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Electronic Supplementary Information

Direct Phosphorylation of Benzylic C-H Bonds under Transition Metal-Free Conditions Forming sp^3C-P Bonds

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General Information

Chemicals were purchased and used as received unless otherwise noted. All reactions were carried out in a sealed Schlenk tubes and monitored by GC and/or GC-MS. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avance-III 400 instrument (400 MHz for 1H, 100 MHz for 13C, and 162 MHz for 31P NMR spectroscopy). $CDCl_3$ was used as the solvent. Chemical shifts for 1H NMR are referred to internal Me4Si (0 ppm) and reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz) and integration. Data for 31P NMR were relative to H_3PO_4 (85% solution in D_2O, 0 ppm). Mass spectra were recorded by GCMS-QP2010 ultra spectrometer. The high-resolution mass spectrum (HRMS) was recorded on MAT 95 XP instrument. The GC yields were determined using dodecane as an internal standard on Shimadzu GC-2014 FID system.

Typical procedure for the direct phosphorylation of benzylic C-H bonds

A mixture of toluene **1a** (0.8 mL), $K_2S_2O_8$ (162 mg, 0.6 mmol), and diphenylphosphine oxide **2a** (40.4 mg, 0.2 mmol), SDBS (35.0 g, 0.1 mmol), H_2O (1.6 mL) was stirred at 120 °C under N_2 in a sealed 25 mL glass tube for 15 min. Then the mixture was extracted with EtOAc. The organic layer was dried over Na_2SO_4, and concentrated under a reduced pressure. The residue was purified by column chromatography on silica gel with EtOAc/petroleum (from 4:1 to 1:1) to afford benzyldiphenylphosphine oxide **3a** in 48% yield.

Characterization and analytical data of products

*Benzyldiphenylphosphine oxide (**3a**):*[1] White solid (28.1 mg, 48%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71-7.67 (m, 4H), 7.53-7.49 (m, 2H), 7.46-7.41 (m, 4H), 7.19-7.17 (m, 3H), 7.11-7.09 (m, 2H), 3.66 (d, J = 13.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 132.1 (d, J = 98.4 Hz), 131.8 (d, J = 2.5 Hz), 131.2 (d, J = 8.9 Hz), 131.1 (d, J = 8.1 Hz), 130.1 (d, J = 5.0 Hz), 128.5 (d, J = 11.5 Hz), 128.4 (d, J = 2.3 Hz), 126.8 (d, J = 2.6 Hz), 38.1 (d, J = 66.2 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 29.8.

*(4-Methylbenzyl)diphenylphosphine oxide (**3b**):*[2] White solid (30.6 mg, 50%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72-7.67 (m, 4H), 7.53-7.49 (m, 2H), 7.46-7.42 (m, 4H), 6.99 (s, 4H), 3.63 (d, J = 13.6 Hz, 2H), 2.26 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 136.4 (d, J = 3.0 Hz), 132.2 (d, J = 94.8 Hz), 131.8 (d, J = 2.5 Hz),

131.2 (d, J = 9.0 Hz), 130.0 (d, J = 5.1 Hz), 129.1 (d, J = 2.3 Hz), 128.5 (d, J = 11.5 Hz), 127.8 (d, J = 8.0 Hz), 37.6 (d, J = 66.2 Hz), 21.0. ^{31}P NMR (162 MHz, $CDCl_3$): δ 29.9.

*(3-Methylbenzyl)diphenylphosphine oxide (**3c**)*:[3] White solid (26.9 mg, 44%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72-7.67 (m, 4H), 7.53-7.50 (m, 2H), 7.46-7.42 (m, 4H), 7.06 (t, J = 7.6 Hz, 1H), 6.98 (d, J = 8.0 Hz, 1H), 6.92 (s, 1H), 6.86 (d, J = 7.6 Hz, 1H), 3.62 (d, J = 13.6 Hz, 2H), 2.22 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.9 (d, J = 2.3 Hz), 132.3 (d, J = 97.1 Hz), 131.8 (d, J = 2.5 Hz), 131.2 (d, J = 8.8 Hz), 131.0 (d, J = 4.8 Hz), 130.9 (d, J = 7.8 Hz), 128.4 (d, J = 11.4 Hz), 128.2 (d, J = 2.2 Hz), 127.6 (d, J = 2.7 Hz), 127.1 (d, J = 5.0 Hz), 38.0 (d, J = 66.0 Hz), 21.3. ^{31}P NMR (162 MHz, $CDCl_3$): δ 29.8.

*(2-Methylbenzyl)diphenylphosphine oxide (**3d**)*:[1] White solid (31.9 mg, 52%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67 (dd, J = 11.2 Hz, 7.6 Hz, 4H), 7.55-7.51 (m, 2H), 7.46-7.42 (m, 4H), 7.12-7.07 (m, 2H), 7.01-6.94 (m, 2H), 3.68 (d, J = 14.0 Hz, 2H), 2.14 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.5 (d, J = 5.2 Hz), 132.4 (d, J = 94.4 Hz), 131.9 (d, J = 2.0 Hz), 131.3 (d, J = 8.9 Hz), 130.7 (d, J = 4.3 Hz), 130.5 (d, J = 2.3 Hz), 129.6 (d, J = 7.8 Hz), 128.5 (d, J = 11.4 Hz), 127.0 (d, J = 2.9 Hz), 125.8 (d, J = 2.5 Hz), 35.3 (d, J = 67.1 Hz), 20.1. ^{31}P NMR (162 MHz, $CDCl_3$): δ 30.0.

*(3,5-Dimethylbenzyl)diphenylphosphine oxide (**3e**):* White solid (32.0 mg, 50%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (dd, J = 11.2 Hz, 7.6 Hz, 4H), 7.53-7.50 (m, 2H), 7.47-7.42 (m, 4H), 6.80 (s, 1H), 6.68 (s, 2H), 3.58 (d, J = 13.6 Hz, 2H), 2.17 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.7 (d, J = 2.4 Hz), 132.2 (d, J = 100.0 Hz), 131.7 (d, J = 2.6 Hz), 131.2 (d, J = 8.9 Hz), 130.6 (d, J = 7.9 Hz), 128.4, 128.4 (d, J = 11.7 Hz), 128.0 (d, J = 5.1 Hz), 37.9 (d, J = 66.3 Hz), 21.1. ^{31}P NMR (162 MHz, $CDCl_3$): δ 30.0. MS (EI): *m/z* (%) 321 (8), 320 (35), 319 (28), 202 (14), 201 (100), 195 (9), 119 (8), 91 (6), 77 (17), 51 (5). HRMS Calcd for $C_{21}H_{21}OP$: 320.1330; found: 320.1315.

*Diphenyl(2,4,5-trimethylbenzyl)phosphine oxide (**3f**):* White solid (28.1 mg, 42%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70-7.65 (m, 4H), 7.54-7.51 (m, 2H), 7.46-7.42 (m, 4H), 6.86 (s, 1H), 6.69 (s, 1H), 3.60 (d, J = 13.6 Hz, 2H), 2.16 (s, 3H), 2.04 (s, 3H), 2.02 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 135.1 (d, J = 5.1 Hz), 134.4 (d, J = 5.6 Hz), 133.7 (d, J = 2.7 Hz), 133.1 (d, J = 2.0 Hz), 132.6 (d, J = 99.6 Hz), 132.0 (d, J = 4.3 Hz), 131.7 (b), 131.3 (d, J = 9.1 Hz), 128.4 (d, J = 11.5 Hz), 127.3 (d, J = 10.0 Hz), 126.4 (d, J = 8.3 Hz), 34.8 (d, J = 66.6 Hz), 19.3, 19.2, 19.0. ^{31}P NMR (162 MHz, $CDCl_3$): δ 29.6. MS (EI): *m/z* (%) 335 (24), 334 (100), 319 (12), 243 (12), 209 (46), 201 (76), 193 (16), 133 (93), 132 (14), 117 (24), 91 (16), 77 (20). HRMS Calcd for $C_{22}H_{23}OP$: 334.1487; found: 334.1480.

*(4-Methoxybenzyl)diphenylphosphine oxide (**3g**)*:[1] White solid (16.1 mg, 25%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71-7.67 (m, 4H), 7.53-7.50 (m, 2H), 7.46-7.42 (m, 4H), 7.02 (d, J = 8.0 Hz, 2H), 6.73 (d, J = 8.0 Hz, 2H), 3.74 (s, 3H), 3.61 (d, J = 13.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.5 (d, J = 2.7 Hz), 132.3

(d, J = 98.3 Hz), 131.7 (d, J = 2.7 Hz), 131.2 (d, J = 9.0 Hz), 131.1 (d, J = 5.0 Hz), 128.5 (d, J = 11.5 Hz), 122.9 (d, J = 8.0 Hz), 113.9 (d, J = 2.5 Hz), 55.2, 37.1 (d, J = 66.9 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 30.1.

*(3-Methoxybenzyl)diphenylphosphine oxide (**3h**):* White solid (13.5 mg, 21%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (dd, J = 11.6 Hz, 7.6 Hz, 4H), 7.53-7.50 (m, 2H), 7.46-7.42 (m, 4H), 7.09 (t, J = 7.6 Hz, 1H), 6.70 (dd, J = 10.8, 10.8 Hz, 2 H), 6.63 (s, 1H), 3.65 (s, 3H), 3.64 (d, J = 14.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.4 (d, J = 2.6 Hz), 132.6 (d, J = 7.9 Hz), 132.3 (d, J = 98.4 Hz), 131.8 (d, J = 2.8 Hz), 131.2 (d, J = 9.1 Hz), 129.3 (d, J = 2.5 Hz), 128.5 (d, J = 11.6 Hz), 122.6 (d, J = 5.4 Hz), 115.2 (d, J = 5.1 Hz), 113.0 (d, J = 2.9 Hz), 55.1, 38.2 (d, J = 66.0 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 29.6. MS (EI): *m/z* (%) 323 (5), 322 (28), 321 (46), 202 (13), 201 (100), 77 (15), 51 (6). HRMS Calcd for $C_{20}H_{18}O_2P$: 322.1123; found: 321.1040 [M-H].

*(4-Fluorobenzyl)diphenylphosphine oxide (**3i**):*[1] White solid (25.4 mg, 41%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71-7.66 (m, 4H), 7.54-7.51 (m, 2H), 7.47-7.43 (m, 4H), 7.09-7.05 (m, 2H), 6.88 (t, J = 8.4 Hz, 2H), 3.62 (d, J = 13.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.9 (dd, J = 243.8, 3.3 Hz), 132.0 (d, J = 99.5 Hz), 131.9 (d, J = 2.7 Hz), 131.6 (dd, J = 7.8, 5.3 Hz), 131.1 (d, J = 9.1 Hz), 128.5 (d, J = 11.6 Hz), 126.8 (dd, J = 7.8, 3.3 Hz), 115.3 (dd, J = 21.4, 2.5 Hz), 37.2 (d, J = 66.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 29.8.

*(4-Chlorobenzyl)diphenylphosphine oxide (**3j**):*[2] White solid (24.1 mg, 37%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71-7.66 (m, 4H), 7.54-7.51 (m, 2H), 7.47-7.43 (m, 4H), 7.16 (d, J = 8.0 Hz, 2H), 7.04 (d, J = 8.0 Hz, 2H), 3.62 (d, J = 13.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 132.9 (d, J = 3.5 Hz), 132.0 (d, J = 2.8 Hz), 131.8 (d, J = 111.1 Hz), 131.4 (d, J = 5.1 Hz), 131.1 (d, J = 9.1 Hz), 129.6 (d, J = 8.1 Hz), 128.6 (d, J = 14.2 Hz) 128.5, 37.4 (d, J = 66.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 29.5.

*(4-Bromobenzyl)diphenylphosphine oxide (**3k**):*[2] White solid (25.9 mg, 35%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71-7.66 (m, 4H), 7.54-7.51 (m, 2H), 7.47-7.43 (m, 4H), 7.30 (d, J = 8.0 Hz, 2H), 6.98 (d, J = 8.0 Hz, 2H), 3.59 (d, J = 13.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 131.9 (d, J = 2.7 Hz), 131.9 (d, J = 98.7 Hz), 131.7 (d, J = 5.2 Hz), 131.4 (d, J = 2.5 Hz), 131.1 (d, J = 9.0 Hz), 130.2 (d, J = 7.7 Hz), 128.6 (d, J = 11.7 Hz), 120.9 (d, J = 3.7 Hz), 37.5 (d, J = 65.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 29.1.

*Diphenyl(1-phenylethyl)phosphine oxide (**3l**):*[4] White solid (18.4 mg, 30%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92-7.88 (m, 2H), 7.58-7.50 (m, 3H), 7.47-7.42 (m, 2H), 7.37-7.34 (m, 1H), 7.28-7.14 (m, 7H), 3.65-3.57 (m, 1H), 1.58 (dd, J = 16.0, 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.9 (d, J = 5.5 Hz), 131.9 (d, J = 97.9 Hz), 131.8 (d, J = 97.9 Hz), 131.7 (d, J = 2.7 Hz), 131.4 (d, J = 8.4 Hz), 131.3 (d, J = 2.6 Hz), 131.1 (d, J = 9.6 Hz), 129.2 (d, J = 5.5 Hz), 128.7 (d, J = 11.1 Hz), 128.2 (d, J = 1.9 Hz), 128.1 (d, J = 11.5 Hz), 126.9 (d, J = 2.6 Hz), 41.0 (d, J = 66.6 Hz), 15.4 (d, J = 2.7 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 33.8.

*(2-Methyl-1-phenylpropyl)diphenylphosphine oxide (**3m**):* White solid (18.1 mg, 27%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98-7.93 (m, 2H), 7.53-7.46 (m, 5H), 7.38 (d, J = 7.6 Hz, 2H), 7.25-7.10 (m, 6H), 3.34 (dd, J = 8.0, 5.6 Hz, 1H), 2.54-2.43 (m, 1H), 1.00 (d, J = 6.8 Hz, 3H), 0.87 (d, J = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 135.1 (d, J = 3.7 Hz), 133.9 (d, J = 92.8 Hz), 133.6 (d, J = 92.8 Hz), 131.4 (d, J = 2.5 Hz), 131.0 (d, J = 2.7 Hz), 130.9 (b), 130.7 (d, J = 8.3 Hz), 130.7 (b), 128.7 (d, J = 11.0 Hz), 127.9 (b), 127.9 (d, J = 13.0 Hz), 126.7 (d, J = 1.6 Hz), 52.4 (d, J = 68.0 Hz), 29.4, 23.4 (d, J = 10.0 Hz), 20.6 (d, J = 5.3 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 31.8. MS (EI): *m/z* (%) 334 (12), 293 (8), 292 (36), 291 (9), 216 (30), 203 (20), 202 (100), 201 (89), 183 (8), 165 (8), 125 (8), 91 (46), 77 (20), 55 (7). HRMS Calcd for $C_{22}H_{23}OP$: 334.1487; found: 334.1481.

*(Naphthalen-1-ylmethyl)diphenylphosphine oxide (**3p**):*[1] White solid (13.7 mg, 20%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, J = 8.0 Hz, 1H), 7.78 (d, J = 7.6 Hz, 1H), 7.71-7.67 (m, 5H), 7.50-7.46 (m, 2H), 7.42-7.35 (m, 6H), 7.29-7.23 (m, 2H), 4.13 (d, J = 14.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 133.8 (d, J = 1.9 Hz), 132.5 (d, J = 4.4 Hz), 132.3 (d, J = 101.4 Hz), 131.8 (d, J = 2.9 Hz), 131.2 (d, J = 9.1 Hz), 128.7 (d, J = 6.2 Hz), 128.5 (d, J = 2.9 Hz), 128.5 (d, J = 11.6 Hz), 127.7 (d, J = 8.4 Hz), 127.7 (d, J = 3.3 Hz), 125.9, 125.6, 125.1 (d, J = 3.3 Hz), 124.3, 34.9 (d, J = 66.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 29.7.

*(Naphthalen-2-ylmethyl)diphenylphosphine oxide (**3q**):*[4] White solid (19.9 mg, 29%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75-7.69 (m, 5H), 7.66 (d, J = 7.6 Hz, 2H), 7.57 (s, 1H), 7.52-7.48 (m, 2H), 7.44-7.39 (m, 6H), 7.25-7.22 (m, 1H), 3.81 (d, J = 14.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 133.3 (d, J = 2.1 Hz), 132.3 (d, J = 94.3 Hz), 132.2 (d, J = 1.4 Hz), 131.9 (d, J = 2.3 Hz), 131.2 (d, J = 9.0 Hz), 129.0 (d, J = 6.6 Hz), 128.8 (d, J = 8.1 Hz), 128.6 (d, J = 11.5 Hz), 128.2 (d, J = 4.0 Hz), 128.0 (d, J = 1.7 Hz), 127.6, 127.6, 126.0, 125.7, 38.4 (d, J = 65.9 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 29.6.

*Diphenyl(quinolin-2-ylmethyl)phosphine oxide (**3r**):*[6] White solid (33.7 mg, 49%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (d, J = 8.4 Hz, 1H), 7.91 (d, J = 8.4 Hz, 1H), 7.80 (dd, J = 12.0, 7.6 Hz, 4H), 7.74 (d, J = 8.0 Hz, 1H), 7.66-7.60 (m, 2H), 7.49-7.45 (m, 3H), 7.43-7.38 (m, 4H), 4.2 (d, J = 14.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.2 (d, J = 6.8 Hz), 147.6, 136.5, 132.2 (d, J = 100.9 Hz), 131.8 (d, J = 2.9 Hz), 131.1 (d, J = 9.5 Hz), 129.5, 128.5, 128.4 (d, J = 11.8 Hz), 127.5, 126.8 (d, J = 1.5 Hz), 126.2, 122.7 (d, J = 2.5 Hz), 41.7 (d, J = 63.1 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 30.1.

*Benzyldi-p-tolylphosphine oxide (**3s**):*[7] White solid (26.3 mg, 41%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 (dd, J = 11.2 Hz, 7.6 Hz, 4H), 7.23 (d, J = 7.2 Hz, 4H), 7.19-7.17 (m, 3H), 7.11 (d, J = 6.0 Hz, 2H), 3.62 (d, J = 13.6 Hz, 2H), 2.38 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.1 (d, J = 2.8 Hz), 131.4 (d, J = 7.8 Hz), 131.1 (d, J = 9.4 Hz), 130.1 (d, J = 5.2 Hz), 129.1 (d, J = 12.0 Hz), 129.1 (d, J = 100.9 Hz), 128.3 (d, J = 2.5 Hz), 126.6 (d, J = 2.9 Hz), 38.2 (d, J = 66.3 Hz), 21.5. ^{31}P NMR (162 MHz, $CDCl_3$): δ 30.2.

*Benzylbis(4-fluorophenyl)phosphine oxide (**3t**):* White solid (29.5 mg, 45%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69-7.63 (m, 4H), 7.21-7.20 (m, 3H), 7.16-7.12 (m, 2H), 7.13 (dd, J = 8.4, 8.4 Hz, 4H), 7.08 (b, 2H), 3.63 (d, J = 13.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.0 (dd, J = 252.2, 3.1 Hz), 133.6 (dd, J = 10.5, 8.7 Hz), 130.7 (d, J = 7.9 Hz), 130.1 (d, J = 5.2 Hz), 128.5 (d, J = 2.5 Hz), 128.0 (dd, J = 103.2, 3.4 Hz), 127.0 (d, J = 3.0 Hz), 116.0 (dd, J = 21.3, 12.7 Hz), 38.4 (d, J = 67.1 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 28.6. MS (EI): *m/z* (%) 328 (16), 327 (33), 238 (13), 237 (100), 189 (4), 95 (9), 91 (10), 77 (4), 75 (6), 65 (7). HRMS Calcd for $C_{19}H_{14}F_2OP$: 327.0829; found: 327.0747 [M-H].

*Benzylbis(4-chlorophenyl)phosphine oxide (**3u**):*[8] White solid (25.9 mg, 36%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62-7.57 (m, 4H), 7.42 (db, J = 7.6 Hz, 4H), 7.27-7.20 (m, 3H), 7.09 (b, 2H), 3.63 (d, J = 13.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.6 (d, J = 3.4 Hz), 132.5 (d, J = 9.9 Hz), 130.3 (d, J = 99.8 Hz), 130.3 (d, J = 8.0 Hz), 130.0 (d, J = 5.4 Hz), 129.0 (d, J = 12.2 Hz), 128.5 (d, J = 2.6 Hz), 127.1 (d, J = 2.9 Hz), 38.0 (d, J = 66.9 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 28.5.

References

1. Montel, S.; Jia, T.; Walsh, P. J. *Org. Lett.* **2014**, *16*, 130.

2. Wang, F.; Qu, M.; Chen, F.; Xu, Q.; Shi, M. *Chem. Commun.* **2012**, *48*, 8580.

3. Shutt, J. R.; Trippett, S. *J. Chem. Soc. C.* **1969**, 2038.

4. Chen, Z.-S.; Zhou, Z.-Z.; Hua, H.-L.; Duan, X.-H.; Luo, J.-Y.; Wang, J.; Zhou, P.-X.; Liang, Y.-M. *Tetrahedron* **2013**, *69*, 1065.

