132.85 (d, *J* = 3.1 Hz), 128.69 (d, *J* = 13.5 Hz), 127.99, 127.36, 126.06, 121.84, 121.55, 117.15, 71.46 (d, *J* = 6.0 Hz), 23.75 (d, *J* = 3.7 Hz), 23.71 (d, *J* = 4.3 Hz), 21.26. $^{31}$P NMR (162 MHz, $CDCl_3$) δ 14.93. HRMS (ESI+): Calculated for $C_{23}H_{27}N_2O^4P$ $[M+H]^+$ 427.1787, Found 427.1783.

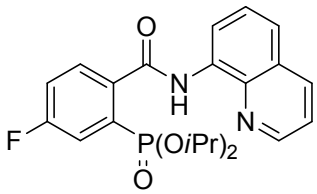
**diisopropyl (5-methoxy-2-(quinolin-8-ylcarbamoyl)phenyl)phosphonate. (3c)**



$R_f$ = 0.36 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.17 (s, 1H), 8.92 (d, *J* =7.2 Hz, 1H), 8.79 (dd, *J* = 4.0, 1.6 Hz, 1H), 8.19 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.69 (dd, *J* = 8.4, 6.0 Hz, 1H), 7.65 - 7.55 (m, 3H), 7.47 - 7.44 (m, 1H), 7.16 - 7.13 (m, 1H), 4.80 - 4.74 (m, 2H), 3.93 (s, 3H), 1.27 (d, *J* = 6.0 Hz, 6H), 1.17 (d, *J* = 6.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 167.34 (d, *J* = 4.3 Hz), 160.19, 160.02, 148.16, 138.78, 136.23, 134.82, 133.24 (d, *J* = 8.6 Hz), 130.63 (d, *J* = 15.2 Hz), 128.01, 127.35, 121.81, 121.54, 118.37 (d, *J* = 10.4 Hz), 117.77 (d, *J* = 2.9 Hz), 117.19, 71.64 (d, *J* = 6.1 Hz), 55.63, 23.77 (d, *J* = 4.6 Hz), 23.72 (d, J = 5.4 Hz). $^{31}$P NMR (162 MHz, $CDCl_3$) δ 14.32. HRMS (ESI+): Calculated for $C_{23}H_{27}N_2O_5P$ $[M+H]^+$ 443.1736, Found

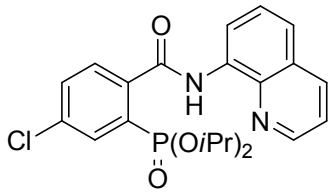
443.1730.

**diisopropyl (5-fluoro-2-(quinolin-8-ylcarbamoyl)phenyl)phosphonate. (3d)**



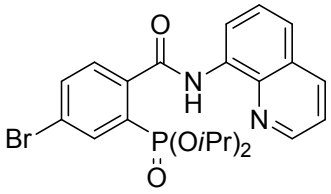
$R_f$ = 0.56 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.18 (s, 1H), 8.93 (dd, *J* = 7.4, 1.4 Hz, 1H), 8.80 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.20 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.85 - 7.69 (m, 2H), 7.67 - 7.55 (m, 2H), 7.47 (dd, *J* = 8.4, 4.2 Hz, 1H), 7.34 - 7.33 (m, 1H), 4.81 - 4.76 (m, 2H), 1.27 (d, *J* = 6.0 Hz, 6H), 1.17 (d, *J* = 6.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.51 (d, *J* = 4.1 Hz), 148.24, 138.72, 137.06 - 136.92 (m), 136.29, 134.60, 131.43 (d, *J* = 4.9 Hz), 131.25 - 131.03 (m), 128.1, 127.36, 122.08, 121.64, 120.61 - 120.27 (m), 119.26 (d, *J* = 4.1 Hz), 119.07, 117.27, 71.98 (d, *J* = 4.9 Hz), 23.77 (d, *J* = 4.1 Hz), 23.71 (d, *J* = 4.5 Hz) $^{31}$P NMR (162 MHz, $CDCl_3$) δ 12.28. HRMS (ESI+): Calculated for $C_{22}H_{24}FN_2O_4P$ $[M+H]^+$ 431.1536, Found 431.1533.

**diisopropyl (5-chloro-2-(quinolin-8-ylcarbamoyl)phenyl)phosphonate. (3e)**



$R_f$ = 0.61 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.17 (s, 1H), 8.92 (dd, *J* = 7.2, 1.6 Hz, 1H), 8.79 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.20 (dd, *J* = 8.4, 1.6 Hz, 1H), 8.09 (dd, *J* = 14.6, 2.0 Hz, 1H), 7.68 (dd, *J* = 8.2, 5.4 Hz, 1H), 7.65 - 7.55 (m, 3H), 7.47 (dd, *J* = 8.4, 4.2 Hz, 1H), 4.80 - 4.75 (m, 2H), 1.27 (d, *J* = 6.0 Hz, 6H), 1.18 (d, *J* = 6.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.41 (d, *J* = 4.4 Hz), 148.25, 139.01 (d, *J* = 8.5 H), 138.67, 136.28, 134.52, 133.31 (d, *J* = 10.3 Hz), 130.59, 130.20 (d, *J* = 14.3 Hz), 138.73, 127.99, 127.34, 122.13, 121.65, 117.24, 71.99 (d, *J* = 6.0 Hz), 23.76 (d, *J* = 4.5 Hz), 23.71 (d, *J* = 5.1 Hz). $^{31}$P NMR (162 MHz, $CDCl_3$) δ 12.34. HRMS (ESI+): Calculated for $C_{22}H_{24}ClN_2O_4P$ $[M+H]^+$ 447.1240, Found 447.1239.

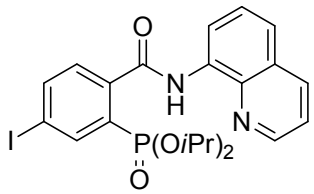
**diisopropyl (5-bromo-2-(quinolin-8-ylcarbamoyl)phenyl)phosphonate. (3f)**



$R_f$ = 0.60 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.16 (s, 1H), 8.92 (dd, *J* = 7.2, 1.6 Hz, 1H), 8.79 (dd, *J* = 4.4, 1.6 Hz, 1H), 8.25 (dd, *J* = 14.4, 2.0 Hz, 1H), 8.19 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.78 (dd, *J* = 8.2, 1.2 Hz, 1H), 7.65 - 7.52 (m, 3H), 7.47 (dd, *J* = 8.4, 4.2 Hz, 1H), 4.80 - 4.75 (m, 2H), 1.27 (d, *J* = 6.0 Hz, 6H), 1.17 (d, *J* = 6.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.44 (d, *J* = 4.3 Hz), 148.25, 139.44 (d, *J* = 8.7 Hz), 138.66, 136.28, 136.13 (d, *J* = 10.2 Hz), 135.23 (d, *J* = 2.8 Hz), 134.50, 130.79, 130.29 (d, *J* = 13.9 Hz), 128.86, 127.99, 127.33, 122.14, 121.65, 117.22, 72.00 (d, *J* = 6.0 Hz), 23.76 (d, *J* = 4.3 Hz), 23.71 (d, *J* = 5.0 Hz). $^{31}$P NMR (162 MHz, $CDCl_3$) δ 12.08. HRMS (ESI+): Calculated for $C_{22}H_{24}BrN_2O_4P$ $[M+H]^+$ 491.0735, Found

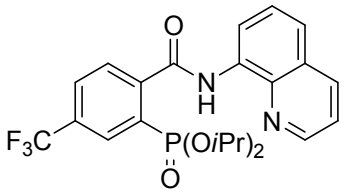
491.0736.

**diisopropyl (5-iodo-2-(quinolin-8-ylcarbamoyl)phenyl)phosphonate. (3g)**



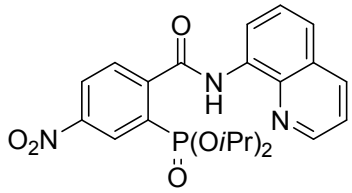
$R_f$ = 0.59 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.16 (s, 1H), 8.92 (dd, *J* = 7.2, 1.6 Hz, 1H), 8.79 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.44 (dd, *J* = 14.0, 1.6 Hz, 1H), 8.20 (dd, *J* = 8.4, 1.6 Hz, 1H), 8.01 - 7.99 (m, 1H), 7.61 - 7.59 (m, 2H), 7.48 - 7.43 (m, 2H), 7.34 - 7.33 (m, 1H), 4.79 - 4.74 (m, 2H), 1.27 (d, *J* = 6.0 Hz, 6H), 1.17 (d, *J* = 6.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.56 (d, *J* = 4.3 Hz), 148.24, 141.85 (d, *J* = 10.2 Hz), 141.19 (d, *J* = 2.6 Hz), 139.97 (d, *J* = 8.5 Hz), 138.66, 136.27, 134.51, 130.57, 130.15 (d, *J* = 3.3 Hz), 128.72, 127.99, 127.33, 122.12, 121.64, 117.22, 71.97 (d, *J* = 6.2 Hz), 23.74 (d, *J* = 4.3 Hz), 23.70 (d, *J* = 5.0 Hz). $^{31}$P NMR (162 MHz, $CDCl_3$) δ 11.72. HRMS (ESI+): Calculated for $C_{22}H_{24}IN_2O_4P$ $[M+H]^+$ 539.0597, Found 539.0591.

**diisopropyl (2-(quinolin-8-ylcarbamoyl)-5-(trifluoromethyl)phenyl)phosphonate. (3h)**



$R_f$ = 0.65 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.21 (s, 1H), 8.94 (dd, *J* = 7.2, 1.8 Hz, 1H), 8.80 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.37 (d, *J* = 14.4 Hz, 1H), 8.21 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.92 (d, *J* = 8.0 Hz, 1H), 7.86 (dd, *J* = 8.0, 4.4 Hz, 1H), 7.70 - 7.52 (m, 2H), 7.48 (dd, *J* = 8.4, 4.2 Hz, 1H), 4.84 - 4.75 (m, 2H), 1.27 (d, *J* = 6.4 Hz, 6H), 1.19 (d, *J* = 6.4 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.09 (d, *J* = 4.7 Hz), 148.33, 143.87 (d, *J* = 7.8 Hz), 138.64, 136.34, 134.38, 130.46 (d, *J* = 3.7 Hz), 130.36 (d, *J* = 3.3 Hz), 129.91, 129.31, 129.18, 129.07 - 128.99 (m), 128.02, 127.35, 122.33, 121.73, 117.31, 72.17 (d, *J* = 6.1 Hz), 23.78 (d, *J* = 3.7 Hz), 23.70 (d, *J* = 4.9 Hz). $^{31}$P NMR (162 MHz, $CDCl_3$) δ 12.13. HRMS (ESI+): Calculated for $C_{23}H_{24}F_3N_2O_4P$ $[M+H]^+$ 481.1504, Found 481.1501.

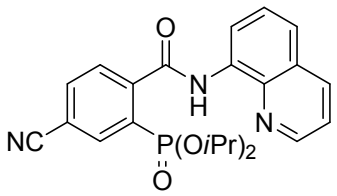
**diisopropyl (5-nitro-2-(quinolin-8-ylcarbamoyl)phenyl)phosphonate. (3i)**



$R_f$ = 0.59 (petroleum ether/EtOAc, 1:1). Yellow solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.26 (s, 1H), 8.94 - 8.92 (m, 1H), 8.89 (s, 1H), 8.80 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.48 (dd, *J* = 8.4, 1.6 Hz, 1H), 8.22 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.91 (dd, *J* = 8.4, 4.8 Hz, 1H), 7.69 - 7.58 (m, 2H), 7.49 (dd, *J* = 8.4, 4.2 Hz, 1H), 4.87 - 4.78 (m, 2H), 1.28 (d, *J* = 6.0 Hz, 6H), 1.22 (d, *J* = 6.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 165.32 (d, *J* = 4.1 Hz), 148.39, 148.90 (d, *J* = 9.1 Hz), 138.58, 136.36, 134.17, 131.22, 130.12 (d, *J* = 13.5 Hz), 129.33, 128.36 (d, *J* = 10.9 Hz), 128.01, 127.31, 126.85 (d, *J* = 2.5 Hz), 122.57, 121.79, 117.37, 72.55 (d, *J* = 6.3 Hz), 23.79 (d, *J* = 4.0 Hz), 23.71 (d, *J* = 5.0 Hz). $^{31}$P NMR (162 MHz, $CDCl_3$) δ 10.69. HRMS (ESI+): Calculated for $C_{22}H_{24}N_3O_6P$ $[M+H]^+$ 458.1481, Found

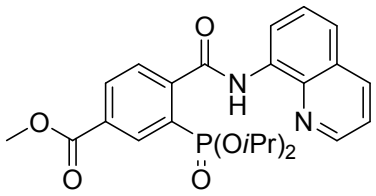
458.1477.

**diisopropyl (5-cyano-2-(quinolin-8-ylcarbamoyl)phenyl)phosphonate. (3j)**



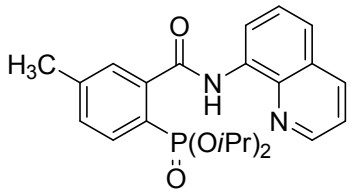
$R_f$ = 0.42 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.22 (s, 1H), 8.92 (dd, *J* = 6.4, 2.4 Hz, 1H), 8.80 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.39 (dd, *J* = 14.0, 1.6 Hz, 1H), 8.22 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.93 (dt, *J* = 8.0, 1.4 Hz, 1H), 7.84 (dd, *J* = 8.0, 4.4 Hz, 1H), 7.69 - 7.56 (m, 2H), 7.49 (dd, *J* = 8.4, 4.2 Hz, 1H), 4.82 - 4.78 (m, 2H), 1.28 (d, *J* = 6.0 Hz, 6H), 1.19 (d, *J* = 6.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 165.54 (d, *J* = 4.4 Hz), 148.37, 144.19 (d, *J* = 8.8 Hz), 138.59, 137.05 (d, *J* = 10.4 Hz), 136.38, 135.43 (d, *J* = 2.9 Hz), 134.21, 130.59, 129.46 (d, *J* = 12.7 Hz), 128.71, 128.02, 127.33, 122.52, 121.78, 117.33, 113.83 (d, *J* = 16.5 Hz), 72.45 (d, *J* = 6.1 Hz), 23.6 (d, *J* = 3.8 Hz), 23.72 (d, *J* = 4.4 Hz). $^{31}$P NMR (162 MHz, $CDCl_3$) δ 10.94. HRMS (ESI+): Calculated for $C_{23}H_{24}N_3O_4P$ $[M+H]^+$ 438.1583, Found 438.1580.

**methyl 3-(diisopropoxyphosphoryl)-4-(quinolin-8-ylcarbamoyl)benzoate. (3k)**



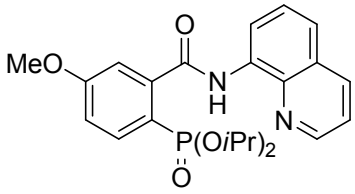
$R_f$ = 0.60 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.21 (s, 1H), 8.95 (d, *J* = 7.2 Hz, 1H), 8.83 - 8.67 (m, 2H), 8.31 (d, *J* = 7.8 Hz, 1H), 8.20 (d, *J* = 8.2 Hz, 1H), 7.81 (dd, *J* = 7.8, 4.8 Hz, 1H), 7.70 - 7.51 (m, 2H), 7.47 (dd, *J* = 8.2, 4.2 Hz, 1H), 4.89 - 4.70 (m, 2H), 4.00 (s, 3H), 1.27 (d, *J* = 6.2 Hz, 6H), 1.20 (d, *J* = 6.2 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.55 (d, *J* = 4.3 Hz), 165.67 (d, *J* = 1.4 Hz), 148.28, 144.44 (d, *J* = 9.6 Hz), 138.66, 136.31, 134.61, 134.49, 133.24 (d, *J* = 2.5Hz), 131.11 (d, *J* = 14.3 Hz), 129.11, 138,91 (d, *J* = 12.7 Hz), 128.01, 127.36, 122.22, 121.67, 117.30, 71.90 (d, *J* = 6.6 Hz), 52.51, 23.80 (d, *J* = 4.1 Hz), 23.71 (d, *J* = 5.3 Hz). $^{31}$P NMR (162 MHz, $CDCl_3$) δ 12.97. HRMS (ESI+): Calculated for $C_{24}H_{27}N_2O_6P$ $[M+H]^+$ 471.1685, Found 471.1681.

**diisopropyl (4-methyl-2-(quinolin-8-ylcarbamoyl)phenyl)phosphonate. (3l)**



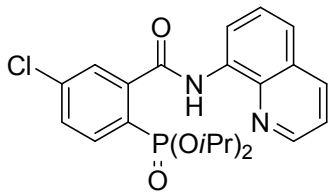
$R_f$ = 0.55 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.14 (s, 1H), 8.95 (d, *J* = 7.4 Hz, 1H), 8.84 - 8.76 (m, 1H), 8.19 (d, *J* = 8.2 Hz, 1H), 7.99 (dd, *J* = 13.8, 7.8 Hz, 1H), 7.69 - 7.50 (m, 3H), 7.50 - 7.43 (m, 1H), 7.39 (d, *J* = 7.8 Hz, 1H), 4.78 - 4.70 (m, 2H), 2.46 (s, 3H), 1.25 (d, *J* = 6.0 Hz, 6H), 1.15 (d, *J* = 6.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 167.64 (d, *J* = 4.4 Hz), 148.15, 142.96 (d, *J* = 3.0 Hz), 140.80 (d, *J* = 9.7 Hz), 138.74, 136.23, 134.74, 133.60 (d, *J* = 9.7 Hz), 130.10 (d, *J* = 4.7 Hz), 129.27 (d, *J* = 13.4 Hz), 127.99, 127.37, 123.12, 121.87, 121.55, 117.22, 71.29 (d, *J* = 6.1 Hz), 23.76 (d, *J* = 4.3 Hz), 23.72 (d, *J* = 5.0 Hz). $^{31}$P NMR (162 MHz, $CDCl_3$) δ 15.04. HRMS (ESI+): Calculated for $C_{23}H_{27}N_2O^4P$ $[M+H]^+$ 427.1787, Found 427.1783.

**diisopropyl (4-methoxy-2-(quinolin-8-ylcarbamoyl)phenyl)phosphonate. (3m)**



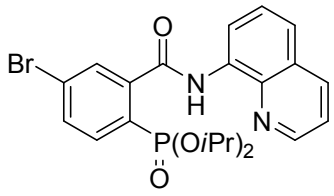
$R_f$ = 0.44 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.17 (s, 1H), 9.00 - 8.89 (m, 1H), 8.80 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.20 (dd, *J* = 8.4, 1.4 Hz, 1H), 8.04 (dd, *J* = 13.6, 8.6 Hz, 1H), 7.63 - 7.57 (m, 2H), 7.47 (dd, *J* = 8.4, 4.2 Hz, 1H), 7.24 - 7.23 (m, 1H), 7.08 (dt, *J* = 8.6, 2.6 Hz, 1H), 4.76 - 4.71 (m, 2H), 3.91 (s, 3H), 1.25 (d, *J* = 6.0 Hz, 6H), 1.15 (d, *J* = 6.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 167.17(d, *J* = 4.1 Hz), 162.48 (d, *J* = 3.3 Hz), 148.21, 142.73 (d, *J* = 11.1 Hz), 138.19, 136.25, 135.64 (d, *J* = 11.0 Hz), 134.70, 128.03, 127.37, 121.98, 121.58, 119.63, 117.30, 115.05 (d, *J* = 16.0 Hz), 114.11 (d, *J* = 13.9 Hz), 71.21 (d, *J* = 6.0 Hz), 55.54, 23.78 (d, *J* = 3.0 Hz), 23.73 (d, *J* = 3.0 Hz). $^{31}$P NMR (162 MHz, $CDCl_3$) δ 15.17. HRMS (ESI+): Calculated for $C_{23}H_{27}N_2O_5P$ $[M+H]^+$ 443.1736, Found 443.1735.

