Supporting Information

t-BuOK-Mediated Reductive Addition of P(O)-H Compounds to Alkynes Forming *β*-Arylphosphine oxides

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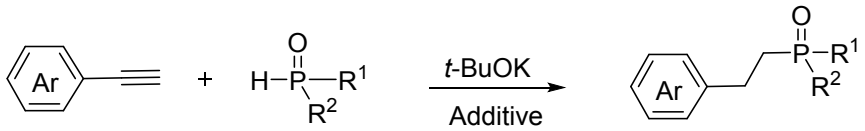
EXPERIMENTAL SECTION

General information: All reactions were carried out in oven-dried Schlenk tubes under N_2 atmosphere. Solvents were treated by the standard procedure. Reagents were used as received unless otherwise noted. The pure products were obtained by means of column chromatography on silica (petroleum ether and ethyl acetate were used as the gradient eluting solvents). ^{1}H NMR, ^{13}C NMR and ^{31}P NMR data were acquired on a 400 MHz spectrometer (400 MHz for ^{1}H, 100.6 MHz for ^{13}C, and 162 MHz for ^{31}P NMR spectroscopy). Chemical shifts for ^{1}H NMR are referred to internal Me_4Si (0 ppm) and reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz) and integration. Data for ^{31}P NMR were referred to H_3PO_4 (85% solution in D_2O, 0 ppm). The ionization method of the high-resolution mass spectrum (HRMS) is Electron Impact (EI). The type of the mass analyzer is quadrupole.

General procedure for the *t*-BuOK-Mediated Reductive Addition of P(O)-H Compounds to Alkynes.



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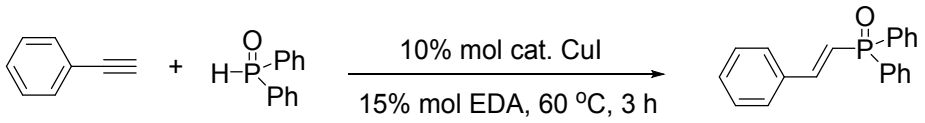
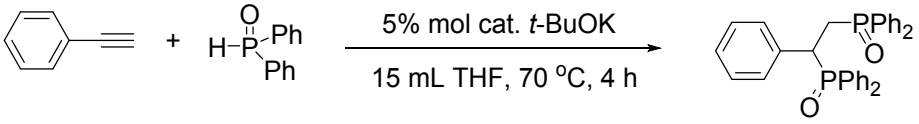
Under N_2 atmosphere, 1.7 mmol P(O)-H compounds, 1 mmol alkynes, 1 mmol KCl, 2 mmol isopropyl alcohol (IA), 0.2 mmol *t*-BuOK and 4.0 mL dioxane were charged into a 25 mL schlenk tube, then the mixture was stirred at 80 °C for 16 h. After removal of the volatiles, the residues were passed through a short silica chromatography column (particle size 37–54 μm, petroleum ether/ethyl acetate as the eluent) to afford analytically pure products **3.**

Synthesis of (*E*)-diphenyl(styryl)phosphine oxide A.[4]



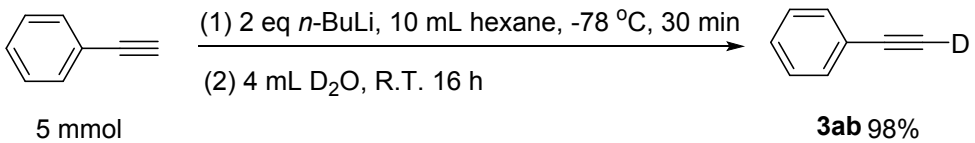
Diphenylphosphine oxide (2 mmol), ethynylbenzene (3 mmol), CuI (0.2 mmol) and ethylenediamine (EDA) (0.3 mmol) were dissolved in 5.0 mL DMSO under nitrogen atmosphere and heated at 60 °C for 3 h. The resulting solution was cooled to room temperature, and then 30 mL chloroform and 30 mL brine were added to the solution, the organic layer was washed with brine (20 mL × 2) and was dried over anhydrous Na_2SO_4. After filtration, the filtrate was concentrated in *vacuo* to give a pale yellow semisolid. The crude product was then purified by silica gel column chromatography using EtOAc/hexane (1:1) as the eluent to provide the pure target product as a pale white solid (*E*)-diphenyl(styryl)phosphine oxide **A** in 90% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (t, 4H, J = 8.0 Hz), 7.47-7.51 (m, 9H), 7.36 (s, 3H), 6.84 (q, 1H, J = 18.0 Hz, J = 22.0 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 24.4; ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 147.6 (d, $J_{P\text{-}C}$ = 3.5 Hz), 135.1 (d, $J_{P\text{-}C}$ = 17.9 Hz), 133.0 (d, $J_{P\text{-}C}$ = 105.8 Hz), 131.9 (d, $J_{P\text{-}C}$ = 2.5 Hz), 131.4 (d, $J_{P\text{-}C}$ = 10.0 Hz), 130.1, 128.9, 128.6 (d, $J_{P\text{-}C}$ = 12.1 Hz), 127.79, 119.3 (d, $J_{P\text{-}C}$ = 104.4 Hz). MS (EI): 304.

Synthesis of Bisphosphorylethane compound B.[6]



A mixture of diphenylphosphine oxide (10 mmol), ethynylbenzene (10 mmol), *t*-BuOK (0.5 mmol) and THF (15.0 mL) was heated at 70 °C for 4 h. The resulting solution was cooled to room temperature and then concentrated in *vacuo* to give a pale solid. The crude product was then purified by silica gel column chromatography using EtOAc/Petroleum ether (from 4:1 to 5:1) as the eluent to provide the pure target product as a pale white solid in 99% yield. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.06 (b, 2H), 7.08-7.78 (m, 20H), 8.85 (b, 3H), 4.30 (b, 1H), 3.18 (b, 1H), 2.88 (b, 1H); ^{31}P NMR ($CDCl_3$, 162 MHz) δ 35.3 (d, J = 46.5 Hz), 30.1 (d, J = 47.1 Hz); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 134.4 (d, $J_{C\text{-}P}$ = 2.5 Hz), 134.1 (d, $J_{C\text{-}P}$ = 2.5 Hz), 134.1 (d, $J_{C\text{-}P}$ = 10.0 Hz), 133.4 (d, $J_{C\text{-}P}$ = 1.2 Hz), 133.4 ($J_{C\text{-}P}$ = 2.9 Hz), 131.9 (d, $J_{C\text{–}P}$ = 38.0 Hz), 131.8 (d, $J_{C\text{-}P}$ = 11.0 Hz), 131.8, 131.6, 131.5, 131.3 (d, $J_{C\text{-}P}$ = 1.4 Hz), 130.9 (d, $J_{C\text{-}P}$ = 10.6 Hz), 130.8, 130.7, 130.3, 130.2, 129.1 (d, $J_{C\text{-}P}$ = 11.5 Hz), 128.7 (d, $J_{C\text{-}P}$ = 11.5 Hz), 127.9 (d, $J_{C\text{-}P}$ = 11.1 Hz), 127.4 (d, $J_{C\text{-}P}$ = 88.8 Hz), 39.3 (d, $J_{C\text{-}P}$ = 64.6 Hz), 30.1 (d, $J_{C\text{-}P}$ = 68.7 Hz).

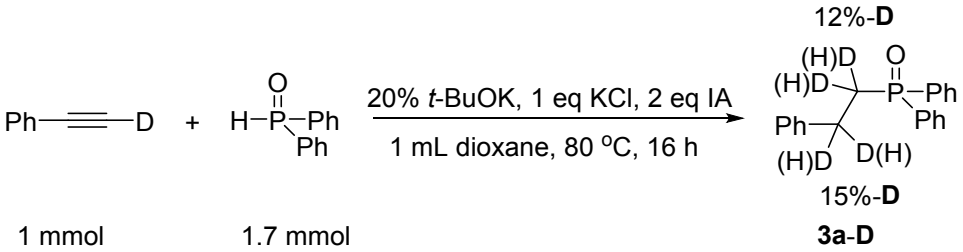
Synthesis of raw materials *sp*C-deuterated phenylacetylene.[5]



To a solution of phenylacetylene (5 mmol) in dry hexane (10 ml) was added *n*-BuLi (1.60 M in hexane solution, 2. 0 equiv) dropwisely at −78 ˚C under N_2 atmosphere. After being stirred at the same temperature for 30 min, D_2O (4 mL) was added at the room temperature under N_2 condition,

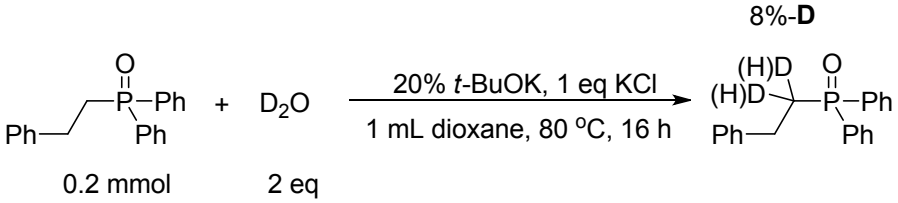
stirred continuously at the same temperature for 16 h and then stopped reaction. The organic layer was separated and the aqueous layer was extracted with CH_2Cl_2. The combined organic layers were washed with CH_2Cl_2 (10 mL× 3), dried over Na_2SO_4 and filtered. The filtrate was concentrated in *vacuo* to afford the corresponding target product with 98% deuterium incoporation.[5] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, 2H, *J* = 6.8 Hz), 7.22-7.28 (m, 3H), 2.99 (s, 0.02H).

The reductive addition of diphenyl phosphine oxide to *sp*C-deuterated phenylacetylene.



Under N_2 atmosphere, 1.7 mmol diphenylphosphine oxide, 1 mmol *sp*C-deuterated phenylacetylene, 1 mmol KCl, 2 mmol isopropyl alcohol (IA), 0.2 mmol *t*-BuOK and 4.0 mL dioxane were charged into a 25 mL schlenk tube, then the mixture was stirred at 80 °C for 16 h. After removal of the volatiles, the residues were passed through a short silica chromatography column (particle size 37–54 μm, petroleum ether/ethyl acetate as the eluent) to afford analytically pure products **3a-D**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (dd, 4H, *J* = 8.4 Hz, *J* = 10.4 Hz), 7.46-7.54 (m, 6H), 7.25 (d, 2H, *J* = 7.6 Hz), 7.15-7.19 (m, 3H), 2.90-2.96 (m, 1.7H), 2.55-2.62 (m, 1.8H). ^{31}P NMR (162 MHz, $CDCl_3$): δ 31.8. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 141.2 (d, J_{C-P} = 15.2 Hz), 132.7 (d, J_{C-P} = 97.8 Hz), 131.9 (d, J_{C-P} = 1.7 Hz), 130.8 (d, J_{C-P} = 9.3 Hz), 128.8, 128.7 (d, J_{C-P} = 7.4 Hz), 128.1, 126.4, 31.9 (31.80) (d, J_{C-P} = 69.4 Hz), 27.5 (dd, J_{C-P} = 3.0 Hz, J_{C-D} = 9.0 Hz).

H-D exchange of 3a with D_2O under the reaction conditions：

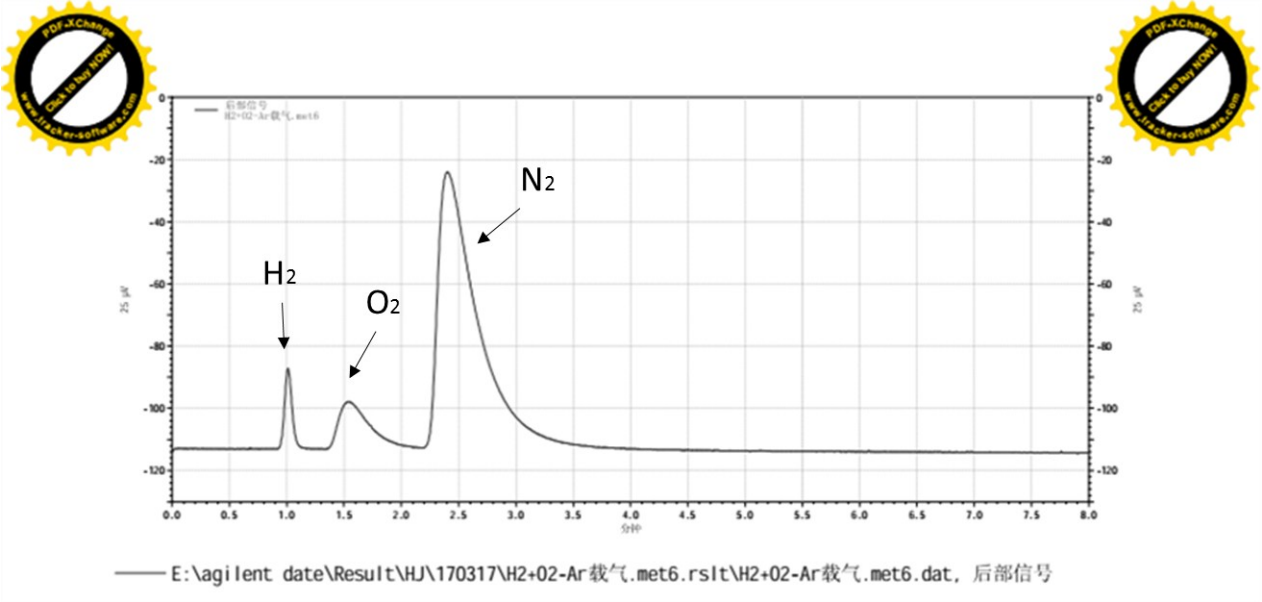


Under N_2 atmosphere, 0.2 mmol **3a**, 0.4 mmol D_2O, 0.2 mmol KCl, 0.04 mmol *t*-BuOK and 1.0 mL dioxane were charged into a 25 mL schlenk tube, then the mixture was stirred at 80 °C for 16 h. After the reaction was cooled, reaction mixture was evaporated under the reduced pressure, as indicated from ^{1} H NMR spectroscopy, the C adjacent to phosphoryl group was deuterated with ca. 8% of deuterium incorporation. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (dd, 4H, *J* = 8.0 Hz, *J* = 11.6 Hz), 7.46-7.55 (m, 6H), 7.26 (dd, 2H, *J* = 7.2 Hz, *J* = 7.6 Hz), 7.15-7.19 (m, 3H), 2.90-2.95 (m, 2H), 2.56-2.63 (m, 1.83H).

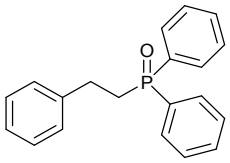
H_2 detection by GC：

Under N_2 atmosphere, 1 mmol ethynylbenzene, 1.7 mmol diphenylphosphine oxide compounds, 1 mmol KCl, 2 mmol isopropyl alcohol (IA), 0.2 mmol *t*-BuOK and 4.0 mL dioxane were charged into a 25 mL schlenk tube, then the mixture was stirred at 80 °C for 16 h. After the reaction was cooled to room temperature, the gas was analyzed by GC (Agilent

Technologies 7820A GC system, TCD detector, AE.5A column, oven temperature: 50 °C, carrier: Ar, 45 mL/min.). The pick time was checked by standard samples.

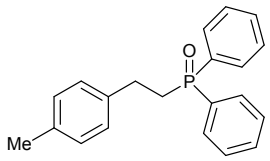


Characterization data of products 3.



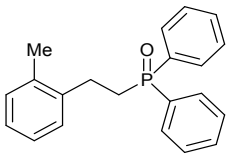
3a

Phenethyldiphenylphosphine oxide (3a).[3] Following the general procedure, **3a** was isolated as a white crystals (275.4 mg, 90%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (dd, 4H, J = 8.0 Hz, J = 11.6 Hz), 7.46-7.55 (m, 6H), 7.26 (dd, 2H, J = 7.2 Hz, J = 7.6 Hz), 7.15-7.19 (m, 3H), 2.90-2.96 (m, 2H), 2.55-2.62 (m, 2H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 31.7; ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 141.2 (d, J_{P-C} = 15.3 Hz), 132.8 (d, J_{P-C} = 97.8 Hz), 131.9 (d, J_{P-C} = 2.8 Hz), 130.8 (d, J_{P-C} = 9.2 Hz), 128.8, 128.7 (d, J_{P-C} = 6.4 Hz), 128.1, 126.4, 31.9 (d, J_{P-C} = 69.5 Hz), 27.6 (d, J_{P-C} = 3.1 Hz). MS (EI): 306. This compound is known.



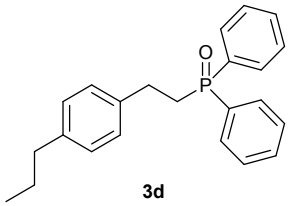
3b

(4-methylphenethyl)diphenylphosphine oxide (3b). Following the general procedure, 1.0 equiv *t*-BuOK was used. **3b** was isolated as a white crystals (294.4 mg, 92%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (dd, 4H, J = 7.6 Hz, J = 11.2 Hz), 7.45-7.54 (m, 6H), 7.06 (b, 4H), 2.83-2.92 (m, 2H), 2.53-2.60 (m, 2H), 2.30 (s, 3H). ^{31}P NMR (162 MHz, $CDCl_3$): δ 31.5. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 138.1 (d, J_{C-P} = 15.4 Hz), 135.9, 132.9 (d, J_{C-P} = 97.5 Hz), 131.8 (d, J_{C-P} = 2.6 Hz), 130.8 (d, J_{C-P} = 9.3 Hz), 129.3, 128.7 (d, J_{C-P} = 11.6 Hz), 127.9, 32.0 (d, J_{C-P} = 69.3 Hz), 27.1 (d, J_{C-P} = 3.0 Hz), 20.9. Melting point: 123.7~123.8 °C. HRMS: Cal. for $C_{21}H_{21}OP$ 320.1330. Found 320.1336.



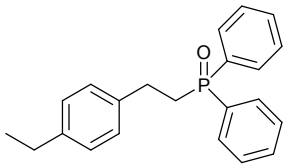
3c

(2-methylphenethyl)diphenylphosphine oxide (3c). Following the general procedure, **3c** was isolated as a white crystals (294.6 mg, 92%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78 (dd, 4H, J = 7.2 Hz, J = 11.2 Hz), 7.45-7.54 (m, 6H), 7.10 (b, 4H), 2.88-2.94 (m, 2H), 2.47-2.55 (m, 2H), 2.20 (s, 3H). ^{31}P NMR (162 MHz, $CDCl_3$): δ 31.6. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 139.4 (d, $J_{C\text{-}P}$ = 14.9 Hz), 135.8, 132.9 (d, $J_{C\text{-}P}$ = 97.5 Hz), 131.8 (d, $J_{C\text{-}P}$ = 2.7 Hz), 130.8 (d, $J_{C\text{-}P}$ = 9.2 Hz), 130.4, 128.7 (d, $J_{C\text{-}P}$ = 11.6 Hz), 128.4, 126.5, 126.3, 30.6 (d, $J_{C\text{-}P}$ = 69.1 Hz), 24.9 (d, $J_{C\text{-}P}$ = 3.0 Hz), 19.1. Melting point: 109.0~109.3 °C. HRMS: Cal. for $C_{21}H_{21}OP$ 320.1330. Found 320.1325.



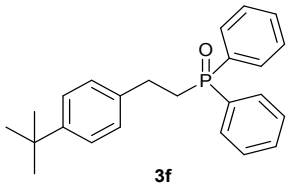
3d

(4-propylphenethyl)diphenylphosphine oxide (3d). Following the general procedure, **3d** was isolated as a white crystals (285.4 mg, 82%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (dd, 4H, J = 7.6 Hz, J = 10.8 Hz), 7.45-7.54 (m, 6H), 7.07 (b, 4H), 2.87-2.93 (m, 2H), 2.51-2.61 (m, 4H), 1.55-1.64 (m, 2H), 0.92 (t, 3H, J = 7.2 Hz). ^{31}P NMR (162 MHz $CDCl_3$): δ 31.7. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 140.7, 138.4 (d, $J_{C\text{-}P}$ = 15.3 Hz), 132.9 (d, $J_{C\text{-}P}$ = 97.2 Hz), 131.8 (d, $J_{C\text{-}P}$ = 2.7 Hz), 130.8 (d, $J_{C\text{-}P}$ = 9.2 Hz), 128.7 (d, $J_{C\text{-}P}$ = 11.5 Hz), 128.7, 127.9, 37.6, 31.9 (d, $J_{C\text{-}P}$ = 69.2 Hz), 27.1 (d, $J_{C\text{-}P}$ = 3.1 Hz), 24.6, 13.8. MS (EI): 348. Melting point: 100.0~101.3°C. HRMS: Cal. for $C_{23}H_{25}OP$ 348.1643. Found 348.1631.



