Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information

Photoredox Catalyzed C–P Bond Forming Reactions – Visible Light Mediated Oxidative Phosphonylations of Amines

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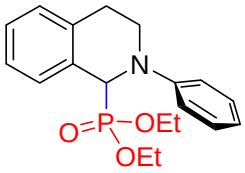
General: Unless otherwise noted, all commercially available compounds were used as provided without further purification. Solvents used in reactions were p.A. grade and dried only if indicated. Solvents for chromatography were technical grade and distilled prior to use. Analytical thin-layer chromatography (TLC) was performed on Merck silica gel aluminum plates with F-254 indicator, visualized by irradiation with UV light. Column chromatography was performed using silica gel Merck 60 (particle size 0.063 – 0.2 mm). Solvent mixtures are understood as volume/volume.

^{1}H-NMR and ^{13}C-NMR were recorded on a Varian VNMR 400 or Mercury 300 spectrometer in $CDCl_3$. Data are reported in the following order: chemical shift (δ) in ppm; multiplicities are indicated br (broadened singlet), s (singlet), d (doublet), t (triplet), q (quartett), m (multiplet); coupling constants (*J*) are in Hertz (Hz). Mass spectra (MS-EI, 70 eV) were conducted on Firma Finnigan MAT SSQ 700. IR spectra were recorded on a Perkin Elmer Spectrum 100 spectrometer and are reported in terms of frequency of absorption (cm^{-1}).

General procedure for the Photoredox Catalyzed C–P Bond Forming Reactions

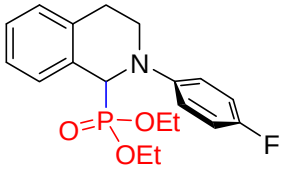
In a vial the appropriate photoredox catalyst and substrate were dissolved in the solvent indicated. Subsequently, 3 equiv. of the phosphite ester were added via syringe and the reaction mixture was stirred for the time indicated under irradiation with the light source as indicated (fluorescent bulb Osram Dulux S 5W, distance app. 5 cm). The reaction was monitored via TLC (hexanes: ethyl acetate). Upon consumption of starting material the crude mixture was purified by column chromatography (hexanes: ethyl acetate) to yield the corresponding products.

Diethyl 2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-ylphosphonate[1] 5a



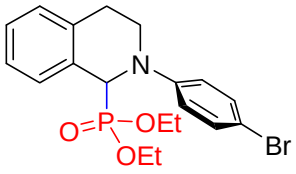
was synthesized according to the general procedure (48h) ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.31-7.29 (m, 1H), 7.20-7.09 (m, 5H), 6.91 (d, 2H, J = 8.1 Hz), 6.72 (t, 1H, J = 7.3 Hz), 5.11 (d, 1H, J = 20.1 Hz), 4.05-3.78 (m, 5H), 3.60-3.52 (m, 1H), 3.05-2.89 (m, 2H), 1.17 (t, 3H, J = 7.1 Hz), 1.06 (t, 3H, J = 7.1 Hz); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 149.3 (d, J = 5.6 Hz), 136.4 (d, J = 5.4 Hz), 130.6, 129.1 (2C), 128.7 (d, J = 2.3 Hz), 128.1 (d, J = 4.6 Hz), 127.4 (d, J = 3.4 Hz), 125.8 (d, J = 2.6 Hz), 118.5, 114.8 (2C), 63.3 (d, J = 7.2 Hz), 62.3 (d, J = 7.7 Hz), 58.8 (d, J = 159.3 Hz), 43.5, 26.7, 16.4 (d, J = 5.6 Hz), 16.3 (d, J = 6.0 Hz); ^{31}P NMR (121 MHz, $CDCl_3$): = δ 22.1; IR (neat): ν = 3460, 2920, 2853, 1592, 1447, 1385, 1242, 1025, 961, 749 cm^{-1}; EI-MS: m/z = 346 (M^++1, 8%) 345 (M^+, 37%) 208 (M^+-$PO(OEt)_2$, 100%).

Diethyl 2-(4-fluorophenyl)-1,2,3,4-tetrahydroisoquinolin-1-ylphosphonate 5b



was synthesized according to the general procedure (48h) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.31-7.29 (m, 1H), 7.19-7.07 (m, 3H), 6.90-6.82 (m, 4H), 4.98 (d, 1H, J = 20.3 Hz), 4.02-3.81 (m, 5H), 3.49-3.44 (m, 1H), 3.01-2.84 (m, 2H), 1.17 (t, 3H, J = 7.1 Hz), 1.08 (t, 3H, J = 7.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 156.4 (d, J = 238.0 Hz), 146.1 (dd, J = 1.9, 6.6 Hz), 136.3 (d, J = 5.6 Hz), 130.4, 128.8 (d, J = 2.5 Hz), 128.1 (d, J = 4.5 Hz), 127.4 (d, J = 3.4 Hz), 125.9 (d, J = 2.9 Hz), 116.5 (d, J = 7.4 Hz), 115.4 (d, J =22.1 Hz), 63.2 (d, J = 7.2 Hz), 62.3 (d, J = 7.6 Hz), 59.3 (d, J = 159.0 Hz), 44.3, 26.5, 16.4 (d, J = 5.6 Hz), 16.3 (d, J = 5.9 Hz); ^{31}P NMR (121 MHz, $CDCl_3$): δ = 22.0; ^{19}F NMR (376 MHz, $CDCl_3$): δ = -126.3; IR (neat): ν = 3445, 3019, 2924, 2854, 1712, 1441, 1384, 1215, 1027, 756 cm^{-1}; EI-MS: m/z = 364 (M^++1, 4%), 363 (M^+, 17%), 226 (M^+-$PO(OEt)_2$, 100%).

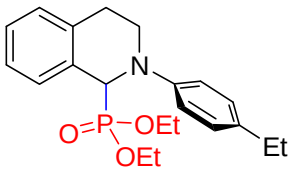
Diethyl 2-(4-bromophenyl)-1,2,3,4-tetrahydroisoquinolin-1-ylphosphonate 5c



was synthesized according to the general procedure (96h) ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.29-7.23 (m, 3H), 7.15-7.11 (m, 3H), 6.79-6.76 (m, 2H), 5.03 (d, 1H, J = 19.2 Hz), 4.06-3.74 (m, 5H), 3.50-3.42 (m, 1H), 3.17-3.04 (m, 1H), 2.95-2.87 (m, 1H), 1.16 (t, 3H, J = 7.1 Hz), 1.07 (t, 3H, J = 7.1 Hz); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 148.3 (d, J = 4.7 Hz), 136.3, 131.8 (2C), 130.4, 128.6 (d, J = 2.6 Hz), 128.1 (d, J = 4.8 Hz), 127.6 (d, J = 3.2 Hz), 126.0 (d, J = 2.7 Hz), 116.1 (2C), 110.3, 63.2 (d, J = 7.4 Hz), 62.4

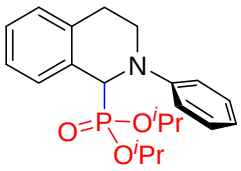
(d, J = 7.6 Hz), 58.7 (d, J = 159.6 Hz), 43.6, 26.9, 16.4 (d, J = 7.0 Hz), 16.3 (d, J = 6.0 Hz); ^{31}P NMR (121 MHz, $CDCl_3$): δ = 21.8; IR (neat): ν = 3442, 3014, 2926, 2856, 1712, 1657, 1488, 1385, 1216, 1024, 756 cm^{-1}; EI-MS: m/z = 425 (M^++2, 4%), 423 (M^+, 4%), 286 (M^+-$PO(OEt)_2$, 100%).

Diethyl 2-(4-ethylphenyl)-1,2,3,4-tetrahydroisoquinolin-1-ylphosphonate 5d



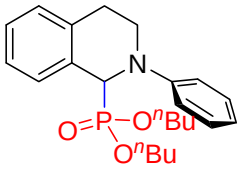
was synthesized according to the general procedure (48h) ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.32-7.28 (m, 1H), 7.12-7.06 (m, 3H), 7.02-6.99 (m, 2H), 6.85-6.82 (m, 2H), 5.06 (d, 1H, J = 20.7 Hz), 4.07-3.80 (m, 5H), 3.57-3.50 (m, 1H), 2.94-2.90 (m, 2H), 2.48 (q, J = 7.6 Hz, 2H), 1.18 (t, 3H, J = 7.1 Hz), 1.11 (t, 3H, J = 7.6 Hz), 1.07 (t, 3H, J = 7.1 Hz); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 147.6 (d, J = 6.9 Hz), 136.4 (d, J = 5.6 Hz), 134.5, 130.6, 128.8 (d, J = 2.5 Hz), 128.4 (2C), 128.1 (d, J = 4.5 Hz), 127.3 (d, J = 3.5 Hz), 125.8 (d, J = 2.9 Hz), 115.3 (2C), 63.3 (d, J = 7.2 Hz), 62.2 (d, J = 7.6 Hz), 59.1 (d, J = 159.3 Hz), 43.7, 27.8, 26.5, 16.5 (d, J = 5.6 Hz), 16.4 (d, J = 6.0 Hz), 15.8; ^{31}P NMR (121 MHz, $CDCl_3$): δ = 22.2; IR (neat): ν = 3450, 3017, 2925, 2856, 1732, 1652, 1447, 1383, 1215, 1029, 757 cm^{-1}; EI-MS: m/z = 374 (M^++1, 3%), 373 (M^+, 12%), 236 (M^+-$PO(OEt)_2$, 100%).

Diisopropyl 2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-ylphosphonate[1] 7a



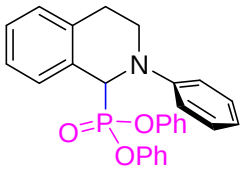
was synthesized according to the general procedure (72h) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.34-7.31 (m, 1H), 7.18-7.04 (m, 5H), 6.88 (d, 2H, J = 8.3 Hz), 6.69 (t, 1H, J = 7.3 Hz), 5.06 (d, 1H, J = 21.2 Hz), 4.59-4.51 (m, 2H), 4.02-3.95 (m, 1H), 3.61-3.55 (m, 1H), 2.96-2.90 (m, 2H), 1.22 (t, 6H, J = 6.4 Hz), 1.08 (d, 3H, J = 6.2 Hz), 0.87 (d, 3H, J = 6.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 149.5 (d, J = 6.6 Hz), 136.4 (d, J = 5.6 Hz), 130.9 (d, J = 1.4 Hz), 129.0 (2C), 128.7 (d, J = 2.6 Hz), 128.4 (d, J = 4.6 Hz), 127.2 (d, J = 4.6 Hz), 125.6 (d, J = 2.8 Hz), 118.3, 115.1 (2C), 72.2 (d, J = 7.8 Hz), 70.8 (d, J = 8.2 Hz), 58.8 (d, J = 164.9 Hz), 43.5, 26.6, 24.6 (d, J = 2.9 Hz), 24.1 (d, J = 3.2 Hz), 23.7 (d, J = 5.7 Hz), 23.3 (d, J = 5.6 Hz); ^{31}P NMR (121 MHz, $CDCl_3$): δ = 22.0; IR (neat): ν = 3467, 2978, 2924, 1597, 1499, 1460, 1381, 1243, 1107, 986, 750, 692, 566 cm^{-1}; EI-MS: m/z = 374 (M^++1, 3%), 373 (M^+, 11%), 208 (M^+-$PO(O^iPr)_2$, 100%).

Dibutyl 2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-ylphosphonate 8a



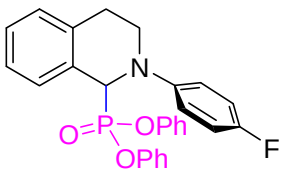
was synthesized according to the general procedure (72h) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.31-7.29 (m, 1H), 7.19-7.06 (m, 5H), 6.90 (d, 2H, J = 8.3 Hz), 6.71 (t, 1H, J = 7.3 Hz), 5.12 (d, 1H, J = 19.9 Hz), 3.98-3.86 (m, 3H), 3.84-3.78 (m, 1H), 3.76-3.68 (m, 1H), 3.59-3.53 (m, 1H), 3.02-2.89 (m, 2H), 1.52-1.45 (m, 2H), 1.41-1.34 (m, 2H), 1.30-1.13 (m, 4H), 0.81 (t, 3H, J = 7.4 Hz), 0.74 (t, 3H, J = 7.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 149.3 (d, J = 5.6 Hz), 136.3 (d, J = 5.6 Hz), 130.7, 129.1 (2C), 128.7 (d, J = 2.6 Hz), 128.1 (d, J = 4.6 Hz), 127.4 (d, J = 3.4 Hz), 125.8 (d, J = 2.7 Hz), 118.4, 114.8 (2C), 66.9 (d, J = 7.6 Hz), 65.9 (d, J = 8.0 Hz), 58.7 (d, J = 158.6 Hz), 43.4, 32.6 (d, J = 5.9 Hz), 32.5 (d, J = 6.1 Hz, 2C), 26.8, 18.6 (d, J = 6.2 Hz, 2C), 13.5 (d, J = 4.1 Hz, 2C); ^{31}P NMR (121 MHz, $CDCl_3$): δ = 23.3; IR (neat): ν = 3485, 2960, 2875, 2425, 1465, 1262, 1069, 1031, 977, 556 cm^{-1}. EI-MS: m/z = 402 (M^++1, 5%), 401 (M^+, 19%), 208 (M^+-PO(O^{n}Bu)$_2$, 100%).

Diphenyl 2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-ylphosphonate 9a



was synthesized according to the general procedure (24h) ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.46-7.42 (m, 1H), 7.22-6.93 (m, 16H), 6.80-6.74 (m, 2H), 5.52 (d, 1H, J = 19.9 Hz), 4.03-3.94 (m, 1H), 3.62-3.55 (m, 1H), 2.97-2.95 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 150.7 (d, J = 10.4 Hz), 150.3 (d, J = 11.4 Hz), 149.2 (d, J = 6.9 Hz), 136.7 (d, J = 5.9 Hz), 129.6 (2C), 129.4 (2C), 129.3, 129.2 (2C), 129.0 (d, J = 2.6 Hz), 128.4 (d, J = 4.9 Hz), 127.9 (d, J = 3.7 Hz), 126.2 (d, J = 2.8 Hz), 125.0, 124.8, 120.6 (d, J = 4.1 Hz, 2C), 120.3 (d, J = 4.1 Hz, 2C), 119.1, 115.4 (2C), 59.1 (d, J = 160.6 Hz), 44.0, 26.6; ^{31}P NMR (121 MHz, $CDCl_3$): δ = 14.7; IR (neat): ν = 3462, 2920, 2852, 1736, 1586, 1448, 1384, 1188, 1025, 930, 756 cm^{-1}; EI-MS: m/z = 442 (M^++1, 5%), 441 (M^+, 17%), 208 (M^+-PO(OPh)$_2$, 100%).

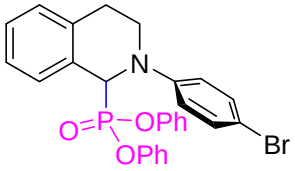
Diphenyl 2-(4-fluorophenyl)-1,2,3,4-tetrahydroisoquinolin-1-ylphosphonate 9b



was synthesized according to the general procedure (12h) ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.46-7.43 (m, 1H), 7.20-6.80 (m, 17H), 5.37 (d, 1H, J = 20.5 Hz), 4.03-3.94 (m, 1H), 3.52-3.44 (m, 1H), 2.92-2.87 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 156.8 (d, J = 238.6 Hz), 150.8 (d, J = 10.5 Hz), 150.2 (d, J = 11.0 Hz), 146.0 (dd, J = 2.0, 8.0 Hz), 136.8 (d, J = 6.2 Hz), 129.6 (2C), 129.5 (2C), 129.2, 129.1 (d, J = 2.6 Hz), 128.4 (d, J = 4.9 Hz), 128.0 (d, J = 3.7 Hz), 126.3 (d, J = 2.9 Hz), 125.0, 124.8, 120.6 (d, J = 4.2 Hz,

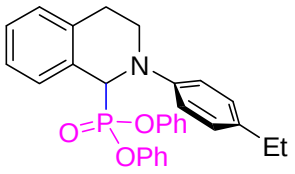
2C), 120.3 (d, J = 4.1 Hz, 2C), 117.5 (d, J = 7.5 Hz, 2C), 115.6 (d, J = 22.2 Hz, 2C), 59.6 (d, J = 160.3 Hz), 44.9, 26.3; ^{31}P NMR (121 MHz, $CDCl_3$): δ 14.4; ^{19}F NMR (376 MHz, $CDCl_3$): δ -125.1; IR (neat): ν = 3448, 2924, 2855, 1730, 1591, 1453, 1213, 1022, 934, 755, 689 cm^{-1}; EI-MS: m/z = 459 (M^+, 2%), 226 (M^+-$PO(OPh)_2$, 100%).

Diphenyl 2-(4-bromophenyl)-1,2,3,4-tetrahydroisoquinolin-1-ylphosphonate 9c



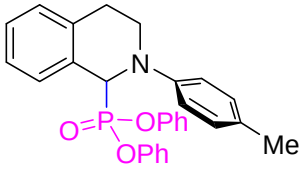
was synthesized according to the general procedure (16h) ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.44-7.41 (m, 1H), 7.28-6.90 (m, 13H), 6.83-6.76 (m, 4H), 5.43 (d, 1H, J = 19.1 Hz), 3.98-3.89 (m, 1H), 3.54-3.46 (m, 1H), 3.08-3.02 (m, 1H), 2.96-2.87 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 150.6 (d, J = 10.5 Hz), 150.2 (d, J = 11.3 Hz), 148.2 (d, J = 5.8 Hz), 136.5 (d, J = 5.6 Hz), 132.0 (2C), 129.6 (2C), 129.5 (2C), 129.3, 129.0 (d, J = 2.6 Hz), 128.4 (d, J = 5.0 Hz) 128.1 (d, J = 3.6 Hz), 126.4 (d, J = 2.6 Hz), 125.1, 124.9, 120.5 (d, J = 4.1 Hz, 2C), 120.2 (d, J = 4.1 Hz, 2C), 116.8 (2C), 111.1, 59.1 (d, J = 160.7 Hz), 44.1, 26.7; ^{31}P NMR (121 MHz, $CDCl_3$): δ = 14.2; IR (neat): ν = 3444, 2921, 2852, 1732, 1589, 1487, 1384, 1267, 1187, 931, 758 cm^{-1}; EI-MS: m/z = 521 (M^++2, 4%), 519 (M^+, 3%), 286 (M^+-$PO(OPh)_2$, 100%).

Diphenyl 2-(4-ethylphenyl)-1,2,3,4-tetrahydroisoquinolin-1-ylphosphonate 9d



was synthesized according to the general procedure (32h) ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.46-7.43 (m, 1H), 7.20-6.93 (m, 13H), 6.90-6.79 (m, 4H), 5.46 (d, 1H, J = 20.6 Hz), 4.03-3.94 (m, 1H), 3.60-3.52 (m, 1H), 2.94-2.89 (m, 2H), 2.49 (q, 2H, J = 7.6 Hz), 1.12 (t, 3H, J = 7.6 Hz); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 150.8 (d, J = 10.6 Hz), 150.3 (d, J = 10.9 Hz), 147.4 (d, J = 7.6 Hz), 136.8 (d, J = 5.8 Hz), 135.3, 129.6 (2C), 129.4 (2C), 129.1 (d, J = 2.5 Hz), 128.6 (2C), 128.4 (d, J = 4.9 Hz), 127.8 (d, J = 3.7 Hz), 126.1 (d, J = 2.8 Hz), 125.0, 124.8, 120.7 (d, J = 4.1 Hz, 2C), 120.4 (d, J = 4.1 Hz, 2C), 116.0 (2C), 115.3, 59.4 (d, J = 160.6 Hz), 44.3, 27.9, 26.4, 15.8; ^{31}P NMR (121 MHz, $CDCl_3$): δ = 14.8; IR (neat): ν = 3329, 2925, 2855, 1730, 1593, 1488, 1214, 1024, 934, 755, 690 cm^{-1}; EI-MS: m/z = 469 (M^+, 4%), 236 (M^+-$PO(OPh)_2$, 100%).

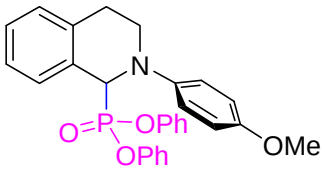
Diphenyl 2-p-tolyl-1,2,3,4-tetrahydroisoquinolin-1-ylphosphonate 9e



was synthesized according to the general procedure (12h) ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.46-7.43 (m, 1H), 7.18-6.94 (m, 13H), 6.87-6.80 (m, 4H), 5.44 (d, 1H, J = 20.9 Hz), 4.03-

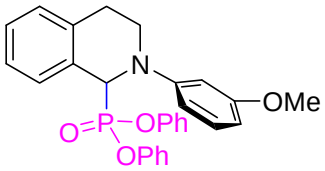
3.94 (m, 1H), 3.59-3.51 (m, 1H), 2.93-2.88 (m, 2H), 2.19 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 150.8 (d, *J* = 10.4 Hz), 150.3 (d, *J* = 10.7 Hz), 147.24 (d, *J* = 8.1 Hz), 136.8 (d, *J* = 6.0 Hz), 131.2, 129.7 (2C), 129.6 (2C), 129.4 (2C), 129.1 (d, *J* = 2.4 Hz), 128.7, 128.4 (d, *J* = 4.8 Hz), 127.8 (d, *J* = 3.6 Hz), 126.1 (d, *J* = 2.9 Hz), 125.0, 124.8, 120.7 (d, *J* = 4.1 Hz, 2C), 120.4 (d, *J* = 4.1 Hz, 2C), 116.1 (2C), 59.4 (d, *J* = 160.9 Hz), 44.4, 26.3, 20.4; ^{31}P NMR (121 MHz, $CDCl_3$): δ = 14.8; IR (neat): ν = 3452, 3019, 2924, 2855, 1711, 1444, 1216, 757 cm^{-1}; EI-MS: m/z = 455 (M^+, 7%), 222 (M^+-$PO(OPh)_2$, 100%).

Diphenyl 2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinolin-1-ylphosphonate 9f



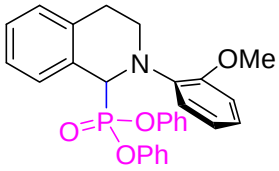
was synthesized according to the general procedure (12h) ^{1}H NMR (300 MHz, $CDCl_3$): *δ* = 7.48-7.44 (m, 1H), 7.20-6.95 (m, 11H), 6.88-6.83 (m, 4H), 6.75-6.72 (m, 2H), 5.32 (d, 1H, *J* = 21.9 Hz), 4.03-3.96 (m, 1H), 3.67 (s, 3H), 3.51-3.43 (m, 1H), 2.85-2.83 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 153.6, 151.0 (d, *J* = 10.5 Hz), 150.3 (d, *J* = 11.2 Hz), 143.9 (d, *J* = 9.8 Hz), 136.7 (d, *J* = 6.3 Hz), 129.6 (2C), 129.4 (2C), 129.2 (d, *J* = 2.4 Hz), 128.4 (d, *J* = 4.6 Hz), 127.8 (d, *J* = 3.6 Hz), 126.1 (d, *J* = 3.0 Hz), 125.0, 124.8, 120.7 (d, *J* = 4.2 Hz, 2C), 120.4 (d, *J* = 4.5 Hz, 2C), 118.6 (2C), 114.5 (2C), 59.8 (d, *J* = 160.4 Hz), 55.6, 45.4, 25.9; ^{31}P NMR (121 MHz, $CDCl_3$): δ = 14.6; IR (neat): ν = 3449, 2921, 2853, 1732, 1585, 1451, 1385, 1183, 958, 753 cm^{-1}; EI-MS: m/z = 471 (M^+, 8%), 238 (M^+-$PO(OPh)_2$, 100%).