5. Yang, J.; Chen, T.; Han, L.-B. *J. Am. Chem. Soc.* **2015**, *137*, 1782.

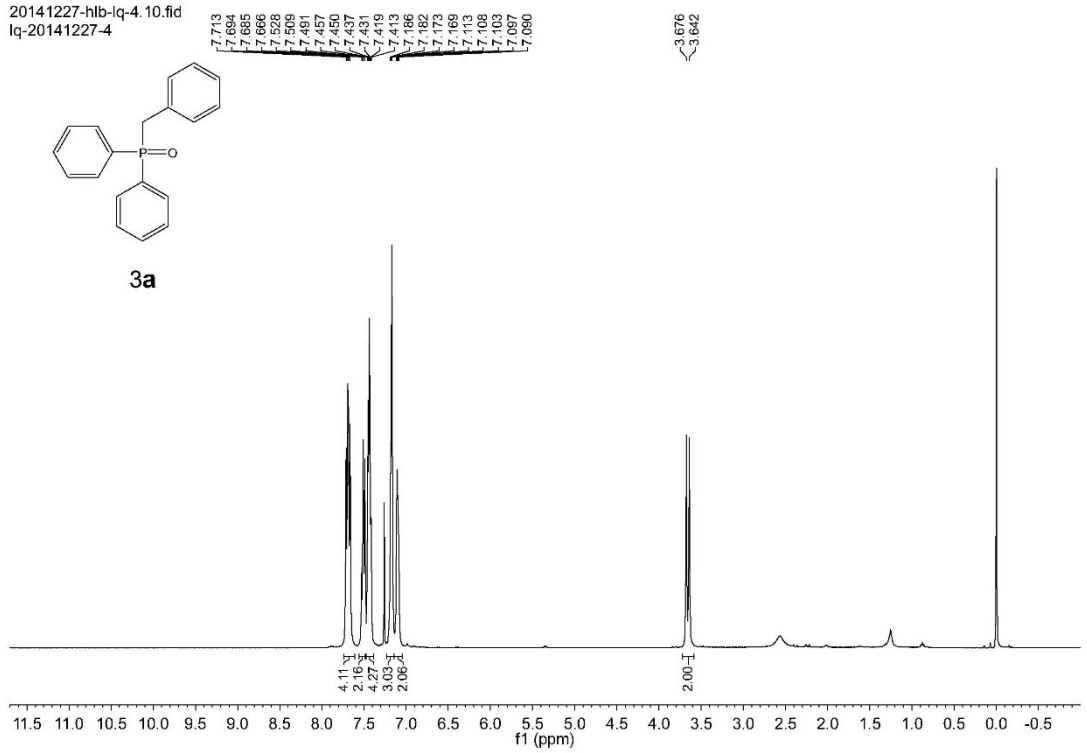
6. Uffmann, T.; Joussen, R. *Chem. Ber.* **1977**, *110*, 3930.

7. Tsvetkov, E. N.; Shvets, A. A.; Korolev, B. A.; Bondarenko, N. A.; Goncharova, L. V.; Osmolovskaya, L. A. *Zh. Obshch. Khim.* **1992**, *62*, 2223.

8. Jugelt, W.; Lamm, W.; Pragst, F. *J. Prakt. Chem.* **1972**, *314*, 193.

Copies of ^{1}H NMR, ^{13}C NMR and ^{31}P NMR spectroscopies

^{1}H NMR



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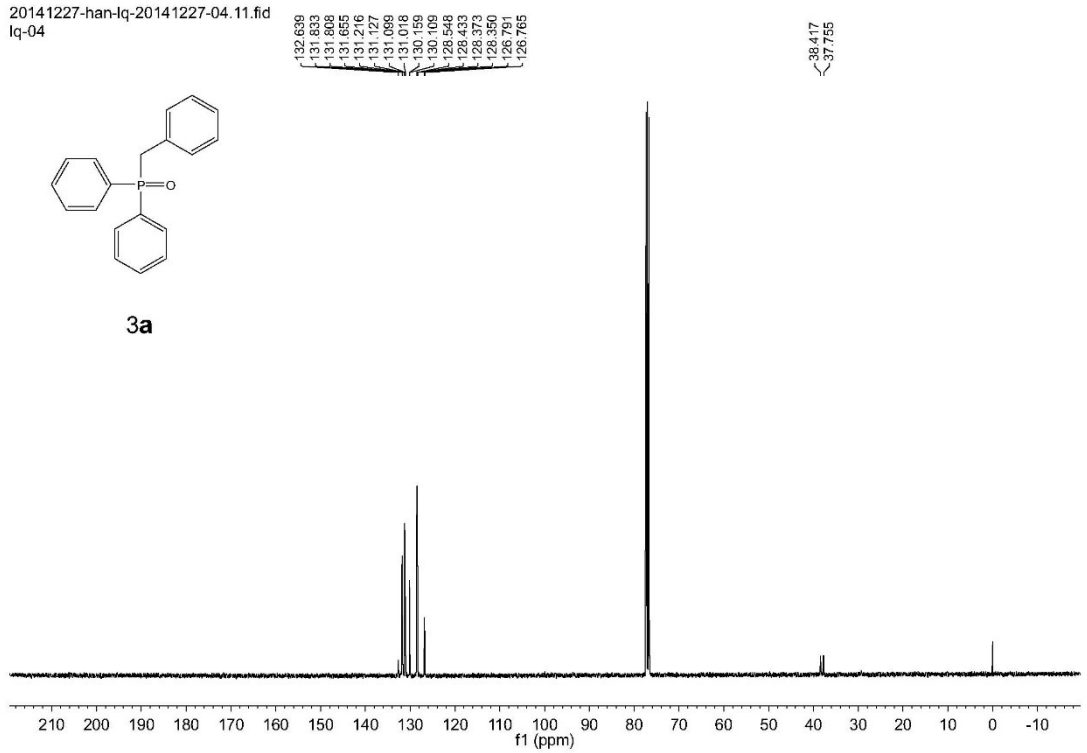

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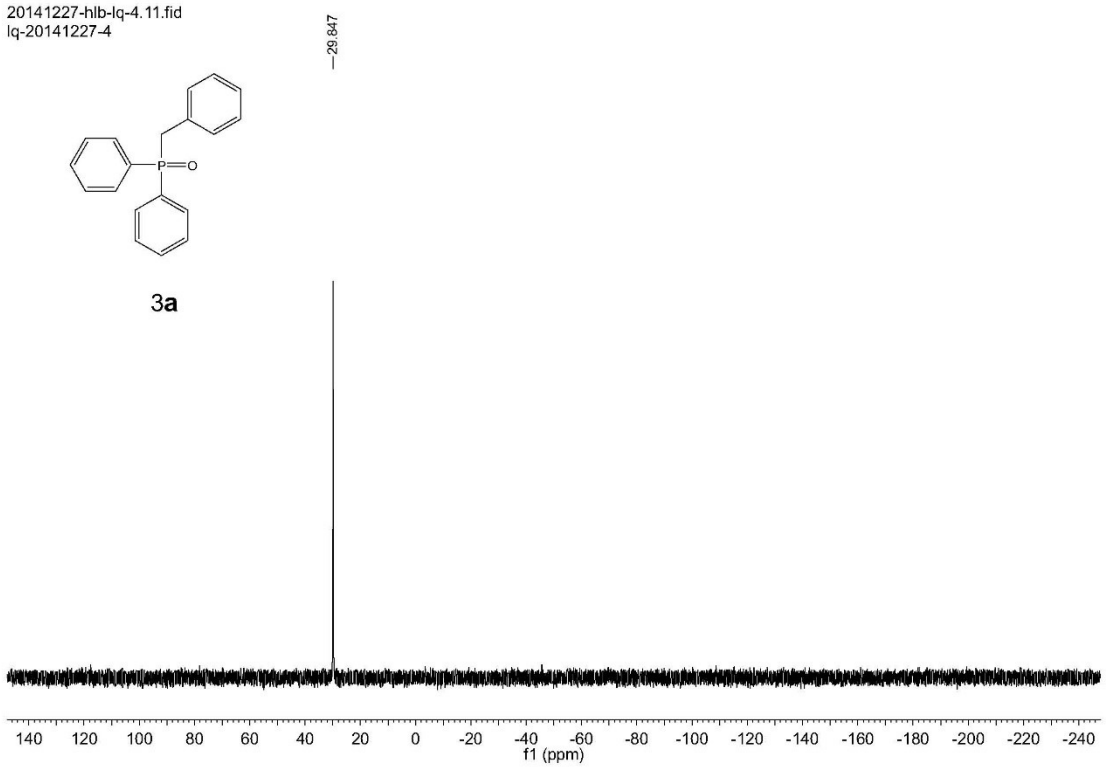

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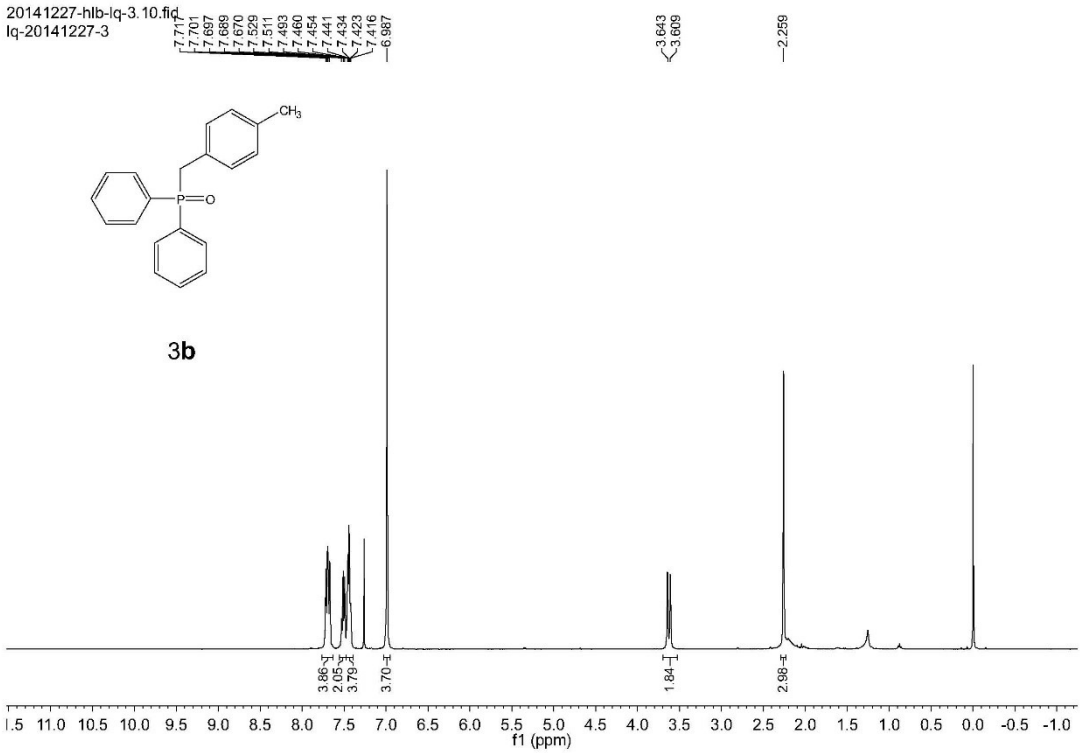
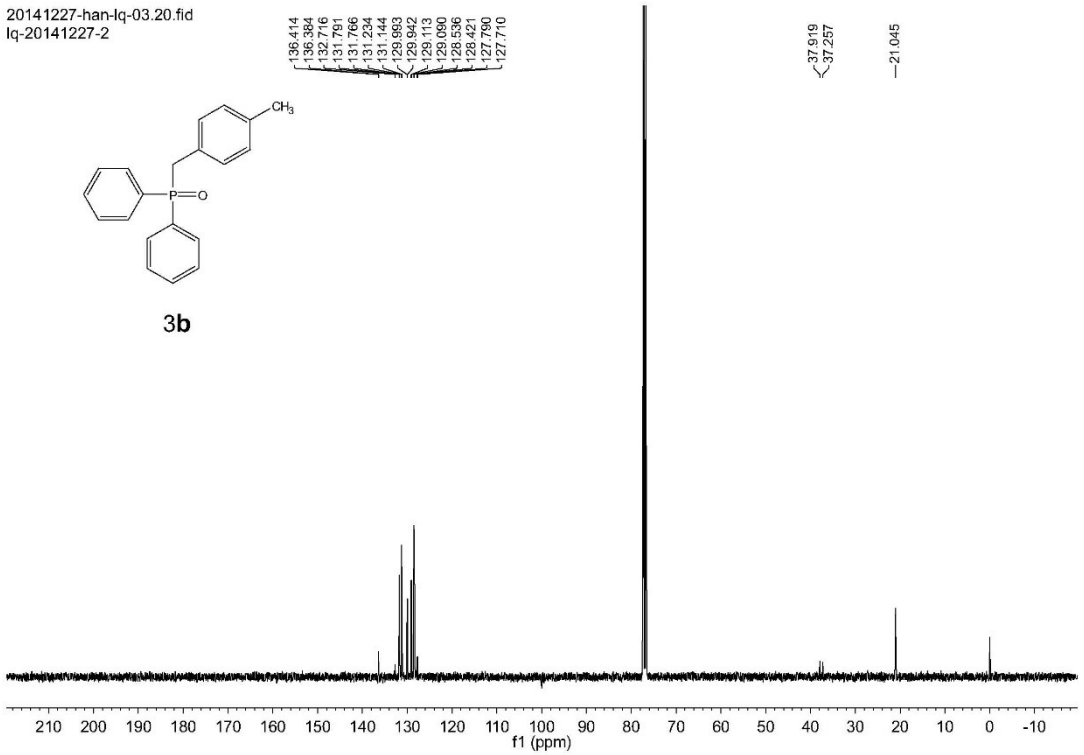
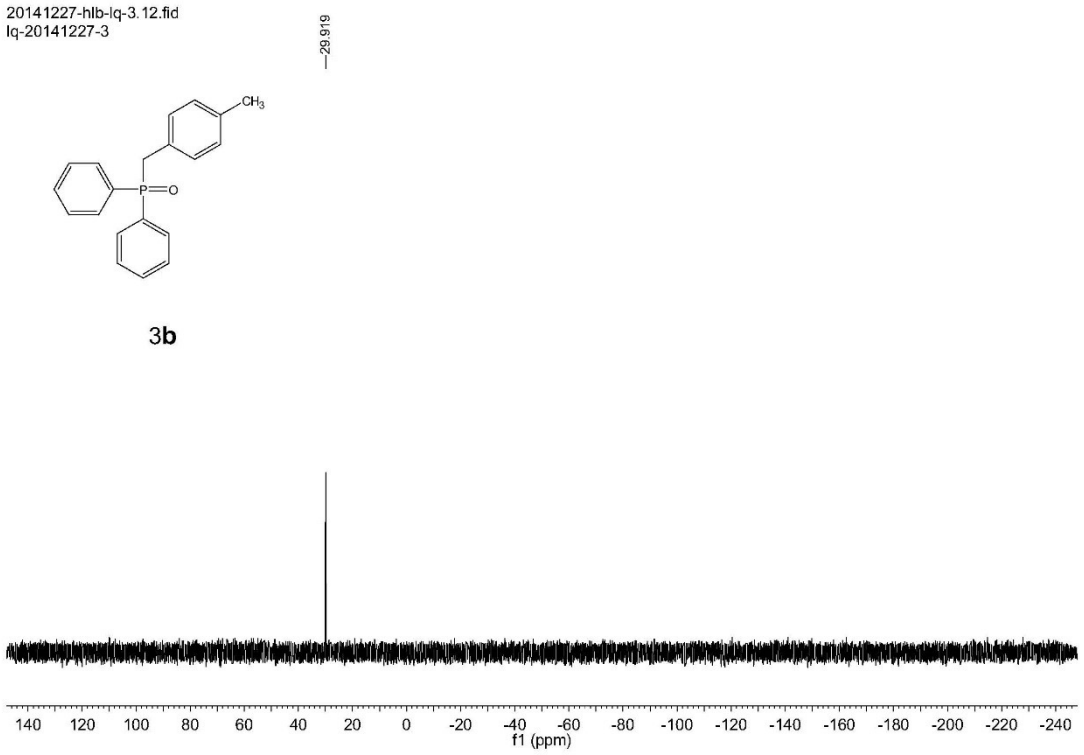


^{13}C NMR



^{31}P NMR



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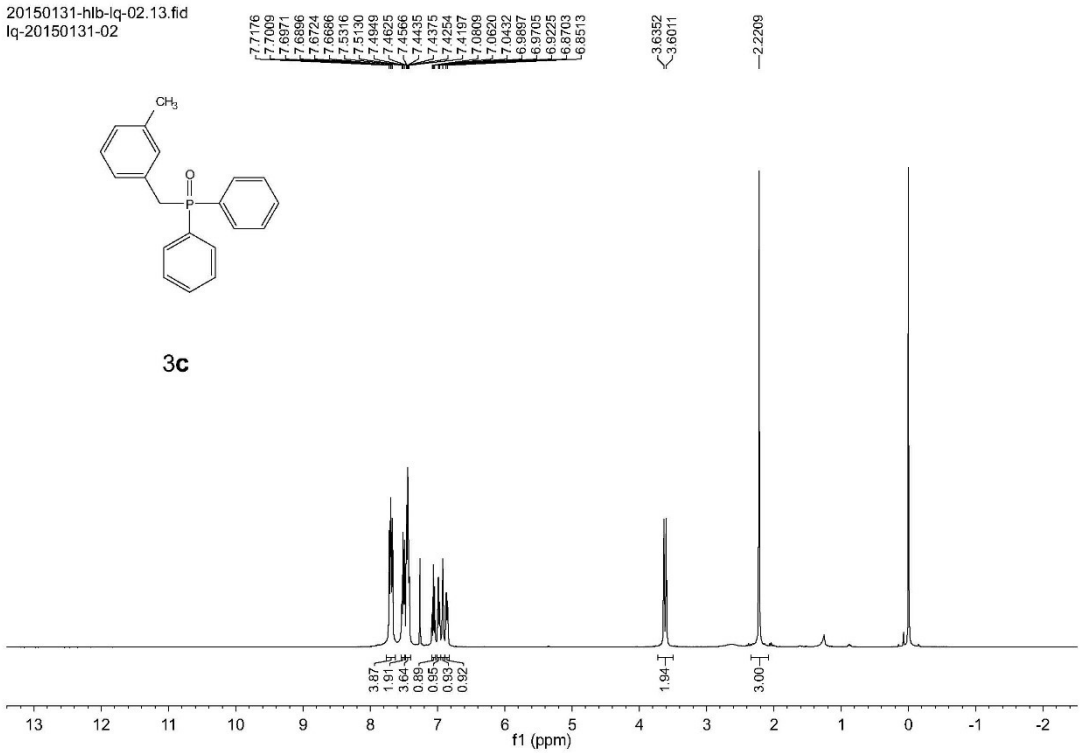

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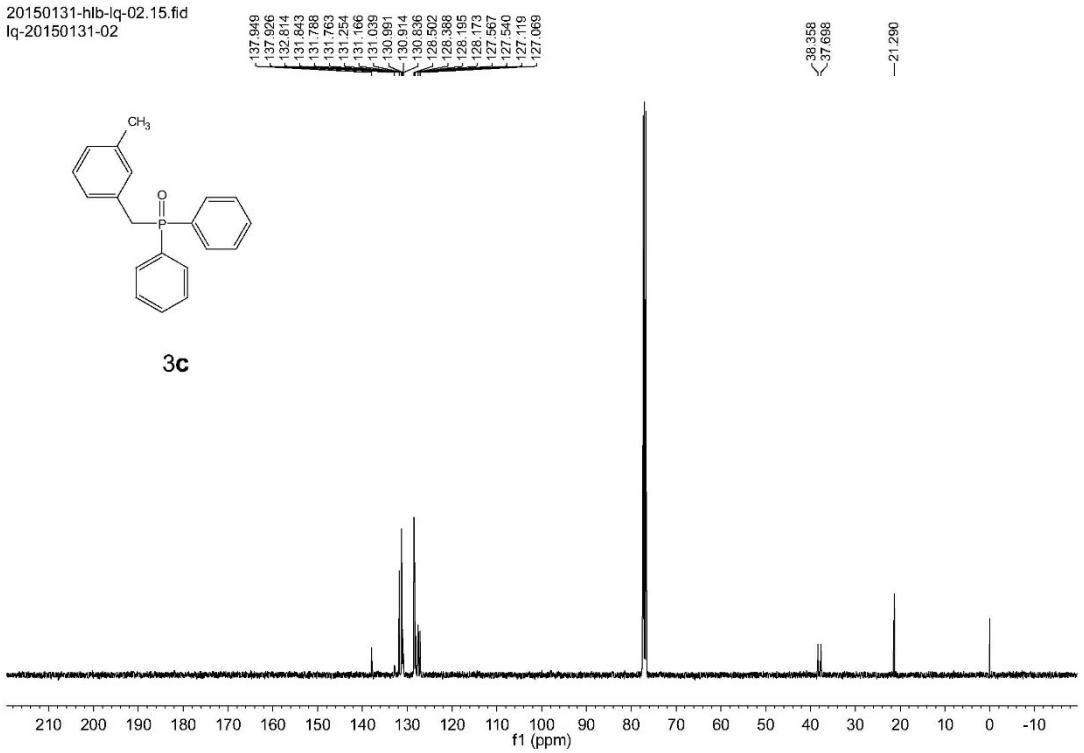