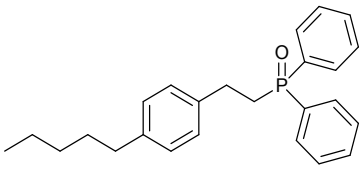
3e

(4-ethylphenethyl)diphenylphosphine oxide (3e). Following the general procedure, **3e** was isolated as a pale white crystals (276 mg, 80%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (dd, 4H, J = 7.6 Hz, J = 10.4 Hz), 7.43-7.54 (m, 6H), 7.08 (b, 4H), 2.87-2.93 (m, 2H), 2.54-2.62 (m, 4H), 1.20 (t, 3H, J = 7.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 31.7. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 142.3, 138.4 (d, $J_{C\text{-}P}$ = 15.2 Hz), 132.9 (d, $J_{C\text{-}P}$ = 97.6 Hz), 131.8 (d, $J_{C\text{-}P}$ = 2.7 Hz), 130.8 (d, $J_{C\text{-}P}$ = 9.3 Hz), 128.7 (d, $J_{C\text{-}P}$ = 11.6 Hz), 128.1, 128.1, 31.9 (d, $J_{C\text{-}P}$ = 69.3 Hz), 28.4, 27.1 (d, $J_{C\text{-}P}$ = 3.0 Hz), 15.7. Melting point: 69 °C. HRMS: Cal. for $C_{22}H_{23}OP$ 334.1487. Found 334.1470.



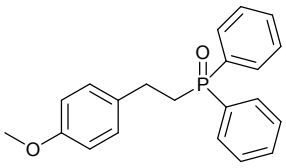
3f

(4-(tert-butyl)phenethyl)diphenylphosphine oxide (3f). Following the general procedure, **3f** was isolated as a white crystals (293.2 mg, 81%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68 (dd, 4H, J = 7.2 Hz, J = 11.2 Hz), 7.36-7.45 (m, 6H), 7.20 (d, 2H, J = 8.0 Hz), 7.02 (d, 2H, J = 8.0 Hz), 2.80-2.86 (m, 2H), 2.47-2.54 (m, 2H), 1.21 (s, 9H). ^{31}P NMR (162 MHz, $CDCl_3$): δ 31.6. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 149.2, 138.1 (d, J_{C-P} = 15.2 Hz), 132.9 (d, J_{C-P} = 97.6 Hz), 131.8 (d, J_{C-P} = 2.7 Hz), 130.8 (d, J_{C-P} = 9.2 Hz), 128.8 (d, J_{C-P} = 11.6 Hz), 127.8, 125.508, 34.4, 31.8 (d, J_{C-P} = 69.4 Hz), 31.4, 26.9 (d, J_{C-P} = 2.1 Hz). Melting point: 118.6 °C. HRMS: Cal. for $C_{24}H_{27}OP$ 362.1800. Found 362.1780.



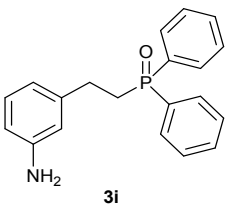
3g

(4-pentylphenethyl)diphenylphosphine oxide (3g). Following the general procedure, 1.0 equiv *t*-BuOK was loaded. **3g** was isolated as a white crystals (315.8 mg, 84%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 (dd, 4H, J = 7.6 Hz, J = 11.2 Hz), 7.44-7.53 (m, 6H), 7.07 (b, 4H), 2.87-2.93 (m, 2H), 2.52-2.61 (m, 4H), 1.54-1.61 (m, 2H), 1.28-1.35 (m, 4H), 0.88 (t, 3H, J = 6.0 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 31.5. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 140.9, 138.3 (d, J_{C-P} = 15.2 Hz), 132.9 (d, J_{C-P} = 97.5 Hz), 131.8 (d, J_{C-P} = 2.6 Hz), 130.8 (d, J_{C-P} = 9.2 Hz), 128.7 (d, J_{C-P} = 11.6 Hz), 128.6, 127.9, 35.5, 31.9 (d, J_{C-P} = 69.3 Hz), 31.5, 31.2, 27.1 (d, J_{C-P} = 3.1 Hz), 22.5, 14.0. Melting point: 100.0~100.2 °C. HRMS: Cal. for $C_{25}H_{29}OP$ 376.1956. Found 376.1944.



3h

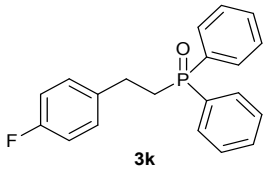
(4-methoxyphenethyl)diphenylphosphine oxide (3h).[2] Following the general procedure, 1.0 equiv *t*-BuOK was loaded. **3h** was isolated as a white crystals (292.3 mg, 87%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (dd, 4H, J = 7.6 Hz, J = 11.2 Hz), 7.46-7.52 (m, 6H), 7.06 (d, 2H, J = 8.4 Hz), 6.78 (d, 2H, J = 8.4 Hz), 3.74 (s, 3H), 2.84-2.90 (m, 2H), 2.51-2.58 (m, 2H). ^{31}P NMR (162 MHz, $CDCl_3$): δ 31.4. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 158.1, 133.2 (d, J_{C-P} = 15.4 Hz), 132.9 (d, J_{C-P} = 97.5 Hz), 131.8 (d, J_{C-P} = 2.6 Hz), 130.8 (d, J_{C-P} = 9.2 Hz), 129.0, 128.7 (d, J_{C-P} = 11.5 Hz), 114.0, 55.3, 32.1 (d, J_{C-P} = 69.0 Hz), 26.7 (d, J_{C-P} = 3.1 Hz). This compound is known.



3i

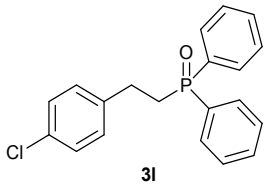
(3-aminophenethyl)diphenylphosphine oxide (3i). Following the general procedure, 1.0 equiv *t*-BuOK was loaded in 6 mL dioxane. **3i** was isolated as a pale yellow crystals (276.1 mg, 86%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 (dd, 4H, J = 8.0 Hz, J = 10.8 Hz), 7.37-7.51 (m, 6H), 7.00 (t, 1H,

J = 7.6 Hz), 6.45-6.52 (m, 3H), 3.67 (b, 2H), 2.77-2.83 (m, 2H), 2.49-2.56 (m, 2H). ^{31}P NMR (162 MHz, $CDCl_3$): δ 31.8. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 146.8, 142.4 (d, J_{C-P} = 15.2 Hz), 132.8 (d, J_{C-P} = 97.7 Hz), 131.8 (d, J_{C-P} = 2.6 Hz), 130.8 (d, J_{C-P} = 9.2 Hz), 129.5, 128.7 (d, J_{C-P} = 11.6 Hz), 118.0, 114.8, 113.1, 31.7 (d, J_{C-P} = 69.4 Hz), 27.5 (d, J_{C-P} = 3.0 Hz). Melting point: 152.2~153.7 °C. HRMS: Cal. for $C_{20}H_{20}NOP$ 321.1283. Found 321.1275.



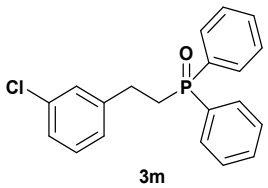
3k

(4-fluorophenethyl)diphenylphosphine oxide (3k).[2] Following the general procedure, **3k** was isolated as a white crystals(181.4 mg, 56%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (dd, 4H, J = 7.6 Hz, J = 10.8 Hz), 7.45-7.54 (m, 6H), 7.10 (dd, 2H, J = 7.6 Hz, J = 5.6 Hz), 6.92 (dd, 2H, J = 8.4 Hz, J = 8.4 Hz), 2.87-2.93 (m, 2H), 2.51-2.58 (m, 2H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 31.5; ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 161.5 (d, J_{F-C} = 242.9 Hz), 136.7 (dd, J_{F-C} = 3.3 Hz, J_{P-C} = 14.9 Hz), 132.6 (d, J_{P-C} = 97.9 Hz), 131.9 (d, J_{P-C} = 2.7 Hz), 130.8 (d, J_{P-C} = 9.3 Hz), 129.5 (d, J_{F-C} = 7.8 Hz), 128.8 (d, J_{P-C} = 11.6 Hz), 115.4 (d, J_{F-C} = 21.1 Hz), 31.9 (d, J_{P-C} = 69.2 Hz), 26.8 (d, J_{P-C} = 2.9 Hz). MS (EI): 324. This compound is known.



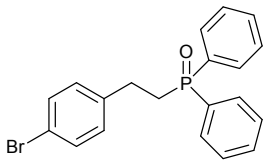
3l

(4-chlorophenethyl)diphenylphoshine oxide (3l).[2] Following the general procedure, 1.0 equiv *t*-BuOK was used. **3l** was isolated as a pale white crystals (278.8 mg, 82%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (dd, 4H, J = 8.0 Hz, J = 8.0 Hz), 7.46-7.56 (m, 6H), 7.21 (d, 2H, J = 8.0 Hz), 7.08 (d, 2H, J = 8.0 Hz), 2.88-2.94 (m, 2H), 2.52-2.58 (m, 2H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 31.4; ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 139.6 (d, J_{P-C} = 14.9 Hz), 132.6 (d, J_{P-C} = 95.7 Hz), 132.1 (d, J_{P-C} = 2.4 Hz), 131.9 (d, J_{P-C} = 2.6 Hz), 130.8 (d, J_{P-C} = 9.2 Hz), 129.5, 128.8 (d, J_{P-C} = 11.5 Hz), 128.7, 31.8 (d, J_{P-C} = 69.5 Hz), 27.0 (d, J_{P-C} = 2.9 Hz). MS (EI): 340. This compound is known.



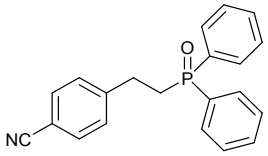
3m

(3-chlorophenethyl)diphenylphoshine oxide (3m). Following the general procedure, **3m** was isolated as a pale white crystals (289.0 mg, 85%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (dd, 4H, J = 7.6 Hz, J = 11.6 Hz), 7.45-7.54 (m, 6H), 7.13-7.18 (m, 3H), 7.03 (d, 1H, J = 7.2 Hz), 2.88-2.94 (m, 2H), 2.52-2.59 (m, 2H). ^{31}P NMR (162 MHz, $CDCl_3$): δ 31.1. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 143.1 (d, J_{C-P} = 15.0 Hz), 134.3, 132.6 (d, J_{C-P} = 98.1 Hz), 131.9 (d, J_{C-P} = 2.7 Hz), 130.8 (d, J_{C-P} = 9.3 Hz), 129.87, 128.8 (d, J_{P-C} = 11.5 Hz), 128.2, 126.6, 126.4, 31.6 (d, J_{C-P} = 68.5 Hz), 27.3 (d, J_{C-P} = 2.9 Hz). Melting point: 125.6~126.9 °C. HRMS: Cal. for $C_{20}H_{18}ClOP$ 340.0784. Found 340.0773.



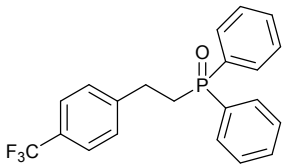
3n

(4-bromophenethyl)diphenylphosphine oxide (3n). Following the general procedure, 1.0 equiv *t*-BuOK was loaded. **3n** was isolated as a white crystals (292.6 mg, 76%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73 (dd, 4H, J = 7.6 Hz, J = 10.8 Hz), 7.42-7.51 (m, 6H), 7.31 (d, 2H, J = 8.0 Hz), 6.99 (d, 2H, J = 7.6 Hz), 2.83-2.89 (m, 2H), 2.48-2.55 (m, 2H). ^{31}P NMR (162 MHz, $CDCl_3$): δ 31.1. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 140.1 (d, J_{C-P} = 14.7 Hz), 132.7 (d, J_{C-P} = 97.9 Hz), 131.9 (d, J_{C-P} = 2.6 Hz), 131.6, 130.7 (d, J_{C-P} = 9.2 Hz), 129.9, 128.8 (d, J_{P-C} = 11.6 Hz), 120.1, 31.7 (d, J_{C-P} = 69.5 Hz), 27.1 (d, J_{C-P} = 3.0 Hz). Melting point: 171.6~172.2 °C. HRMS: Cal. for $C_{20}H_{18}BrOP$ 384.0279. Found 384.0280.



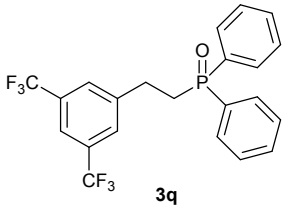
3o

4-(2-diphenylphosphoryl)ethyl)benzonitrile (3o). Following the general procedure, **3o** was isolated as a white crystals (244.9 mg, 74%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (dd, 4H, J = 7.6 Hz, J = 11.6 Hz), 7.47-7.57 (m, 8H), 7.27 (d, 2H, J = 8.0 Hz), 2.93-3.04 (m, 2H), 2.54-2.61 (m, 2H). ^{31}P NMR (162 MHz, $CDCl_3$): δ 30.8. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 146.6 (d, J_{C-P} = 14.2 Hz), 132.4 (d, J_{C-P} = 98.4 Hz), 132.4, 132.0 (d, J_{C-P} = 2.7 Hz), 130.7 (d, J_{C-P} = 9.3 Hz), 129.0, 128.8 (d, J_{C-P} = 11.7 Hz), 118.8, 110.3, 31.3 (d, J_{C-P} = 69.5 Hz), 27.8 (d, J_{C-P} = 2.8 Hz). Melting point: 196.3~197.5 °C. HRMS: Cal. for $C_{21}H_{18}NOP$ 331.1126. Found 331.1112.



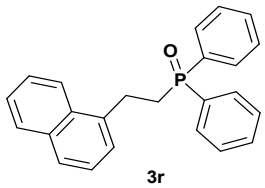
3p

Diphenyl(4-trifluoromethyl)phosphine oxide (3p). Following the general procedure, **3p** was isolated as a white crystals (269.3 mg, 72%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (dd, 4H, J = 7.6 Hz, J = 11.2 Hz), 7.09-7.20 (m, 8H), 6.90 (d, 2H, J = 7.6 Hz), 2.61-2.68 (m, 2H), 2.19-2.26 (m, 2H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 30.99; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.43; ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.16 (db, J_{P-C} = 14.2 Hz), 132.49 (d, J_{P-C} = 98.1 Hz), 131.92 (d, J_{P-C} = 2.7 Hz), 131.34 (q, J_{C-F} = 10.0 Hz), 130.73 (d, J_{P-C} = 9.3 Hz), 128.77 (d, J_{P-C} = 11.6 Hz), 128.50, 125.47(q, J_{F-C} = 3.8 Hz), 123.91 (q, J_{F-C} = 270.2 Hz), 31.50 (d, J_{P-C} = 69.6 Hz), 27.49 (d, J_{P-C} = 3.0 Hz). Melting point: 99.1~99.8 °C. MS (EI): 374. HRMS: Cal. for $C_{21}H_{18}F_3OP$ 374.1047. Found 374.1027.



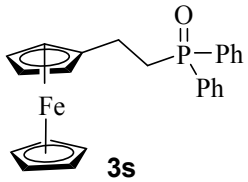
3q

(3, 5-bis(trifluoromethyl)phenethyl)diphenylphosphine oxide (3q). Following the general procedure (80 °C, 16 h), 1.0 equiv *t*-BuOK was loaded. **3q** was isolated as a white crystals (344.7 mg, 78%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (dd, 4H, *J* = 8.4 Hz), 7.67 (s, 1H), 7.60 (s, 2H), 7.46-7.56 (m, 6H), 3.104 (dt, 2H, *J* = 7.6 Hz, *J* = 8.8 Hz), 2.62 (dt, 2H, *J* = 7.6 Hz, *J* = 10.0 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 30.5; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.9; ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 143.3 (db, J_{P-C} = 13.6 Hz), 132.2 (d, J_{P-C} = 98.8 Hz), 132.1 (d, J_{P-C} = 2.7 Hz), 131.8 (q, J_{F-C} = 32.9 Hz), 131.72 (d, J_{P-C} = 9.4Hz), 128.6 (d, J_{P-C} = 11.7 Hz), 128.5 (q, J_{F-C} = 2.8 Hz), 123.2 (q, J_{F-C} = 271.0 Hz), 120.5 (dq, J_{P-C} = 3.7 Hz, J_{F-C} = 3.7 Hz), 31.3 (d, J_{P-C} = 69.6 Hz), 27.5 (d, J_{P-C} = 2.9 Hz). Melting point: 151.6~152.8 °C. MS (EI): 442. HRMS: Cal. for $C_{22}H_{17}F_6OP$ 442.0921. Found 442.0907.



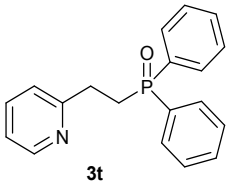
3r

(2-(naphthalene-1-yl)ethyl)diphenylphosphine oxide (3r). Following the general procedure, **3r** was isolated as a pale yellow crystals (284.8 mg, 80%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78-7.87 (m, 6H), 7.70 (d, 1H, *J* = 8.0 Hz), 7.46-7.55 (m, 8H), 7.29-7.37 (m, 2H), 3.36-3.42 (m, 2H), 2.66-2.72 (m, 2H). ^{31}P NMR (162 MHz, $CDCl_3$): δ 31.5. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 137.3 (d, J_{C-P} = 14.5 Hz), 133.9, 132.9 (d, J_{C-P} = 97.6 Hz), 131.9 (d, J_{C-P} = 2.6 Hz), 131.3, 130.8 (d, J_{C-P} = 9.3 Hz), 128.9, 128.8 (d, J_{C-P} = 11.5 Hz), 127.2, 126.19, 125.8, 125.7, 125.6, 123.3, 31.2 (d, J_{C-P} = 69.0 Hz), 24.8 (d, J_{C-P} = 2.8 Hz). Melting point: 122.9~123.3 °C. HRMS: Cal. for $C_{24}H_{21}OP$ 356.1330. Found 356.1318.



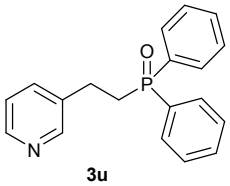
3s

Ferrocenetheyldiphenylphosphine oxide (3s). Following the general procedure, **3s** was isolated as a yellow crystals (339.4 mg, 82%). ^{1}H NMR (400 MHz $CDCl_3$): δ 7.76 (dd, 4H, *J* = 7.6 Hz, *J* = 7.6 Hz), 7.48-7.54 (m, 6H), 4.06 (s, 5H), 4.03 (b, 4H), 2.62-2.68 (m, 2H), 2.46-2.52 (m, 2H). ^{31}P NMR (162 MHz $CDCl_3$): δ 31.59. ^{13}C NMR (100 MHz $CDCl_3$): δ 132.89 (d, J_{C-P} = 97.6 Hz), 131.81 (d, J_{C-P} = 2.7 Hz), 130.78 (d, J_{C-P} = 9.3 Hz), 128.74 (d, J_{C-P} = 11.5 Hz), 88.30 (d, J_{C-P} = 18.1 Hz), 68.60, 67.70, 67.44, 31.47 (d, J_{C-P} = 70.0 Hz), 21.55 (d, J_{C-P} = 2.8 Hz). Melting point: 170.8~170.9 °C.



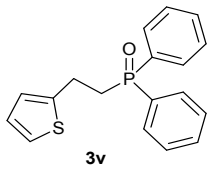
3t

Diphenyl(2-(priding-2-yl)ethyl)phosphine oxide (3t).[1, 2] Following the general procedure. **3t** was isolated as a pale yellow crystals (214.9 mg, 70%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.47 (d, 2H, J = 4.8 Hz), 7.77 (dd, 4H, J = 7.6 Hz, J = 10.8 Hz), 7.42-7.53 (m, 7H), 7.11 (d, 1H, J = 8.0 Hz), 7.06 (dd, 1H, J = 6.4 Hz, J = 6.4 Hz), 3.07-3.13 (m, 2H), 2.74-2.81 (m, 2H). ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.3. ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.2 (d, $J_{C\text{-}P}$ = 14.4 Hz), 149.3, 136.5, 132.8 (d, $J_{C\text{-}P}$ = 98.9 Hz), 131.7 (d, $J_{C\text{-}P}$ = 2.7 Hz), 130.8 (d, $J_{C\text{-}P}$ = 9.3 Hz), 128.7 (d, $J_{C\text{-}P}$ = 11.6 Hz), 123.1, 121.5, 29.7 (d, $J_{C\text{-}P}$ = 2.7 Hz), 29.2 (d, $J_{C\text{-}P}$ = 71.1 Hz). This compound is known.