Diphenyl 2-(3-methoxyphenyl)-1,2,3,4-tetrahydroisoquinolin-1-ylphosphonate 9g



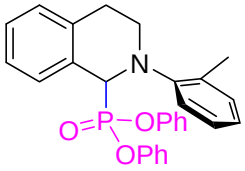
was synthesized according to the general procedure (20h) ^{1}H NMR (300 MHz, $CDCl_3$): *δ* = 7.44-7.41 (m, 1H), 7.17-6.93 (m, 12H), 6.81-6.78 (m, 2H), 6.63-6.56 (m, 1H), 6.51 (t, 1H, *J* = 2.3 Hz), 6.33 (dd, 1H, *J* = 2.3, 8.1 Hz), 5.51 (d, 1H, *J* = 19.8 Hz), 4.00-3.91 (m, 1H), 3.70 (s, 3H), 3.61-3.55 (m, 1H), 2.98-2.95 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 160.7, 150.6 (d, *J* = 7.0 Hz), 150.3 (d, *J* = 11.2 Hz), 136.7 (d, *J* = 5.6 Hz) 129.9, 129.6 (2C), 129.4 (2C), 129.0 (d, *J* = 2.6 Hz), 128.4 (d, *J* = 5.0 Hz), 128.0 (d, *J* = 3.5 Hz), 126.2 (d, *J* = 2.6 Hz), 125.1, 124.9, 120.6 (d, *J* = 4.1 Hz, 2C), 120.4 (d, *J* = 4.0 Hz, 2C), 120.0, 115.3, 108.2, 103.8, 102.1, 59.2 (d, *J* = 160.9 Hz), 55.2, 44.0, 26.7; ^{31}P NMR (121 MHz, $CDCl_3$): δ = 14.6; IR (neat): ν = 2924, 2853, 1734, 1590, 1488, 1251, 1191, 1162, 924, 755, 687 cm^{-1}; EI-MS: m/z = 471 (M^+, 3%), 238 (M^+-$PO(OPh)_2$, 100%)

Diphenyl 2-(2-methoxyphenyl)-1,2,3,4-tetrahydroisoquinolin-1-ylphosphonate 9h



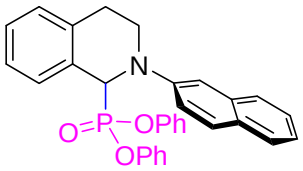
was synthesized according to the general procedure (22h) ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.53-7.49 (m, 1H), 7.18-7.07 (m, 7H), 7.03-6.77 (m, 10H), 5.49 (d, 1H, J = 21.0 Hz), 4.11-4.01 (m, 1H), 3.72 (s, 3H), 3.65-3.57 (m, 1H), 2.91-2.83 (m, 1H), 2.77-2.68 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 152.6, 150.7 (d, J = 10.5 Hz), 150.4 (d, J = 10.9 Hz), 139.6 (d, J = 8.4 Hz), 136.2 (d, J = 6.4 Hz), 130.1, 129.6, 129.4 (2C), 129.3 (2C), 128.5 (d, J = 4.2 Hz), 127.5 (d, J = 3.7 Hz), 125.8 (d, J = 3.3 Hz), 124.7, 124.6, 123.5, 122.1, 120.9, 120.6 (d, J = 4.1 Hz, 4C), 111.7, 59.1 (d, J = 147.7 Hz), 55.4, 44.8, 26.7; ^{31}P NMR (121 MHz, $CDCl_3$): δ = 16.1; IR (neat): ν = 3064, 2925, 1590, 1490, 1457, 1240, 1186, 1024, 924, 748, 689 cm^{-1}; EI-MS: m/z = 472 (M^++1, 2%), 471 (M^+, 7%), 238 (M^+-$PO(OPh)_2$, 100%)

Diphenyl 2-o-tolyl-1,2,3,4-tetrahydroisoquinolin-1-ylphosphonate 9i



was synthesized according to the general procedure (36h) ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.57-7.53 (m, 1H), 7.18-7.10 (m, 8H), 7.03-6.78 (m, 9H), 5.10 (d, 1H, J = 25.1 Hz), 4.07-3.98 (m, 1H), 3.22-3.15 (m, 1H), 2.82-2.59 (m, 2H), 2.22 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 150.9 (d, J = 10.7 Hz), 150.5 (d, J = 10.6 Hz), 150.1 (d, J = 12.3 Hz), 137.0 (d, J = 6.8 Hz), 133.7, 131.3, 129.5 (5C), 129.3 (d, J = 1.5 Hz), 128.5 (d, J = 4.0 Hz), 127.5 (d, J = 3.7 Hz), 126.6, 126.1 (d, J = 3.2 Hz), 124.8, 124.7, 124.1, 122.7, 120.6 (d, J = 4.1 Hz, 2C), 120.4 (d, J = 4.2 Hz, 2C), 60.3 (d, J = 160.4 Hz), 45.9, 25.1, 18.0; ^{31}P NMR (121 MHz, $CDCl_3$): δ = 14.8; IR (neat): ν = 3064, 2924, 1591, 1487, 1271, 1186, 925, 754, 688 cm^{-1}; EI-MS: m/z = 456 (M^++1, 1%), 455 (M^+, 5%), 222 (M^+-$PO(OPh)_2$, 100%).

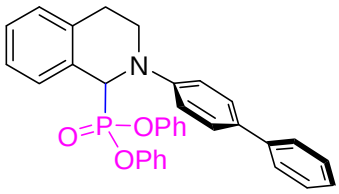
Diphenyl 2-(naphthalen-2-yl)-1,2,3,4-tetrahydroisoquinolin-1-ylphosphonate 9j



was synthesized according to the general procedure (48h) ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.70-6.50 (m, 5H), 7.20-6.94 (m, 14 H), 6.82-6.78 (m, 2H), 5.66 (d, 1H, J = 20.7 Hz), 4.15-4.07 (m, 1H), 3.79-3.71 (m, 1H), 3.01-2.96 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 150.8 (d, J = 10.6 Hz), 150.3 (d, J = 11.1 Hz), 147.0 (d, J = 7.3 Hz), 136.6 (d, J = 5.7 Hz), 134.6, 132.1, 129.6 (2C), 129.4 (2C), 129.2 (d, J = 2.4 Hz), 129.1, 128.5 (d, J = 4.9 Hz), 128.1, 128.0 (d, J = 3.7 Hz), 127.4, 126.6, 126.4, 126.3, 125.1, 124.9, 123.2, 120.6 (d, J = 4.1 Hz, 2C), 120.3 (d, J = 4.1 Hz, 2C), 118.2, 110.3, 59.1 (d, J = 159.9 Hz), 44.3, 26.5; ^{31}P NMR (121 MHz, $CDCl_3$): δ = 14.6; IR (neat): ν = 3415, 2921,

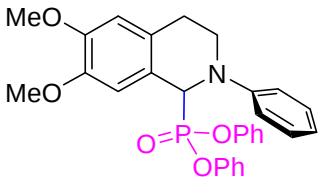
2852, 1736, 1627, 1592, 1488, 1386, 1213, 1189, 1004, 926, 754, 686 cm^{-1}; EI-MS: m/z = 491 (M^+). 491 (M^+, 3%), 258 (M^+-$PO(OPh)_2$, 100%).

Diphenyl 2-(biphenyl-4-yl)-1,2,3,4-tetrahydroisoquinolin-1-ylphosphonate 9k



was synthesized according to the general procedure (26h) ^1H NMR (300 MHz, $CDCl_3$): δ = 7.50-7.44 (m, 5H), 7.35-7.30 (m, 2H), 7.22-6.93 (m, 15H), 6.82-6.79 (m, 2H), 5.60 (d, 1H, J = 19.4 Hz), 4.06-3.94 (m, 1H), 3.67-3.61 (m, 1H), 3.11-2.91 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 150.2, 148.5 (d, J = 6.2 Hz), 140.8, 136.7 (d, J = 5.9 Hz), 131.7, 129.6 (2C), 129.5 (2C), 129.0 (d, J = 2.4 Hz), 128.7 (2C), 128.4 (d, J = 5.3 Hz), 128.0 (d, J = 3.2 Hz), 127.8 (2C), 127.6, 127.3, 126.4 (3C), 126.3 (d, J = 2.5 Hz), 125.5, 125.1, 124.9, 120.6 (d, J = 4.2 Hz, 2C), 120.3 (d, J = 4.0 Hz, 2C), 115.4 (2C), 59.1 (d, J = 160.3 Hz), 43.9, 26.8; ^{31}P NMR (121 MHz, $CDCl_3$): δ = 14.6; IR (neat): ν = 2921, 2853, 1737, 1593, 1487, 1380, 1213, 1190, 928, 756, 691 cm^{-1}; EI-MS: m/z = 517 (M^+, 5%), 284 (M^+-$PO(OPh)_2$, 100%).

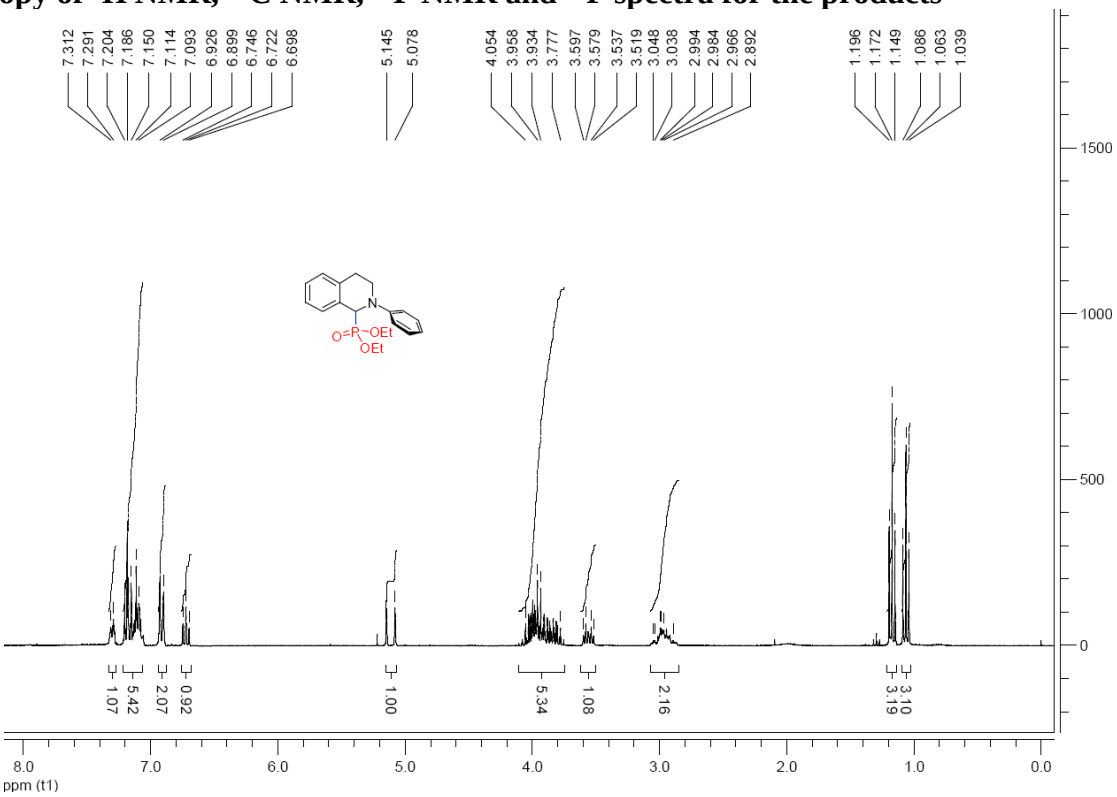
Diphenyl 6,7-dimethoxy-2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-ylphosphonate 9l

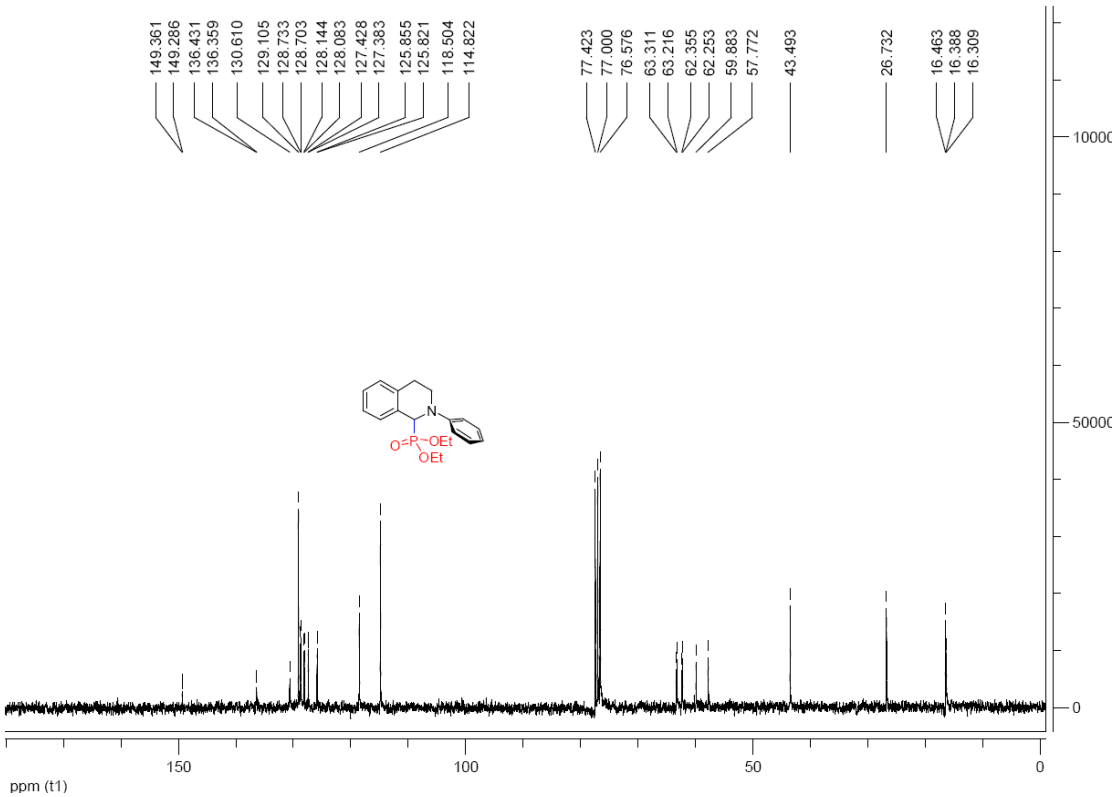


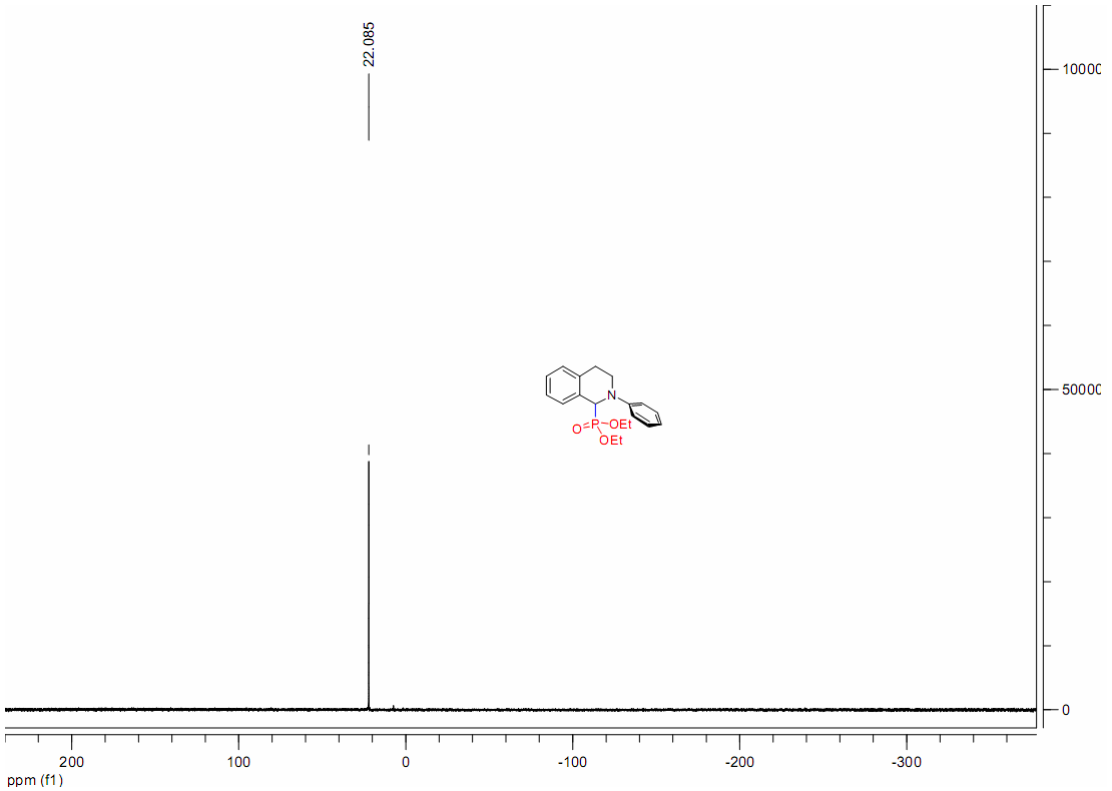
was synthesized according to the general procedure (20h) ^1H NMR (300 MHz, $CDCl_3$): δ = 7.22-6.94 (m, 14H), 6.84-6.77 (m, 3H), 6.59 (s, 1H), 5.42 (d, 1H, J = 20.1 Hz), 4.04-3.95 (m, 1H), 3.79 (s, 3H), 3.69 (s, 3H), 3.67-3.61 (m, 1H), 2.92-2.74 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 150.8 (d, J = 10.5 Hz), 150.4 (d, J = 11.1 Hz), 149.4 (d, J = 8.3 Hz), 148.7 (d, J = 3.6 Hz), 147.3 (d, J = 2.7 Hz), 129.7 (2C), 129.4 (2C), 129.2 (2C), 128.9 (d, J = 6.8 Hz), 125.1, 124.8, 120.7, 120.5 (d, J = 4.1 Hz, 2C), 120.4 (d, J = 4.1 Hz, 2C), 119.4, 116.0 (2C), 111.8 (d, J = 2.2 Hz), 111.1 (d, J = 3.9 Hz), 58.7 (d, J = 160.8 Hz), 56.0, 55.9, 44.0, 25.8; ^{31}P NMR (121 MHz, $CDCl_3$): δ = 14.9; IR (neat): ν = 3330, 2919, 2853, 1738, 1593, 1460, 1376, 1189, 1091, 939, 754, 690 cm^{-1}; EI-MS: m/z = 501 (M^+, 1%), 268 (M^+-$PO(OPh)_2$, 100%).