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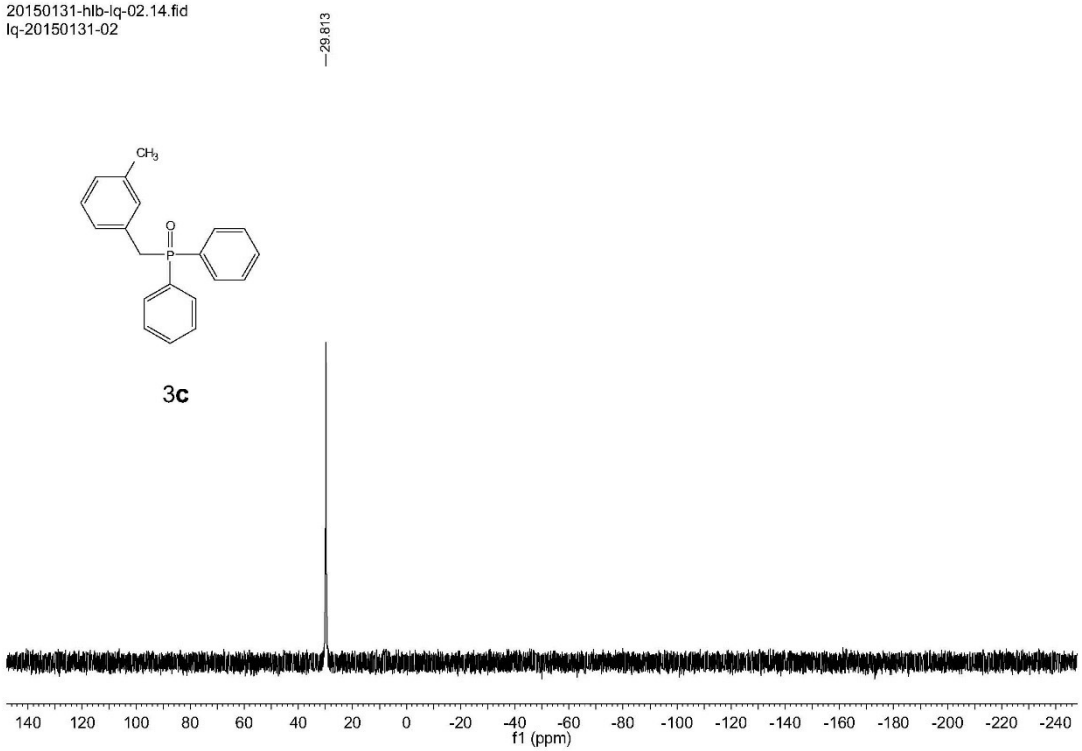

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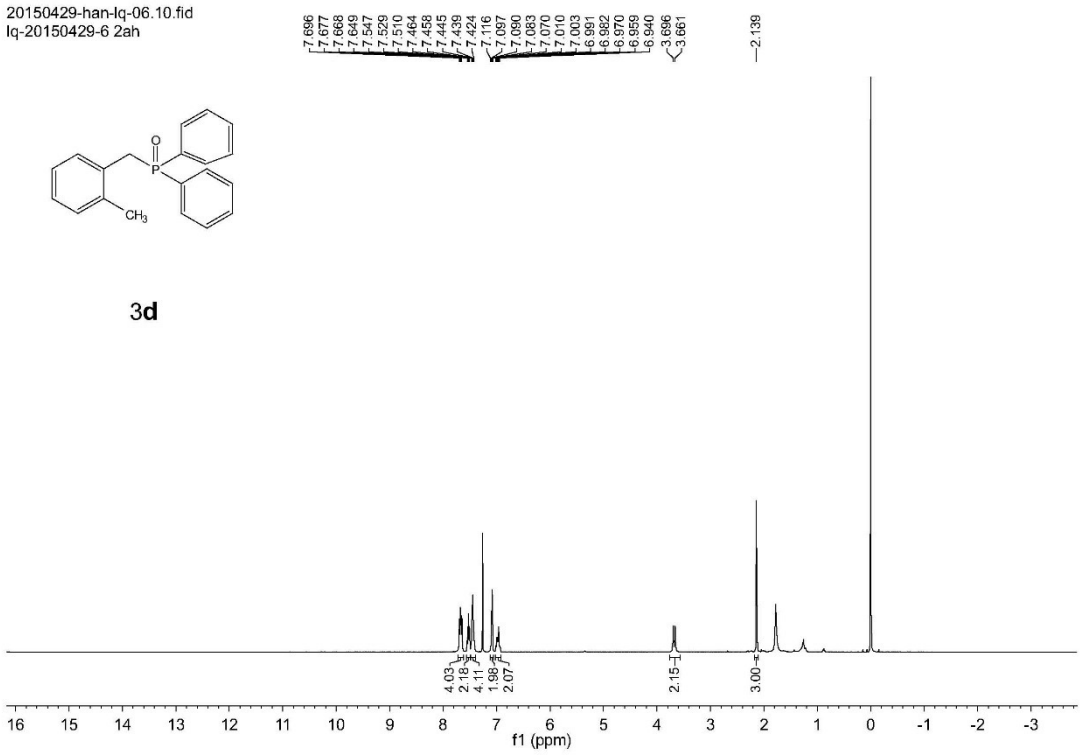

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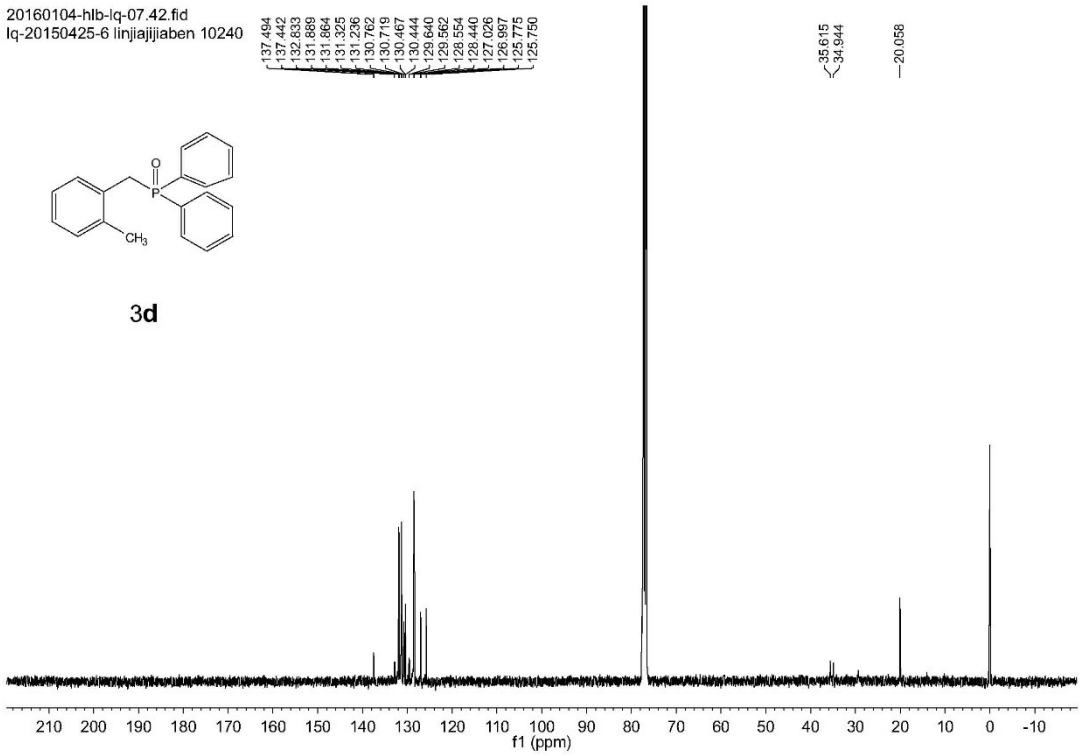

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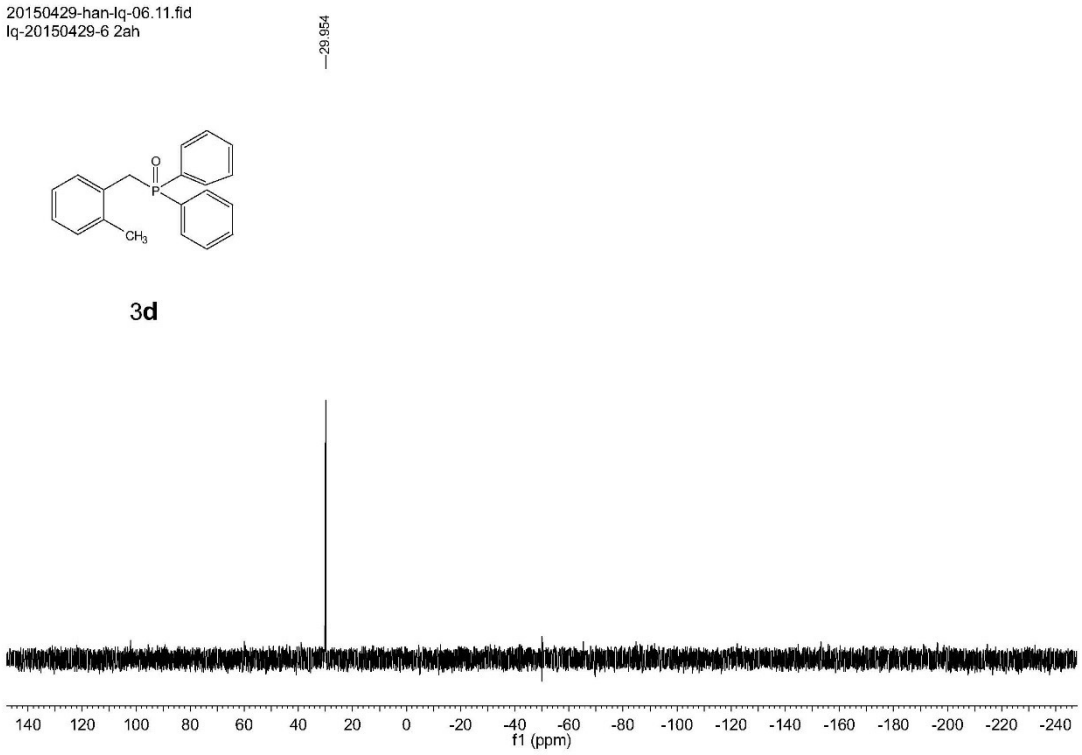

3d

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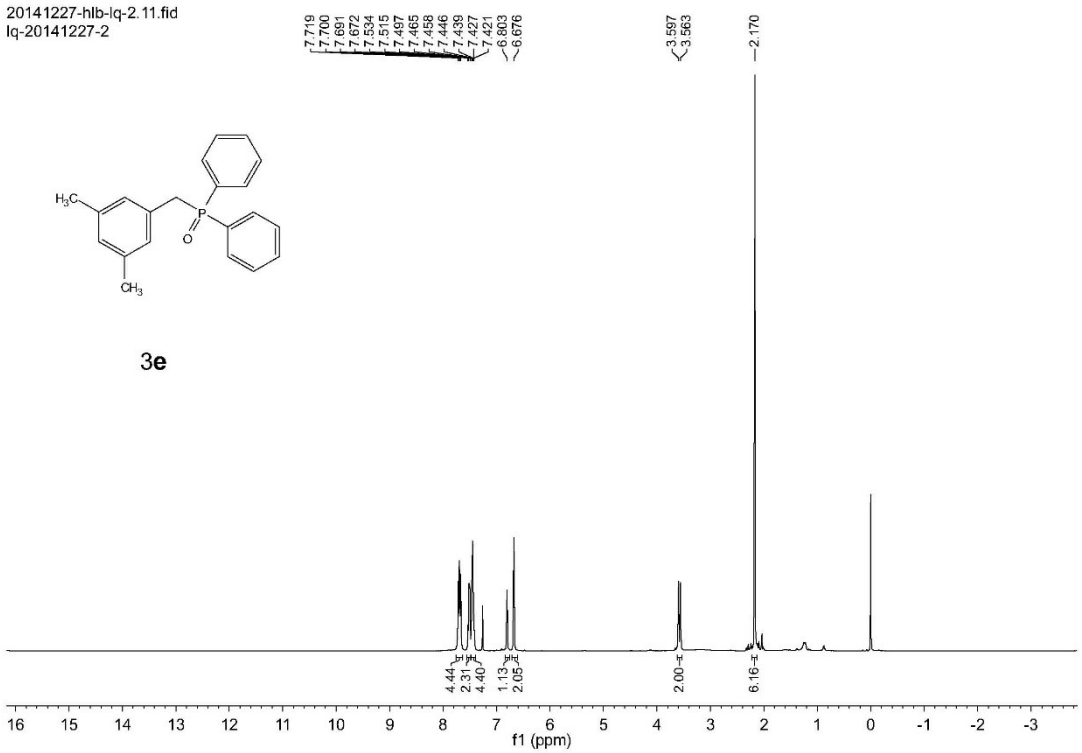

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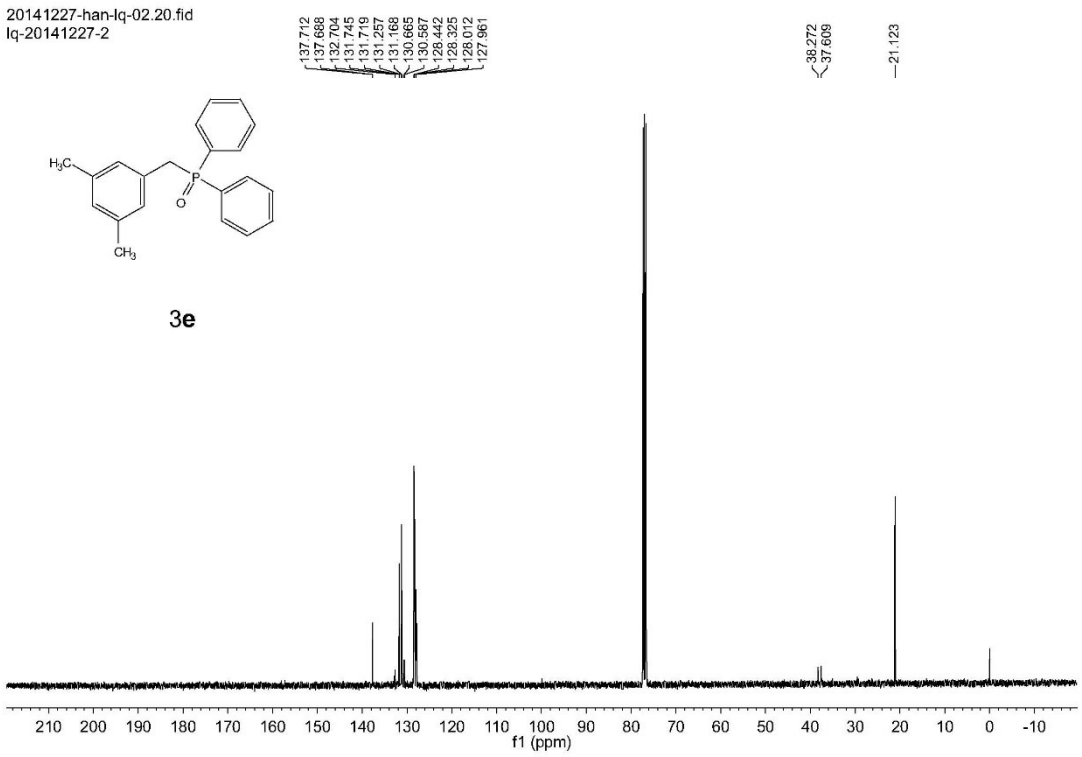

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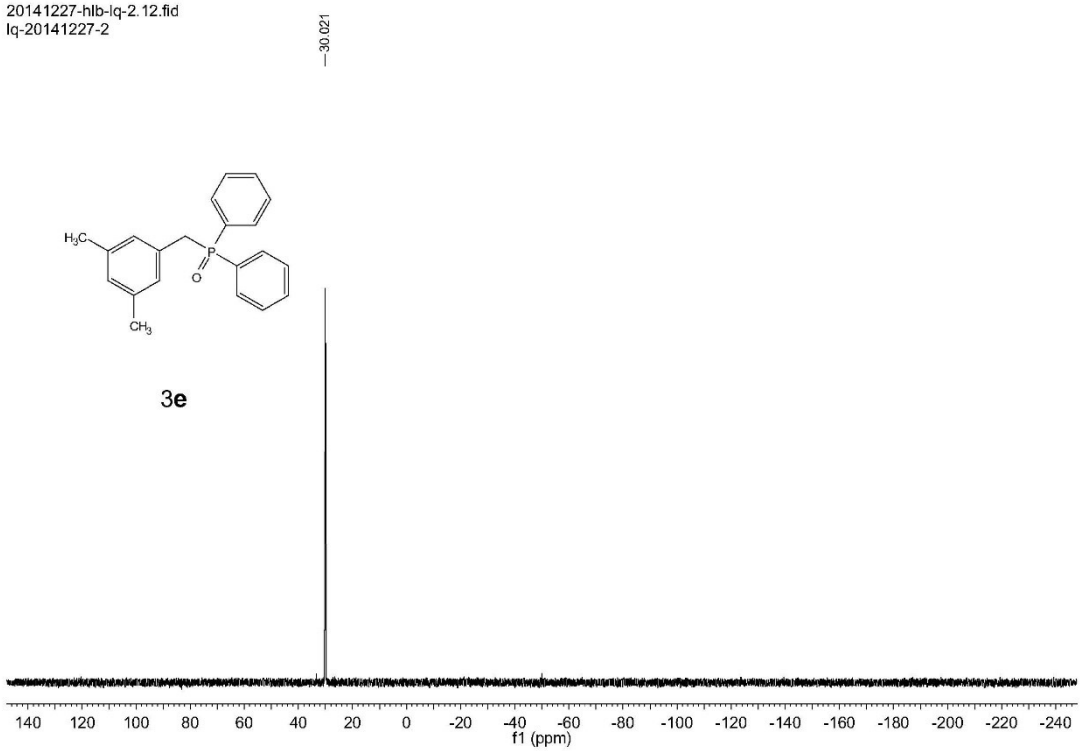

3e

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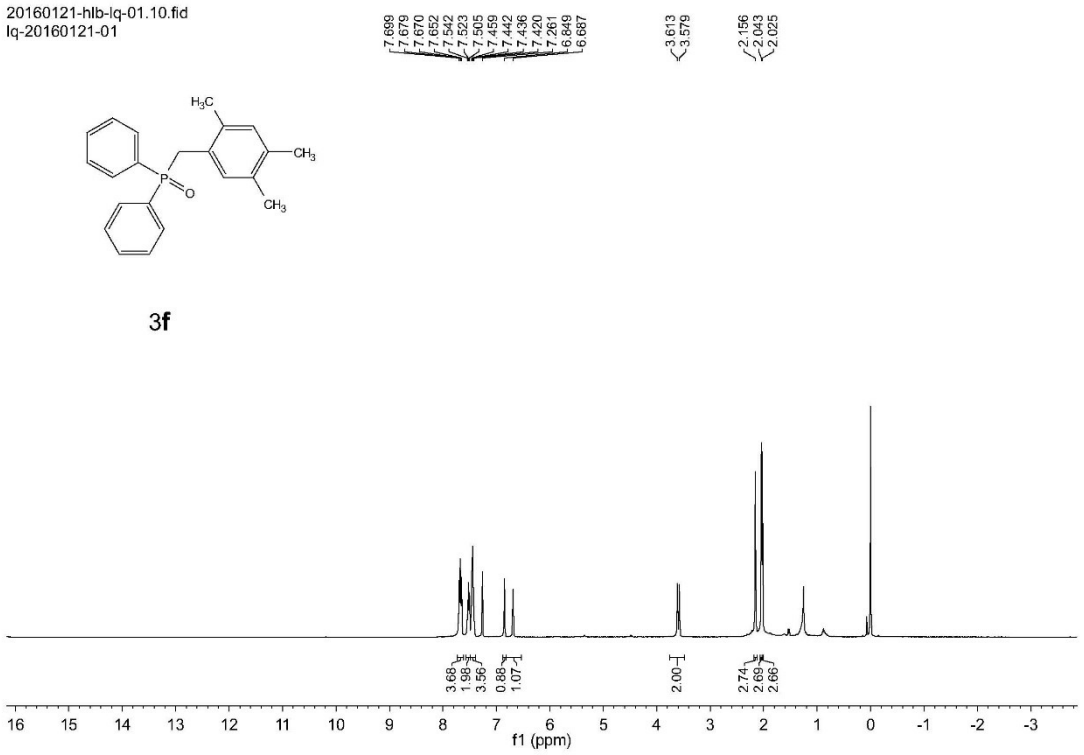

