Supporting Information

Efficient Visible Light-Mediated Cross-Dehydrogenative Coupling Reactions of Tertiary Amines Catalyzed by a Polymer-Immobilized Iridium-Based Photocatalyst

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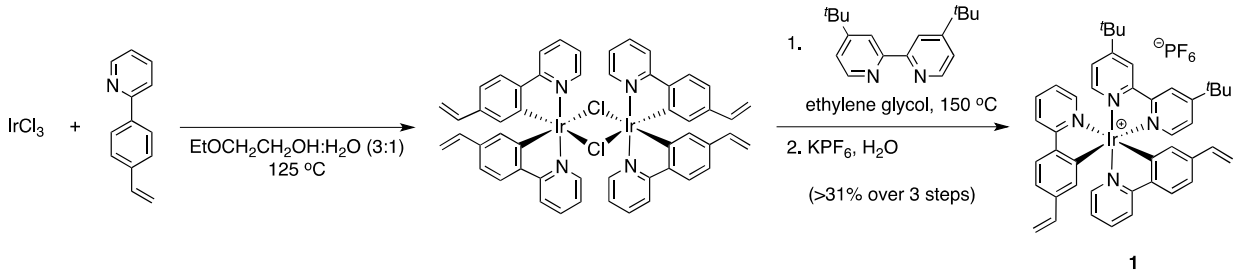
Contents

General Information: ^{1}H and ^{13}C NMR spectra were recorded on a JEOL ECX-400 in $CDCl_3$. Chemical shifts were reported in parts per million (ppm) from tetramethylsilane using the solvent resonance as the internal standard (chloroform: δ 7.26 ppm) for ^{1}H NMR and (deuterochloroform: δ 77.0 ppm) for ^{13}C NMR. ^{31}P NMR spectra were referenced to external H_3PO_4 (δ 0 ppm). ^{19}F NMR spectra were referenced to external benzotrifluoride (δ -63.7 ppm). IR spectra were measured on a JASCO FT/IR-610 spectrometer. High-resolution mass spectrometry was carried out using a JEOL JMS-T100TD (ESI). Preparative thin-layer chromatography (PTLC) was carried out using Wakogel B-5F from Wako Pure Chemical Industries, Ltd.

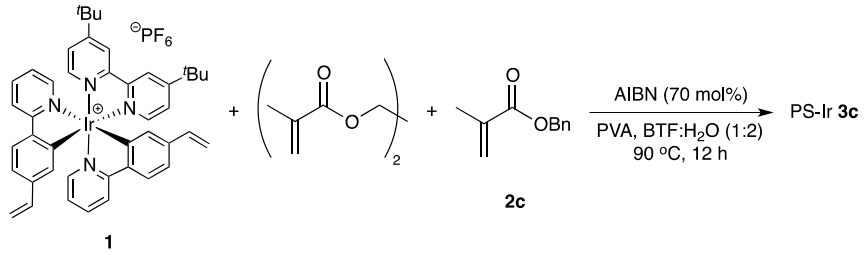
Reagents: Unless stated otherwise, commercial reagents were used as received. 2-(4-vinylphenyl)pyridine,[1] *N*-aryl tetrahydroisoquinolines **4a-e**,[2] and secondary phosphine oxides **5f-k**[3] were prepared according to known literature procedures.

Part I: Preparation of the Polymer-Supported Iridium Photocatalyst



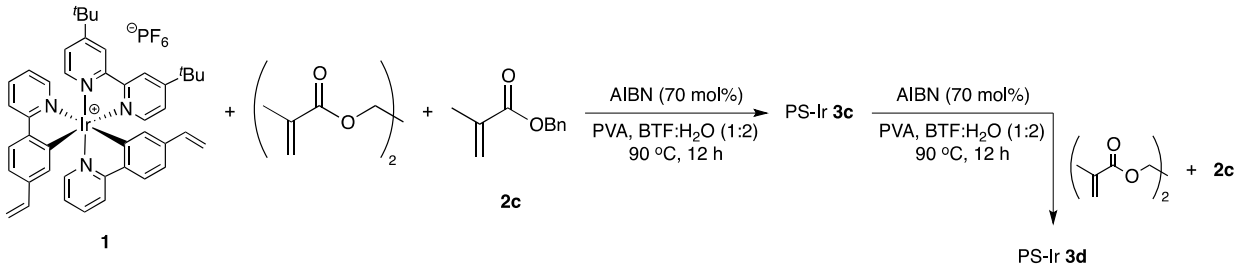
$[Ir(vppy)_2Cl]_2$. In a 50 mL round-bottom flask, $IrCl_3$ (0.2470 g, 0.8273 mmol) and 2-(4-vinylphenyl)pyridine (0.5738 g, 3.166 mmol) was dissolved in a solvent mixture of 2-ethoxyethanol (19 mL) and water (6.4 mL). The reaction vessel was fitted with a reflux condenser, flushed with argon gas, and placed in an oil bath set at 125 °C for 16 hours. The reaction mixture was allowed to cool to room temperature, filtered, and washed with H_2O, EtOH, and hexane. The yellow solid was allowed to dry under light vacuum and this crude product (403.6 mg, 0.3425 mmol, <41%) was used directly for the next procedure.

$Ir(vppy)_2(dtbbpy)PF_6$ (1). In a 30 mL round-bottom flask was added $[Ir(vppy)_2Cl]_2$ (0.4036 g, 0.3425 mmol), 4,4'-di-*tert*-butyl-2,2'-bipyridine (0.2023 g, 0.7537 mmol), and ethylene glycol (17 mL). The reaction flask was fitted with a reflux condenser, flushed with argon gas, and heated to 150 °C for 16 hours. The reaction mixture was allowed to cool to room temperature and then was poured into a KPF_6 solution (1.261 g in 170 mL of water). The resulting precipitate was filtered and then was purified by column chromatography (basic Al_2O_3, MeCN) to provide a bright yellow gel. The resulting gel was re-dissolved in a minimal amount of acetone, and then was precipitated in Et_2O to provide a bright yellow solid (0.2530 g, 0.2619 mmol, <76%). This crude solid was used directly for the suspension polymerization reaction.



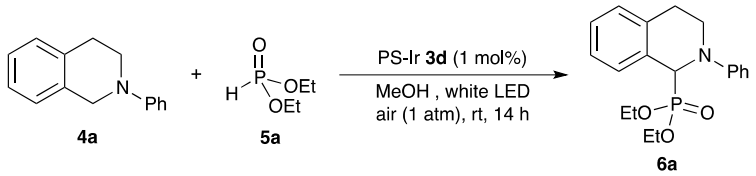
PS-Ir (3c). In a 10 mL test tube was added the crude Ir-based monomer **1** (0.0400 g, 0.0414 mmol), ethylene glycol dimethacrylate (24 µL, 0.13 mmol), benzyl methacrylate (**2c**) (400 µL, 2.35 mmol), azobisisobutyronitrile (AIBN) (0.0048 g, 0.0210 mmol), polyvinyl alcohol (0.0048 g), benzotrifluoride (BTF) (0.4 mL), and water (0.8 mL). The reaction vessel was degassed and was allowed to stir at 90 °C for 12 hours under an atmosphere of argon. The resulting solid was filtered and washed successively with water, MeOH, and DCM. The light orange cross-linked polymer was then lightly grounded with a mortar and pestle to give a bright yellow solid. The yellow polymer was washed with DCM and dried under high vacuum to provide **3c** (448.5 mg).

The iridium content of **3c** was determined using the following procedure: In a test tube containing 14.2 mg of **3c** was added conc. H_2SO_4 (1 mL) and then was heated to 200 °C for 1 hour in a heating block. Then conc. HNO_3 was added dropwise until the black solution turned into a light transparent yellow solution. After being heated for an additional 1 hour at 200 °C, the acidic solution was allowed to cool to room temperature. Then, 1 mL of aqua regia was added and then the solution was diluted to 50 mL with de-ionized using a volumetric flask. ICP analysis of the acid digested polymer **3c** was performed and the Ir content was determined using a calibration curve (2.85291 ppm Ir, 0.0523 mmol/g, expected Ir loading: 0.0851 mmol/g).



PS-Ir (3d). In a 50 mL round-bottom was added the crude Ir-based monomer **1** (0.3706 g, 0.3836 mmol), ethylene glycol dimethacrylate (235 µL, 1.246 mmol), benzyl methacrylate (**2c**) (3.8 mL, 22 mmol), azobisisobutyronitrile (AIBN) (0.0441 g, 0.2686 mmol), polyvinyl alcohol (0.0441 g), benzotrifluoride (BTF) (3.8 mL), and water (7.6 mL). The reaction vessel was degassed and was allowed to stir at 90 °C for 12 hours under an atmosphere of argon. The resulting solid was filtered and washed successively with water, MeOH, and DCM. The light orange cross-linked polymer was then lightly grounded with a mortar and pestle to give a bright yellow solid. The yellow polymer was washed with DCM and dried under high vacuum at 90 °C to provide PS-Ir **3c** (4.381 g). The dried yellow solid was then transferred to a 50 mL round-bottom flask and the above procedure was repeated to provide the double-coated cross-linked polymer **3d** (8.4226 g, 0.0255 mmol/g, expected Ir loading: 0.0437 mmol/g).

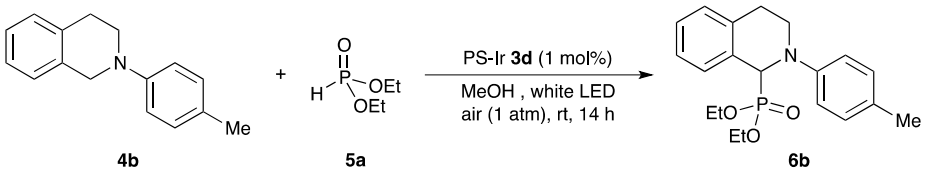
Part II: Substrate Scope for the CDC Reaction of *N*-Aryl Tetrahydroisoquinolines with P-H Nucleophiles



4a 5a 6a

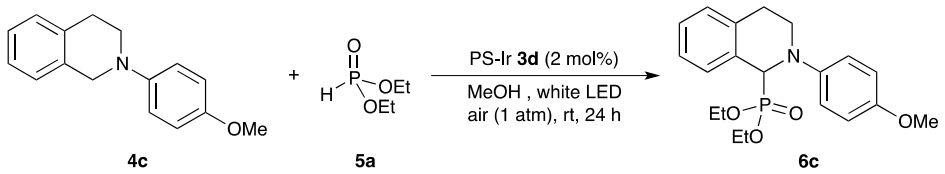
In a 2-necked test-tube was added PS-Ir **3d** (0.1996 g, 0.0255 mmol/g, 0.005 mmol, 1 mol%) and *N*-aryl tetrahydroisoquinoline **4a** (0.1046 g, 0.500 mmol). The reaction vessel was dried under high vacuum, and then re-pressurized with dry air. Then dialkyl phosphite **5a** (65 μL, 0.5046 mmol) and MeOH (1.6 mL) was added and the reaction mixture was allowed to stir for 14 h under a white LED lamp (Toshiba E-CORE LDA7N/2). Next, the heterogeneous Ir catalyst **3d** was filtered and washed with MeOH. The filtrate was concentrated under reduced pressure, and the crude product was purified by preparative TLC (1:1 EtOAc:hexane) to afford the desired α-amino phosphonate **6a** (0.1499 g, 0.4340 mmol, 87%) as a white solid.

Diethyl (2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (6a) (Table 2, entry 1). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.38-7.37 (m, 1H), 7.26-7.13 (m, 5H), 6.97 (d, 2H, J = 8.3 Hz), 6.78 (t, 1H, J = 7.4 Hz), 5.19 (d, 1H, J = 20.2 Hz), 4.11-3.86 (m, 5H), 3.65-3.59 (m, 1H), 3.10-2.98 (m, 2H), 1.24 (t, 3H, J = 6.9 Hz), 1.13 (t, 3H, J = 6.8 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 149.3 (d, $J_{C\text{-}P}$ = 5.7 Hz), 136.3 (d, $J_{C\text{-}P}$ =5.7 Hz), 130.6, 129.0, 128.6 (d, $J_{C\text{-}P}$ = 2.9 Hz), 128.0 (d, $J_{C\text{-}P}$ = 4.8 Hz), 127.3 (d, $J_{C\text{-}P}$ = 3.8 Hz), 125.8 (d, $J_{C\text{-}P}$ = 2.9 Hz), 118.4, 114.7, 63.2 (d, $J_{C\text{-}P}$ = 7.6 Hz), 62.2 (d, $J_{C\text{-}P}$ = 7.6 Hz), 58.7 (d, $J_{C\text{-}P}$ =160.2 Hz), 43.4, 26.7; ^{31}P NMR ($CDCl_3$, 160 MHz) δ 23.3. This is a known compound and the spectral data are identical to those reported in the literature.[4]

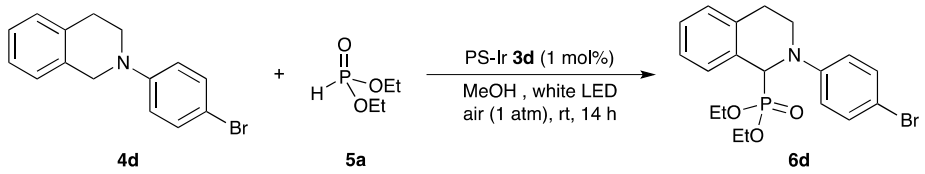


4b 5a 6b

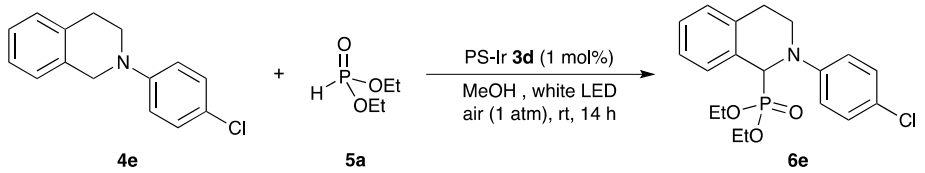
Diethyl (2-(*p*-tolyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (6b) (Table 2, entry 2). Following the above general procedure with **4b** (111.7 mg, 0.500 mmol) and **5a** (65 μL, 0.505 mmol) for 14 h. The crude reaction mixture was purified by preparative TLC (1:1 EtOAc:hexane) to provide **6b** (0.1736 g, 0.483 mmol, 97%) as a white solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ□□□□□□□□□□□□□□m, 1H), 7.17-7.10 (m, 3H), 7.03 (d, 2H, J = 8.3 Hz), 6.87 (d, 2H, J = 8.2 Hz), 5.11 (d, 1H, J = 20.6 Hz), 4.16-3.86 (m, 5H), 3.60-3.56 (m, 1H), 2.98-2.97 (m, 2H), 2.24 (s, 3H), 1.24 (t, 3H, J = 6.9 Hz), 1.16 (t, 3H, J = 6.9 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 147.4, 136.4 (d, $J_{C\text{-}P}$ = 5.7 Hz), 130.6, 129.6, 128.8 (d, $J_{C\text{-}P}$ = 1.9 Hz), 128.1, 127.9, 127.3 (d, $J_{C\text{-}P}$ = 3.8 Hz), 125.7 (d, $J_{C\text{-}P}$ = 2.9 Hz), 115.3, 63.3 (d, $J_{C\text{-}P}$ = 6.7 Hz), 62.2 (d, $J_{C\text{-}P}$ = 7.6 Hz), 59.0 (d, $J_{C\text{-}P}$ = 157.3 Hz), 43.8, 26.4, 20.2, 16.4 (d, $J_{C\text{-}P}$ = 5.7 Hz), 16.3 (d, $J_{C\text{-}P}$ = 5.7 Hz); ^{31}P NMR ($CDCl_3$, 160 MHz) δ 22.9. This is a known compound and the spectral data are identical to those reported in the literature.[5]



Diethyl (2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (6c) (Table 2, entry 3). Following the above general procedure with **4c** (119.7 mg, 0.500 mmol), **5a** (65 μL, 0.505 mmol), and heterogeneous catalyst **3d** (0.3992 g, 0.0255 mmol/g, 0.010 mmol, 2 mol%) for 24 h. The crude reaction mixture was purified by preparative TLC (1:1 EtOAc:hexane) to provide **6c** (0.1641 g, 0.437 mmol, 87%) as a white solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.38-7.36 (m, 1H), 7.17-7.10 (m, 3H), 6.90 (d, 2H, J = 8.7 Hz), 6.79 (d, 2H, J = 8.7 Hz), 5.00 (d, 1H, J = 21.5 Hz), 4.13-3.87 (m, 5H), 3.72 (s, 3H), 3.53-3.49 (m, 1H), 2.91-2.93 (m, 2H), 1.23 (t, 3H, J = 7.3 Hz), 1.14 (t, 3H, J = 7.3 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 153.1, 144.2, 136.4 (d, $J_{C\text{-}P}$ = 5.7 Hz), 130.5, 128.9 (d, $J_{C\text{-}P}$ = 2.9 Hz), 128.1 (d, $J_{C\text{-}P}$ = 4.8 Hz), 127.2 (d, $J_{C\text{-}P}$ = 2.9 Hz), 125.8 (d, $J_{C\text{-}P}$ = 2.9 Hz), 117.6, 114.5, 63.3 (d, $J_{C\text{-}P}$ = 6.7 Hz), 62.2 (d, $J_{C\text{-}P}$ = 7.6 Hz), 59.4 (d, $J_{C\text{-}P}$ = 157.2 Hz), 55.6, 44.6, 26.1, 16.4 (d, $J_{C\text{-}P}$ = 5.7 Hz), 16.3 (d, $J_{C\text{-}P}$ = 5.7 Hz); ^{31}P NMR ($CDCl_3$, 160 MHz) δ 22.8. This is a known compound and the spectral data are identical to those reported in the literature.[4]

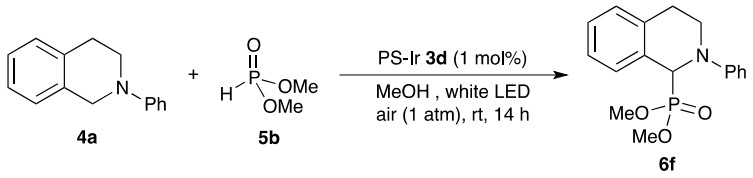


Diethyl (2-(4-bromophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (6d) (Table 2, entry 4). Following the above general procedure with **4d** (144.1 mg, 0.500 mmol) and **5a** (65 μL, 0.505 mmol) for 14 h. The crude reaction mixture was purified by preparative TLC (1:1 EtOAc:hexane) to provide **6d** (0.0827 g, 0.1949 mmol, 39%) as a white solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.34-7.29 (m, 3H), 7.21-7.13 (m, 3H), 6.84-6.81 (m, 2H), 5.08 (d, 1H, J = 19.3 Hz), 4.08-3.81 (m, 5H), 3.54-3.49 (m, 1H), 3.15-3.11 (m, 1H), 2.98-2.93 (m, 1H), 1.21 (t, 3H, J = 7.3 Hz), 1.19 (t, 3H, J = 7.3 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 148.3 (d, $J_{C\text{-}P}$ = 4.8 Hz), 136.3, 131.7, 130.4, 128.6 (d, $J_{C\text{-}P}$ = 2.9 Hz), 128.1 (d, $J_{C\text{-}P}$ = 4.8 Hz), 127.6 (d, $J_{C\text{-}P}$ = 2.9 Hz), 126.0 (d, $J_{C\text{-}P}$ = 2.9 Hz), 116.1, 110.3, 63.2 (d, $J_{C\text{-}P}$ = 7.6 Hz), 62.4 (d, $J_{C\text{-}P}$ = 7.6 Hz), 58.7 (d, $J_{C\text{-}P}$ = 158.3 Hz), 43.6, 26.9, 16.4 (d, $J_{C\text{-}P}$ = 5.7 Hz), 16.3 (d, $J_{C\text{-}P}$ = 3.7 Hz); ^{31}P NMR ($CDCl_3$, 160 MHz) δ 22.4. This is a known compound and the spectral data are identical to those reported in the literature.[6]



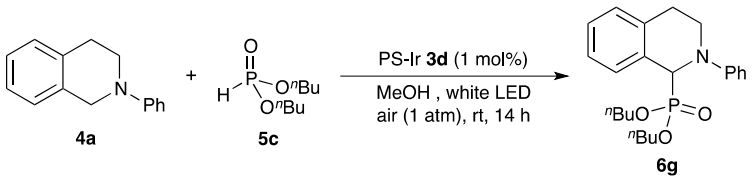
Diethyl (2-(4-chlorophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (6e) (Table 2, entry 5). Following the above general procedure with **4e** (121.9 mg, 0.500 mmol) and **5a** (65 μL, 0.505 mmol) for 14 h. The crude reaction mixture was purified by preparative TLC (1:1 EtOAc:hexane) to provide **6e** (0.0889 g, 0.2341 mmol, 47%) as a white solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.35-7.33 (m, 1H), 7.21-7.13 (m, 5H), 6.88-6.86 (m, 2H), 5.09 (d, 1H, J = 19.2 Hz),

4.10-3.81 (m, 5H), 3.55-3.49 (m, 1H), 3.14-3.10 (m, 1H), 2.98-2.93 (m, 1H), 1.21 (t, 3H, J = 7.3 Hz), 1.12 (t, 3H, J = 6.9 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 147.9 (d, $J_{C\text{-}P}$ = 4.8 Hz), 136.2 (d, $J_{C\text{-}P}$ = 5.7 Hz), 130.4, 128.6 (d, $J_{C\text{-}P}$ = 2.9 Hz), 128.1 (d, $J_{C\text{-}P}$ = 4.8 Hz), 127.6 (d, $J_{C\text{-}P}$ = 3.8 Hz), 126.0 (d, $J_{C\text{-}P}$ = 2.9 Hz), 123.1, 115.7, 63.2 (d, $J_{C\text{-}P}$ = 6.7 Hz), 62.4 (d, $J_{C\text{-}P}$ = 7.6 Hz), 58.8 (d, $J_{C\text{-}P}$ = 160.2 Hz), 43.7, 26.9, 16.4 (d, $J_{C\text{-}P}$ = 4.8 Hz), 16.3 (d, $J_{C\text{-}P}$ = 5.7 Hz); ^{31}P NMR ($CDCl_3$, 160 MHz) δ 22.5. This is a known compound and the spectral data are identical to those reported in the literature.[5]



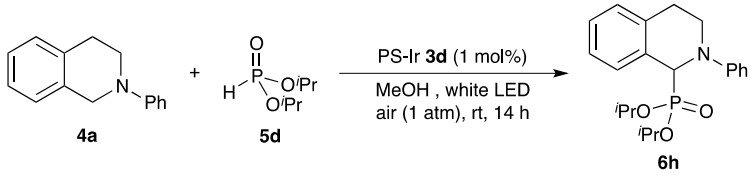
4a 5b 6f

Dimethyl (2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (6f) (Table 2, entry 6). Following the above general procedure with **4a** (104.6 mg, 0.500 mmol) and **5b** (46 μL, 0.502 mmol) for 14 h. The crude reaction mixture was purified by preparative TLC (1:1 EtOAc:hexane) to provide **6f** (0.1511 g, 0.4762 mmol, 95%) as a white solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.28-7.26 (m, 1H), 7.19-7.06 (m, 5H), 6.89 (d, 2H, J = 8.7 Hz), 6.72 (t, 1H, J = 7.3 Hz), 5.12 (d, 1H, J = 19.7 Hz), 3.96-3.90 (m, 1H), 3.58-3.54 (m, 7H), 3.02-2.87 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 149.2 (d, $J_{C\text{-}P}$ = 6.7 Hz), 136.3 (d, $J_{C\text{-}P}$ = 4.8 Hz), 130.4, 129.2, 128.8 (d, $J_{C\text{-}P}$ = 1.9 Hz), 127.9 (d, $J_{C\text{-}P}$ = 4.8 Hz), 127.5 (d, $J_{C\text{-}P}$ = 3.8 Hz), 126.0 (d, $J_{C\text{-}P}$ = 2.9 Hz), 118.6, 114.7, 58.7 (d, $J_{C\text{-}P}$ = 158.3 Hz), 53.9 (d, $J_{C\text{-}P}$ = 1.9 Hz), 53.8 (d, $J_{C\text{-}P}$ = 1.9 Hz), 43.5, 26.6; ^{31}P NMR ($CDCl_3$, 160 MHz) δ 25.0. This is a known compound and the spectral data are identical to those reported in the literature.[4]



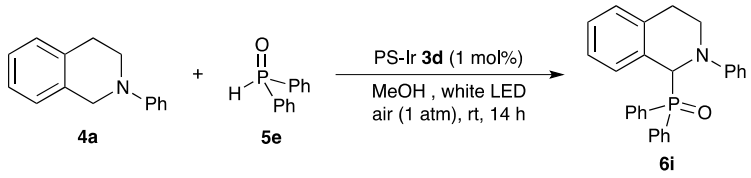
4a 5c 6g

Dibutyl (2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (6g) (Table 2, entry 7). Following the above general procedure with **4a** (104.6 mg, 0.500 mmol) and **5c** (98 μL, 0.502 mmol) for 14 h. The crude reaction mixture was purified by preparative TLC (1:1 EtOAc:hexane) to provide **6g** (0.1766 g, 0.4399 mmol, 88%) as a white solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.39-7.37 (m, 1H), 7.26-7.13 (m, 5H), 6.97 (d, 2H, J = 8.2 Hz), 6.78 (t, 1H, J = 7.3 Hz), 5.20 (d, 1H, J = 19.7 Hz), 4.06-3.76 (m, 5H), 3.66-3.60 (m, 1H), 3.15-2.97 (m, 2H), 1.60-1.51 (m, 2H), 1.49-1.42 (m, 2H), 1.38-1.20 (m, 4H), 0.88 (t, 3H, J = 7.3 Hz), 0.82 (t, 3H, J = 7.3 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 149.3, 136.4, 130.7, 129.0, 128.7 (d, $J_{C\text{-}P}$ = 1.9 Hz), 128.1 (d, $J_{C\text{-}P}$ = 4.8 Hz), 127.3 (d, $J_{C\text{-}P}$ = 2.9 Hz), 125.8 (d, $J_{C\text{-}P}$ = 2.9 Hz), 118.3, 114.7, 66.8 (d, $J_{C\text{-}P}$ = 7.6 Hz), 65.9 (d, $J_{C\text{-}P}$ = 7.6 Hz), 58.7 (d, $J_{C\text{-}P}$ = 157.3 Hz), 43.4, 32.5 (d, $J_{C\text{-}P}$ = 5.7 Hz), 32.4, 26.8, 18.6 (d, $J_{C\text{-}P}$ = 6.7 Hz), 13.5 (d, $J_{C\text{-}P}$ = 4.8 Hz); ^{31}P NMR ($CDCl_3$, 160 MHz) δ 23.0. This is a known compound and the spectral data are identical to those reported in the literature.[6]