1. O. Baslé and C. J. Li, *Chem. Commun.* 2009, 4124-4126.

Copy of ^{1}H NMR, ^{13}C NMR, ^{31}P NMR and ^{19}F spectra for the products



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100
0
-100
-200
-300
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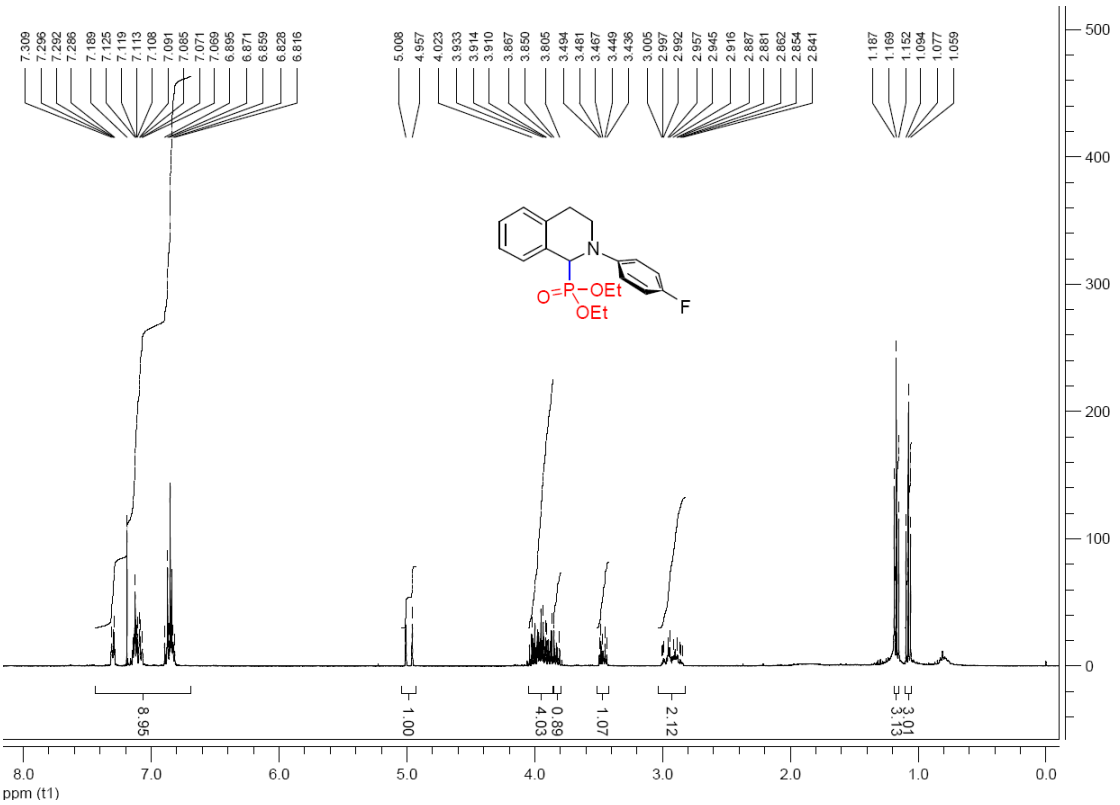
OEt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7.292
7.286
7.189
7.125
7.119
7.113
7.108
7.091
7.085
7.071
7.069
6.895
6.871
6.859
6.828
6.816
5.008
4.957
4.023
3.933
3.914
3.910
3.867
3.850
3.805
3.494
3.481
3.467
3.449
3.436
3.005
2.997
2.992
2.957
2.945
2.916
2.887
2.881
2.862
2.854
2.841
1.187
1.169
1.152
1.094
1.077
1.059
O=P–OEt
OEt
F
8.95
1.00
4.03
0.89
1.07
2.12
3.13
3.01
500
400
300
200
100
0
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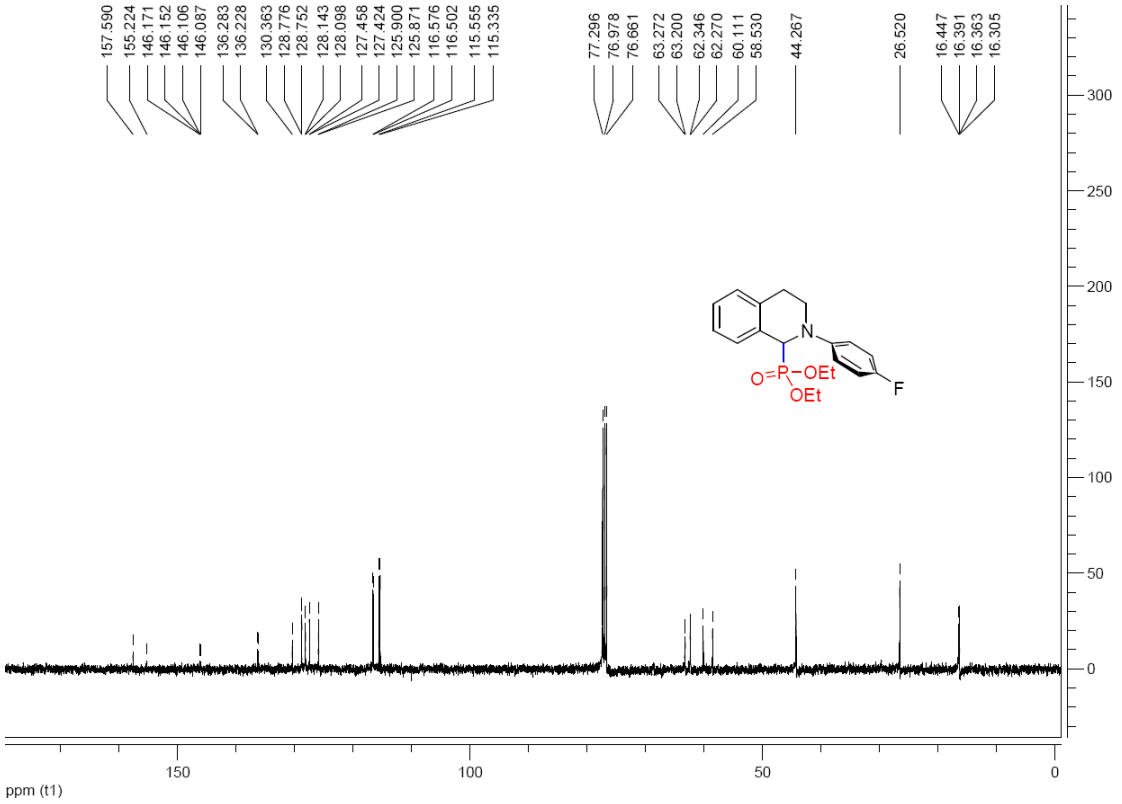
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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127.458
127.424
125.900
125.871
116.576
116.502
115.555
115.335
77.296
76.978
76.661
63.272
63.200
62.346
62.270
60.111
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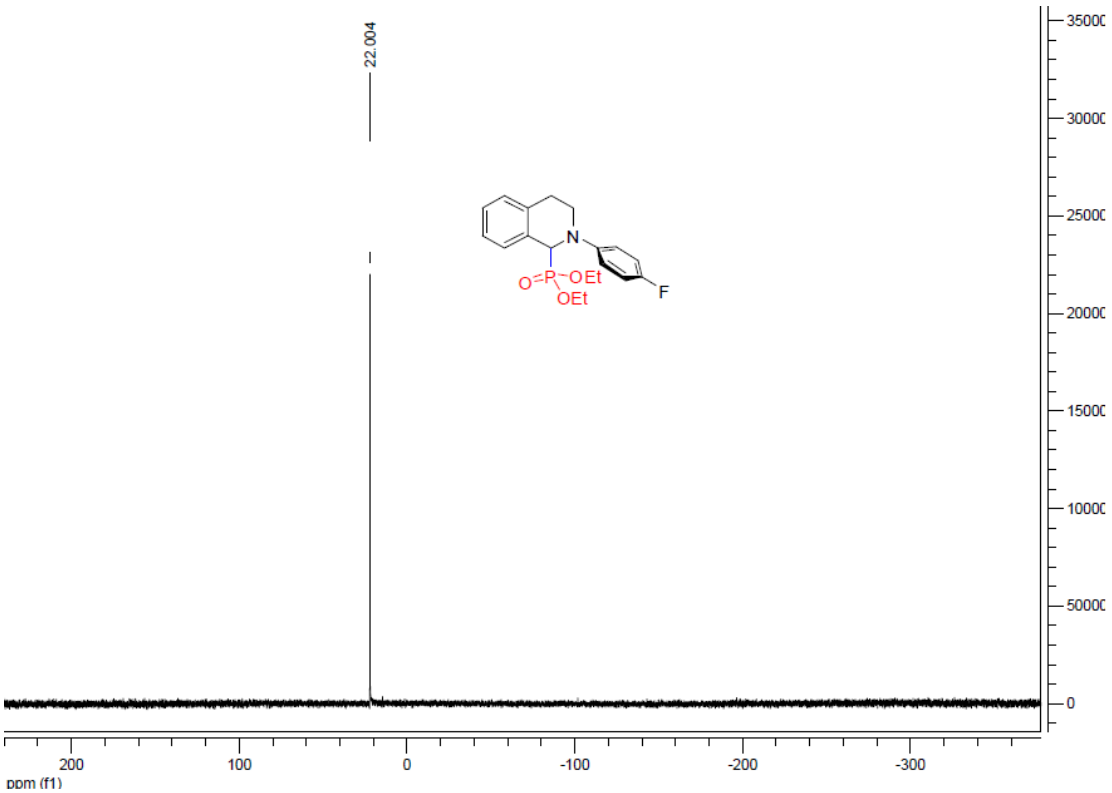
26.520
16.447
16.391
16.363
16.305
O=P-OEt
OEt
N
F
ppm (t1)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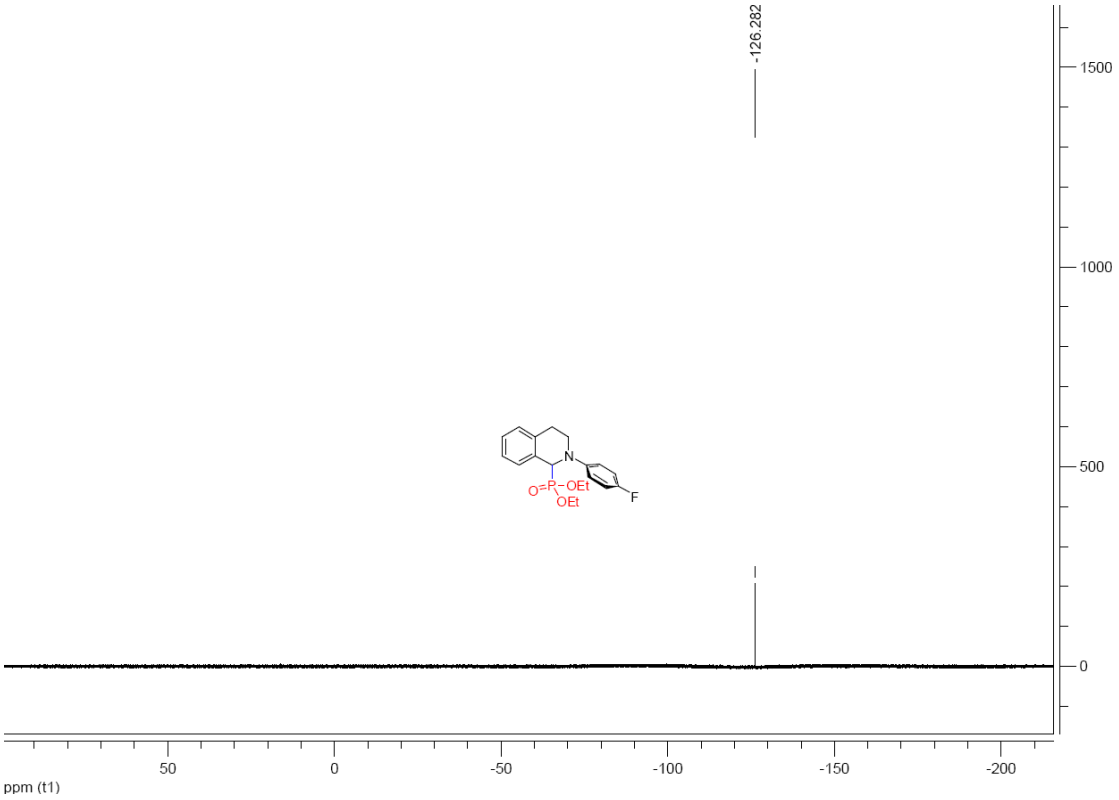
OEt
N
F
ppm (f1)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500
0
50
0
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-150
-200
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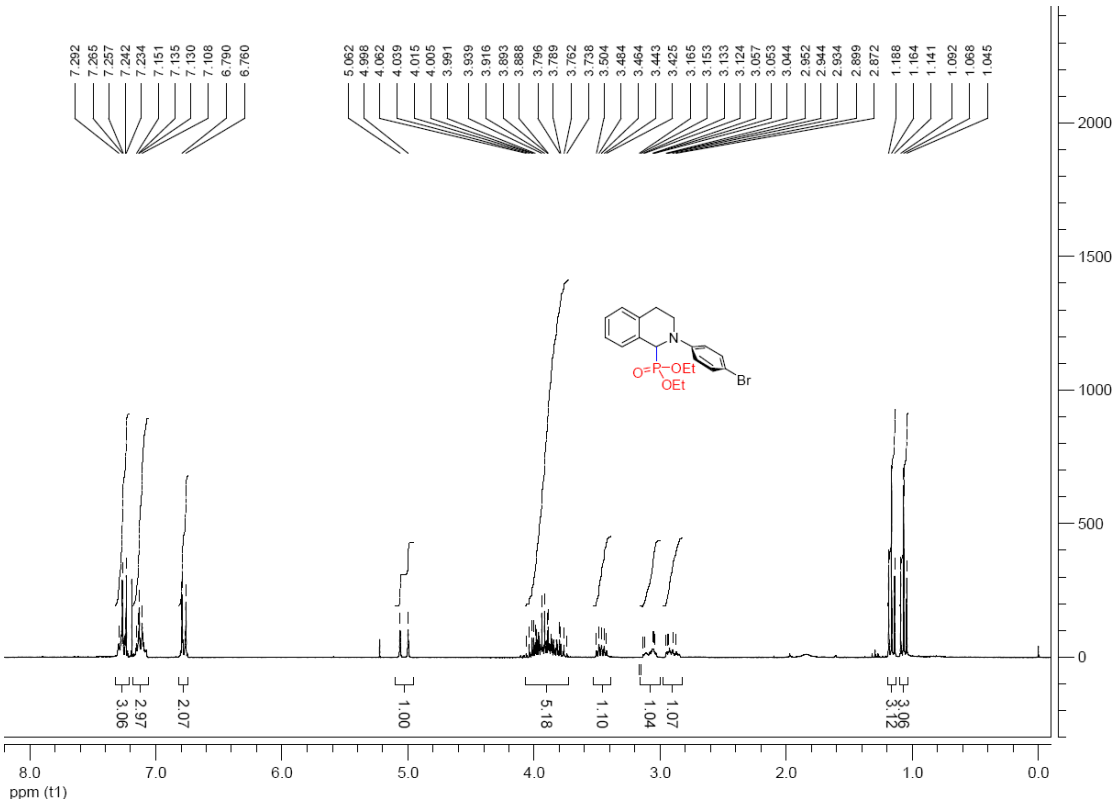
OEt
F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7.135
7.130
7.108
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6.760
5.062
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4.062
4.039
4.015
4.005
3.991
3.939
3.916
3.893
3.888
3.796
3.789
3.762
3.738
3.504
3.484
3.464
3.443
3.425
3.165
3.153
3.133
3.124
3.057
3.053
3.044
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2.944
2.934
2.899
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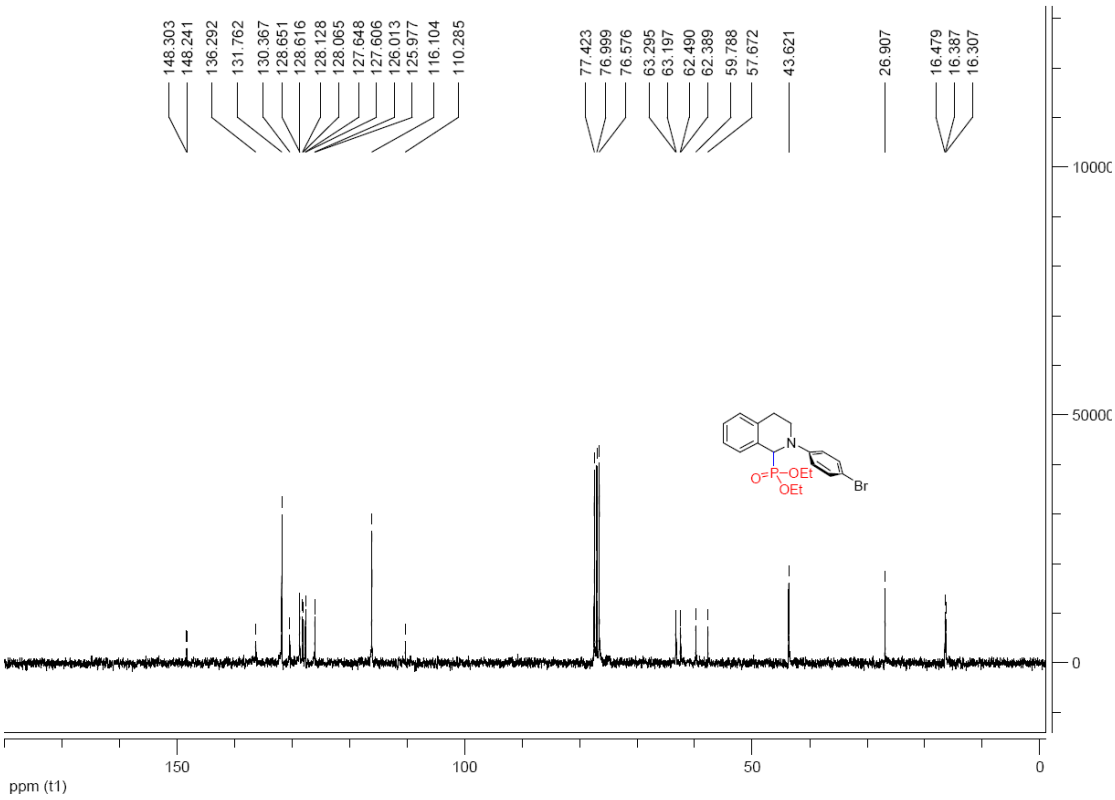
1.188
1.164
1.141
1.092
1.068
1.045
2000
1500
1000
500
0
3.06
2.97
2.07
1.00
5.18
1.10
1.04
1.07
3.06
3.12
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)
O=P-OEt
OEt
Br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130.367
128.651
128.616
128.128
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127.606
126.013
125.977
116.104
110.285
77.423
76.999
76.576
63.295
63.197
62.490
62.389
59.788
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26.907
16.479
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16.307
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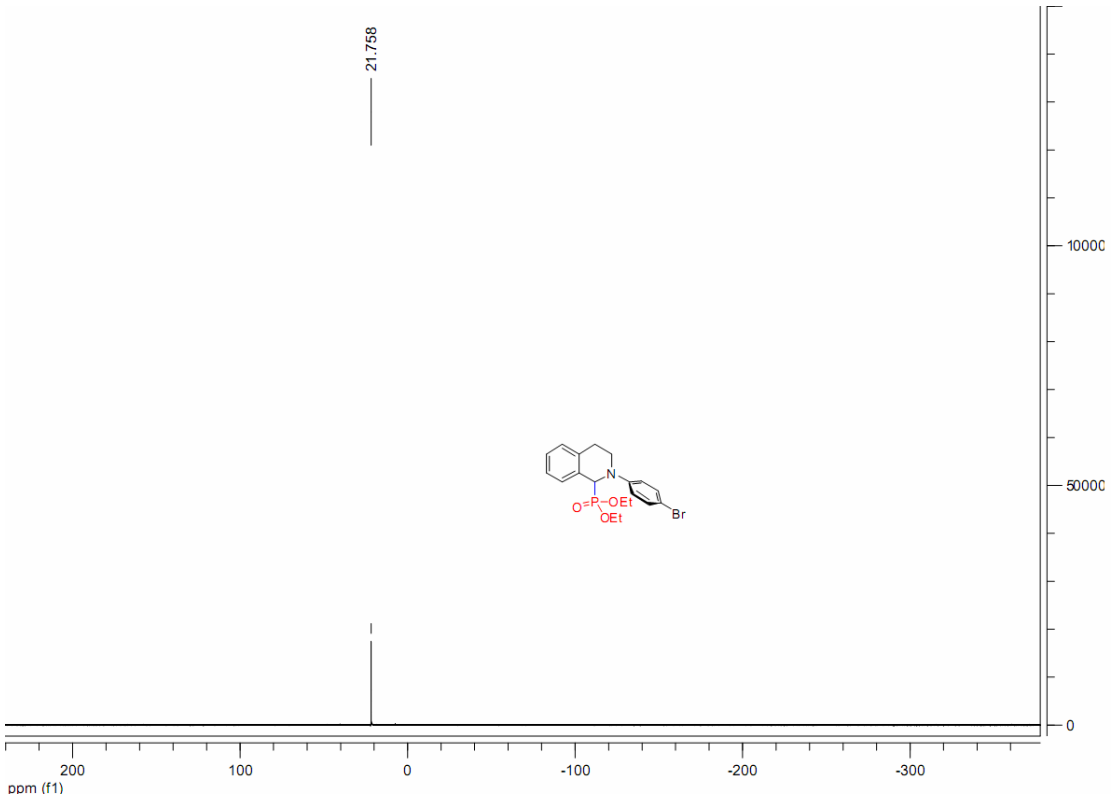
50
0
ppm (t1)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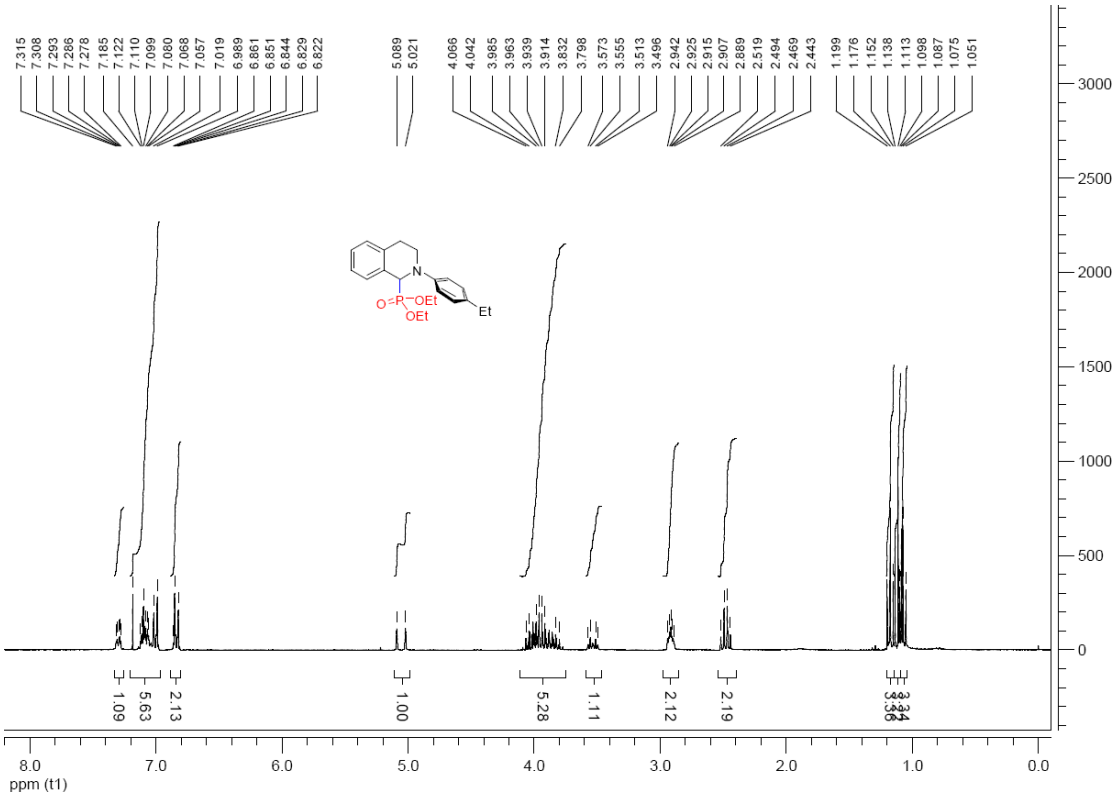
ppm (f1)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7.057
7.019
6.989
6.861
6.851
6.844
6.829
6.822
5.089
5.021
4.066
4.042
3.985
3.963
3.939
3.914
3.832
3.798
3.573
3.555
3.513
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2.494
2.469
2.443
1.199
1.176
1.152
1.138
1.113
1.098
1.087
1.075
1.051
O=P(OEt)OEt
Et
1.09
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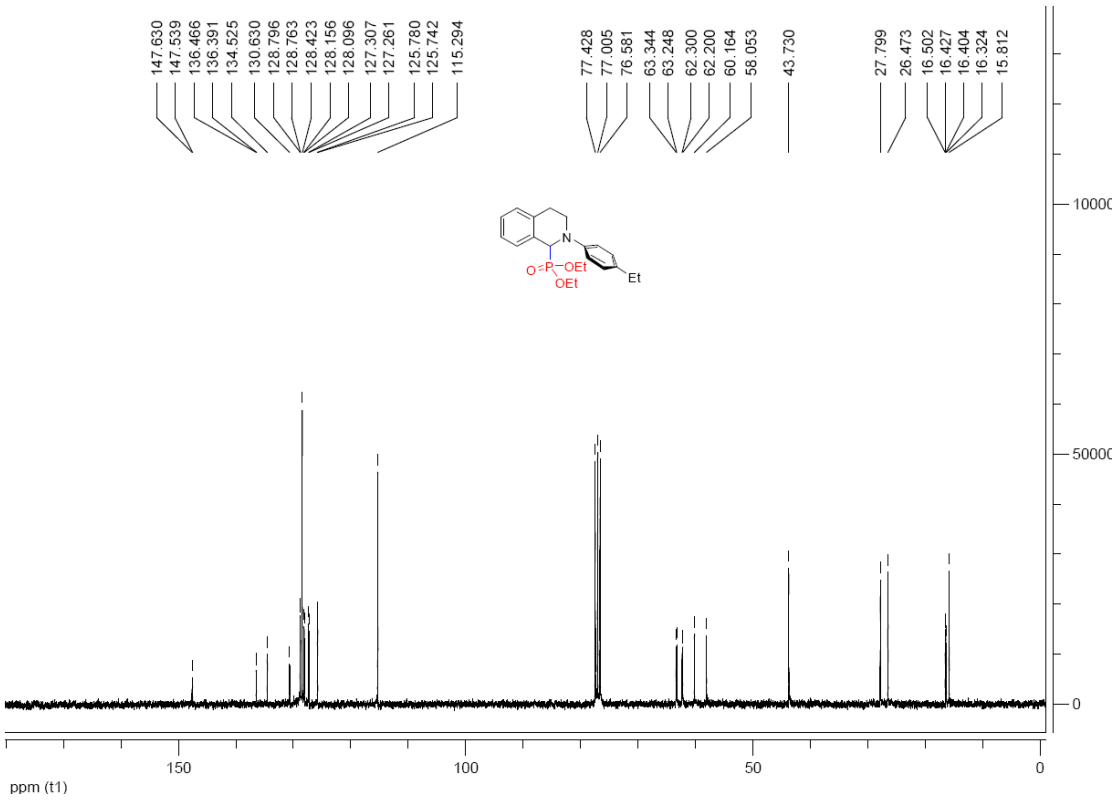
2.13
1.00
5.28
1.11
2.12
2.19
3.34
3.36
ppm (t1)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136.391
134.525
130.630
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128.763
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127.307
127.261
125.780
125.742
115.294
77.428
77.005
76.581
63.344
63.248
62.300
62.200
60.164
58.053
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16.404
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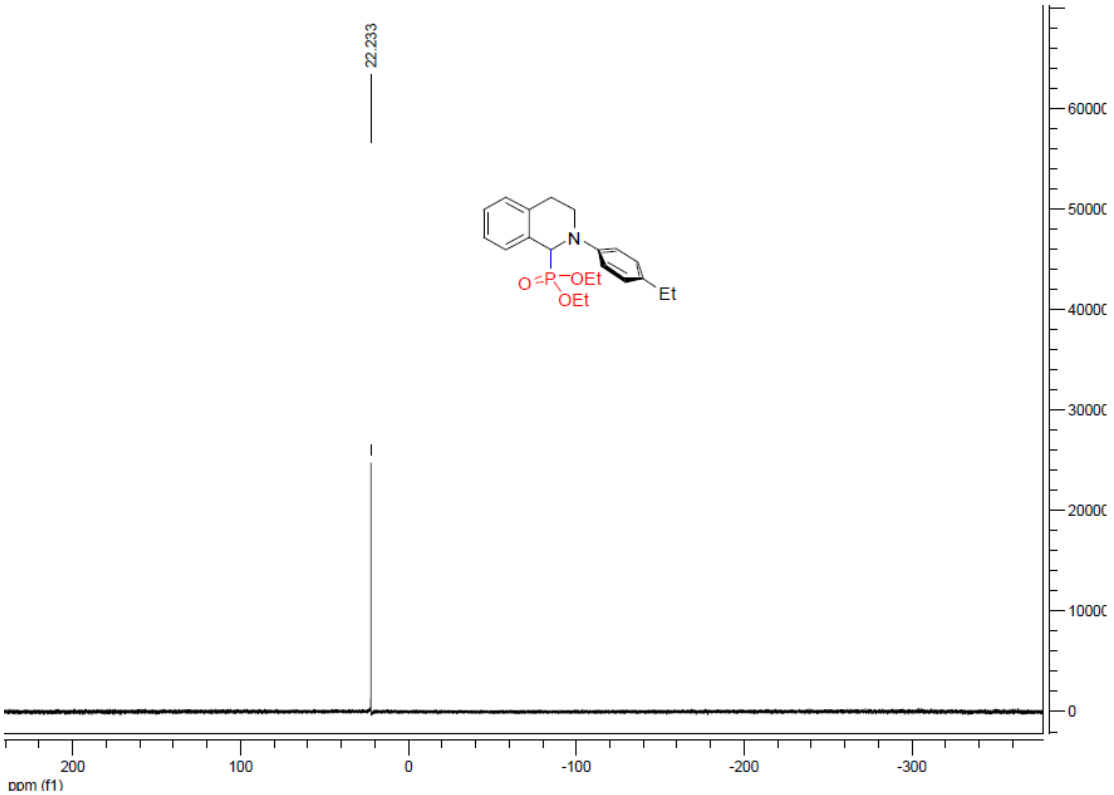
O=P(OEt)OEt
Et
ppm (t1)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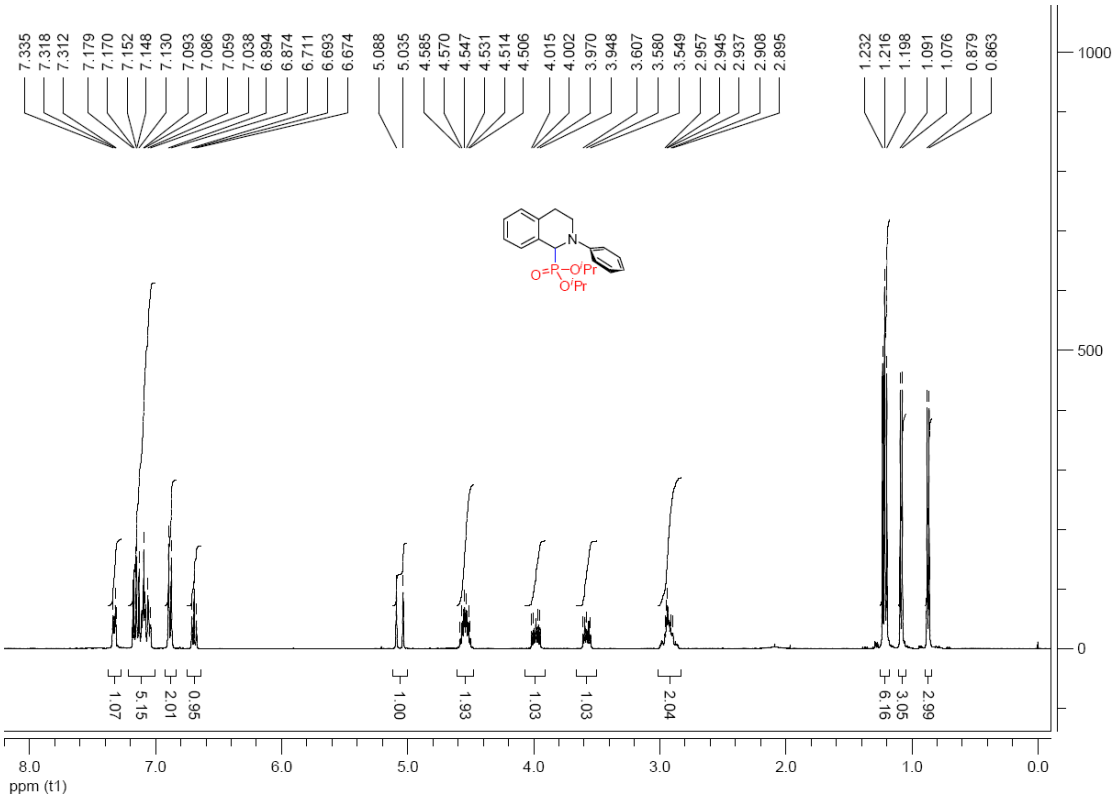
OEt
N
Et
ppm (f1)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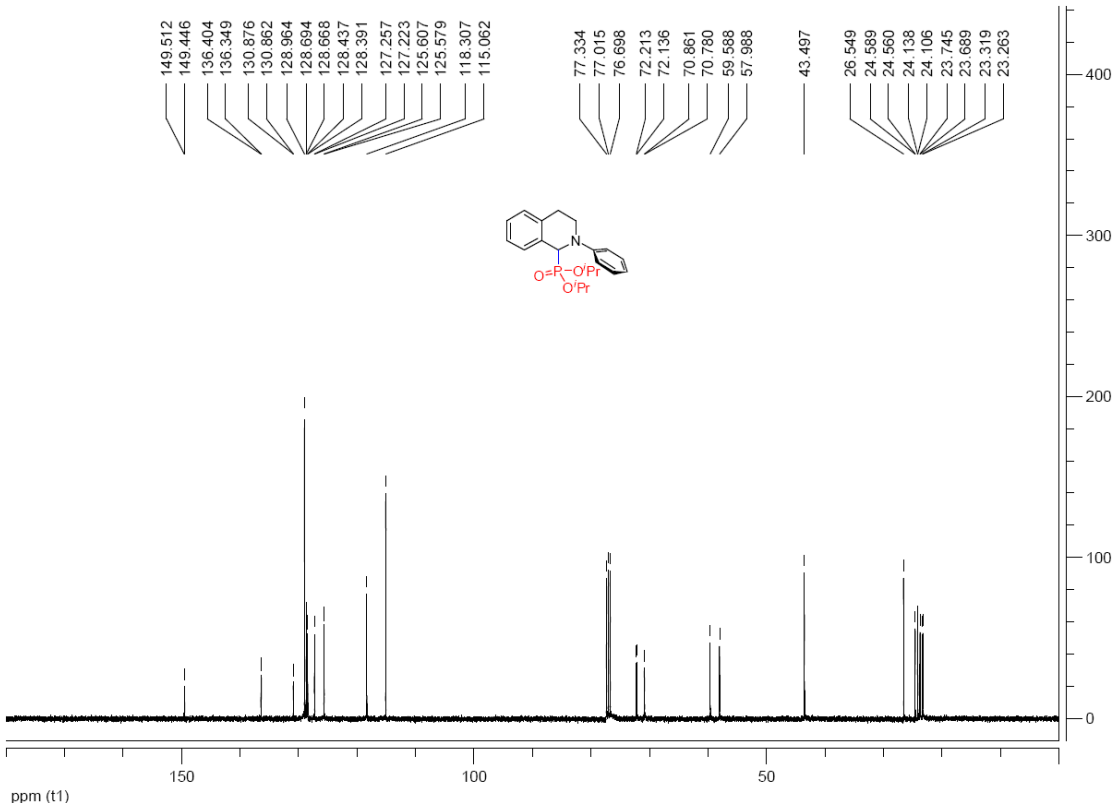
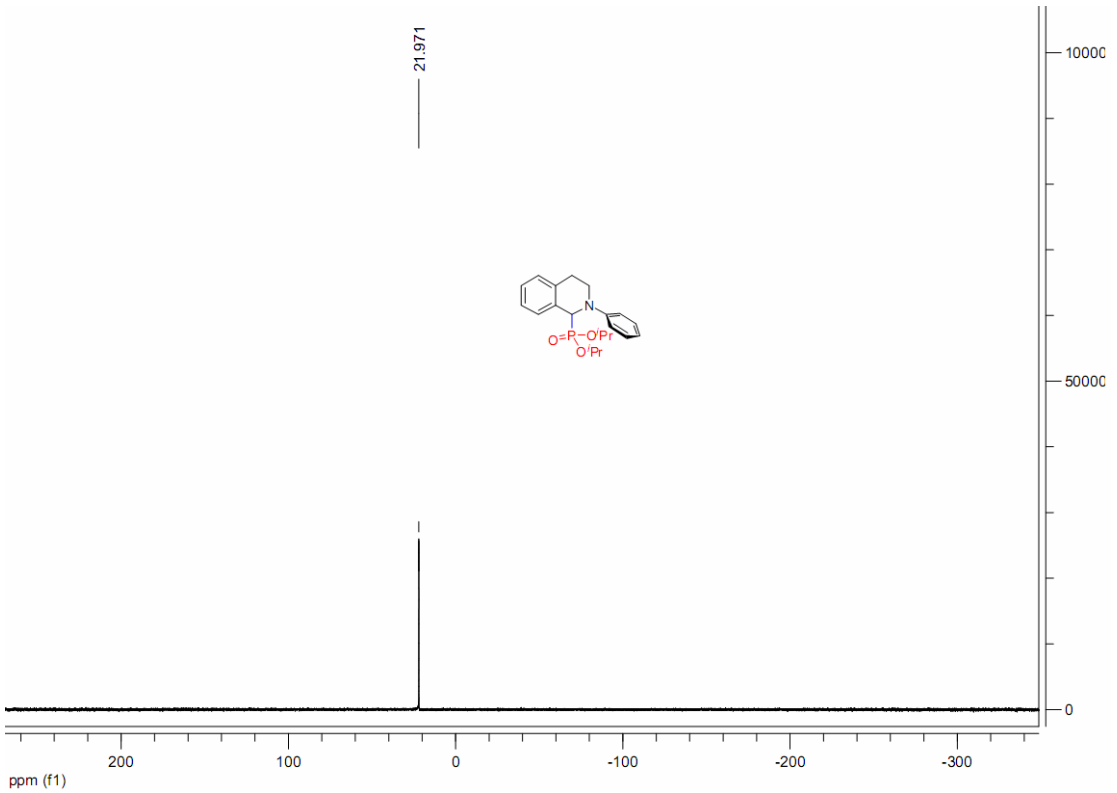
7.318
7.312
7.179
7.170
7.152
7.148
7.130
7.093
7.086
7.059
7.038
6.894
6.874
6.711
6.693
6.674
5.088
5.035
4.585
4.570
4.547
4.531
4.514
4.506
4.015
4.002
3.970
3.948
3.607
3.580
3.549
2.957
2.945
2.937
2.908
2.895
1.232
1.216
1.198
1.091
1.076
0.879
0.863
O=P–O'Pr
O'Pr
1.07
5.15
2.01
0.95
1.00
1.93
1.03
1.03
2.04
6.16
3.05
2.99
ppm (t1)