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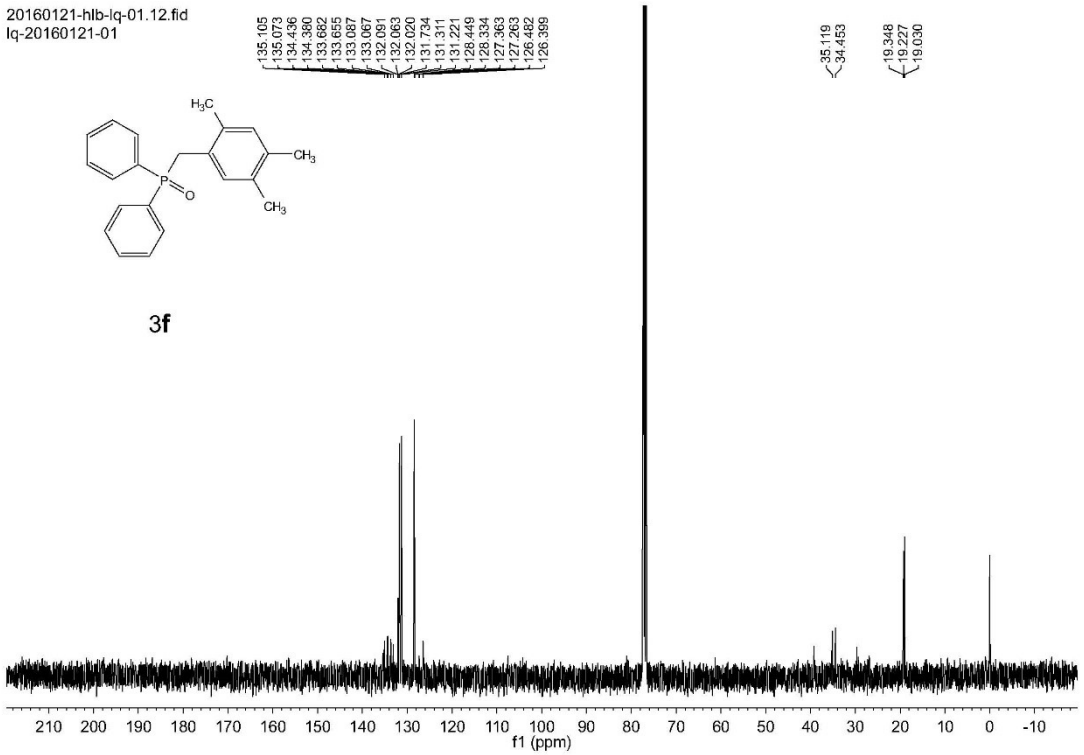

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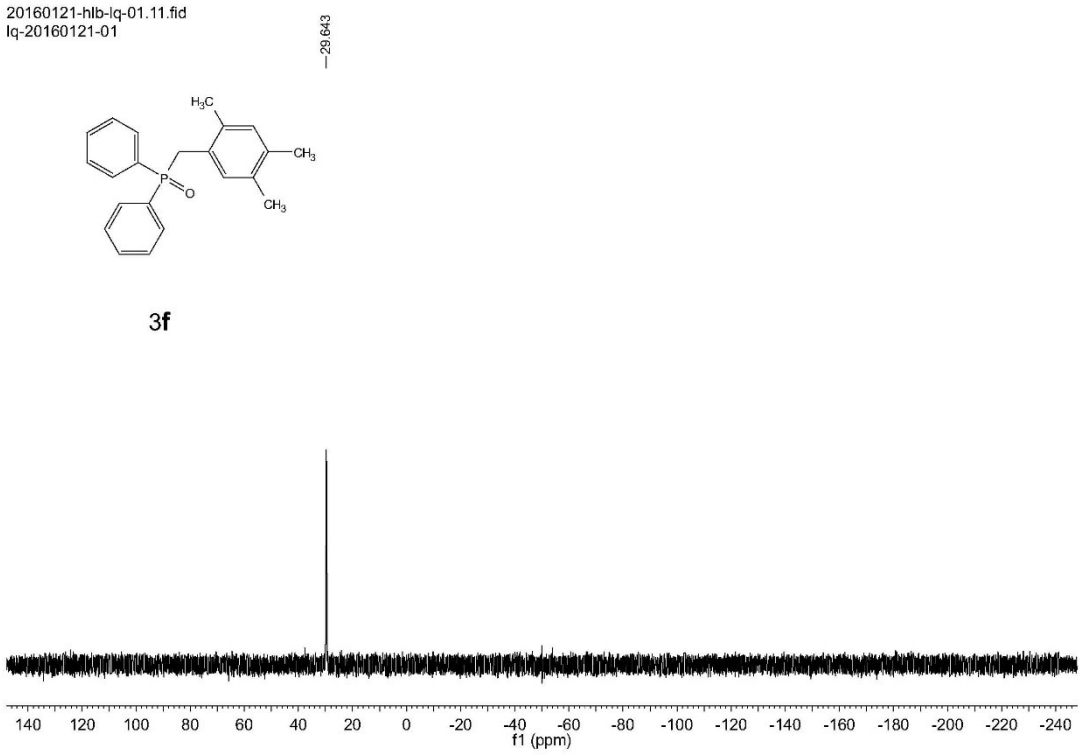

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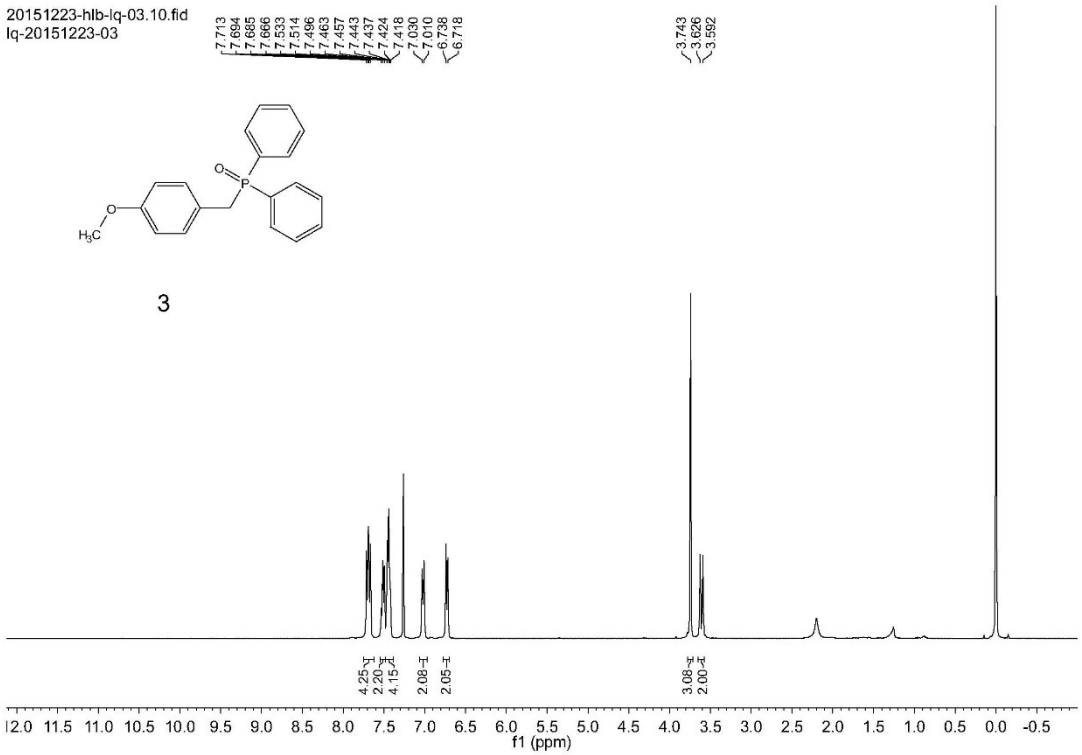

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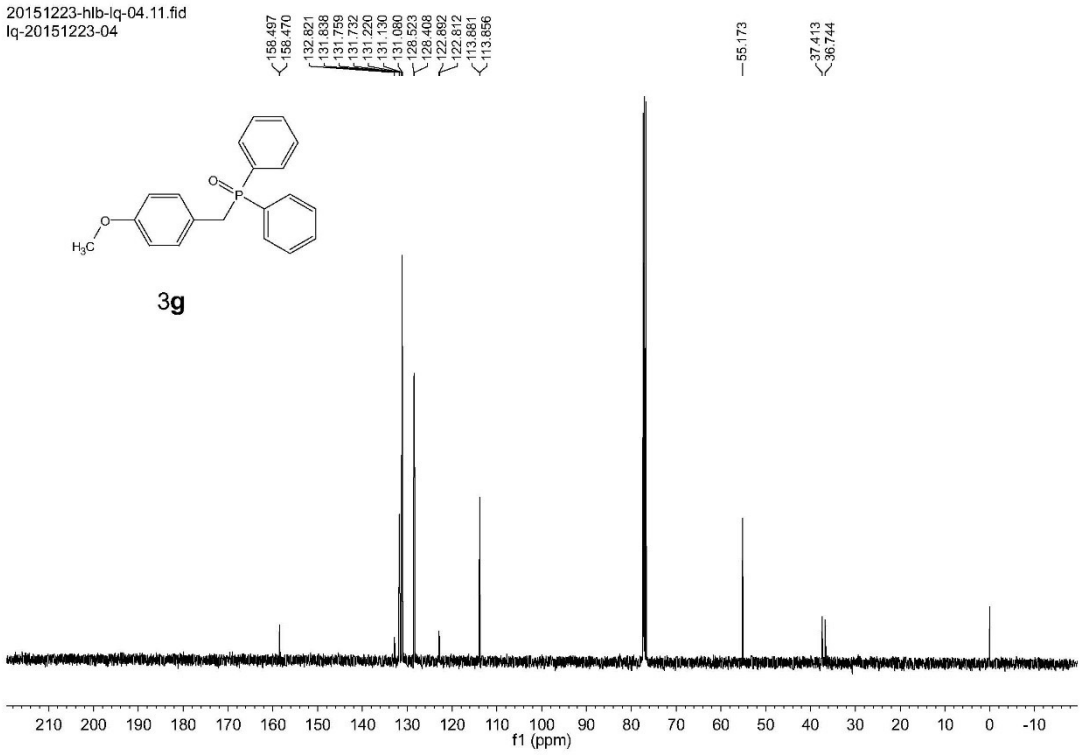

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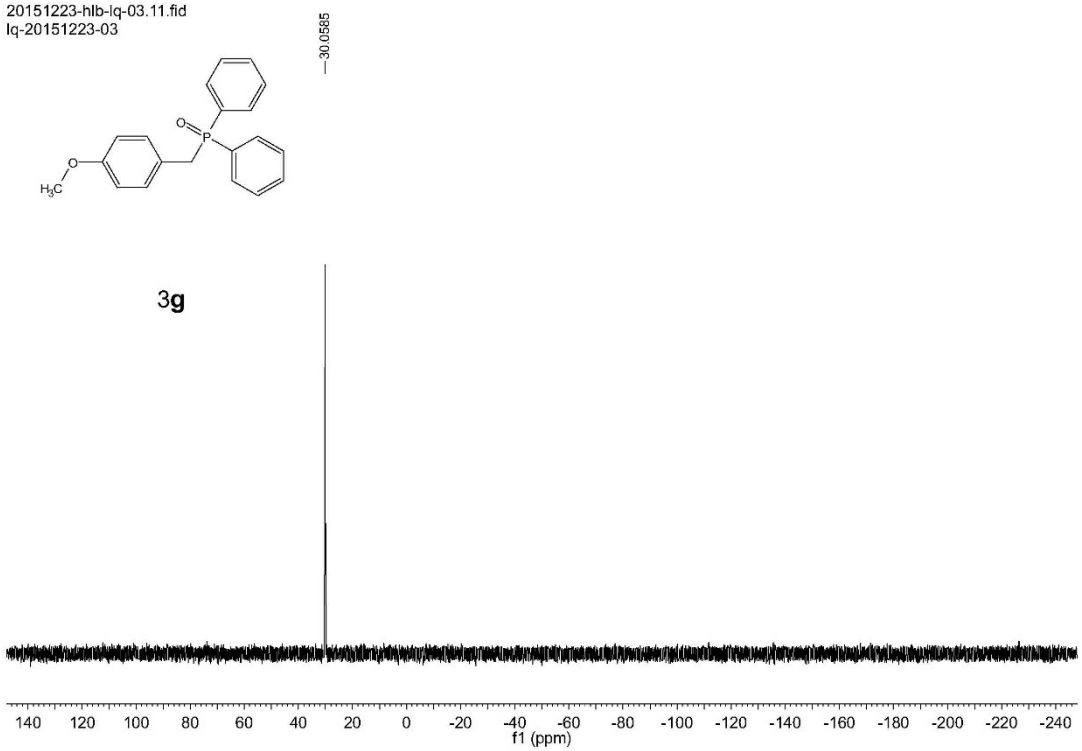

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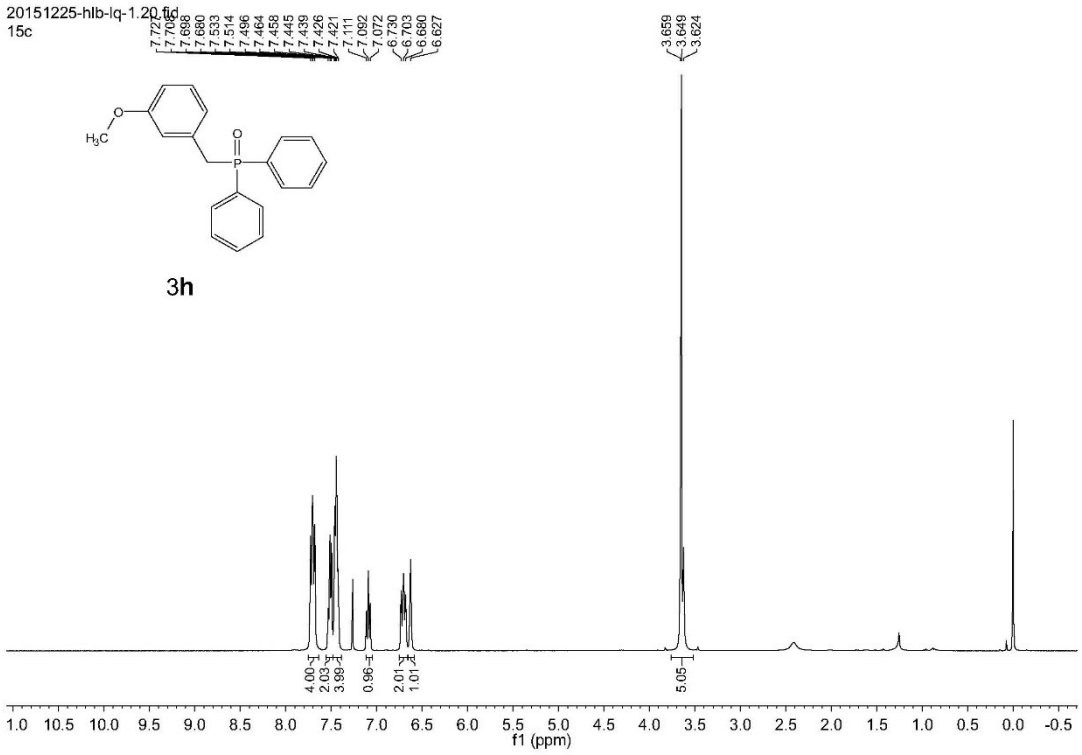

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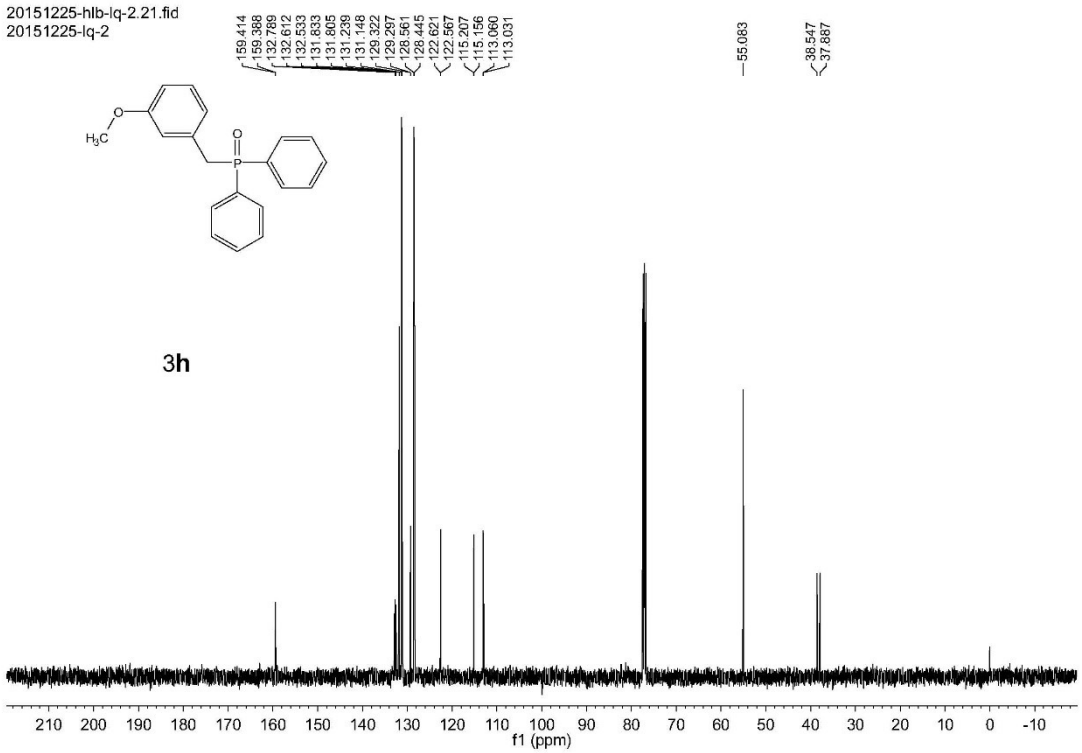

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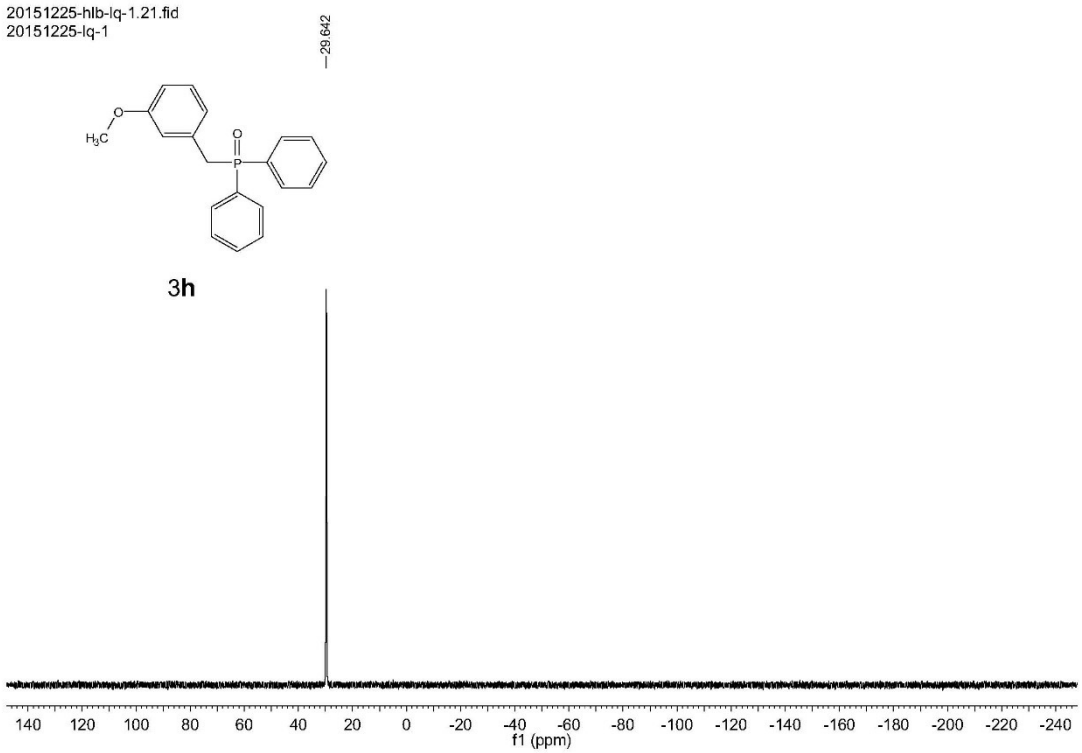