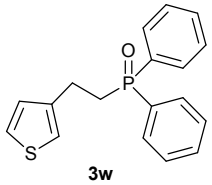
3u

Diphenyl(2-(priding-3-yl)ethyl)phosphine oxide (3u). Following the general procedure, **3u** was isolated as a pale yellow crystals (211.8 mg, 69%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.39 (b, 2H), 7.74 (dd, 4H, J = 8.4 Hz, J = 10.0 Hz), 7.46-7.53 (m, 7H), 7.14 (b, 1H), 2.92 (dt, 2H, J = 7.6 Hz, J = 8.0 Hz), 2.55 (dt, 2H, J = 6.8 Hz, J = 10.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 31.3. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 149.5, 147.8, 136.4 (d, $J_{C\text{-}P}$ = 14.3 Hz), 135.7, 132.4 (d, $J_{C\text{-}P}$ = 98.3 Hz), 132.0 (d, $J_{C\text{-}P}$ = 2.6 Hz), 130.7 (d, $J_{C\text{-}P}$ = 9.3 Hz), 128.8 (d, $J_{C\text{-}P}$ = 11.6 Hz), 123.5, 31.5 (d, $J_{C\text{-}P}$ = 69.6 Hz), 24.9 (d, $J_{C\text{-}P}$ = 2.9 Hz). Melting point: 104.0~104.6 ^{o}C. HRMS: Cal. for $C_{19}H_{18}NOP$ 307.1126. Found 307.1120.



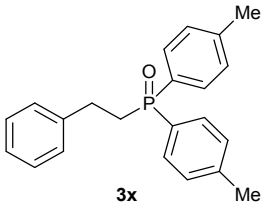
3v

Diphenyl(2-thiophen-2-yl)ethyl)phosphine oxide (3v). Following the general procedure, **3v** was isolated as a pale yellow crystals (227.7 mg, 73%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (dd, 4H, J = 7.6 Hz, J = 10.8 Hz), 7.44-7.53 (m, 6H), 7.07 (d, 1H, J = 4.8 Hz), 6.84 (dd, 1H, J = 4.0 Hz, J = 4.0 Hz), 6.76 (b, 1H), 3.10-3.16 (m, 2H), 2.61-2.68 (m, 2H), ^{31}P NMR (162 MHz, $CDCl_3$): δ 31.2. ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.8 (d, $J_{C\text{-}P}$ = 17.3 Hz), 132.5 (d, $J_{C\text{-}P}$ = 97.1 Hz), 131.9 (d, $J_{C\text{-}P}$ = 2.5 Hz), 130.8 (d, J = 9.4 Hz), 128.8 (d, J = 11.7 Hz), 126.9, 124.6, 123.6, 32.2 (d, $J_{C\text{-}P}$ = 69.2 Hz), 22.1 (d, $J_{C\text{-}P}$ = 2.4 Hz). Melting point: 125.2~127.9 ^{o}C. HRMS: Cal. for $C_{18}H_{17}OPS$ 312.0738. Found 312.0731.



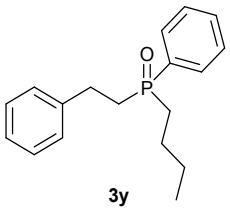
3w

Diphenyl(thiophen-3-yl)ethyl)phosphine oxide (3w). Following the general procedure, 1.0 equiv *t*-BuOK was loaded. **3w** was isolated as a pale yellow crystals (268.3 mg, 86%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (dd, 4H, J = 7.6 Hz, J = 10.2 Hz), 7.45-7.54 (m, 6H), 7.22 (b, 1H), 6.94 (b, 1H), 6.90 (d, 1H, J = 4.8 Hz), 2.93-2.99 (m, 2H), 2.55-2.62 (m, 2H). ^{31}P NMR (162 MHz, $CDCl_3$): δ 31.47. ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.33 (d, $J_{C\text{-}P}$ = 15.7 Hz), 132.75 (d, $J_{C\text{-}P}$ = 97.8 Hz), 131.84 (d, $J_{C\text{-}P}$ = 2.7 Hz), 130.79 (d, $J_{C\text{-}P}$ = 9.3 Hz), 128.74 (d, $J_{C\text{-}P}$ = 11.6 Hz), 127.73, 125.90, 120.53, 30.97 (d, $J_{C\text{-}P}$ = 69.8 Hz), 22.22 (d, $J_{C\text{-}P}$ = 2.8 Hz). Melting point: 111.2~111.7 °C. HRMS: Cal. for $C_{18}H_{17}OPS$ 312.0738. Found 312.0722.



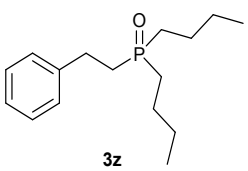
3x

Phenethyldi-*p*-tolylphosphine oxide (3x). Following the general procedure, 1.0 equiv *t*-BuOK was loaded. **3x** was isolated as a white crystals (273.8 mg, 82%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.65 (dd, 4H, J = 8.0 Hz, J = 10.8 Hz), 7.22-7.27 (m, 6H), 7.14-7.16 (m, 3H), 2.89-2.95 (m, 2H), 2.50-2.57 (m, 2H), 2.37 (s, 6H). ^{31}P NMR (162 MHz, $CDCl_3$): δ 31.8. ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.2 (d, $J_{C\text{-}P}$ = 2.7 Hz), 141.4 (d, $J_{C\text{-}P}$ = 15.3 Hz), 130.8 (d, $J_{C\text{-}P}$ = 9.6 Hz), 129.7 (d, $J_{C\text{-}P}$ = 100.1 Hz), 129.4 (d, $J_{C\text{-}P}$ = 11.9 Hz), 128.6, 128.1, 126.3, 32.1 (d, $J_{C\text{-}P}$ = 69.6 Hz), 27.6 (d, $J_{C\text{-}P}$ = 3.0 Hz), 21.6. Melting point: 99.5~100.4 °C. HRMS: Cal. for $C_{22}H_{23}OP$ 334.1487. Found 334.1480.



3y

Butyl(phenethyl)(phenyl)phosphine oxide (3y). Following the general procedure, 0.5 equiv *t*-BuOK was loaded. **3y** was isolated as a colorless oil (225.9 mg, 79%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (dd, 2H, J = 8.0 Hz, J = 8.0 Hz), 7.43-7.50 (m, 3H), 7.20 (dd, 2H, J = 7.6 Hz, J = 7.6 Hz), 7.08-7.13 (m, 3H), 2.89-2.99 (m, 1H), 2.61-2.71 (m, 1H), 2.27-2.33 (m, 1H), 2.07-2.17 (m, 1H), 1.78-1.99 (m, 2H), 1.54-1.62 (m, 1H), 1.27-1.43 (m, 3H), 0.82 (t, 3H, J = 7.2 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.7. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 141.2 (d, $J_{C\text{-}P}$ = 14.2 Hz), 132.3 (d, $J_{C\text{-}P}$ = 91.6 Hz), 131.6 (d, $J_{C\text{-}P}$ = 2.6 Hz), 130.4 (d, $J_{C\text{-}P}$ = 8.7 Hz), 128.7 (d, $J_{C\text{-}P}$ = 11.1 Hz), 128.6, 128.0, 126.3, 31.9 (d, $J_{C\text{-}P}$ = 65.7 Hz), 29.8 (d, $J_{C\text{-}P}$ = 68.1 Hz), 27.5 (d, $J_{C\text{-}P}$ = 3.3 Hz), 24.0 (d, $J_{C\text{-}P}$ = 14.4 Hz), 23.5 (d, $J_{C\text{-}P}$ = 4.1 Hz), 13.5. HRMS: Cal. for $C_{18}H_{23}OP$ 286.1487. Found 286.1474.



3z

Dibutyl(phenethyl)phosphine oxide (3z). Following the general procedure, 1.0 equiv *t*-BuOK was loaded. **3z** was isolated as a colorless oil (170.2 mg, 64%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 (dd, 2H, J = 6.0 Hz, J = 6.0 Hz), 7.23 (parad, 3H, J = 6.0 Hz), 2.90-2.95 (m, 2H), 1.99-2.10 (m, 2H), 1.68-1.73 (m, 4H), 1.53-1.57 (m, 4H), 1.38-1.45 (m, 4H), 0.93 (t, 6H, J = 6.0 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 48.2. ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 141.2 (d, $J_{C\text{-}P}$ = 10.3 Hz), 128.7, 128.1, 126.4, 29.7 (d, $J_{C\text{-}P}$ = 49.7 Hz), 27.8 (d, $J_{C\text{-}P}$ = 51.7 Hz), 27.7 (d, $J_{C\text{-}P}$ = 2.5 Hz), 24.3 (d, $J_{C\text{-}P}$ = 11.4 Hz), 23.8 (d, $J_{C\text{-}P}$ = 3.0 Hz), 13.6. MS (EI): 266.

References:

(1) Jr R. A. S.; Taylor R. I.; Thompson L. E.; Patel P. B. *Org. Lett.* **2005**, *7*, 851.

(2) Isley N. A.; Linstadt R. T. H.; Slack E. D.; Lipshutz B. H. *Dalton Trans.* **2014**, *43*, 13196.

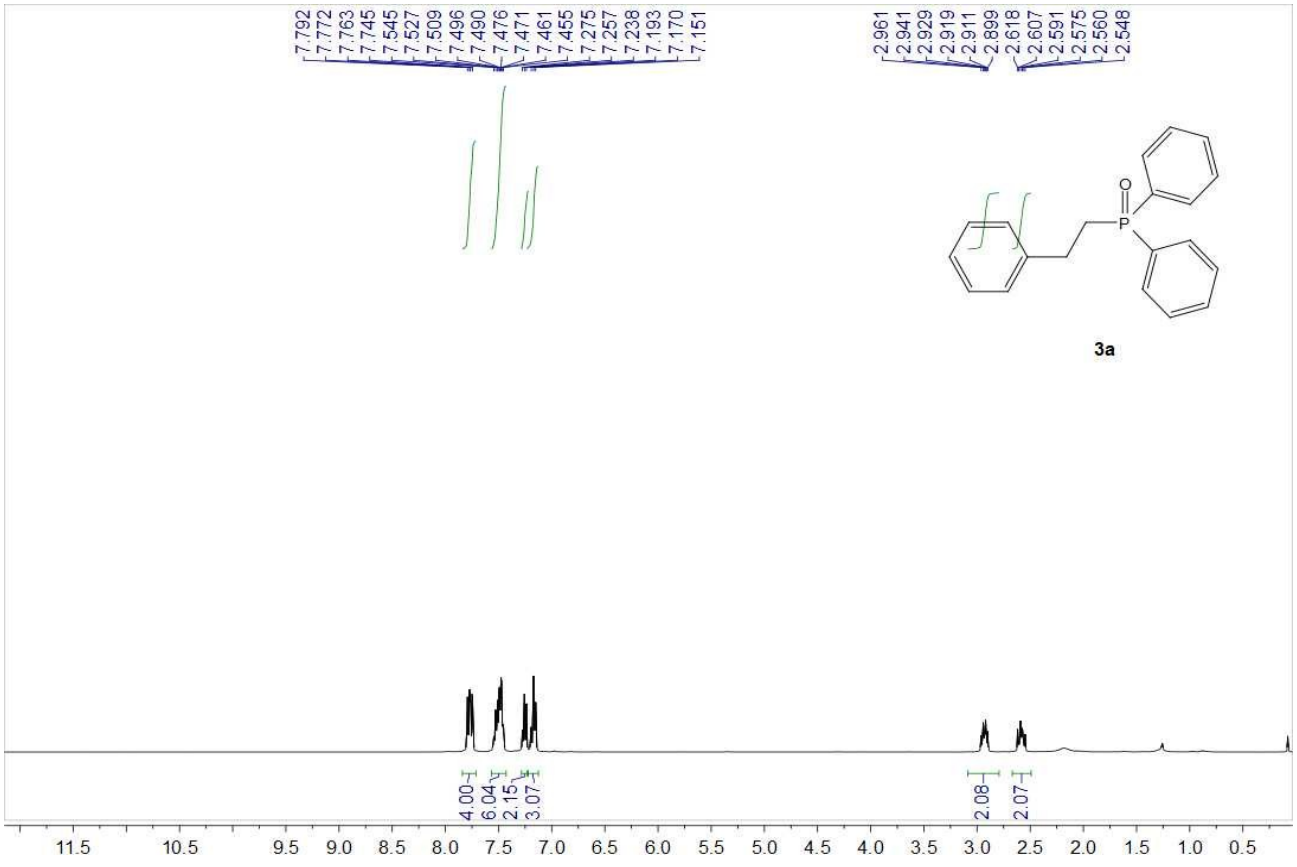
(3) Miao W.; Gao Y.; Li X.; Gao Y.; Tang G. Zhao Y. *Adv. Synth. Catal.* **2012**, *354*, 2659.

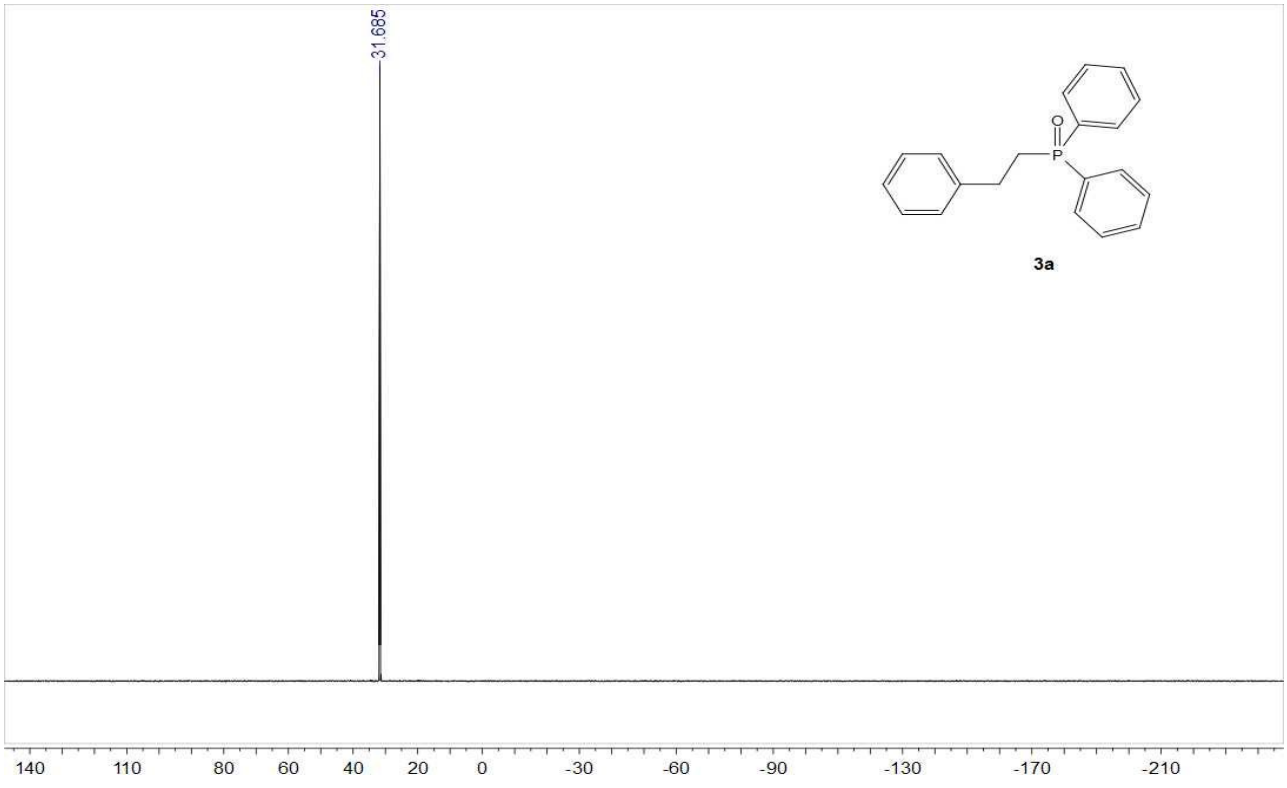
(4) Niu M.; Fu H.; Jiang Y.; Zhao Y. *Chem. Commun.* **2007**, 272. DOI: 10.1039/B613416E.

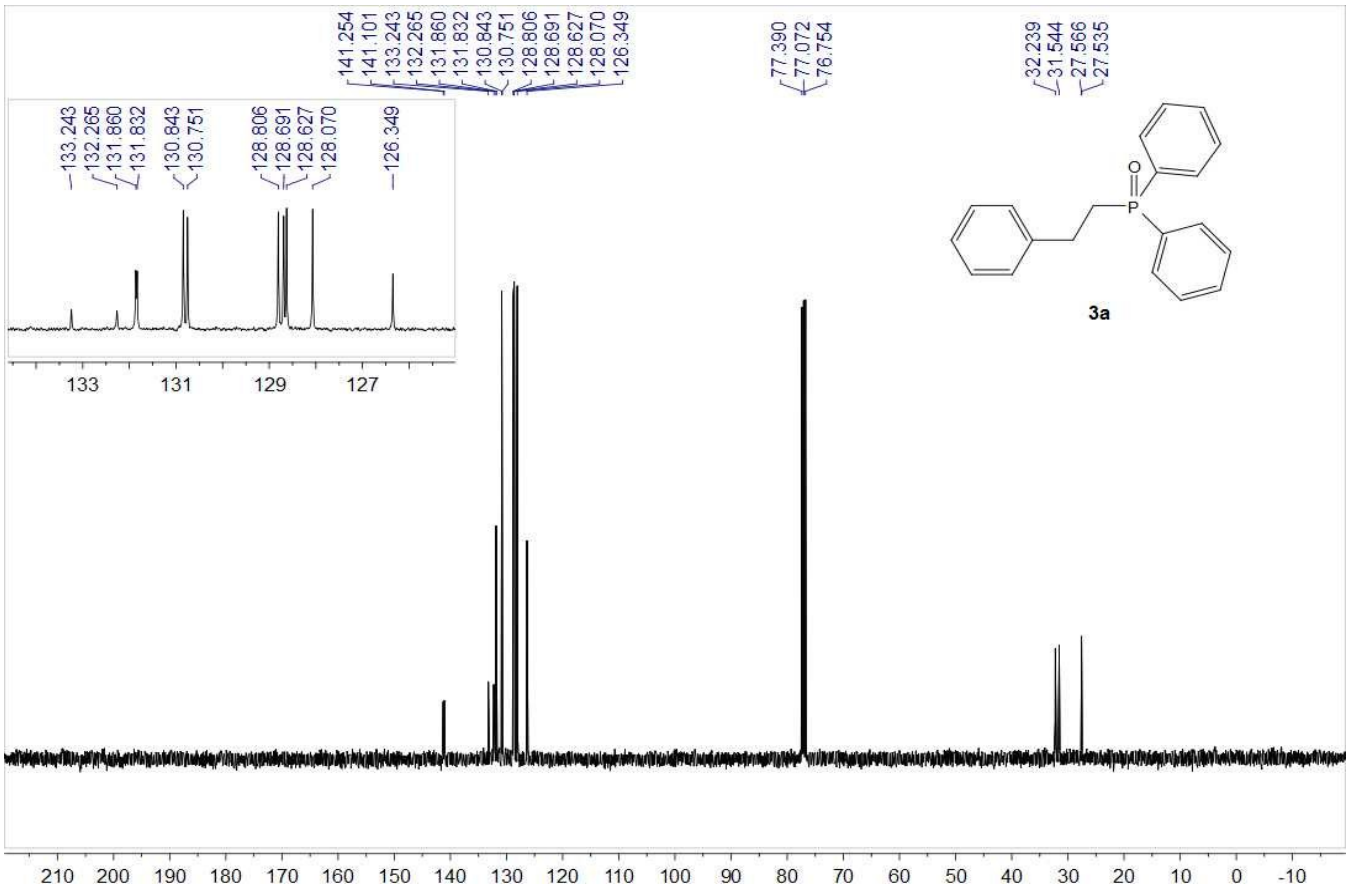
(5) Eisch J. J.; Liu W.; Zhu L.; Rheingold A. L. *Eur. J. Org. Chem.* **2015**, *2015*, 7384.

(6) A. Yoshimura, Y. Saga, Y. Sato, A. Ogawa, T. Chen and L.-B. Han, *Tetrahedron Lett.*, **2016**, 57, 3382.