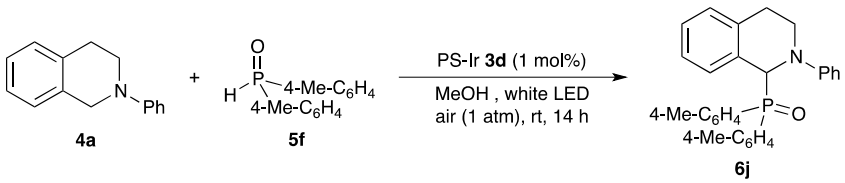


4a 5d 6h

Diisopropyl (2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (6h) (Table 2, entry 8). Following the above general procedure with **4a** (104.6 mg, 0.500 mmol) and **5d** (84 μL, 0.504 mmol) for 14 h. The crude reaction mixture was purified by preparative TLC (1:1 EtOAc:hexane) to provide **6h** (0.1260 g, 0.3374 mmol, 67%) as a white solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.43-7.41 (m, 1H), 7.27-7.13 (m, 5H), 6.97 (d, 2H, J = 8.2 Hz), 6.78 (t, 1H, J = 7.3 Hz), 5.16 (d, 1H, J = 21.1 Hz), 4.68-4.60 (m, 2H), 4.10-4.03 (m, 1H), 3.76-3.64 (m, 1H), 3.07-2.96 (m, 2H), 1.31 (t, 6H, J = 6.9 Hz), 1.18 (d, 3H, J = 6.0 Hz), 0.97 (d, 3H, J = 6.4 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 149.5 (d, $J_{C\text{-}P}$ = 6.7 Hz), 136.4 (d, $J_{C\text{-}P}$ = 5.7 Hz), 130.9, 128.9, 128.6 (d, $J_{C\text{-}P}$ = 2.9 Hz), 128.4 (d, $J_{C\text{-}P}$ = 4.8 Hz), 127.2 (d, $J_{C\text{-}P}$ = 2.9 Hz), 125.6 (d, $J_{C\text{-}P}$ = 2.9 Hz), 118.2, 115.0, 72.1 (d, $J_{C\text{-}P}$ = 7.6 Hz), 70.8 (d, $J_{C\text{-}P}$ = 8.6 Hz), 58.7 (d, $J_{C\text{-}P}$ = 159.2 Hz), 43.4, 26.5, 24.5 (d, $J_{C\text{-}P}$ = 2.9 Hz), 24.1 (d, $J_{C\text{-}P}$ = 3.8 Hz), 23.7 (d, $J_{C\text{-}P}$ = 4.8 Hz), 23.3 (d, $J_{C\text{-}P}$ = 5.7 Hz); ^{31}P NMR ($CDCl_3$, 160 MHz) δ 21.5. This is a known compound and the spectral data are identical to those reported in the literature.[6]

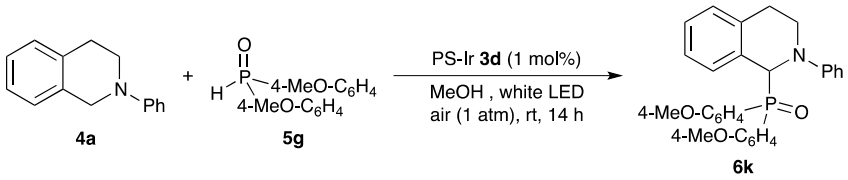


Diphenyl (2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphine oxide (6i) (Table 2, entry 9). Following the above general procedure with **4a** (104.6 mg, 0.500 mmol) and **5e** (101.1 mg, 0.500 mmol) for 14 h. The crude reaction mixture was purified by preparative TLC (1:49 MeOH:CH_2Cl_2) to provide **6i** (0.1874 g, 0.4577 mmol, 92%) as a white solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.83-7.67 (m, 4H), 7.53-7.29 (m, 6H), 7.14-7.05 (m, 4H), 6.92 (t, 1H, J = 7.4 Hz), 6.80-6.74 (m, 3H), 6.64 (d, 1H, J = 7.8 Hz). 5.56 (d, 1H, J = 10.5 Hz), 4.06-3.99 (m, 1H), 3.59-3.53 (m, 1H), 2.85-2.78 (m, 1H), 2.69-2.64 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 149.9, 136.8, 132.6, 132.1 (d, $J_{C\text{-}P}$ = 7.6 Hz), 131.8 (d, $J_{C\text{-}P}$ = 2.9 Hz), 131.7 (d, $J_{C\text{-}P}$ = 6.7 Hz), 131.6 (d, $J_{C\text{-}P}$ = 4.8 Hz), 130.8, 129.8, 129.1 (d, $J_{C\text{-}P}$ = 1.9 Hz), 129.0, 128.4, 128.3 (d, $J_{C\text{-}P}$ = 5.7 Hz), 128.1, 127.7 (d, $J_{C\text{-}P}$ = 2.9 Hz), 127.3 (d, $J_{C\text{-}P}$ = 2.9 Hz), 125.4 (d, $J_{C\text{-}P}$ = 2.9 Hz), 119.5, 116.7, 61.9 (d, $J_{C\text{-}P}$ = 79.1 Hz), 45.1, 25.5; ^{31}P NMR ($CDCl_3$, 160 MHz) δ 31.4. This is a known compound and the spectral data are identical to those reported in the literature.[5]

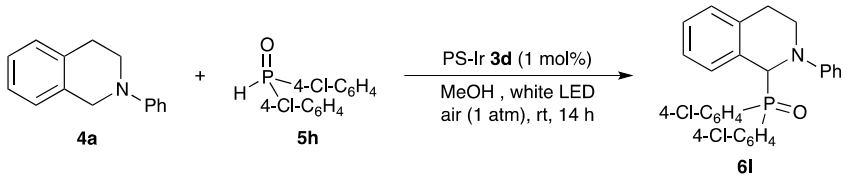


(2-Phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)di-*p*-tolylphosphine oxide (6j) (Table 2, entry 10). Following the above general procedure with **4a** (104.6 mg, 0.500 mmol) and **5f** (115.1 mg, 0.500 mmol) for 14 h. The crude reaction mixture was purified by preparative TLC (1:49 MeOH:CH_2Cl_2) to provide **6j** (0.1860 g, 0.4251 mmol, 85%) as a white solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.59-7.43 (m, 4H), 7.16-7.14 (m, 2H), 7.06-7.00 (m, 6H), 6.73-6.61 (m, 4H), 5.44 (d, 1H, J = 11.0 Hz), 3.95-3.88 (m, 1H), 3.50-3.47 (m, 1H), 2.75-2.71 (m, 1H), 2.58-2.53 (m, 1H), 2.30 (s, 3H), 2.23 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 149.9 (d, $J_{C\text{-}P}$ = 8.6 Hz), 142.2 (d, $J_{C\text{-}P}$ = 2.9 Hz), 141.9 (d, $J_{C\text{-}P}$

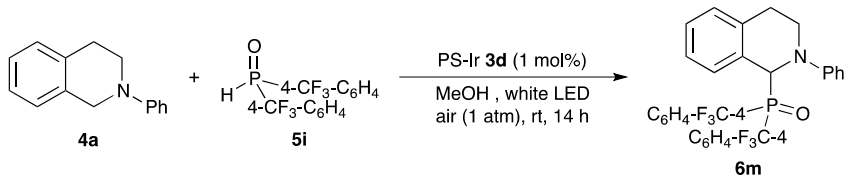
= 2.9 Hz); 136.7, 132.1 (d, $J_{C\text{-}P}$ = 8.6 Hz), 131.6 (d, $J_{C\text{-}P}$ = 9.5 Hz), 130.1, 129.6, 129.11, 129.08 (d, $J_{C\text{-}P}$ = 1.9 Hz), 128.97 (d, $J_{C\text{-}P}$ = 1.9 Hz), 128.8, 128.6 (d, $J_{C\text{-}P}$ = 5.7 Hz), 127.8 (d, $J_{C\text{-}P}$ = 2.9 Hz), 127.6, 127.2 (d, $J_{C\text{-}P}$ = 2.9 Hz), 125.4 (d, $J_{C\text{-}P}$ = 2.9 Hz), 119.2, 116.5, 61.9 (d, $J_{C\text{-}P}$ = 79.1 Hz), 44.8, 25.5, 21.5 (d, $J_{C\text{-}P}$ = 8.6 Hz); ^{31}P NMR ($CDCl_3$, 160 MHz) δ 31.8. This is a known compound and the spectral data are identical to those reported in the literature.[5]



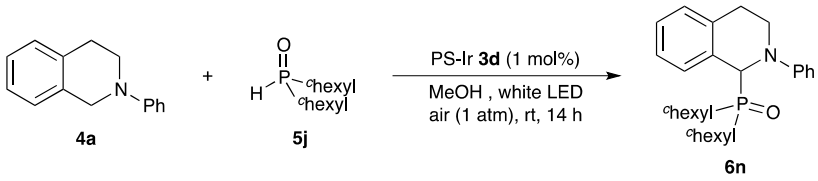
Bis(4-methoxyphenyl)(2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphine oxide (6k) (Table 2, entry 11). Following the above general procedure with **4a** (104.6 mg, 0.500 mmol) and **5g** (131.1 mg, 0.500 mmol) for 14 h. The crude reaction mixture was purified by preparative TLC (1:49 MeOH:CH_2Cl_2) to provide **6k** (0.2092 g, 0.4456 mmol, 89%) as a white solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.70-7.65 (m, 2H), 7.58-7.53 (m, 2H), 7.14-7.10 (m, 3H), 7.05-7.03 (m, 1H), 6.98-6.91 (m, 3H), 6.83-6.72 (m, 6H), 5.48 (d, 1H, J = 11.5 Hz), 3.98-3.89 (m, 1H), 3.80 (s, 3H), 3.74 (s, 3H), 3.60-3.52 (m, 1H), 2.84-2.78 (m, 1H), 2.63-2.58 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 162.3 (d, $J_{C\text{-}P}$ = 2.9 Hz), 162.1 (d, $J_{C\text{-}P}$ = 2.9 Hz), 149.9 (d, $J_{C\text{-}P}$ = 7.6 Hz), 136.7, 134.0 (d, $J_{C\text{-}P}$ = 9.5 Hz), 133.4 (d, $J_{C\text{-}P}$ = 9.5 Hz), 130.2, 129.0, 127.9 (d, $J_{C\text{-}P}$ = 2.9 Hz), 127.2 (d, $J_{C\text{-}P}$ = 2.9 Hz), 125.4 (d, $J_{C\text{-}P}$ = 2.9 Hz), 124.1, 123.1, 122.6, 121.7, 119.2, 116.3, 113.9 (d, $J_{C\text{-}P}$ = 11.4 Hz), 62.3 (d, $J_{C\text{-}P}$ = 80.1 Hz), 55.2, 55.1, 44.6, 25.6; ^{31}P NMR ($CDCl_3$, 160 MHz) δ 31.6; IR (KBr) cm^{-1} 3423 (m), 3023 (m), 2966 (m), 2840 (m), 1570 (s), 1502 (s), 1297 (s), 1255 (s), 1024 (s); ESI-HRMS (m/z) calcd. for $C_{29}H_{29}N_1O_3P_1$ [$(M+H)^+$]: 470.18850, found: 470.18622.



Bis(4-chlorophenyl)(2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphine oxide (6l) (Table 2, entry 12). Following the above general procedure with **4a** (104.6 mg, 0.500 mmol) and **5h** (135.5 mg, 0.500 mmol) for 14 h. The crude reaction mixture was purified by preparative TLC (1:49 MeOH:CH_2Cl_2) to provide **6l** (0.2080 g, 0.4348 mmol, 87%) as a white solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.70 (t, 2H, J = 8.2 Hz), 7.57 (t, 2H, J = 8.2 Hz), 7.43-7.40 (m, 2H), 7.31-7.28 (m, 2H), 7.17-7.06 (m, 3H), 6.98 (t, 1H, J = 7.8 Hz), 6.82-6.71 (m, 4H), 5.50 (d, 1H, J = 11.0 Hz), 4.03-3.91 (m, 1H), 3.56-3.51 (m, 1H), 2.84-2.77 (m, 1H), 2.66-2.61 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 149.8 (d, $J_{C\text{-}P}$ = 8.6 Hz), 138.7 (d, $J_{C\text{-}P}$ = 3.8 Hz), 138.4 (d, $J_{C\text{-}P}$ = 3.8 Hz), 136.7, 133.4 (d, $J_{C\text{-}P}$ = 9.5 Hz), 132.9 (d, $J_{C\text{-}P}$ = 9.5 Hz), 130.9, 130.1, 129.9, 129.3 (d, $J_{C\text{-}P}$ = 2.9 Hz), 129.2, 128.9, 128.7 (d, $J_{C\text{-}P}$ = 9.5 Hz), 128.6, 127.6 (d, $J_{C\text{-}P}$ = 2.9 Hz), 125.7 (d, $J_{C\text{-}P}$ = 2.9 Hz), 120.1, 117.2, 61.9 (d, $J_{C\text{-}P}$ = 80.1 Hz), 45.4, 25.4; ^{31}P NMR ($CDCl_3$, 160 MHz) δ 30.5. This is a known compound and the spectral data are identical to those reported in the literature.[5]



(2-Phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)bis(4-(trifluoromethyl)phenyl)phosphine oxide (6m) (Table 2, entry 13). Following the above general procedure with **4a** (104.6 mg, 0.500 mmol) and **5i** (169.1 mg, 0.500 mmol) for 14 h. The crude reaction mixture was purified by preparative TLC (1:49 MeOH:CH_2Cl_2) to provide **6m** (0.1658 g, 0.3040 mmol, 61%) as a white solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.92 (t, 2H, J = 9.6 Hz), 7.81 (t, 2H, J = 9.2 Hz), 7.71 (d, 2H, J = 6.4 Hz), 7.59 (d, 2H, J = 7.8 Hz), 7.20-7.09 (m, 4H), 6.98 (t, 1H, J = 7.8 Hz), 6.84-6.77 (m, 3H), 6.64 (d, 1H, J = 7.8 Hz), 5.59 (d, 1H, J = 10.1 Hz), 4.05-3.95 (m, 1H), 3.58-3.53 (m, 1H), 2.85-2.77 (m, 1H), 2.71-2.66 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 149.8, 149.7, 136.88, 136.84,136.5, 136.2, 135.6, 135.3, 134.16, 134.14, 133.85, 133.83, 133.53, 133.50, 132.5, 132.4, 132.1, 132.0, 129.58, 129.56, 129.3, 128.7, 127.90, 127.88, 127.42, 127.38, 125.81, 125.79, 125.52, 125.48, 125.45, 125.41, 125.38, 125.30, 125.26, 125.23, 125.19, 125.15, 125.11, 125.07, 125.04, 124.8, 122.1, 120.7, 117.7, 62.1, 61.3, 45.9, 25.4; ^{31}P NMR ($CDCl_3$, 160 MHz) δ 29.5; ^{19}F NMR ($CDCl_3$, 376 MHz) δ -64.2, -64.3; IR (KBr) cm^{-1} 3434 (m), 3061 (m), 2940 (m), 1598 (m), 1506 (m), 1327 (s), 1172 (s), 1065 (s), 1018 (m); ESI-HRMS (m/z) calcd. for $C_{29}H_{23}N_1O_1F_6P_1$ [(M+H)$^+$]: 546.14214, found: 546.14106.



Dicyclohexyl(2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphine oxide (6n) (Table 2, entry 14). Following the above general procedure with **4a** (104.6 mg, 0.500 mmol) and **5j** (107.1 mg, 0.500 mmol) for 14 h. The crude reaction mixture was purified by preparative TLC (1:49 MeOH:CH_2Cl_2) to provide **6n** (0.1616 g, 0.3833 mmol, 77%) as a white solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.41-7.40 (m, 1H), 7.20-7.12 (m, 4H), 7.05-7.03 (m, 1H), 6.89 (d, 2H, J = 8.2 Hz), 6.82 (t, 1H, J = 7.3 Hz), 5.05 (d, 1H, J = 13.8 Hz), 4.12-4.05 (m, 1H), 3.73-3.69 (m, 1H), 2.76-2.74 (m, 2H), 2.08-1.91 (m, 4H), 1.85-1.07 (m, 18H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 150.4 (d, $J_{C\text{-}P}$ = 9.5 Hz), 136.2 (d, $J_{C\text{-}P}$ = 3.8 Hz), 131.7, 129.4 (d, $J_{C\text{-}P}$ = 1.9 Hz), 129.2, 127.5 (d, $J_{C\text{-}P}$ = 3.8 Hz), 127.0 (d, $J_{C\text{-}P}$ = 2.9 Hz), 125.9 (d, $J_{C\text{-}P}$ = 1.9 Hz), 120.3, 118.3, 57.5 (d, $J_{C\text{-}P}$ = 1.9 Hz), 56.8 (d, $J_{C\text{-}P}$ = 2.9 Hz), 46.9, 37.8, 37.4, 36.8, 27.0 (d, $J_{C\text{-}P}$ = 7.6 Hz), 26.9 (d, $J_{C\text{-}P}$ = 3.8 Hz), 26.8 (d, $J_{C\text{-}P}$ = 2.9 Hz), 26.2 (d, $J_{C\text{-}P}$ = 3.8 Hz), 26.1, 26.0, 25.9 (d, $J_{C\text{-}P}$ = 3.8 Hz), 25.0; ^{31}P NMR ($CDCl_3$, 160 MHz) δ 52.9; IR (KBr) cm^{-1} 3426 (m), 3056 (w), 3032 (w), 2926 (s), 2852 (s), 1595 (s), 1505 (s), 1449 (m), 1312 (m), 1151 (s); ESI-HRMS (m/z) calcd. for $C_{27}H_{37}N_1O_1P_1$ [(M+H)$^+$]: 422.26128, found: 422.25985.

Part III: NMR Spectra of **6a-n**

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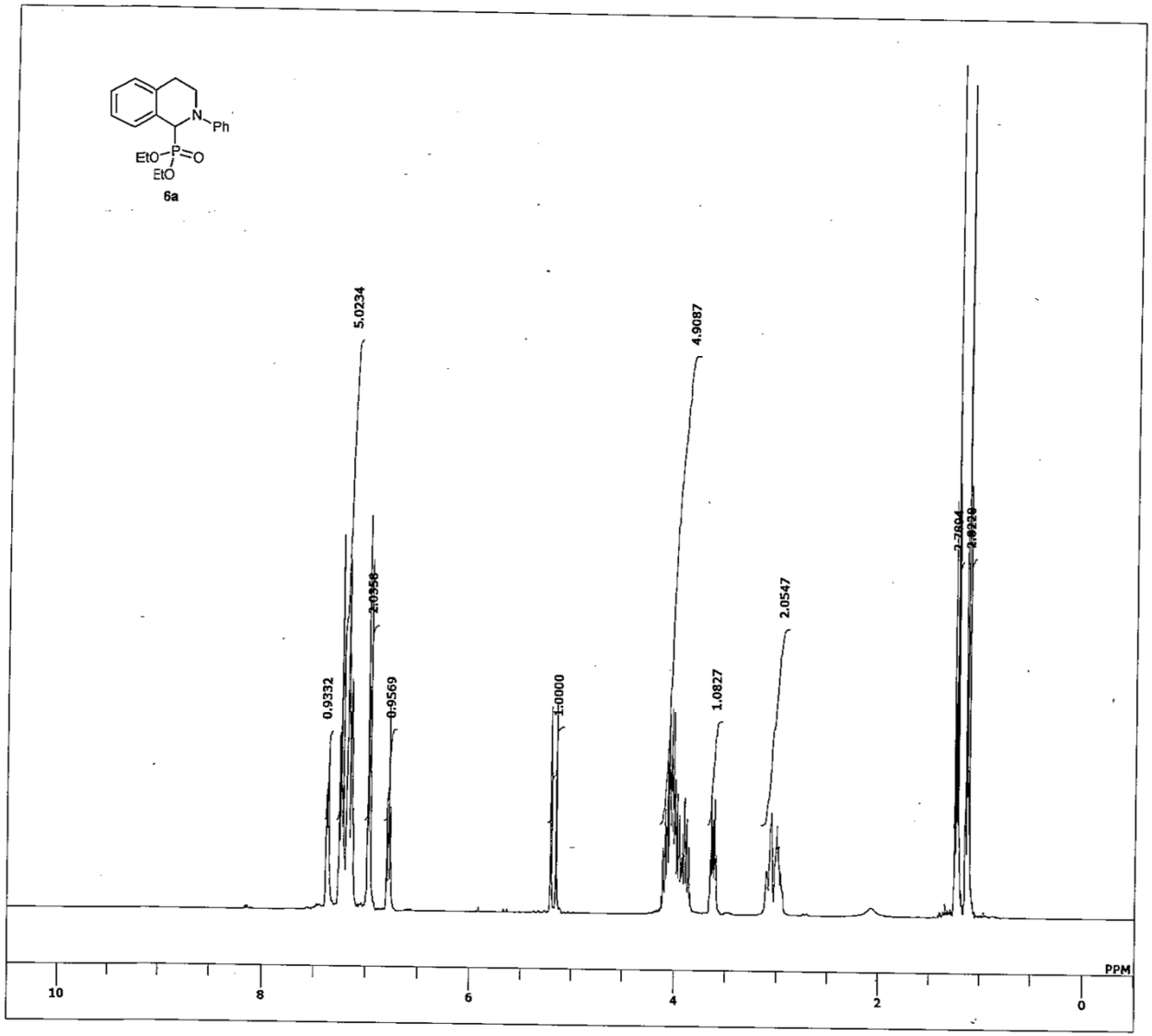

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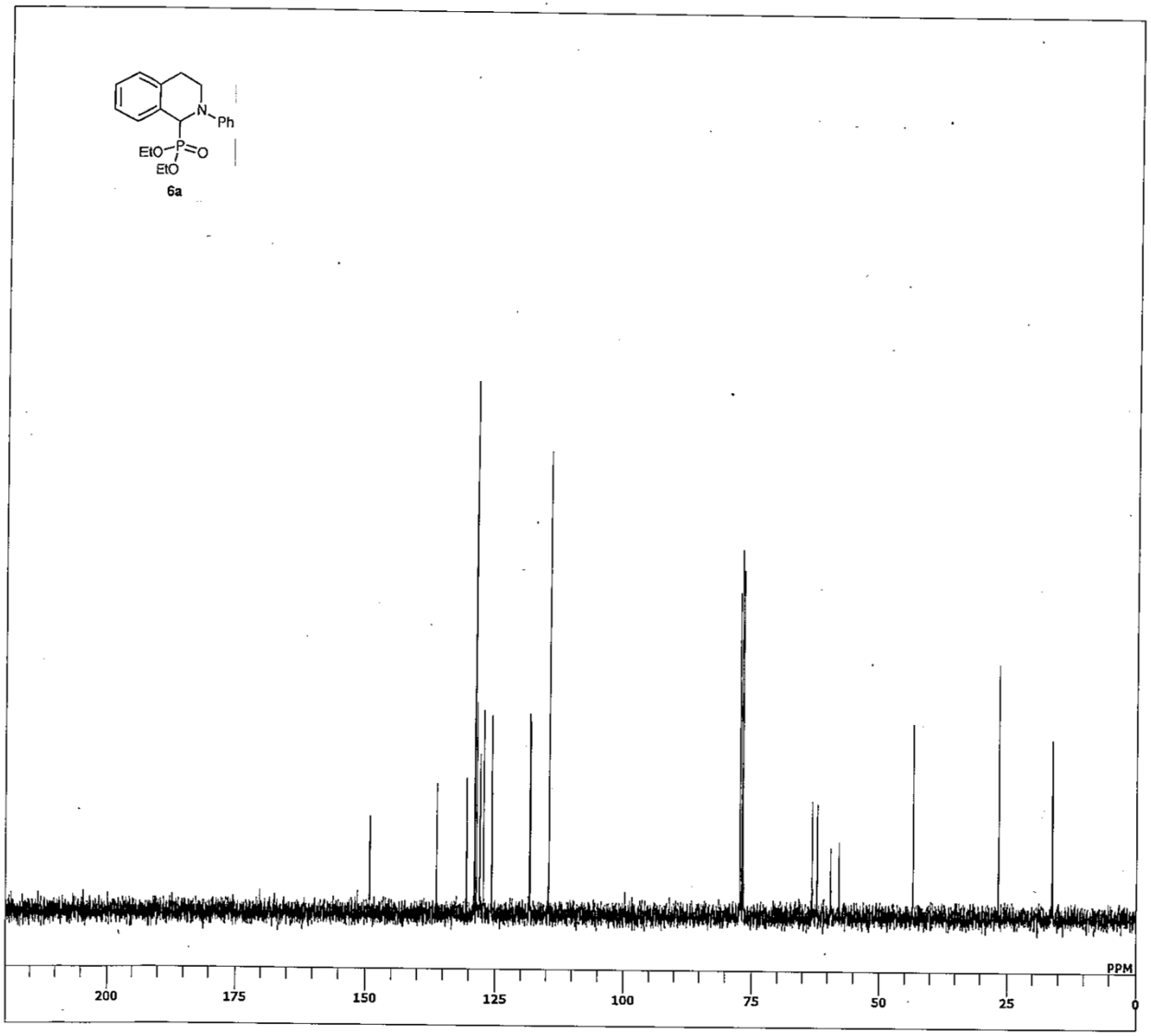

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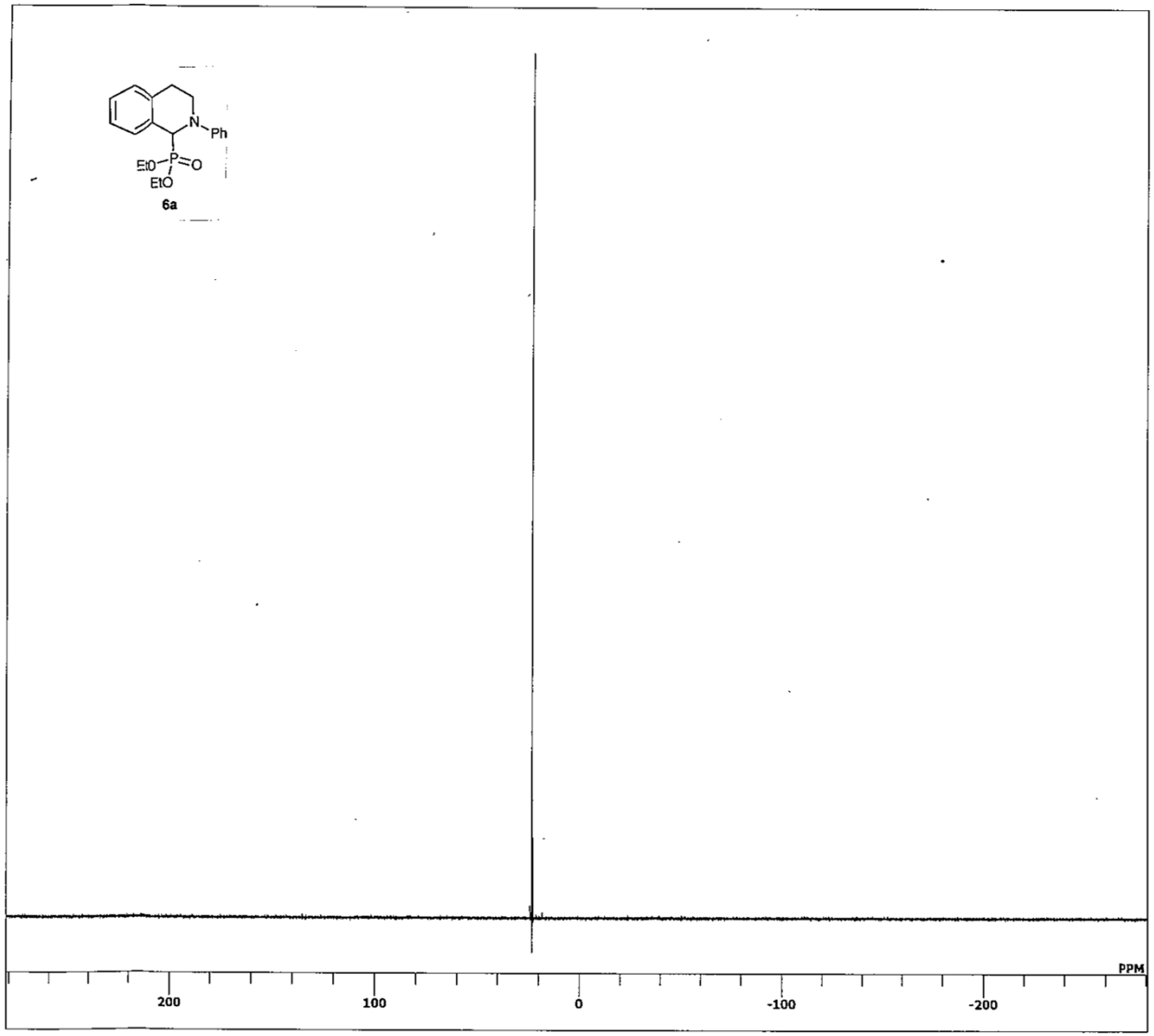