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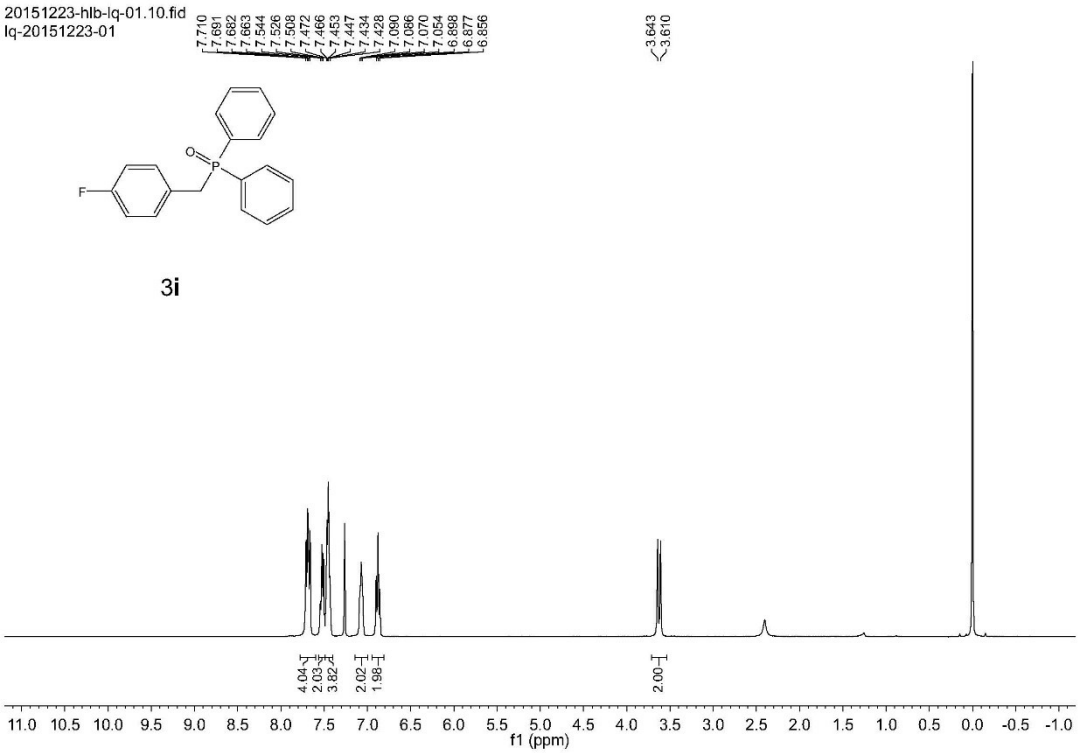

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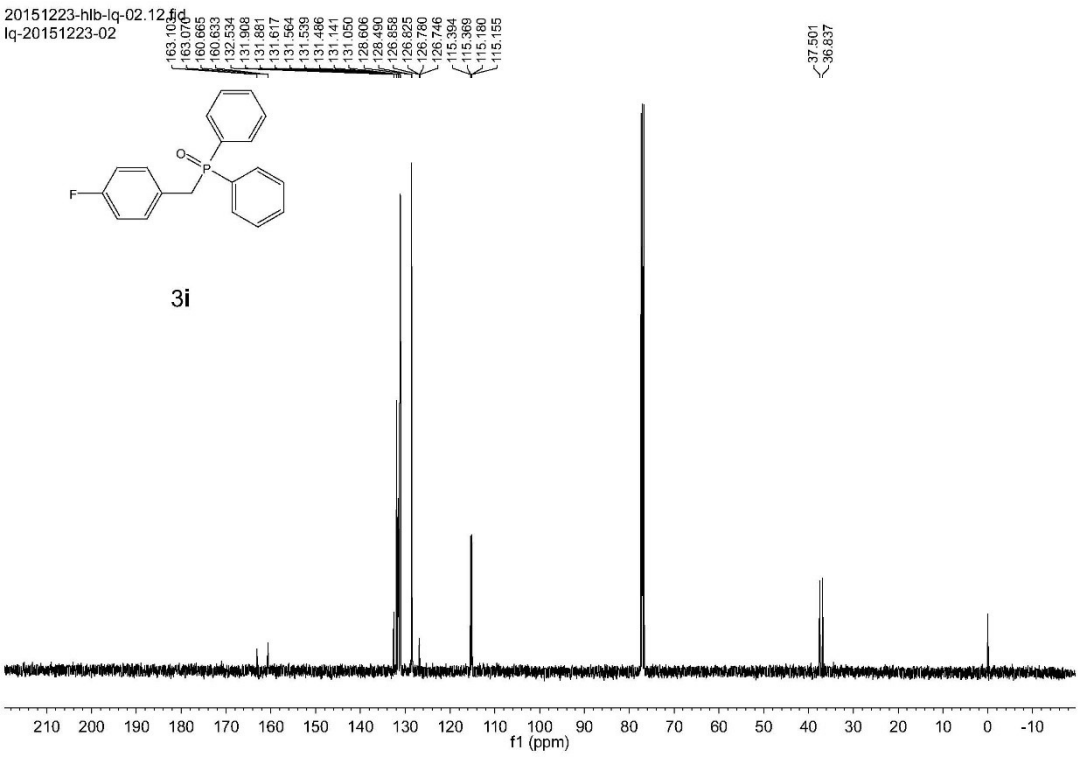

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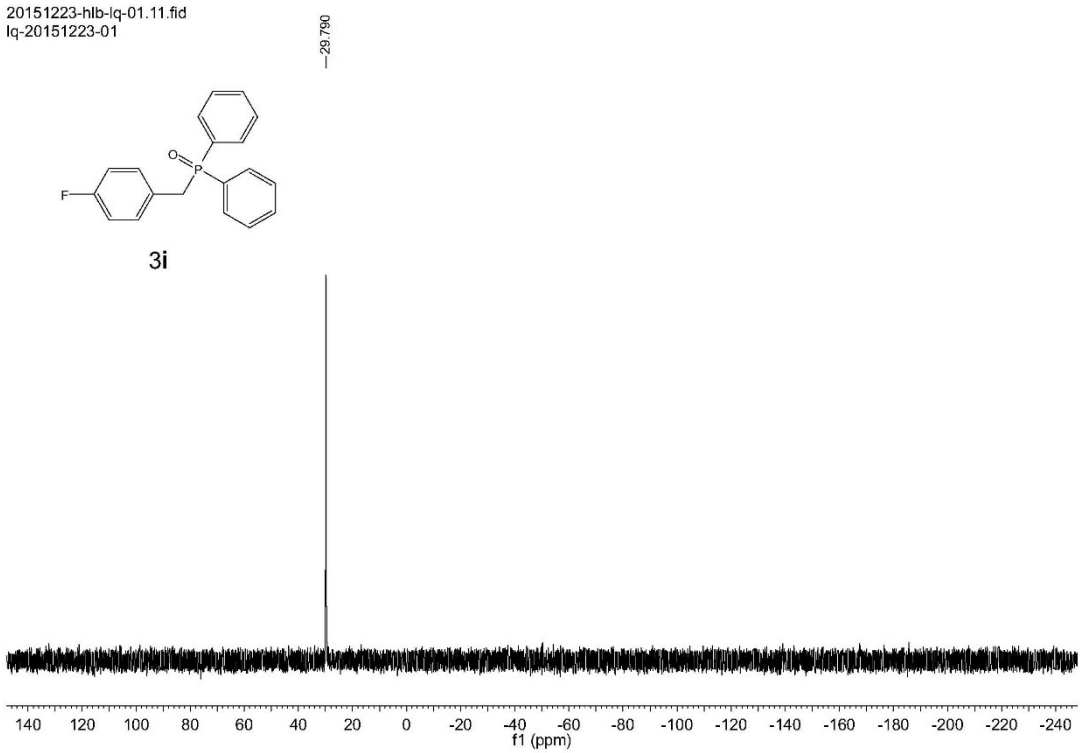

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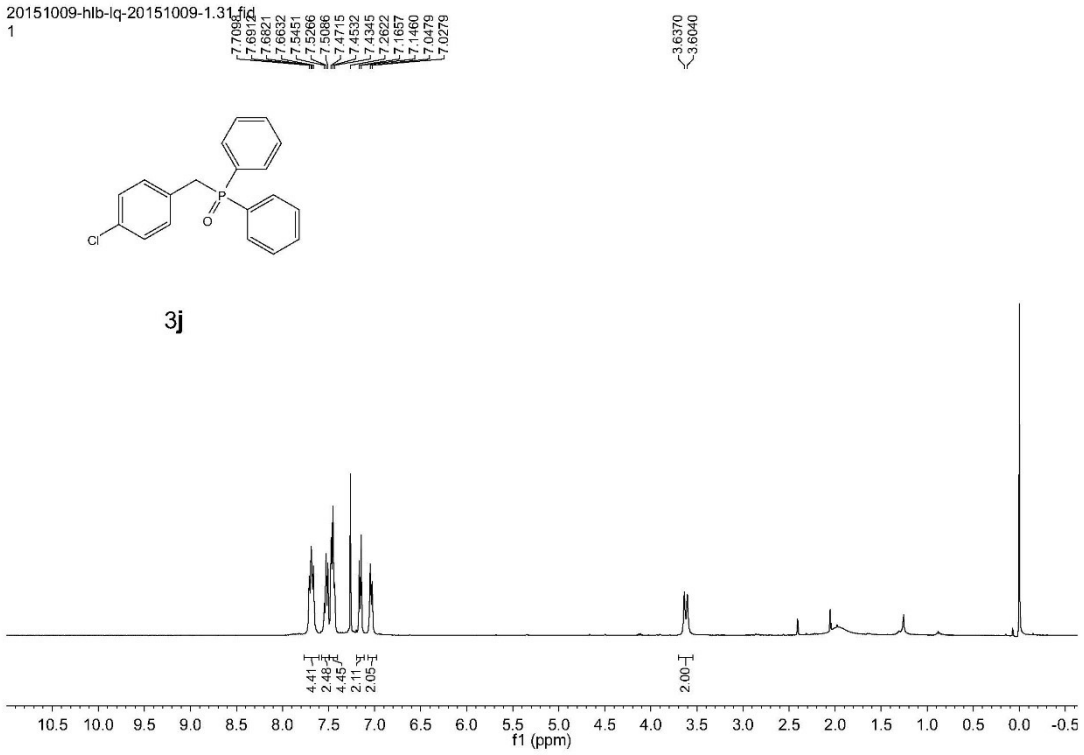

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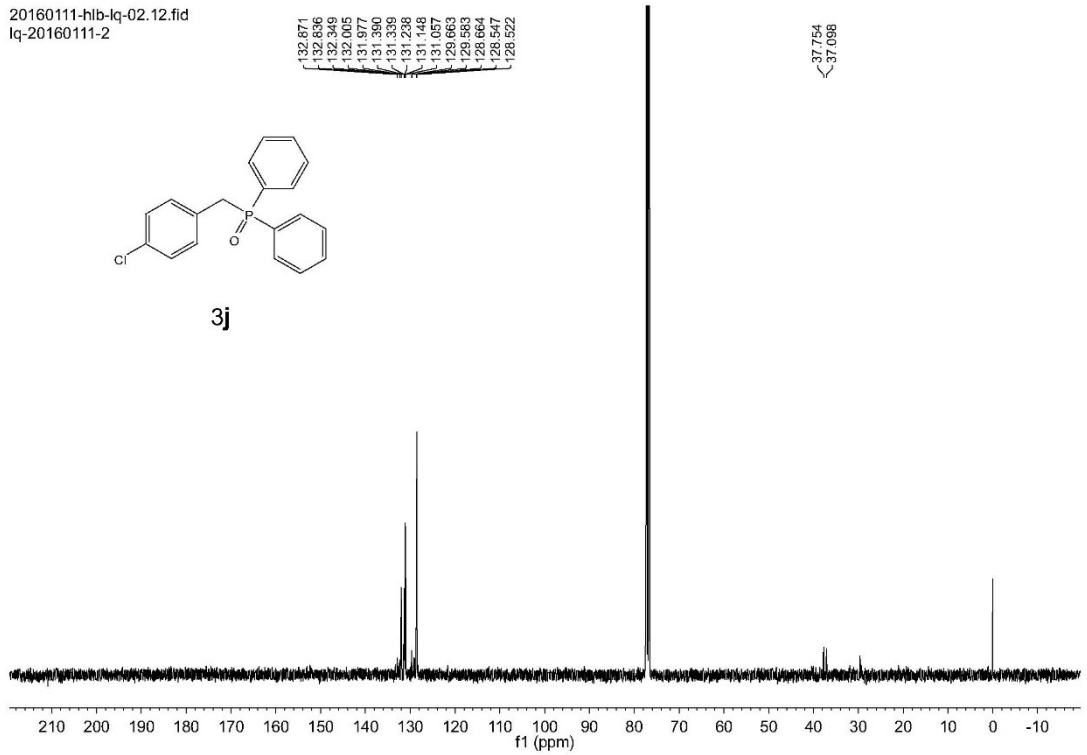

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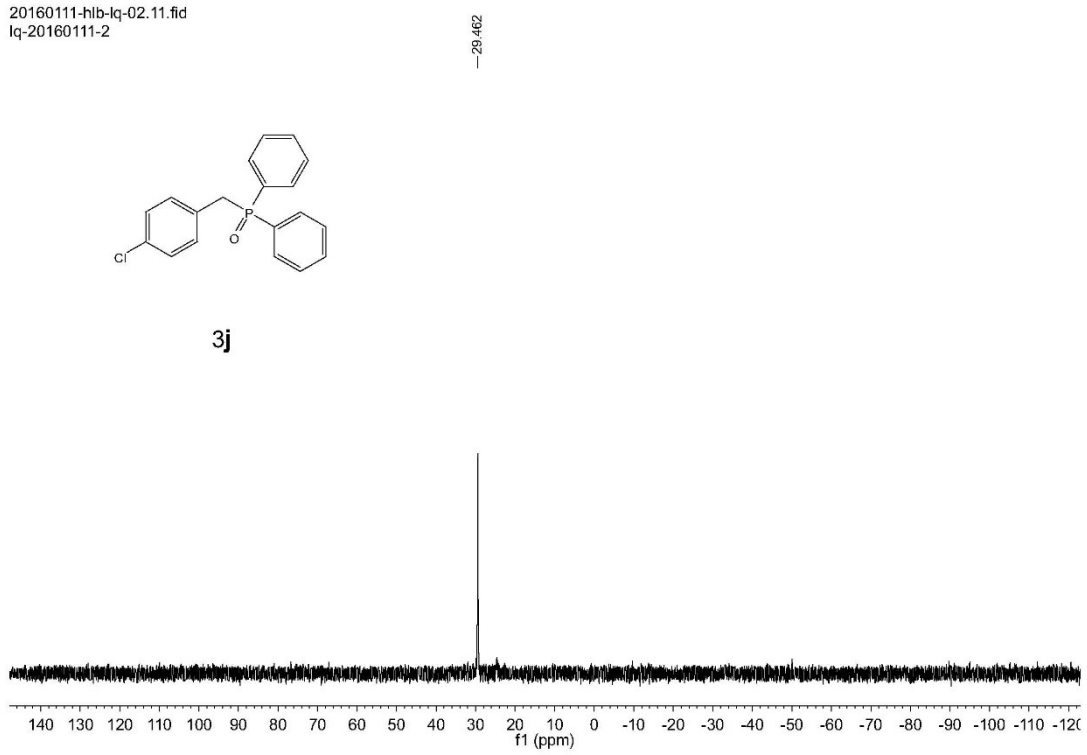

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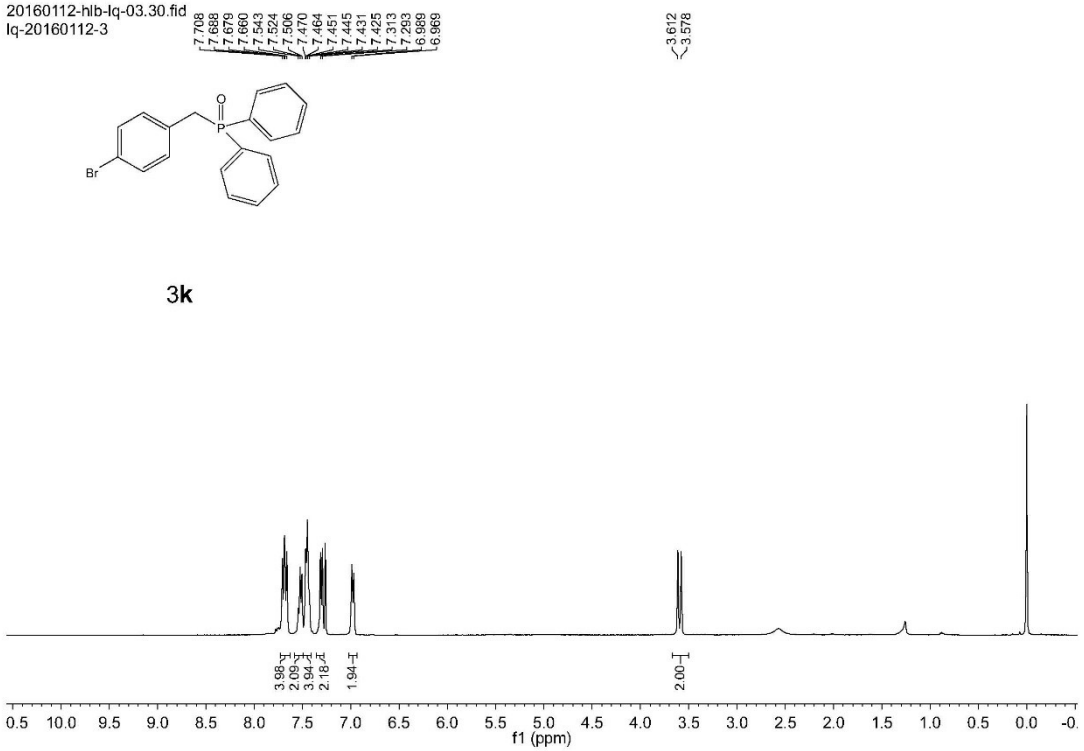
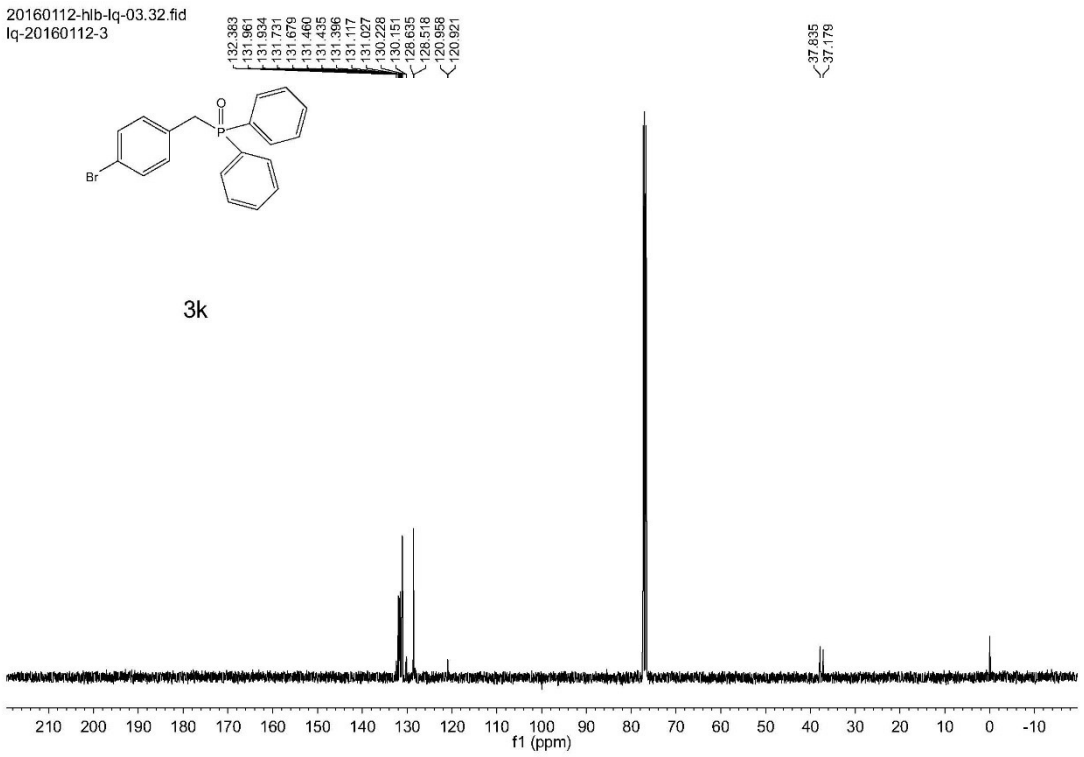