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



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77.072
76.754
32.239
31.544
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P
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131
129
127
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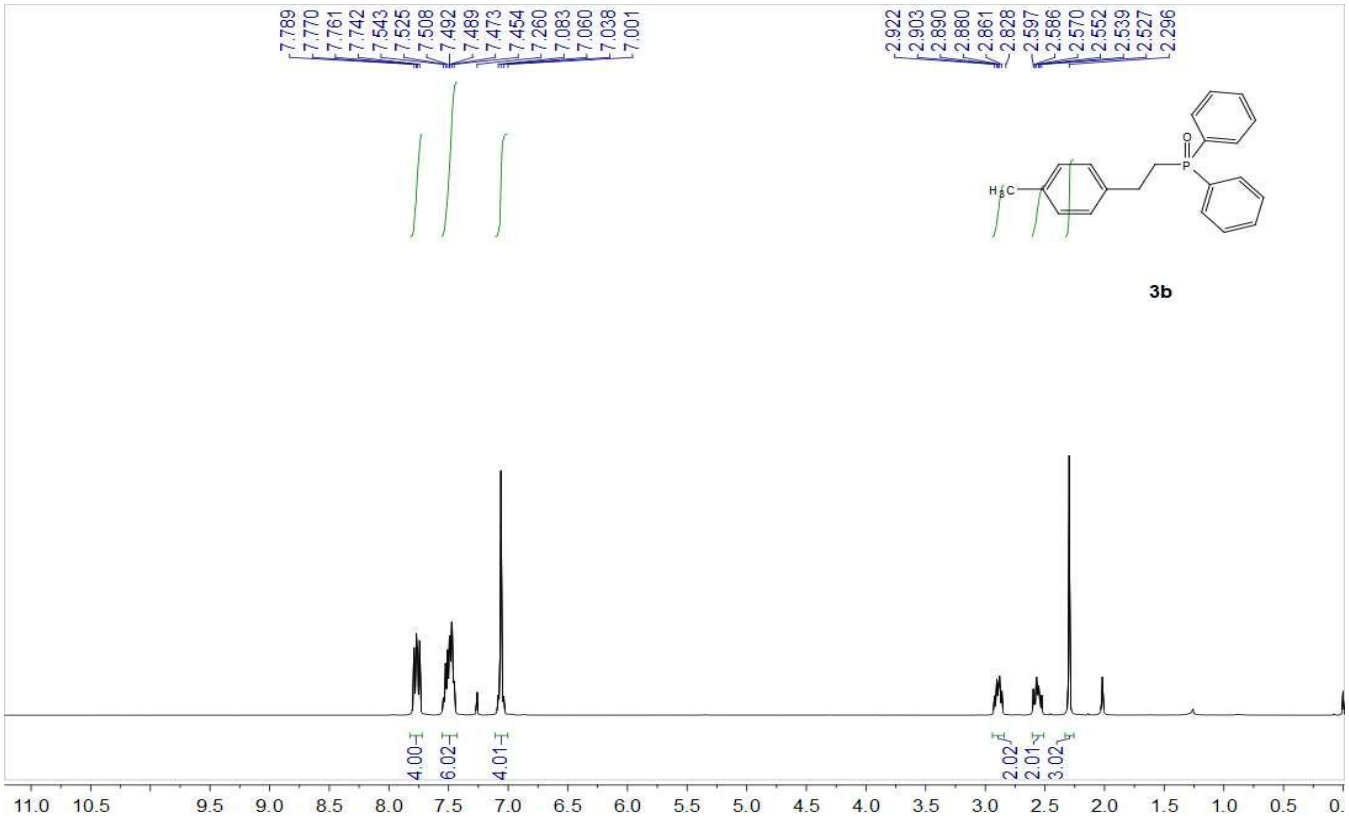
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2.828
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2.527
2.296
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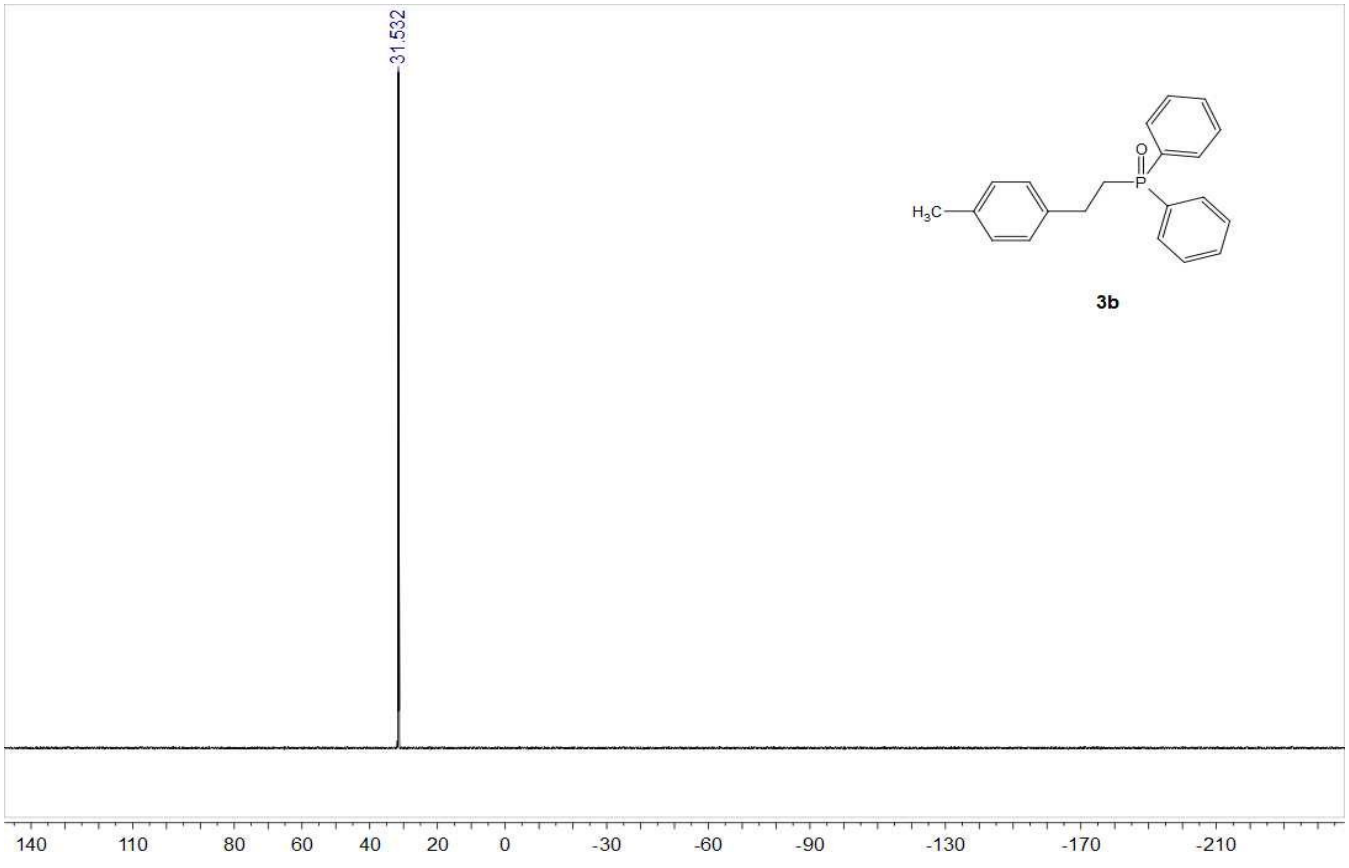
P
H3C
3b
4.00
6.02
4.01
2.02
2.01
3.02
11.0 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5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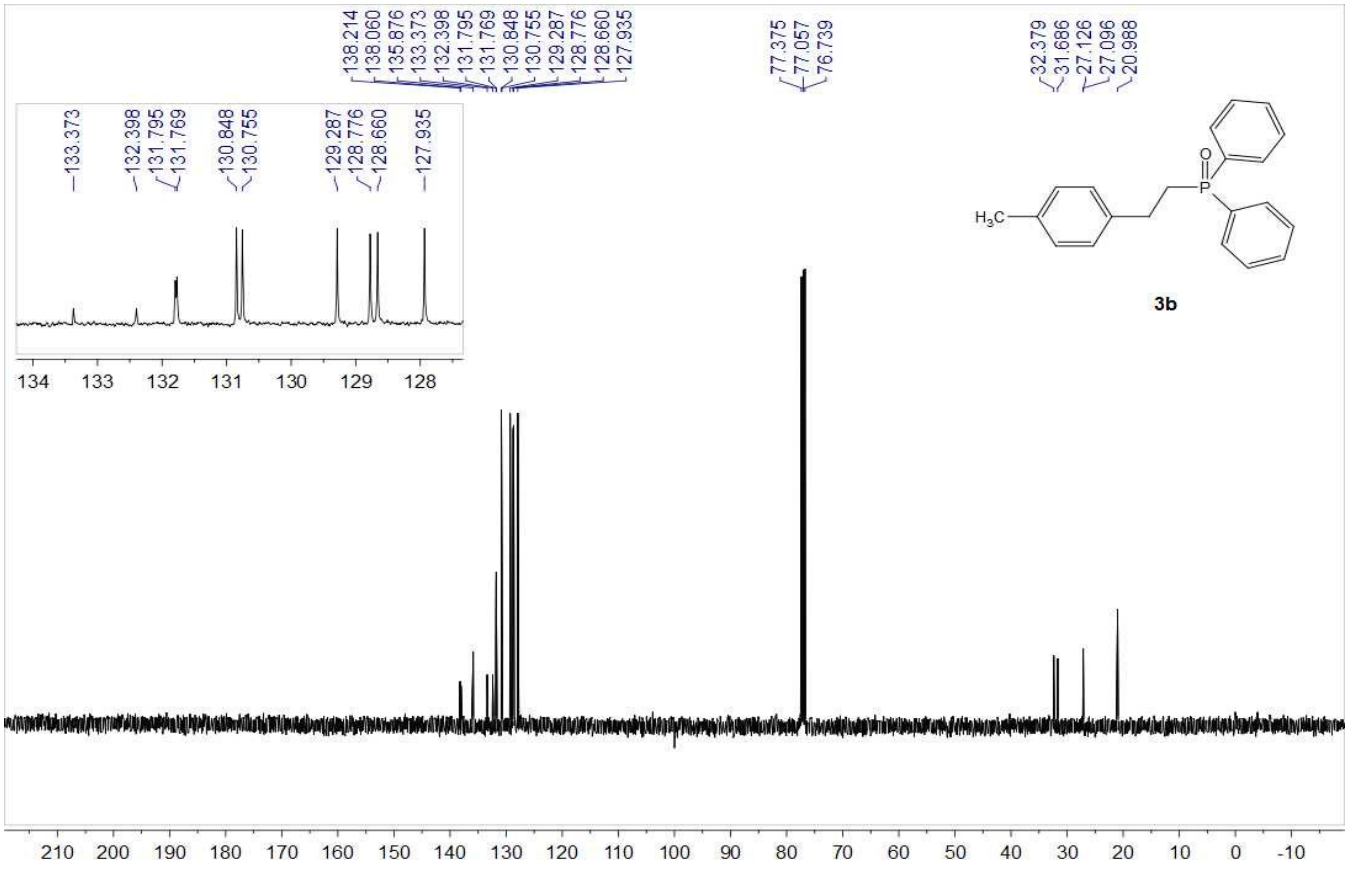
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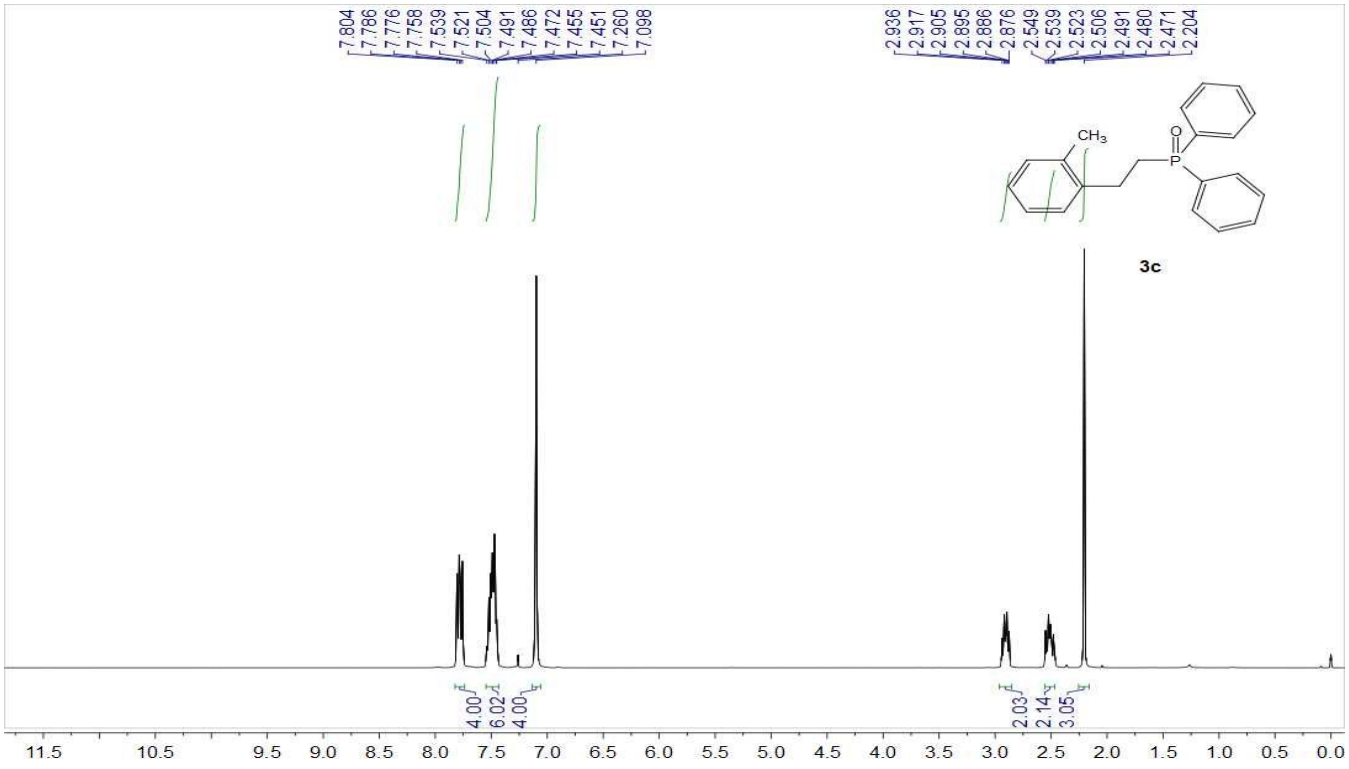
P
3b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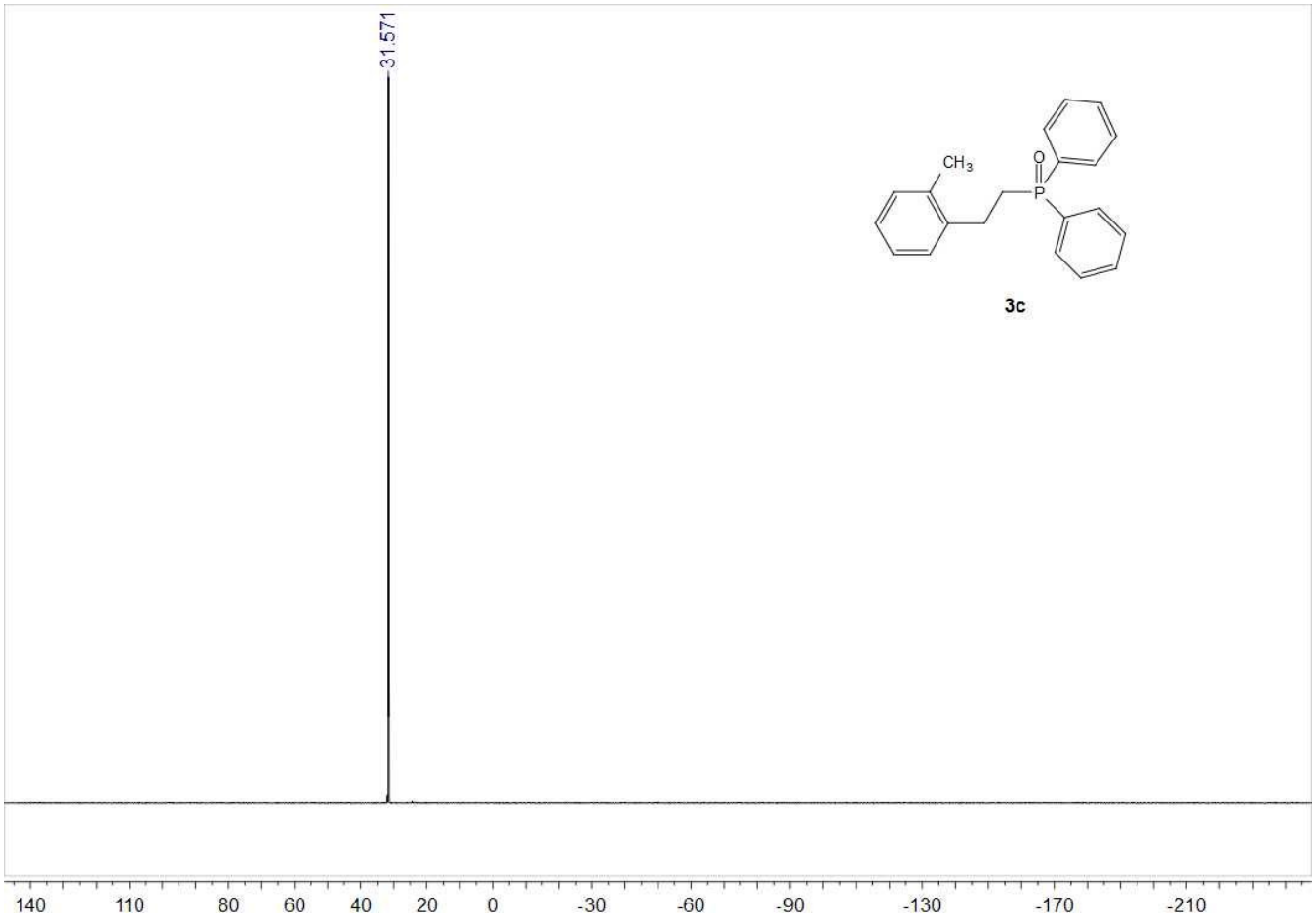
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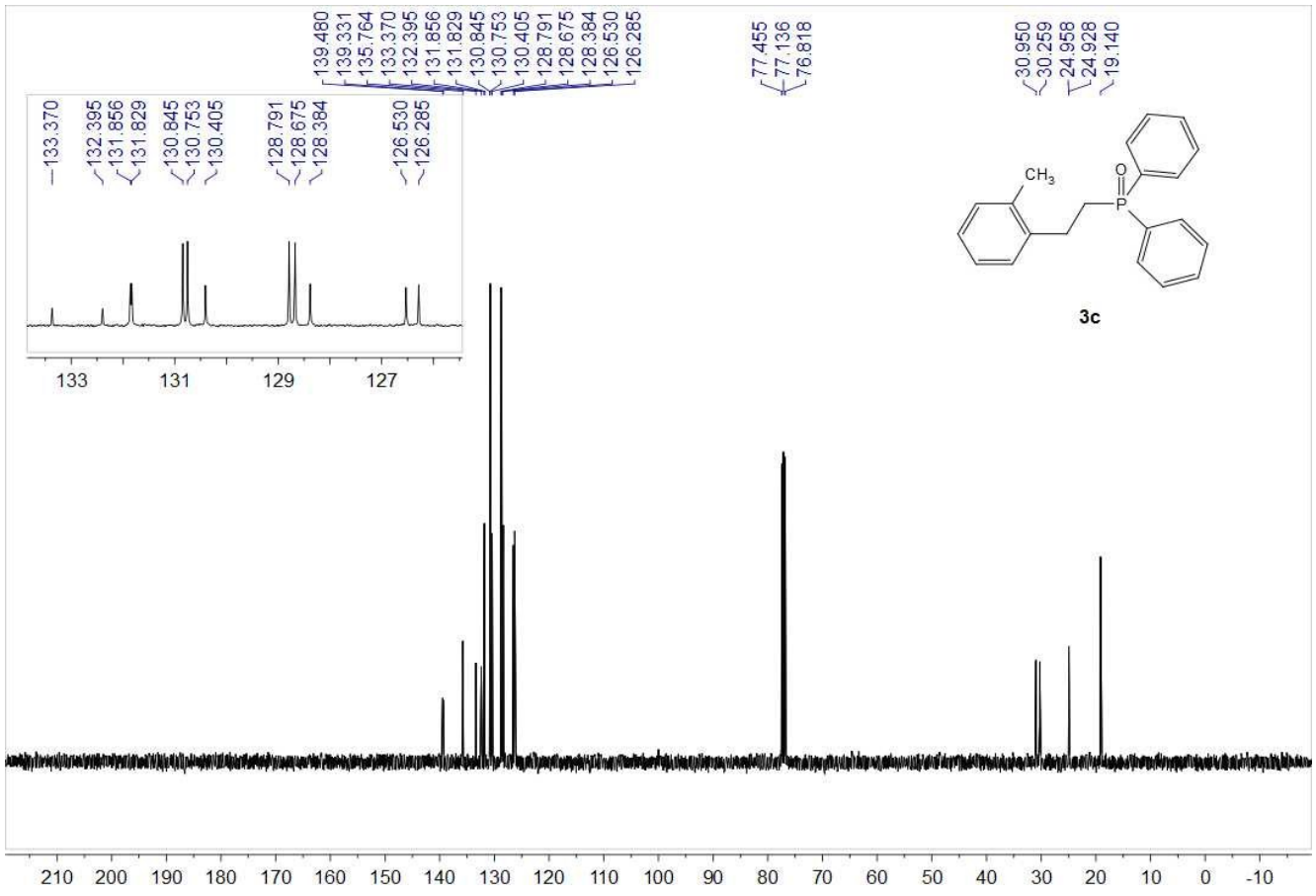
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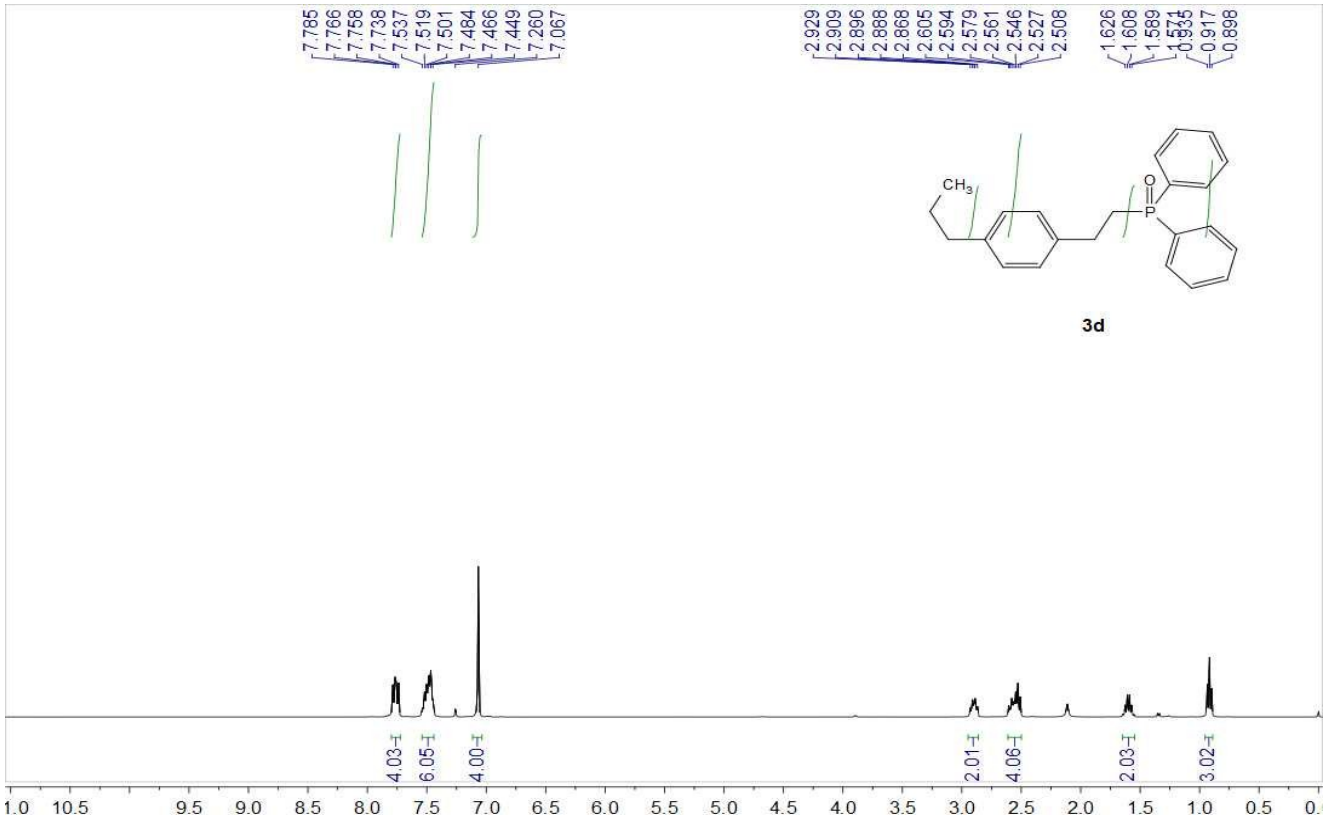
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3.02
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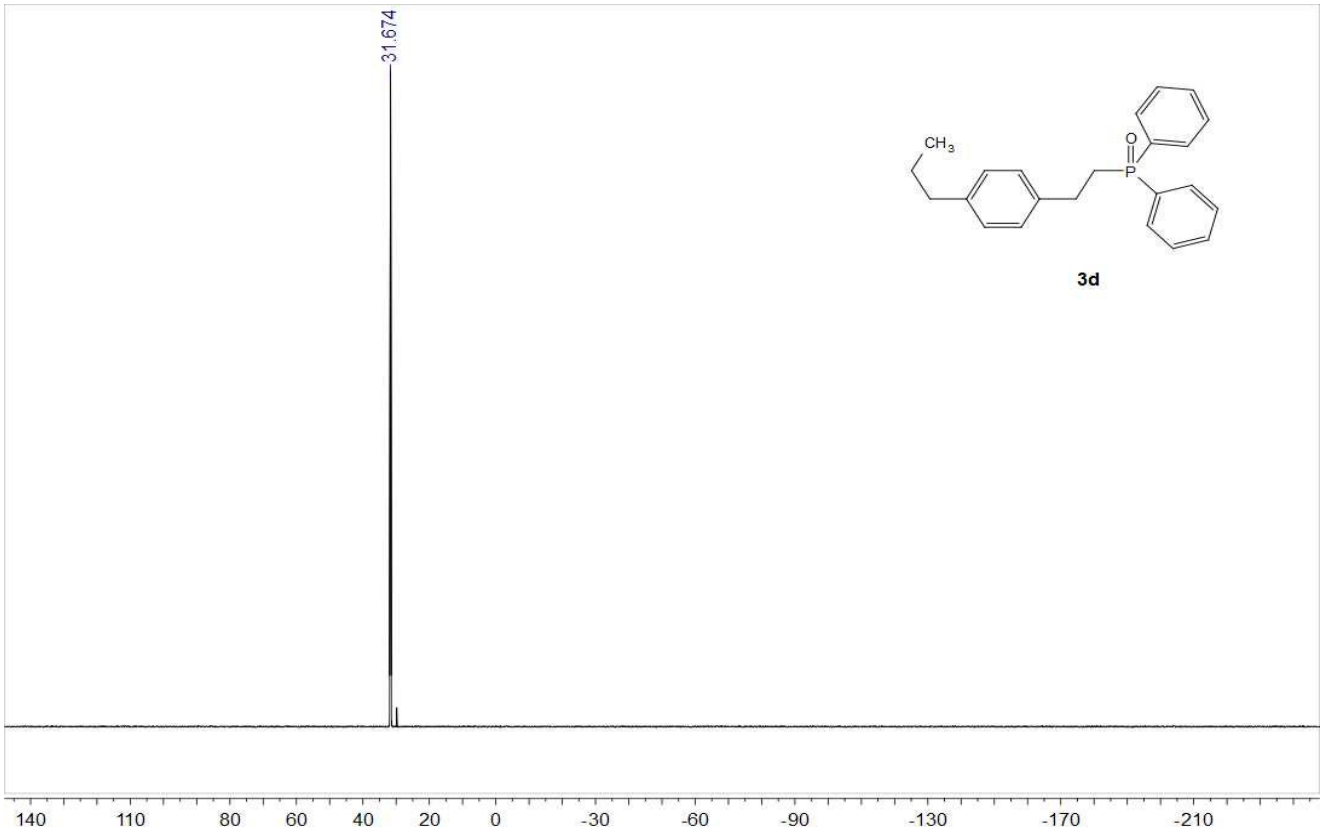