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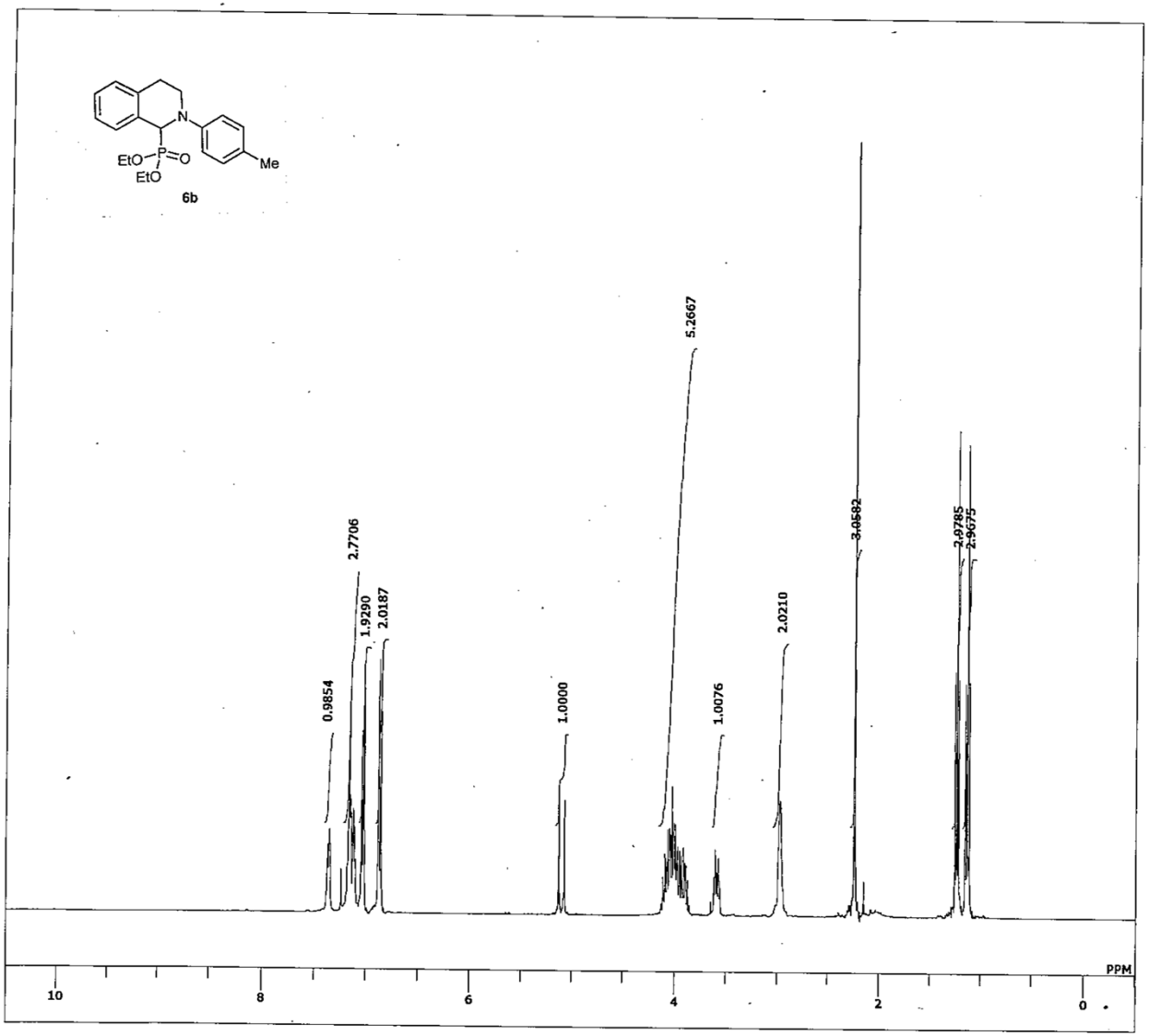

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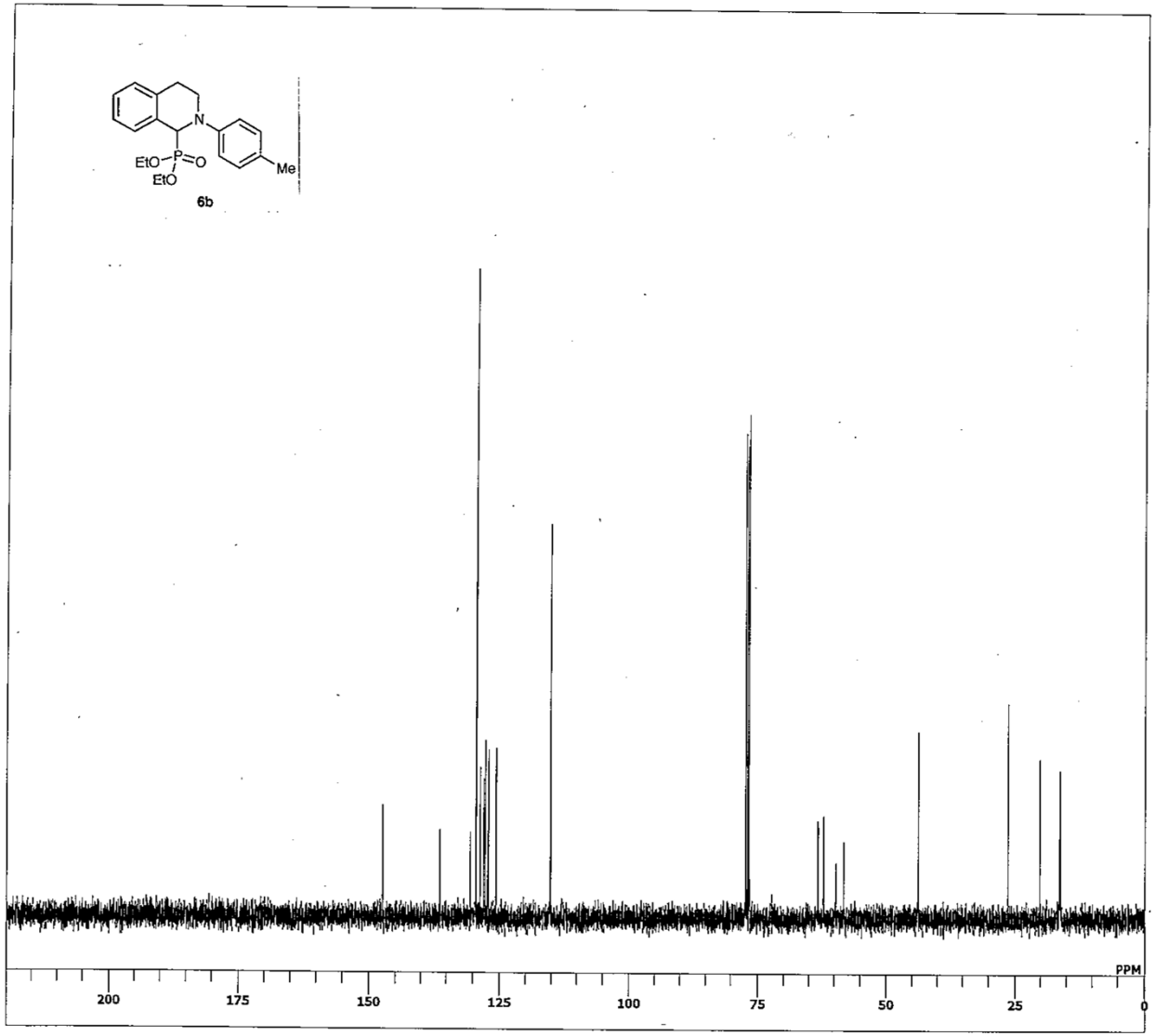

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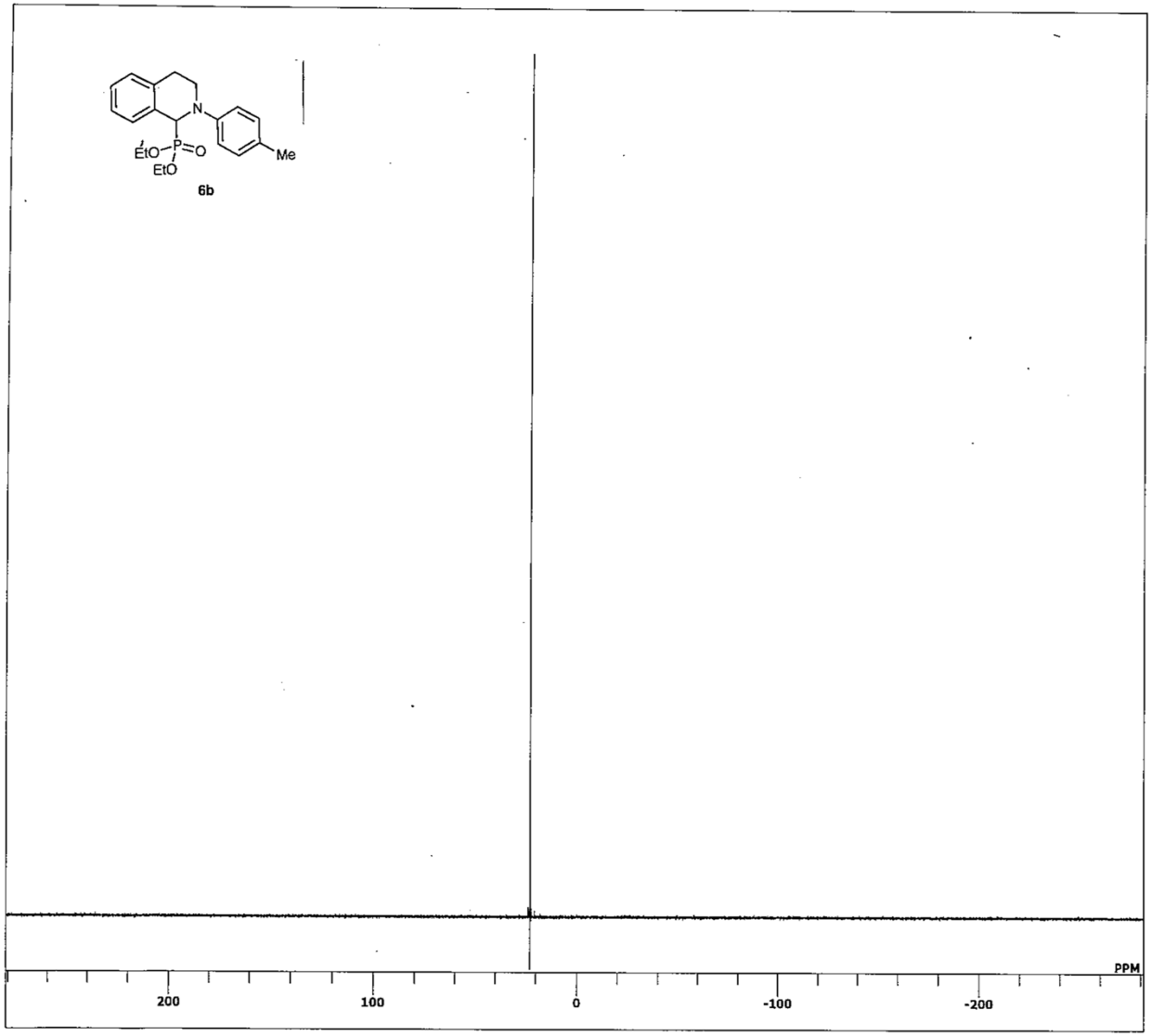

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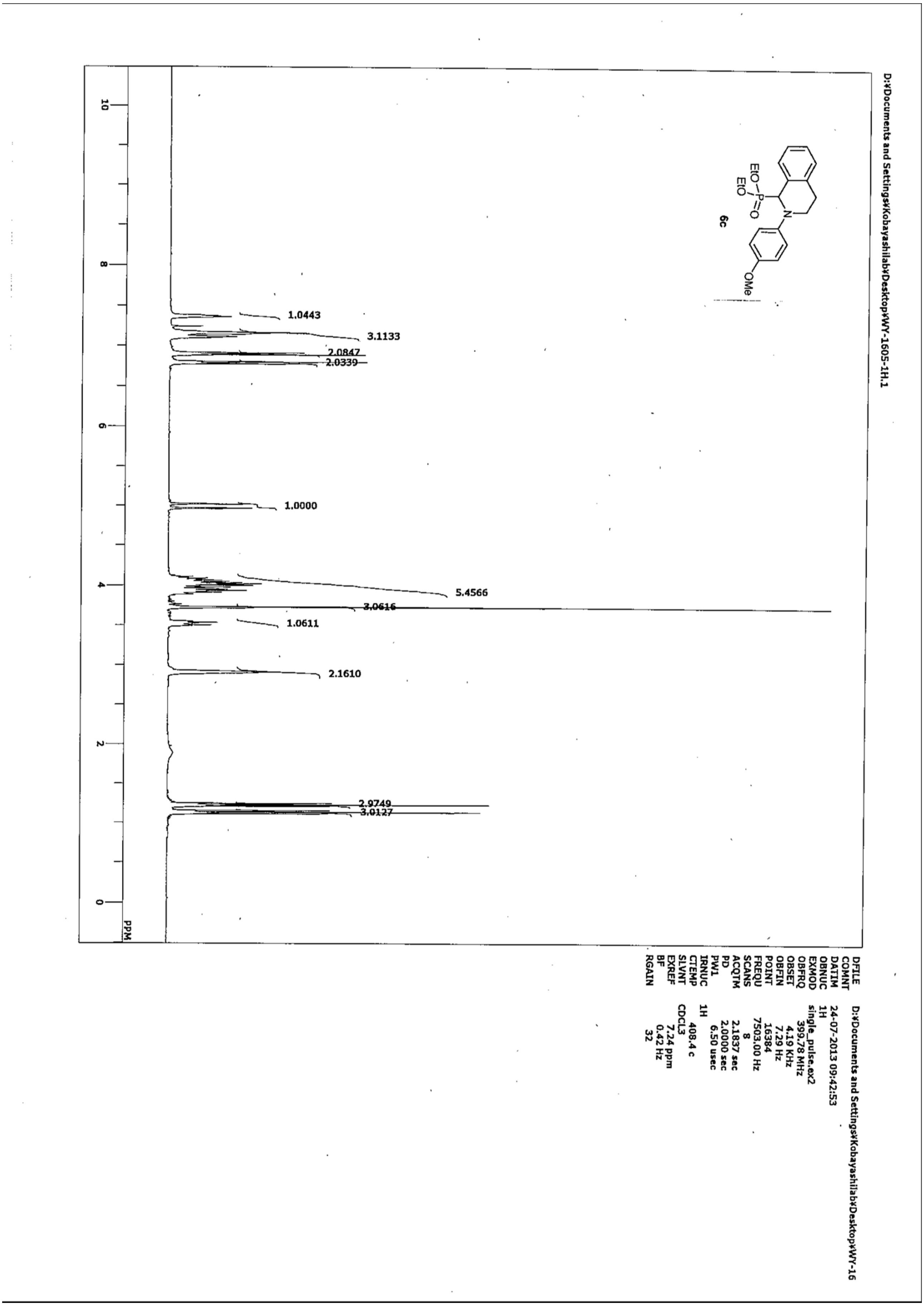
RGAIN	56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RGAIN 32

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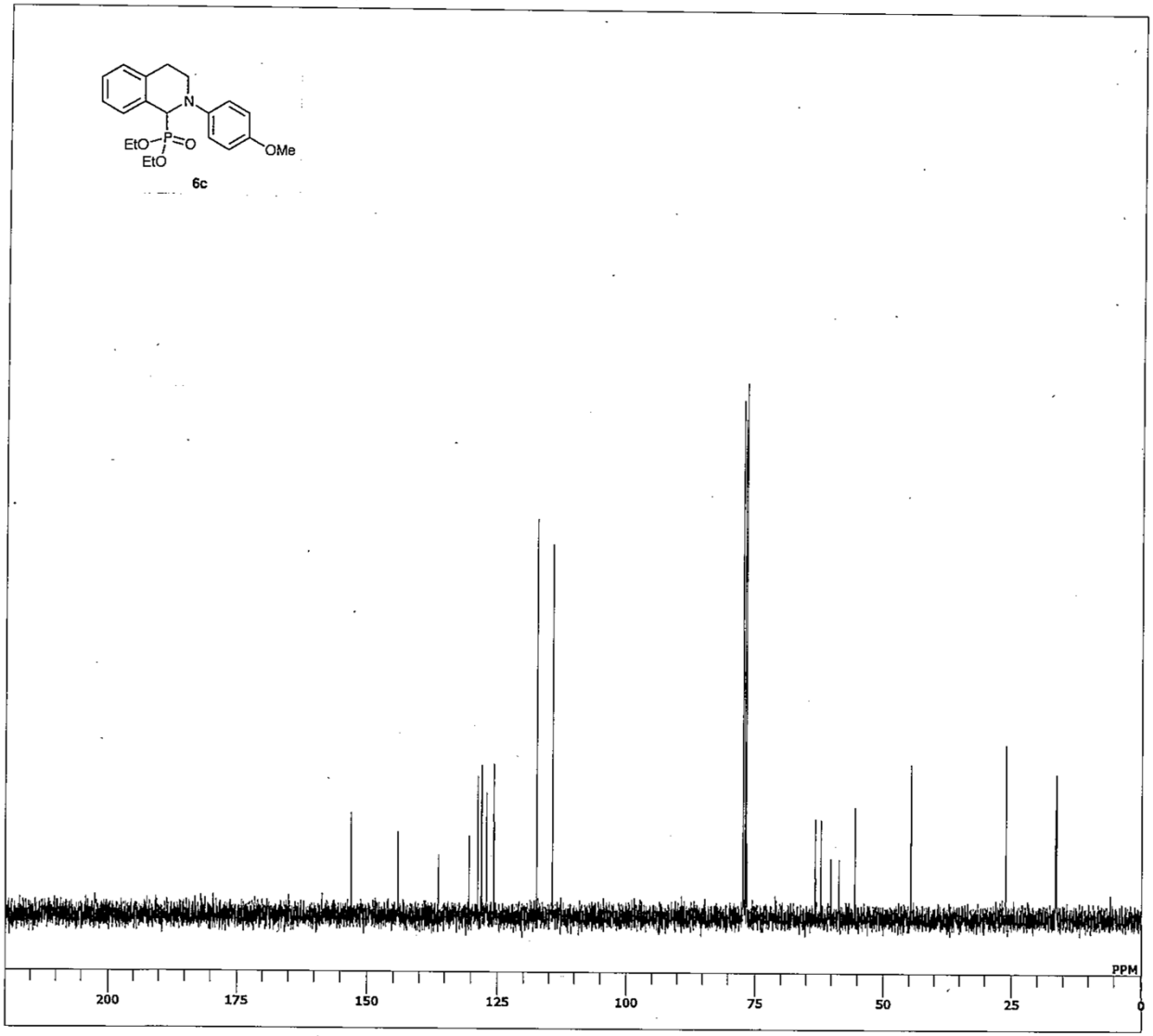
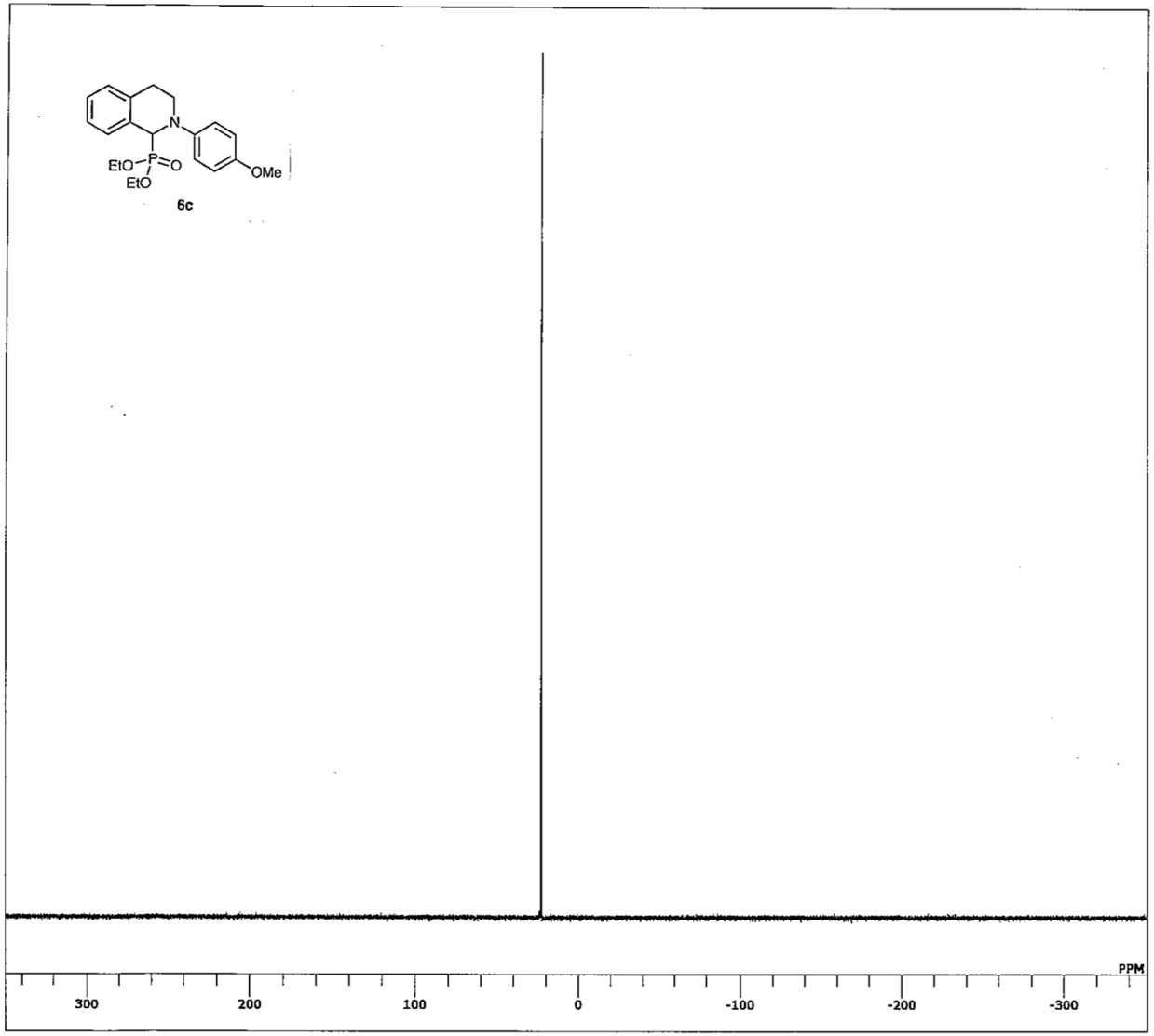
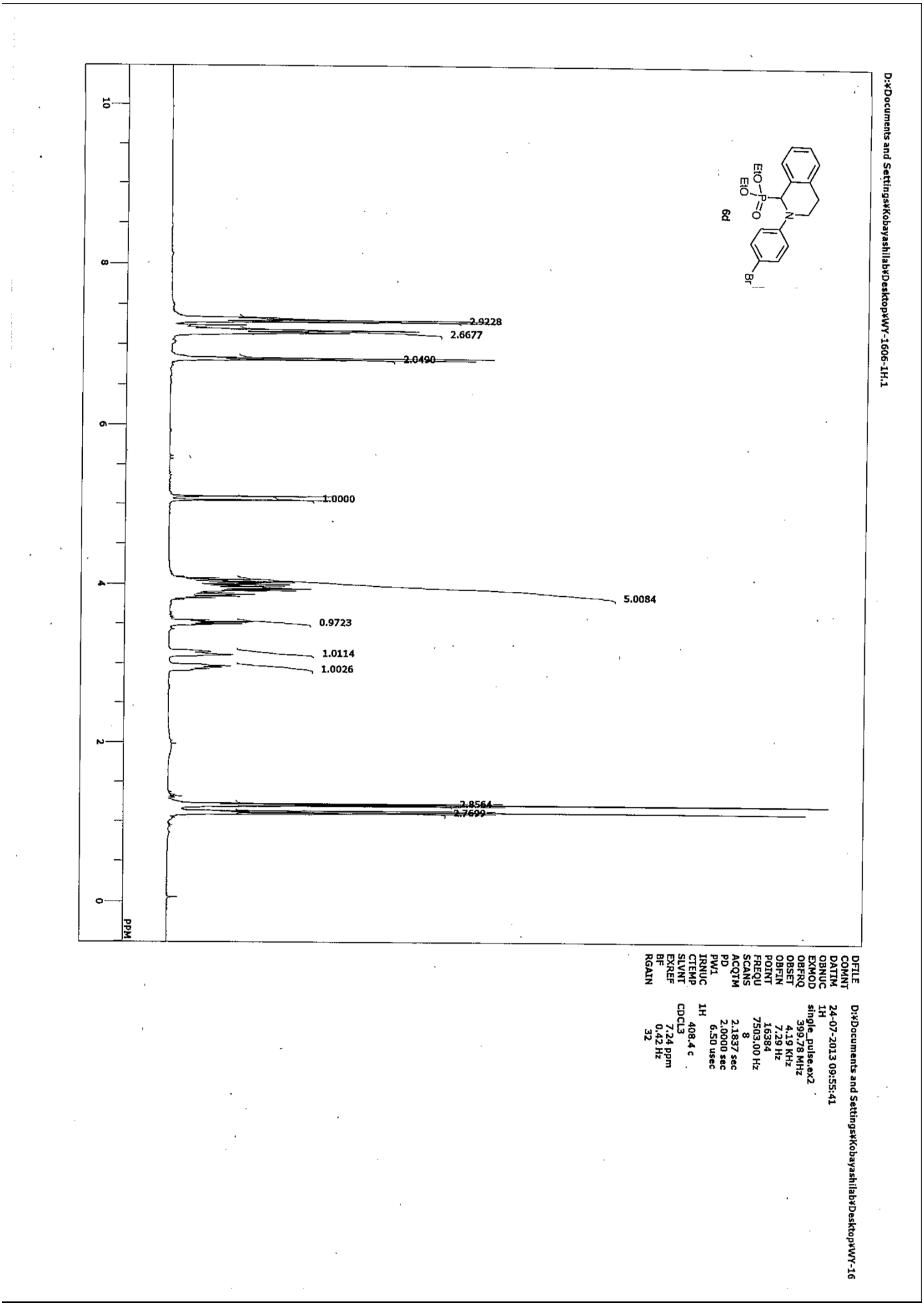


DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	24-07-2013 09:49:20
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.03 Hz
SCANS	76
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	408.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.42 Hz
RGAIN	60

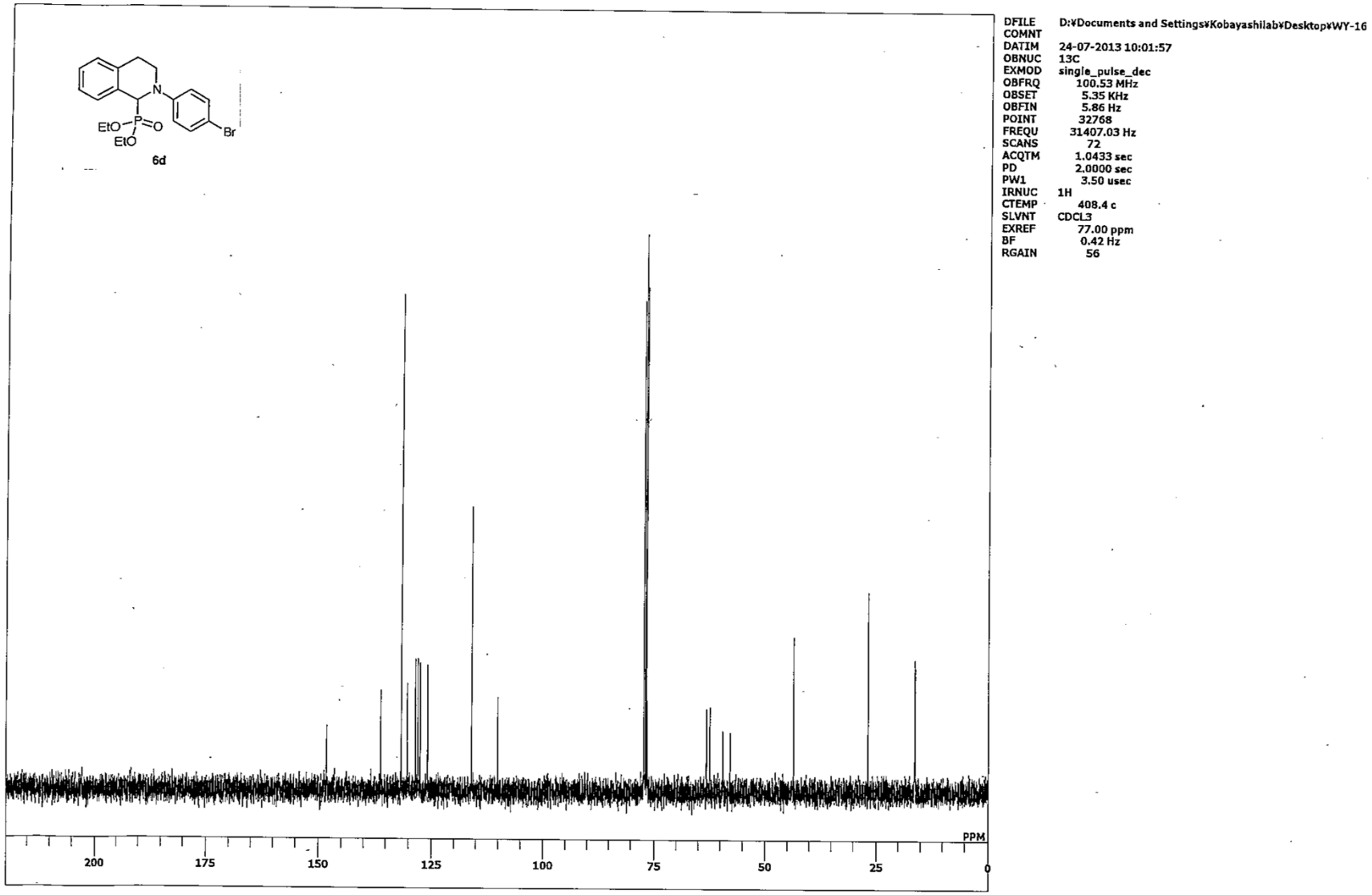
D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1605-31P.1



DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	24-07-2013 09:52:02
OBNUC	31P
EXMOD	single_pulse_dec
OBFRQ	161.83 MHz
OBSET	4.69 KHz
OBFIN	3.09 Hz
POINT	32768
FREQU	142045.45 Hz
SCANS	13
ACQTM	0.2307 sec
PD	2.0000 sec
PW1	5.80 usec
IRNUC	1H
CTEMP	408.4 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.42 Hz
RGAIN	56

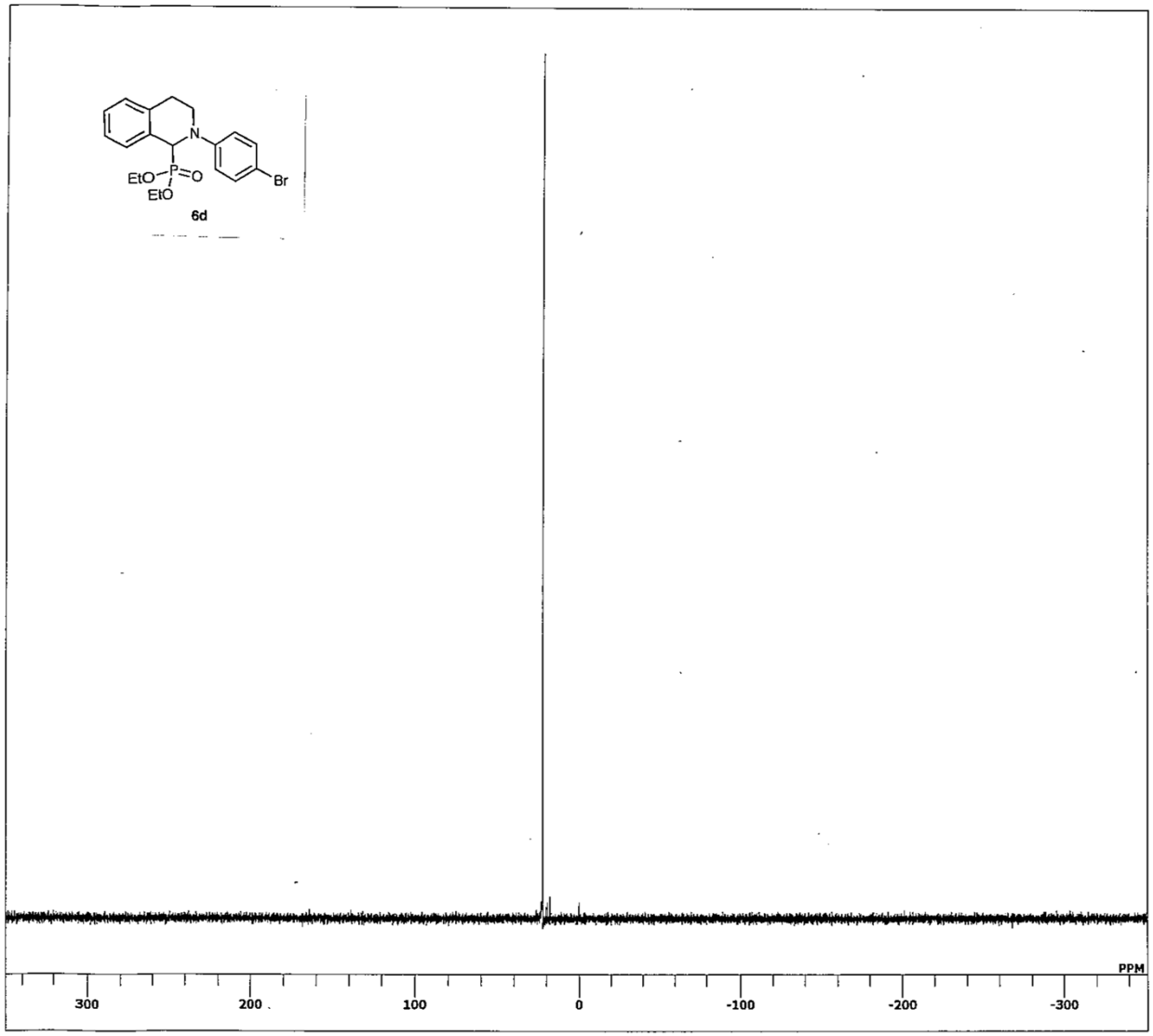

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1606-1H.1
EtO
EtO
P
O
N
Br
6d
2.9228
2.6677
2.0490
1.0000
5.0084
0.9723
1.0114
1.0026
2.8564
2.7699
PPM
DFILE D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT
DATIM 24-07-2013 09:55:41
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 8
ACQTM 2.1837 sec
PD 2.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 408.4 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.42 Hz
RGAIN 32

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1606-13C.1

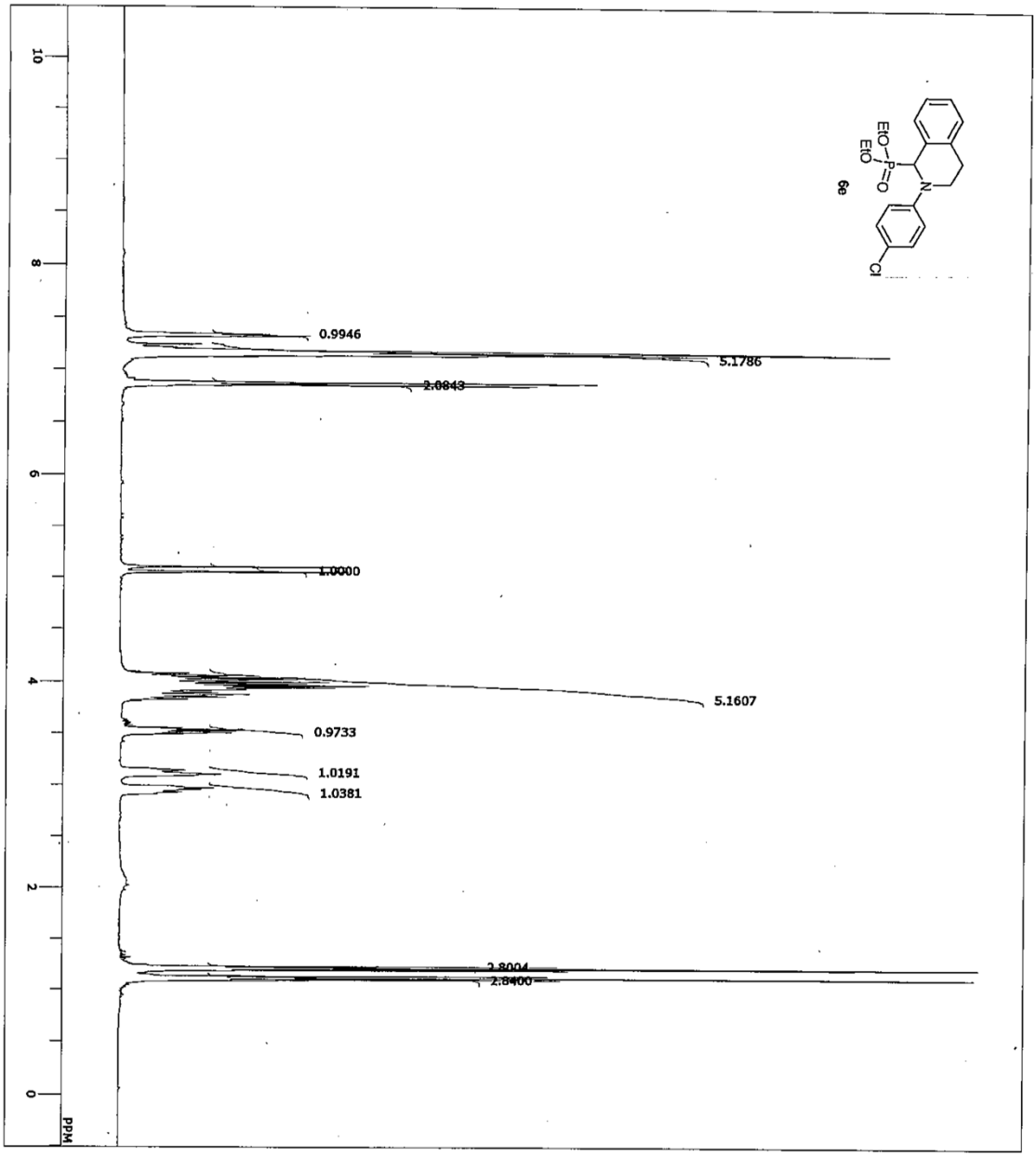


DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	24-07-2013 10:01:57
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.03 Hz
SCANS	72
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	408.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.42 Hz
RGAIN	56

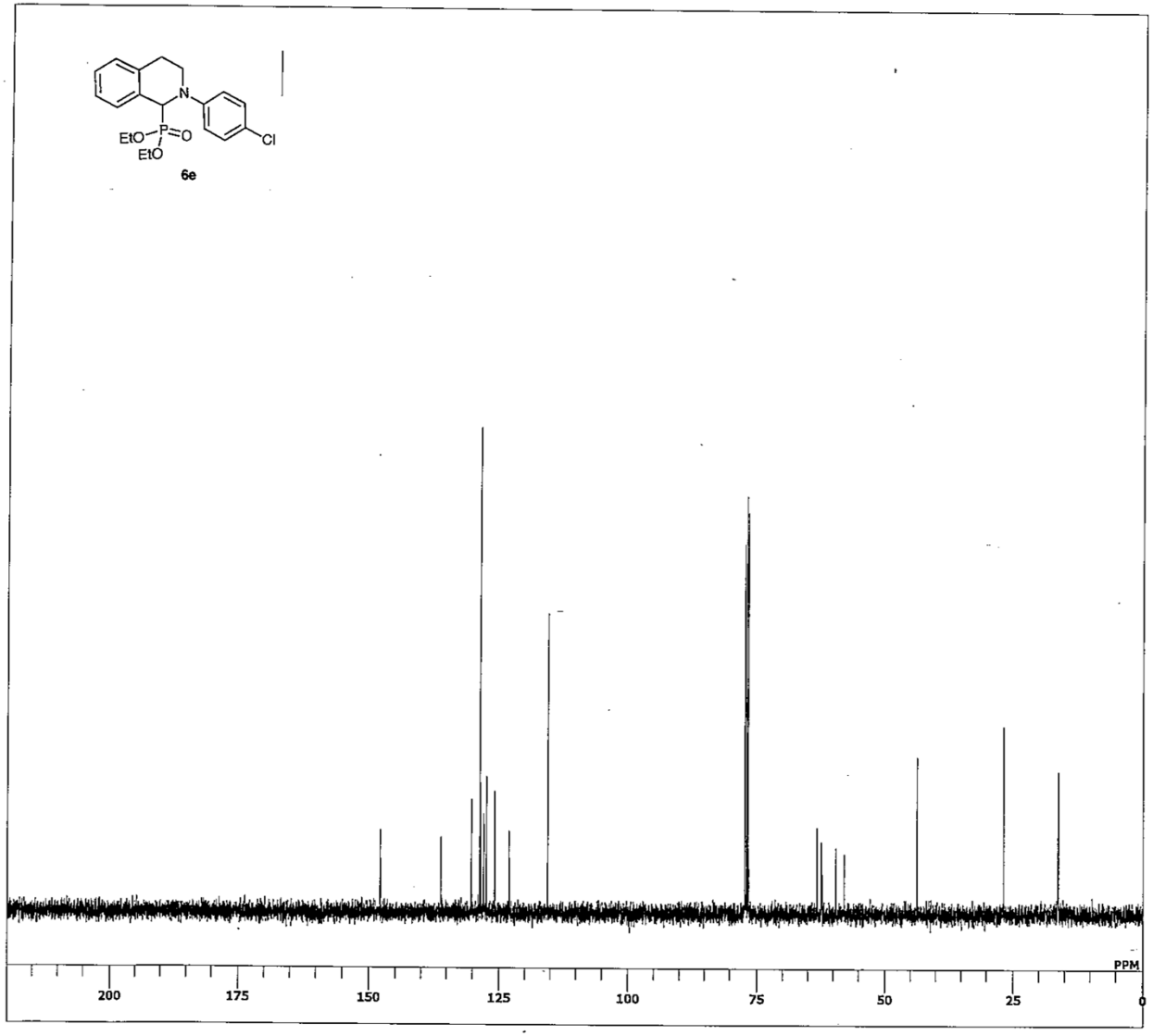
D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1606-31P.1



DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	24-07-2013 10:04:42
OBNUC	31P
EXMOD	single_pulse_dec
OBFRQ	161.83 MHz
OBSET	4.69 KHz
OBFIN	3.09 Hz
POINT	32768
FREQU	142045.45 Hz
SCANS	14
ACQTM	0.2307 sec
PD	2.0000 sec
PW1	5.80 usec
IRNUC	1H
CTEMP	408.4 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.42 Hz
RGAIN	56

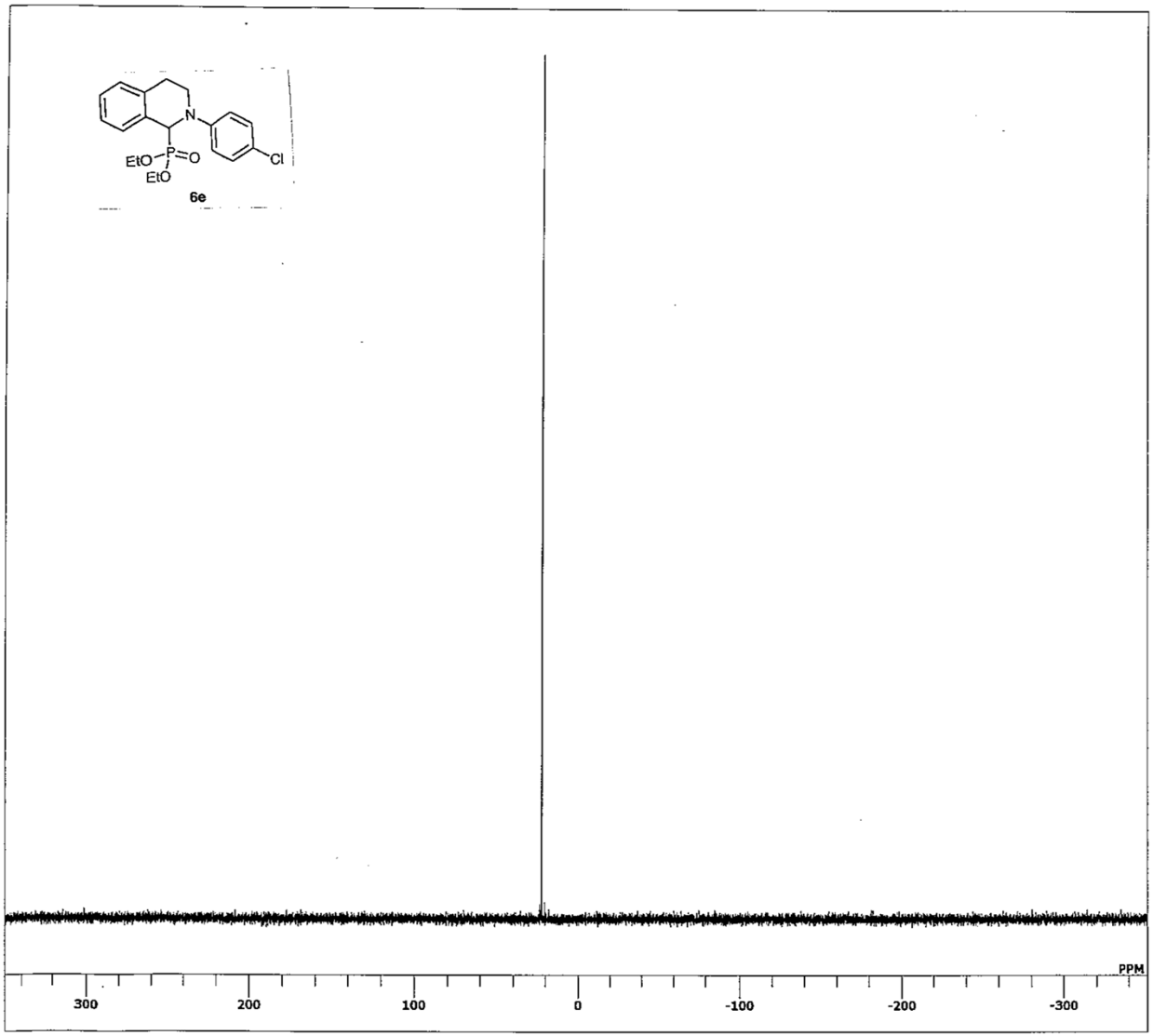

D:\Documents and Settings\Kobayashilab\Desktop\WY-1607-1H.1
EtO
EtO
P
O
N
Cl
6e
0.9946
5.1786
2.0843
1.0000
5.1607
0.9733
1.0191
1.0381
2.8004
2.8400
PPM
DFILE D:\Documents and Settings\Kobayashilab\Desktop\WY-16
COMNT
DATIM 24-07-2013 10:08:07
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 8
ACQTM 2.1837 sec
PD 2.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 408.4 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.42 Hz
RGAIN 32

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1607-13C.1



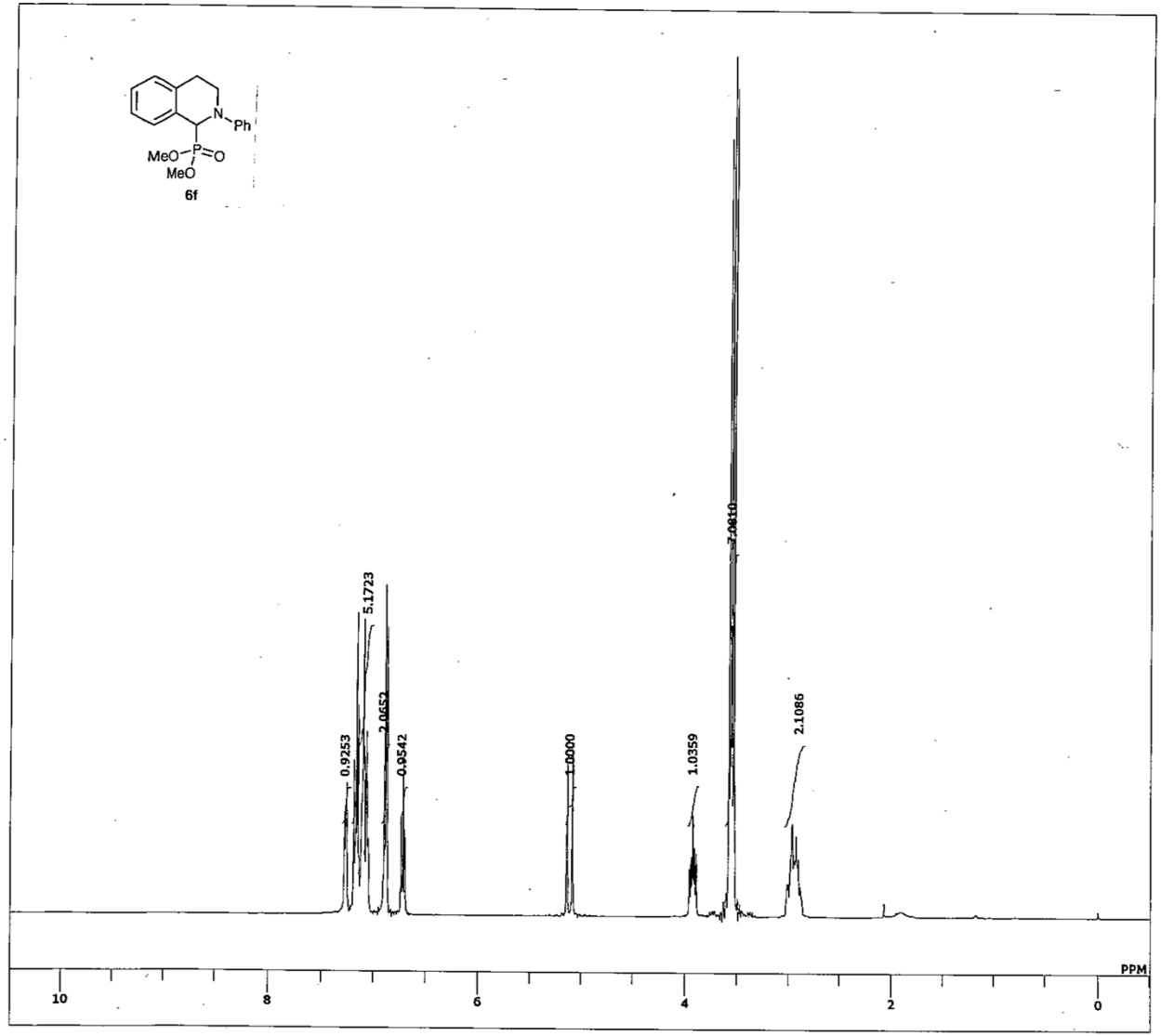
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	24-07-2013 10:15:03
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.03 Hz
SCANS	82
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	408.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.42 Hz
RGAIN	54