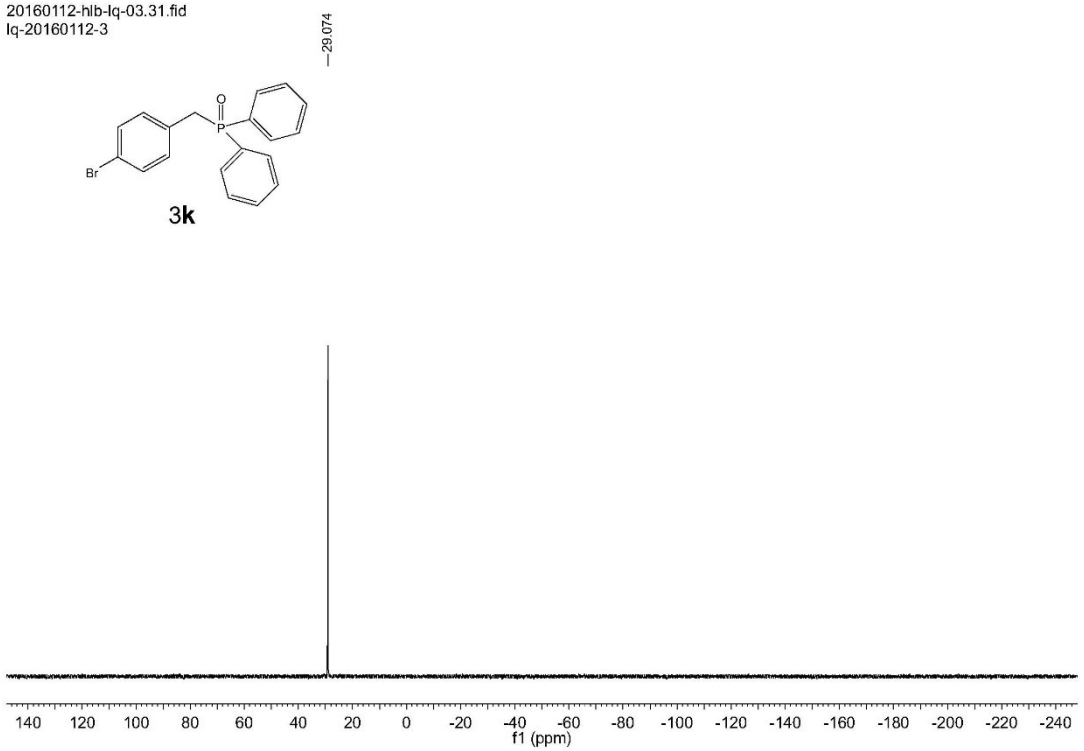
3**k**

^{13}C NMR



3k

^{31}P NMR



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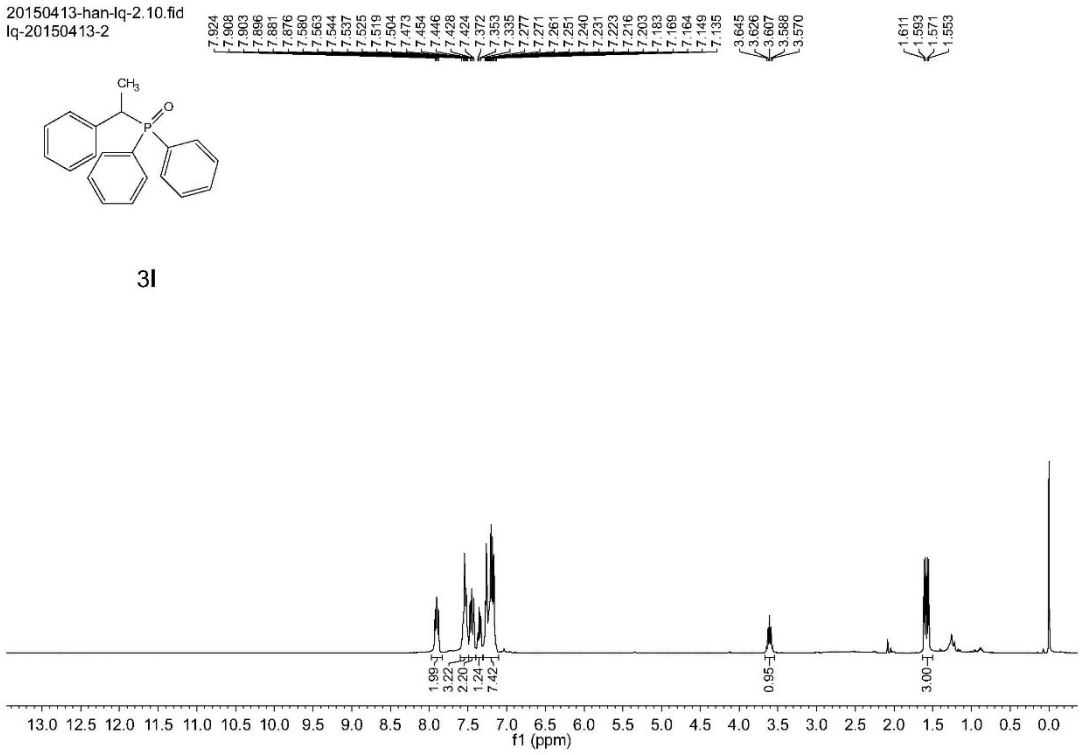

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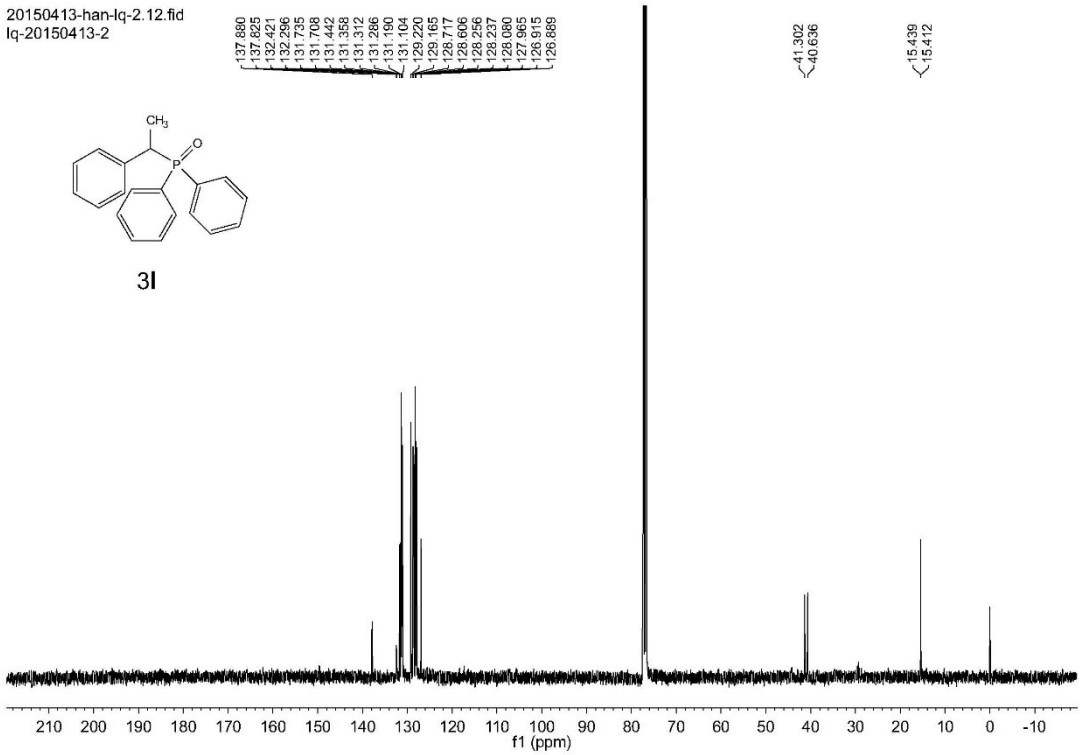

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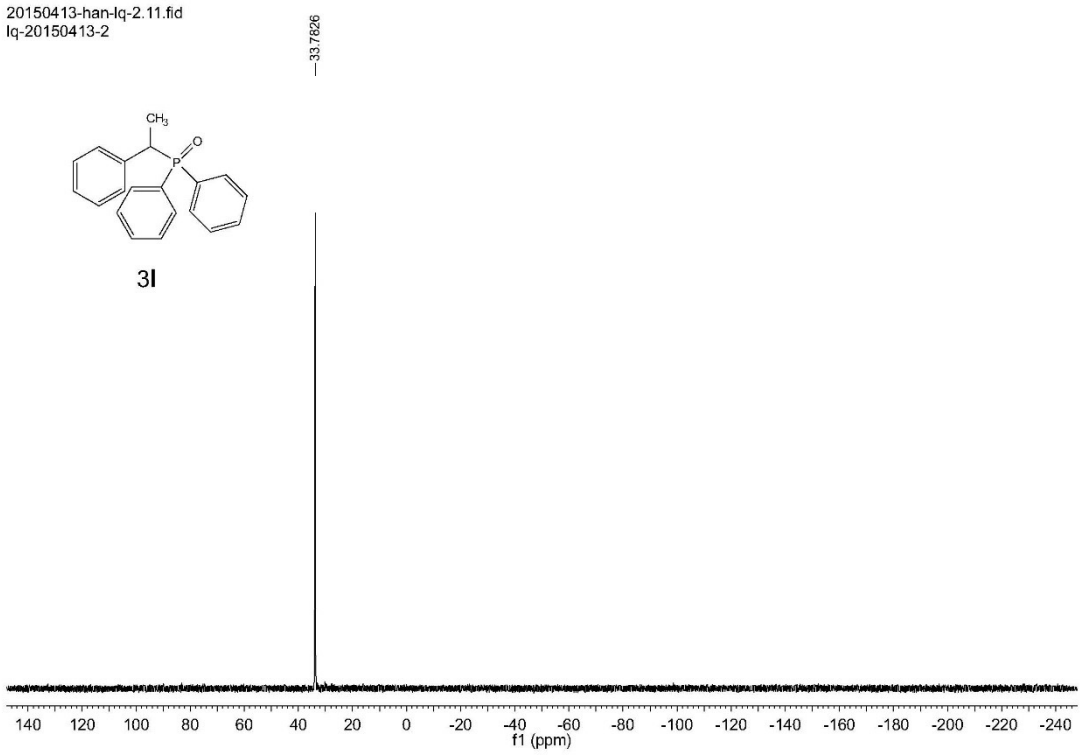

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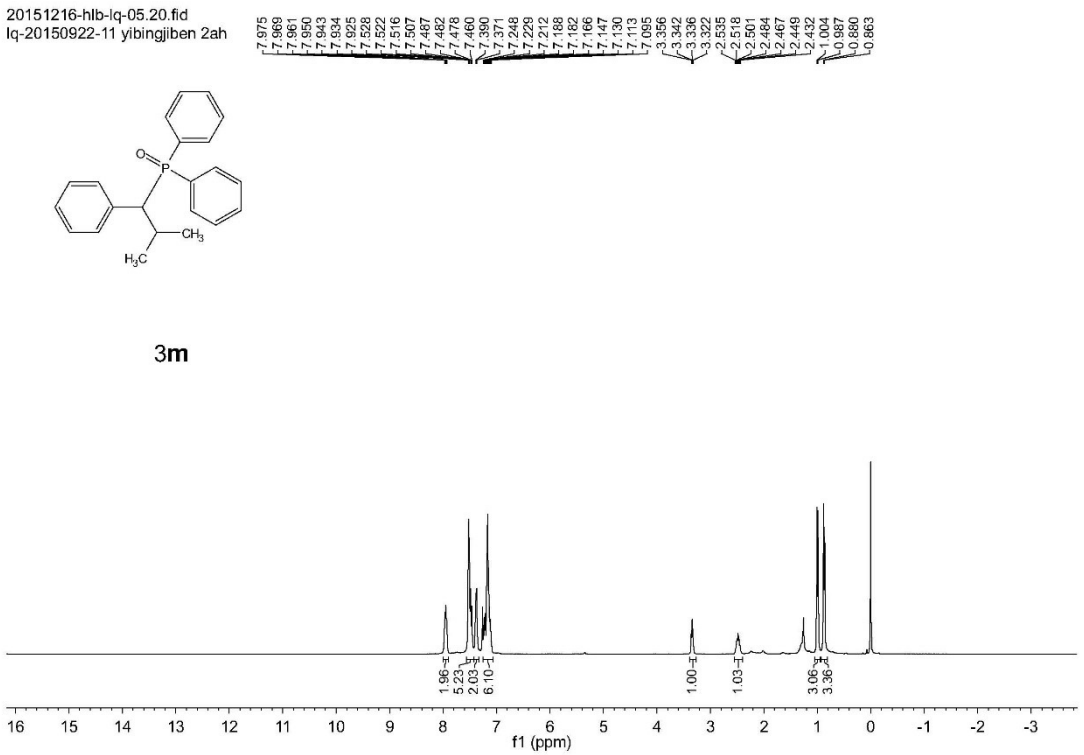
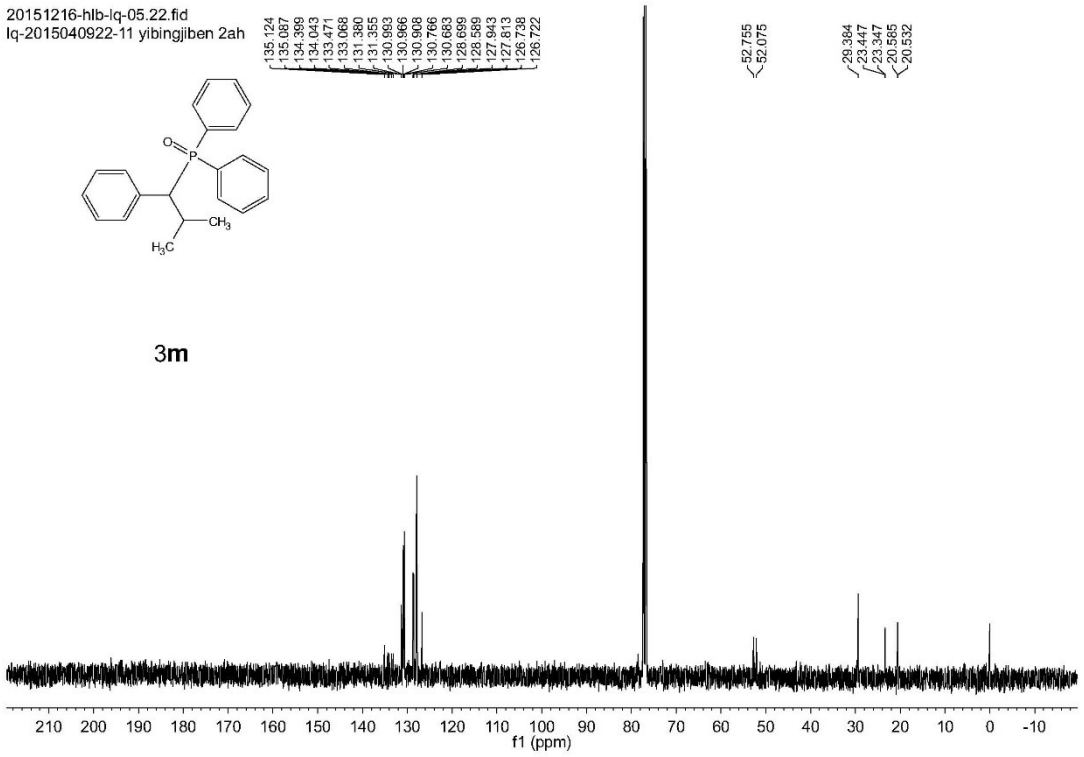


3m

^{13}C NMR



3m

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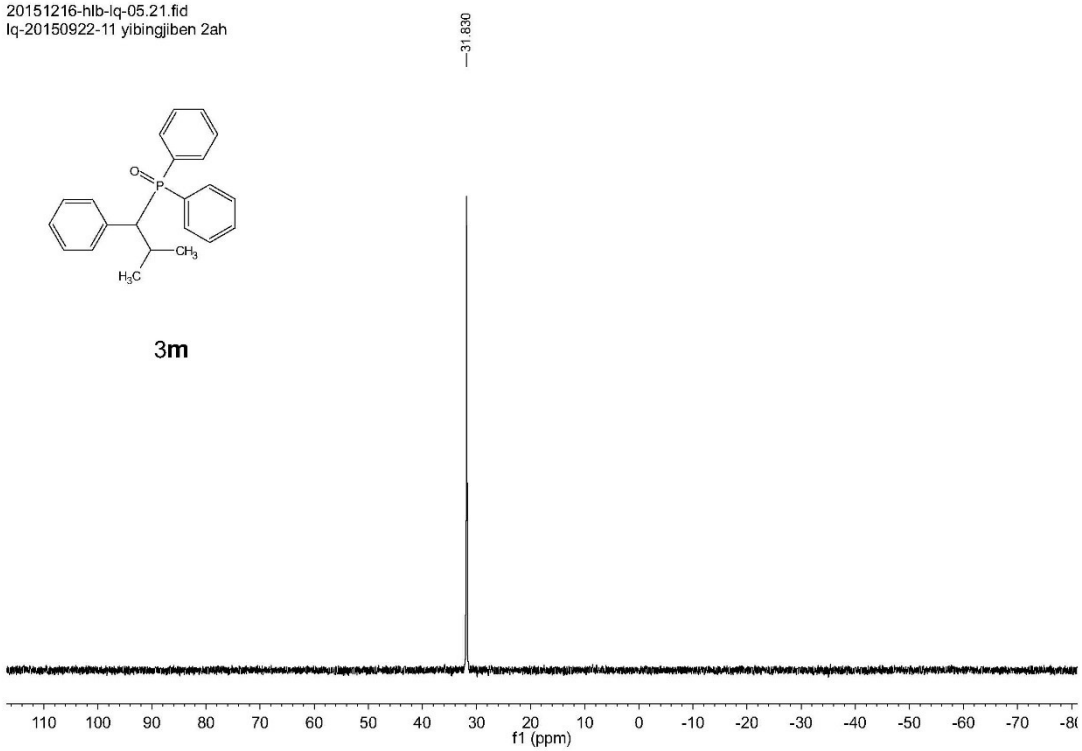

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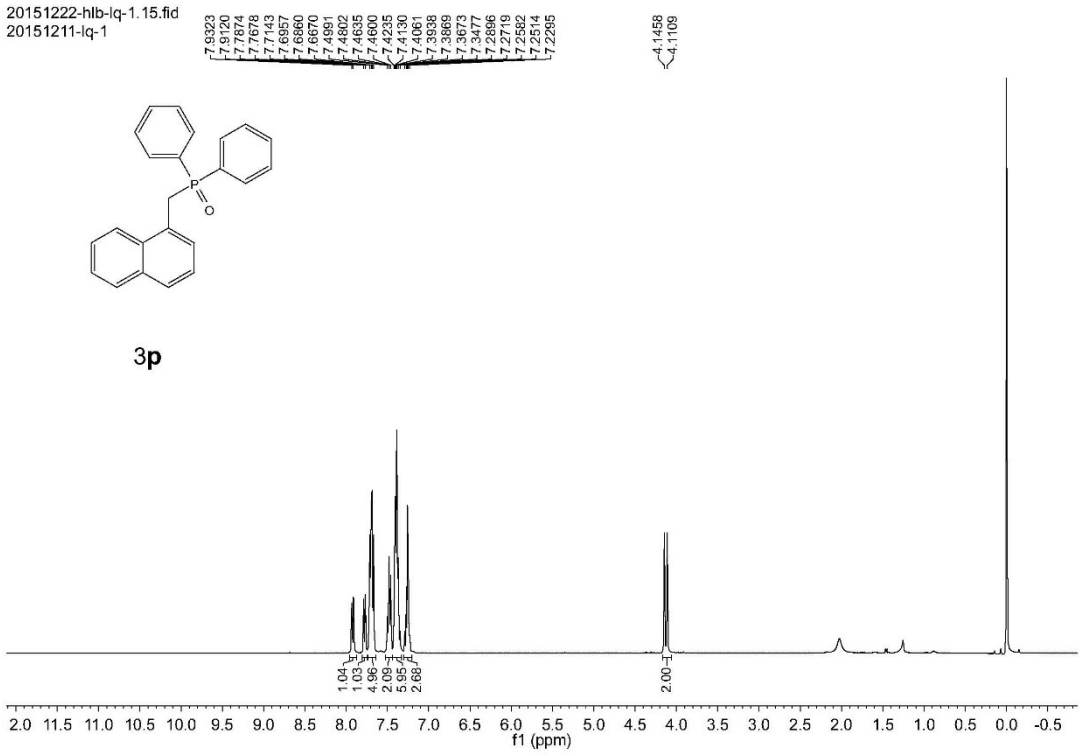

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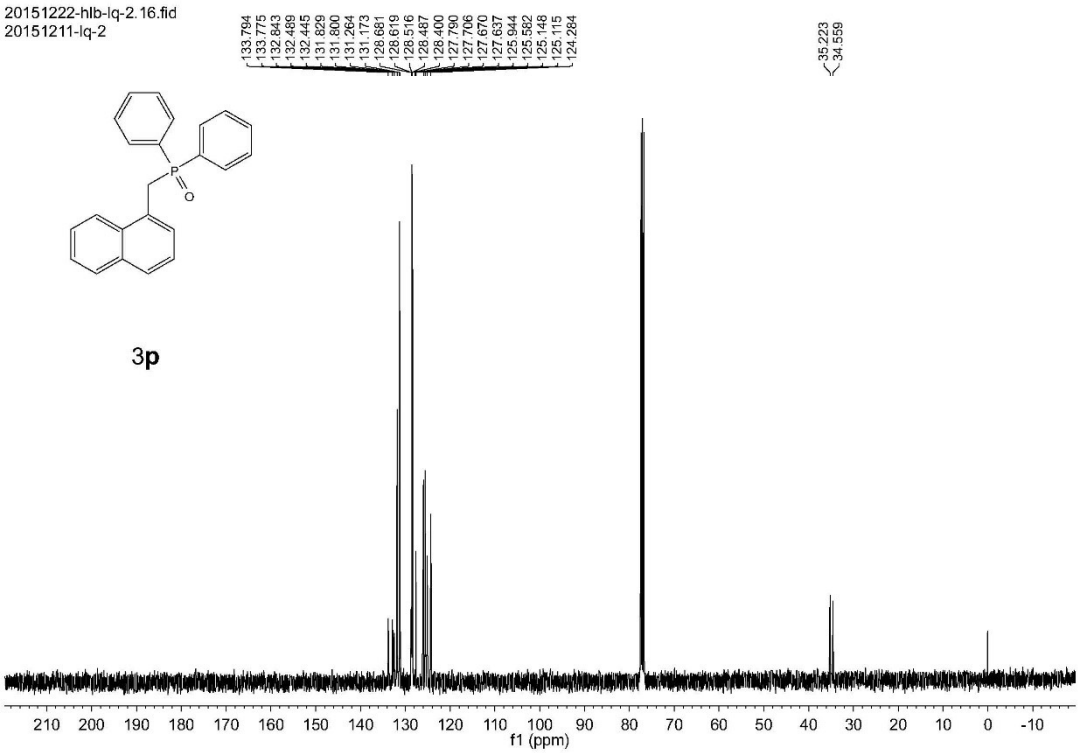