**diisopropyl (4-chloro-2-(quinolin-8-ylcarbamoyl)phenyl)phosphonate. (3n)**



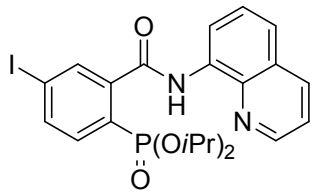
$R_f$ = 0.68 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.17 (s, 1H), 8.93 (dd, *J* = 7.2, 1.6 Hz, 1H), 8.80 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.21 (dd, *J* = 8.2, 1.6 Hz, 1H), 8.05 (dd, *J* = 13.6, 8.2 Hz, 1H), 7.71 (dd, *J* = 4.2, 2.0 Hz, 1H), 7.65 - 7.52 (m, 3H), 7.48 (dd, *J* = 8.2, 4.2 Hz, 1H), 4.79 - 4.74 (m, 2H), 1.26 (d, *J* = 6.0 Hz, 6H), 1.16 (d, *J* = 6.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 165.89 (d, *J* = 4.5 Hz), 148.27, 142.30 (d, *J* = 10.6 Hz), 138.78 (d, *J* = 4.0 Hz), 138.69, 136.30, 134.98 (d, *J* = 10.3 Hz), 134.48, 129.62 (d, *J* = 13.8 Hz), 128.85 (d, *J* = 12.7 Hz), 128.02, 127.35, 124.99, 122.22, 121.68, 117.33, 71.81 (d, *J* = 6.4 Hz), 23.75 (d, *J* = 4.2 Hz), 23.76 (d, *J* = 4.5 Hz). $^{31}$P NMR (162 MHz, $CDCl_3$) δ 13.31. HRMS (ESI+): Calculated for $C_{22}H_{24}ClN_2O_4P$ $[M+H]^+$ 447.1240, Found 447.1238.

**diisopropyl (4-bromo-2-(quinolin-8-ylcarbamoyl)phenyl)phosphonate. (3o)**



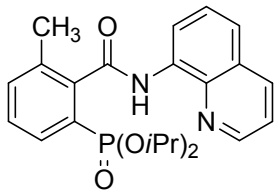
$R_f$ = 0.69 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.16 (s, 1H), 8.92 (dd, *J* = 7.2, 1.6 Hz, 1H), 8.81 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.21 (dd, *J* = 8.2, 1.6 Hz, 1H), 7.97 (dd, *J* = 13.6, 8.2 Hz, 1H), 7.87 (dd, *J* = 4.2, 1.8 Hz, 1H), 7.75 - 7.74 (m, 1H), 7.62 - 7.60 (m, 2H), 7.48 (dd, *J* = 8.2, 4.2 Hz, 1H), 4.78 - 4.73 (m, 2H), 1.26 (d, *J* = 6.0 Hz, 6H), 1.16 (d, *J* = 6.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 165.77 (d, *J* = 4.3 Hz), 148.27, 142.29 (d, *J* = 10.0 Hz), 138.68 Hz, 136.31, 134.99 (d, *J* = 10.3 Hz), 134.47, 132.61 (d, *J* = 14.6 Hz), 131.68 (d, *J* = 13.4 Hz), 128.02, 127.35, 127.20 (d, *J* = 3.7 Hz), 125.46, 122.21, 121.67, 117.32, 71.82 (d, *J* = 6.1 Hz), 23.76 (d, *J* = 3.8 Hz), 23.72 (d, *J* = 4.5 Hz). $^{31}$P NMR (162 MHz, $CDCl_3$) δ 13.46. HRMS (ESI+): Calculated for $C_{22}H_{24}BrN_2O_4P$ $[M+Na]^+$ 513.0555, Found 513.0490.

**diisopropyl (4-iodo-2-(quinolin-8-ylcarbamoyl)phenyl)phosphonate. (3p)**



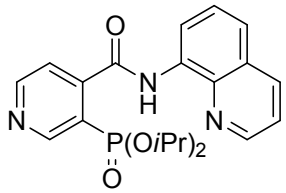
$R_f$ = 0.55 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.14 (s, 1H), 8.92 (dd, *J* = 7.2, 1.6 Hz, 1H), 8.80 (dd, *J* = 4.4, 1.6 Hz, 1H), 8.20 (dd, *J* = 8.4, 1.6 Hz, 1H), 8.06 (dd, *J* = 4.4, 1.6 Hz, 1H), 7.95 - 7.93 (m, 1H), 7.80 (dd, *J* = 13.6, 8.0 Hz, 1H), 7.69 - 7.54 (m, 2H), 7.48 (dd, *J* = 8.4, 4.2 Hz, 1H), 4.78 - 4.73 (m, 2H), 1.25 (d, *J* = 6.0 Hz, 6H), 1.16 (d, *J* = 6.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 165.65 (d, *J* = 4.2 Hz), 148.25, 142.00 (d, *J* = 9.5 Hz), 138.66 (d, *J* = 1.3 Hz), 138.50, 137.37 (d, *J* = 13.2 Hz), 136.28, 134.69 (d, *J* = 10.1 Hz), 134.47, 128.00, 127.85, 127.35, 125.96, 122.18, 121.65, 117.28, 71.81 (d, *J* = 5.7 Hz), 23.76 (d, *J* = 0.3 Hz), 23.72 (d, *J* = 7.1 Hz). $^{31}$P NMR (162 MHz, $CDCl_3$) δ 13.79. HRMS (ESI+): Calculated for $C_{22}H_{24}IN_2O_4P$ $[M+H]^+$ 539.0597, Found 539.0539.

**diisopropyl (3-methyl-2-(quinolin-8-ylcarbamoyl)phenyl)phosphonate. (3q)**



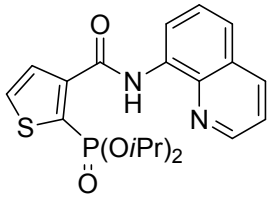
$R_f$ = 0.44 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 9.99 (s, 1H), 9.04 (dd, *J* = 7.4, 1.4 Hz, 1H), 8.77 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.20 (dd, *J* = 8.2, 1.6 Hz, 1H), 7.94-7.89 (m, 1H), 7.65-7.56 (m, 2H), 7.47-7.44 (m, 3H), 4.74-4.69 (m, 2H), 2.49 (s, 3H), 1.22 (d, *J* = 6.0 Hz, 6H), 1.13 (d, *J* = 6.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.95 (d, *J* = 4.9 Hz), 148.07, 138.56, 136.27, 136.14, 134.71, 134.36 (d, *J* = 4.1 Hz), 130.68 (d, *J* = 9.8 Hz), 128.68 (d, *J* = 13.9 Hz), 128.02, 127.98, 127.48, 126.17, 121.78, 121.57, 116.94, 71.26 (d, *J* = 5.7 Hz), 23.72 (d, *J* = 3.3 Hz), 23.68 (d, *J* = 4.5 Hz), 19.55. $^{31}$P NMR (162 MHz, $CDCl_3$) δ 14.87. HRMS (ESI+): Calculated for $C_{23}H_{27}N_2O^4P$ $[M+H]^+$ 427.1787, Found 427.1783.

**diisopropyl (4-(quinolin-8-ylcarbamoyl)pyridin-3-yl)phosphonate. (3r)**



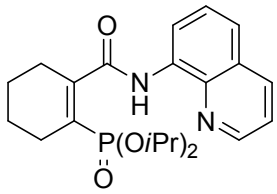
$R_f$ = 0.20 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.25 (s, 1H), 9.25 (d, *J* = 7.2 Hz, 1H), 9.02 - 8.87 (m, 2H), 8.81 (dd, *J* = 4.0, 1.6 Hz, 1H), 8.21 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.69 - 7.56 (m, 3H), 7.49 (dd, *J* = 8.4, 4.4 Hz, 1H), 4.86 - 4.77 (m, 2H), 1.28 (d, *J* = 6.0 Hz, 6H), 1.22 (d, *J* = 6.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 165.01 (d, *J* = 4.7 Hz), 153.72 (d, *J* = 11.9 Hz), 153.46, 148.39, 147.55 (d, *J* = 7.9 Hz), 138.61, 136.33, 134.16, 128.00, 127.29, 123.46, 122.54, 122.20 (d, *J* = 10.1 Hz), 121.76, 117.42, 72.15 (d, *J* = 6.2 Hz), 23.81 (d, *J* = 4.0 Hz), 23.73 (d, *J* = 4.8 Hz). $^{31}$P NMR (162 MHz, $CDCl_3$) δ 11.46. HRMS (ESI+): Calculated for $C_{21}H_{24}N_3O_4P$ $[M+H]^+$ 414.1583, Found 414.1581.

**diisopropyl (3-(quinolin-8-ylcarbamoyl)thiophen-2-yl)phosphonate. (3s)**



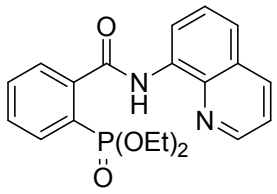
$R_f$ = 0.45 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.61 (s, 1H), 8.93 - 8.83 (m, 2H), 8.20 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.67 (t, *J* = 5.2 Hz, 1H), 7.65 - 7.55 (m, 3H), 7.48 (dd, *J* = 8.4, 4.2 Hz, 1H), 4.88 - 4.80 (m, 2H), 1.33 (d, *J* = 6.0 Hz, 6H), 1.30 (d, *J* = 6.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$ , ppm) δ 162.02 (d, *J* = 2.9 Hz), 148.43, 143,94 (d, *J* = 11.1 Hz), 139.17, 136.21, 134.50, 131.83 (d, *J* = 7.8 Hz), 130.63, 130.46, 128.12, 127.19, 122.34, 121.61, 118.04, 72.40 (d, *J* = 5,7 Hz), 23.94 (d, *J* = 4.1 Hz), 23.69 (d, *J* = 5.3 Hz). $^{31}$P NMR (162 MHz, $CDCl_3$ , ppm) δ 7.17. HRMS (ESI+): Calculated for $C_{20}H_{23}N_2O_4PS$ $[M+H]^+$ 419.1194, Found 419.1194.

**diisopropyl (2-(quinolin-8-ylcarbamoyl)cyclohex-1-en-1-yl)phosphonate. (3t)**



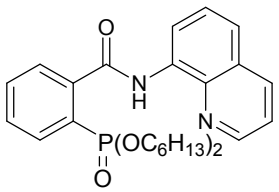
$R_f$ = 0.39 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 9.91 (s, 1H), 8.88 (dd, *J* = 7.2, 1.2 Hz, 1H), 8.83 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.18 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.64 - 7.51 (m, 2H), 7.46 (dd, *J* = 8.4, 4.2 Hz, 1H), 4.71 - 4.63 (m, 2H), 2.59 (s, 2H), 2.42 (s, 2H), 1.77 (s, 4H), 1.27 (d, *J* = 6.2 Hz, 6H), 1.17 (d, *J* = 6.2 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 169.08 (d, *J* = 11.5 Hz), 148.03, 138.62, 134.77, 132.45 (d, *J* = 8.2 Hz), 127.9, 127.45, 125.65, 121.61, 121.47, 117.00, 70.63 (d, *J* = 6.6 Hz), 29.12 (d, *J* = 14.7), 25.89 (d, *J* = 8.2 Hz), 23.85 (d, *J* = 3.7 Hz), 23.79 (d, *J* = 5.2 Hz), 21.62 (d, *J* = 8.2 Hz), 21.40. $^{31}$P NMR (162 MHz, $CDCl_3$) δ 14.09. HRMS (ESI+): Calculated for C22H29N2O4P $[M+H]^+$ 417.1943, Found 417.1940.

**diethyl (2-(quinolin-8-ylcarbamoyl)phenyl)phosphonate. (4b)**



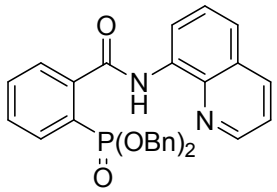
$R_f$ = 0.26 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.23 (s, 1H), 8.98 (dd, *J* = 7.4, 1.6 Hz, 1H), 8.79 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.20 (dd, *J* = 8.2, 1.6 Hz, 1H), 8.14 - 8.08 (m, 1H), 7.80 – 7.72 (m, 1H), 7.71-7.67 (m, 1H), 7.66 - 7.55 (m, 3H), 7.48 - 7.45 (m, 1H), 4.18 - 4.05 (m, 4H), 1.23 (t, *J* = 7.1 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 167.36 (d, *J* = 4.9 Hz), 148.20, 140.95 (d, *J* = 9.0 Hz), 138.65, 136.28, 134.70, 133.85 (d, *J* = 9.7 Hz), 132.59 (d, *J* = 2.9 Hz), 129.60 (d, *J* = 14.1 Hz), 128.48 (d, *J* = 12.7 Hz), 128.03, 127.40, 124.91, 122.01, 121.63, 116.97, 62.65 (d, *J* = 5.7 Hz), 16.12 (d, *J* = 6.6 Hz). $^{31}$P NMR (162 MHz, $CDCl_3$) δ 16.56. HRMS (ESI+): Calculated for $C_{20}H_{21}N_2O_4P$ $[M+H]^+$ 385.1317, Found 385.1315.

**dihexyl (2-(quinolin-8-ylcarbamoyl)phenyl)phosphonate. (4c)**



$R_f$ = 0.65 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ10.21 (s, 1H), 8.98 (dd, *J* = 7.2, 1.2 Hz, 1H), 8.78 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.20 (dd, *J* = 8.2, 1.6 Hz, 1H), 8.1 - 8.06 (m, 1H), 7.79 - 7.72 (m, 1H), 7.68 (dd, *J* = 10.6, 4.4 Hz, 1H), 7.64 - 7.53 (m, 3H), 7.47 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.04 (q, *J* = 6.8 Hz, 4H), 1.59-1.53 (m, 4H), 1.25 - 1.11 (m, 12H), 0.81 (t, *J* = 6.9 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 167.31 (d, *J* = 4.5 Hz), 148.17, 140.91 (d, *J* = 9.4 Hz), 138.65, 136.24, 134.71, 133.83 (d, *J* = 9.2 Hz), 132.50 (d, *J* = 2.8 Hz), 129.55 (d, *J* = 14.1 Hz), 128.41 (d, *J* = 13.0 Hz), 128.03, 127.36, 125.01, 121.96, 121.57, 117.02, 66.72 (d, *J* = 6.1 Hz), 31.23, 30.27 (d, *J* = 6.1 Hz), 25.03, 22.35, 13.88. $^{31}$P NMR (162 MHz, $CDCl_3$) δ 16.67. HRMS (ESI+): Calculated for $C_{28}H_{37}N_2O_4P$ $[M+H]^+$ 497.2569, Found 497.2566.

**dibenzyl (2-(quinolin-8-ylcarbamoyl)phenyl)phosphonate. (4e)**

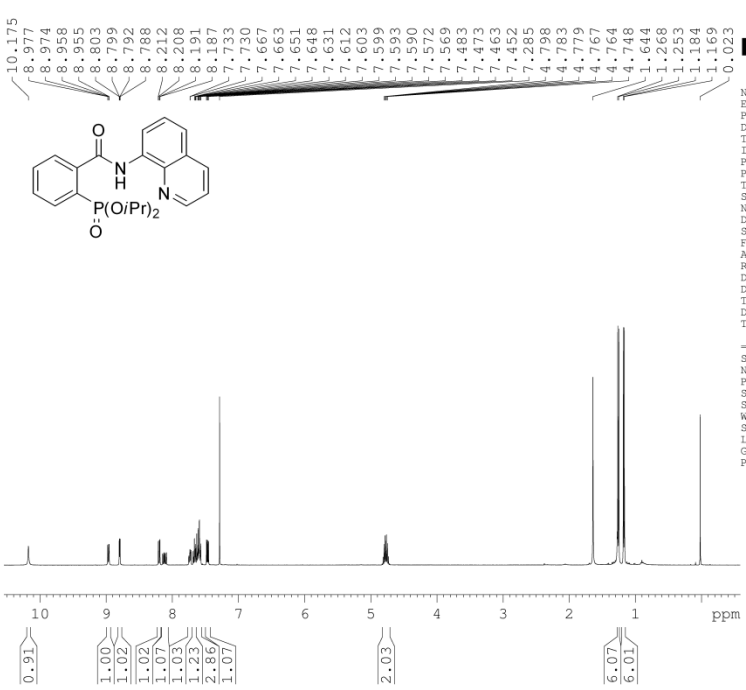


$R_f$ = 0.51 (petroleum ether/EtOAc, 1:1). White solid. $^1$H NMR (400 MHz, $CDCl_3$) δ 10.20 (s, 1H), 8.91 - 8.81 (m, 1H), 8.61 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.17 (dd, *J* = 8.4, 1.6 Hz, 1H), 8.07 (dd, *J* = 14.4, 7.6 Hz, 1H), 7.80 - 7.73 (m, 1H), 7.68 (t, *J* = 7.5 Hz, 1H), 7.59 - 7.55 (m, 2H), 7.41 (dd, *J* = 8.4, 4.2 Hz, 1H), 7.36 - 7.29 (m, 2H), 7.24 - 7.19 (m, 4H), 7.19 - 7.13 (m, 5H), 5.05 (dt, *J* = 6.8, 3.6 Hz, 4H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 167.17 (d, *J* = 4.5 Hz), 148.17, 140.97 (d, *J* = 9.7 Hz), 138.56, 136.14, 135.96 (d, *J* = 6.6 Hz), 134.53, 133.91 (d, *J* = 9.1 Hz), 132.68 (d, *J* = 2.8 Hz), 129.66 (d, *J* = 14.4 Hz), 128.50 (d, *J* = 2.4 Hz), 128.39, 128.31, 128.18, 128.03, 127.91, 127.34, 121.96, 121.53, 117.06, 68.43 (d, *J* = 5.8 Hz). $^{31}$P NMR (162 MHz, $CDCl_3$ , ppm) δ 17.47. HRMS (ESI+): Calculated for $C_{30}H_{25}N_2O_4P$ $[M+H]^+$ 509.1630, Found 509.1626.

**References.**

[1] F.-R. Gou, X.-C. Wang, P.-F. Huo, H.-P. Bi, Z.-H. Guan, Y.-M. Liang, *Org. Lett.* **2009**, *11*, 5726.

[2] S. Inoue, H. Shiota, Y. Fukumoto, N. Chatani, *J. Am. Chem. Soc.* **2009**, *131*, 6898.