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1607-31P.1



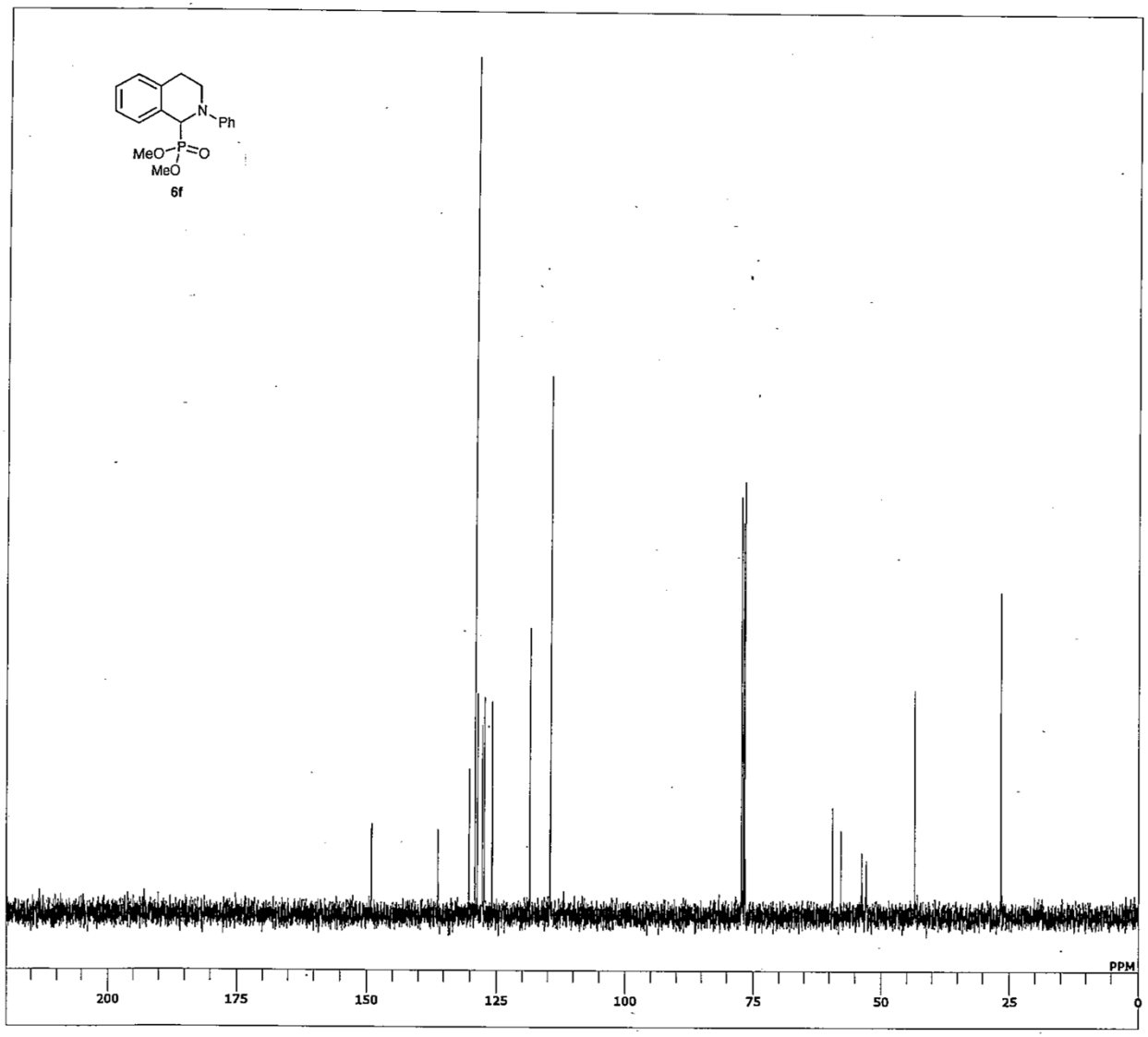
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	24-07-2013 10:17:25
OBNUC	31P
EXMOD	single_pulse_dec
OBFRQ	161.83 MHz
OBSET	4.69 KHz
OBFIN	3.09 Hz
POINT	32768
FREQU	142045.45 Hz
SCANS	3
ACQTM	0.2307 sec
PD	2.0000 sec
PW1	5.80 usec
IRNUC	1H
CTEMP	408.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.42 Hz
RGAIN	56

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1602-1H.1



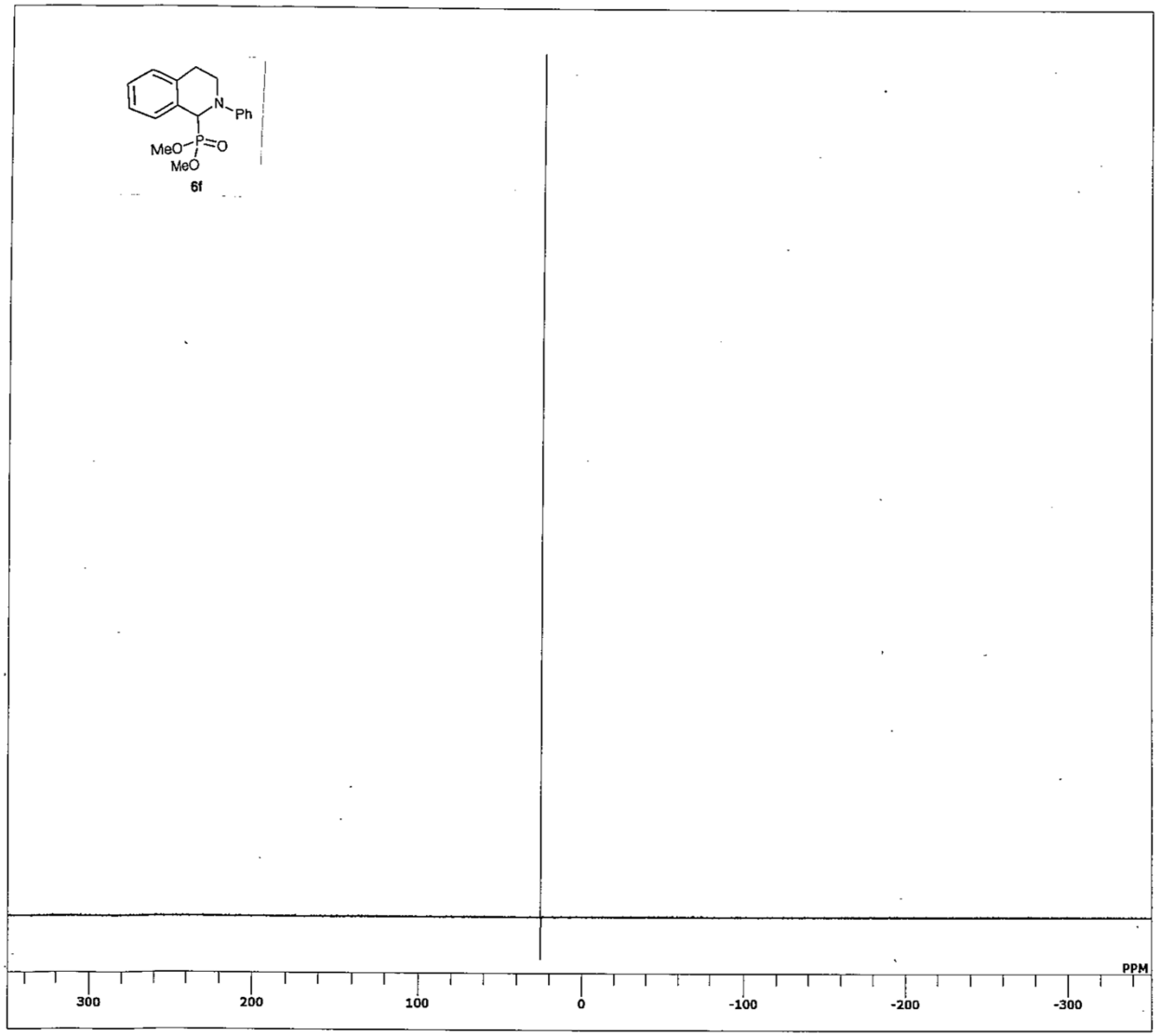
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	23-07-2013 14:26:29
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	6.50 usec
IRNUC	1H
CTEMP	408.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.42 Hz
RGAIN	28

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1602-13C.1



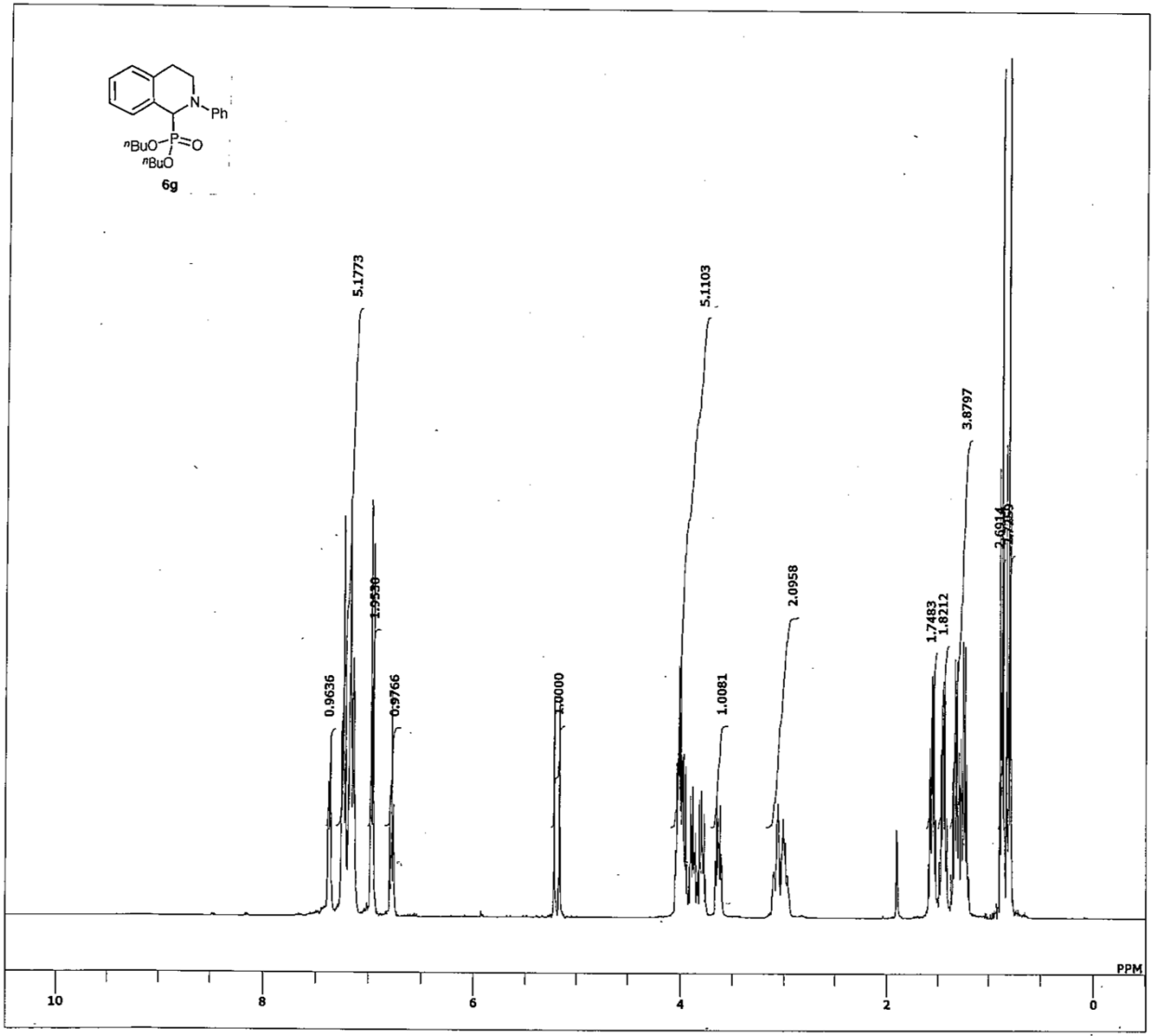
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	23-07-2013 14:30:59
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.03 Hz
SCANS	42
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	408.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.42 Hz
RGAIN	54

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1602-31P.1



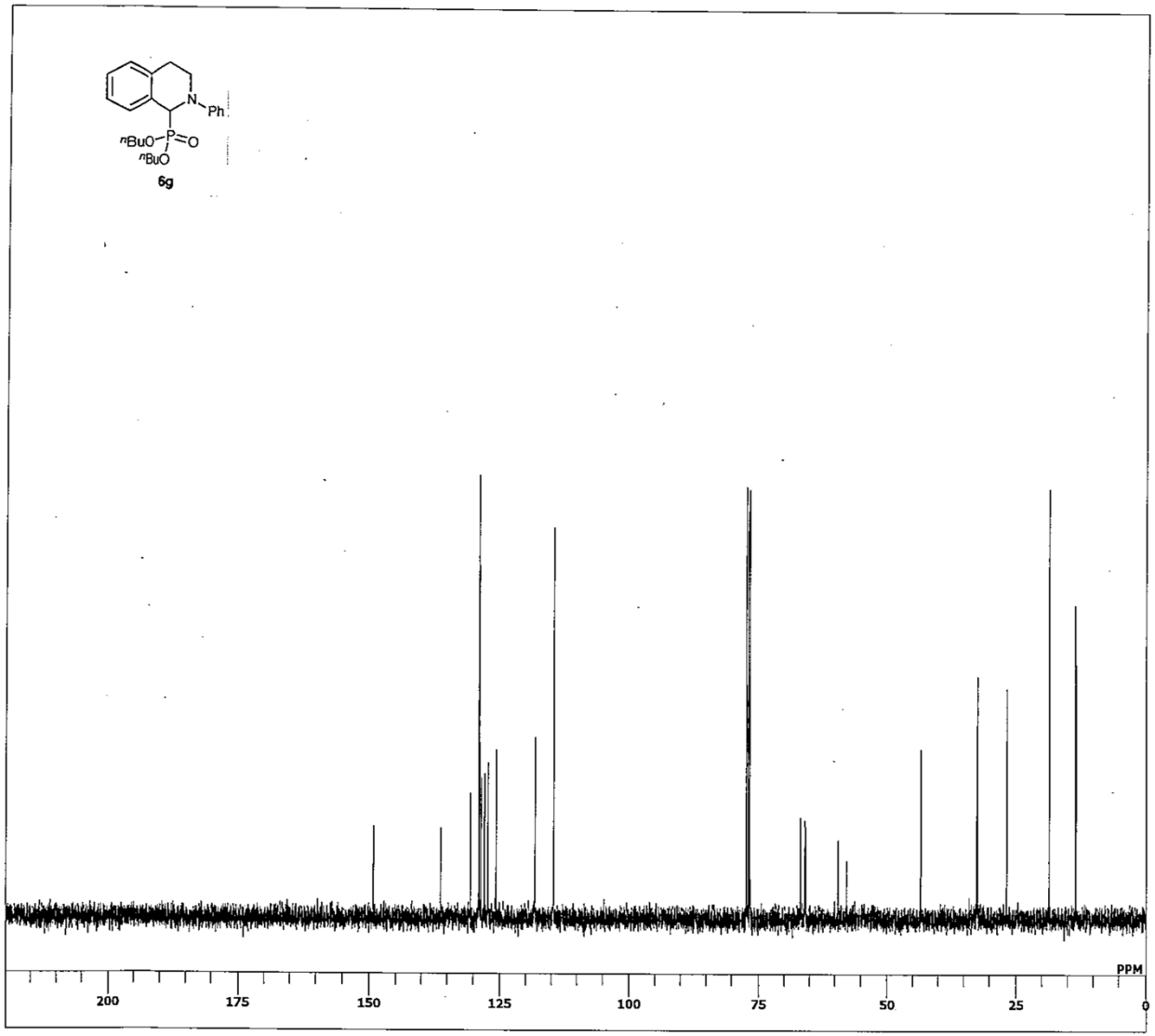
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	23-07-2013 14:33:40
OBNUC	31P
EXMOD	single_pulse_dec
OBFRQ	161.83 MHz
OBSET	4.69 KHz
OBFIN	3.09 Hz
POINT	32768
FREQU	142045.45 Hz
SCANS	9
ACQTM	0.2307 sec
PD	2.0000 sec
PW1	5.80 usec
IRNUC	1H
CTEMP	408.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.42 Hz
RGAIN	54

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1609-1H.1



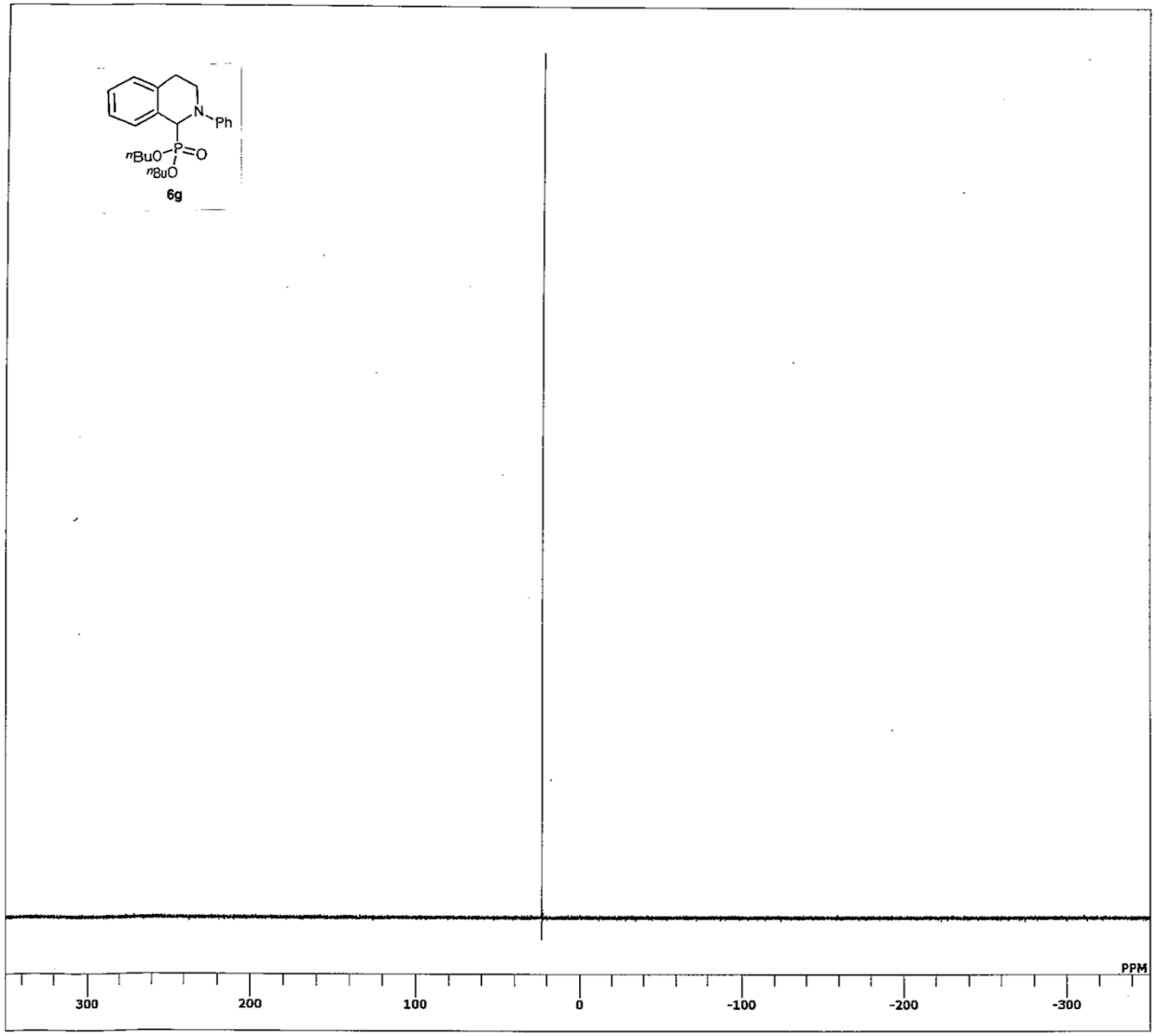
DFILE D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT
DATIM 25-07-2013 16:34:31
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 8
ACQTM 2.1837 sec
PD 2.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 408.7 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.42 Hz
RGAIN 28

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1609-13C.1



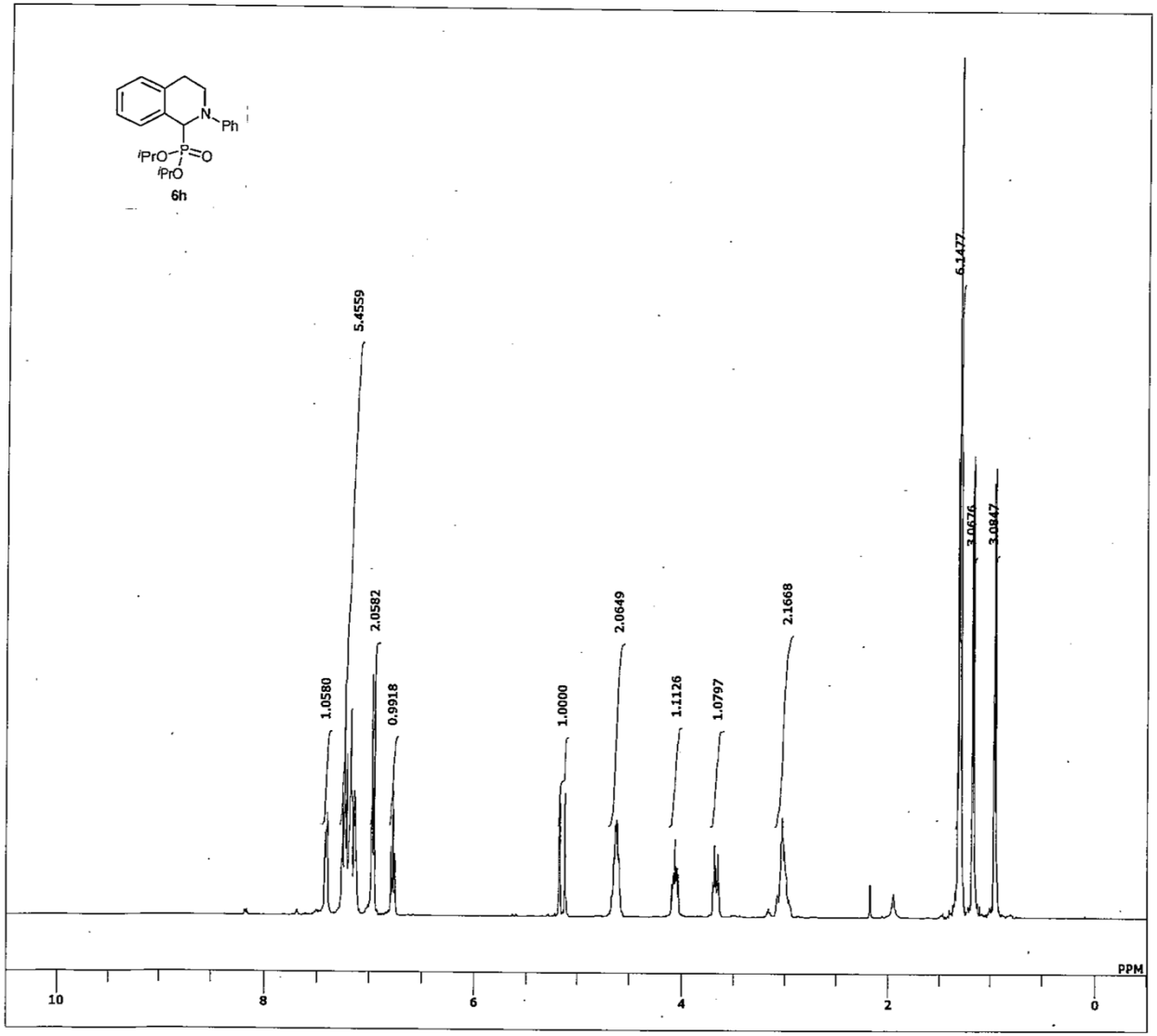
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	25-07-2013 16:40:34
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.03 Hz
SCANS	56
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	408.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.42 Hz
RGAIN	60

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1609-31P.1



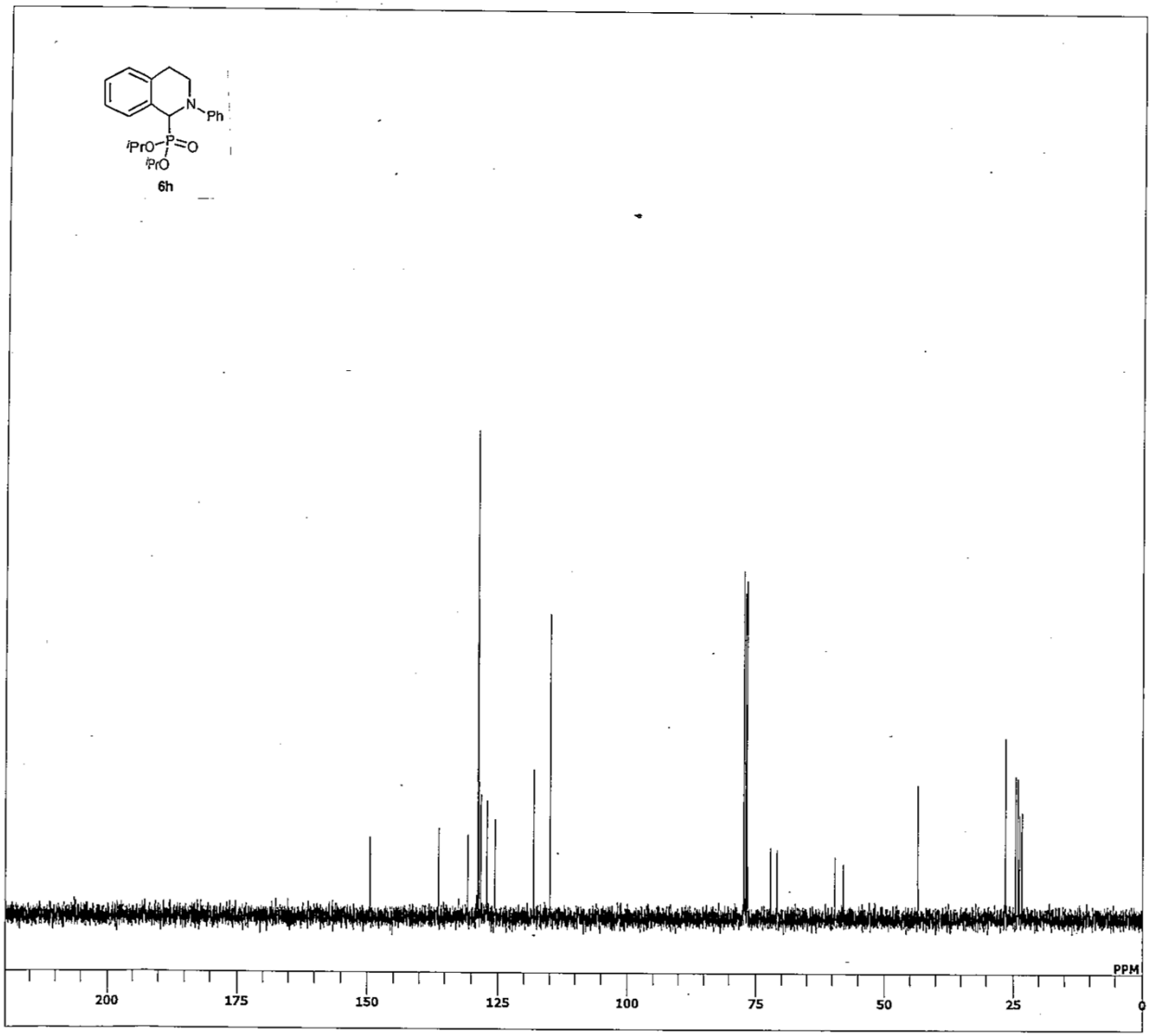
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	25-07-2013 16:43:04
OBNUC	31P
EXMOD	single_pulse_dec
OBFRQ	161.83 MHz
OBSET	4.69 KHz
OBFIN	3.09 Hz
POINT	32768
FREQU	142045.45 Hz
SCANS	5
ACQTM	0.2307 sec
PD	2.0000 sec
PW1	5.80 usec
IRNUC	1H
CTEMP	408.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.42 Hz
RGAIN	54