149.512
149.446
136.404
136.349
130.876
130.862
128.964
128.694
128.668
128.437
128.391
127.257
127.223
125.607
125.579
118.307
115.062
77.334
77.015
76.698
72.213
72.136
70.861
70.780
59.588
57.988
43.497
26.549
24.589
24.560
24.138
24.106
23.745
23.689
23.319
23.263
400
300
200
100
0
150
100
50
ppm (t1)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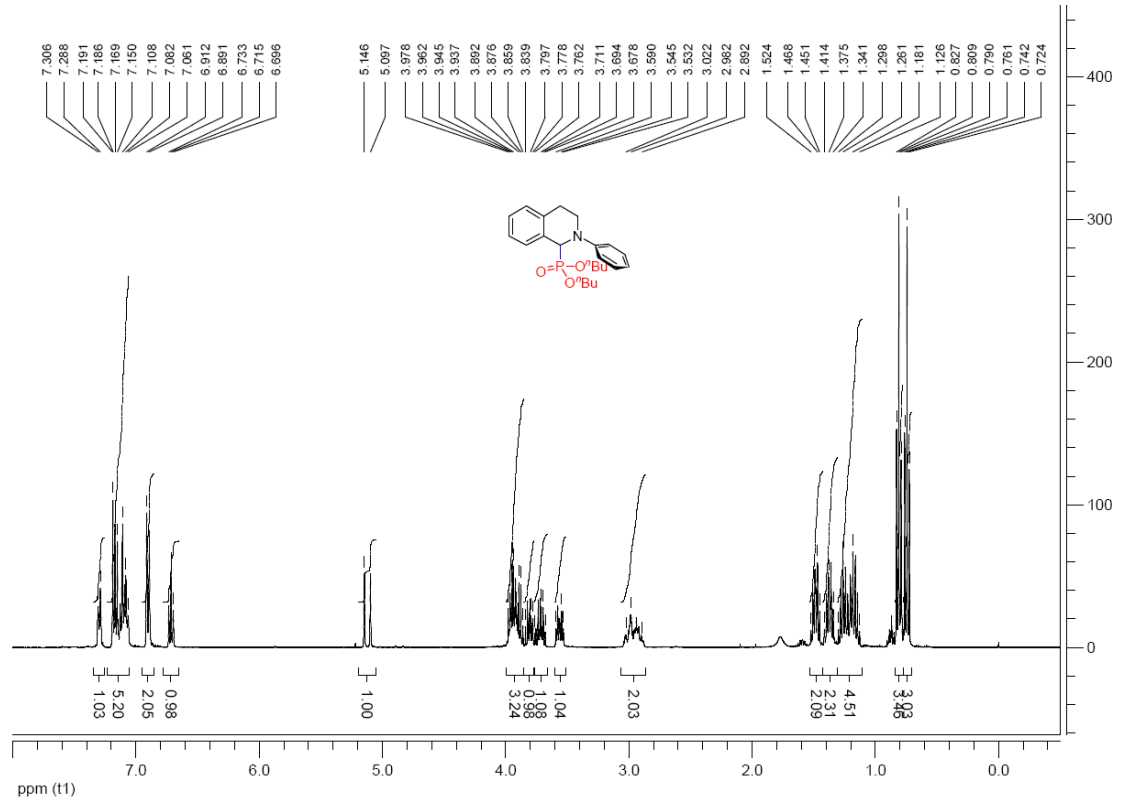
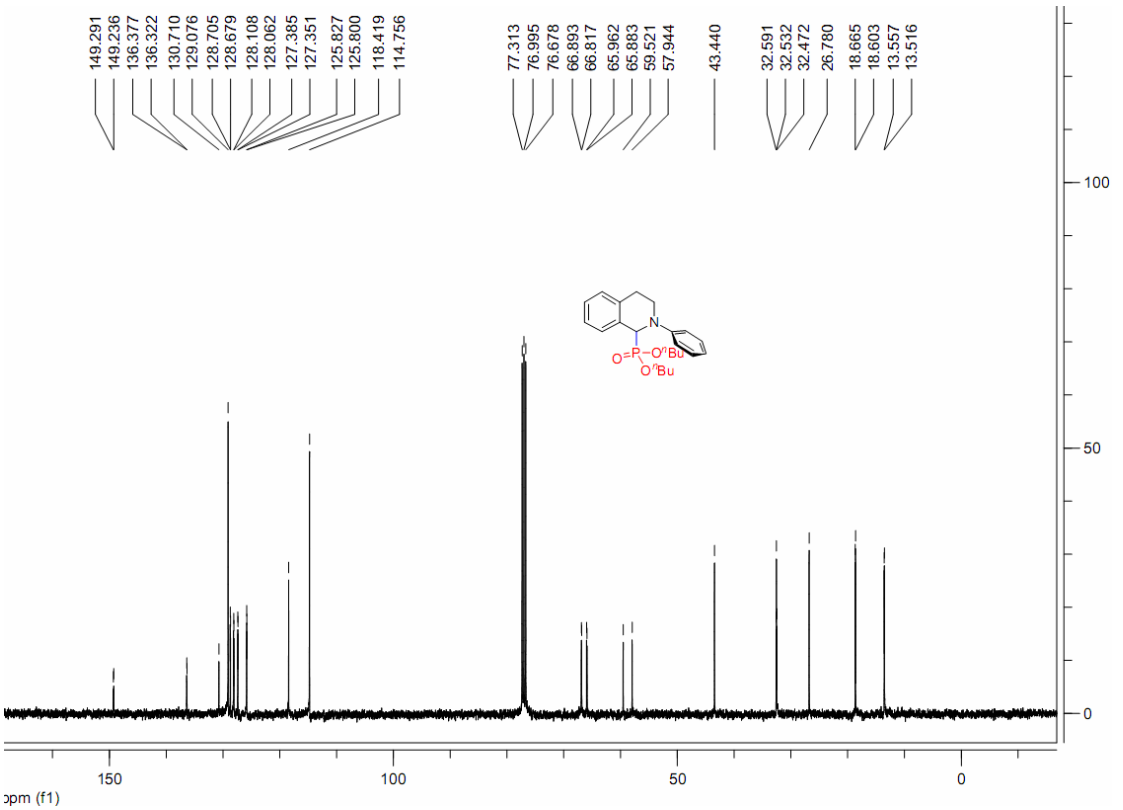
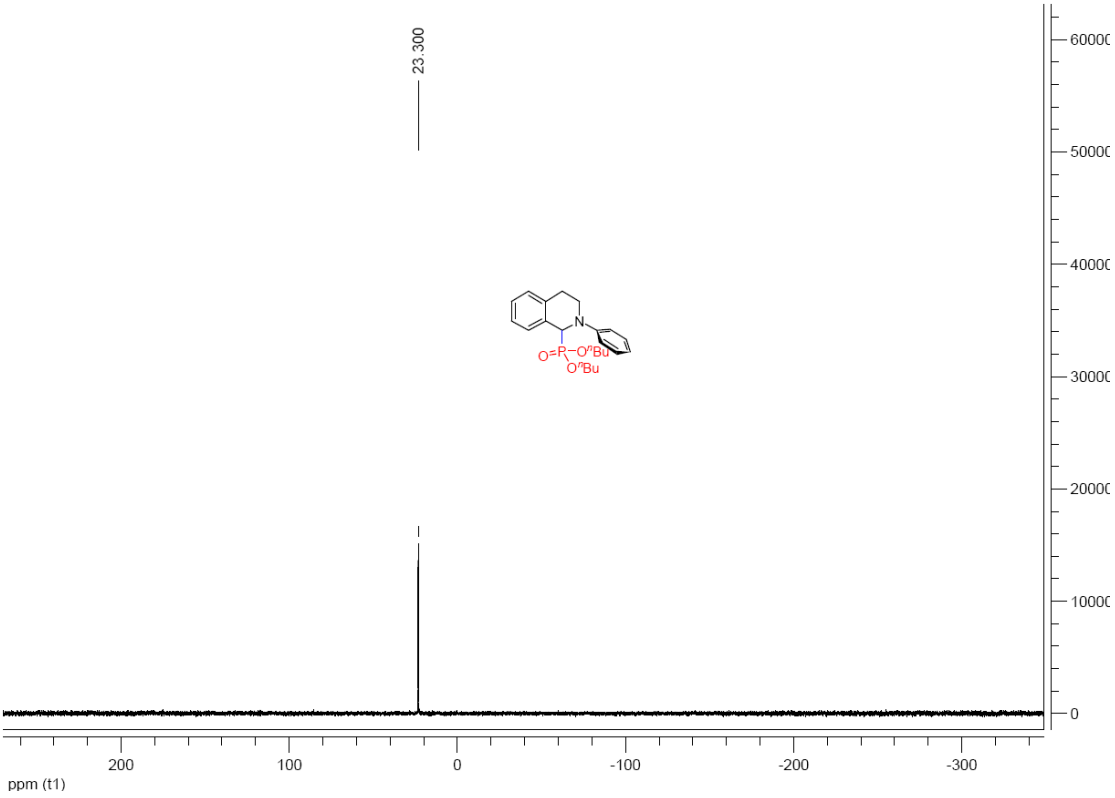
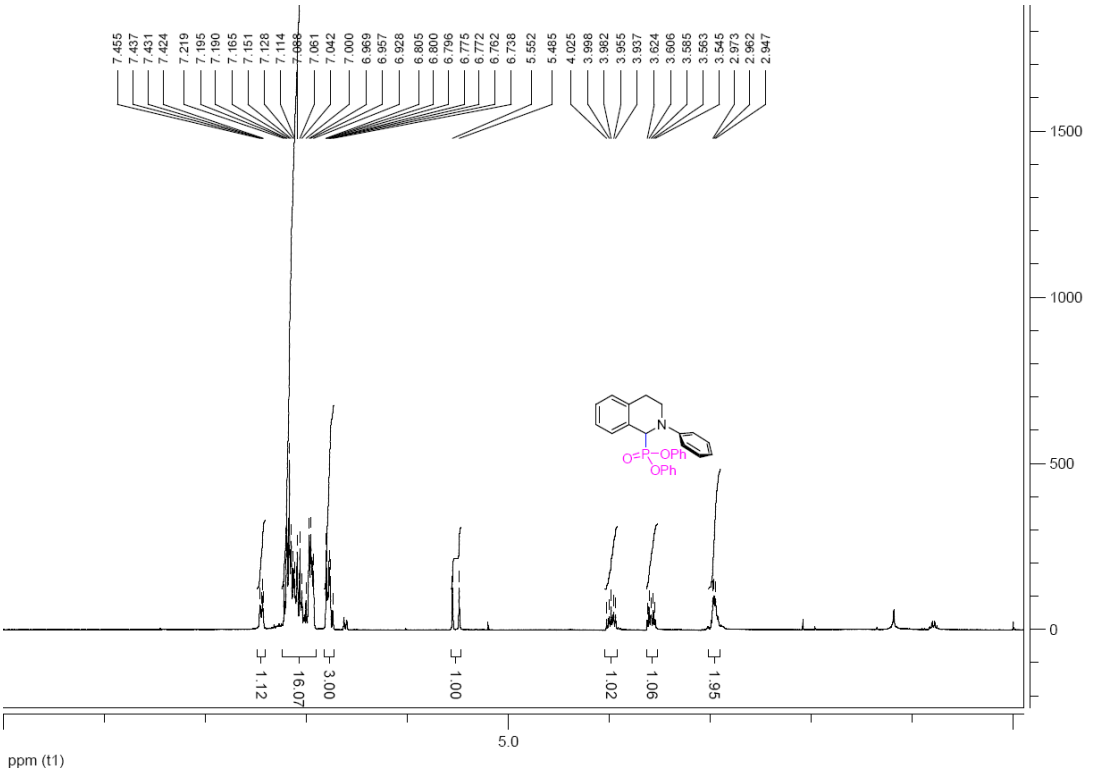
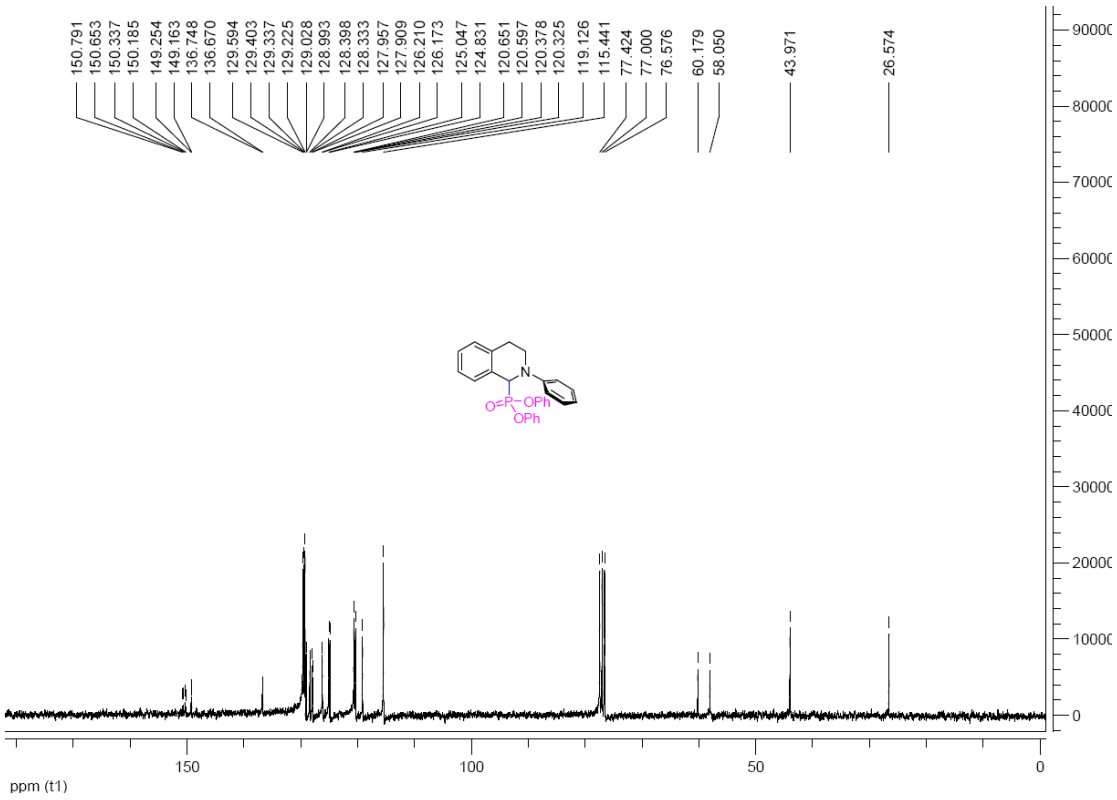
10000
50000
0
200
100
0
-100
-200
-300
ppm (f1)

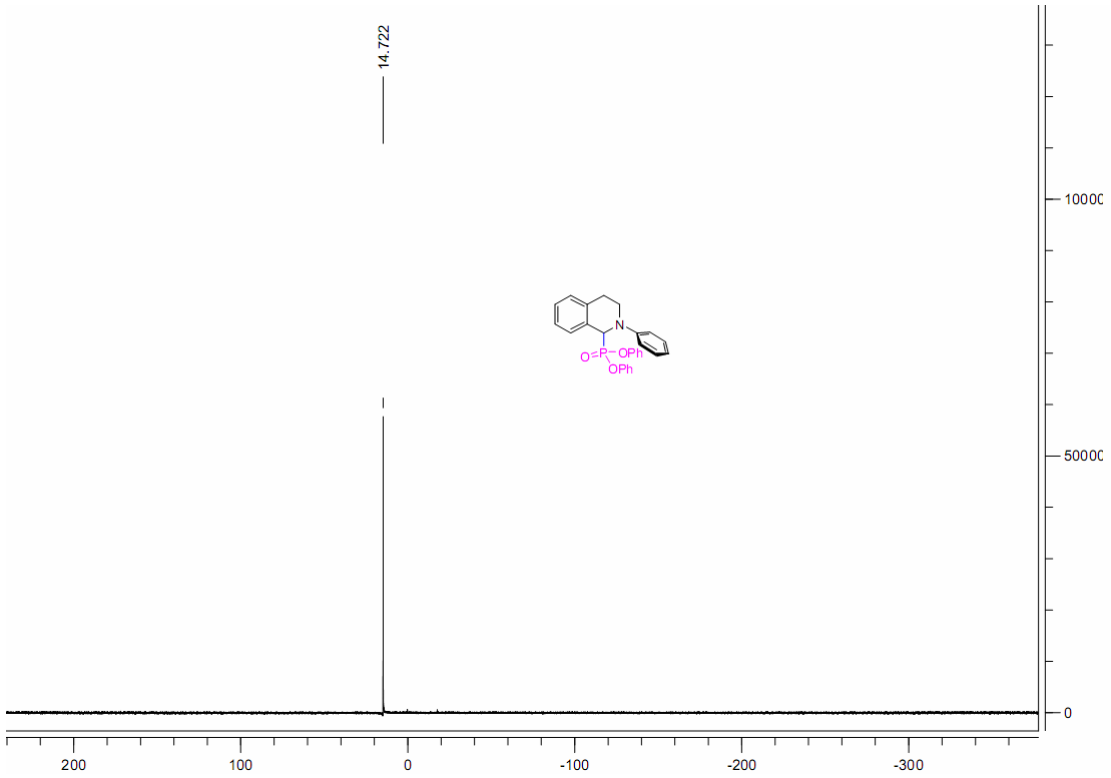

7.306
7.288
7.191
7.186
7.169
7.150
7.108
7.082
7.061
6.912
6.891
6.733
6.715
6.696
5.146
5.097
3.978
3.962
3.945
3.937
3.892
3.876
3.859
3.839
3.797
3.778
3.762
3.711
3.694
3.678
3.590
3.545
3.532
3.022
2.982
2.892
1.524
1.468
1.451
1.414
1.375
1.341
1.298
1.261
1.181
1.126
0.827
0.809
0.790
0.761
0.742
0.724
O=P–OnBu
OnBu
1.03
5.20
2.05
0.68
1.00
3.24
1.04
2.03
2.09
2.31
4.51
3.46
ppm (t1)