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DE                           6.50 usec
TE                          297.9 K
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TD0                              1

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GB                               0
PC                            1.00
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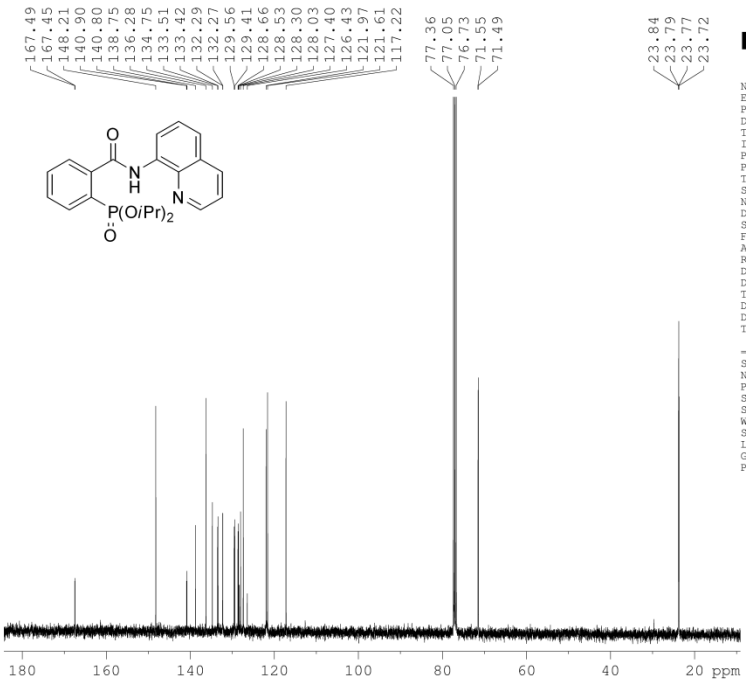


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PULPROG                   zgpg30
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SOLVENT                     CDCl3
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DS                               4
SWH                      24038.461 Hz
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AQ                        1.3631988 sec
RG                           203.91
DW                          20.800 usec
DE                           6.50 usec
TE                          298.5 K
D1                        2.00000000 sec
D11                       0.03000000 sec
TD0                              1

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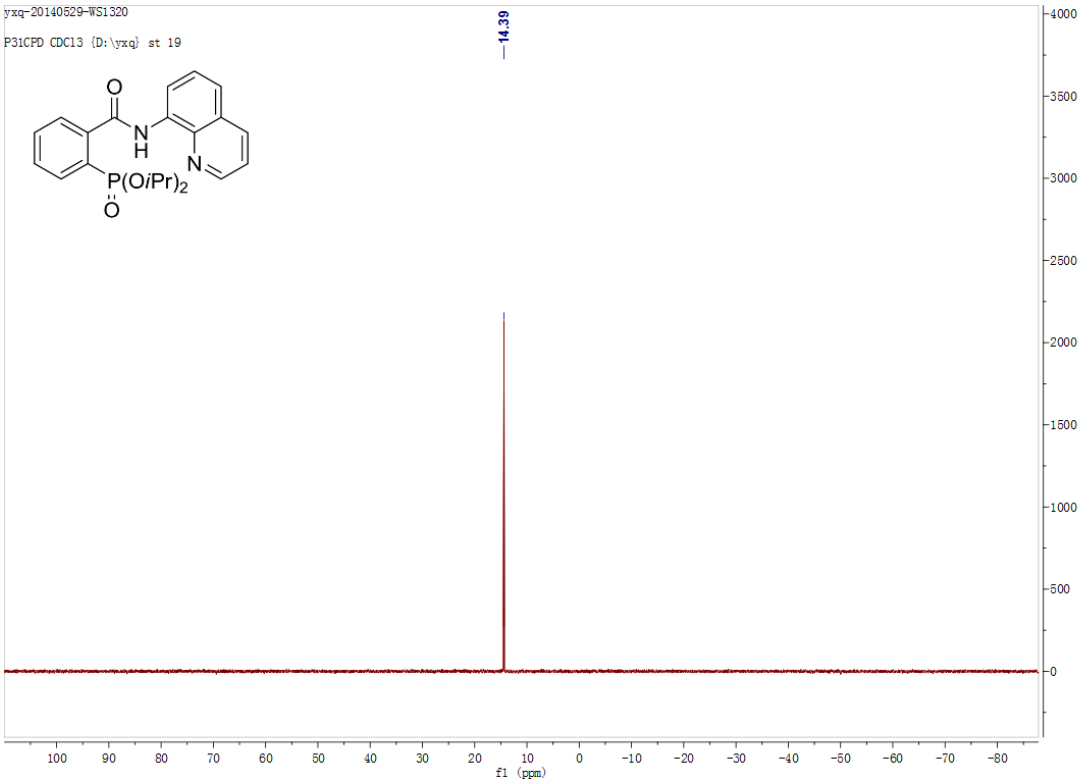


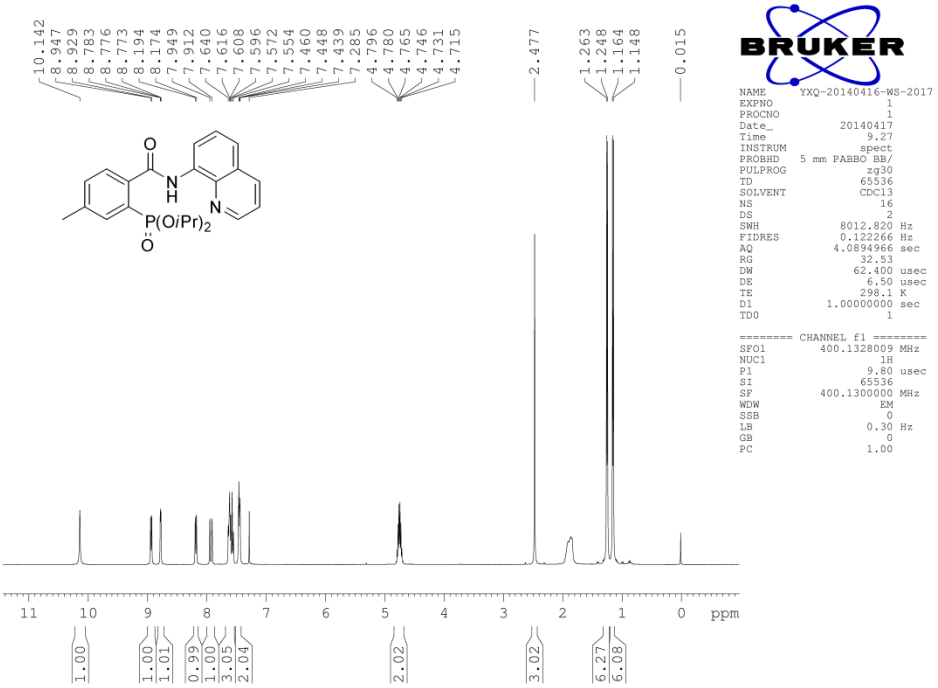


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PROCNO                   1
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PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH               8012.820 Hz
FIDRES            0.122266 Hz
AQ                4.0894966 sec
RG                    32.53
DW                  62.400 usec
DE                   6.50 usec
TE                 298.1 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
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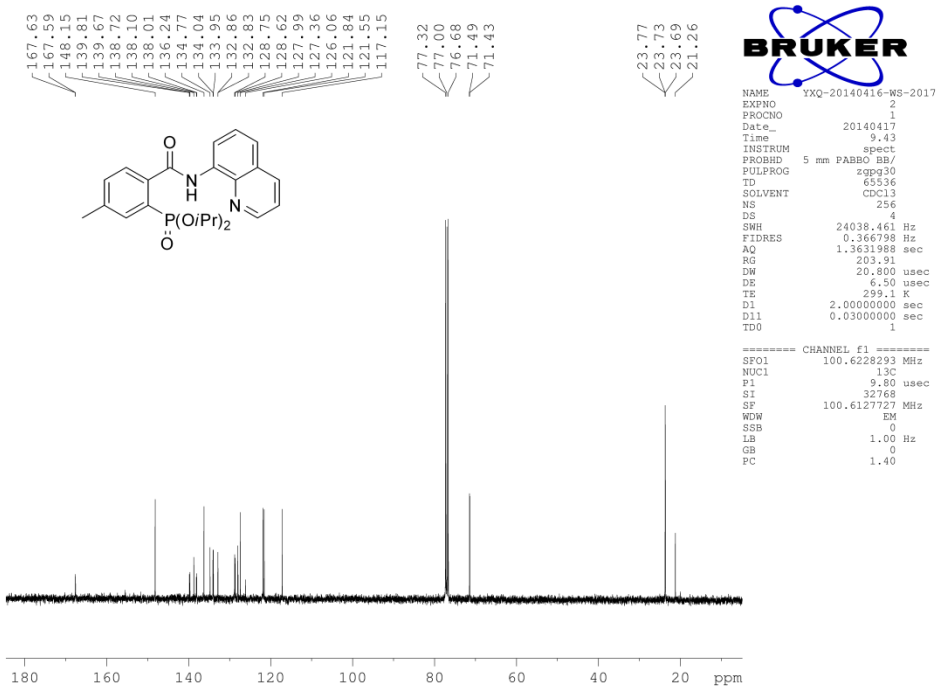
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H
N
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120
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BRUKER
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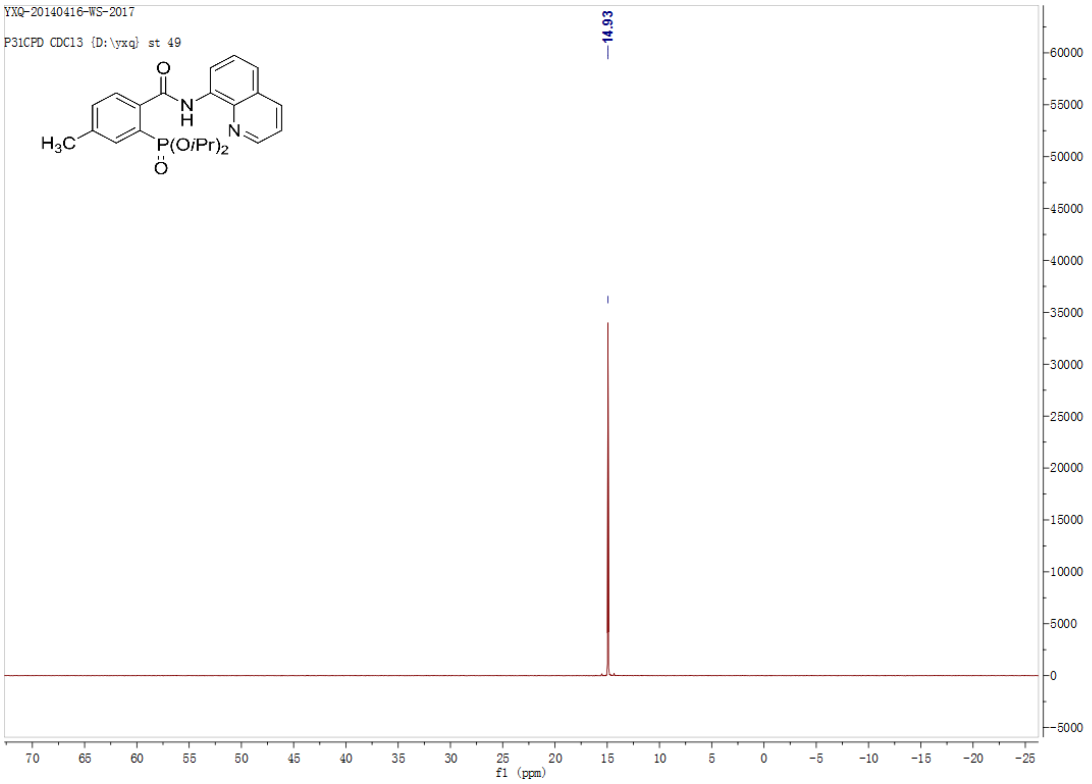
YXQ-20140416-WS-2017
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N
H3C
P(O*i*Pr)2
O
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60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
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65
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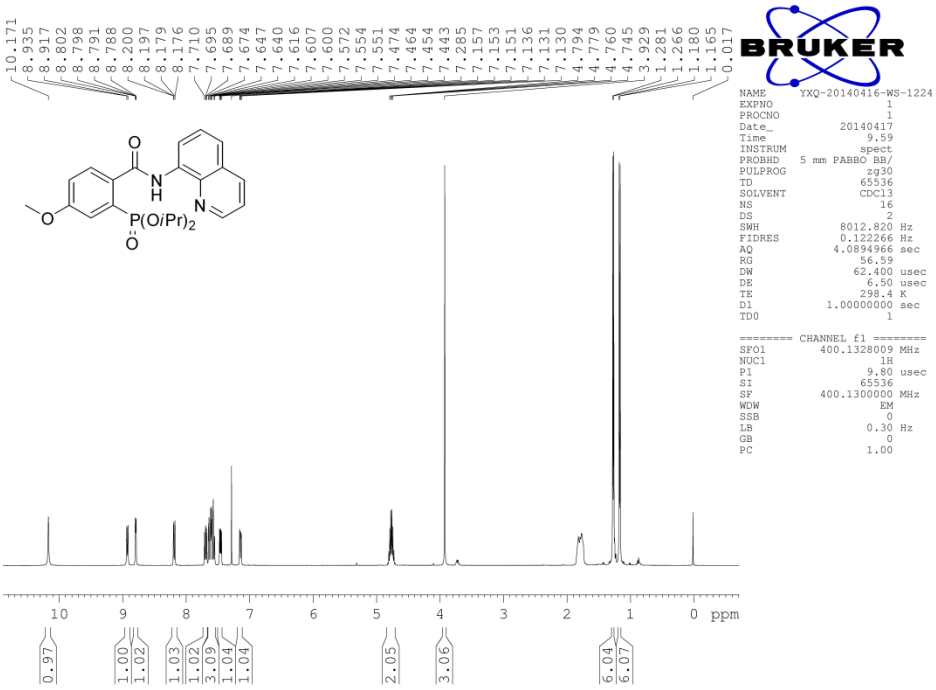
f1 (ppm)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Date_ 20140417
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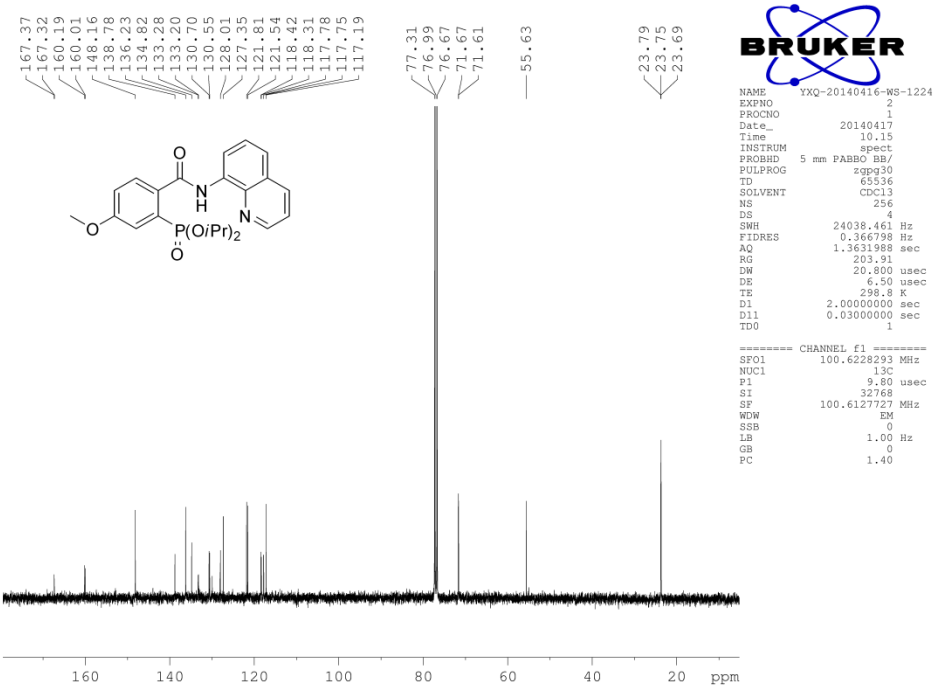
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TE 298.8 K
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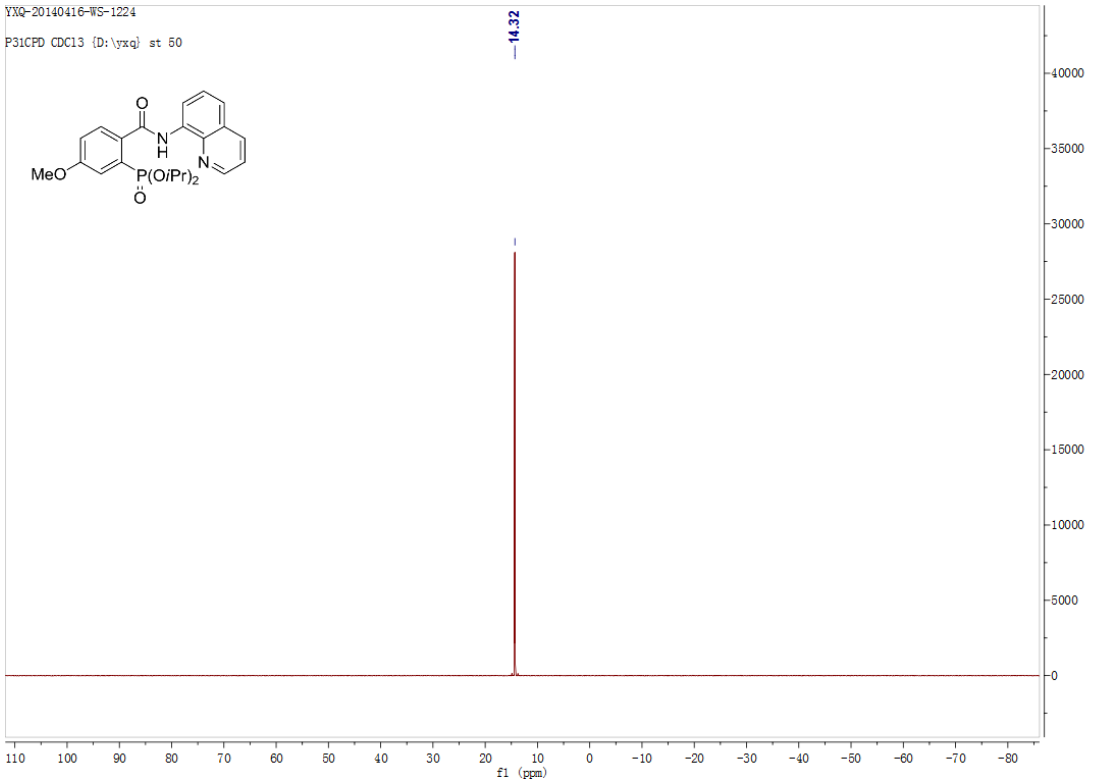
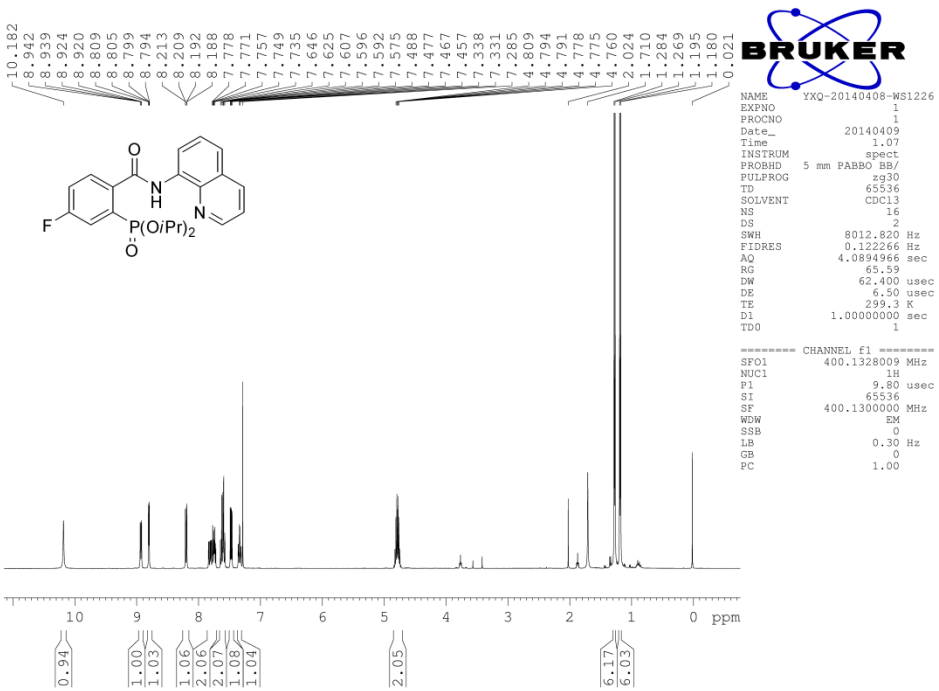
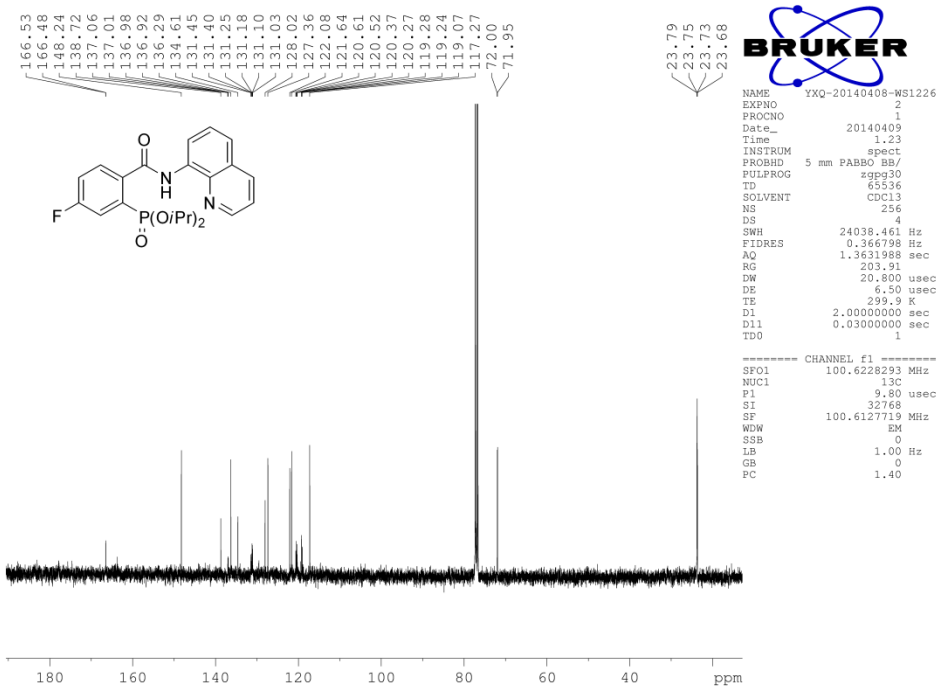
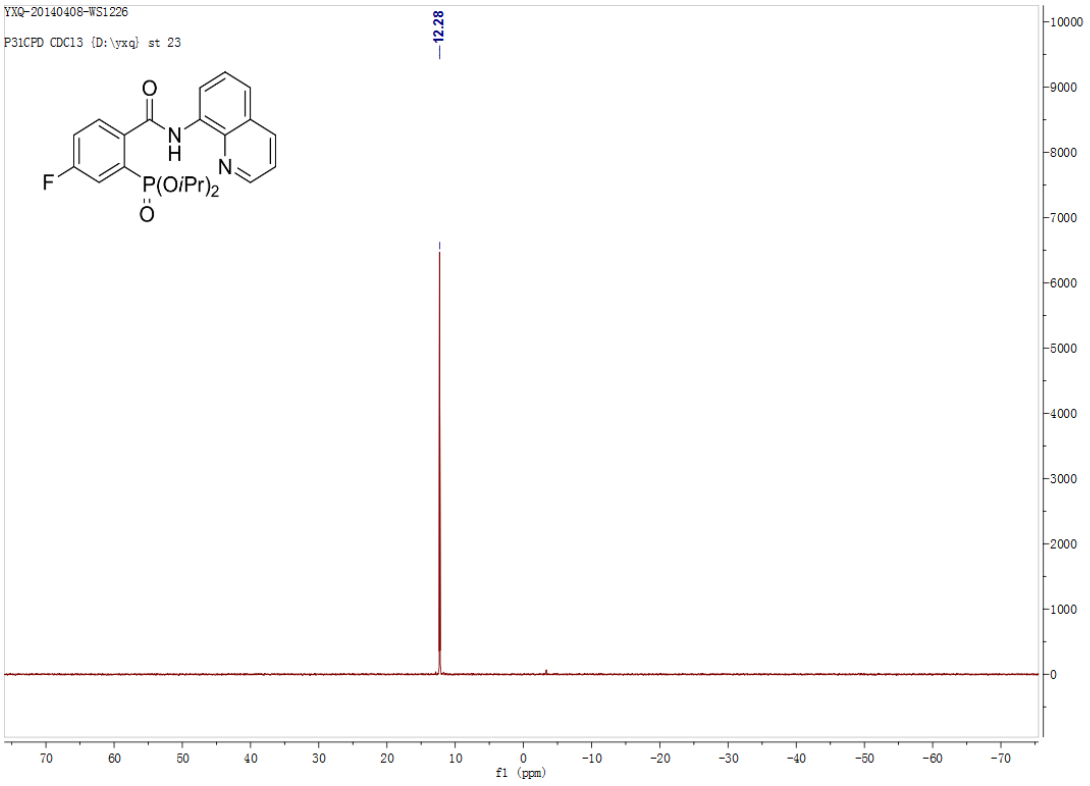
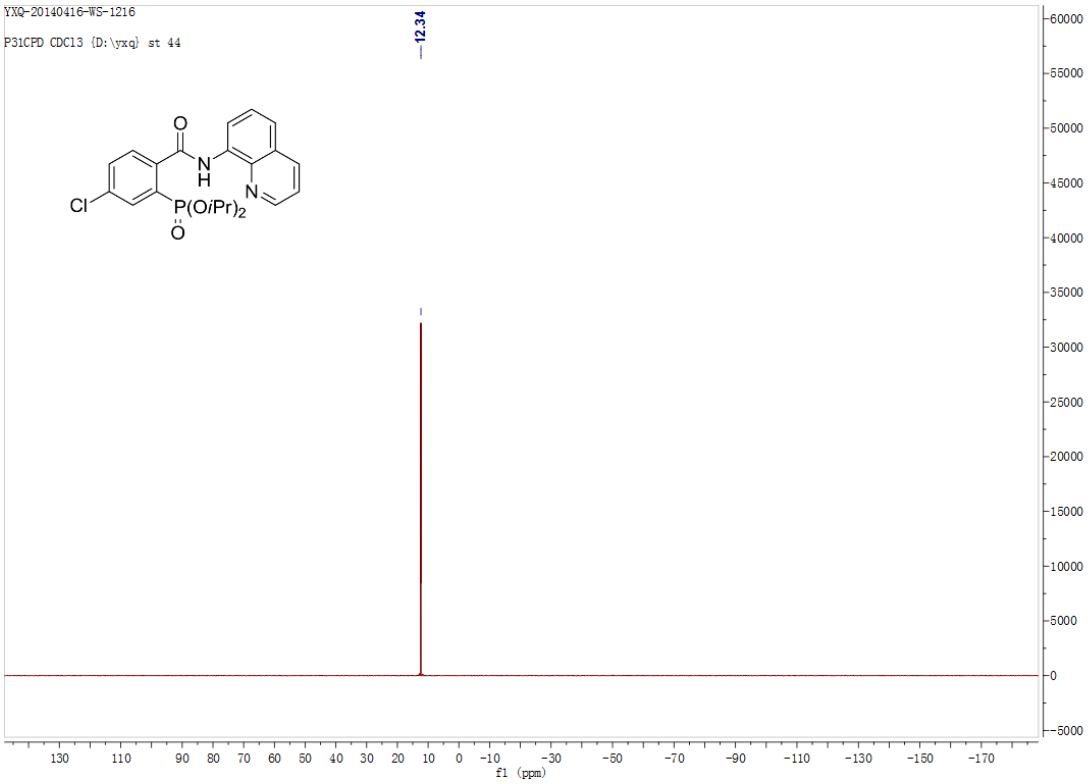
20
ppm