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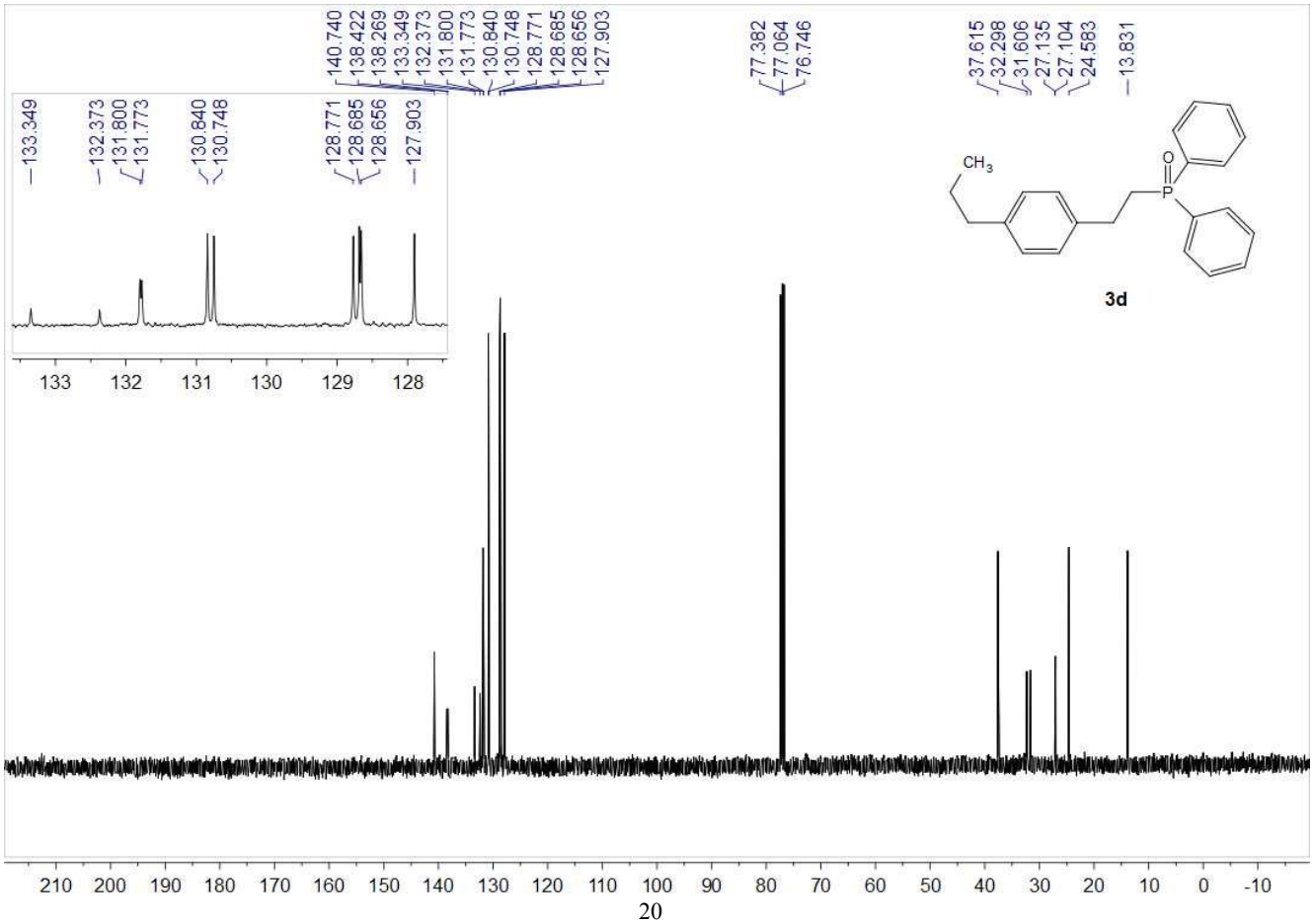
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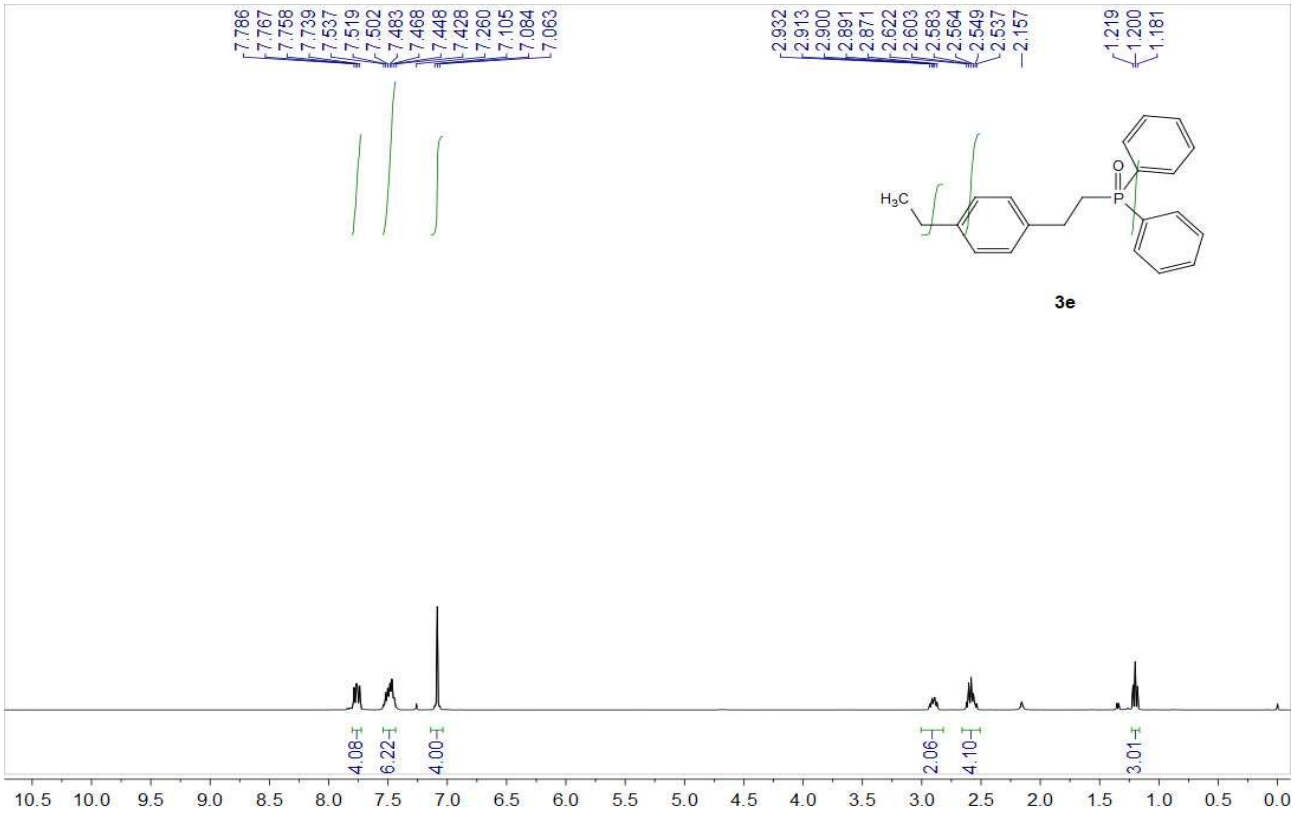
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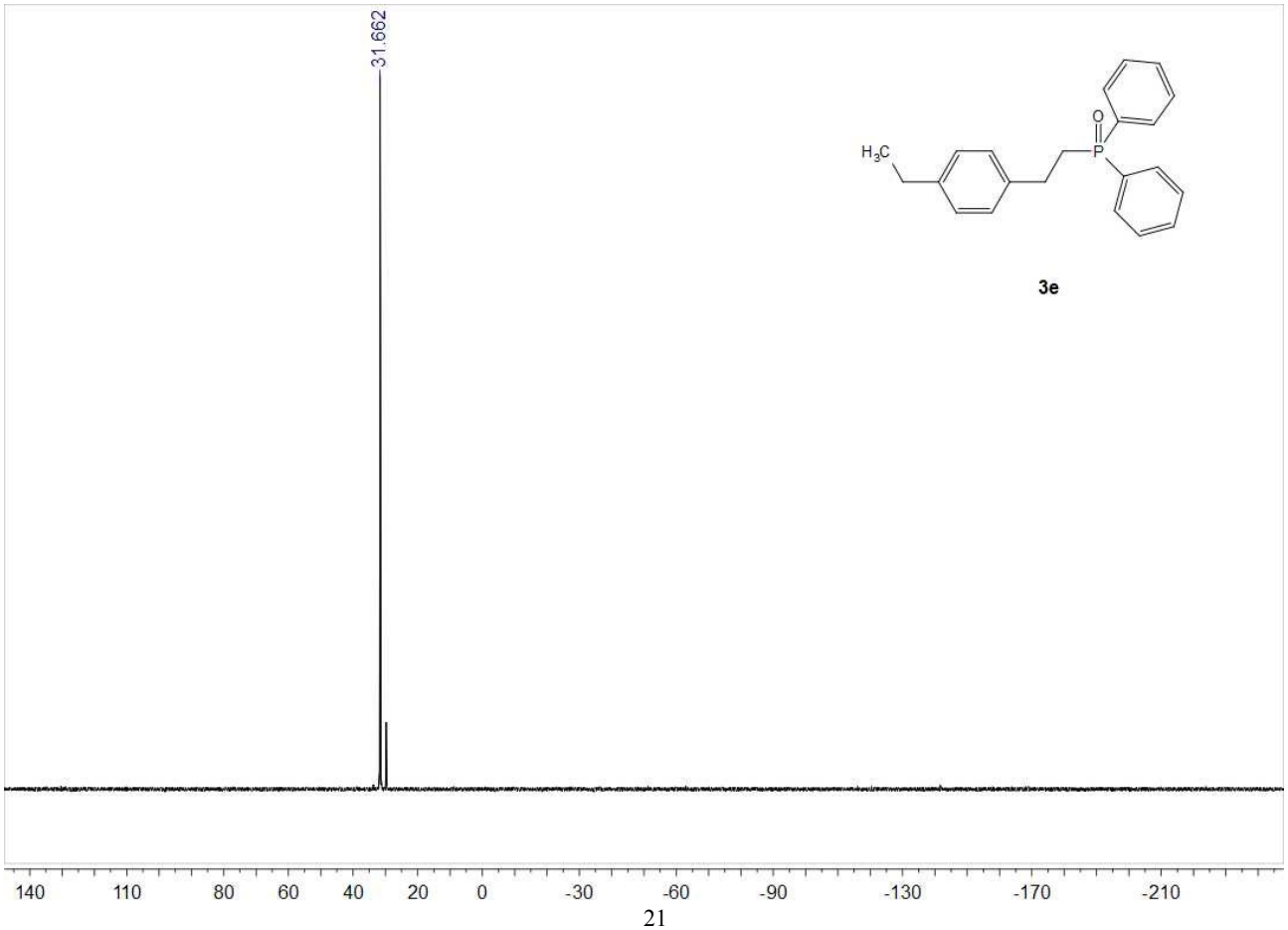
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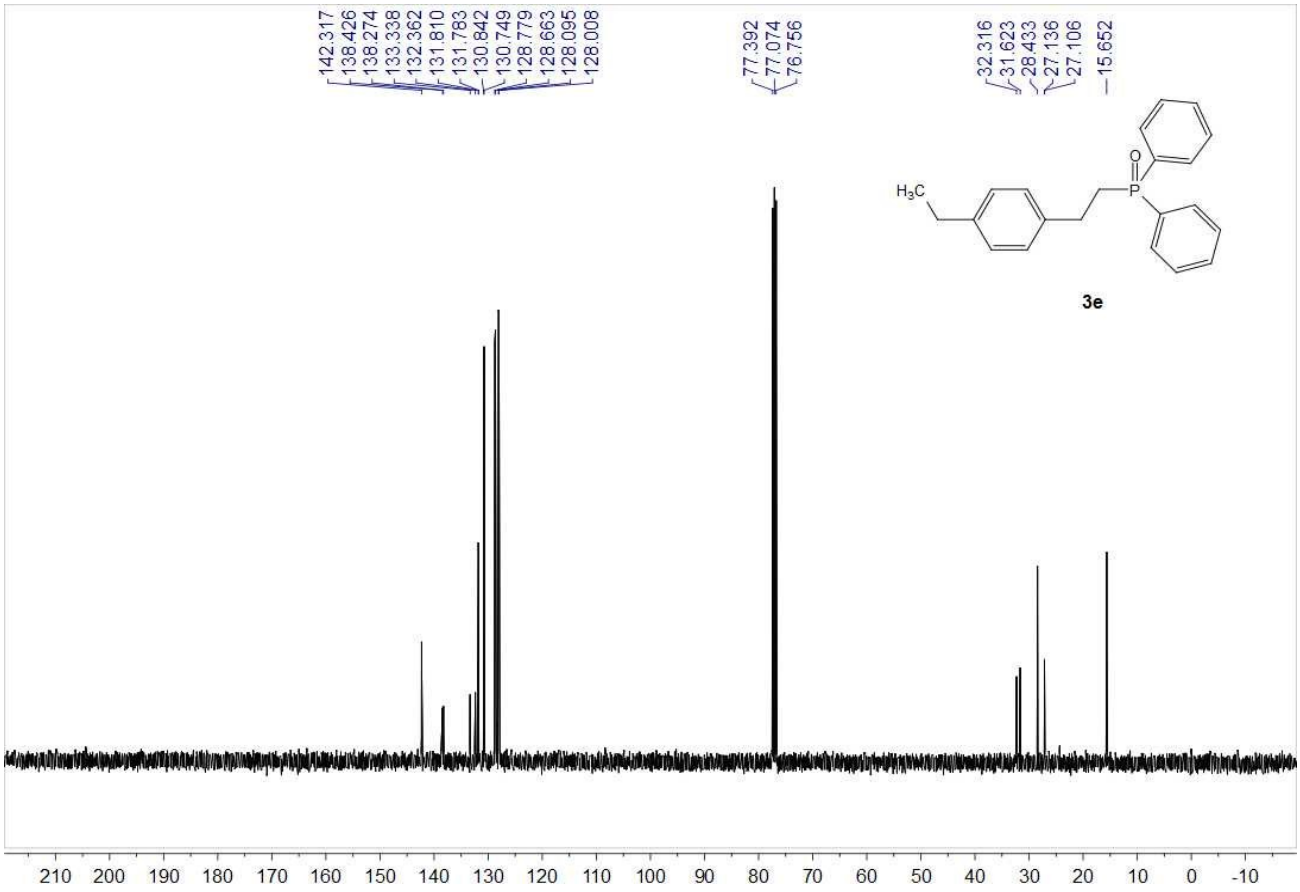
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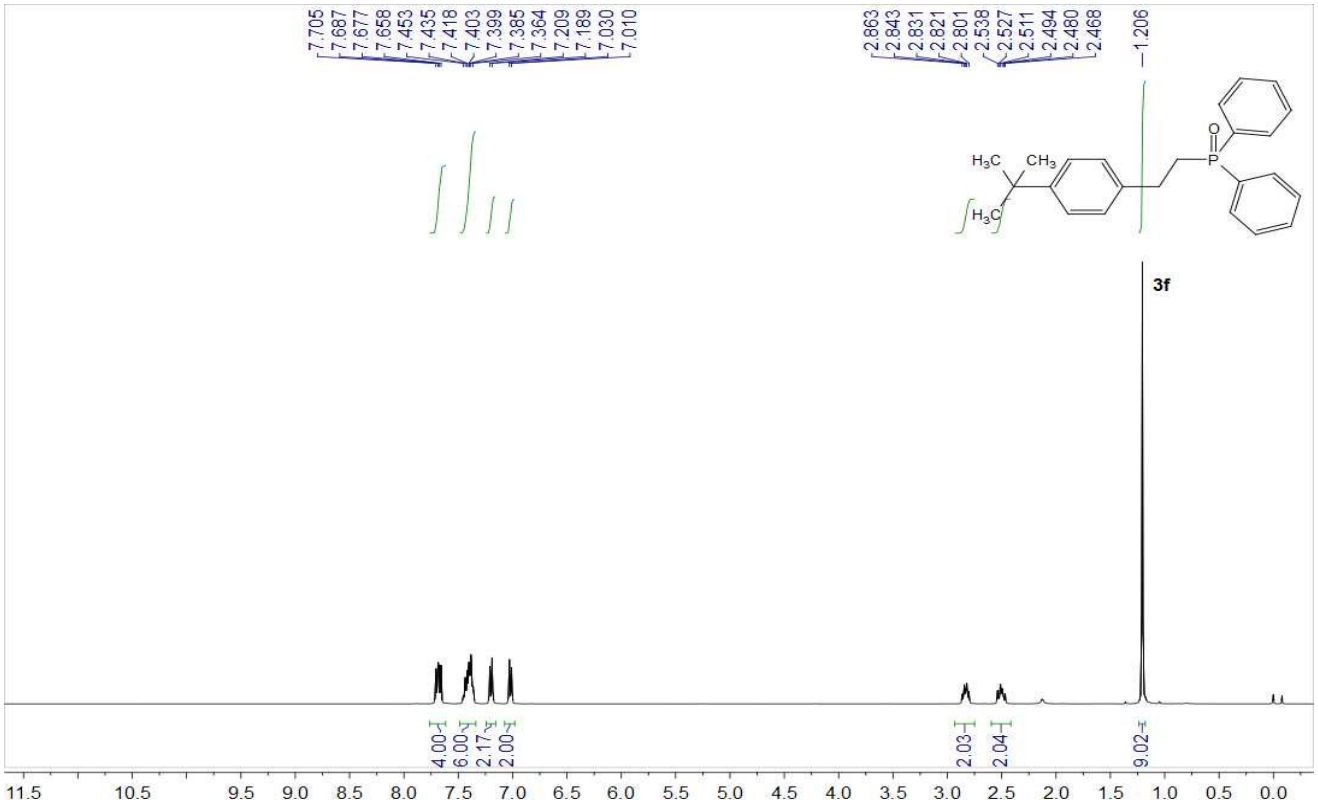
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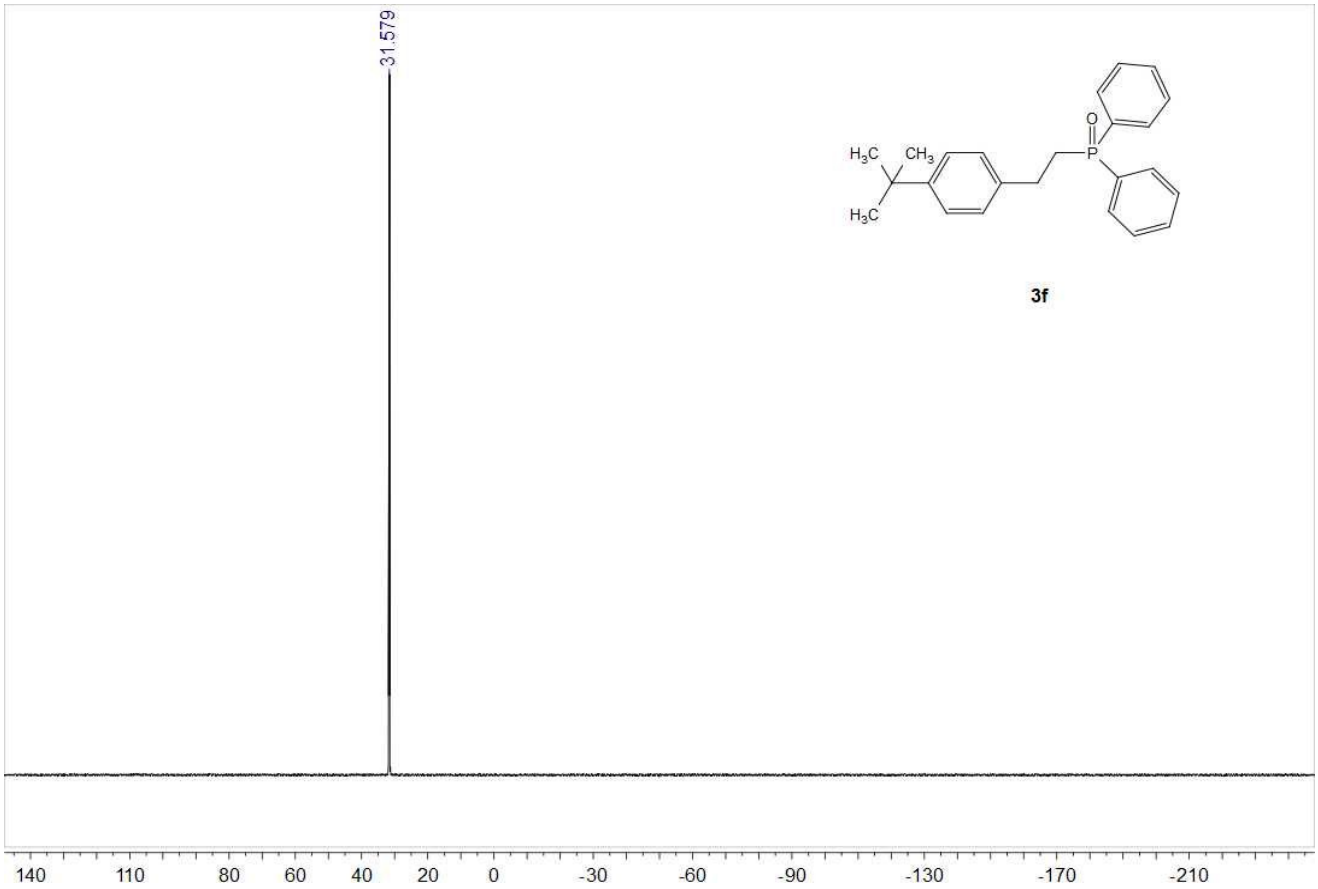
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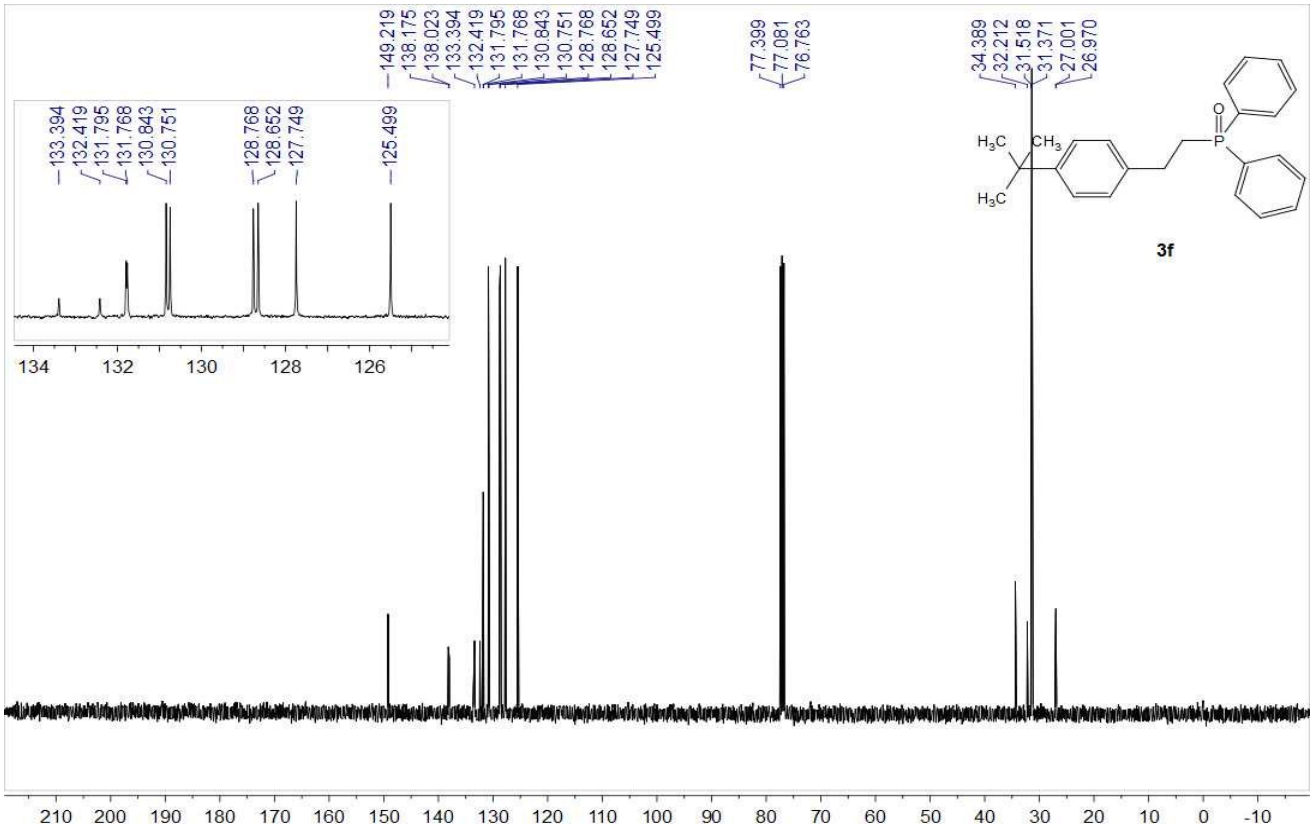
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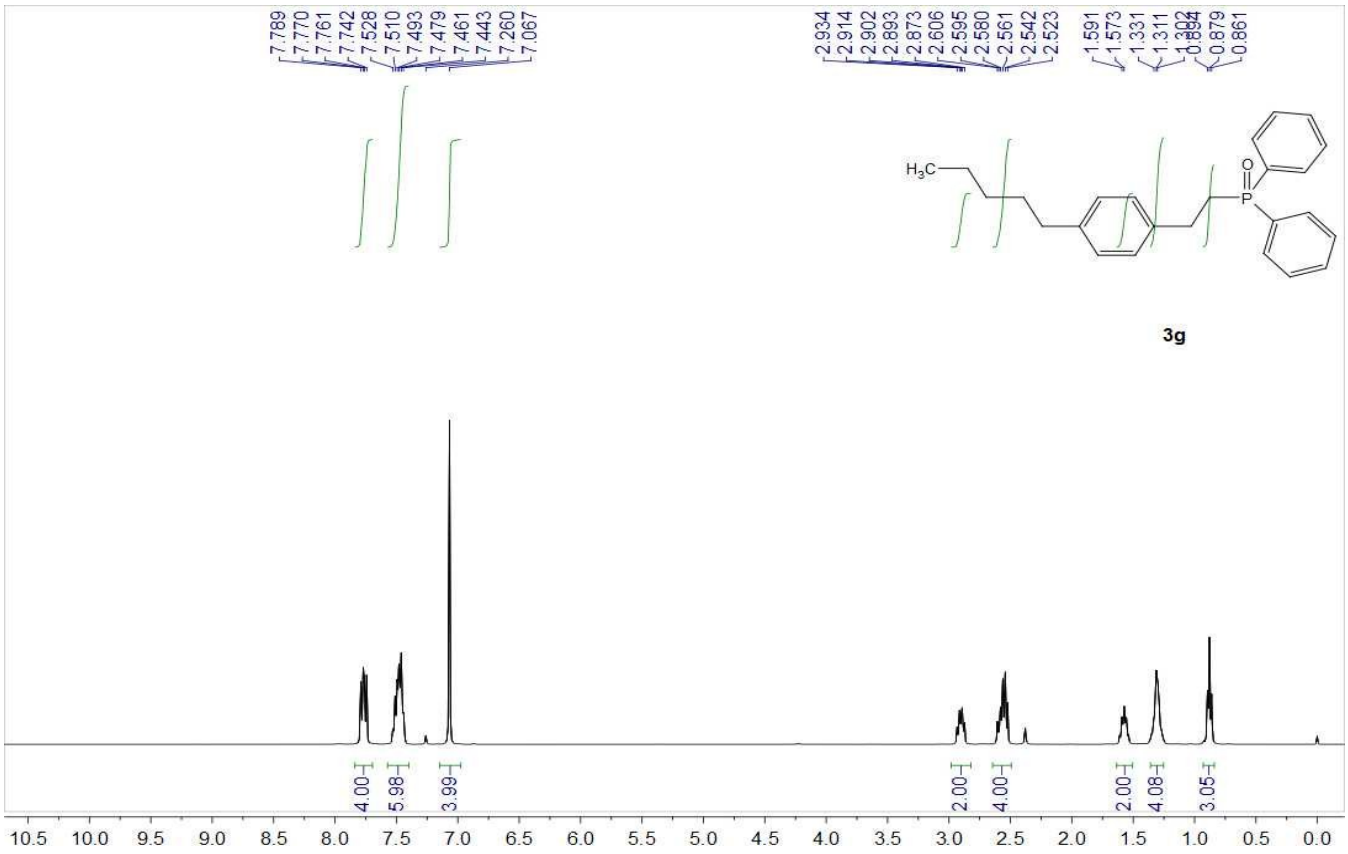
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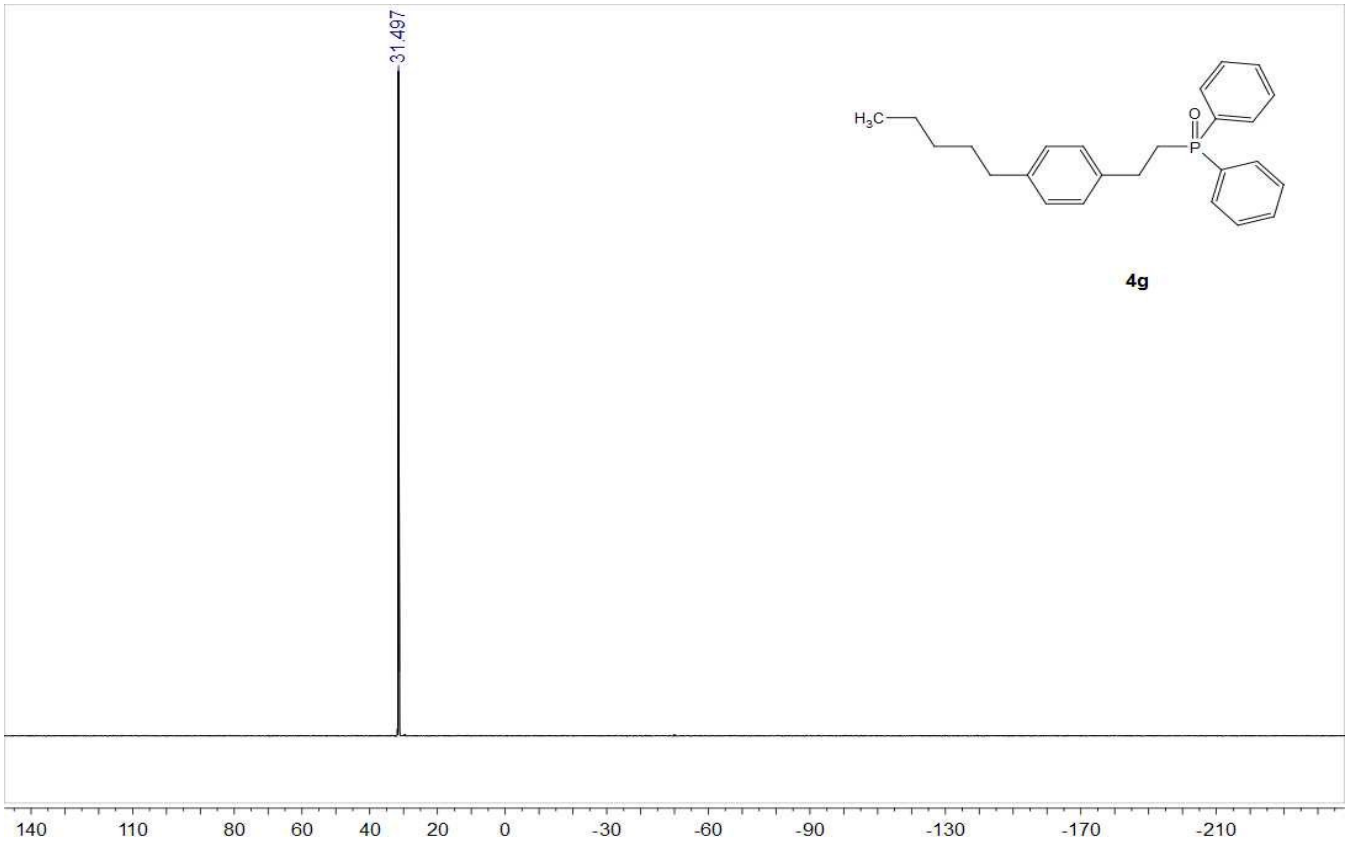
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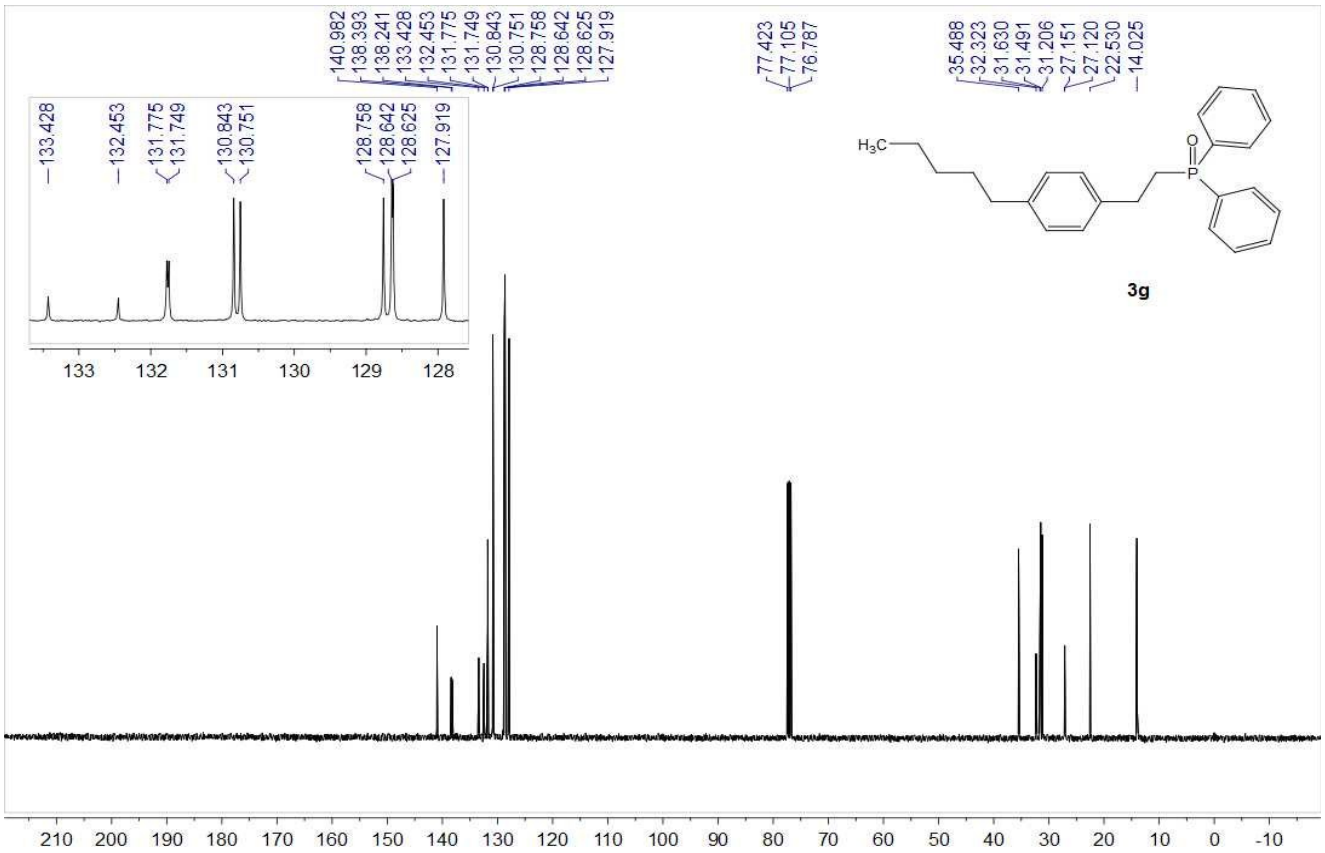
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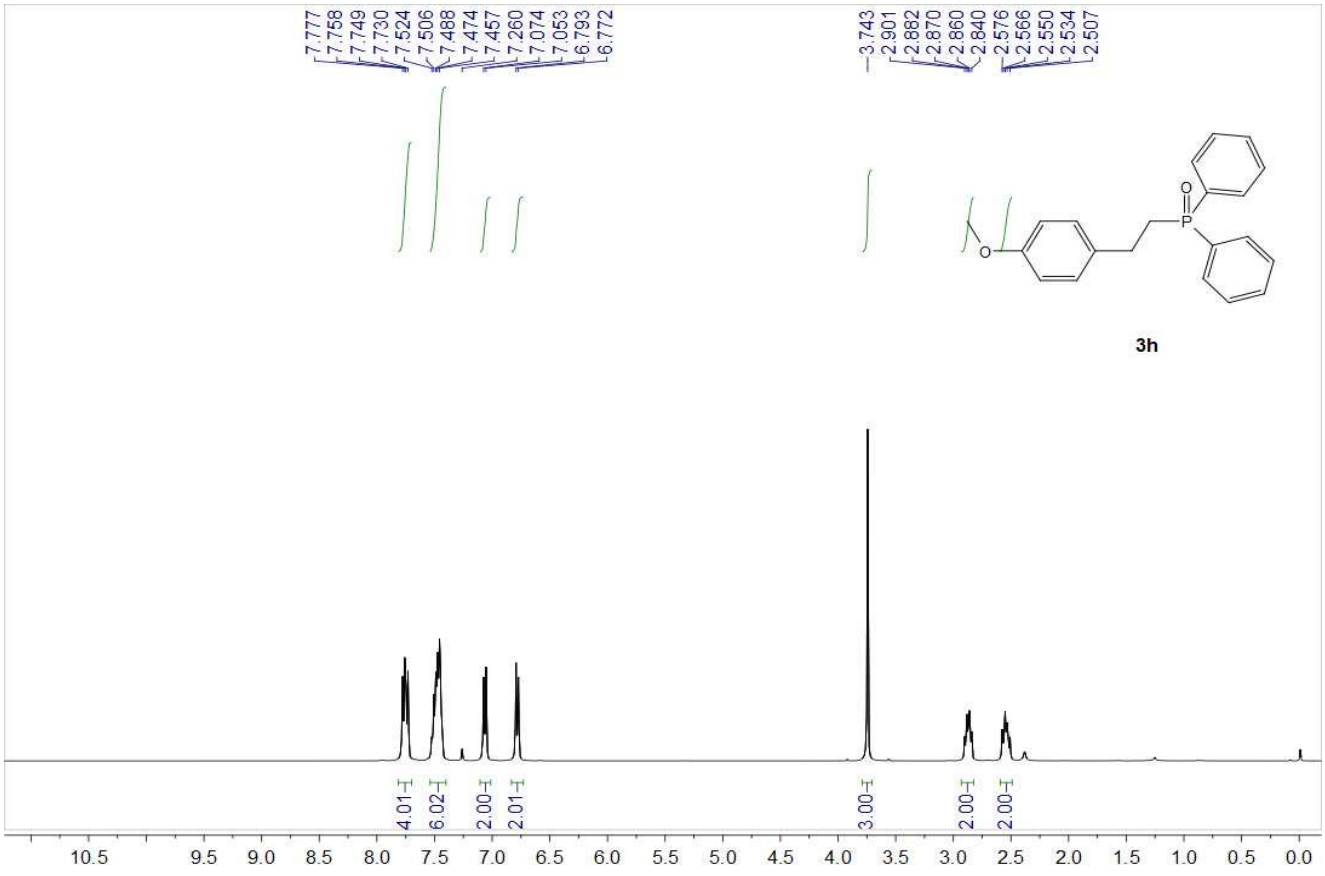
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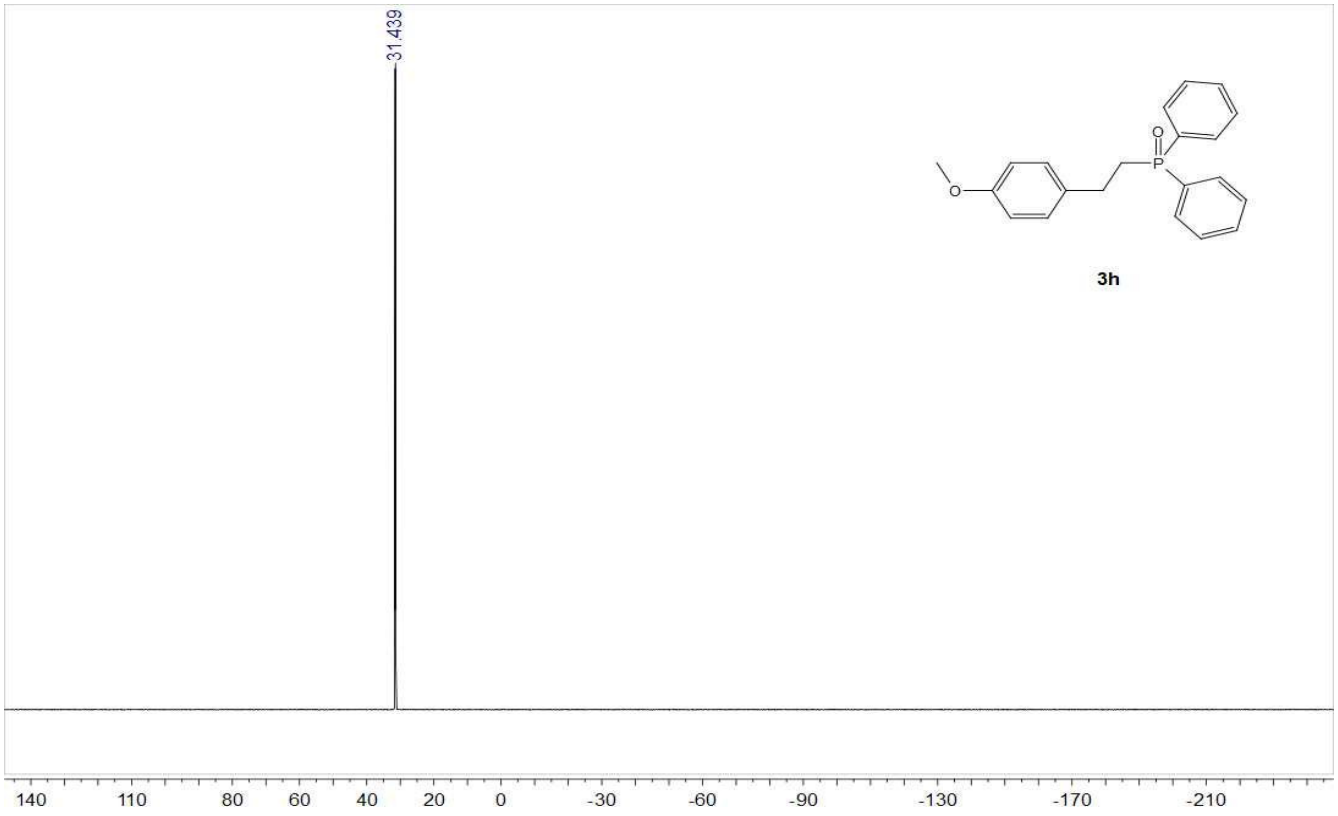
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P
O
3h
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80