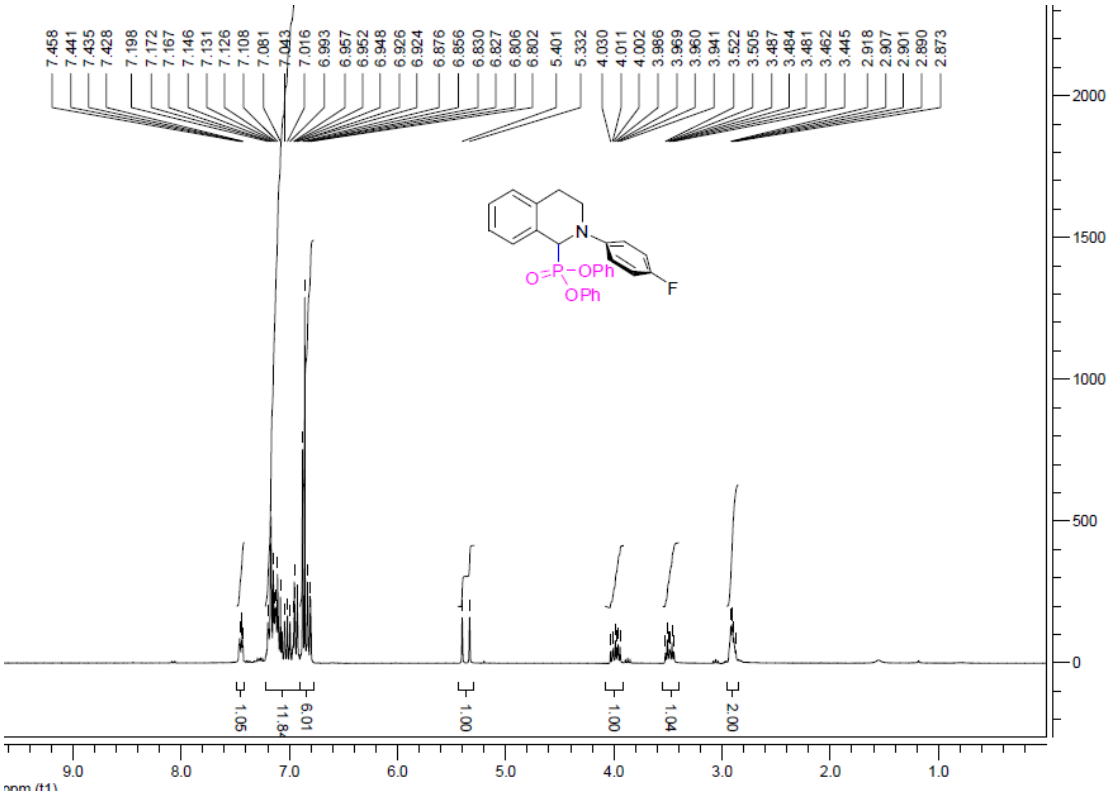

149.291
149.236
136.377
136.322
130.710
129.076
128.705
128.679
128.108
128.062
127.385
127.351
125.827
125.800
118.419
114.756
77.313
76.995
76.678
66.893
66.817
65.962
65.883
59.521
57.944
43.440
32.591
32.532
32.472
26.780
18.665
18.603
13.557
13.516
O=P–OnBu
OnBu
ppm (f1)

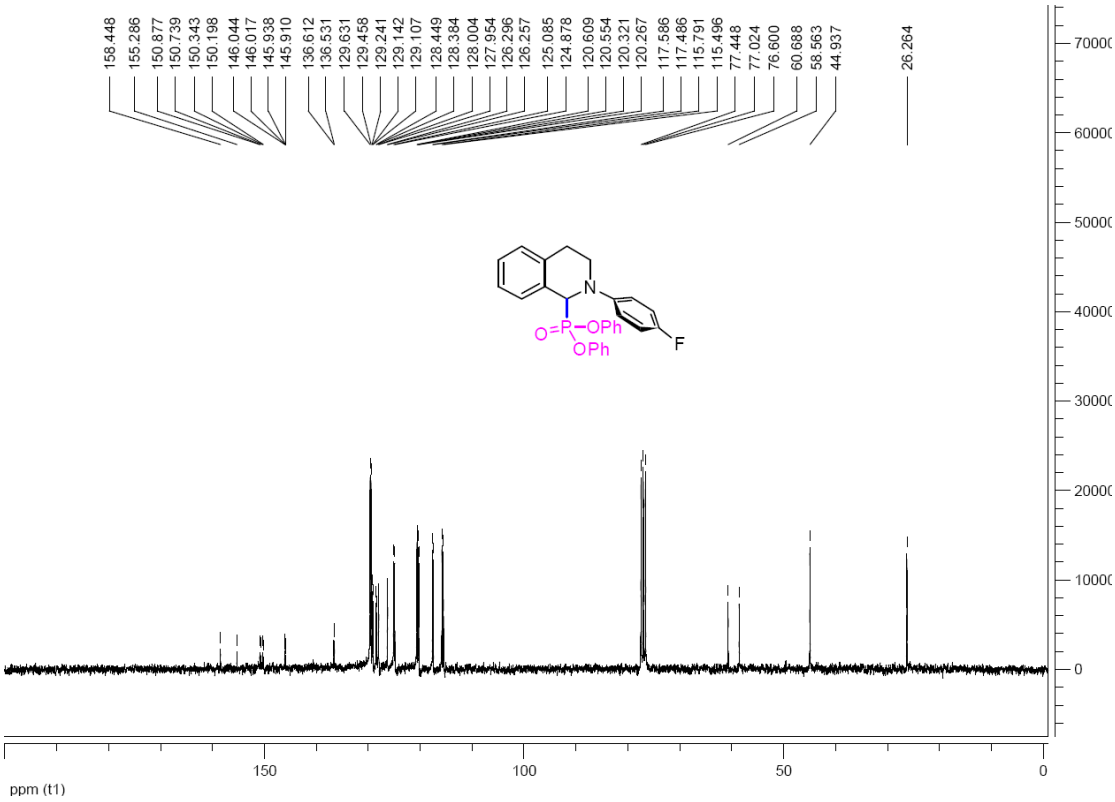

23.300
60000
50000
40000
30000
20000
10000
0
200
100
0
-100
-200
-300
ppm (t1)

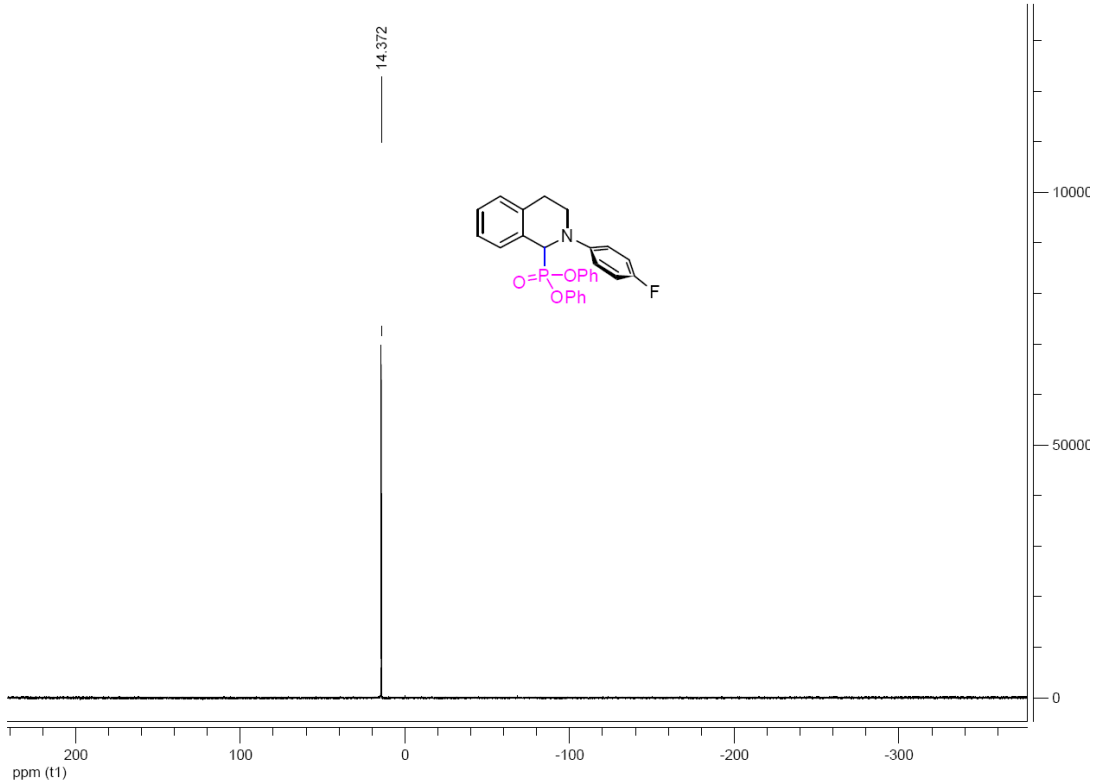

7.455
7.437
7.431
7.424
7.219
7.195
7.190
7.165
7.151
7.128
7.114
7.088
7.061
7.042
7.000
6.969
6.957
6.928
6.805
6.800
6.796
6.775
6.772
6.762
6.738
5.552
5.485
4.025
3.998
3.982
3.955
3.937
3.624
3.606
3.585
3.563
3.545
2.973
2.962
2.947
1500
1000
500
0
1.12
16.07
3.00
1.00
1.02
1.06
1.95
5.0
ppm (t1)

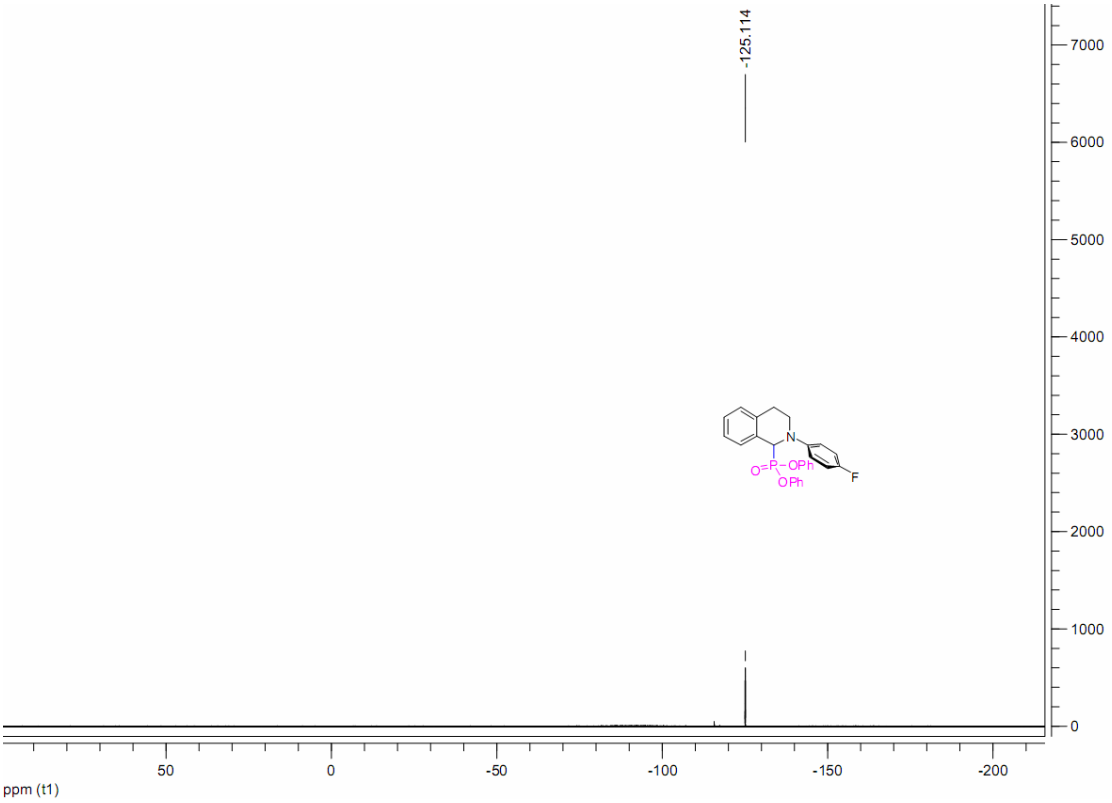

150.791
150.653
150.337
150.185
149.254
149.163
136.748
136.670
129.594
129.403
129.337
129.225
129.028
128.993
128.398
128.333
127.957
127.909
126.210
126.173
125.047
124.831
120.651
120.597
120.378
120.325
119.126
115.441
77.424
77.000
76.576
60.179
58.050
43.971
26.574
O=P-OPh
OPh
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
150
100
50
0
ppm (t1)

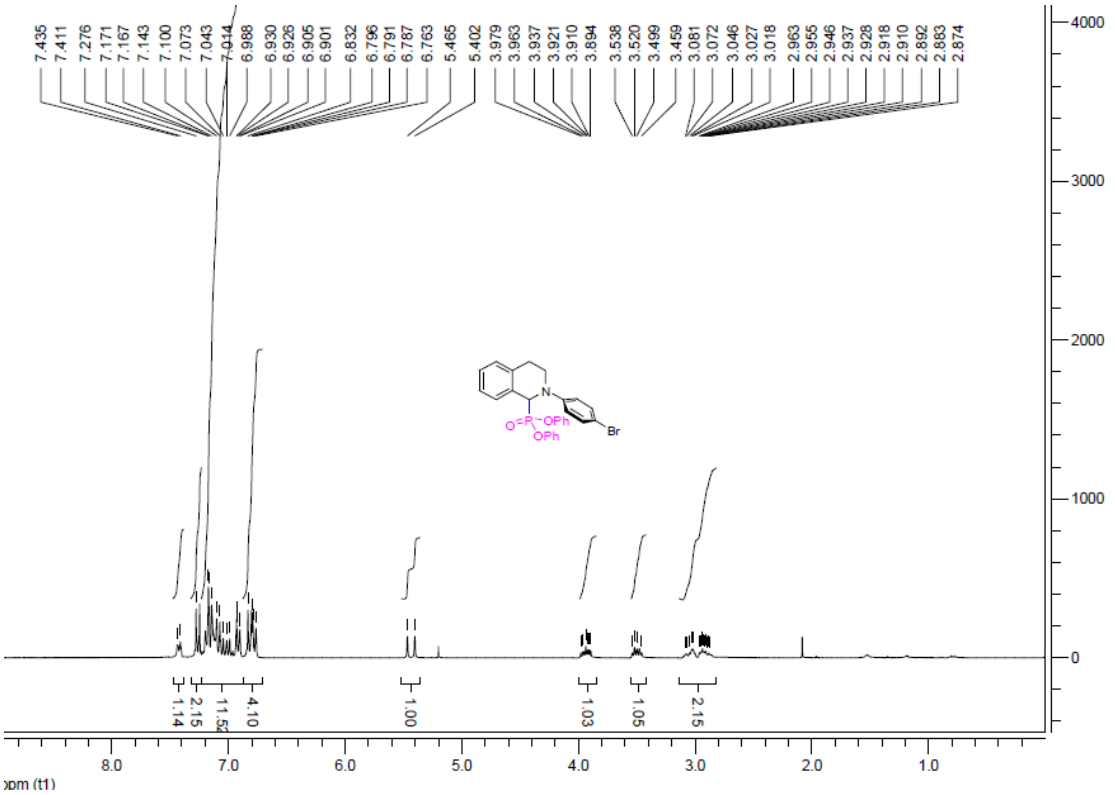

14.722
O=P-OPh
OPh
10000
5000
0
200
100
0
-100
-200
-300

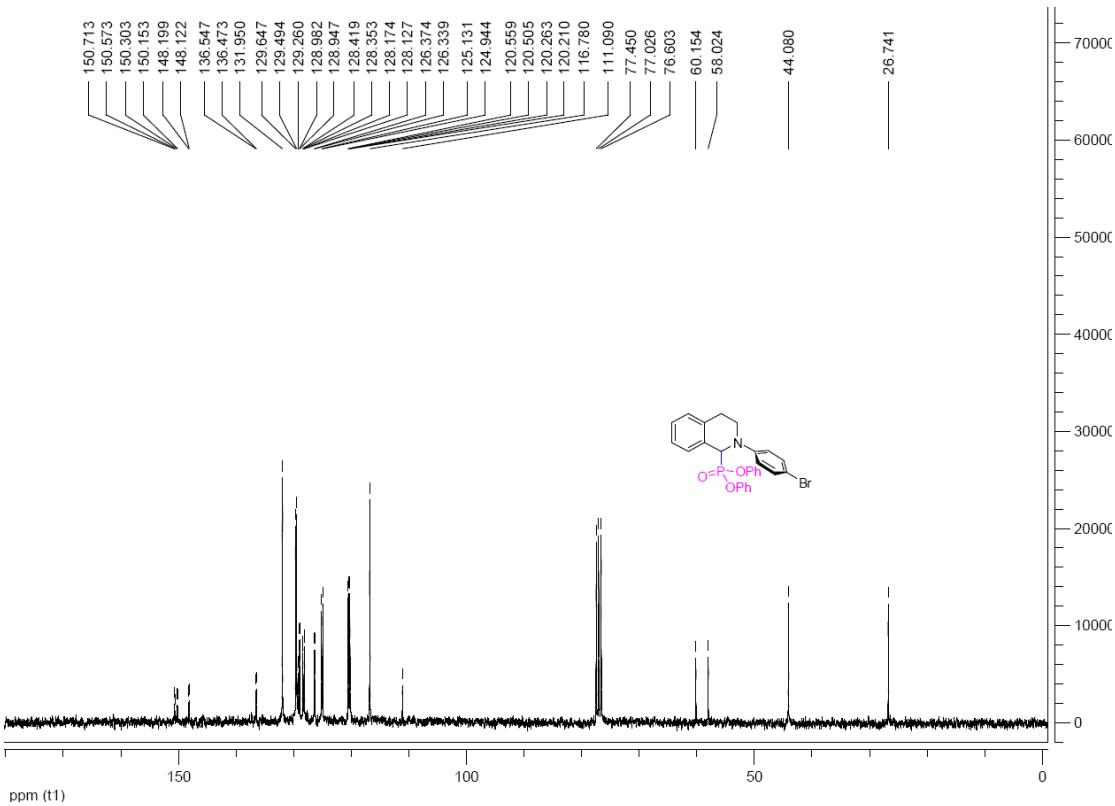

7.458
7.441
7.435
7.428
7.198
7.172
7.167
7.146
7.131
7.126
7.108
7.081
7.043
7.016
6.993
6.957
6.952
6.948
6.926
6.924
6.876
6.856
6.830
6.827
6.806
6.802
5.401
5.332
4.030
4.011
4.002
3.986
3.969
3.960
3.941
3.522
3.505
3.487
3.484
3.481
3.462
3.445
2.918
2.907
2.901
2.890
2.873
N
O=P–OPh
OPh
F
1.05
11.84
6.01
1.00
1.00
1.04
2.00
2000
1500
1000
500
0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0

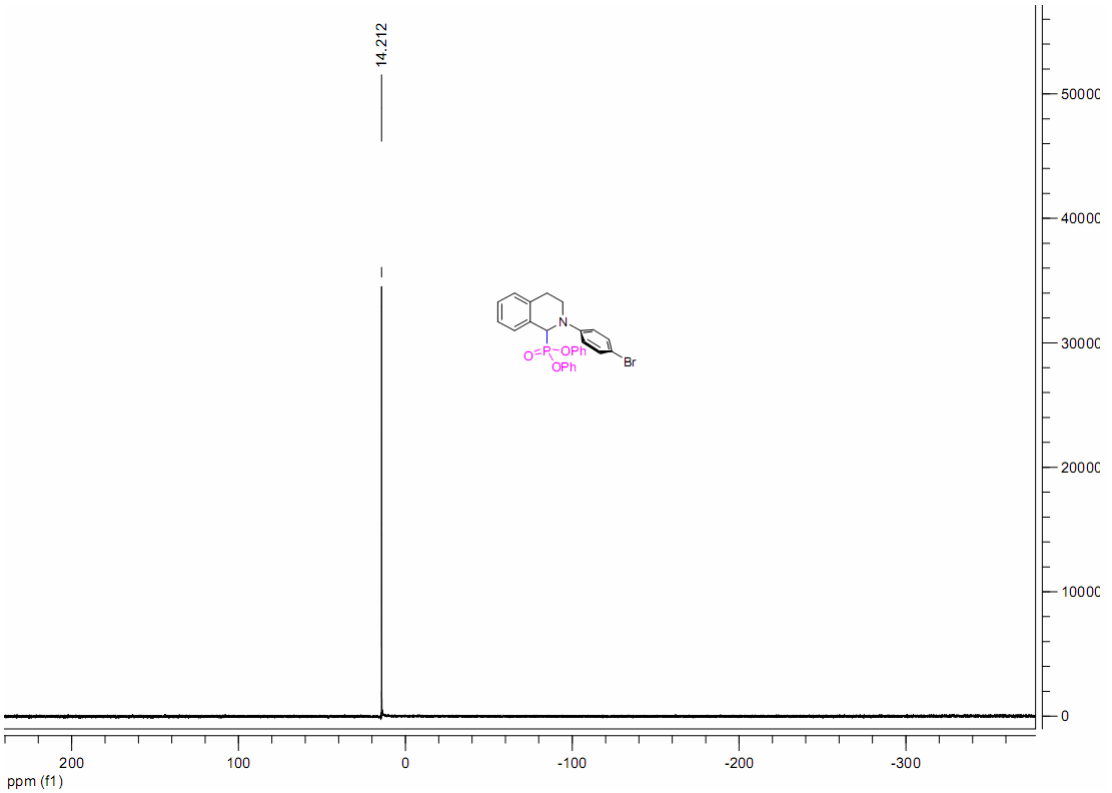

158.448
155.286
150.877
150.739
150.343
150.198
146.044
146.017
145.938
145.910
136.612
136.531
129.631
129.458
129.241
129.142
129.107
128.449
128.384
128.004
127.954
126.296
126.257
125.085
124.878
120.609
120.554
120.321
120.267
117.586
117.486
115.791
115.496
77.448
77.024
76.600
60.688
58.563
44.937
26.264
N
O=P–OPh
OPh
F
70000
60000
50000
40000
30000
20000
10000
0
150
100
50
0
ppm (t1)