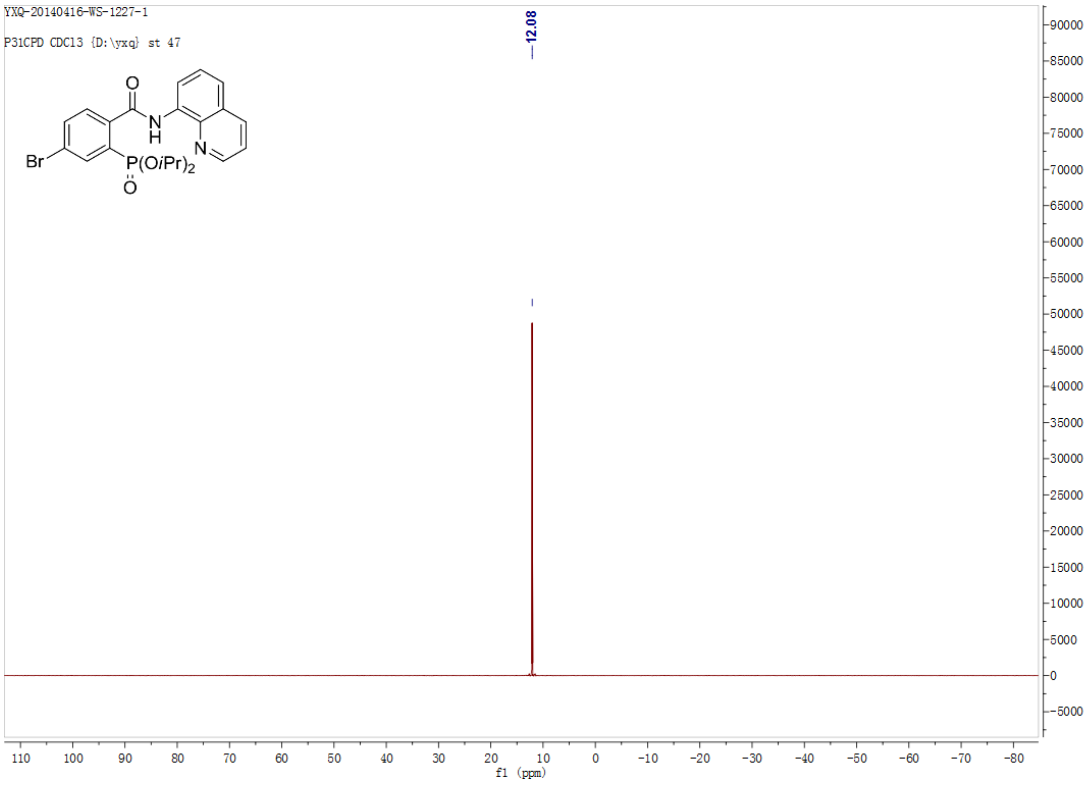

YXQ-20140416-WS-1224
P31CPD CDCl3 (D:\yxq) st 50
14.32
MeO
P(O*i*Pr)2
f1 (ppm)

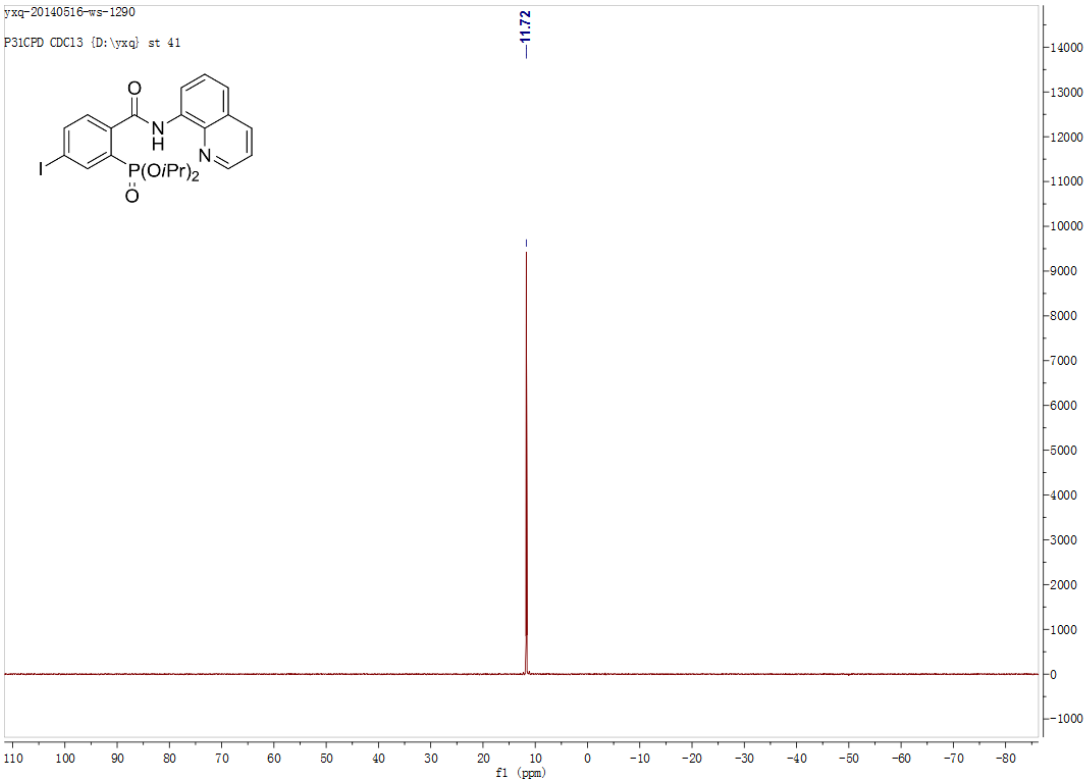

10.182
8.942
8.939
8.924
8.920
8.809
8.805
8.799
8.794
8.213
8.209
8.192
8.188
7.778
7.771
7.757
7.749
7.735
7.646
7.625
7.607
7.596
7.592
7.575
7.488
7.477
7.467
7.457
7.338
7.331
7.285
4.809
4.794
4.791
4.778
4.775
4.760
2.024
1.710
1.284
1.269
1.195
1.180
0.021
F
P(O*i*Pr)2
BRUKER
NAME YXQ-20140408-WS1226
EXPNO 1
PROCNO 1
Date_ 20140409
Time 1.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 65.59
DW 62.400 usec
DE 6.50 usec
TE 299.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1328009 MHz
NUC1 1H
P1 9.80 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
0.94
1.00
1.03
1.06
2.06
2.07
1.08
1.04
2.05
6.17
6.03

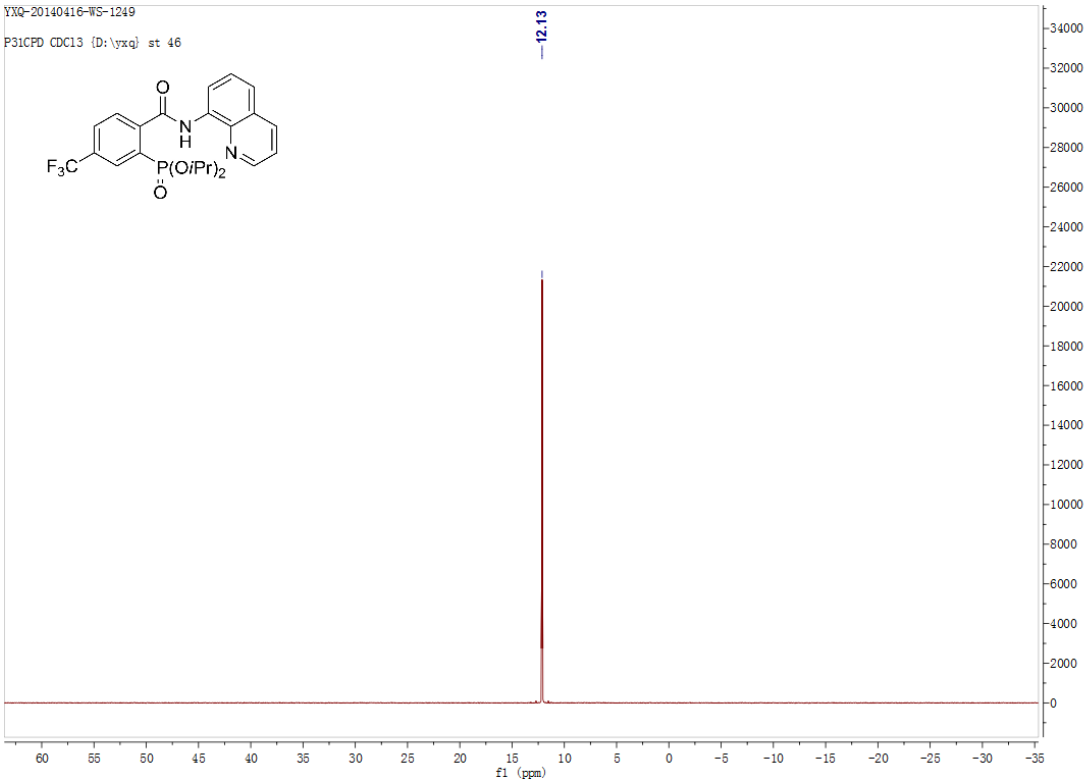

166.53
166.48
148.24
138.72
137.06
137.01
136.98
136.92
136.29
134.61
131.45
131.40
131.25
131.18
131.10
131.03
128.02
127.36
122.08
121.64
120.61
120.52
120.37
120.27
119.28
119.24
119.07
117.27
72.00
71.95
23.79
23.75
23.73
23.68
BRUKER
NAME YXQ-20140408-WS1226
EXPNO 2
PROCNO 1
Date_ 20140409
Time 1.23
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203.91
DW 20.800 usec
DE 6.50 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.80 usec
SI 32768
SF 100.6127719 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
N
H
N
F
P(OiPr)2
O
180
160
140
120
100
80
60
40
ppm

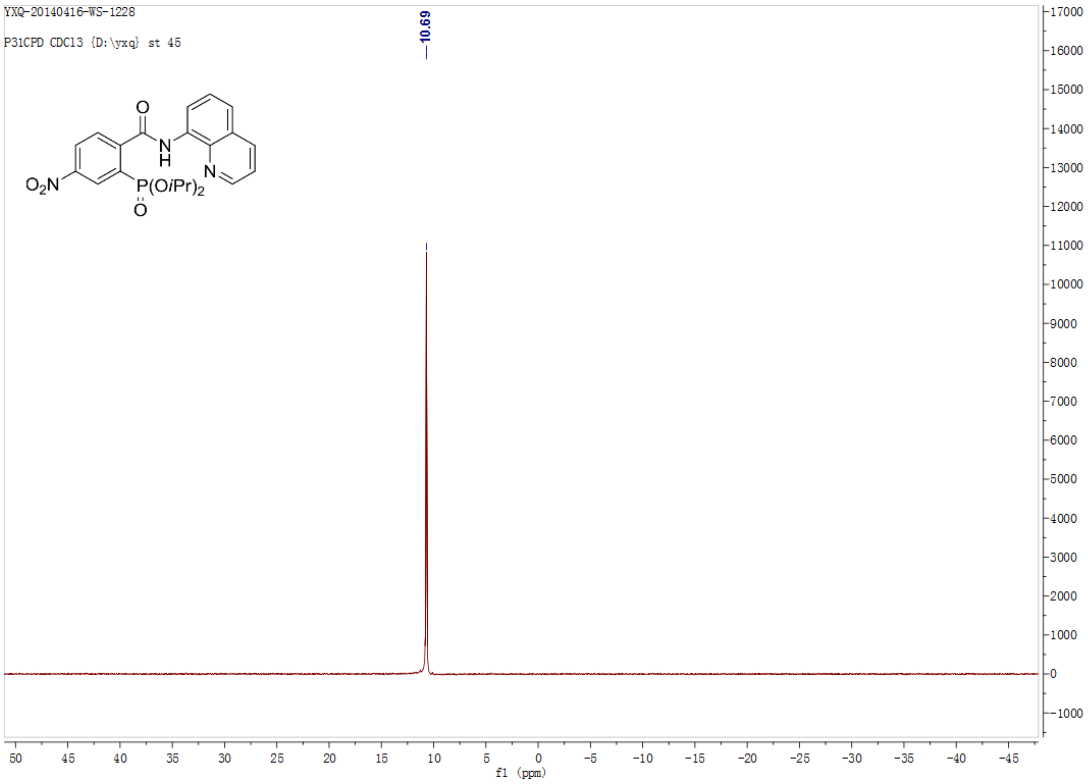

YXQ-20140408-WS1226
P31CPD CDCl3 {D:\yxq} st 23
12.28
O
N
H
N
F
P(OiPr)2
O
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
f1 (ppm)