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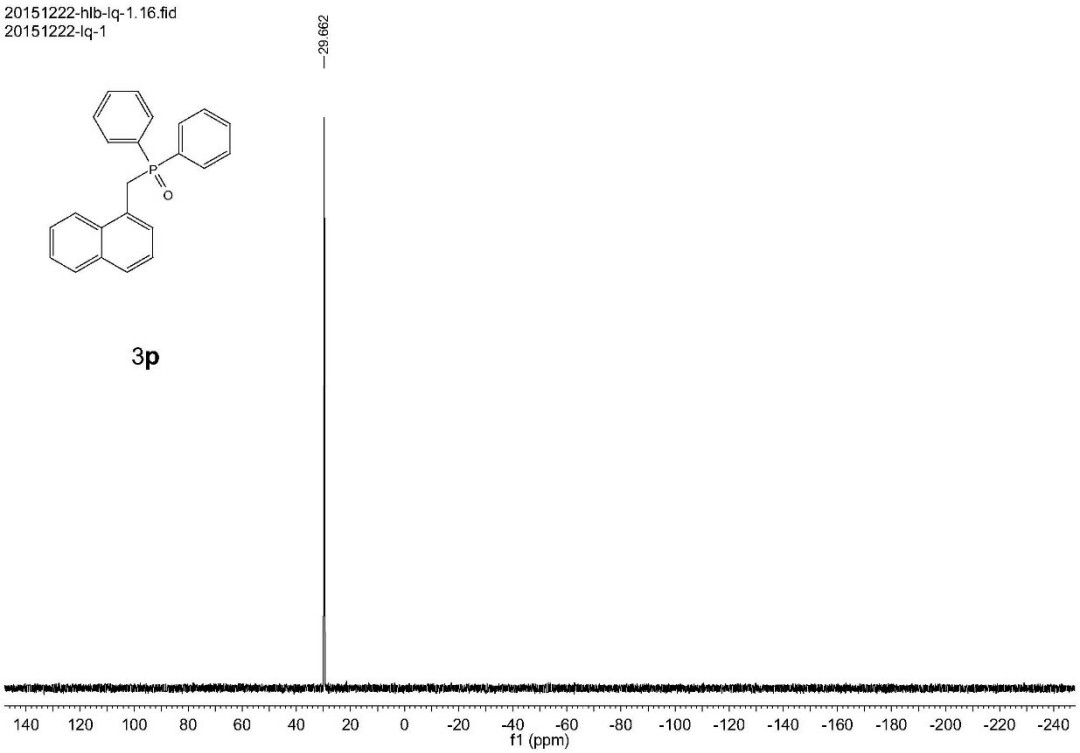

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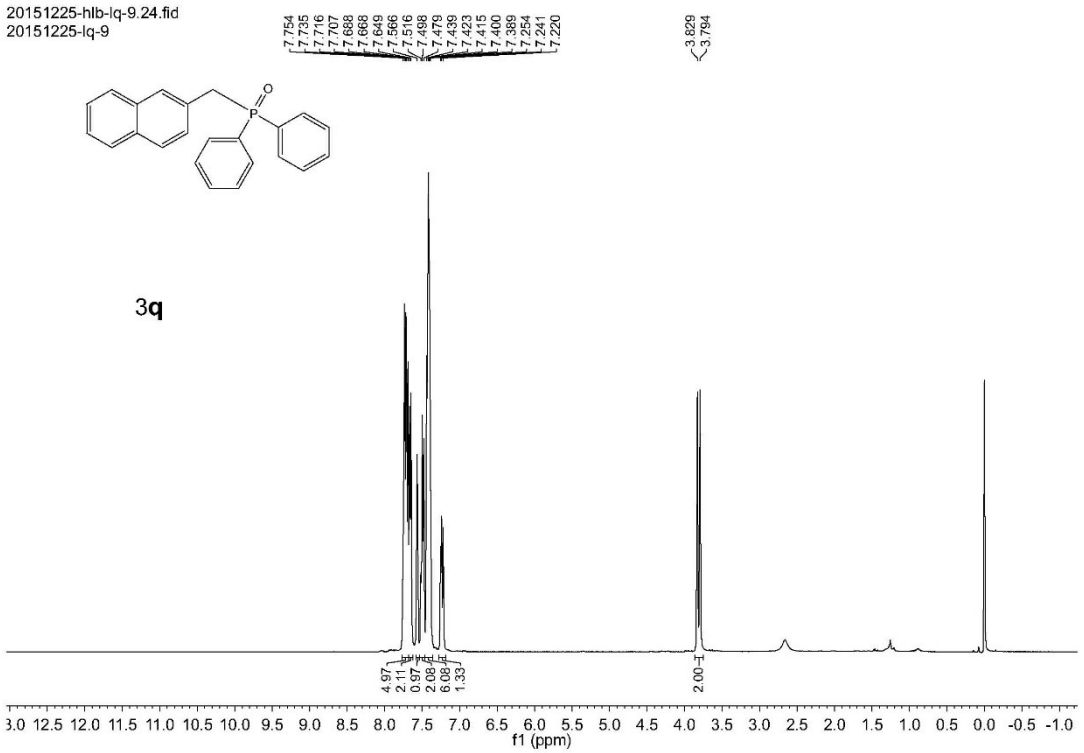

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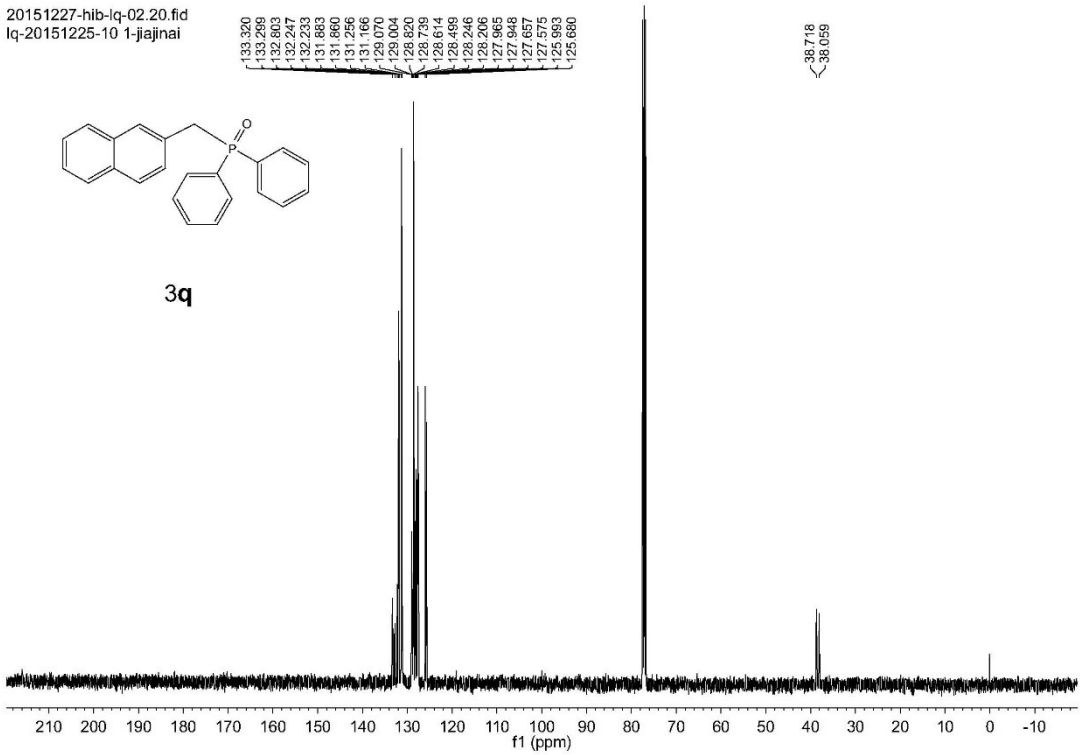

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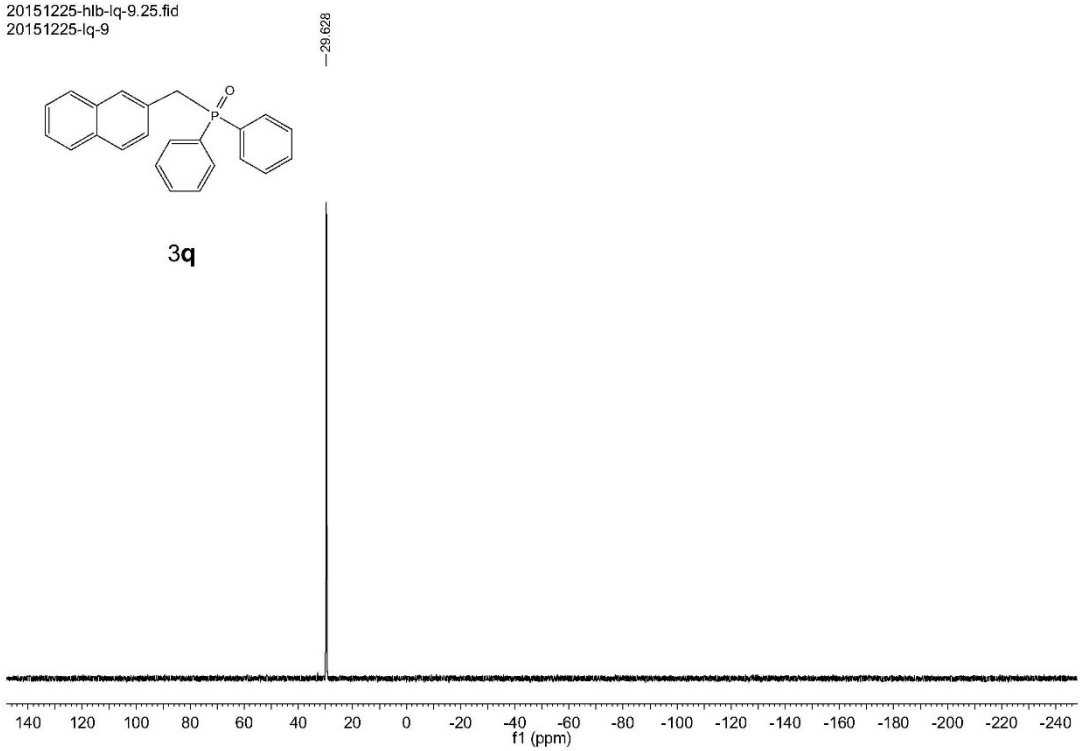

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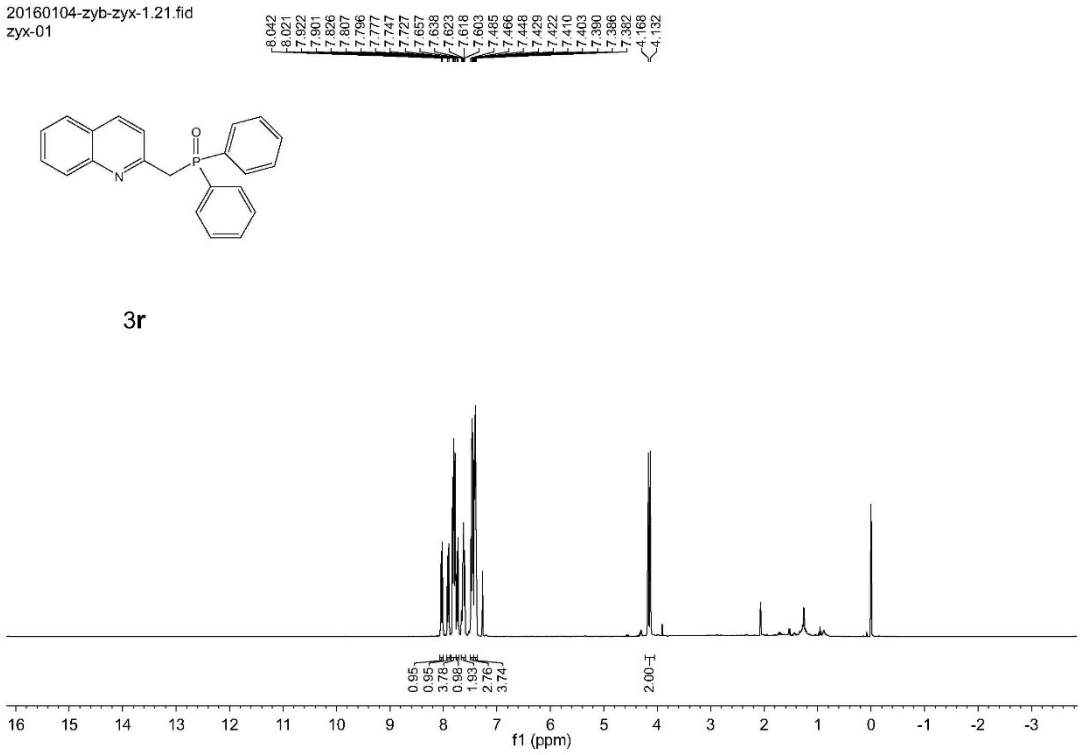

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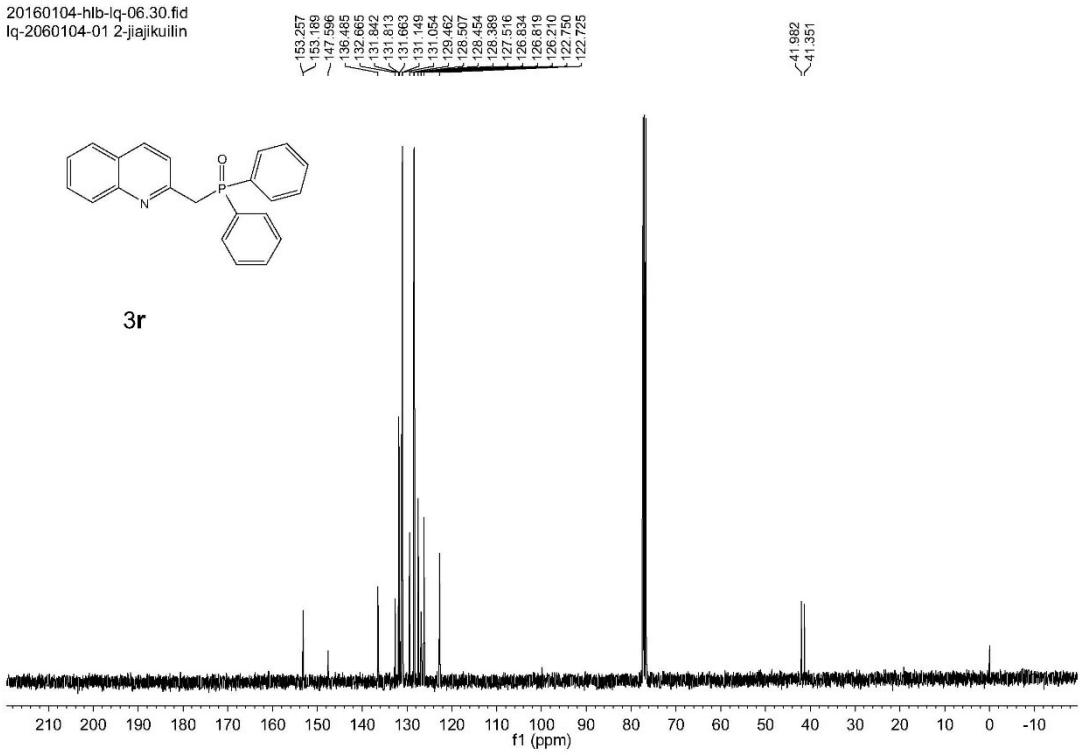

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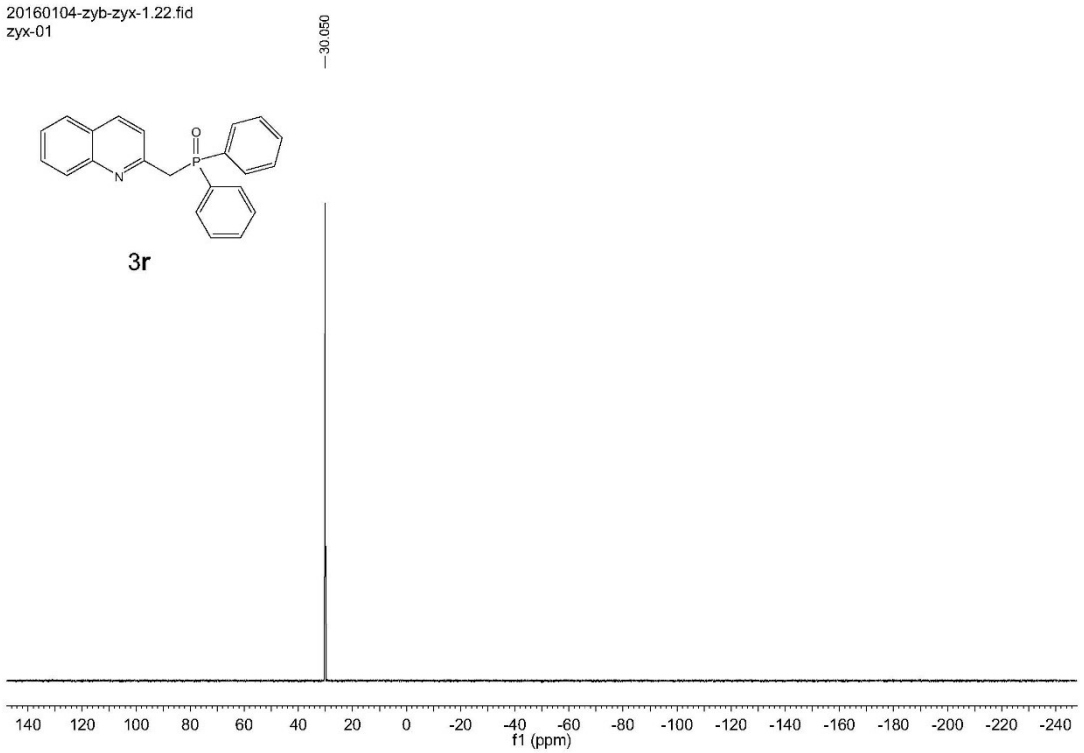

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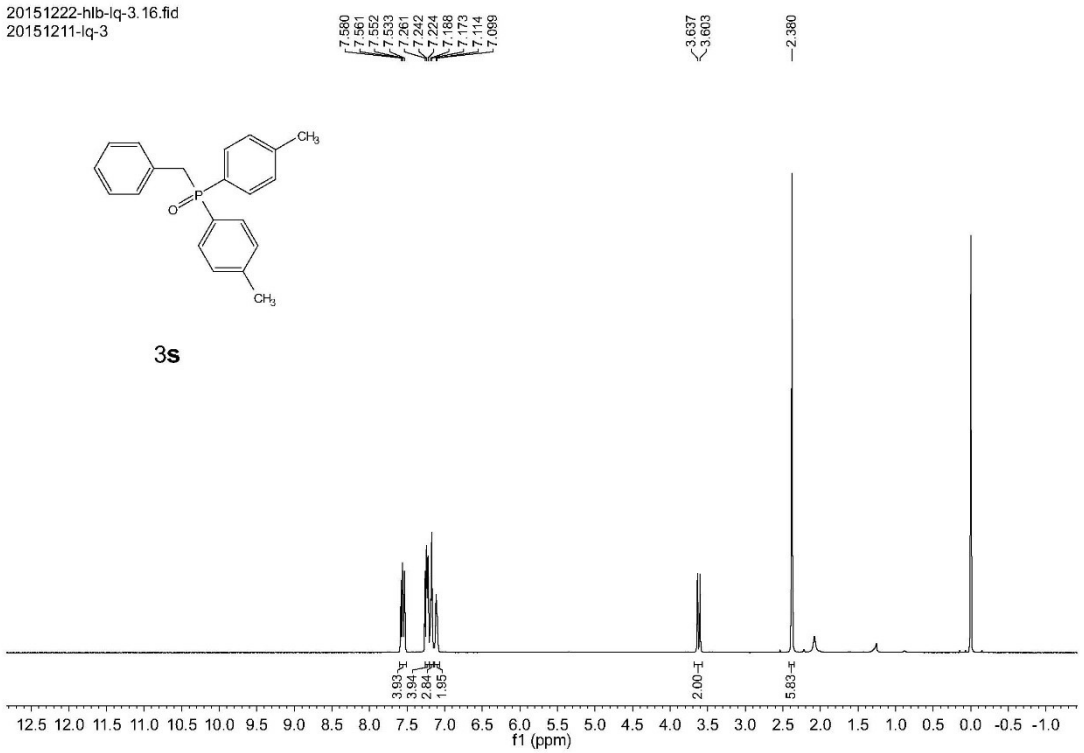