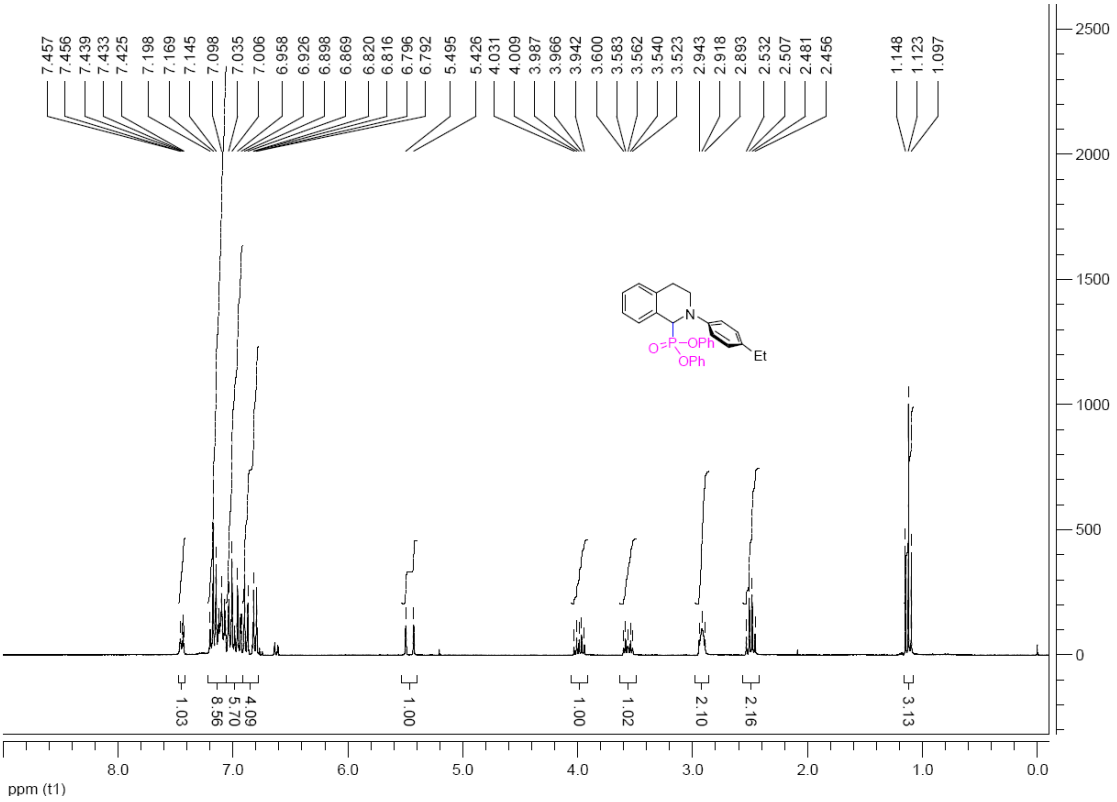

14.372
10000
50000
0
200
100
0
-100
-200
-300
ppm (t1)
N
O=P–OPh
OPh
F

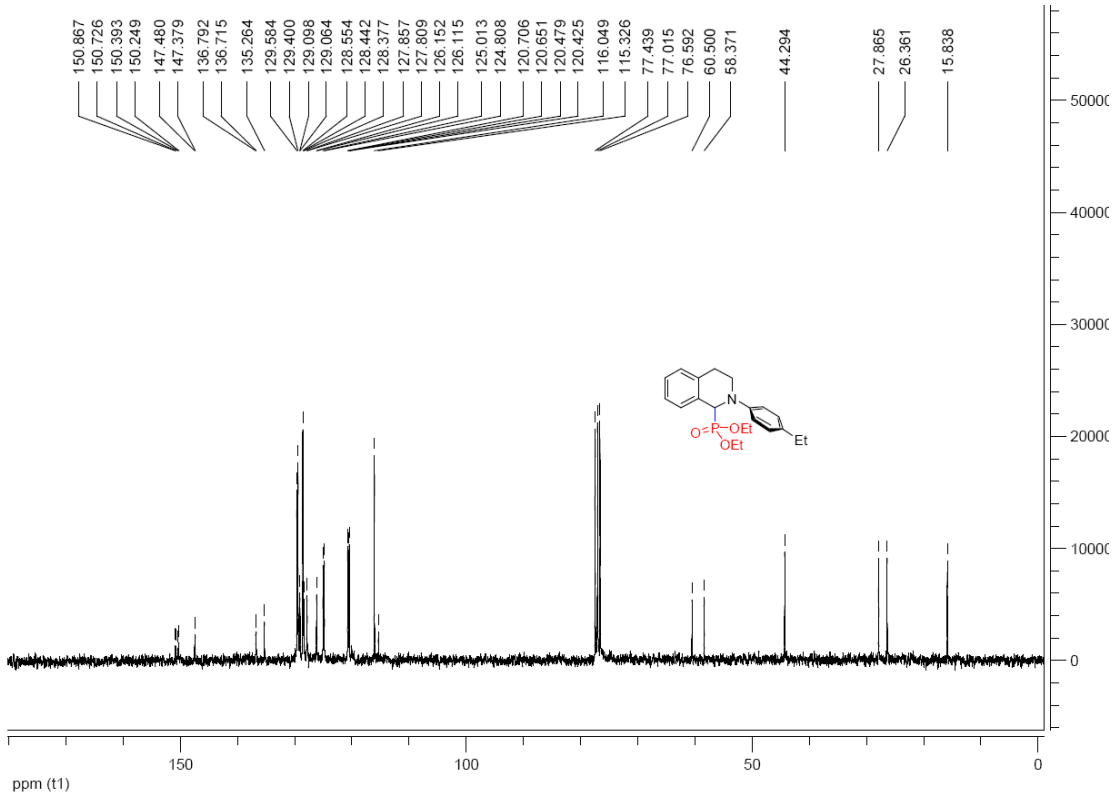

-125.114
7000
6000
5000
4000
3000
2000
1000
0
50
0
-50
-100
-150
-200
ppm (t1)
N
O=P–OPh
OPh
F

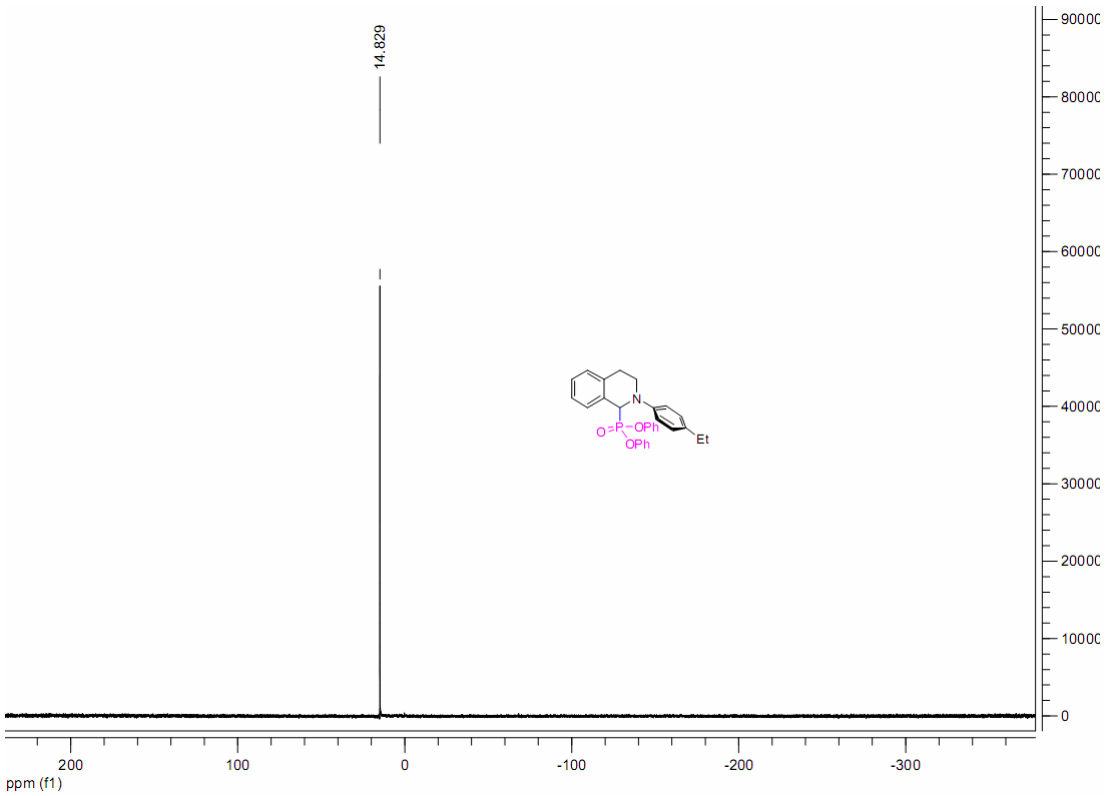

7.435
7.411
7.276
7.171
7.167
7.143
7.100
7.073
7.043
7.014
6.988
6.930
6.926
6.905
6.901
6.832
6.796
6.791
6.787
6.763
5.465
5.402
3.979
3.963
3.937
3.921
3.910
3.894
3.538
3.520
3.499
3.459
3.081
3.072
3.046
3.027
3.018
2.963
2.955
2.946
2.937
2.928
2.918
2.910
2.892
2.883
2.874
4000
3000
2000
1000
0
1.14
2.15
11.52
4.10
1.00
1.03
1.05
2.15
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)
N
O=P
OPh
OPh
Br

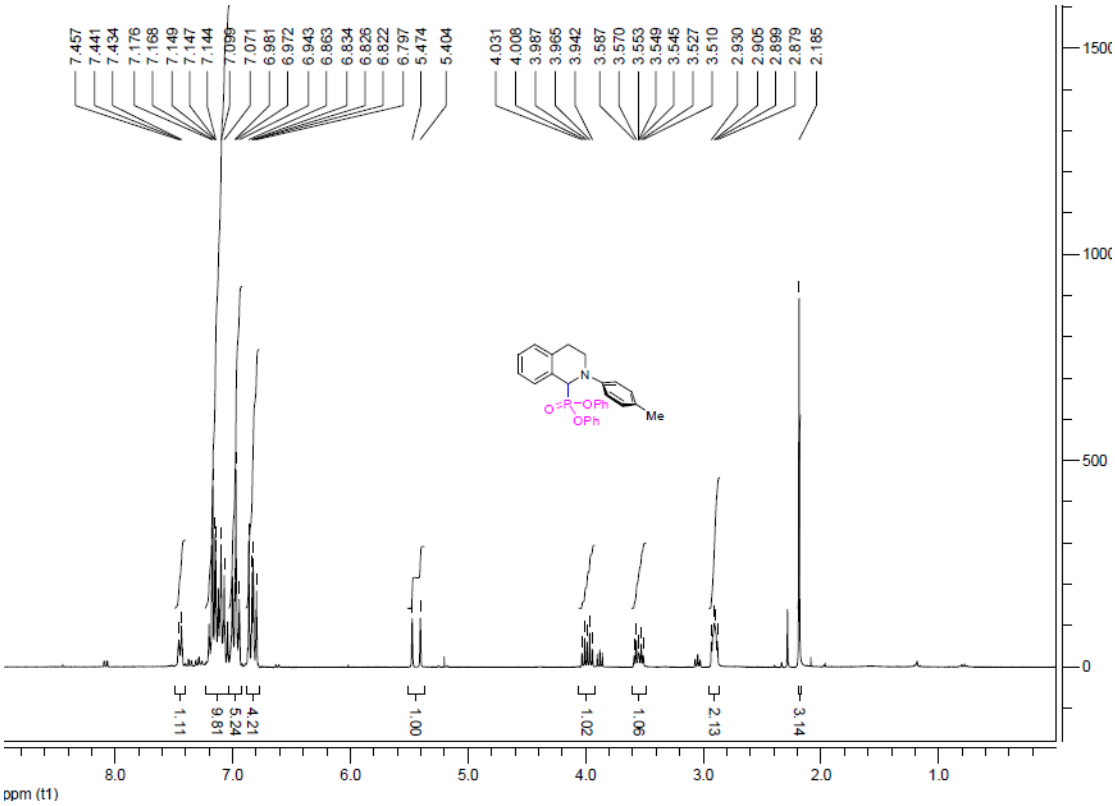

150.713
150.573
150.303
150.153
148.199
148.122
136.547
136.473
131.950
129.647
129.494
129.260
128.982
128.947
128.419
128.353
128.174
128.127
126.374
126.339
125.131
124.944
120.559
120.505
120.263
120.210
116.780
111.090
77.450
77.026
76.603
60.154
58.024
44.080
26.741
70000
60000
50000
40000
30000
20000
10000
0
150
100
50
0
ppm (t1)
N
O=P
OPh
OPh
Br

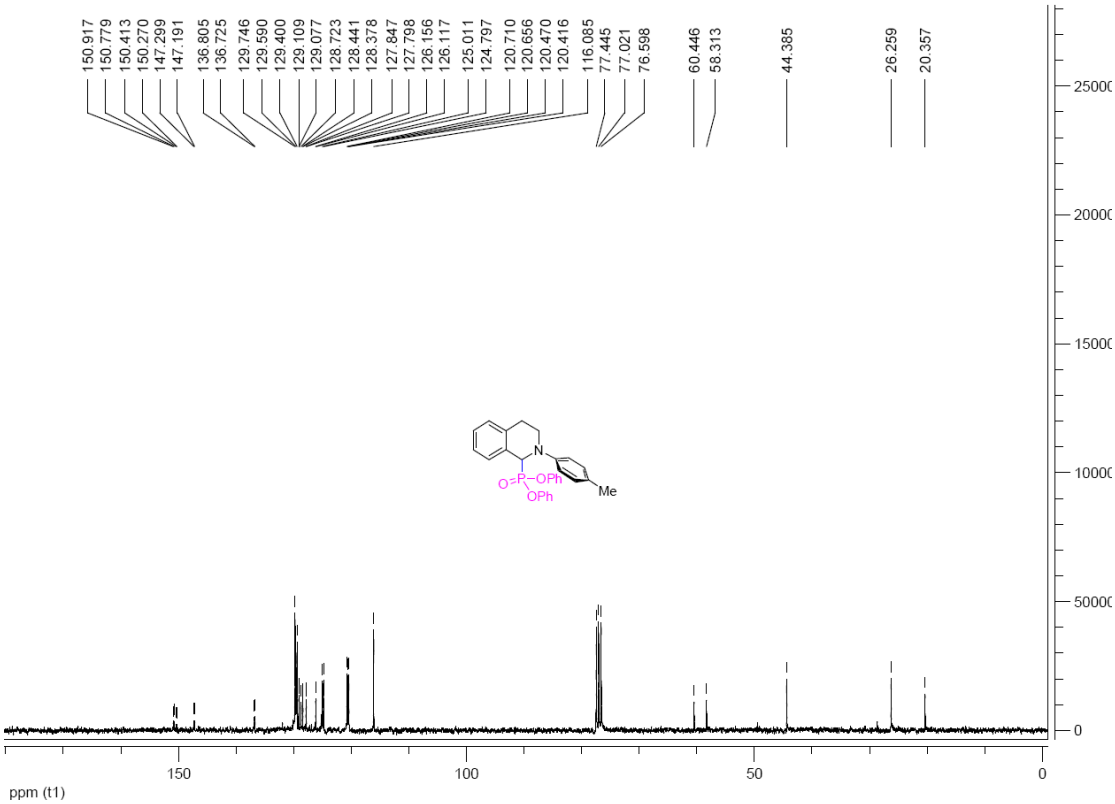

14.212
O=P-OPh
OPh
Br
ppm (f1)

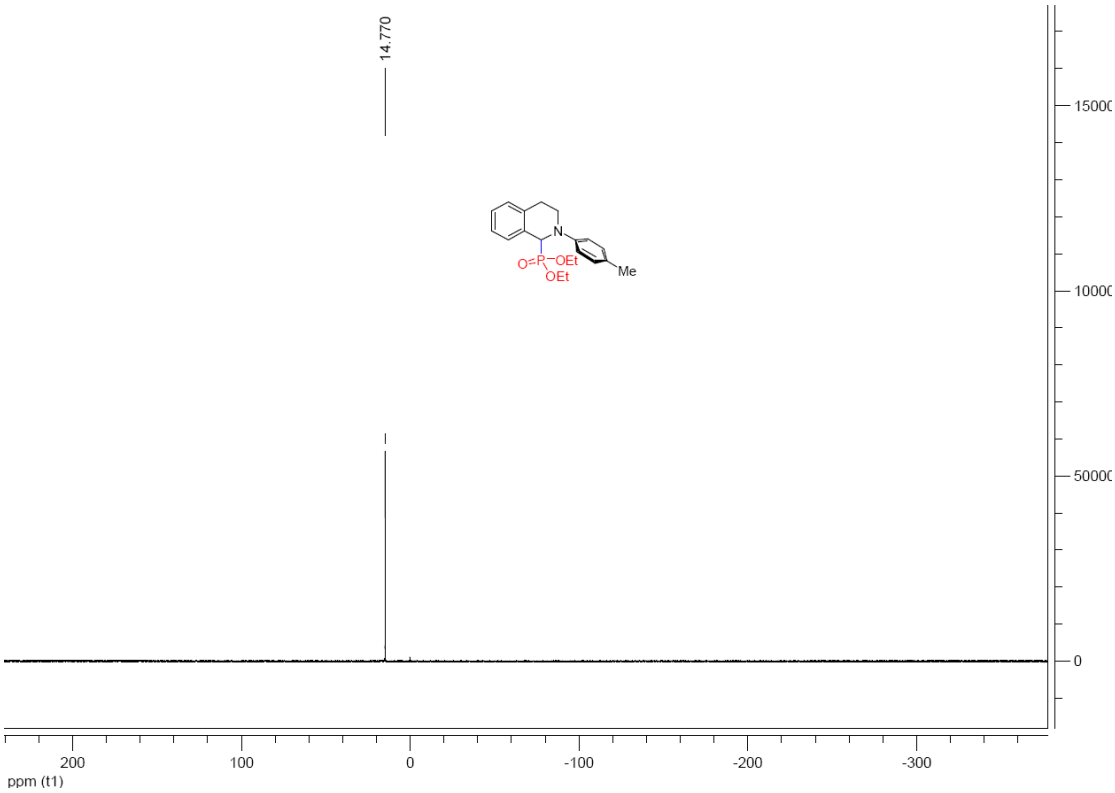

7.457
7.456
7.439
7.433
7.425
7.198
7.169
7.145
7.098
7.035
7.006
6.958
6.926
6.898
6.869
6.820
6.816
6.796
6.792
5.495
5.426
4.031
4.009
3.987
3.966
3.942
3.600
3.583
3.562
3.540
3.523
2.943
2.918
2.893
2.532
2.507
2.481
2.456
1.148
1.123
1.097
O=P-OPh
OPh
Et
1.03
8.56
5.70
4.09
1.00
1.00
1.02
2.10
2.16
3.13
ppm (t1)

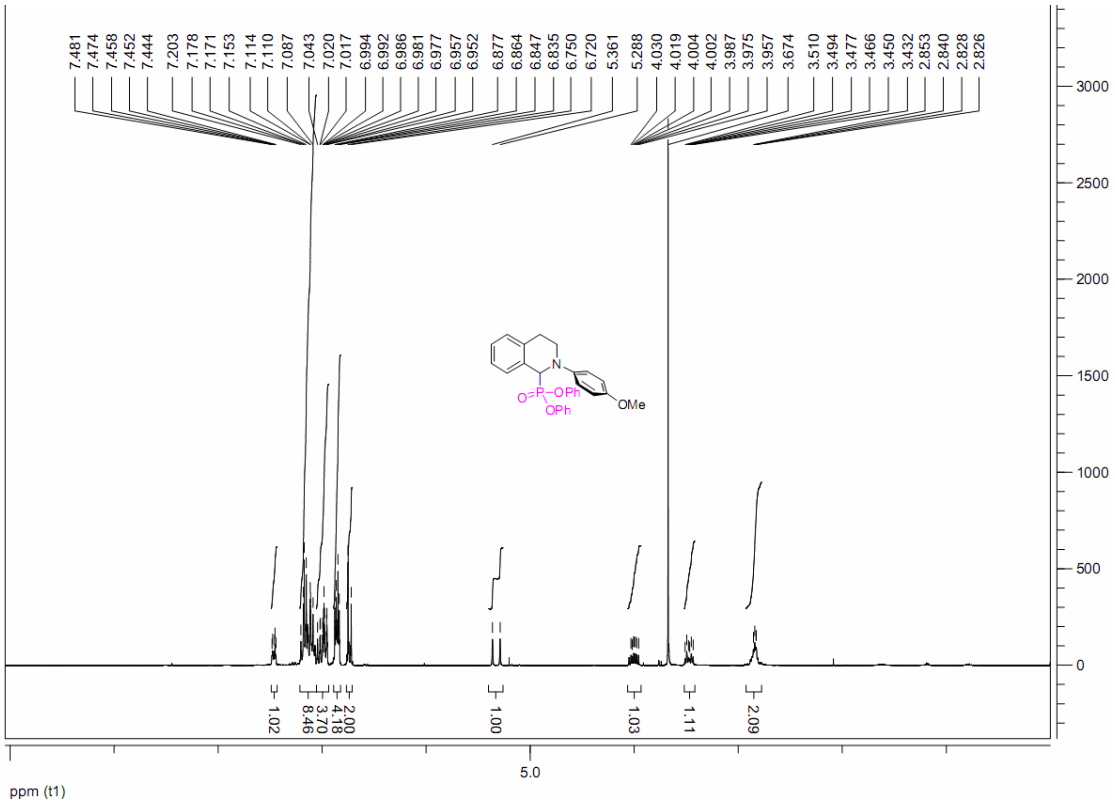

150.867
150.726
150.393
150.249
147.480
147.379
136.792
136.715
135.264
129.584
129.400
129.098
129.064
128.554
128.442
128.377
127.857
127.809
126.152
126.115
125.013
124.808
120.706
120.651
120.479
120.425
116.049
115.326
77.439
77.015
76.592
60.500
58.371
44.294
27.865
26.361
15.838
O=P-OEt
OEt
Et
ppm (t1)

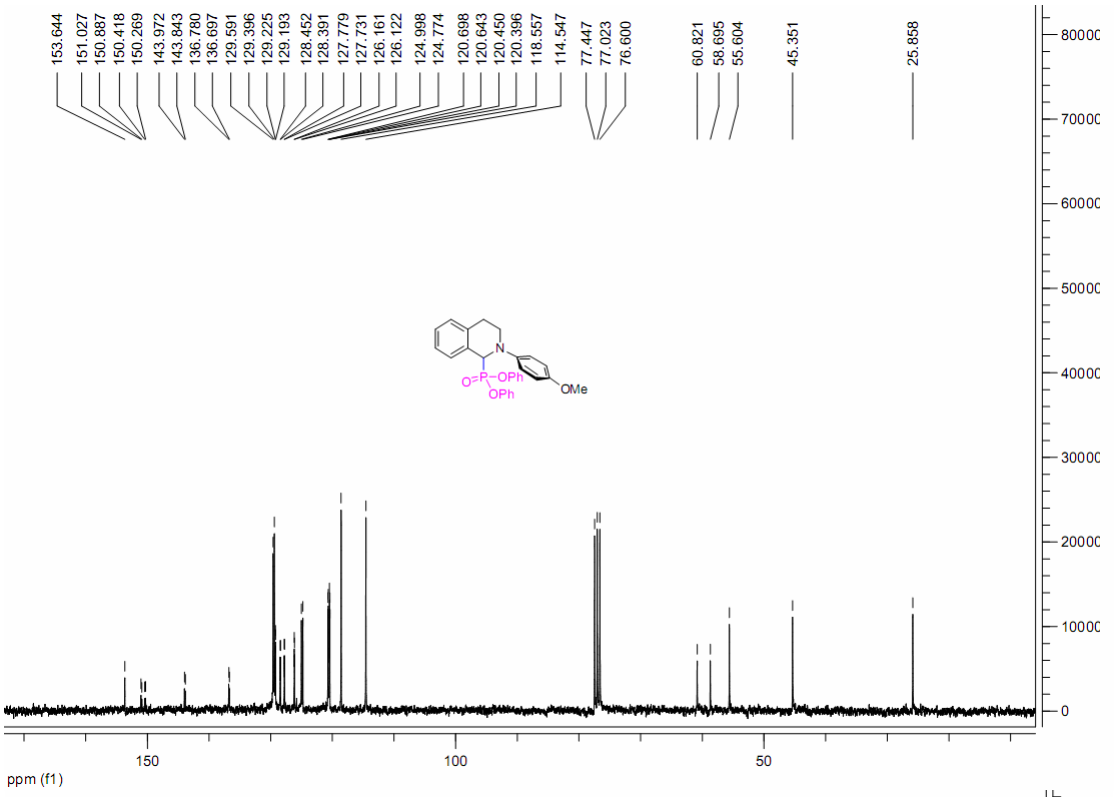

14.829
O=P-OPh
OPh
Et
ppm (f1)

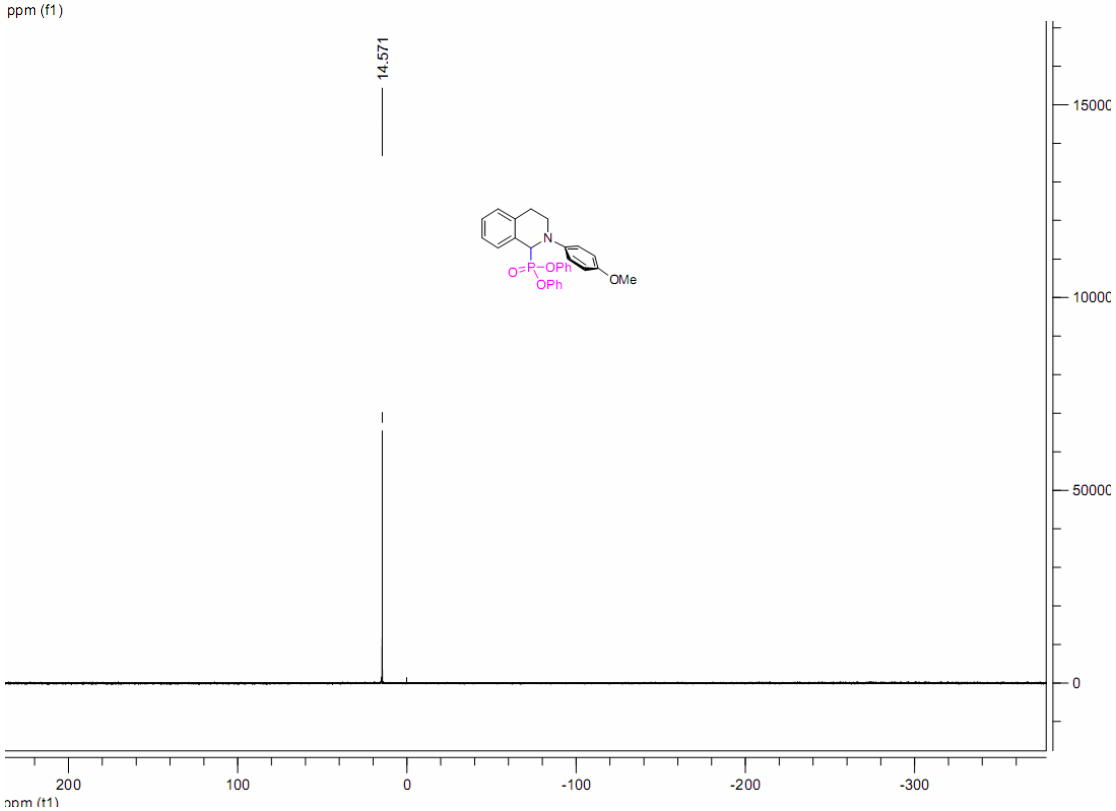

7.457
7.441
7.434
7.176
7.168
7.149
7.147
7.144
7.099
7.071
6.981
6.972
6.943
6.863
6.834
6.826
6.822
6.797
5.474
5.404
4.031
4.008
3.987
3.965
3.942
3.587
3.570
3.553
3.549
3.545
3.527
3.510
2.930
2.905
2.899
2.879
2.185
1.11
9.81
5.42
4.21
1.00
1.02
1.06
2.13
3.14
Me
ppm (t1)