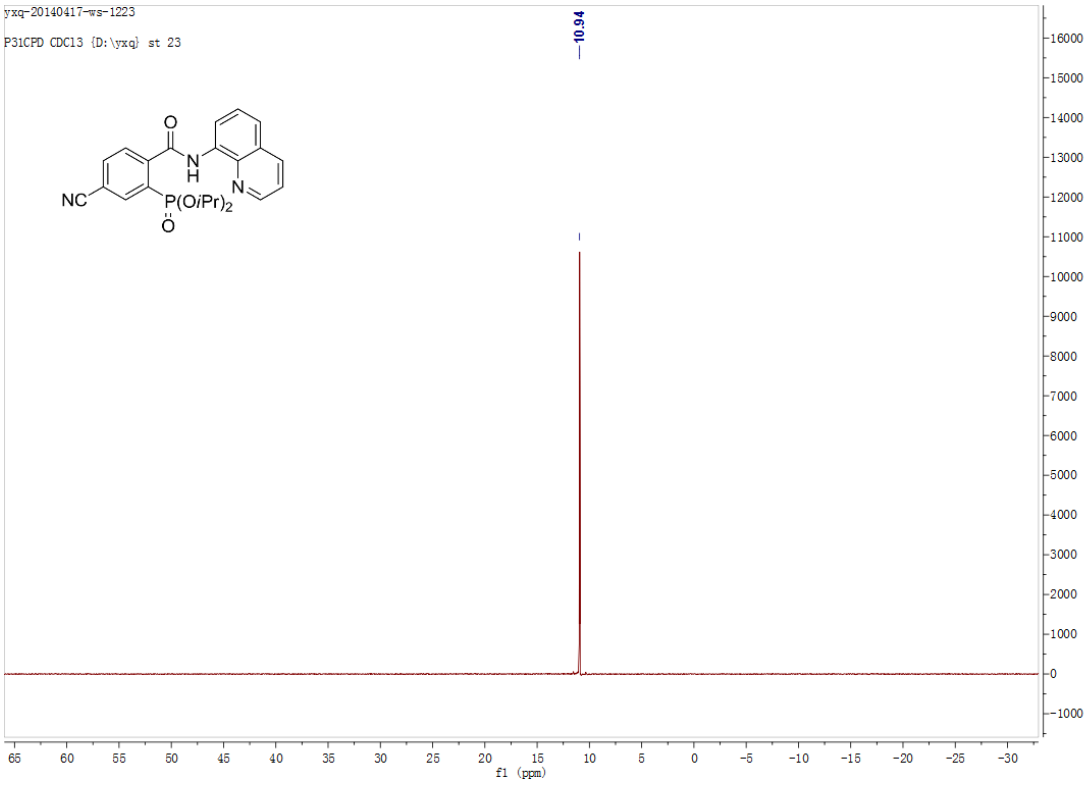

YXQ-20140416-WS-1216
P31CPD CDCl3 (D:\yxq) st 44
12.34
O
N
H
N
Cl
P(OiPr)2
O
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
130
110
90
80
70
60
50
40
30
20
10
0
-10
-30
-50
-70
-90
-110
-130
-150
-170
f1 (ppm)

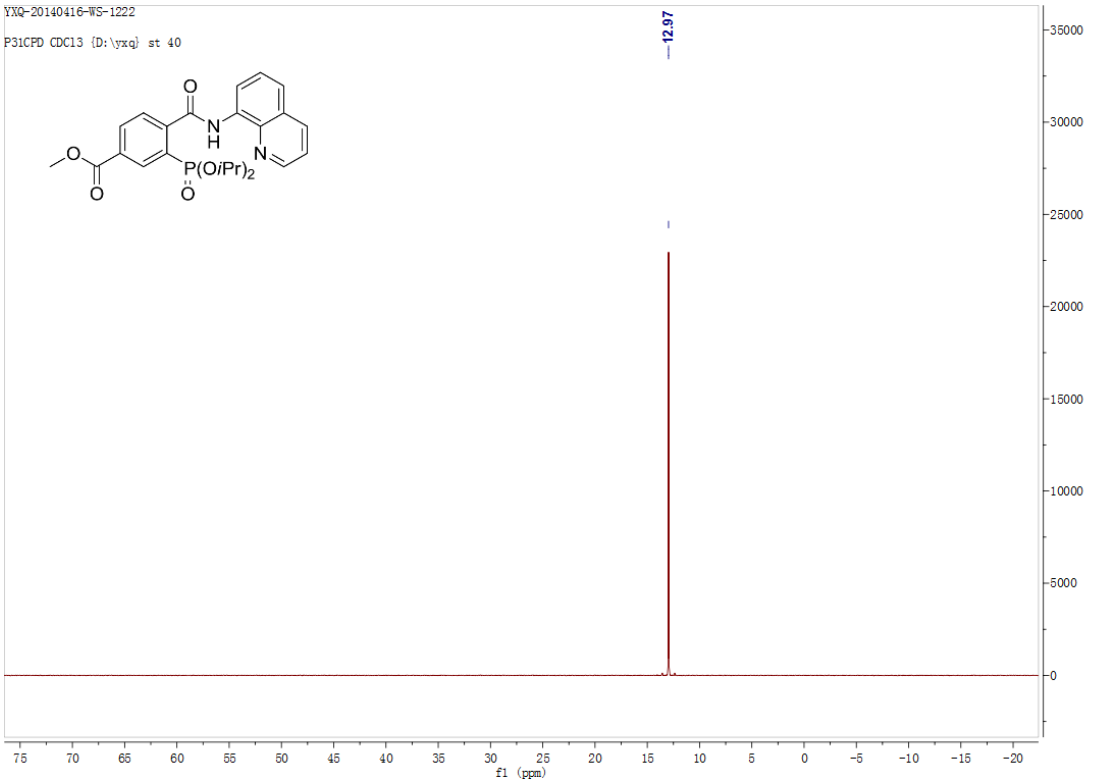

YXQ-20140416-WS-1227-1
P31CPD CDCl3 {D:\yxq} st 47
12.08
Br
O
N
H
N
P(OiPr)2
O
f1 (ppm)

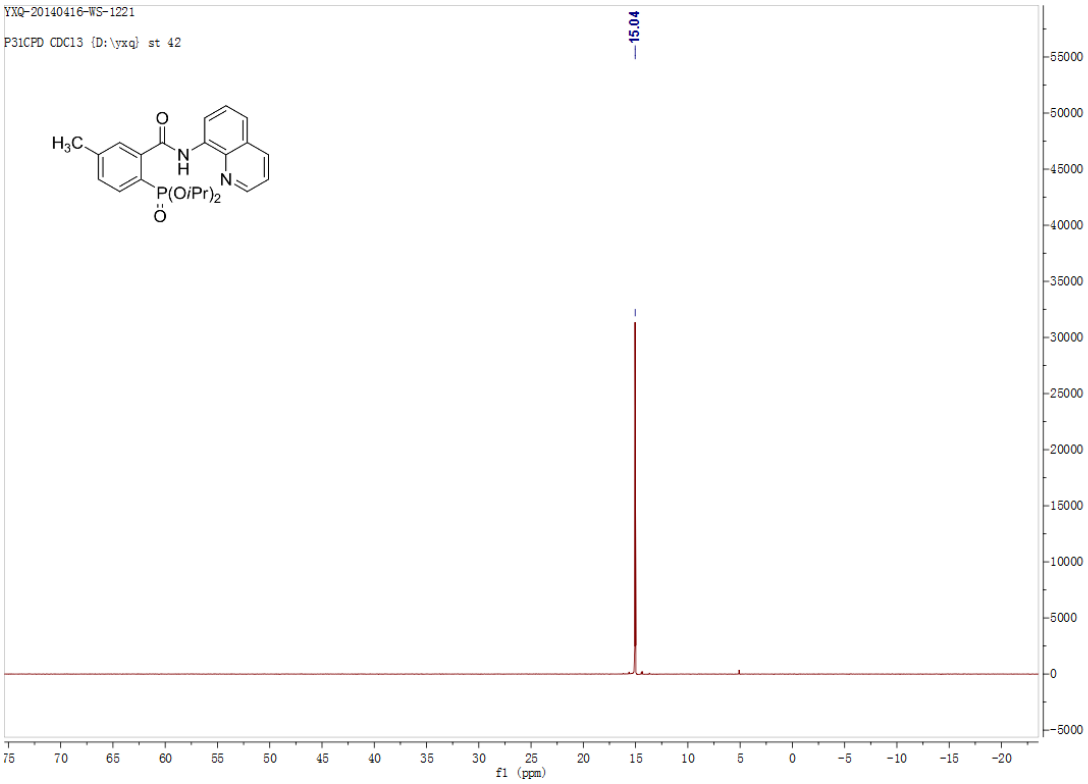

yxq-20140516-ws-1290
P31CPD CDCl3 (D:\yxq) st 41
11.72
O
N
H
N
I
P(OiPr)2
O
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80
f1 (ppm)

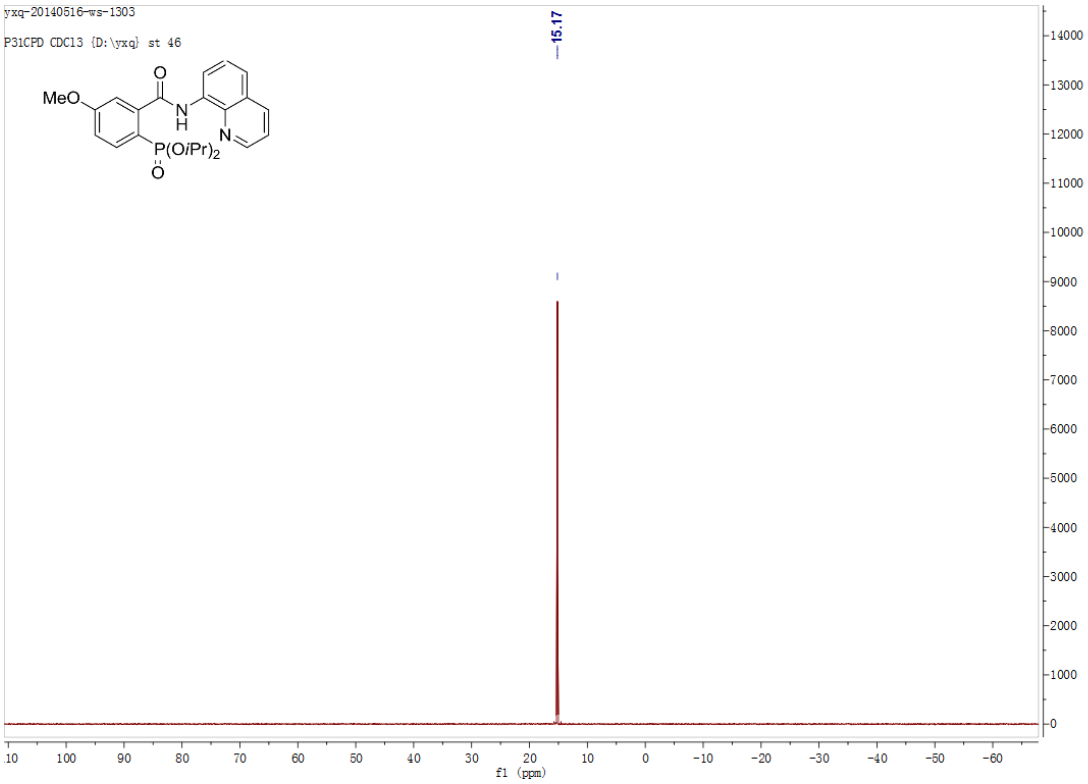

YXQ-20140416-WS-1249
P31CPD CDCl3 {D:\yxq} st 46
12.13
O
N
H
N
F3C
P(OiPr)2
O
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
f1 (ppm)

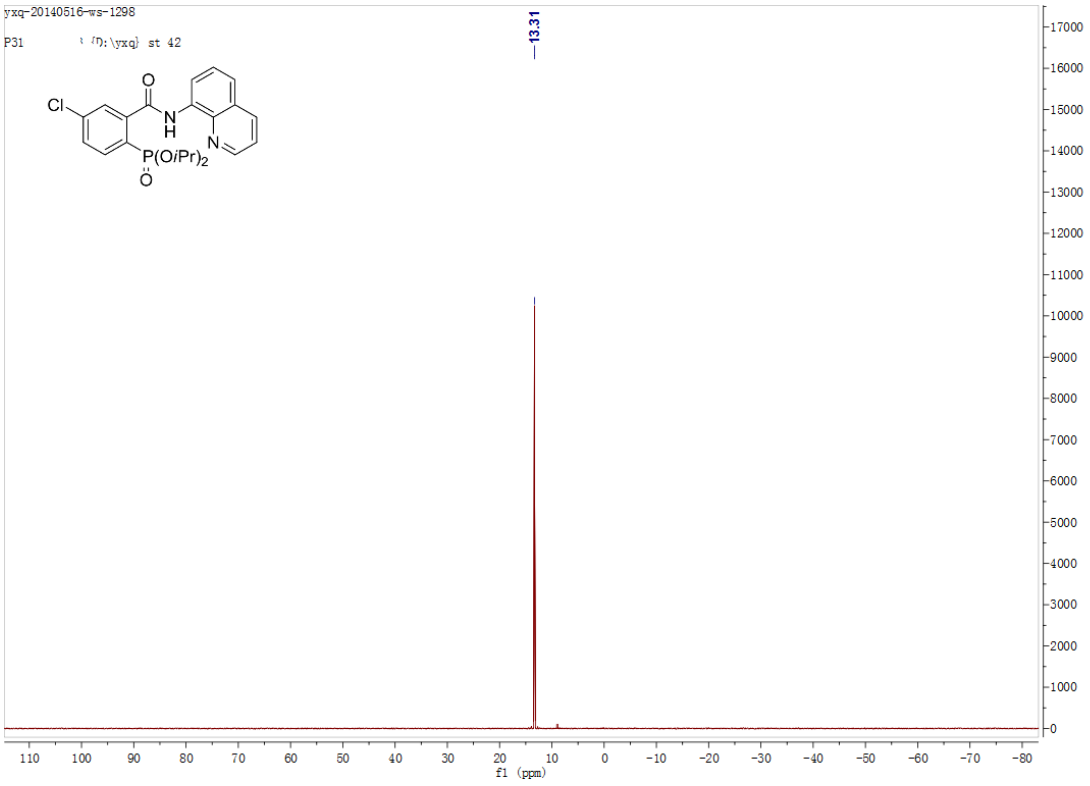

YXQ-20140416-WS-1228
P31CPD CDCl3 (D:\yxq) st 45
10.69
O
N
H
O2N
P(OiPr)2
O
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
50 45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45
f1 (ppm)

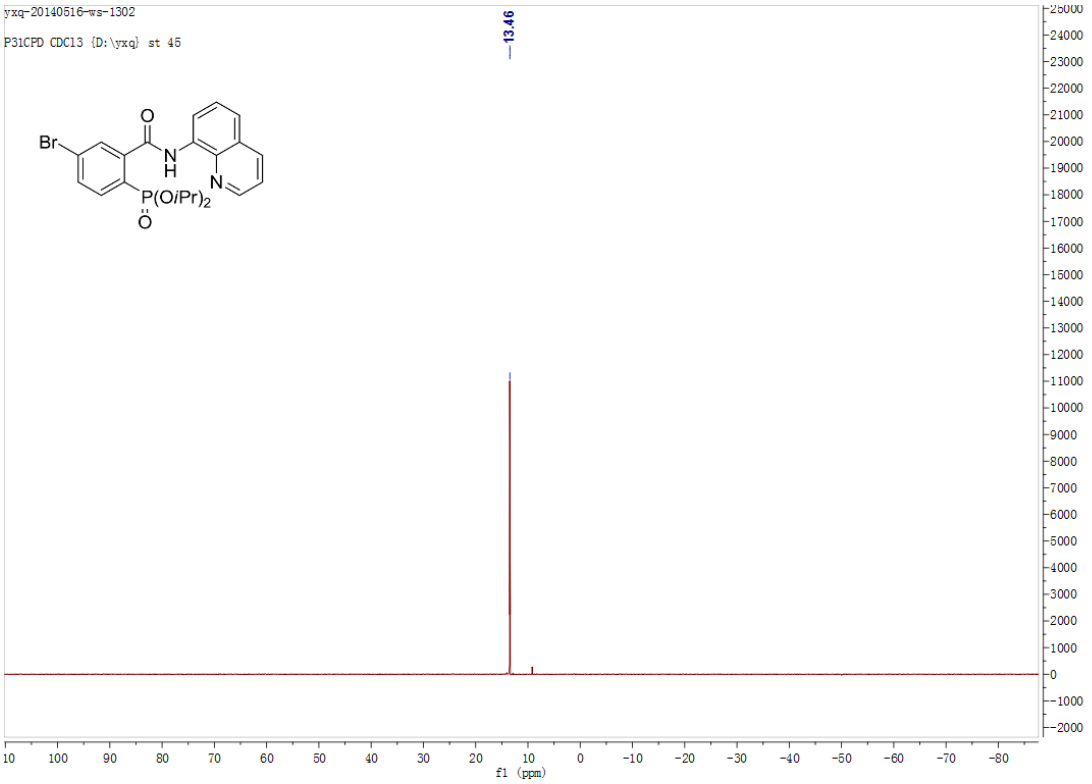

yxq-20140417-ws-1223
P31CPD CDCl3 (D:\yxq) st 23
10.94
NC
P(O/Pr)2
O
N
H
N
f1 (ppm)

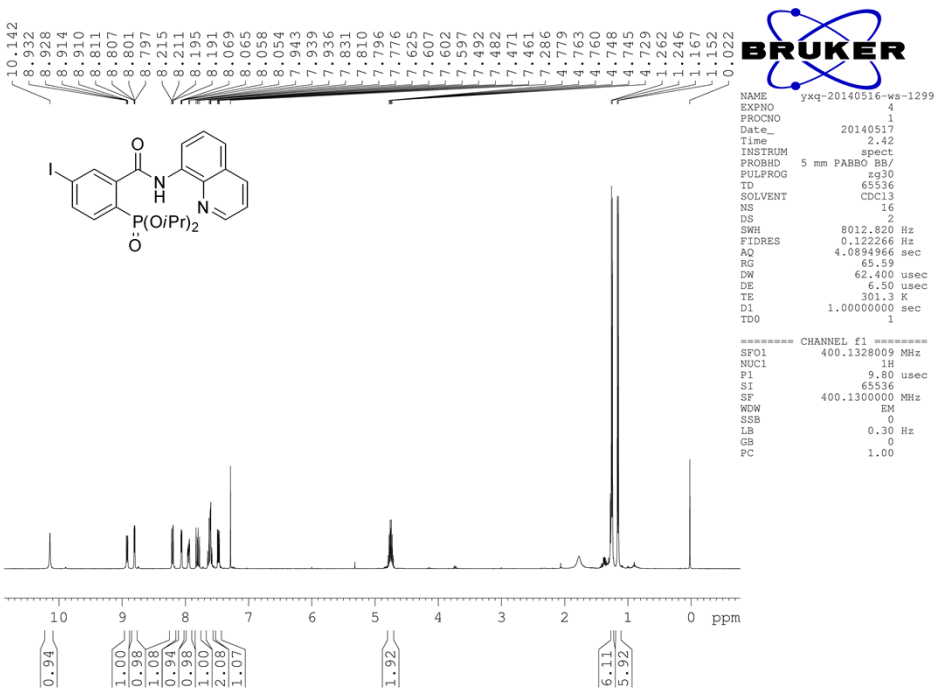

YXQ-20140416-WS-1222
P31CPD CDCl3 (D:\yxq) st 40
12.97
O
N
H
N
O
O
P(OiPr)2
O
35000
30000
25000
20000
15000
10000
5000
0
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
f1 (ppm)

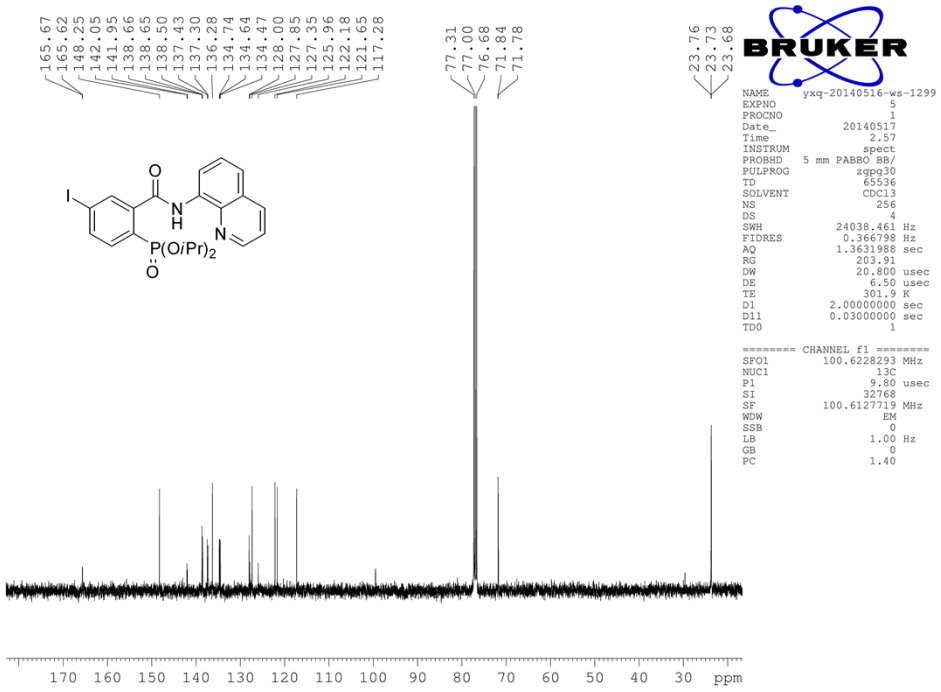

YXQ-20140416-WS-1221
P31CPD CDCl3 {D:\yxq} st 42
15.04
O
H3C
N
H
N
P(OiPr)2
O
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20
f1 (ppm)