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1603-1H.1



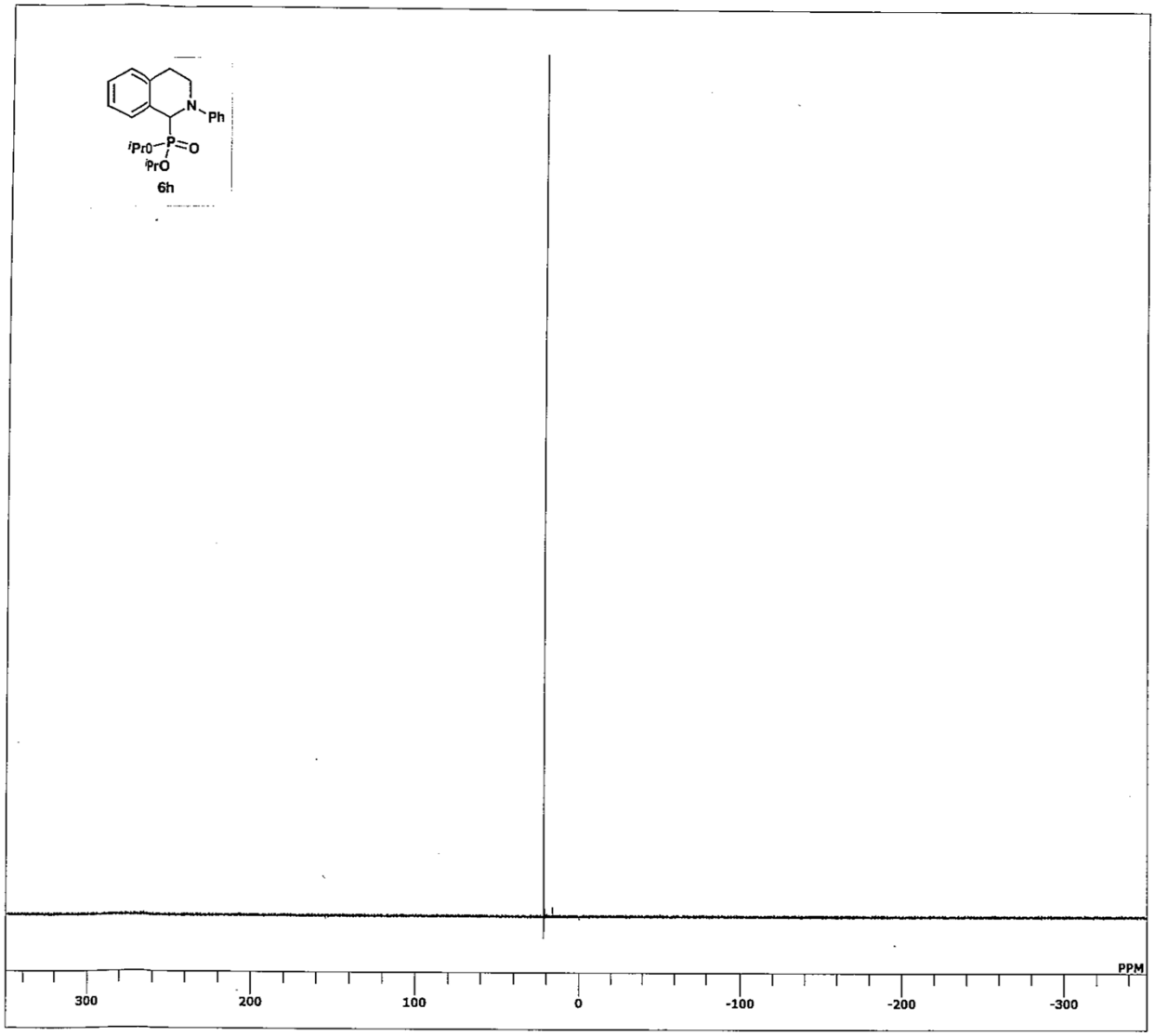
DFILE D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT
DATIM 23-07-2013 14:37:04
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 8
ACQTM 2.1837 sec
PD 2.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 408.6 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.42 Hz
RGAIN 28

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1603-13C.1



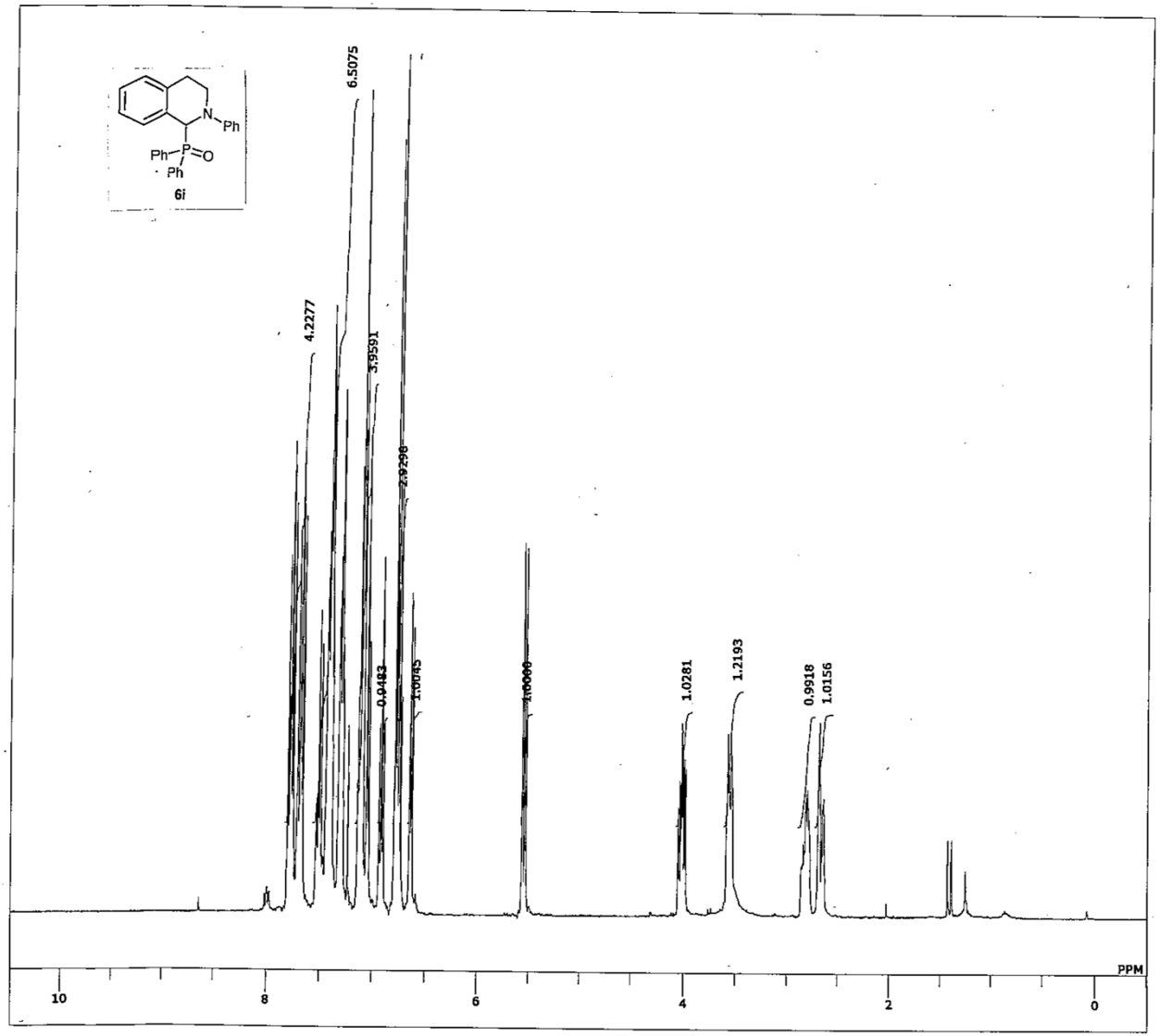
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	23-07-2013 14:42:33
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.03 Hz
SCANS	45
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	408.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.42 Hz
RGAIN	60

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1603-31P,1



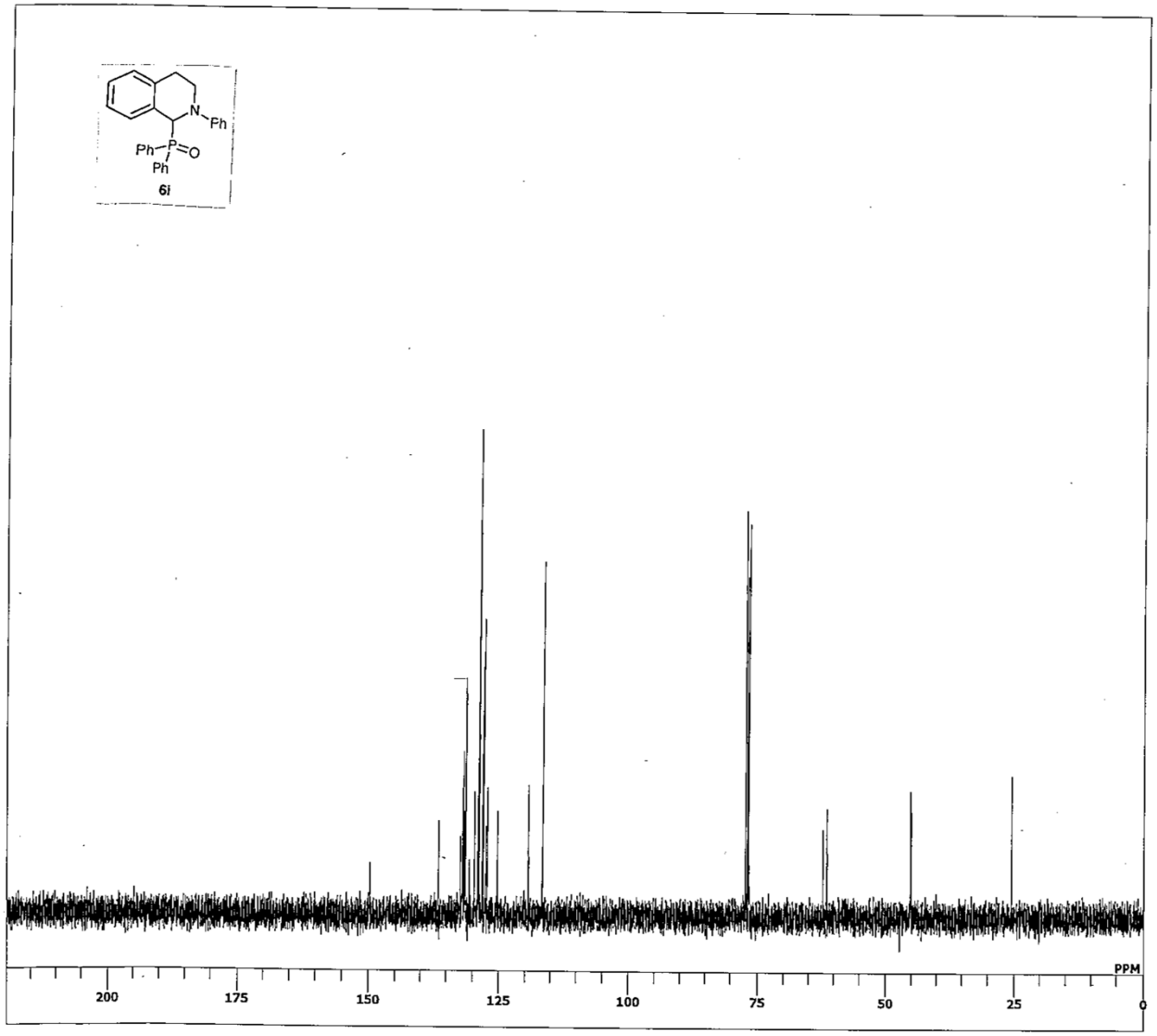
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	23-07-2013 14:45:39
OBNUC	31P
EXMOD	single_pulse_dec
OBFRQ	161.83 MHz
OBSET	4.69 KHz
OBFIN	3.09 Hz
POINT	32768
FREQU	142045.45 Hz
SCANS	11
ACQTM	0.2307 sec
PD	2.0000 sec
PW1	5.80 usec
IRNUC	1H
CTEMP	408.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.42 Hz
RGAIN	54

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1612-1H.1



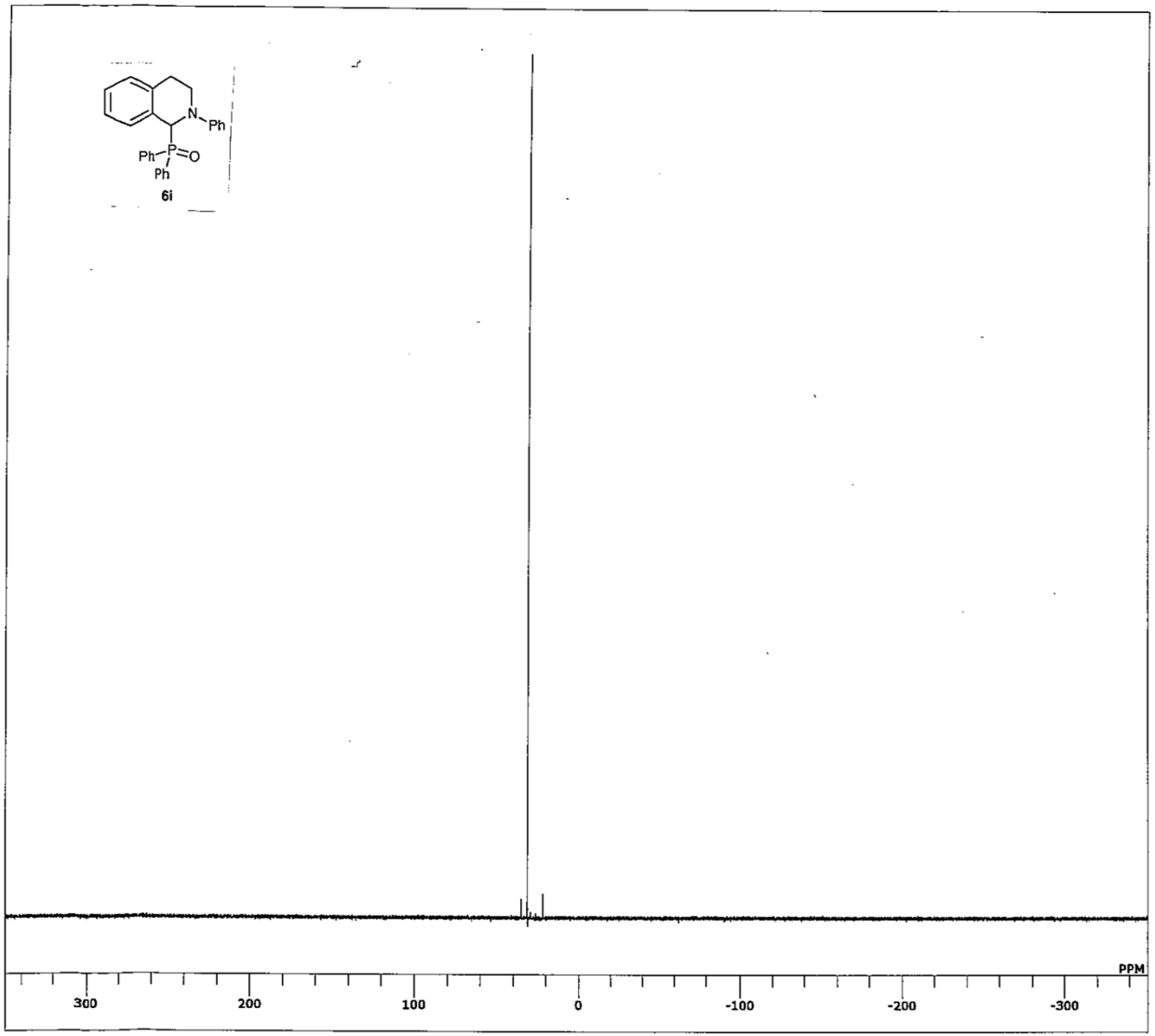
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	27-07-2013 16:41:07
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	6.50 usec
IRNUC	1H
CTEMP	408.2 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	28

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1612-13C.1



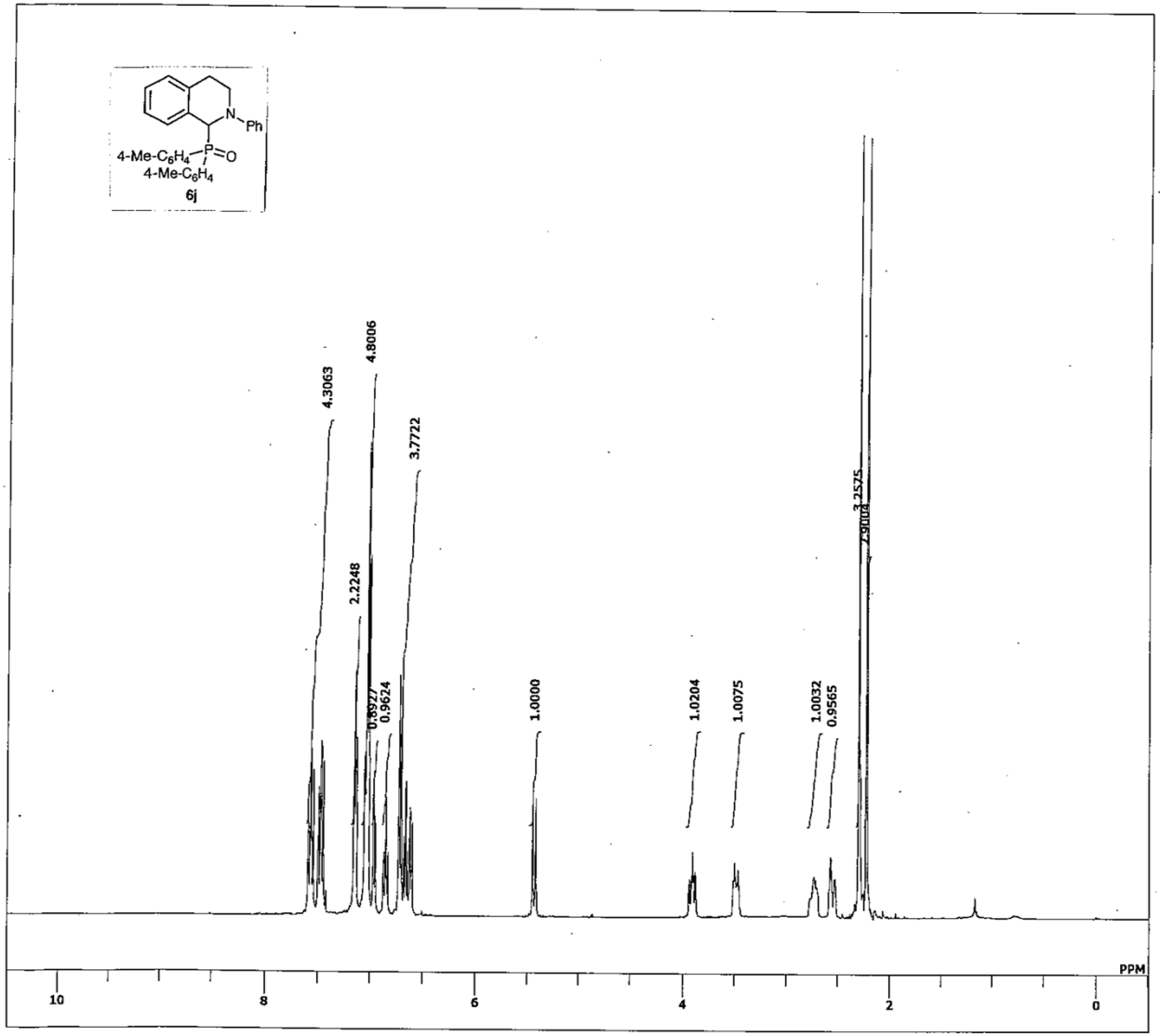
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	27-07-2013 16:45:13
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.03 Hz
SCANS	33
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	408.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1612-31P.1



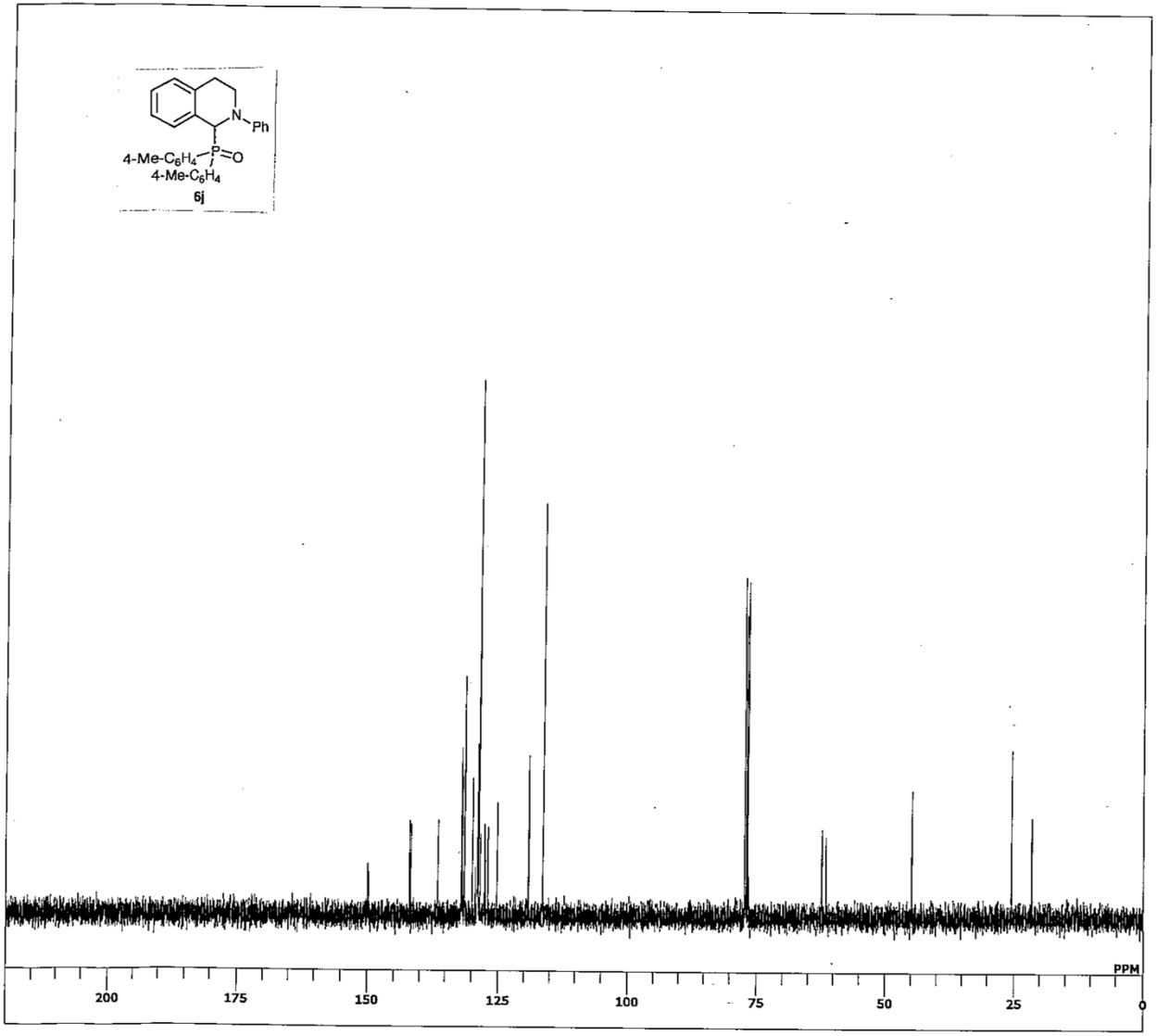
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	27-07-2013 16:48:18
OBNUC	31P
EXMOD	single_pulse_dec
OBFRQ	161.83 MHz
OBSET	4.69 KHz
OBFIN	3.09 Hz
POINT	32768
FREQU	142045.45 Hz
SCANS	11
ACQTM	0.2307 sec
PD	2.0000 sec
PW1	5.80 usec
IRNUC	1H
CTEMP	408.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.42 Hz
RGAIN	56

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1613-1H.1



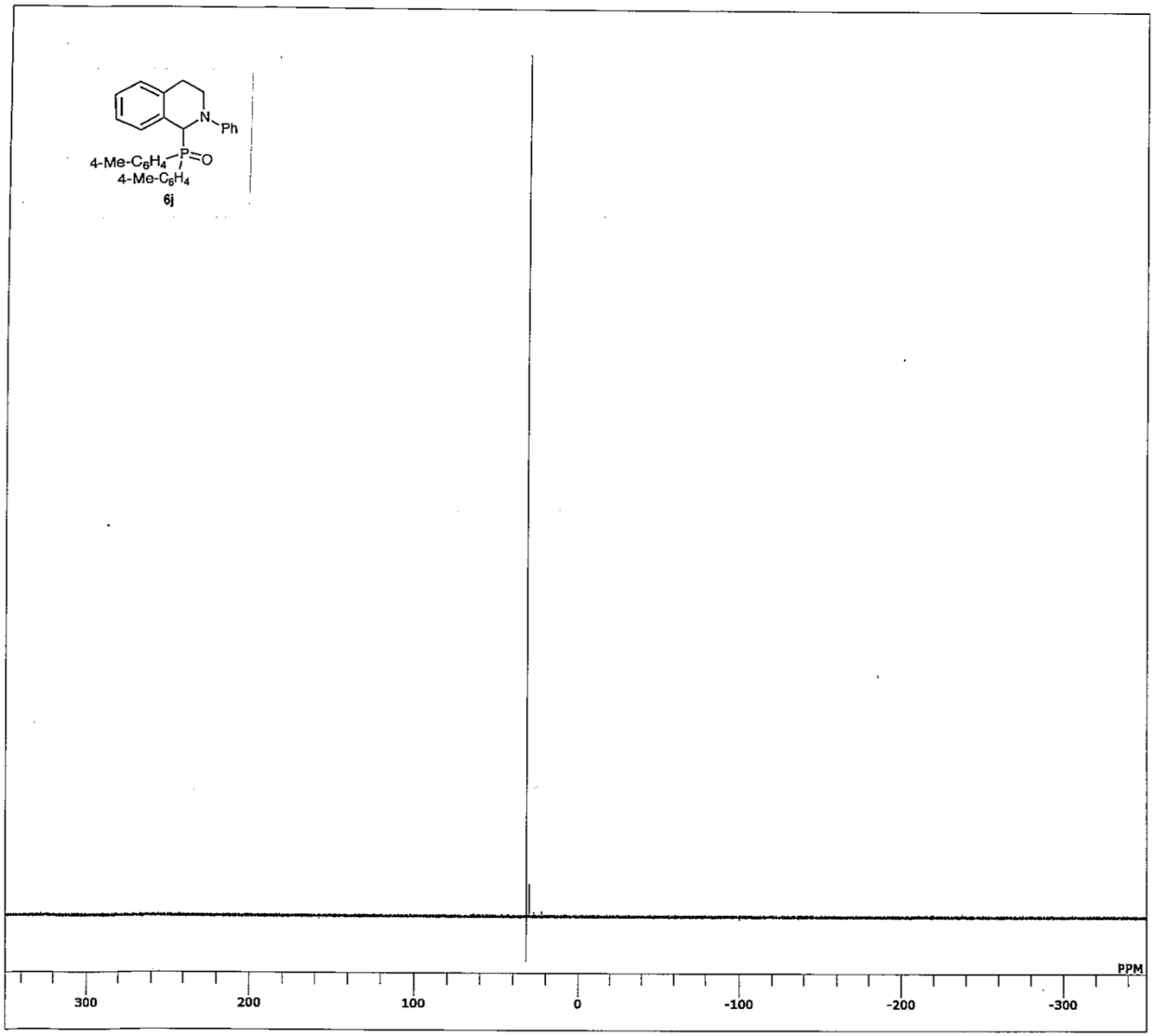
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	27-07-2013 16:52:41
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	6.50 usec
IRNUC	1H
CTEMP	408.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	28