60
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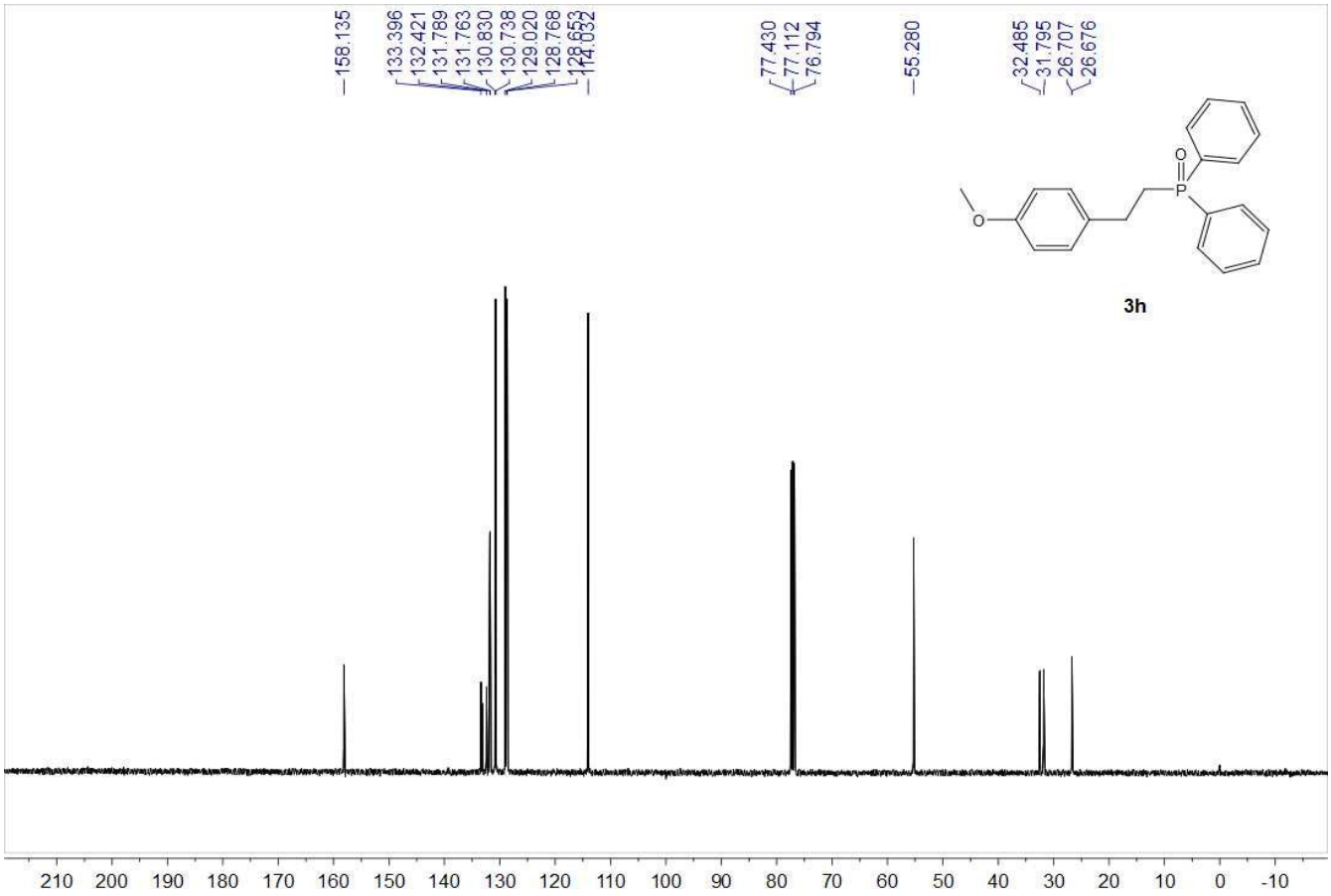
0
-30
-60
-90
-130
-170
-210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129.020
128.768
128.653
114.032
77.430
77.112
76.794
55.280
32.485
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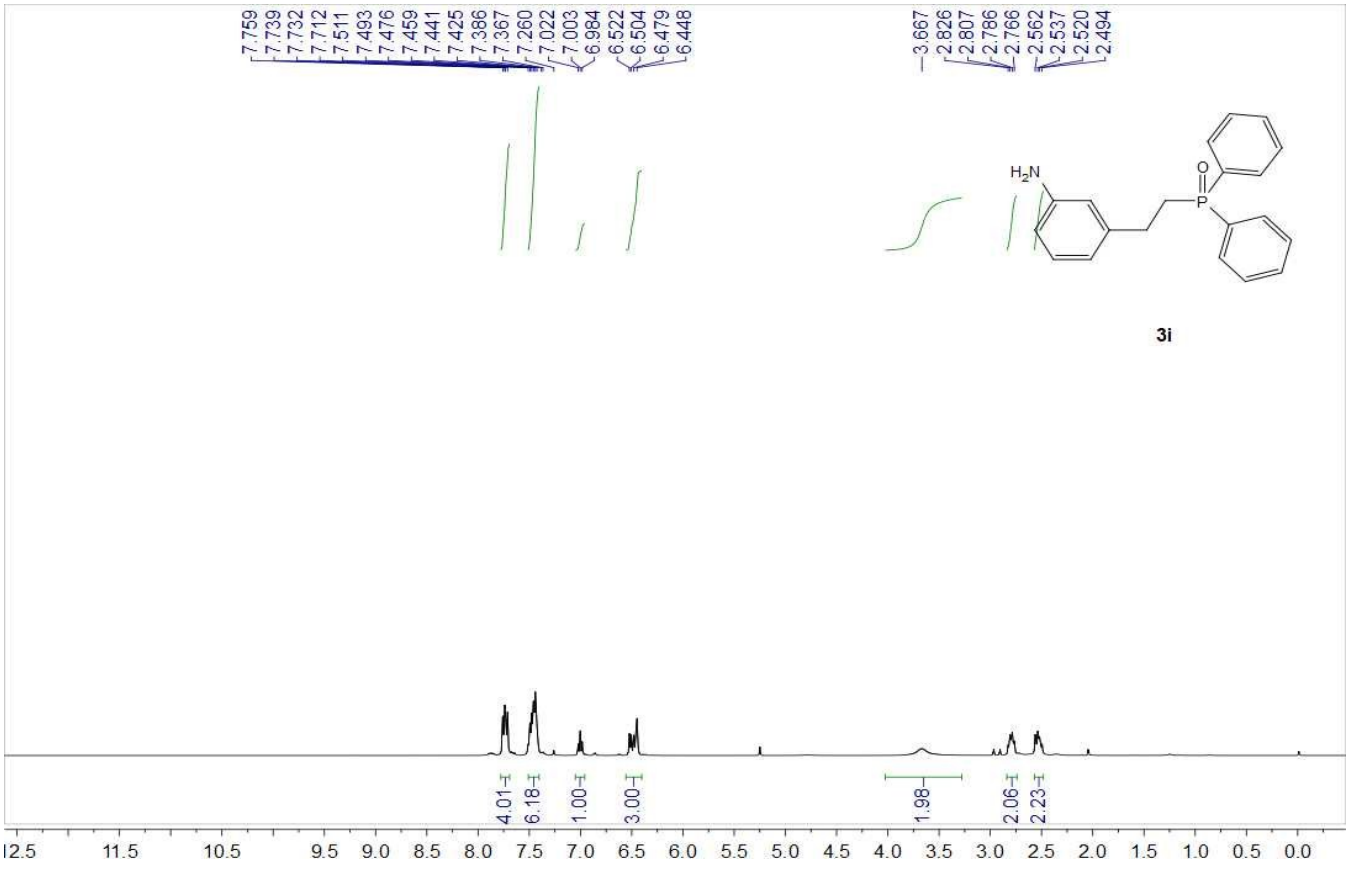
26.707
26.676
O
P
O
3h
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10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7.739
7.732
7.712
7.511
7.493
7.476
7.459
7.441
7.425
7.386
7.367
7.260
7.022
7.003
6.984
6.522
6.504
6.479
6.448
3.667
2.826
2.807
2.786
2.766
2.562
2.537
2.520
2.494
H2N
O
P
3i
4.01
6.18
1.00
3.00
1.98
2.06
2.23
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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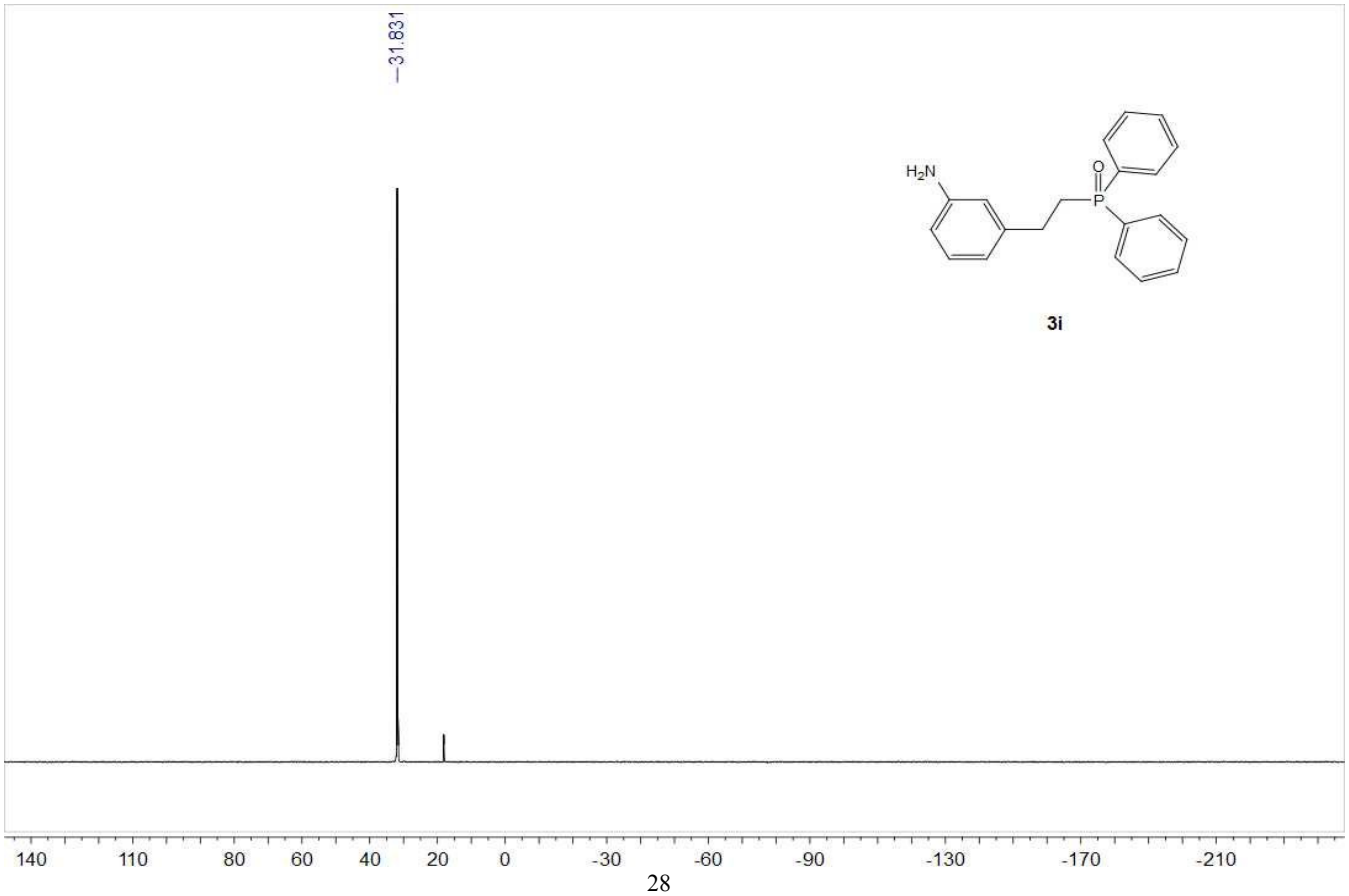
0.0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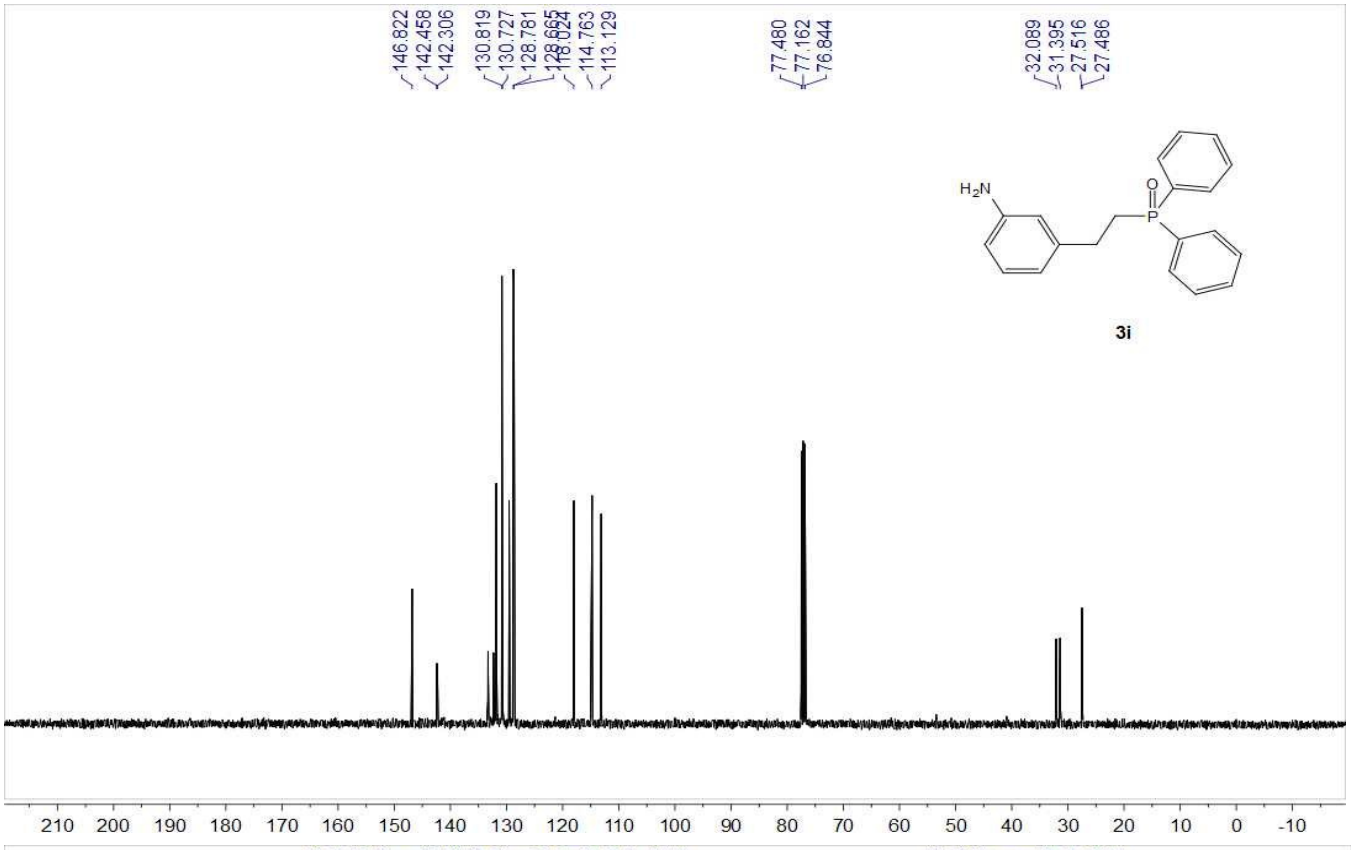
-170
-210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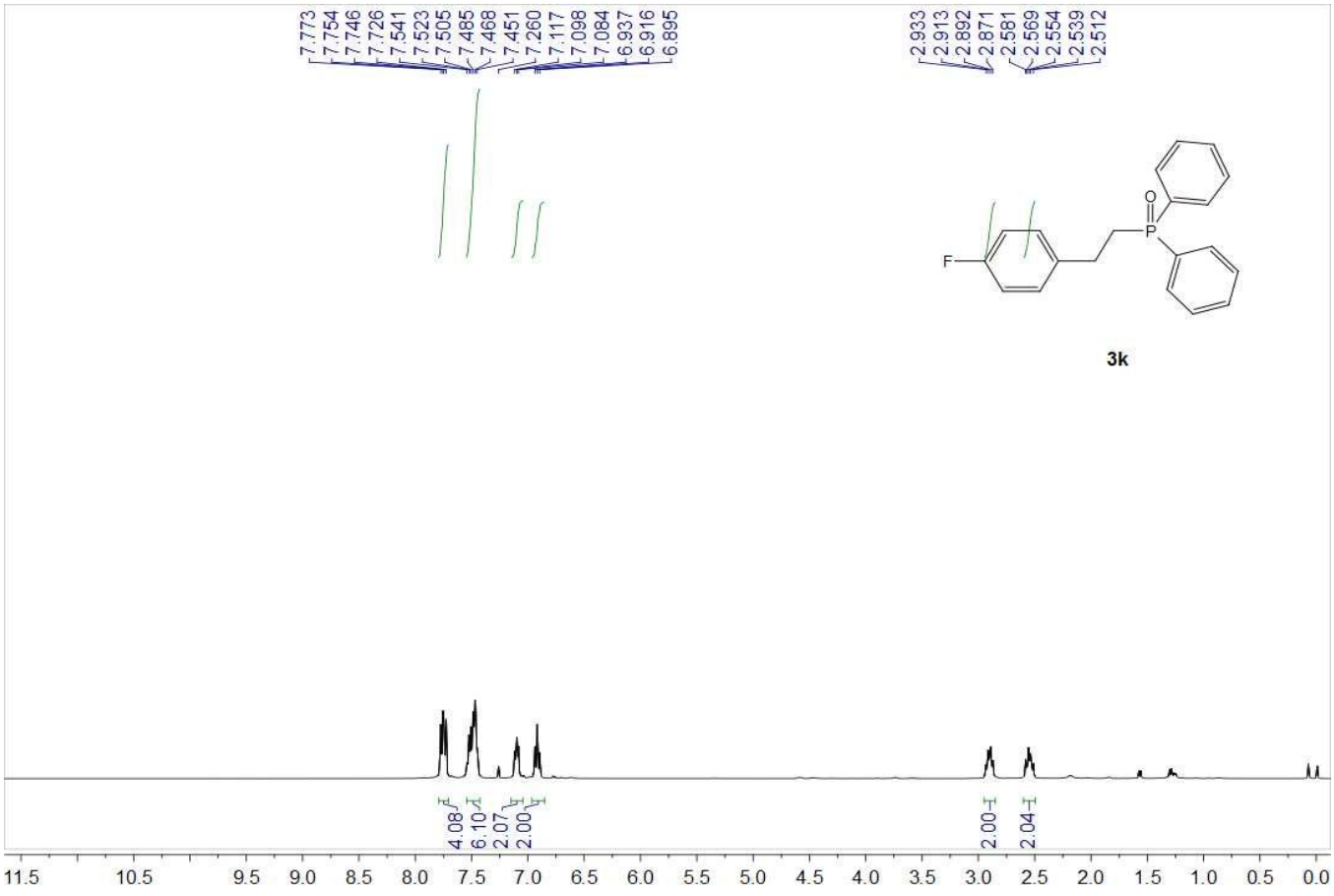
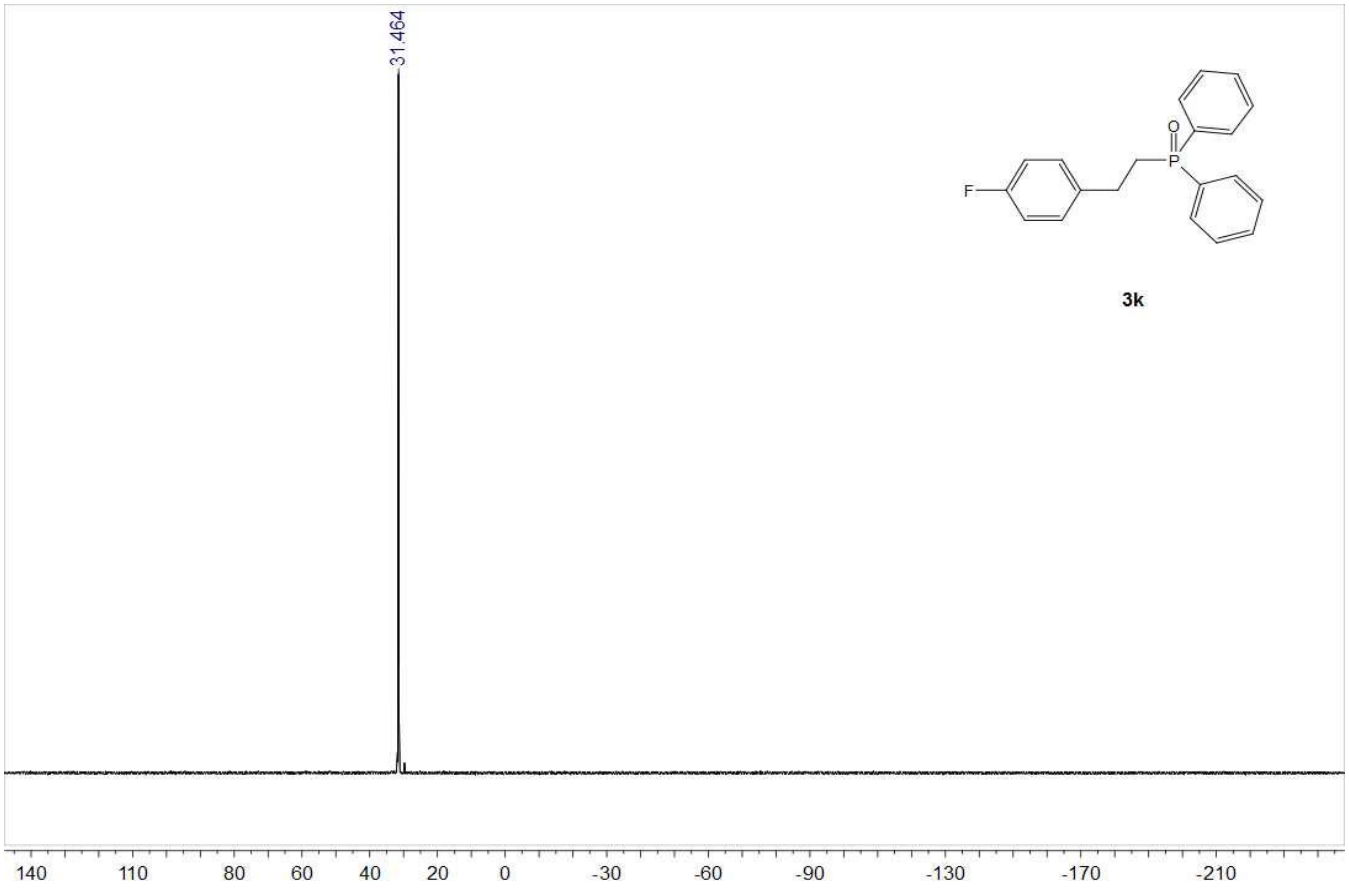
142.306
130.819
130.727
128.781
128.665
128.024
118.024
114.763
113.129
77.480
77.162
76.844
32.089
31.395
27.516
27.486
H2N
O
P
3i
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10