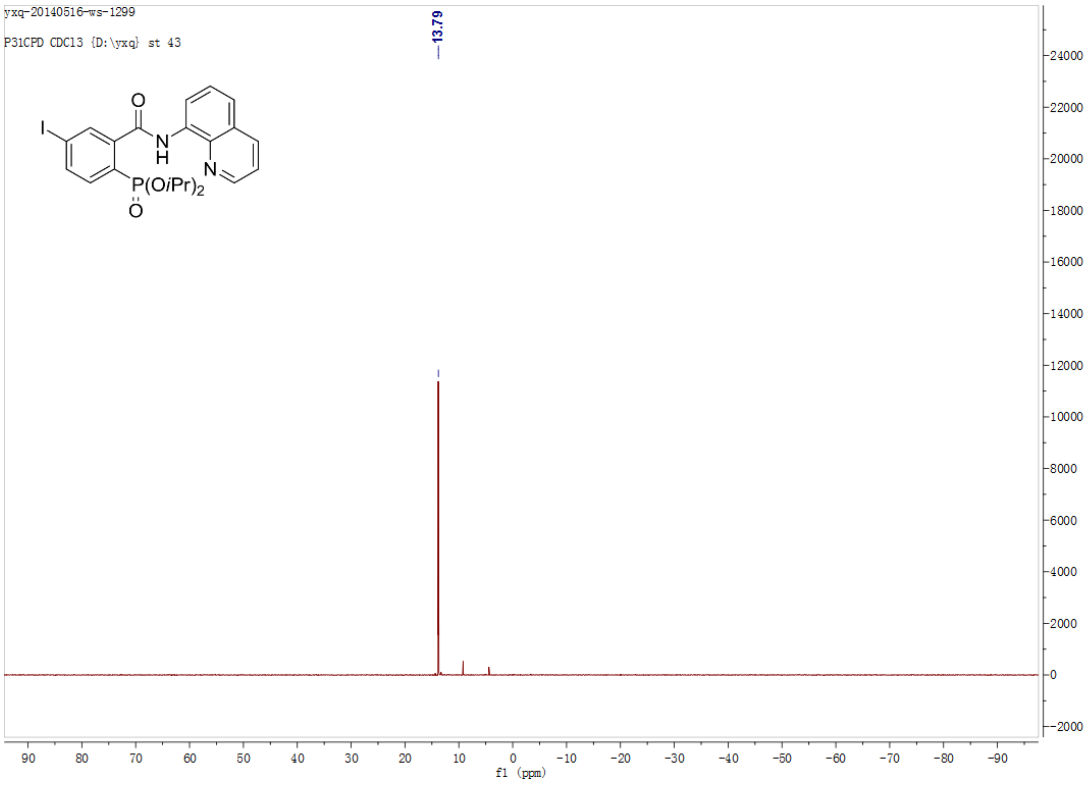

yxq-20140516-ws-1303
P31CPD CDCl3 (D:\yxq) st 46
MeO
O
N
H
N
P(OiPr)2
O
15.17
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
10
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
f1 (ppm)

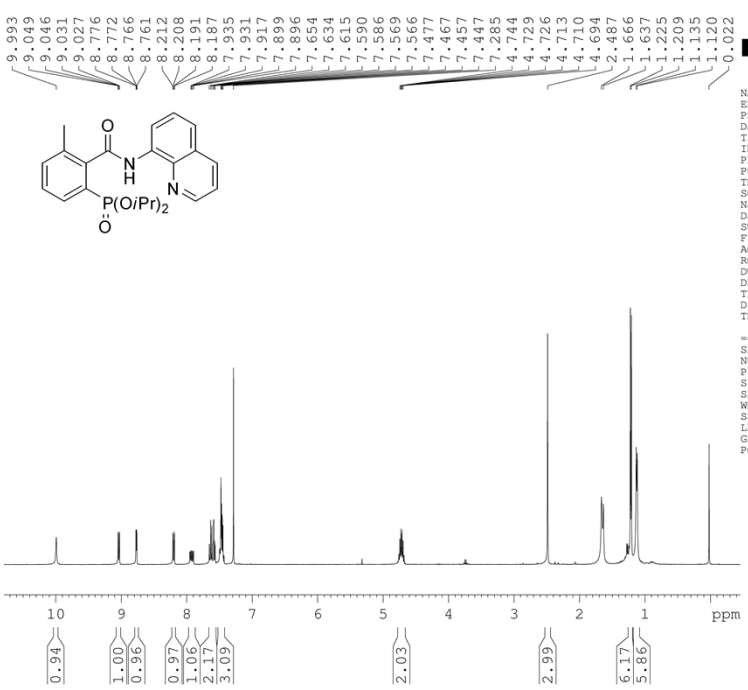

yxq-20140516-ws-1298
P31
13.31
Cl
O
N
H
N
P(OiPr)2
O
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
f1 (ppm)


yxq-20140516-ws-1302
P31CPD CDCl3 (D:\yxq) st 45
13.46
Br
O
N
H
N
P(OiPr)2
O
f1 (ppm)


10.142
8.932
8.928
8.914
8.910
8.811
8.807
8.801
8.797
8.215
8.211
8.195
8.191
8.069
8.065
8.058
8.054
7.943
7.939
7.936
7.831
7.810
7.796
7.776
7.625
7.607
7.602
7.597
7.492
7.482
7.471
7.461
7.286
4.779
4.763
4.760
4.748
4.745
4.729
1.262
1.246
1.167
1.152
0.022
I
O
N
H
N
P(OiPr)2
O
BRUKER
NAME yxq-20140516-ws-1299
EXPNO 4
PROCNO 1
Date_ 20140517
Time 2.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 65.59
DW 62.400 usec
DE 6.50 usec
TE 301.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1328009 MHz
NUC1 1H
P1 9.80 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
0.94
1.00
0.98
1.08
0.94
0.98
1.00
2.08
1.07
1.92
6.11
5.92

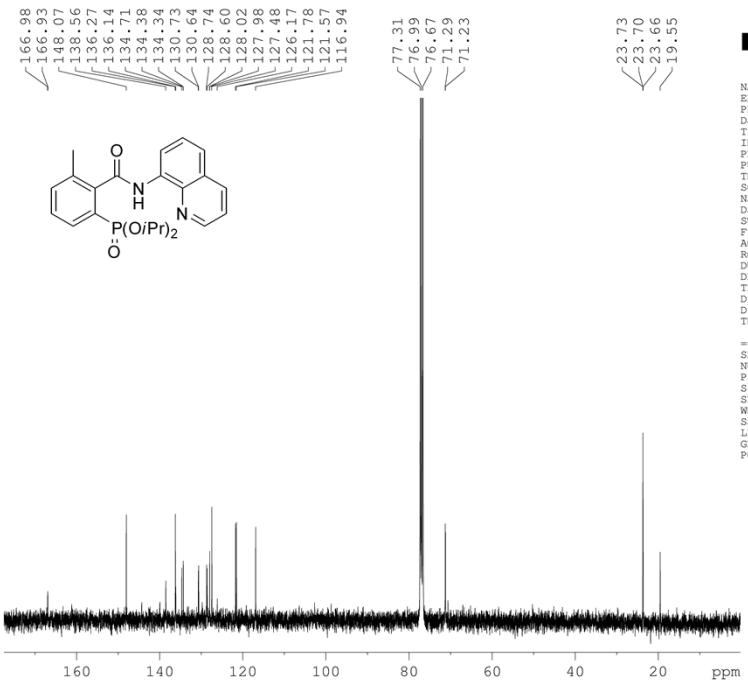

165.67
165.62
148.25
142.05
141.95
138.66
138.65
138.50
137.43
137.30
136.28
134.74
134.64
134.47
128.00
127.85
127.35
125.96
122.18
121.65
117.28
77.31
77.00
76.68
71.84
71.78
23.76
23.73
23.68
BRUKER
NAME yxq-20140516-ws-1299
EXPNO 5
PROCNO 1
Date_ 20140517
Time 2.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203.91
DW 20.800 usec
DE 6.50 usec
TE 301.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.80 usec
SI 32768
SF 100.6127719 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
I
O
N
H
N
P(O*i*Pr)2
O
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm


yxq-20140516-ws-1299
P31CPD CDCl3 {D:\yxq} st 43
13.79
I
O
N
H
N
P(O*i*Pr)2
O
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)



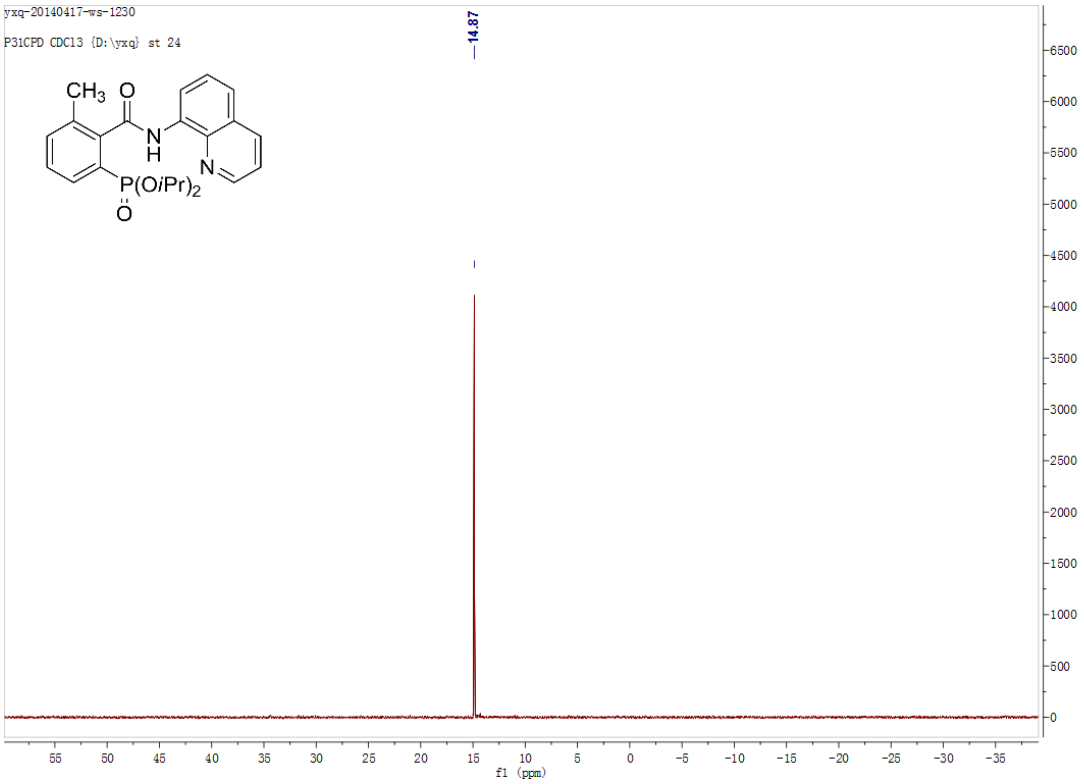

BRUKER

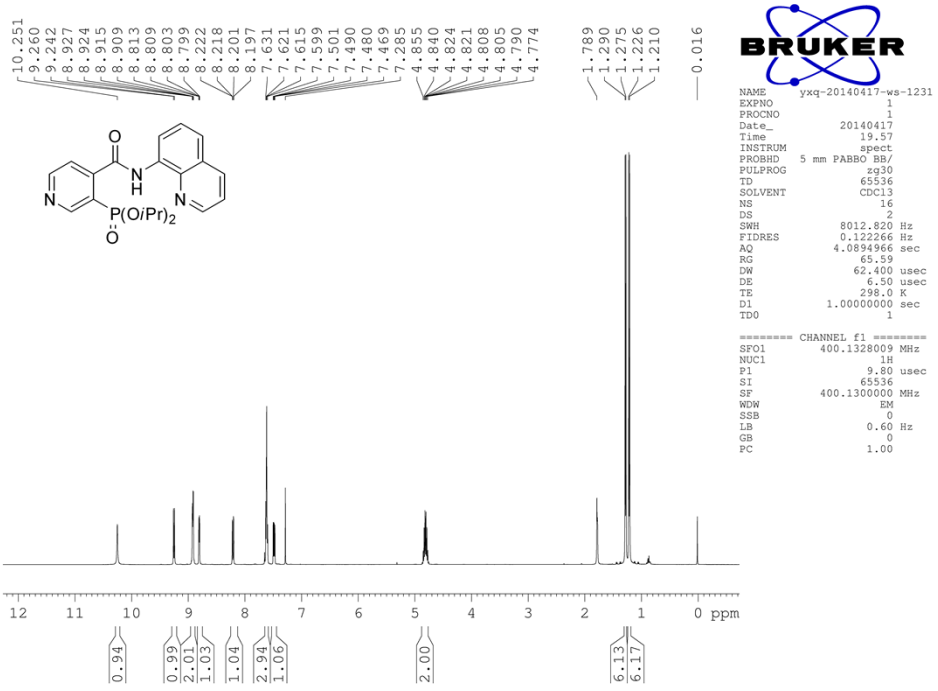
```
NAME          yxq-20140417-ws-1230
EXPNO                           1
PROCNO                          1
Date_                    20140417
Time                        22.18
INSTRUM                     spect
PROBHD   5 mm PABBO BB/
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                            16
DS                             2
SWH                     8012.820 Hz
FIDRES                  0.122266 Hz
AQ                    4.0894966 sec
RG                         91.97
DW                        62.400 usec
DE                         6.50 usec
TE                        298.2 K
D1                  1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                400.1328009 MHz
NUC1                        1H
P1                        9.80 usec
SI                         65536
SF                  400.1300000 MHz
WDW                           EM
SSB                            0
LB                         0.30 Hz
GB                             0
PC                          1.00
```

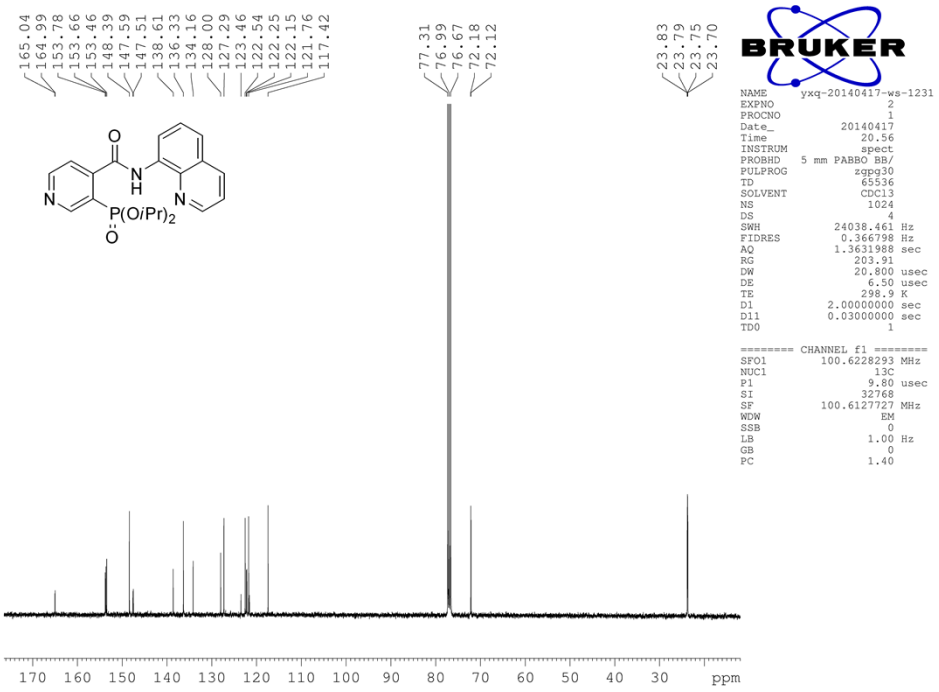


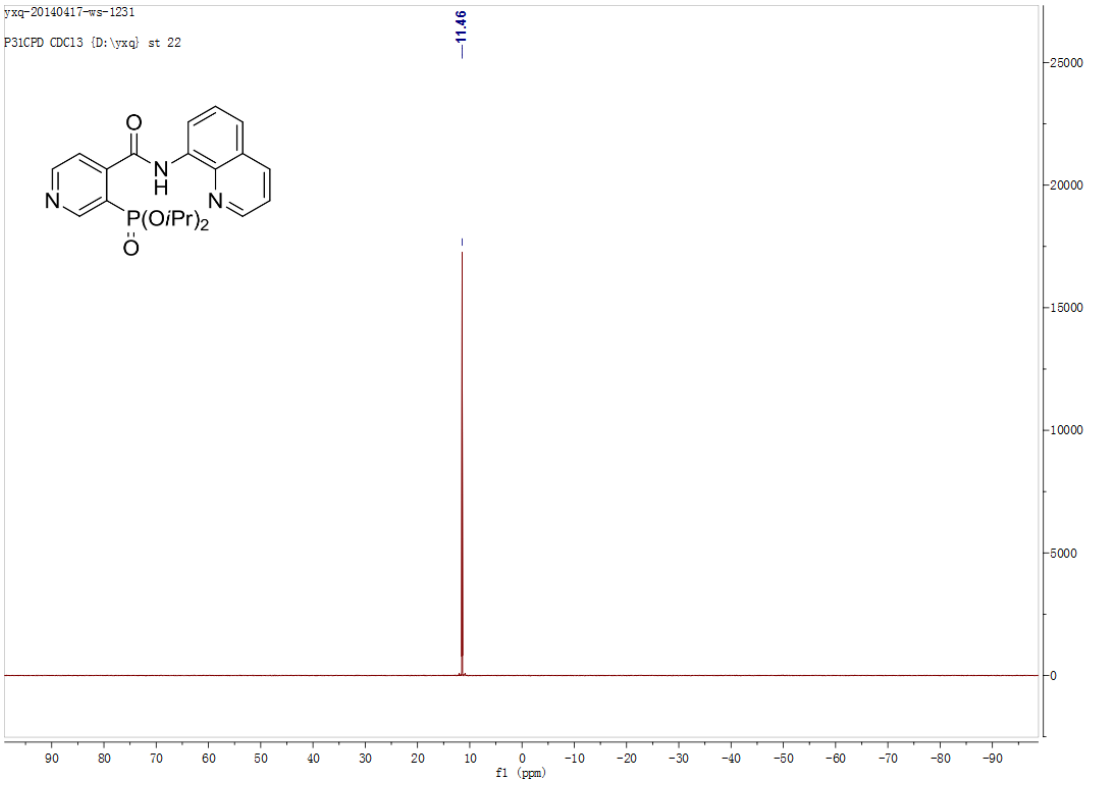

BRUKER

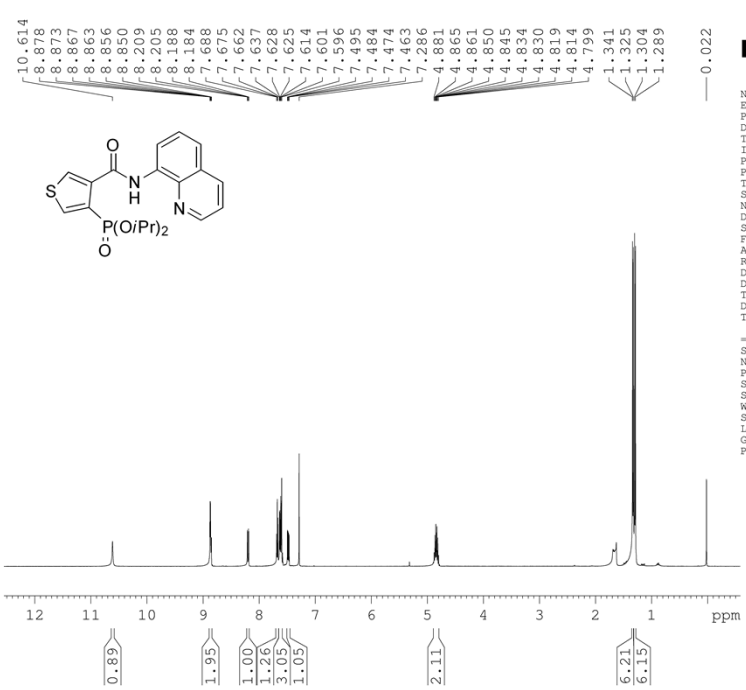
```
NAME          yxq-20140417-ws-1230
EXPNO                           2
PROCNO                          1
Date_                    20140417
Time                        23.18
INSTRUM                     spect
PROBHD   5 mm PABBO BB/
PULPROG                   zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                          1024
DS                             4
SWH                    24038.461 Hz
FIDRES                  0.366798 Hz
AQ                    1.3631988 sec
RG                        203.91
DW                        20.800 usec
DE                         6.50 usec
TE                        298.9 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                100.6228293 MHz
NUC1                        13C
P1                        9.80 usec
SI                         32768
SF                  100.6127727 MHz
WDW                           EM
SSB                            0
LB                         1.00 Hz
GB                             0
PC                          1.40
```


yxq-20140417-ws-1230
P31CPD CDCl3 (D:\yxq) st 24
14.87
f1 (ppm)