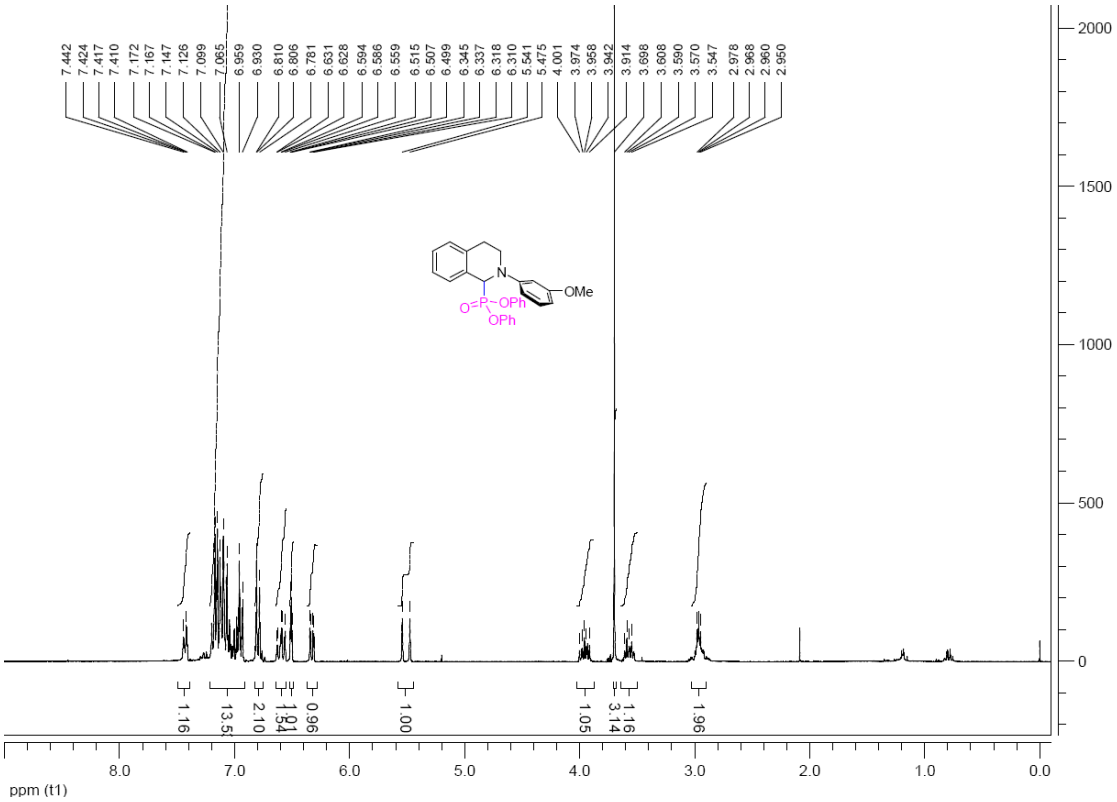

150.917
150.779
150.413
150.270
147.299
147.191
136.805
136.725
129.746
129.590
129.400
129.109
129.077
128.723
128.441
128.378
127.847
127.798
126.156
126.117
125.011
124.797
120.710
120.656
120.470
120.416
116.085
77.445
77.021
76.598
60.446
58.313
44.385
26.259
20.357
Me
ppm (t1)

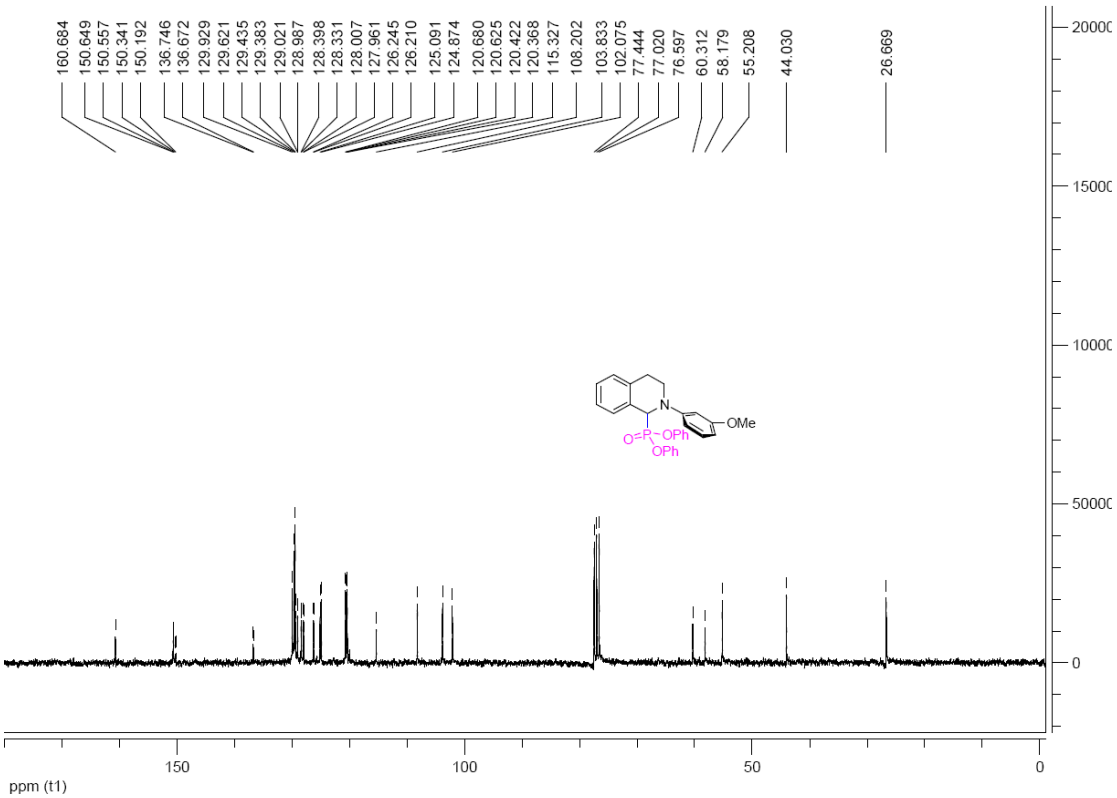

14.770
O=P–OEt
OEt
N
Me
15000
10000
5000
0
200
100
0
-100
-200
-300
ppm (t1)


7.481
7.474
7.458
7.452
7.444
7.203
7.178
7.171
7.153
7.114
7.110
7.087
7.043
7.020
7.017
6.994
6.992
6.986
6.981
6.977
6.957
6.952
6.877
6.864
6.847
6.835
6.750
6.720
5.361
5.288
4.030
4.019
4.004
4.002
3.987
3.975
3.957
3.674
3.510
3.494
3.477
3.466
3.450
3.432
2.853
2.840
2.828
2.826
O=P–OPh
OPh
N
OMe
3000
2500
2000
1500
1000
500
0
1.02
8.46
3.70
4.18
2.00
1.00
1.03
1.11
2.09
5.0
ppm (t1)


153.644
151.027
150.887
150.418
150.269
143.972
143.843
136.780
136.697
129.591
129.396
129.225
129.193
128.452
128.391
127.779
127.731
126.161
126.122
124.998
124.774
120.698
120.643
120.450
120.396
118.557
114.547
77.447
77.023
76.600
60.821
58.695
55.604
45.351
25.858
O=P–OPh
OPh
OMe
ppm (f1)


14.571
O=P–OPh
OPh
OMe


7.442
7.424
7.417
7.410
7.172
7.167
7.147
7.126
7.099
7.065
6.959
6.930
6.810
6.806
6.781
6.631
6.628
6.594
6.586
6.559
6.515
6.507
6.499
6.345
6.337
6.318
6.310
5.541
5.475
4.001
3.974
3.958
3.942
3.914
3.698
3.608
3.590
3.570
3.547
2.978
2.968
2.960
2.950
OMe
O=P
OPh
OPh
1.16
13.5
2.10
1.94
1.91
0.96
1.00
1.05
3.14
1.16
1.96
2000
1500
1000
500
0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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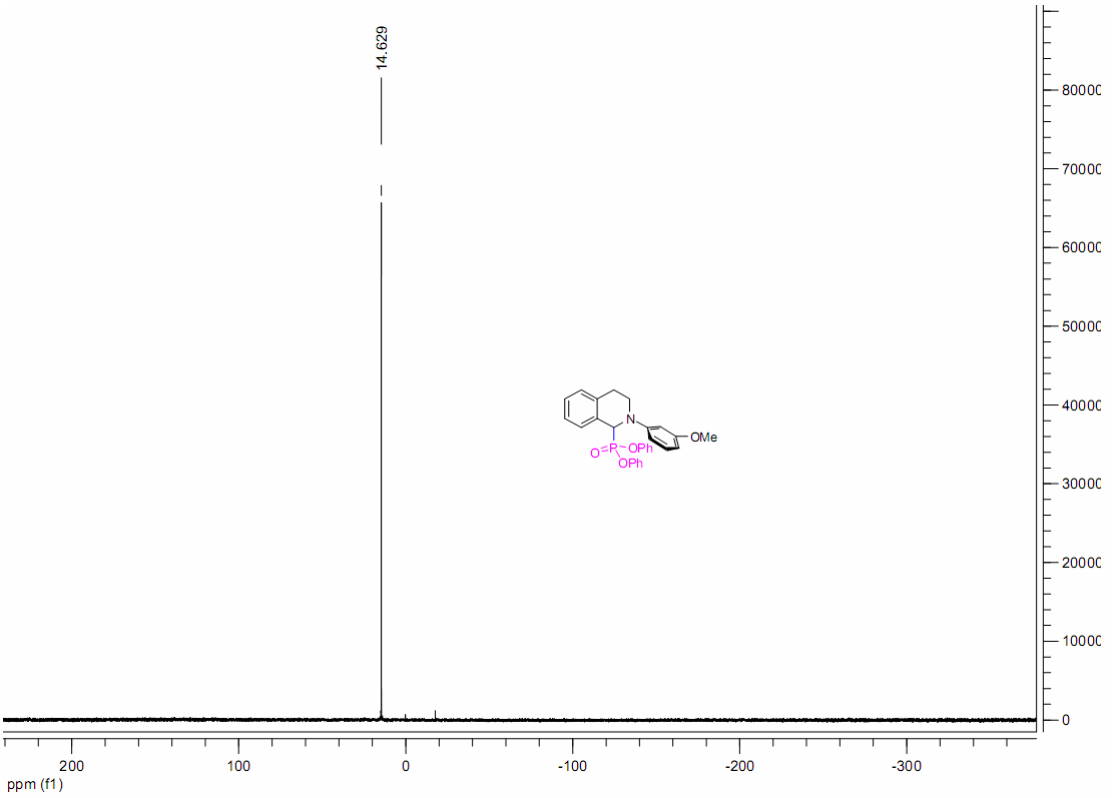
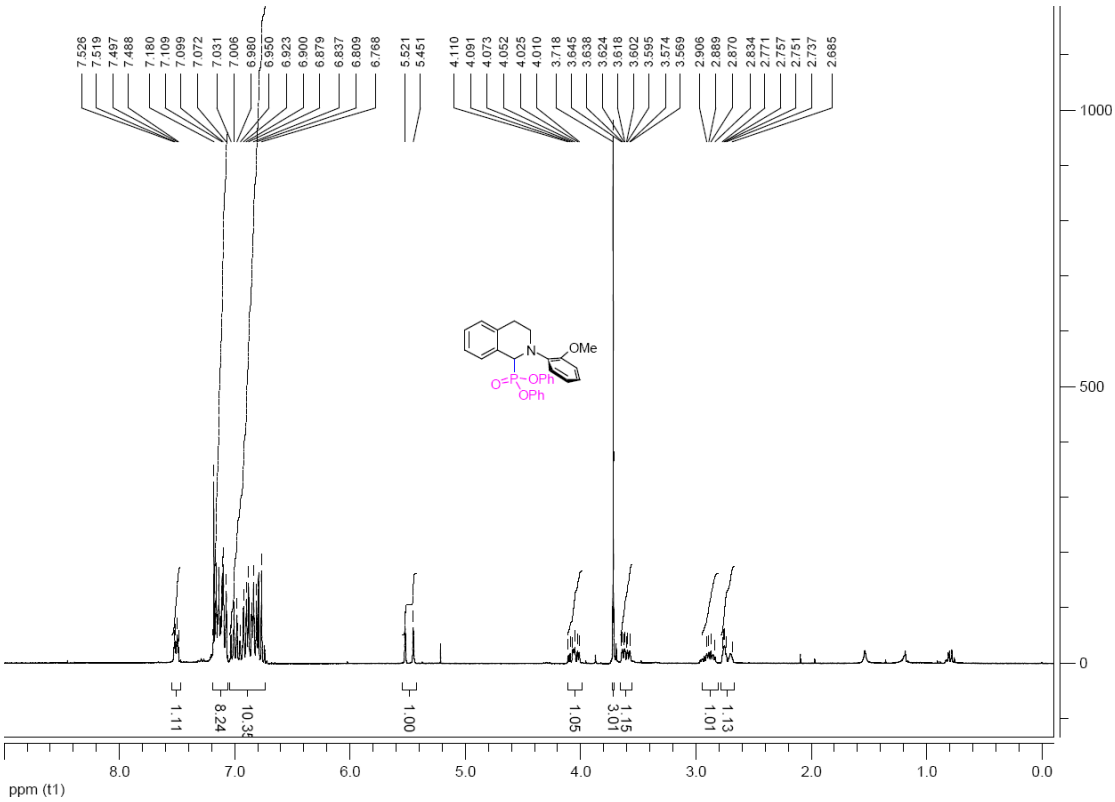
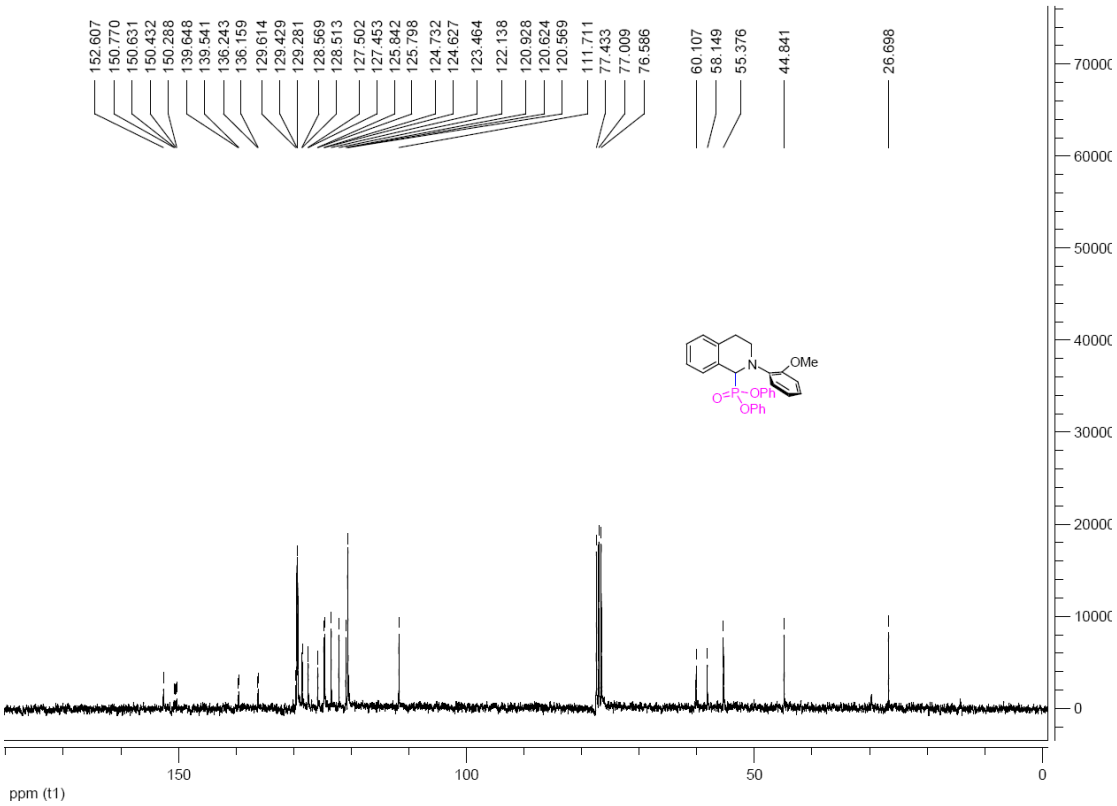
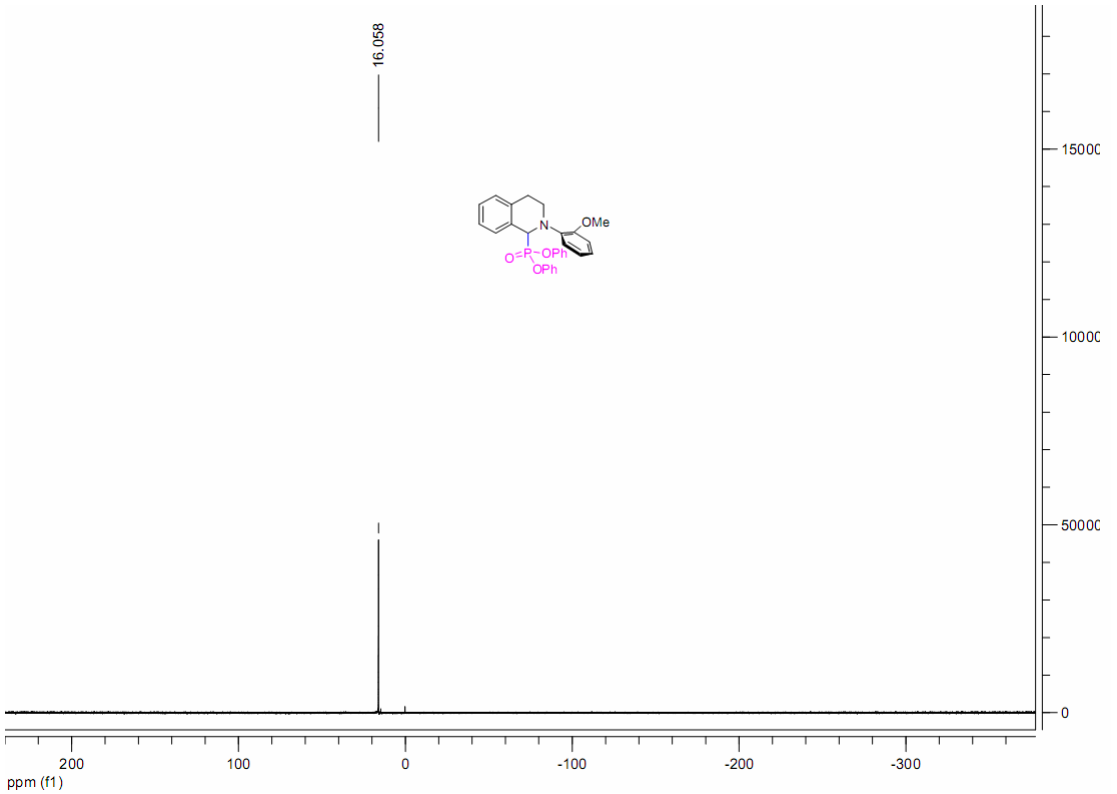
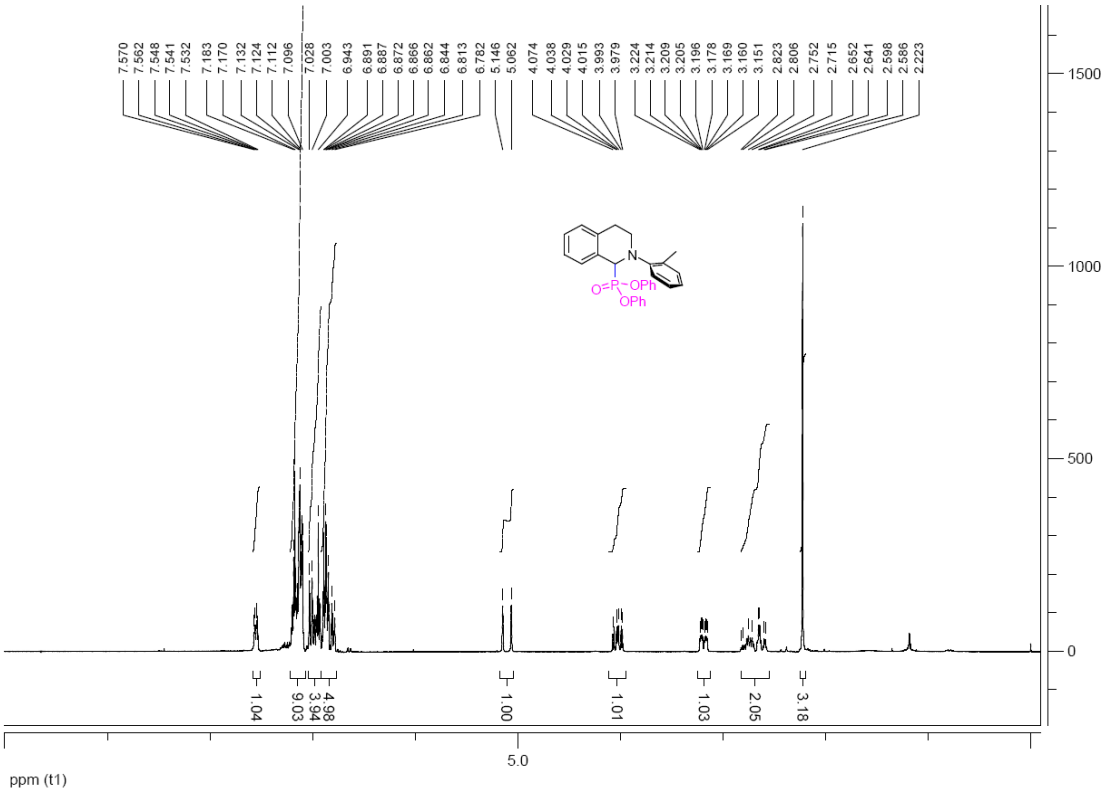
150.649
150.557
150.341
150.192
136.746
136.672
129.929
129.621
129.435
129.383
129.021
128.987
128.398
128.331
128.007
127.961
126.245
126.210
125.091
124.874
120.680
120.625
120.422
120.368
115.327
108.202
103.833
102.075
77.444
77.020
76.597
60.312
58.179
55.208
44.030
26.669
OMe
O=P
OPh
OPh
20000
15000
10000
5000
0
150
100
50
0
ppm (t1)