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1613-13C.1

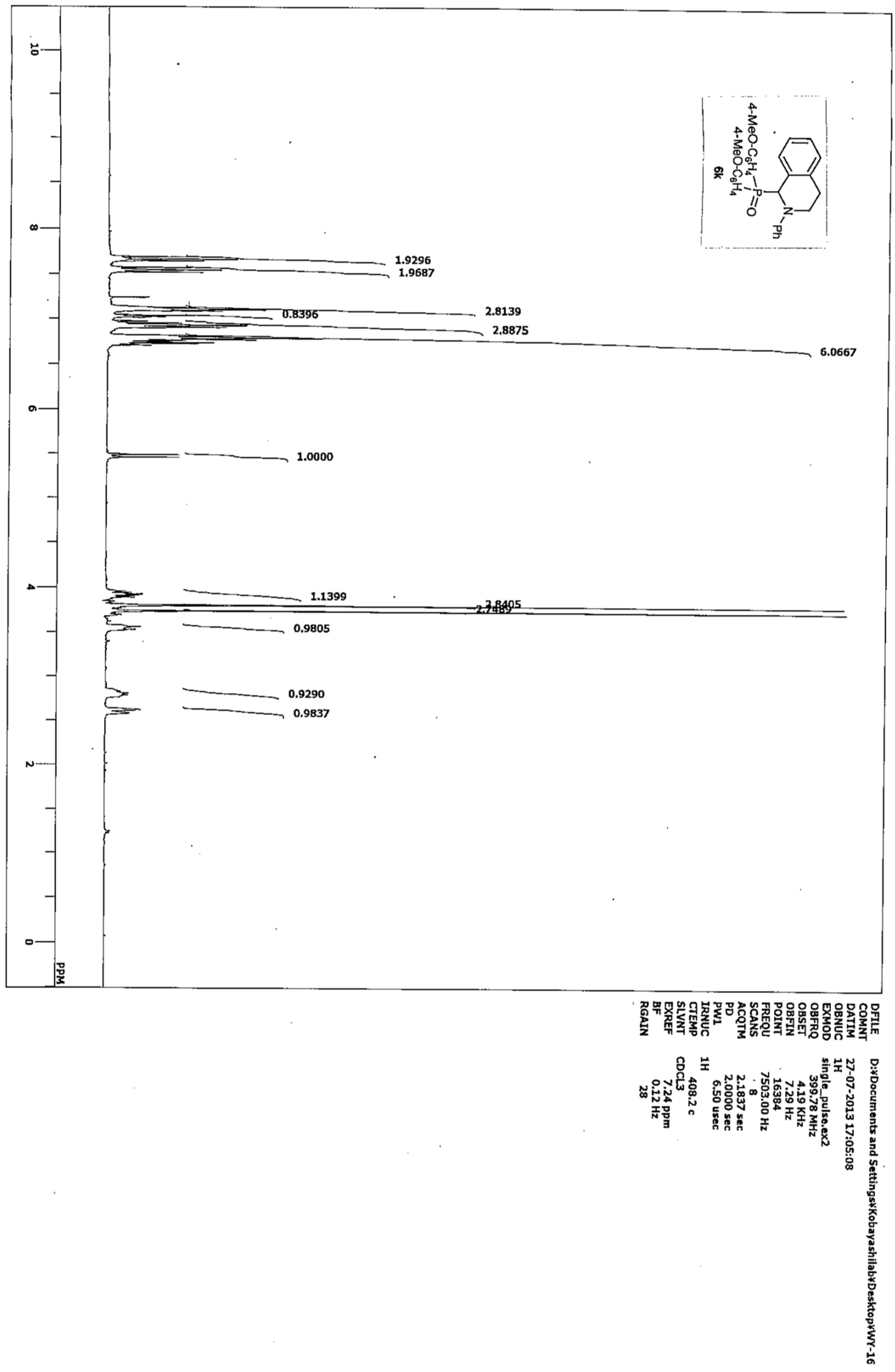


DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	27-07-2013 16:57:35
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.03 Hz
SCANS	38
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	408.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	58

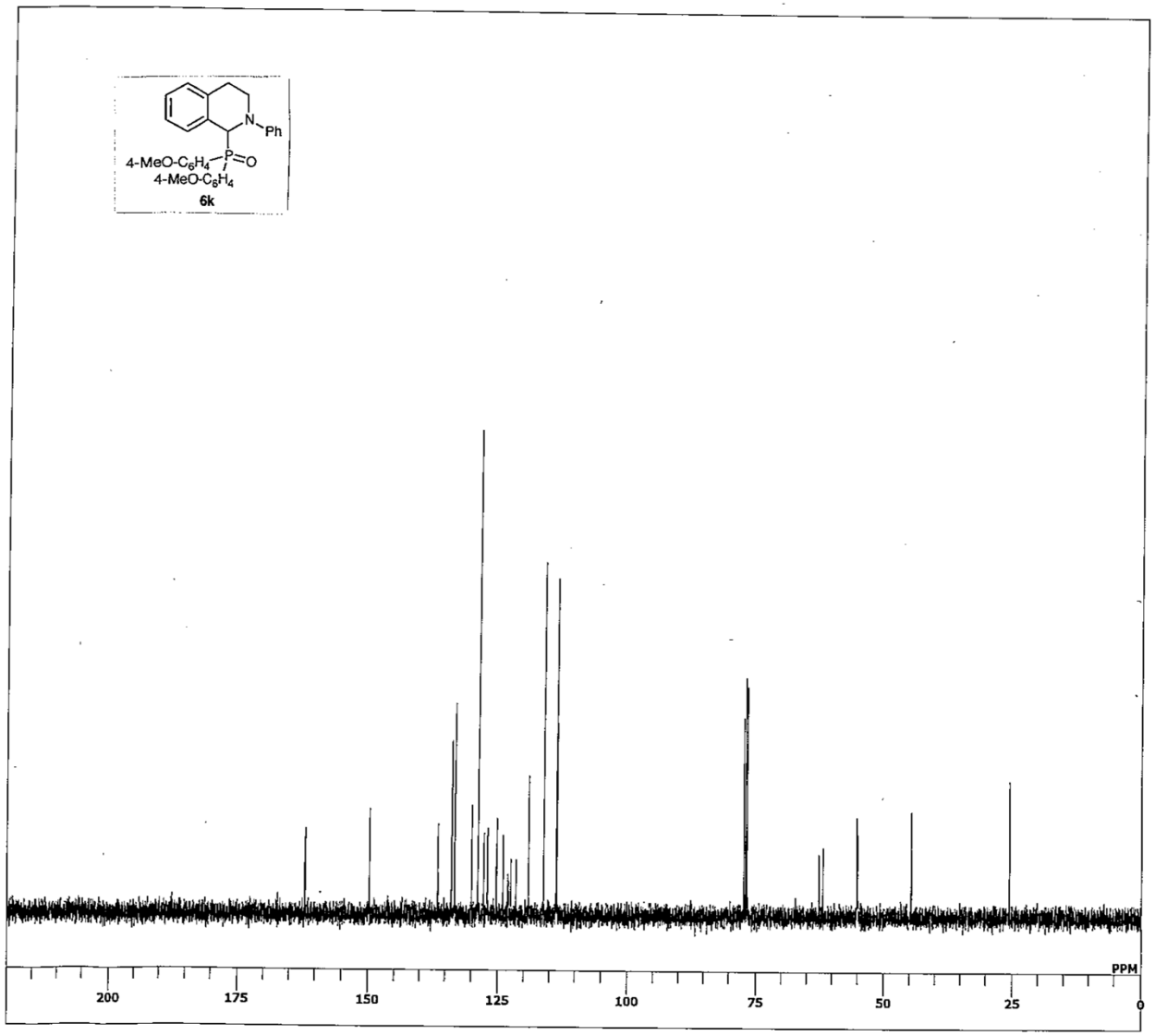
D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1613-31P.1



DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	27-07-2013 17:00:55
OBNUC	31P
EXMOD	single_pulse_dec
OBFRQ	161.83 MHz
OBSET	4.69 KHz
OBFIN	3.09 Hz
POINT	32768
FREQU	142045.45 Hz
SCANS	6
ACQTM	0.2307 sec
PD	2.0000 sec
PW1	5.80 usec
IRNUC	1H
CTEMP	408.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.42 Hz
RGAIN	54

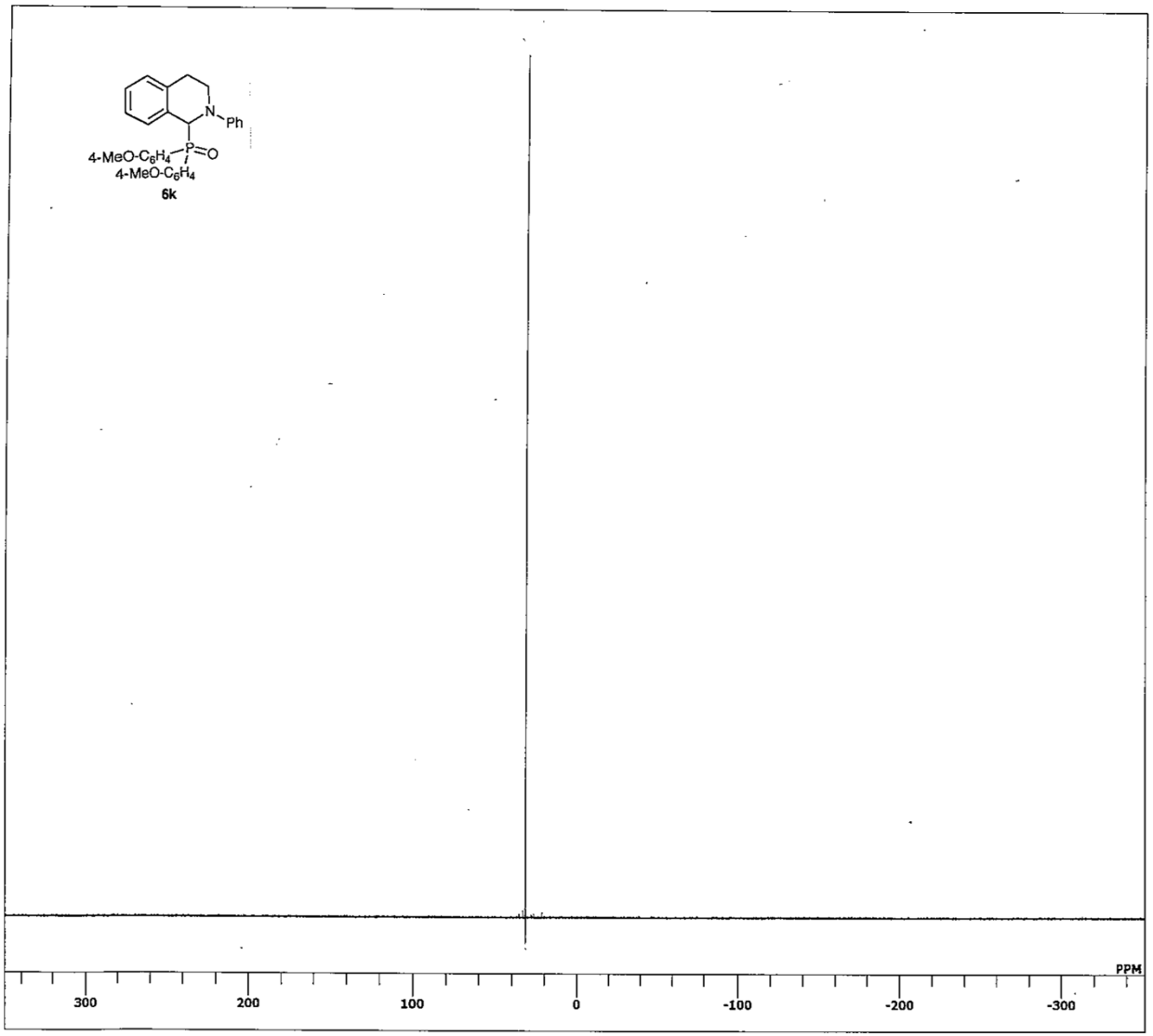

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1614-1H.1
N–Ph
P=O
4-MeO-C6H4
4-MeO-C6H4
6k
1.9296
1.9687
0.8396
2.8139
2.8875
6.0667
1.0000
1.1399
2.8405
2.7489
0.9805
0.9290
0.9837
10
8
6
4
2
0
PPM
DFILE D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT
DATIM 27-07-2013 17:05:08
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 8
ACQTM 2.1837 sec
PD 2.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 408.2 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 28

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1614-13C.1



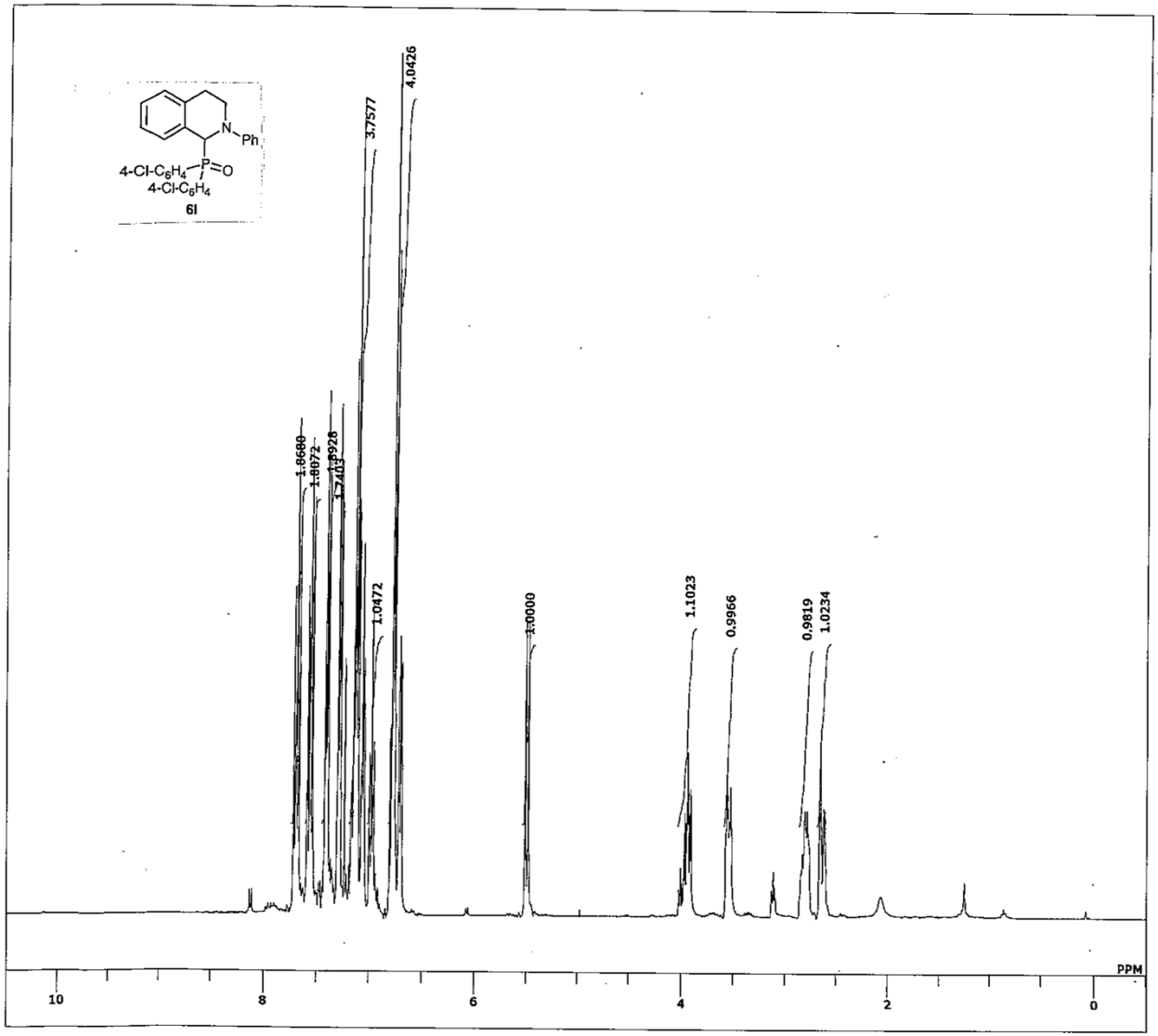
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	27-07-2013 17:09:58
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.03 Hz
SCANS	36
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	408.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	56

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1614-31P.1



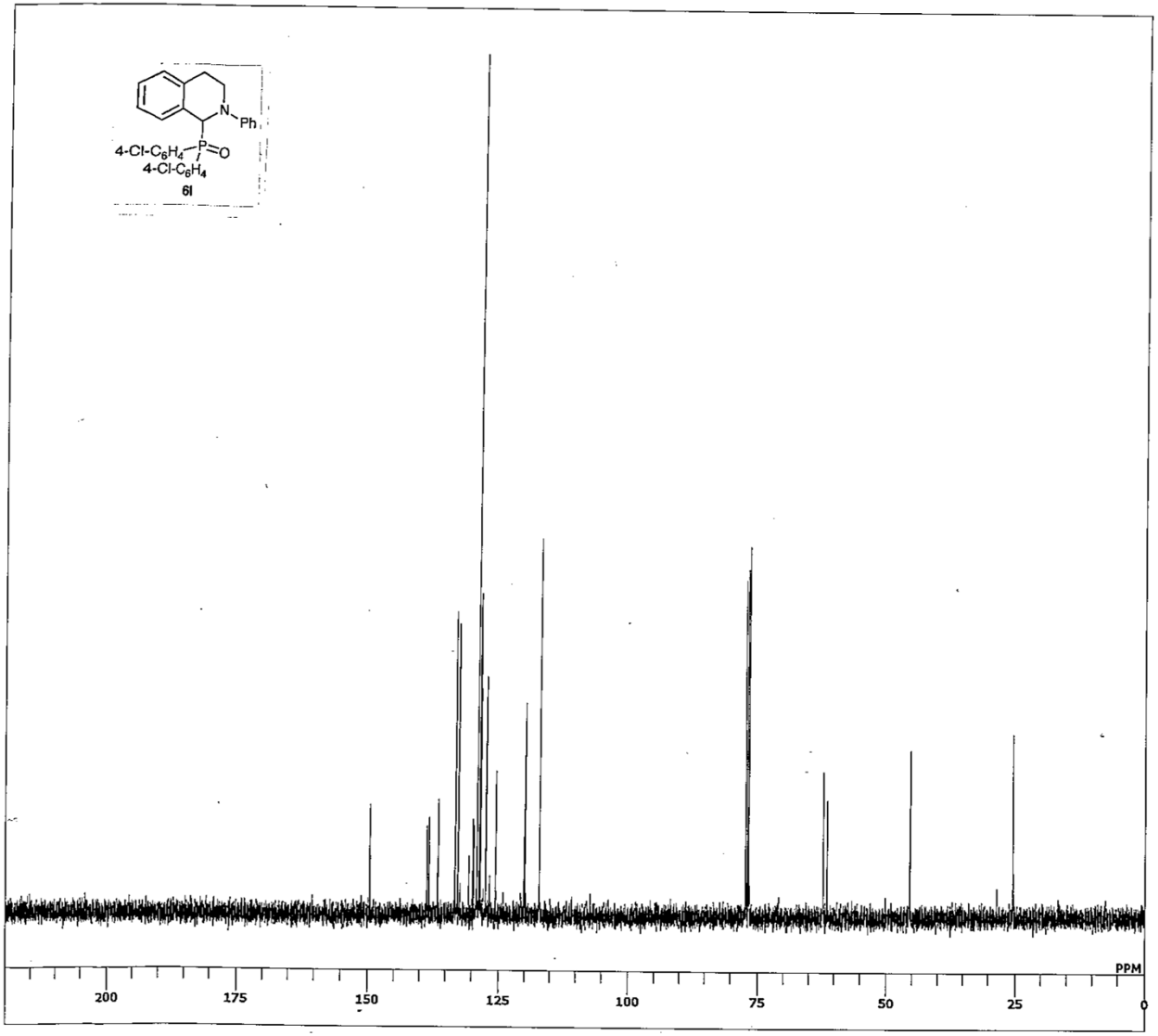
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	27-07-2013 17:13:04
OBNUC	31P
EXMOD	single_pulse_dec
OBFRQ	161.83 MHz
OBSET	4.69 KHz
OBFIN	3.09 Hz
POINT	32768
FREQU	142045.45 Hz
SCANS	10
ACQTM	0.2307 sec
PD	2.0000 sec
PW1	5.80 usec
IRNUC	1H
CTEMP	408.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.42 Hz
RGAIN	54

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1619-1H.2



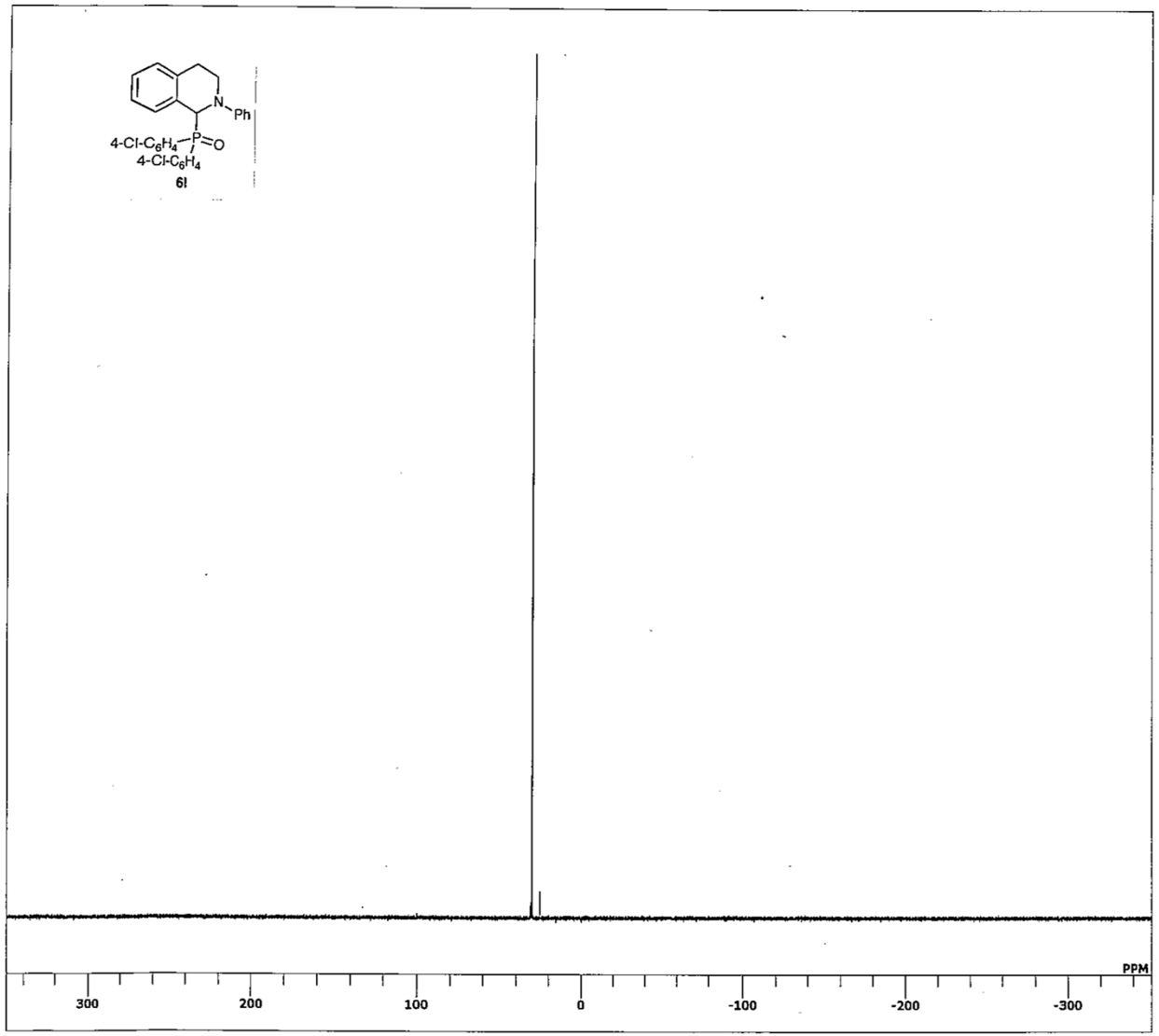
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	14-08-2013 14:44:26
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	6.50 usec
IRNUC	1H
CTEMP	400.8 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	28

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1619-13C.1



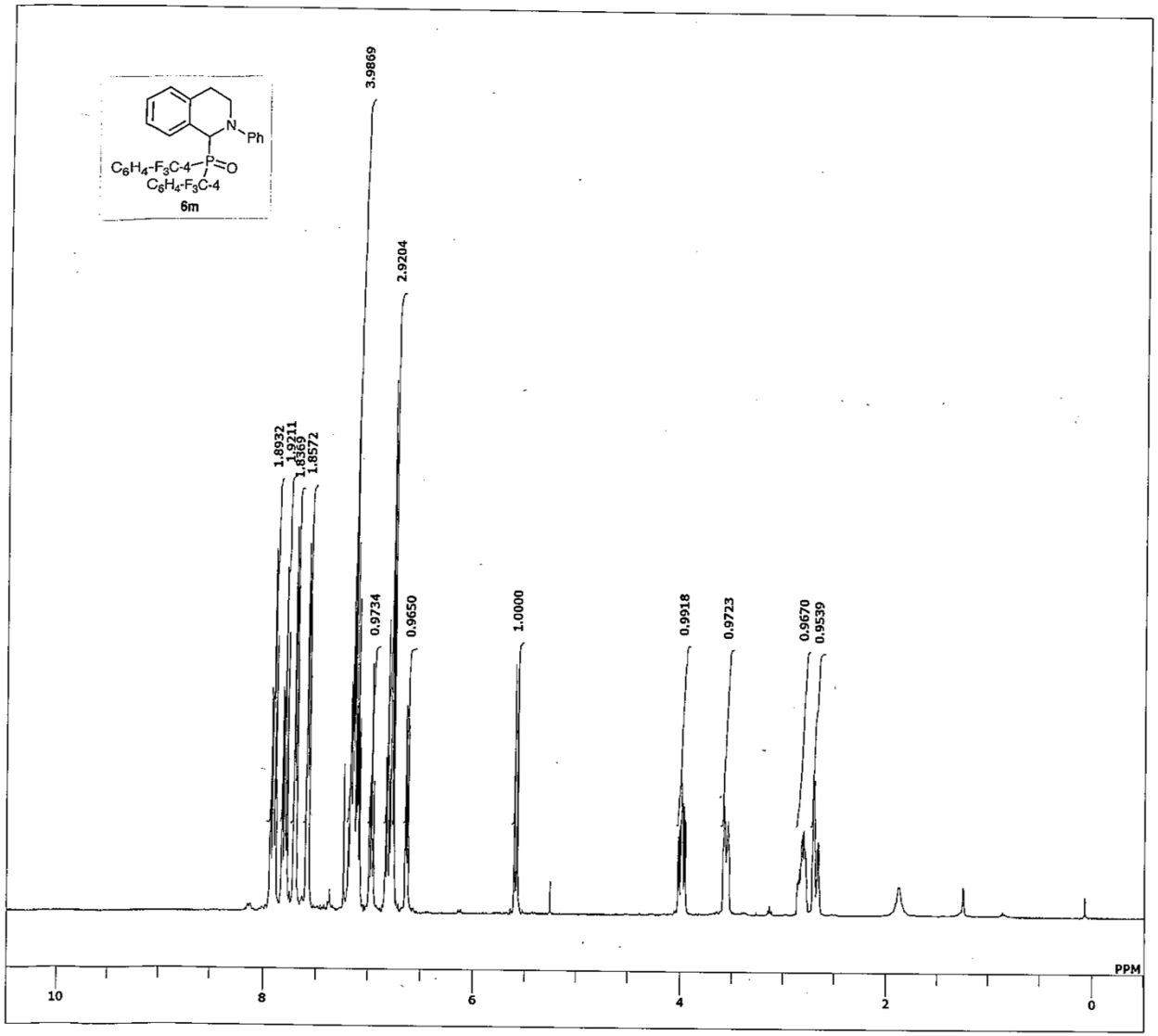
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	14-08-2013 14:50:35
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.03 Hz
SCANS	71
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	401.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	56