10.251
9.260
9.242
8.927
8.924
8.915
8.909
8.813
8.809
8.803
8.799
8.222
8.218
8.201
8.197
7.631
7.621
7.615
7.599
7.501
7.490
7.480
7.469
7.285
4.855
4.840
4.824
4.821
4.808
4.805
4.790
4.774
1.789
1.290
1.275
1.226
1.210
0.016
BRUKER
NAME yxq-20140417-ws-1231
EXPNO 1
PROCNO 1
Date_ 20140417
Time 19.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 65.59
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1328009 MHz
NUC1 1H
P1 9.80 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00
ppm
0.94
0.99
2.01
1.03
1.04
2.94
1.06
2.00
6.13
6.17

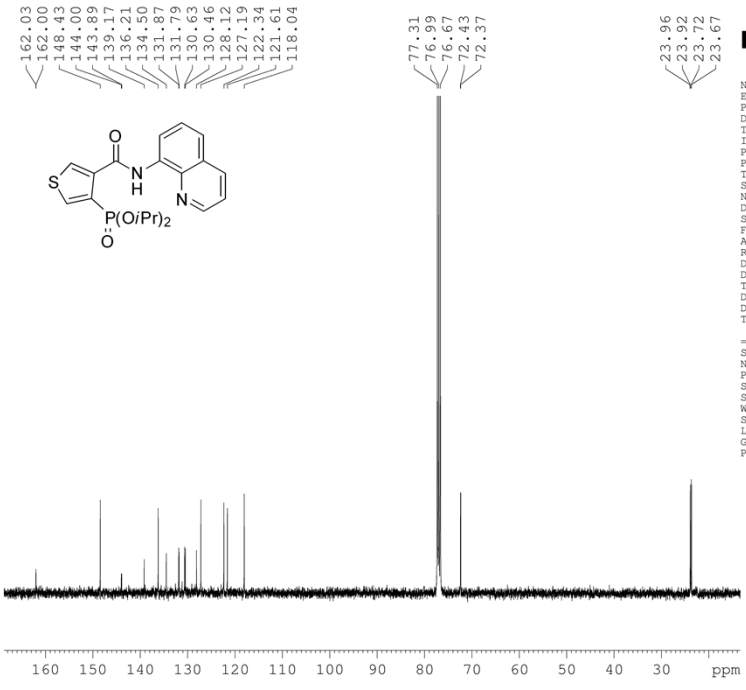

165.04
164.99
153.78
153.66
153.46
148.39
147.59
147.51
138.61
136.33
134.16
128.00
127.29
123.46
122.54
122.25
122.15
121.76
117.42
77.31
76.99
76.67
72.18
72.12
23.83
23.79
23.75
23.70
BRUKER
NAME yxq-20140417-ws-1231
EXPNO 2
PROCNO 1
Date_ 20140417
Time 20.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203.91
DW 20.800 usec
DE 6.50 usec
TE 298.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.80 usec
SI 32768
SF 100.6127727 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
N
H
N
N
P(OiPr)2
O
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm


yxq-20140417-ws-1231
P31CPD CDCl3 {D:\yxq} st 22
11.46
O
N
H
N
N
P(OiPr)2
O
25000
20000
15000
10000
5000
0
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)



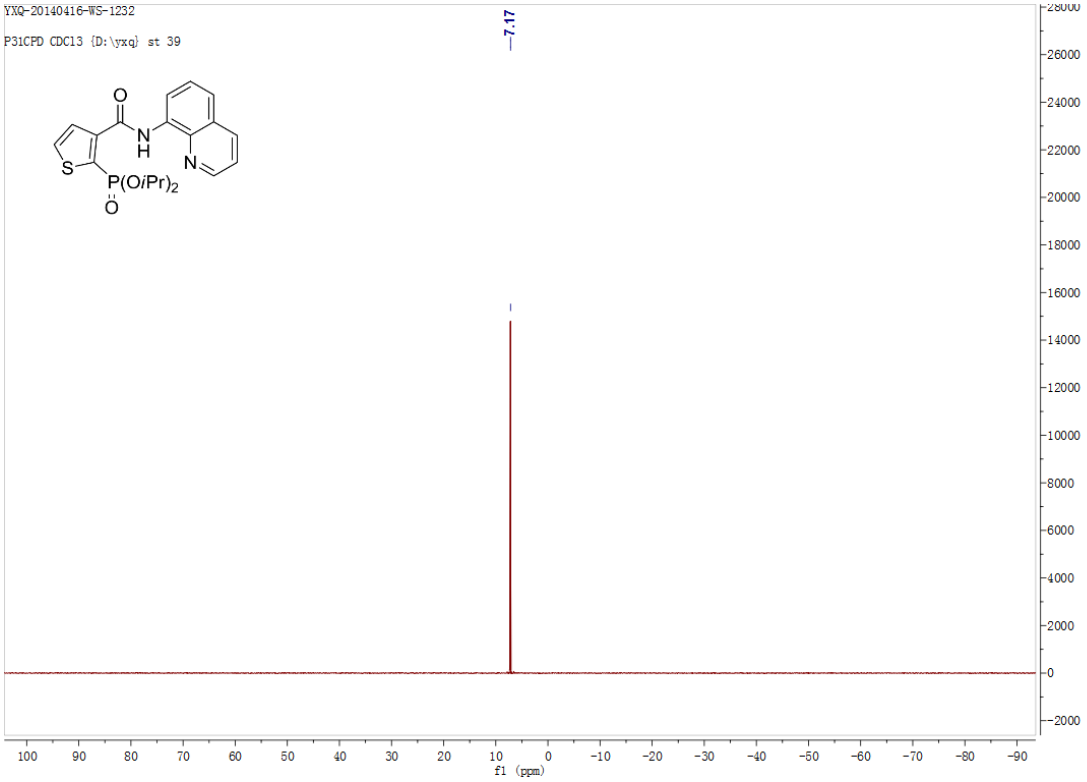

BRUKER

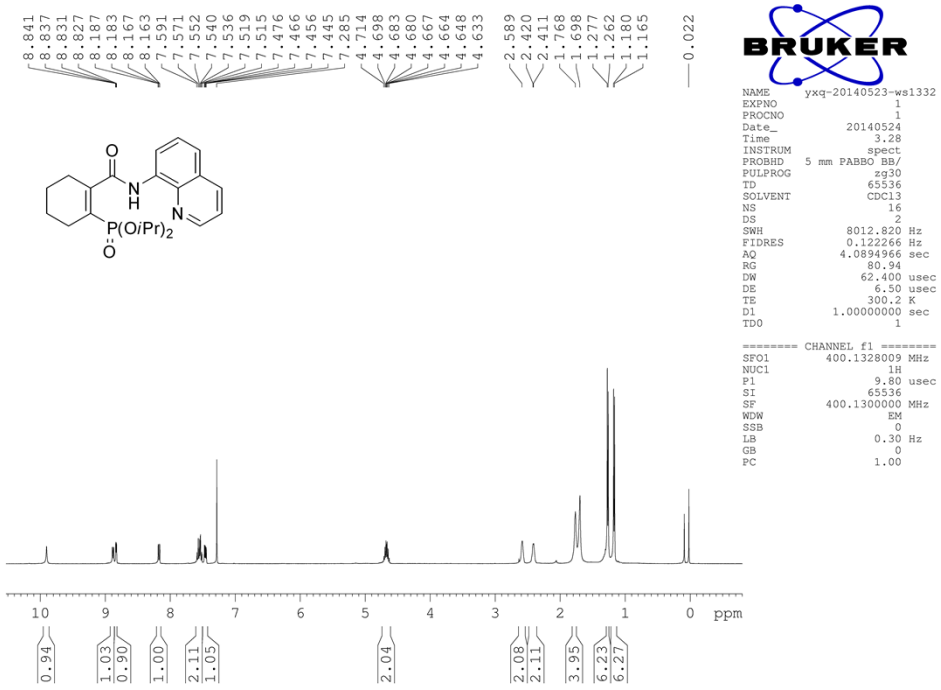
```
NAME          YXQ-20140416-WS-1232
EXPNO                           1
PROCNO                          1
Date_                    20140416
Time                        22.24
INSTRUM                     spect
PROBHD    5 mm PABBO BB/
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                            16
DS                             2
SWH                     8012.820 Hz
FIDRES                  0.122266 Hz
AQ                      4.0894966 sec
RG                          80.94
DW                         62.400 usec
DE                          6.50 usec
TE                         298.2 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                400.1328009 MHz
NUC1                        1H
P1                         9.80 usec
SI                        65536
SF                  400.1300000 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



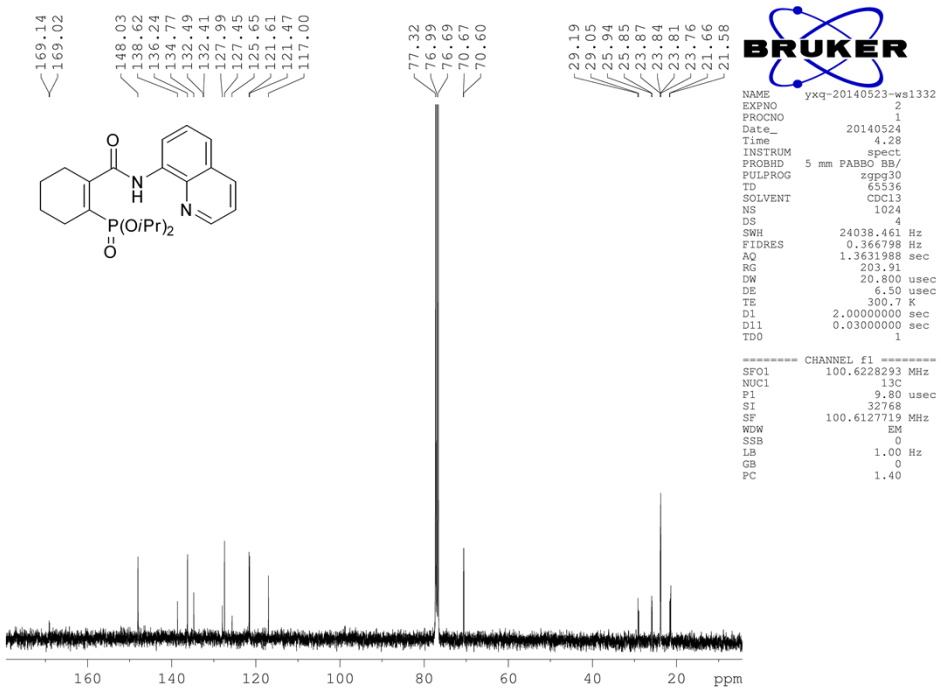

BRUKER

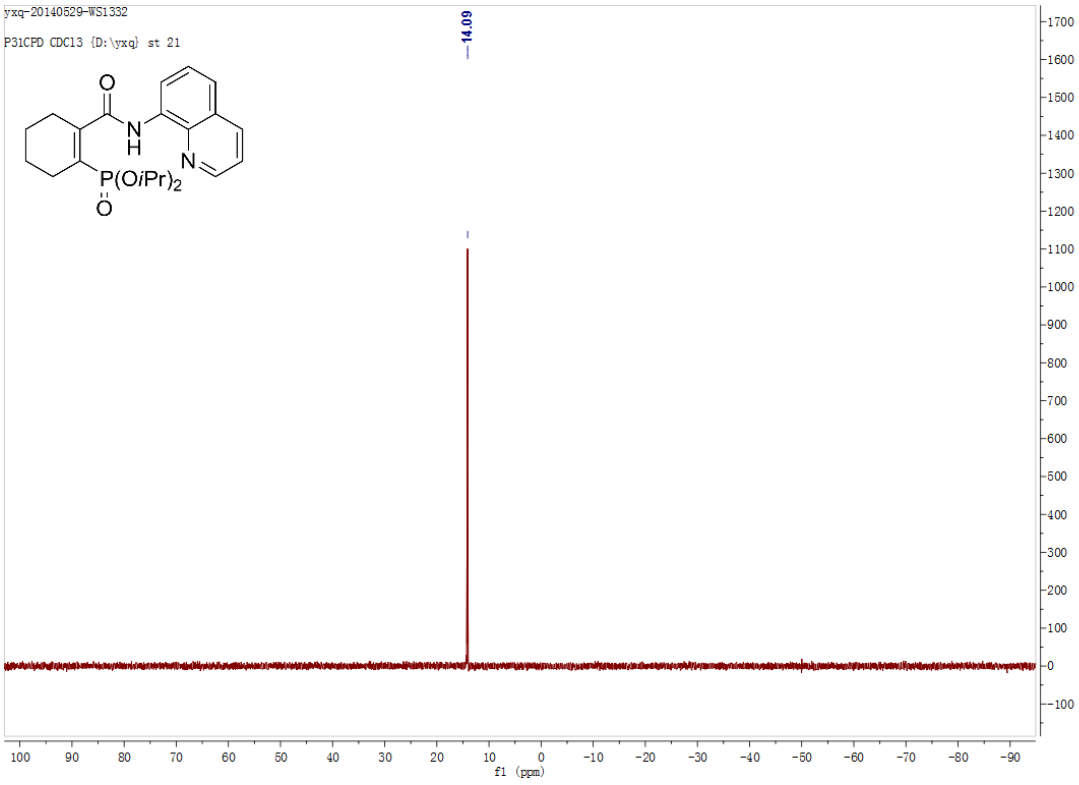
```
NAME          YXQ-20140416-WS-1232
EXPNO                           2
PROCNO                          1
Date_                    20140416
Time                        23.24
INSTRUM                     spect
PROBHD    5 mm PABBO BB/
PULPROG                   zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                          1024
DS                             4
SWH                    24038.461 Hz
FIDRES                  0.366798 Hz
AQ                      1.3631988 sec
RG                         203.91
DW                         20.800 usec
DE                          6.50 usec
TE                         299.3 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                100.6228293 MHz
NUC1                        13C
P1                         9.80 usec
SI                        32768
SF                  100.6127719 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

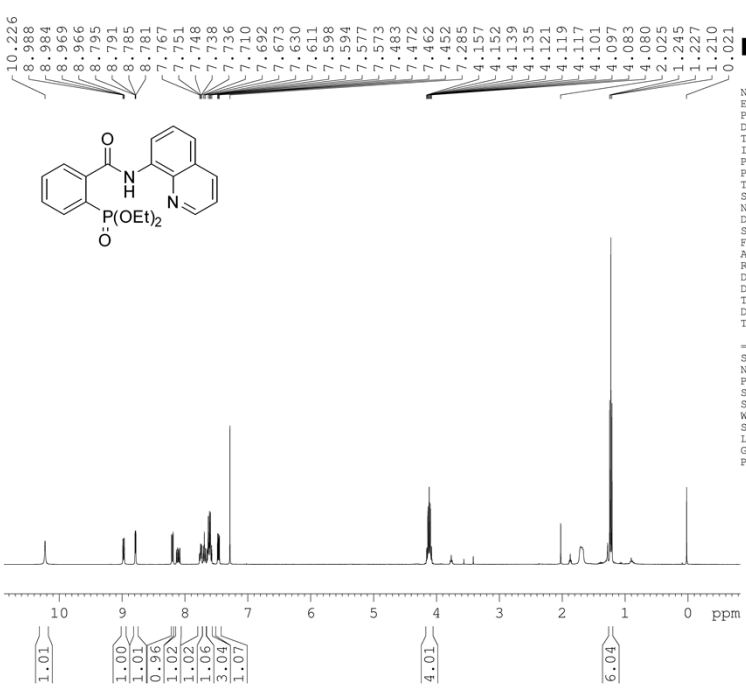



```
NAME          yxq-20140523-ws1332
EXPNO                           1
PROCNO                          1
Date_                    20140524
Time                         3.28
INSTRUM                      spect
PROBHD   5 mm PABBO BB/
PULPROG                      zg30
TD                          65536
SOLVENT                     CDCl3
NS                             16
DS                              2
SWH                    8012.820 Hz
FIDRES                 0.122266 Hz
AQ                     4.0894966 sec
RG                         80.94
DW                        62.400 usec
DE                         6.50 usec
TE                        300.2 K
D1                    1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
SFO1                 400.1328009 MHz
NUC1                          1H
P1                         9.80 usec
SI                          65536
SF                   400.1300000 MHz
WDW                            EM
SSB                             0
LB                         0.30 Hz
GB                              0
PC                         1.00
```

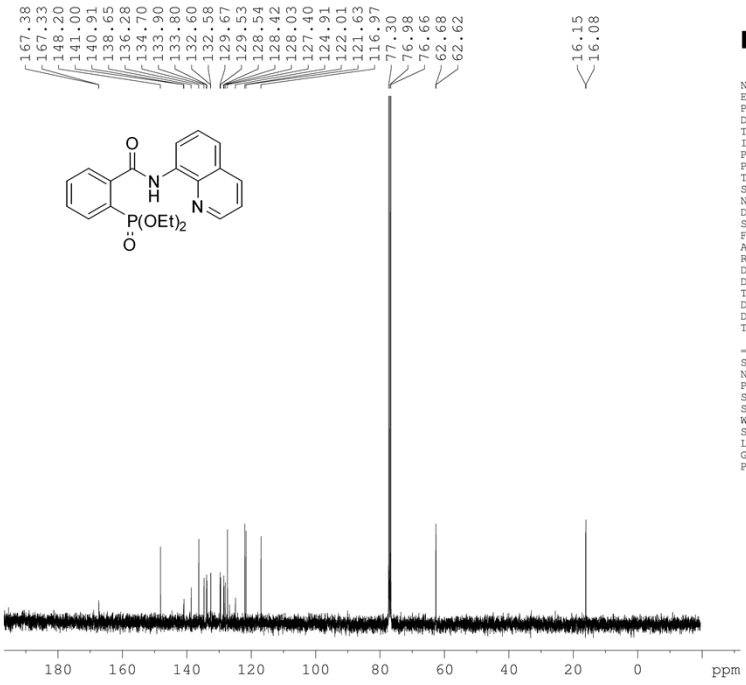

169.14
169.02
148.03
138.62
136.24
134.77
132.49
132.41
127.99
127.45
125.65
121.61
121.47
117.00
77.32
76.99
76.69
70.67
70.60
29.19
29.05
25.94
25.85
23.87
23.84
23.81
23.76
21.66
21.58
O
N
H
N
P(OiPr)2
O
BRUKER
NAME yxq-20140523-ws1332
EXPNO 2
PROCNO 1
Date_ 20140524
Time 4.28
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203.91
DW 20.800 usec
DE 6.50 usec
TE 300.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.80 usec
SI 32768
SF 100.6127719 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
140
120
100
80
60
40
20
ppm


yxq-20140529-WS1332
P31CPD CDC13 {D:\yxq} st 21
14.09
O
N
H
N
P(OiPr)2
O
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
f1 (ppm)