7.773
7.754
7.746
7.726
7.541
7.523
7.505
7.485
7.468
7.451
7.260
7.117
7.098
7.084
6.937
6.916
6.895
2.933
2.913
2.892
2.871
2.581
2.569
2.554
2.539
2.512
O
P
F
3k
4.08
6.10
2.07
2.00
2.00
2.04
11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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P
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3k
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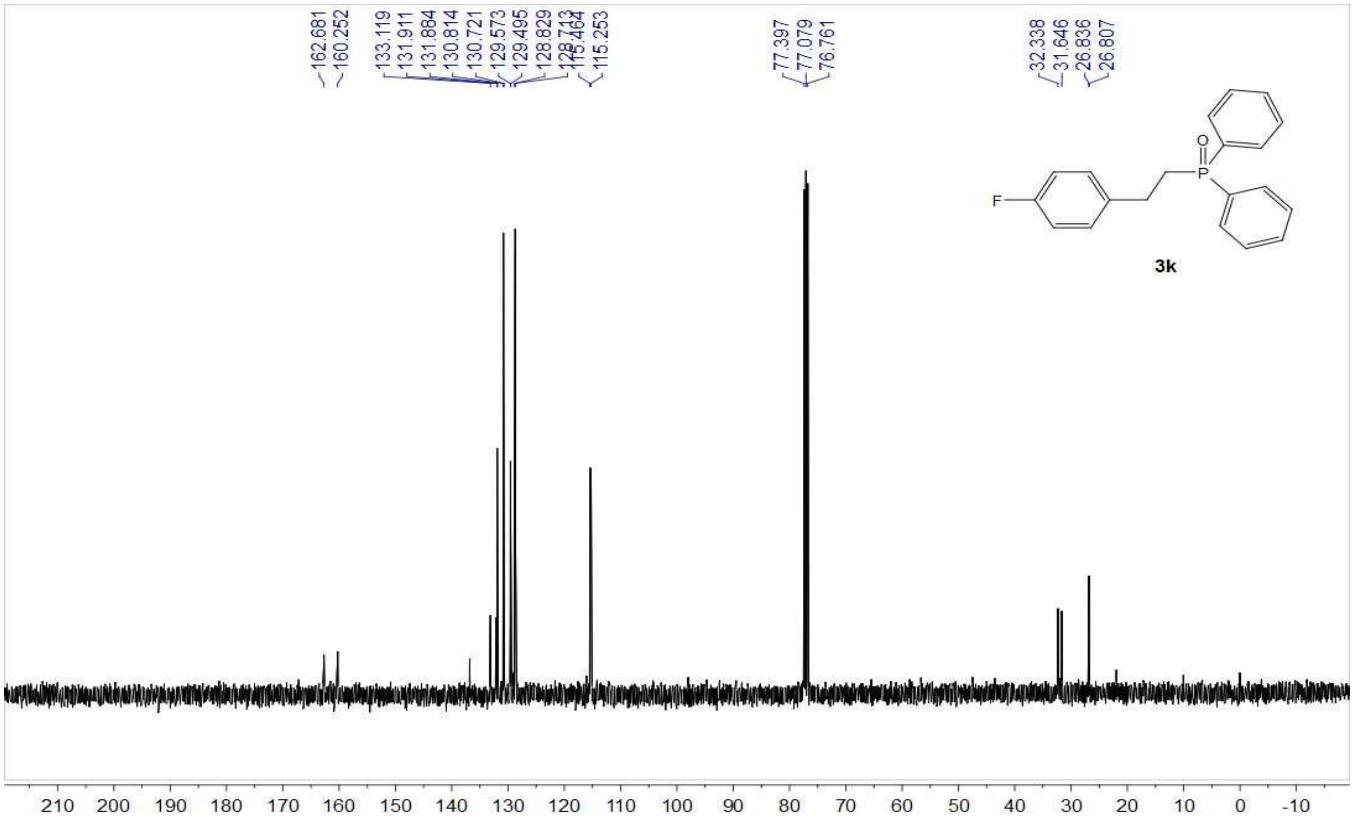
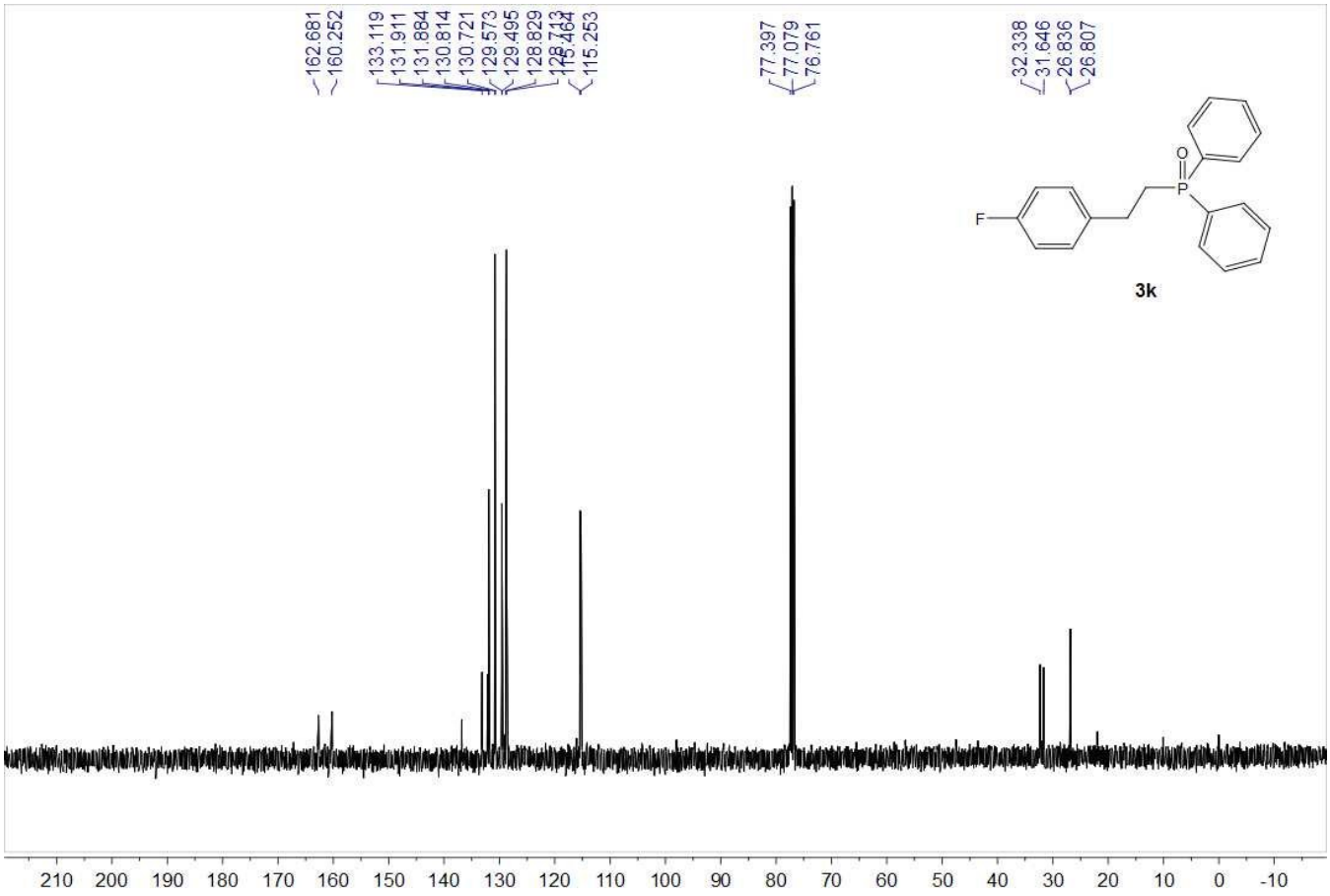
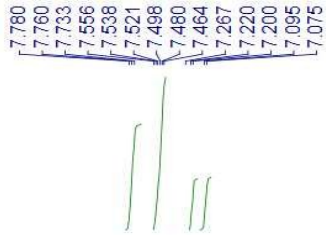
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210