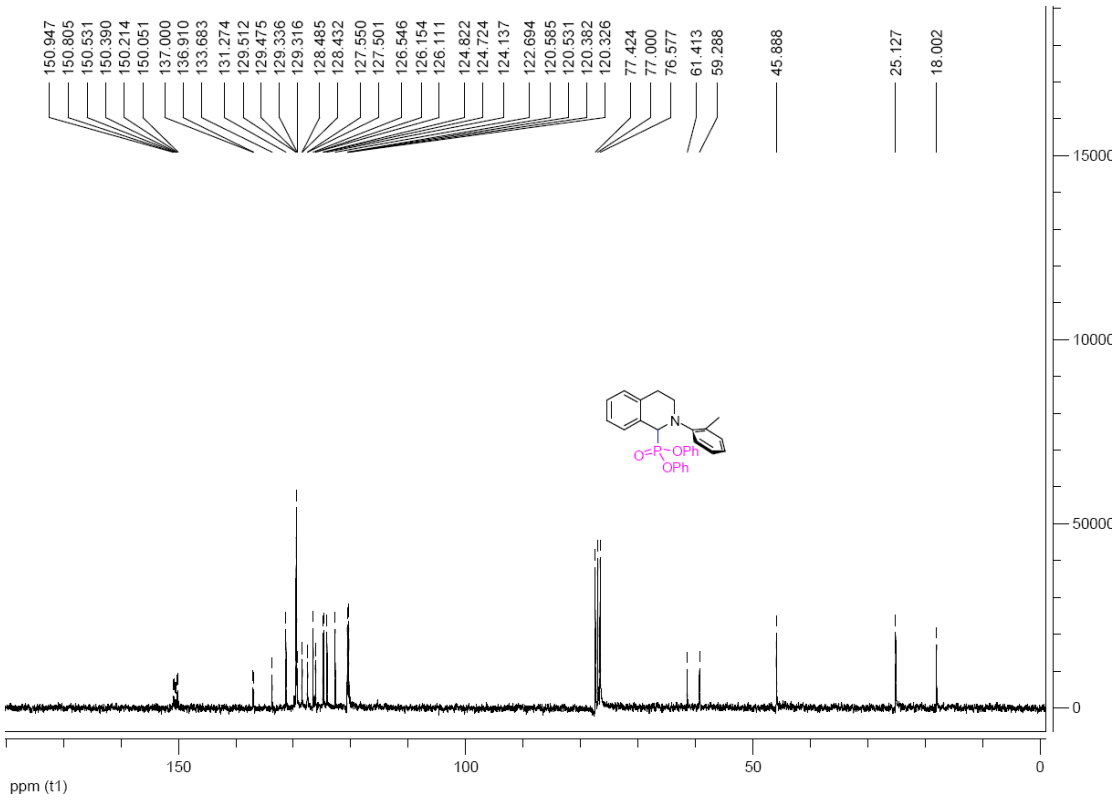

14.629
OMe
O=P(OPh)OPh
ppm (f1)

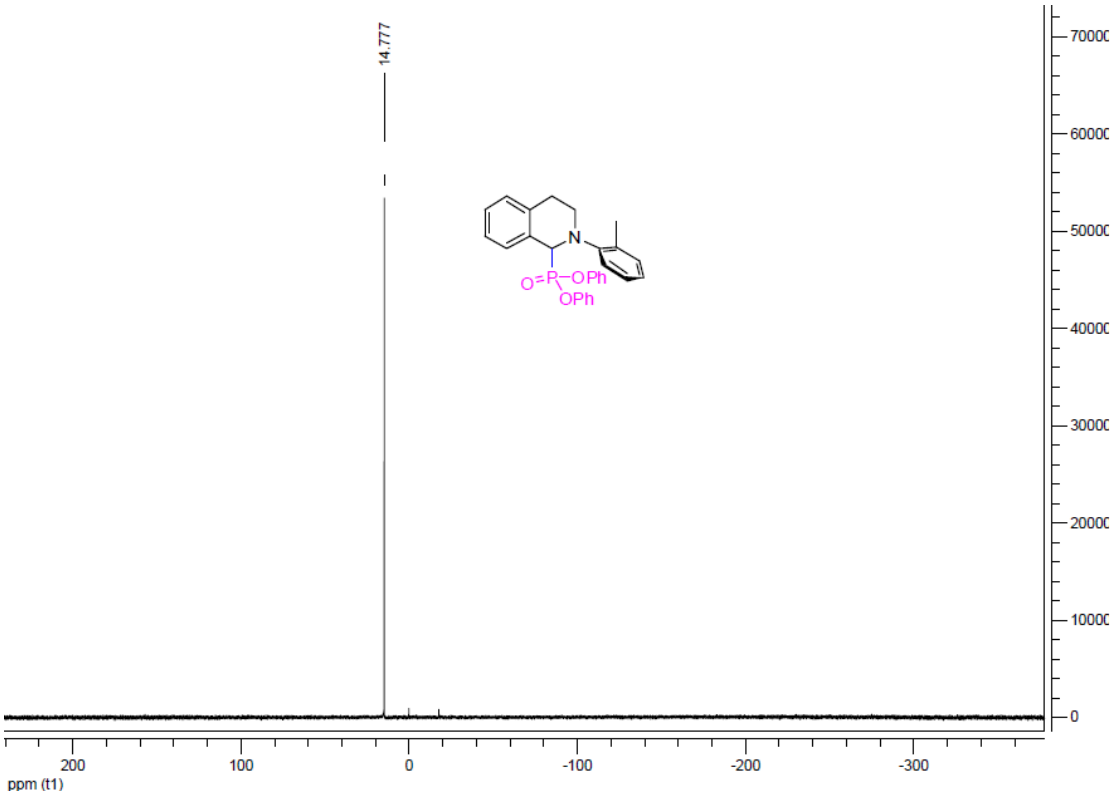

7.526
7.519
7.497
7.488
7.180
7.109
7.099
7.072
7.031
7.006
6.980
6.950
6.923
6.900
6.879
6.837
6.809
6.768
5.521
5.451
4.110
4.091
4.073
4.052
4.025
4.010
3.718
3.645
3.638
3.624
3.618
3.602
3.595
3.574
3.569
2.906
2.889
2.870
2.834
2.771
2.757
2.751
2.737
2.685
OMe
O=P(OPh)OPh
1.11
8.24
10.34
1.00
1.05
1.15
3.01
1.01
1.13
ppm (t1)

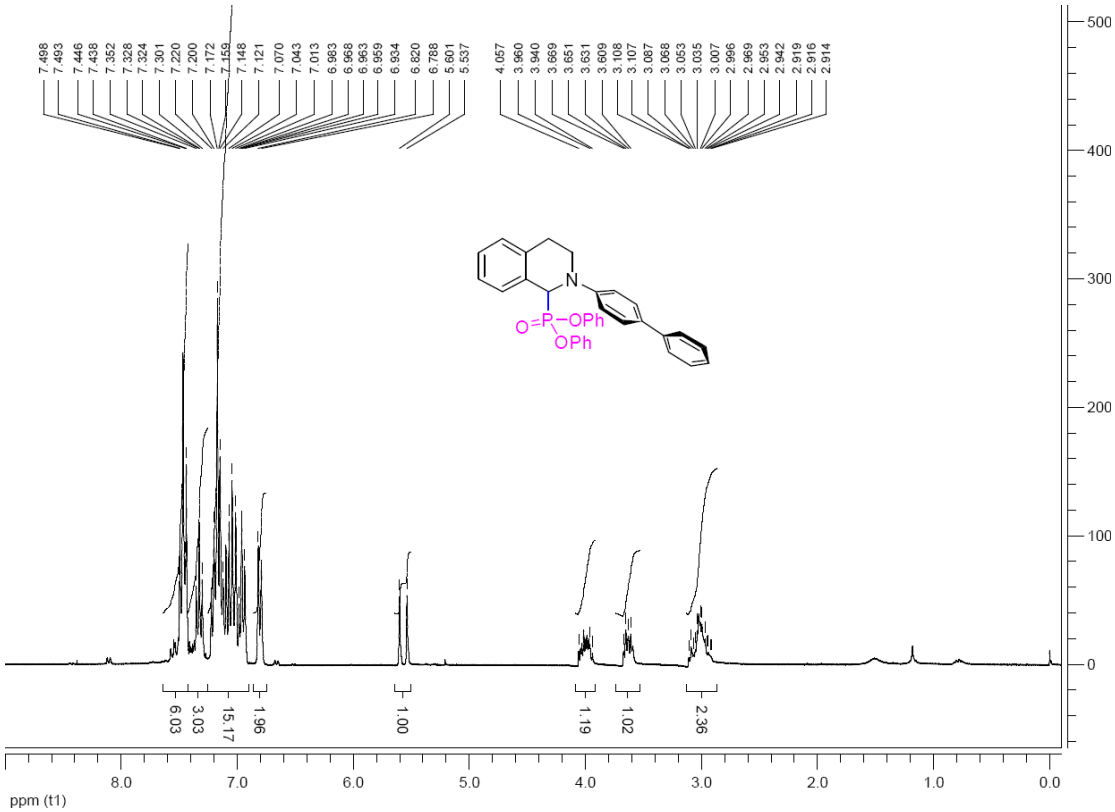

152.607
150.770
150.631
150.432
150.288
139.648
139.541
136.243
136.159
129.614
129.429
129.281
128.569
128.513
127.502
127.453
125.842
125.798
124.732
124.627
123.464
122.138
120.928
120.624
120.569
111.711
77.433
77.009
76.586
60.107
58.149
55.376
44.841
26.698
OMe
O=P-OPh
OPh
ppm (t1)

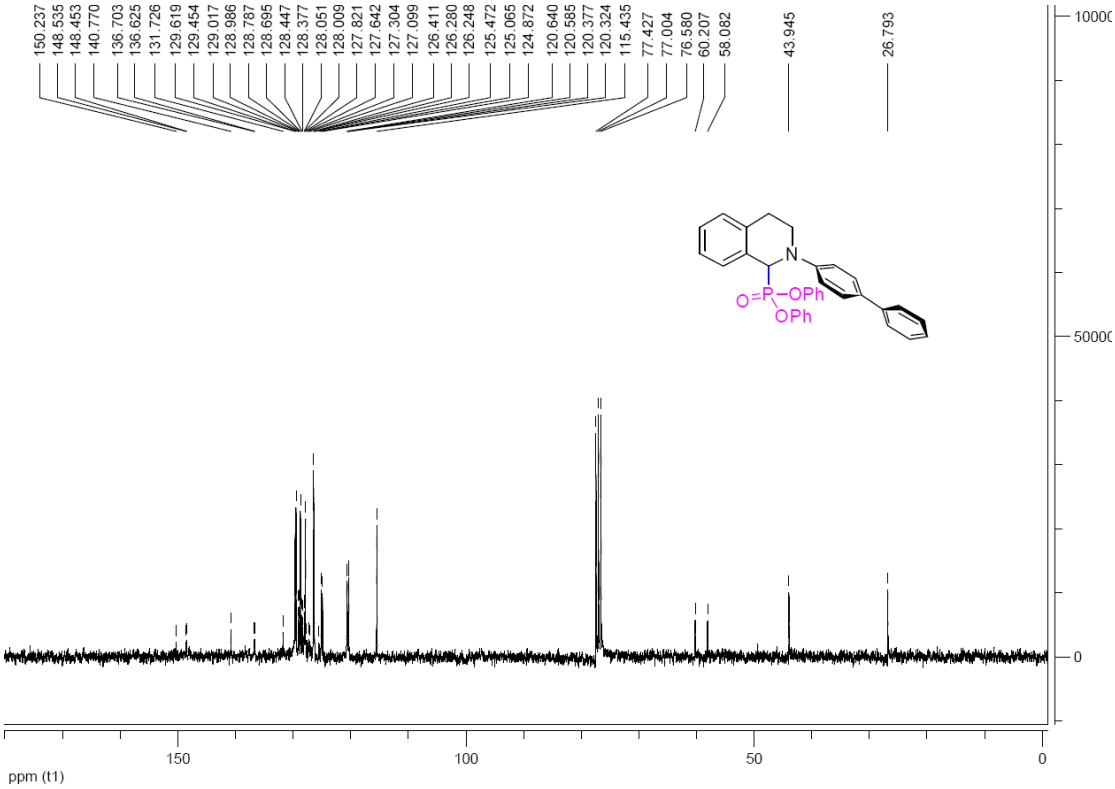

16.058
OMe
O=P-OPh
OPh
ppm (f1)

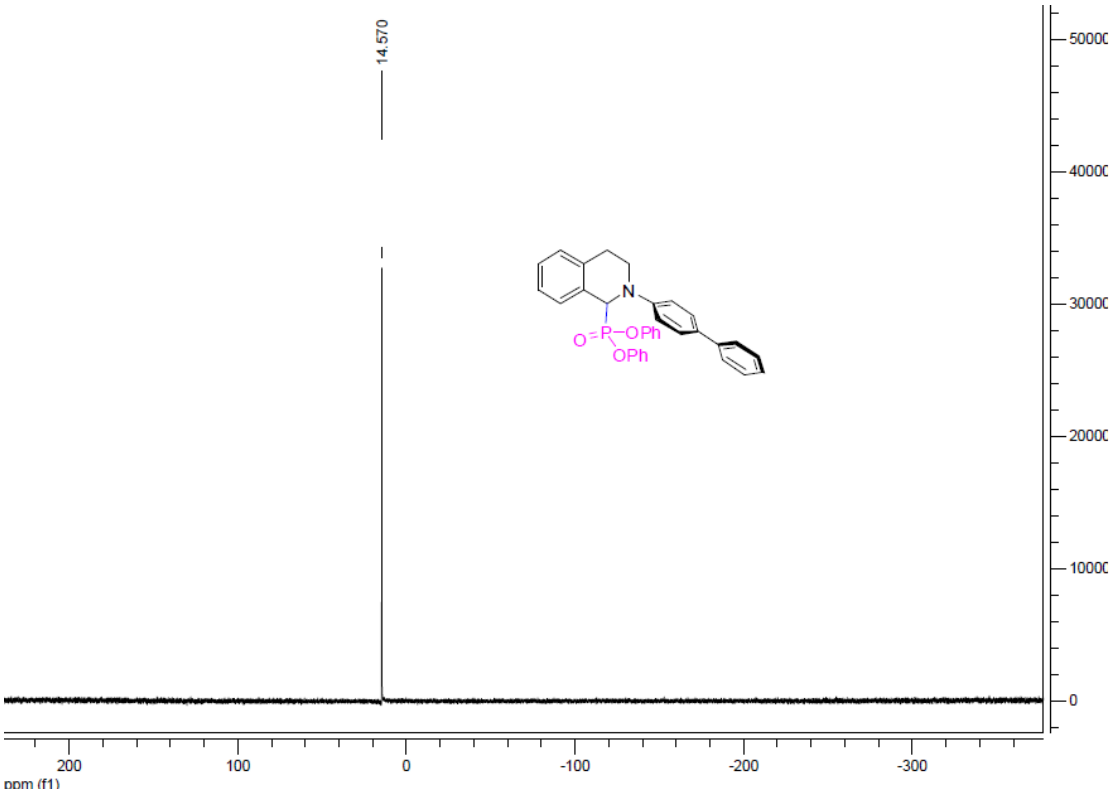

7.570
7.562
7.548
7.541
7.532
7.183
7.170
7.132
7.124
7.112
7.096
7.028
7.003
6.943
6.891
6.887
6.872
6.866
6.862
6.844
6.813
6.782
5.146
5.062
4.074
4.038
4.029
4.015
3.993
3.979
3.224
3.214
3.209
3.205
3.196
3.178
3.169
3.160
3.151
2.823
2.806
2.752
2.715
2.652
2.641
2.598
2.586
2.223
1500
1000
500
0
1.04
9.03
3.46
4.88
1.00
1.01
1.03
2.05
3.18
5.0
ppm (t1)

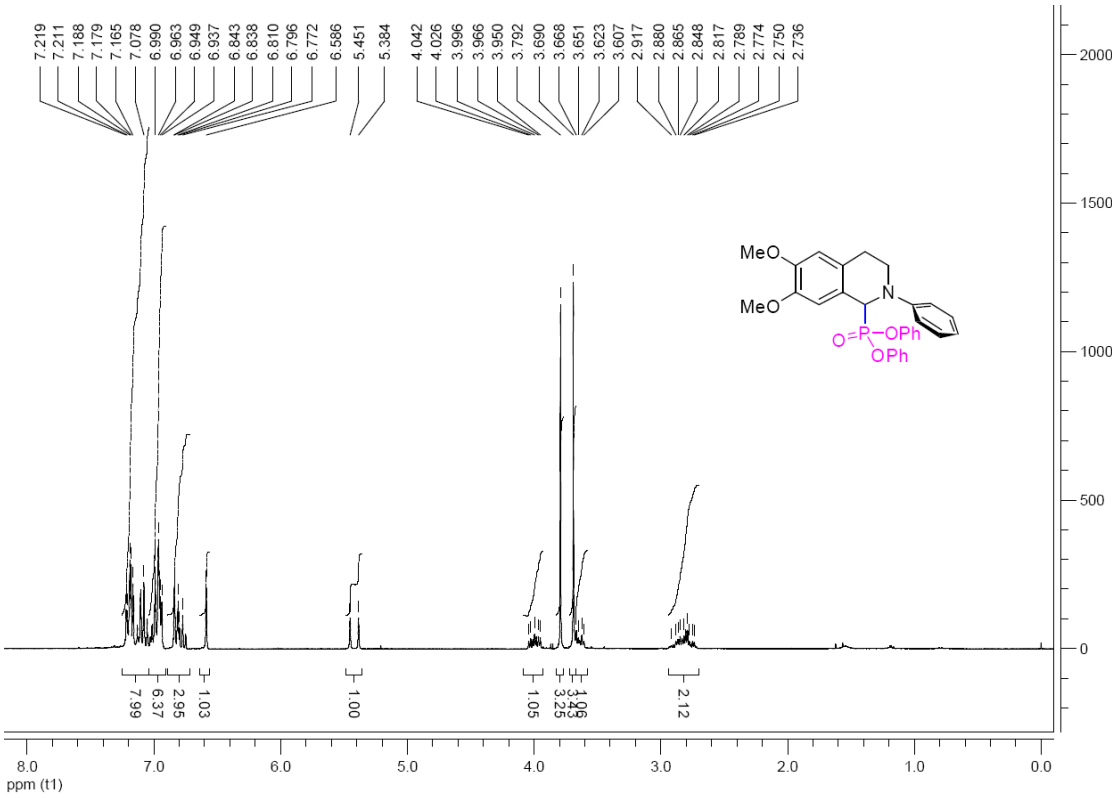

150.947
150.805
150.531
150.390
150.214
150.051
137.000
136.910
133.683
131.274
129.512
129.475
129.336
129.316
128.485
128.432
127.550
127.501
126.546
126.154
126.111
124.822
124.724
124.137
122.694
120.585
120.531
120.382
120.326
77.424
77.000
76.577
61.413
59.288
45.888
25.127
18.002
15000
10000
5000
0
150
100
50
0
ppm (t1)

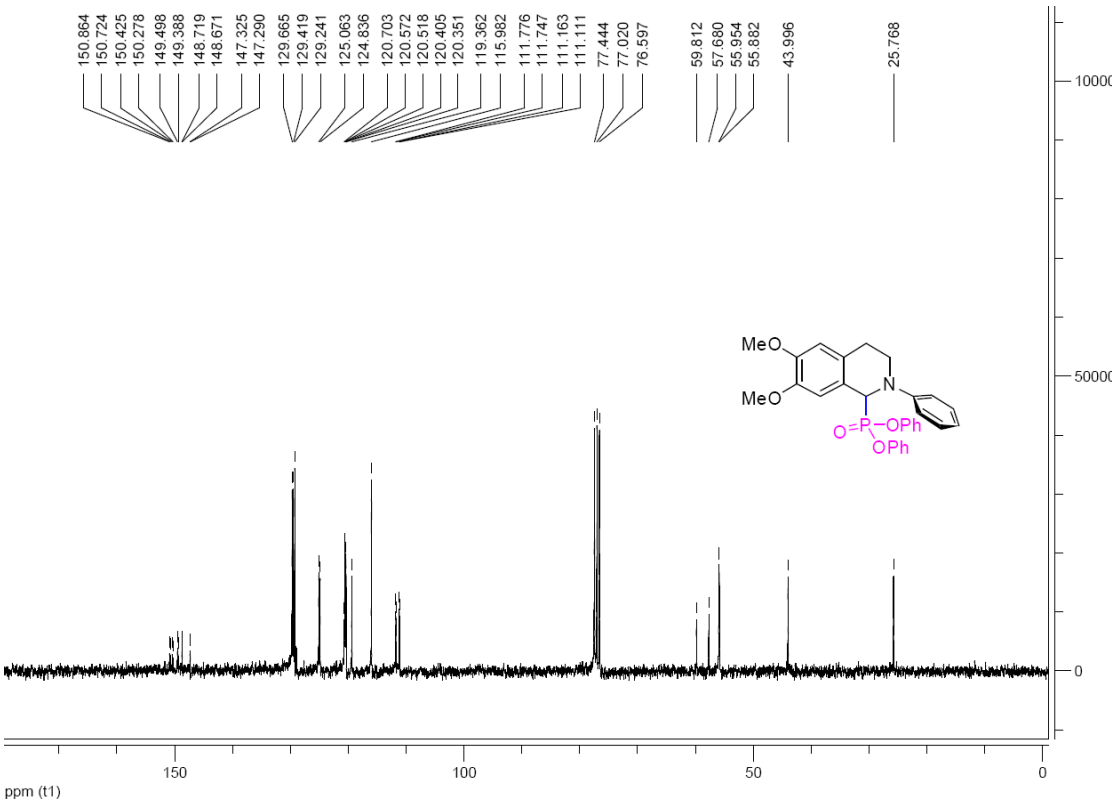

14.777
O=P–OPh
OPh
ppm (t1)

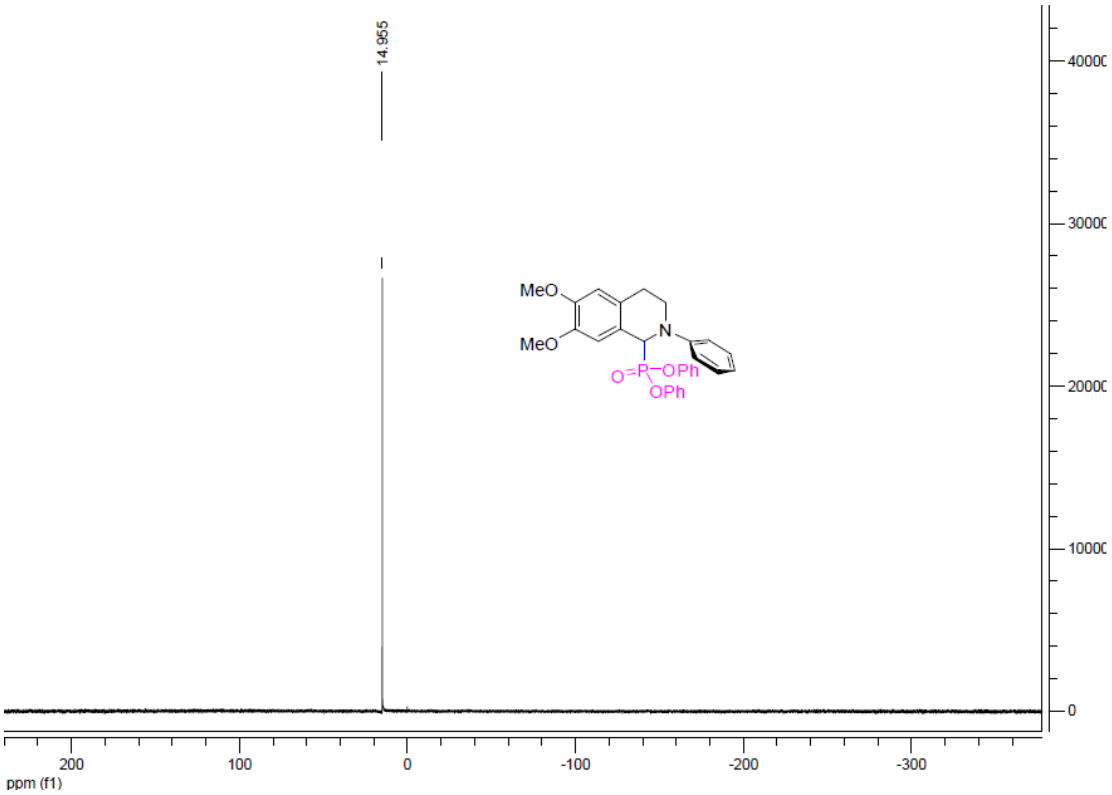

7.498
7.493
7.446
7.438
7.352
7.328
7.324
7.301
7.220
7.200
7.172
7.158
7.148
7.121
7.070
7.043
7.013
6.983
6.968
6.963
6.959
6.934
6.820
6.788
6.601
5.537
4.057
3.960
3.940
3.869
3.651
3.631
3.609
3.108
3.107
3.087
3.068
3.053
3.035
3.007
2.996
2.969
2.953
2.942
2.919
2.916
2.914
O=P–OPh
OPh
6.03
3.03
15.17
1.96
1.00
1.19
1.02
2.36
ppm (t1)


150.237
148.535
148.453
140.770
136.703
136.625
131.726
129.619
129.454
129.017
128.986
128.787
128.695
128.447
128.377
128.051
128.009
127.821
127.642
127.304
127.099
126.411
126.280
126.248
125.472
125.065
124.872
120.640
120.585
120.377
120.324
115.435
77.427
77.004
76.580
60.207
58.082
43.945
26.793
O=P–OPh
OPh
ppm (t1)


14.570
O=P–OPh
OPh
ppm (f1)


7.219
7.211
7.188
7.179
7.165
7.078
6.990
6.963
6.949
6.937
6.843
6.838
6.810
6.796
6.772
6.586
5.451
5.384
4.042
4.026
3.996
3.966
3.950
3.792
3.690
3.668
3.651
3.623
3.607
2.917
2.880
2.865
2.848
2.817
2.789
2.774
2.750
2.736
MeO
MeO
O=P–OPh
OPh
7.69
6.37
2.65
1.03
1.00
1.05
3.25
2.12
ppm (t1)


150.864
150.724
150.425
150.278
149.498
149.388
148.719
148.671
147.325
147.290
129.665
129.419
129.241
125.063
124.836
120.703
120.572
120.518
120.405
120.351
119.362
115.982
111.776
111.747
111.163
111.111
77.444
77.020
76.597
59.812
57.680
55.954
55.882
43.996
25.768
MeO
MeO
O=P–OPh
OPh
ppm (t1)


14.955
MeO
MeO
N
O=P–OPh
OPh
40000
30000
20000
10000
0
200
100
0
-100
-200
-300
ppm (f1)