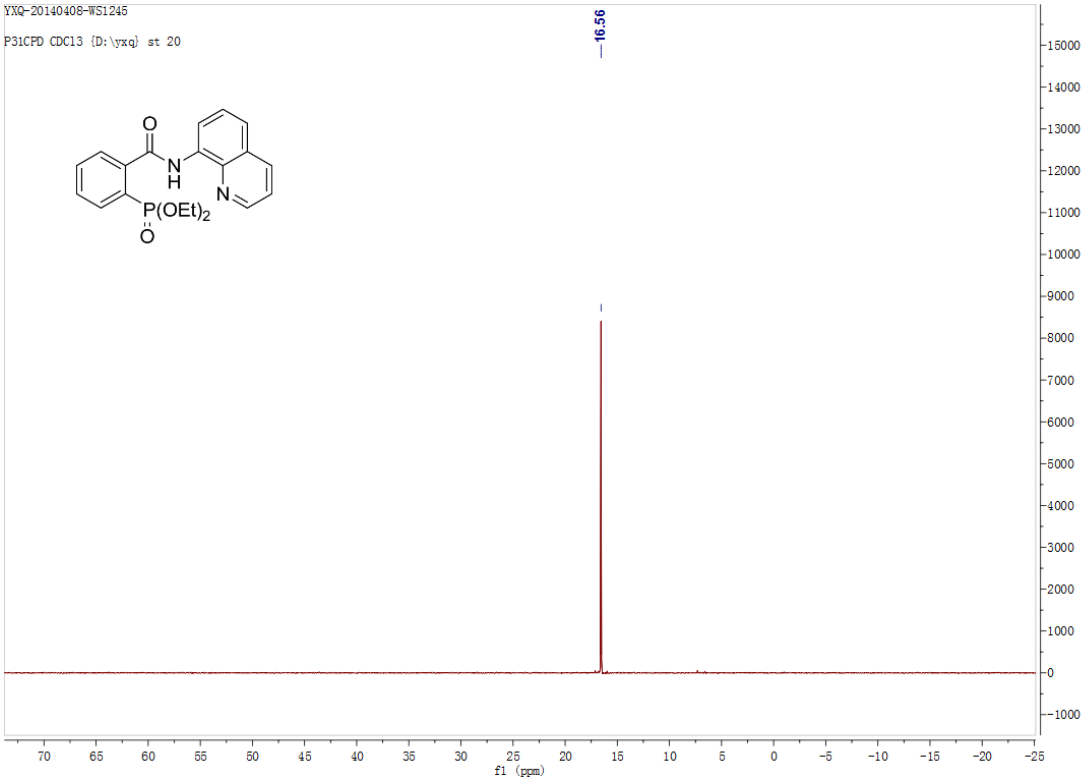

BRUKER

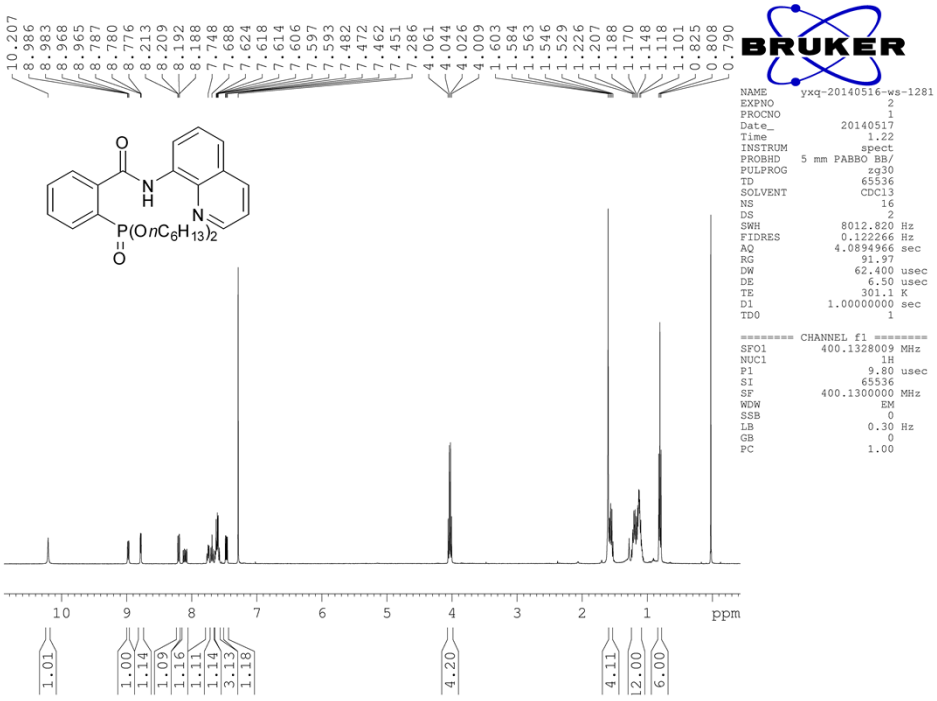
```
NAME          YXQ-20140408-WS1245
EXPNO                          1
PROCNO                         1
Date_                   20140408
Time                       23.03
INSTRUM                    spect
PROBHD    5 mm PABBO BB/
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                            16
DS                             2
SWH                     8012.820 Hz
FIDRES                     0.122266 Hz
AQ                      4.08894966 sec
RG                         73.76
DW                         62.400 usec
DE                          6.50 usec
TE                        299.3 K
D1                     1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                400.1328009 MHz
NUC1                        1H
P1                        9.80 usec
SI                         65536
SF                  400.1300000 MHz
WDW                           EM
SSB                            0
LB                         0.30 Hz
GB                             0
PC                         1.00
```




BRUKER

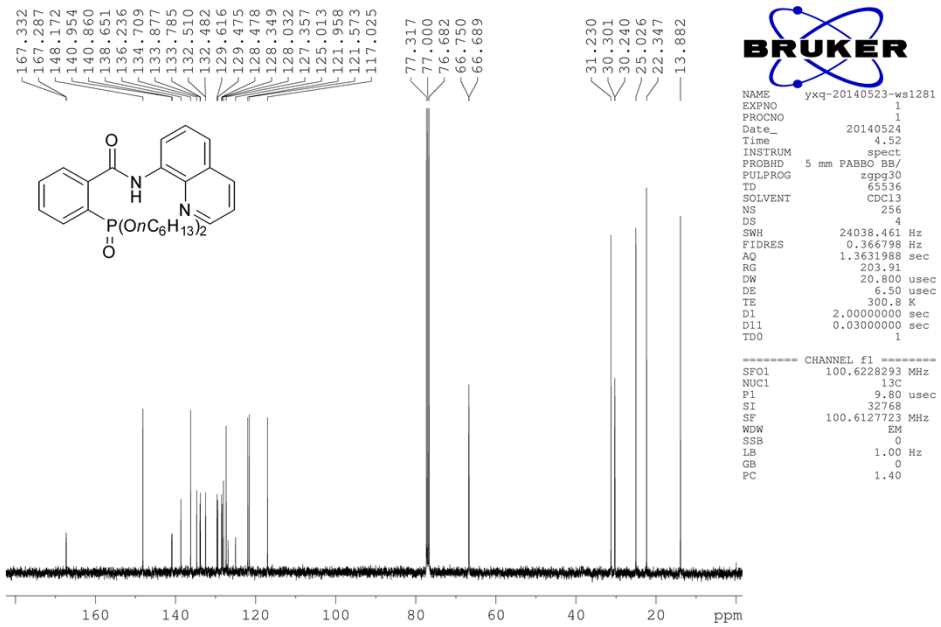
```
NAME          YXQ-20140408-WS1245
EXPNO                          2
PROCNO                         1
Date_                   20140408
Time                       23.19
INSTRUM                    spect
PROBHD    5 mm PABBO BB/
PULPROG                  zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                           256
DS                             4
SWH                    24038.461 Hz
FIDRES                     0.366798 Hz
AQ                      1.3631988 sec
RG                        203.91
DW                         20.800 usec
DE                          6.50 usec
TE                        299.9 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                100.6228293 MHz
NUC1                        13C
P1                        9.80 usec
SI                         32768
SF                  100.6127723 MHz
WDW                           EM
SSB                            0
LB                         1.00 Hz
GB                             0
PC                         1.40
```



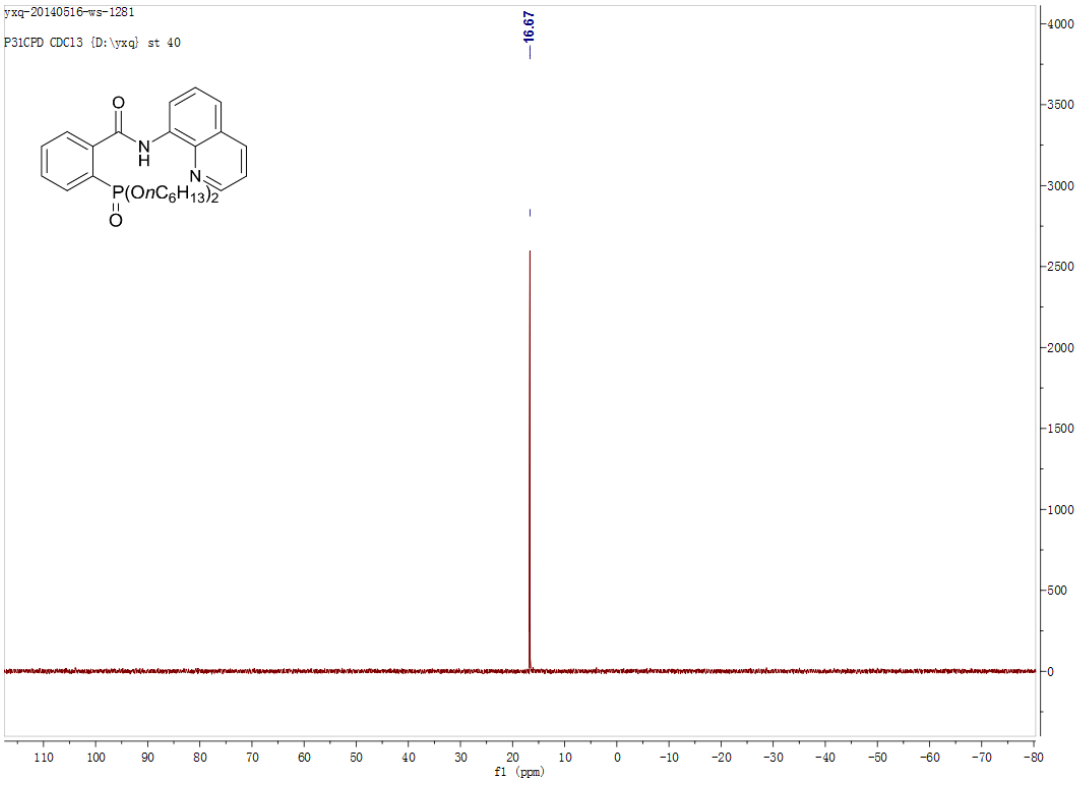



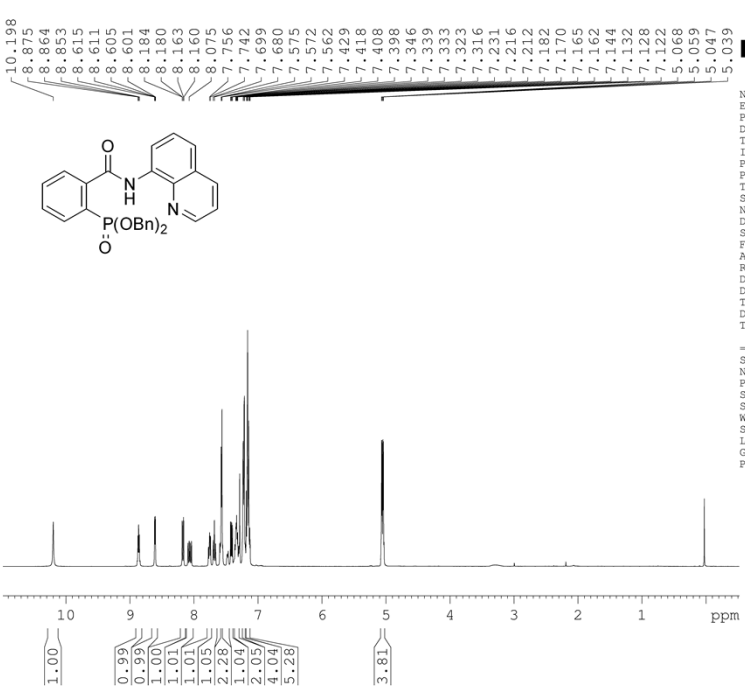

BRUKER

```
NAME          yxq-20140516-ws-1281
EXPNO                           2
PROCNO                          1
Date_                    20140517
Time                         1.22
INSTRUM                     spect
PROBHD   5 mm PABBO BB/
PULPROG                      zg30
TD                          65536
SOLVENT                     CDCl3
NS                             16
DS                              2
SWH                    8012.820 Hz
FIDRES                 0.122266 Hz
AQ                     4.0894966 sec
RG                         91.97
DW                        62.400 usec
DE                         6.50 usec
TE                        301.1 K
D1                    1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
SFO1                 400.1328009 MHz
NUC1                         1H
P1                         9.80 usec
SI                        65536
SF                   400.1300000 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```

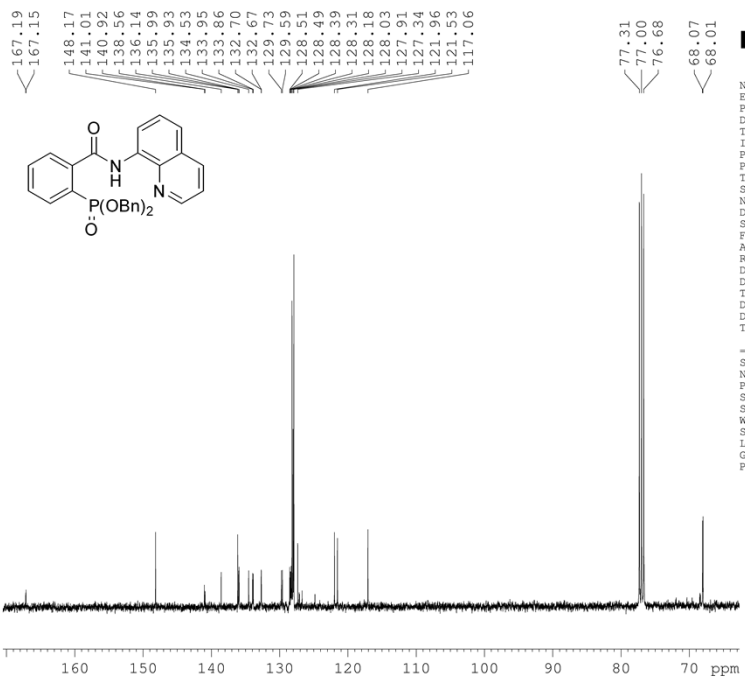



```
NAME            yxq-20140523-ws1281
EXPNO                            1
PROCNO                           1
Date_                     20140524
Time                          4.52
INSTRUM                      spect
PROBHD          5 mm PABBO BB/
PULPROG                   zgpg30
TD                             65536
SOLVENT                      CDCl3
NS                               256
DS                                 4
SWH                    24038.461 Hz
FIDRES                     0.366798 Hz
AQ                         1.3631988 sec
RG                            203.91
DW                            20.800 usec
DE                             6.50 usec
TE                            300.8 K
D1                        2.00000000 sec
D11                       0.03000000 sec
TD0                              1

======== CHANNEL f1 ========
SFO1                 100.6228293 MHz
NUC1                         13C
P1                            9.80 usec
SI                           32768
SF                   100.6127723 MHz
WDW                            EM
SSB                             0
LB                          1.00 Hz
GB                              0
PC                           1.40
```

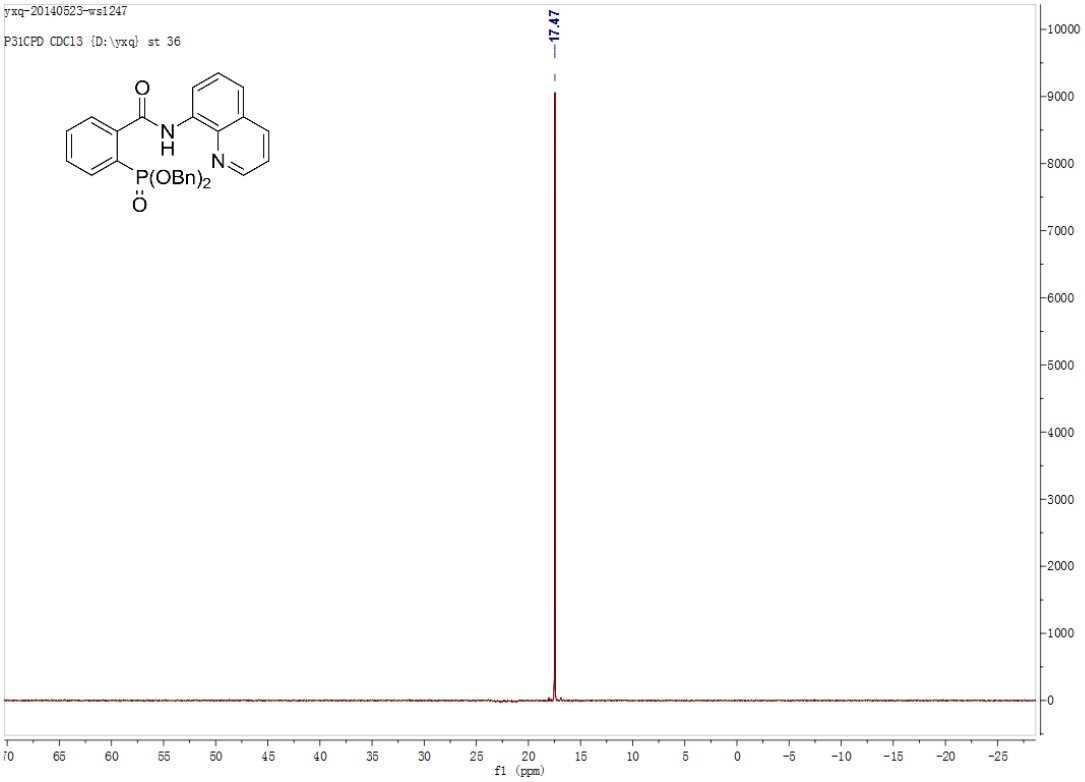





```
NAME          20140604-WS-1334
EXPNO                        1
PROCNO                       1
Date_             20140604
Time                    17.12
INSTRUM                 spect
PROBHD   5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 8012.820 Hz
FIDRES              0.122266 Hz
AQ                  4.08894966 sec
RG                      56.59
DW                    62.400 usec
DE                     6.50 usec
TE                     299.3 K
D1                  1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1           400.1328009 MHz
NUC1                      1H
P1                      9.80 usec
SI                     65536
SF             400.1300000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```




```
NAME          20140604-WS-1334
EXPNO                        3
PROCNO                       1
Date_             20140604
Time                    17.35
INSTRUM                 spect
PROBHD   5 mm PABBO BB/
PULPROG              zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       256
DS                         4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631988 sec
RG                     203.91
DW                    20.800 usec
DE                     6.50 usec
TE                     300.1 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1           100.6228293 MHz
NUC1                      13C
P1                      9.80 usec
SI                     32768
SF             100.6127727 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```


yxq-20140623-ws1247
P31CPD CDCl3 (D:\yxq) st 36
17.47
O
N
H
N
P(OBn)2
O
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25
f1 (ppm)