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1619-31P.1



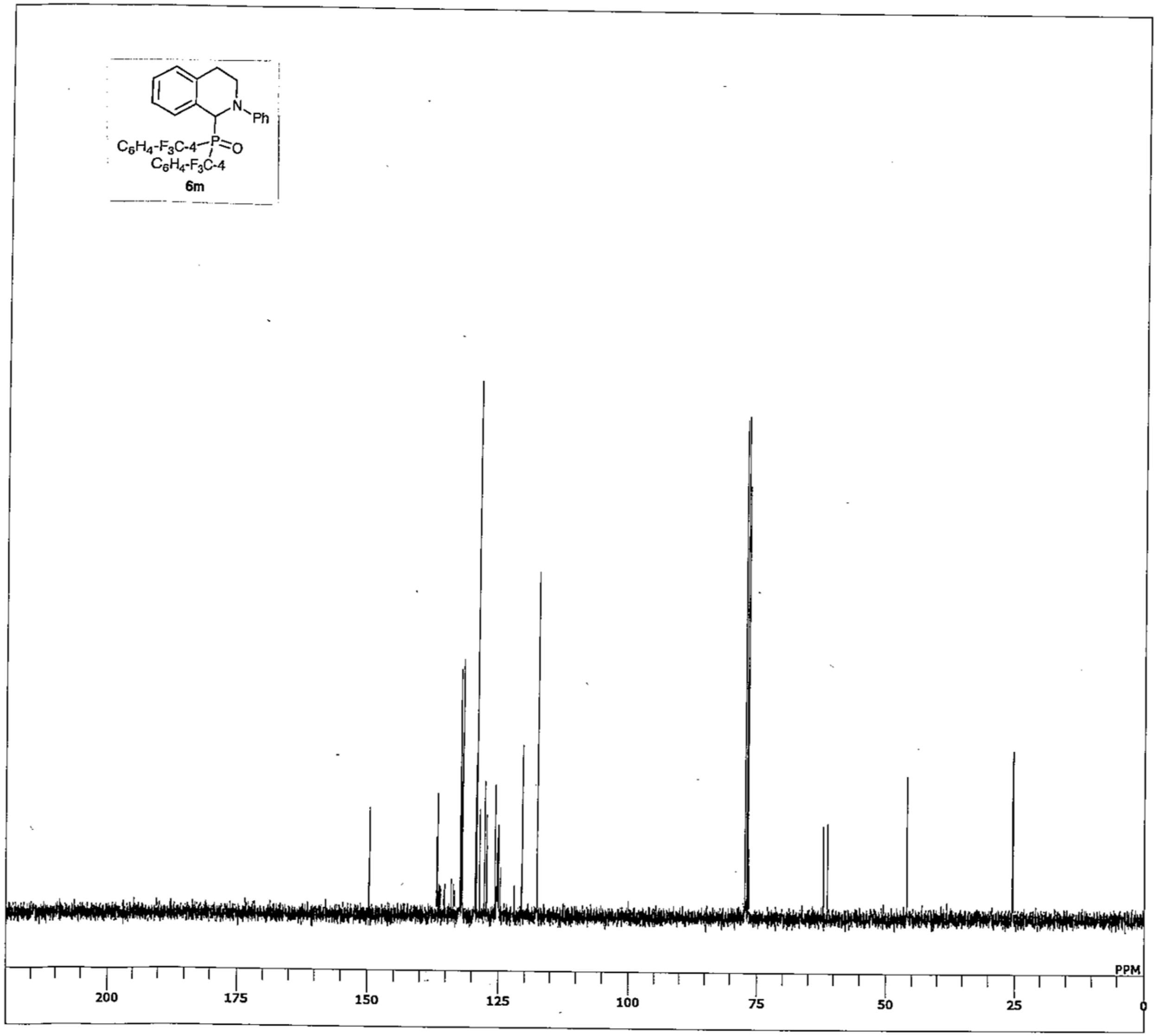
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	14-08-2013 14:53:41
OBNUC	31P
EXMOD	single_pulse_dec
OBFRQ	161.83 MHz
OBSET	4.69 KHz
OBFIN	3.09 Hz
POINT	32768
FREQU	142045.45 Hz
SCANS	11
ACQTM	0.2307 sec
PD	2.0000 sec
PW1	5.80 usec
IRNUC	1H
CTEMP	401.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.42 Hz
RGAIN	54

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1616-1H.1



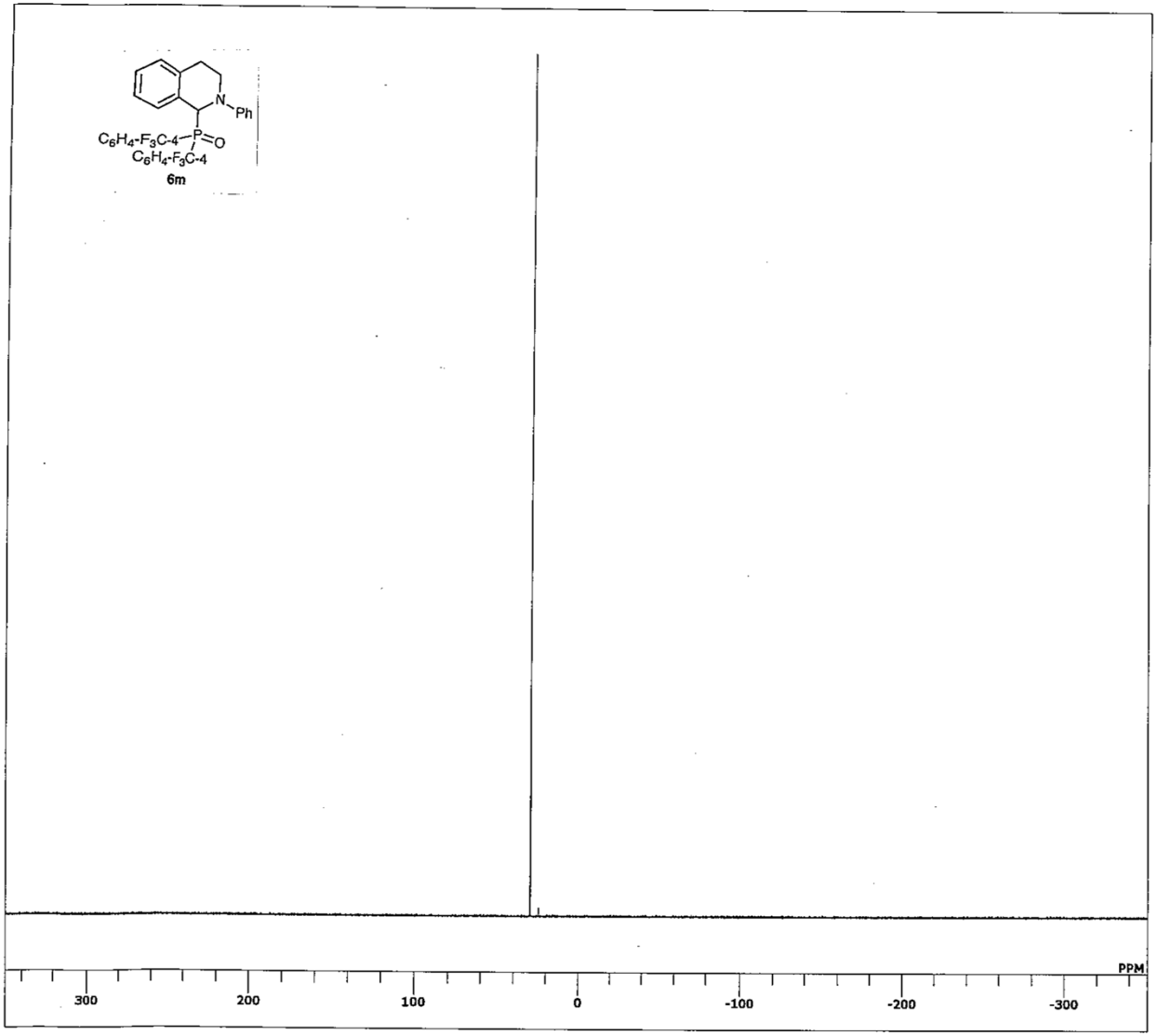
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	09-08-2013 09:41:16
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	6.50 usec
IRNUC	1H
CTEMP	400.8 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	32

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1616-13C.1



DFILE D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT
DATIM 09-08-2013 09:55:19
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 32768
FREQU 31407.03 Hz
SCANS 234
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.50 usec
IRNUC 1H
CTEMP 402.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 56

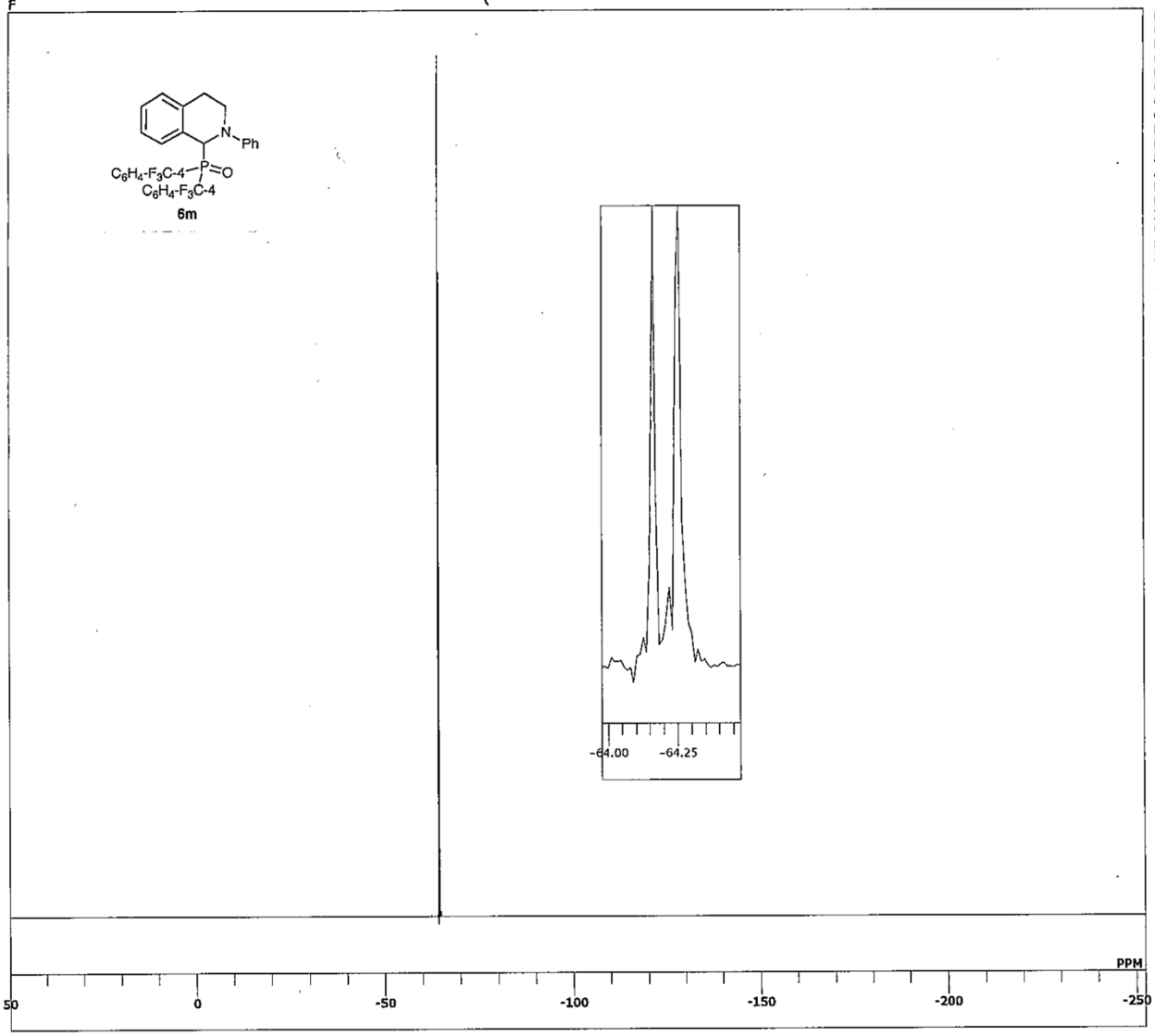
D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1616-31P.1



DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	09-08-2013 09:59:48
OBNUC	31P
EXMOD	single_pulse_dec
OBFRQ	161.83 MHz
OBSET	4.69 KHz
OBFIN	3.09 Hz
POINT	32768
FREQU	142045.45 Hz
SCANS	61
ACQTM	0.2307 sec
PD	2.0000 sec
PW1	5.80 usec
IRNUC	1H
CTEMP	402.4 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.42 Hz
RGAIN	56

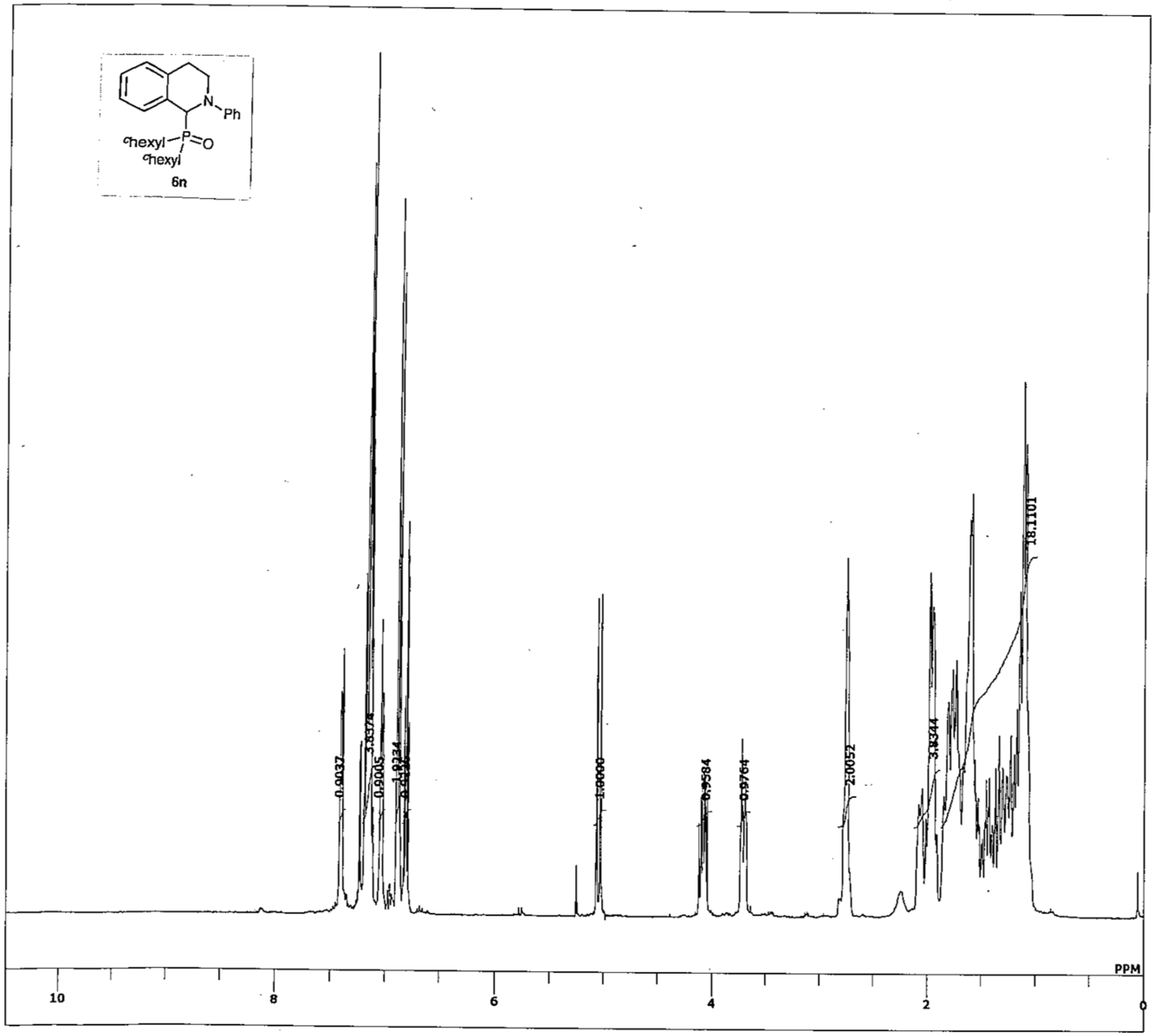
D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1616-19F.1

F



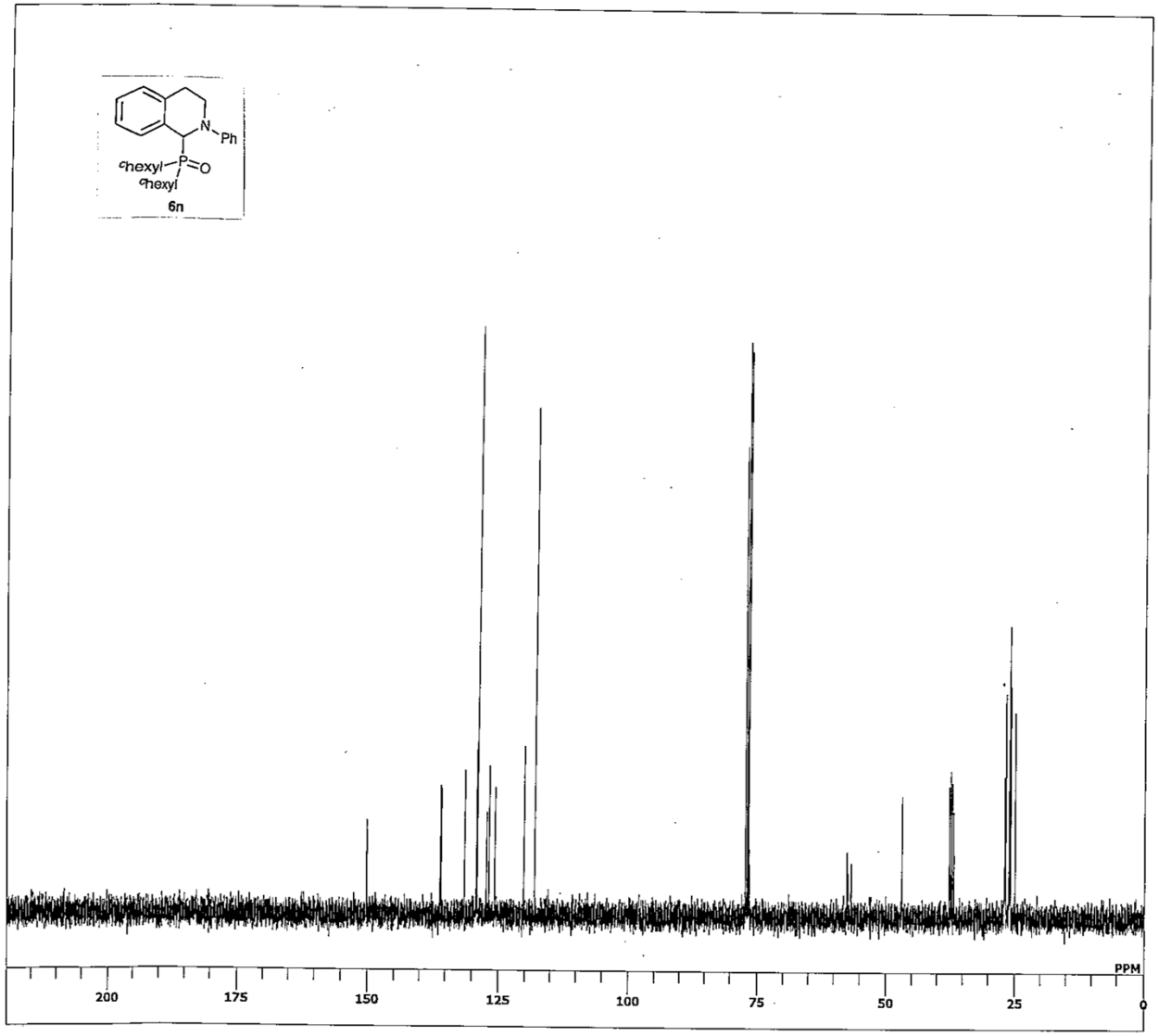
DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	F
DATIM	09-08-2013 10:03:20
OBNUC	19F
EXMOD	single_pulse_man
OBFRQ	376.13 MHz
OBSET	3.43 KHz
OBFIN	6.82 Hz
POINT	32768
FREQU	142045.45 Hz
SCANS	8
ACQTM	0.2307 sec
PD	7.0000 sec
PW1	6.70 usec
IRNUC	19F
CTEMP	402.7 c
SLVNT	CDCL3
EXREF	-64.16 ppm
BF	0.12 Hz
RGAIN	50

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1617-1H.1



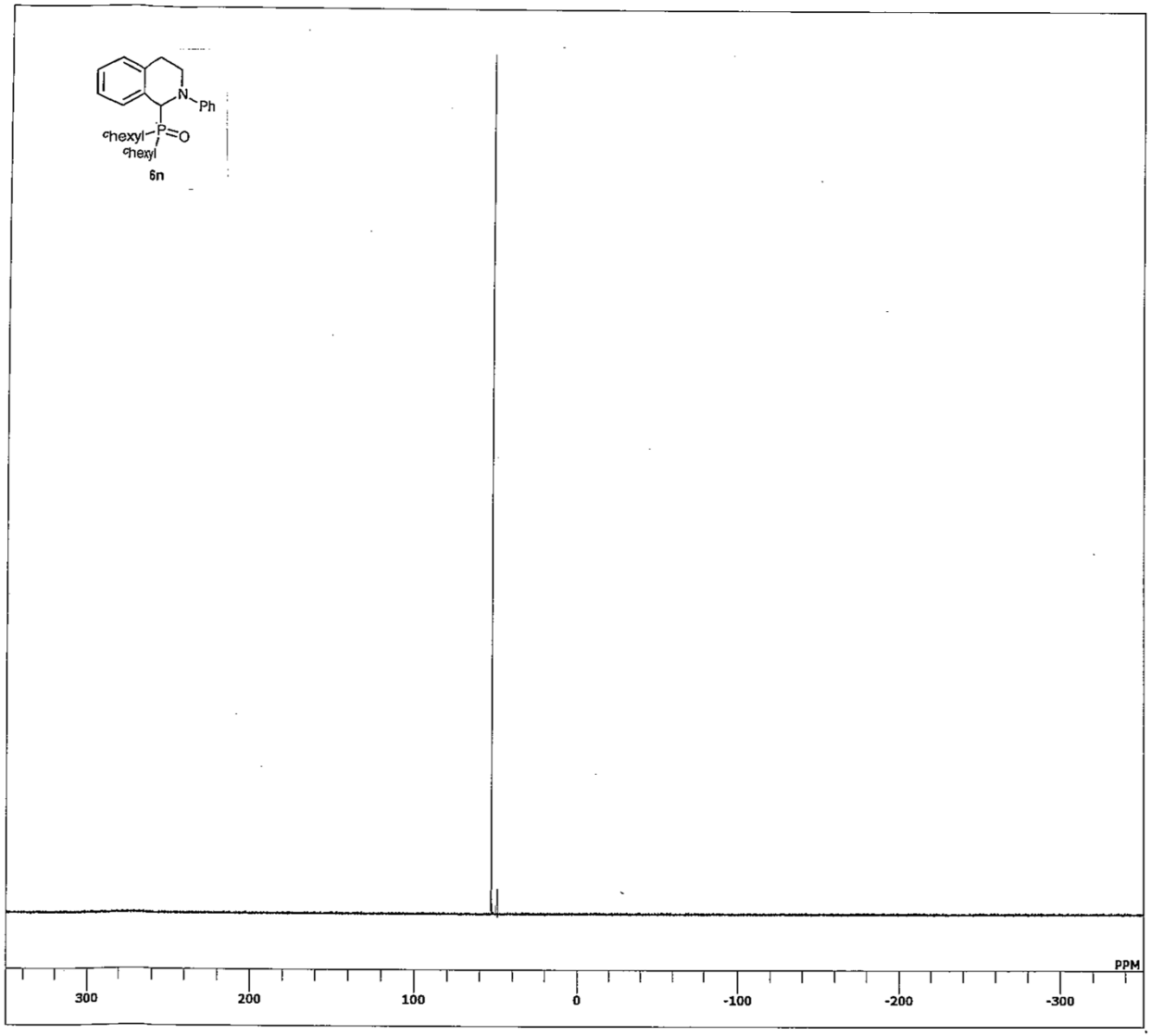
DFILE D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT
DATIM 09-08-2013 10:08:58
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 8
ACQTM 2.1837 sec
PD 2.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 403.2 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 28

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1617-13C.1



DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	09-08-2013 10:16:48
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.03 Hz
SCANS	91
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	403.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	58

D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-1617-31P.1



DFILE	D:¥Documents and Settings¥Kobayashilab¥Desktop¥WY-16
COMNT	
DATIM	09-08-2013 10:20:22
OBNUC	31P
EXMOD	single_pulse_dec
OBFRQ	161.83 MHz
OBSET	4.69 KHz
OBFIN	3.09 Hz
POINT	32768
FREQU	142045.45 Hz
SCANS	28
ACQTM	0.2307 sec
PD	2.0000 sec
PW1	5.80 usec
IRNUC	1H
CTEMP	404.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.42 Hz
RGAIN	54

Part III: References

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