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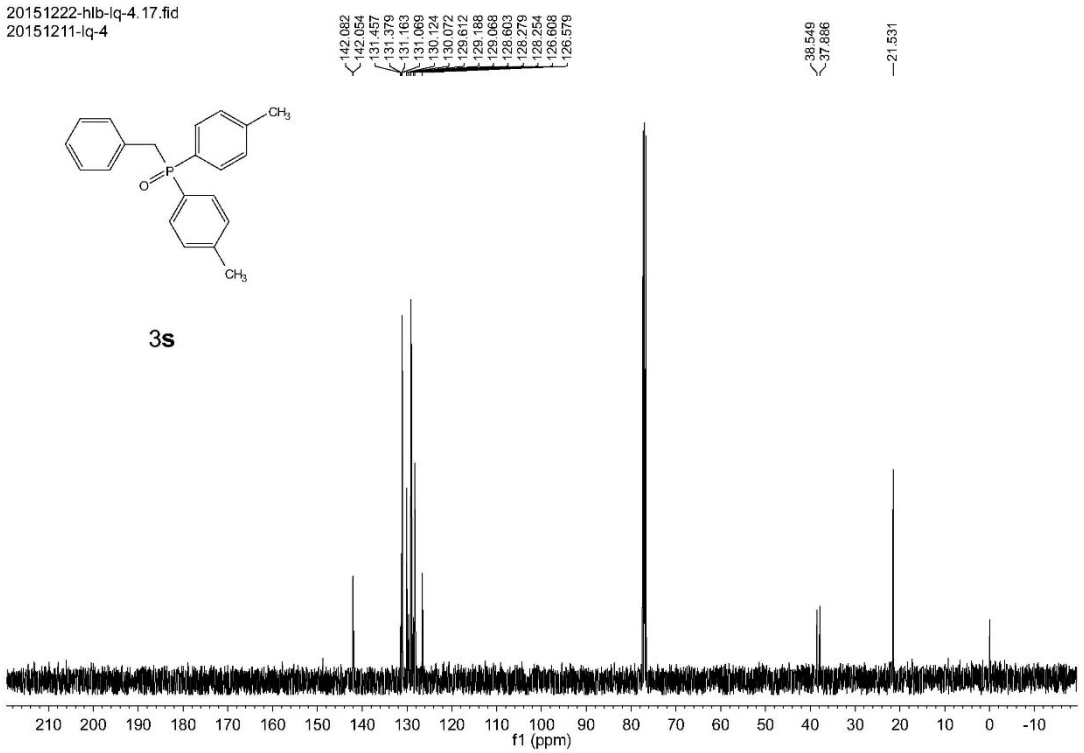

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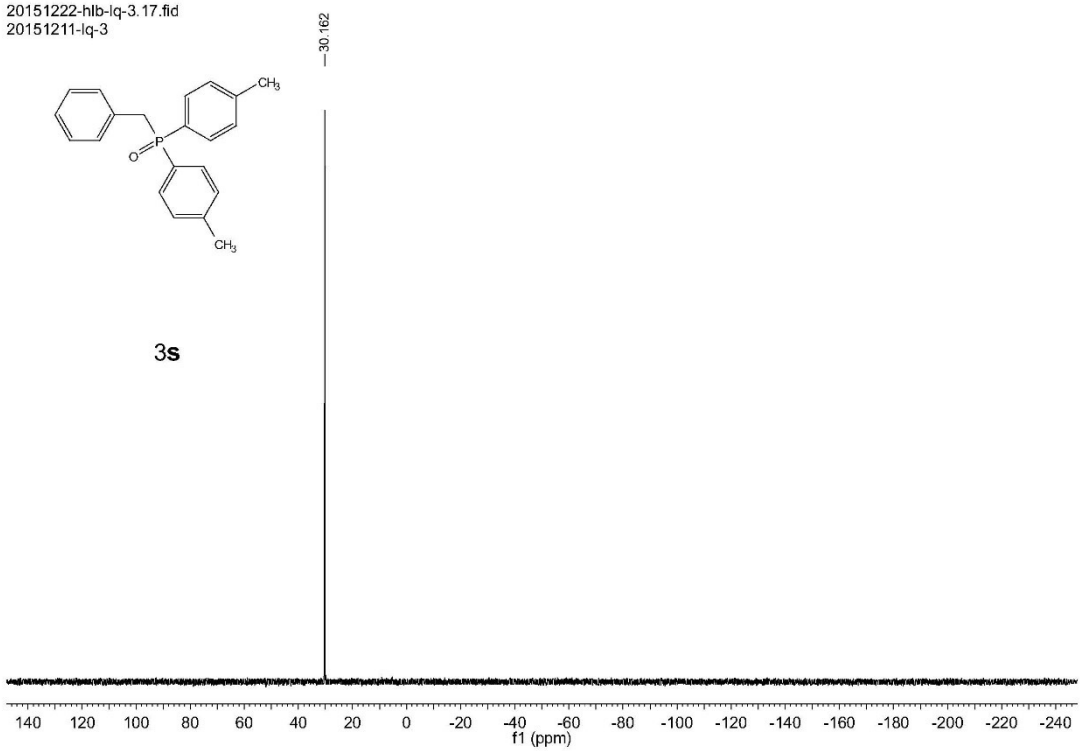

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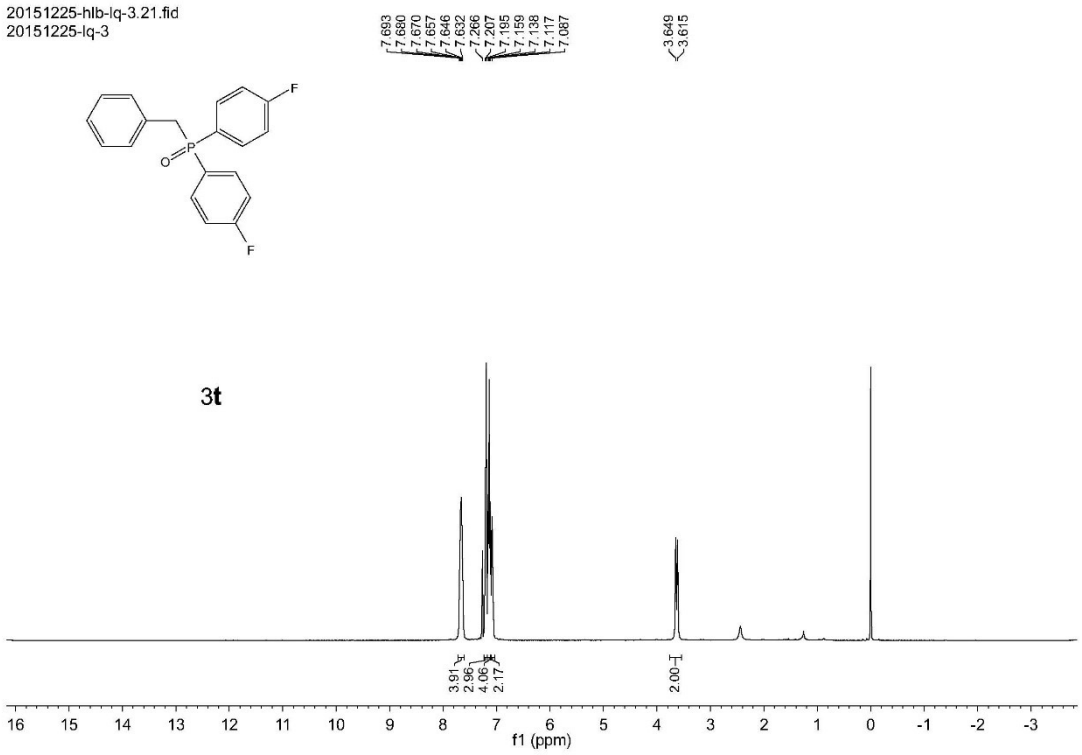

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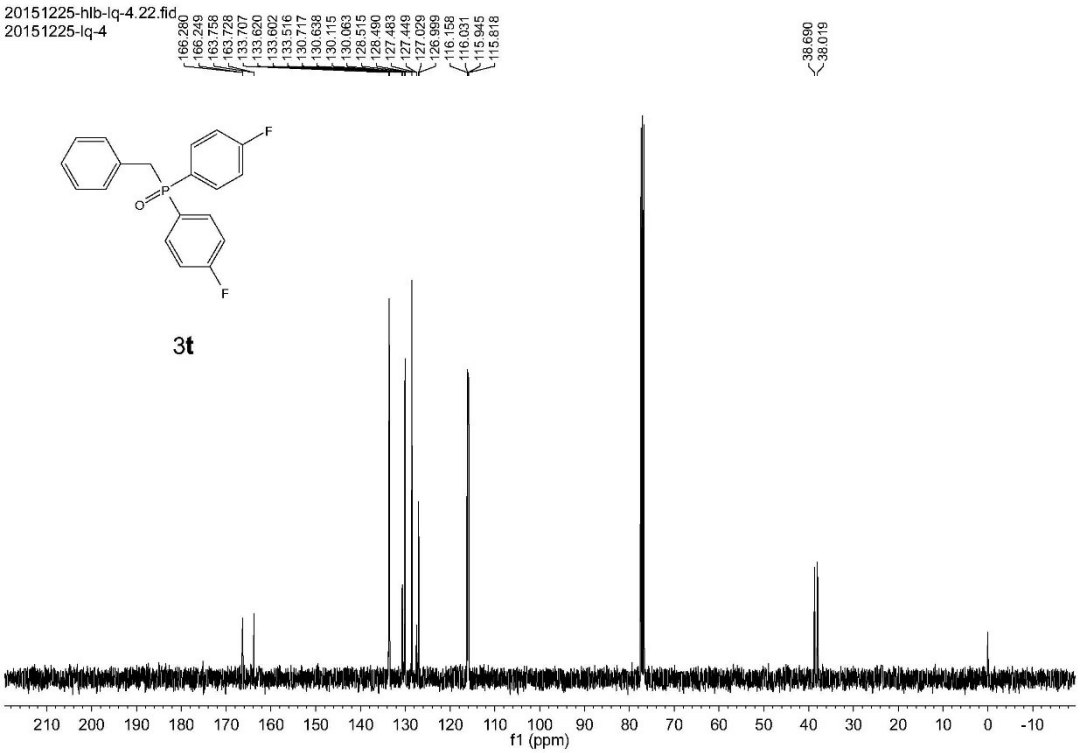

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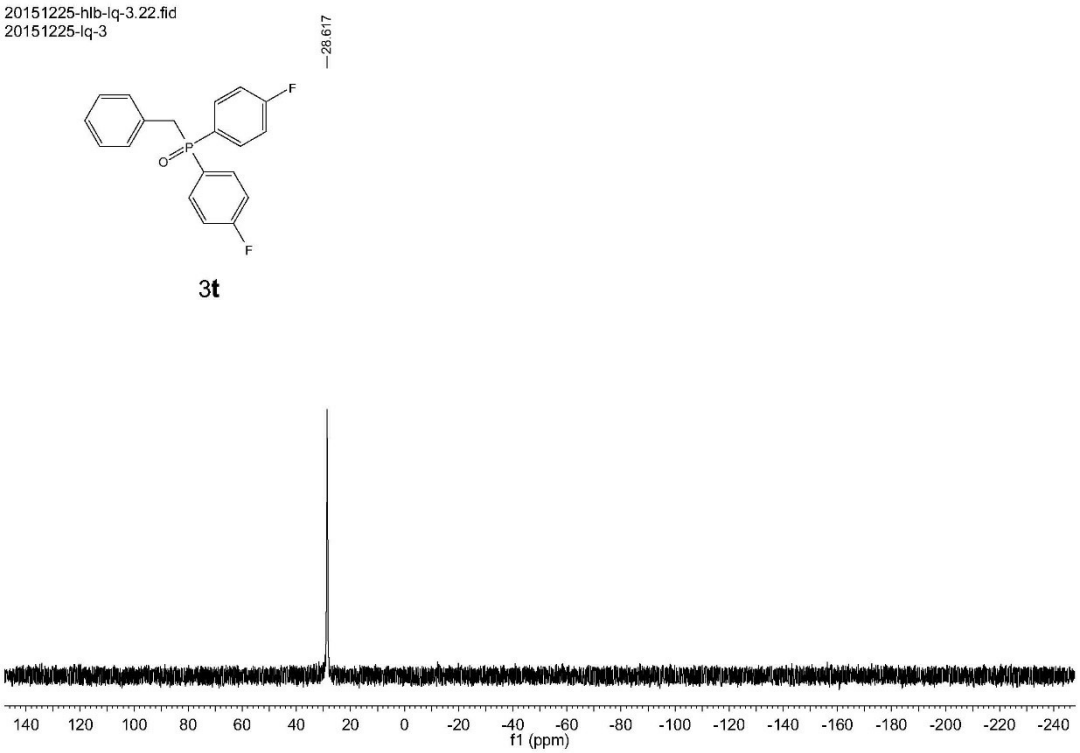

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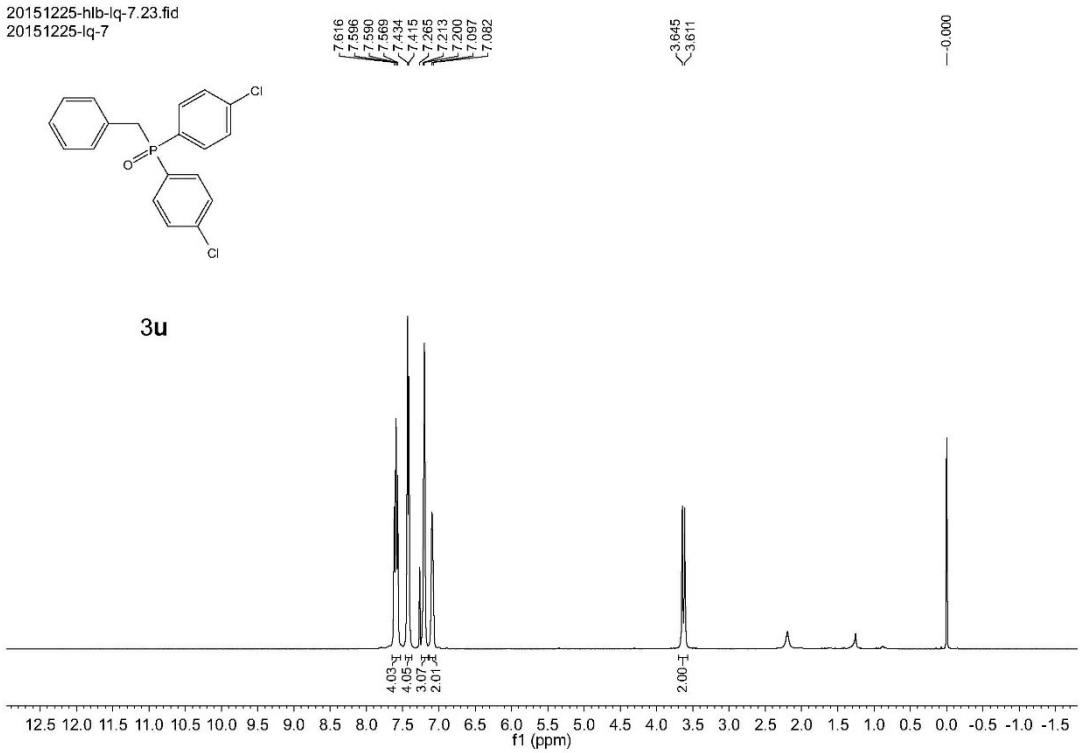

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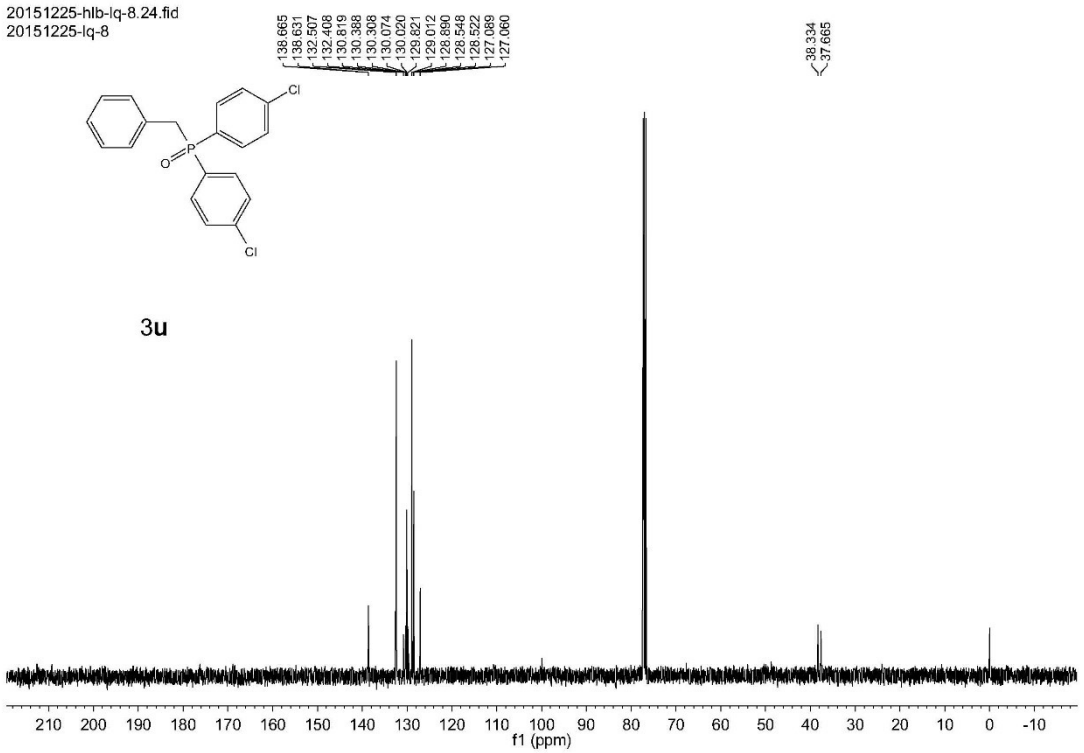

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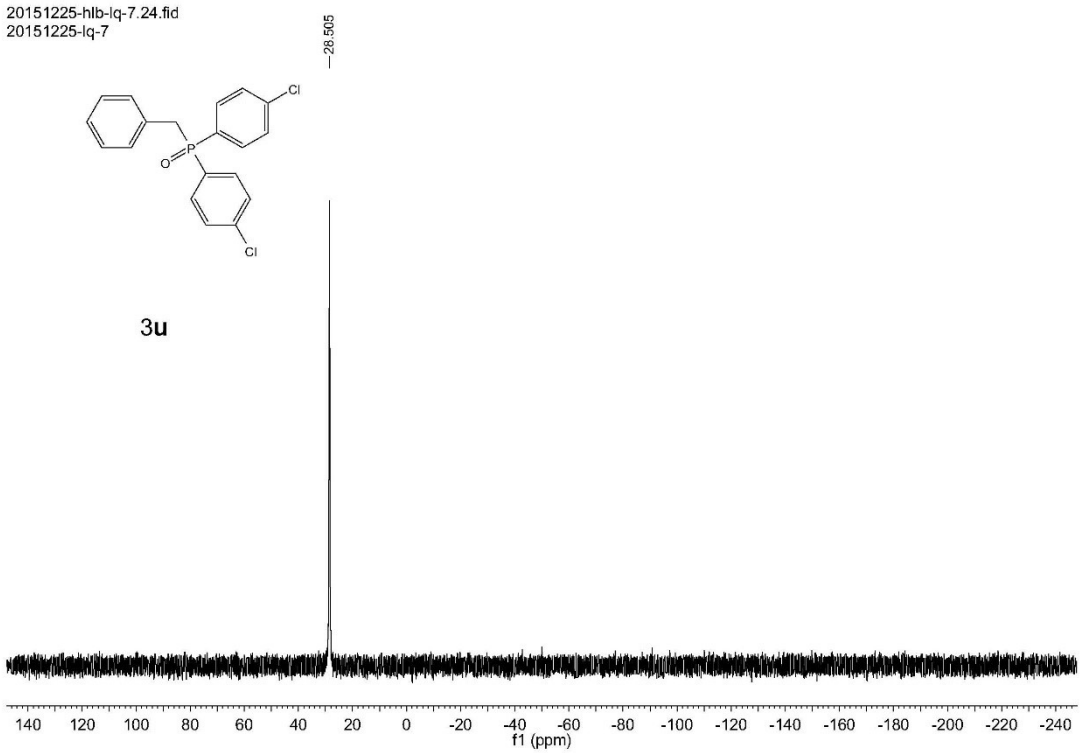
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