162.681
160.252
133.119
131.911
131.884
130.814
130.721
129.573
129.495
128.829
115.253
77.397
77.079
76.761
32.338
31.646
26.836
26.807
O
P
F
3k
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10


162.681
160.252
133.119
131.911
131.884
130.814
130.721
129.573
129.495
128.829
115.253
77.397
77.079
76.761
32.338
31.646
26.836
26.807
O
P
F
3k
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10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7.760
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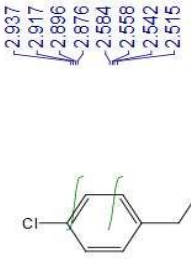
7.556
7.538
7.521
7.498
7.480
7.464
7.267
7.220
7.200
7.095
7.075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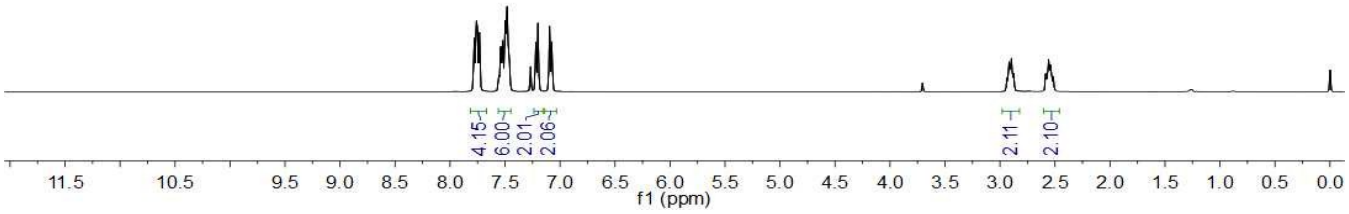
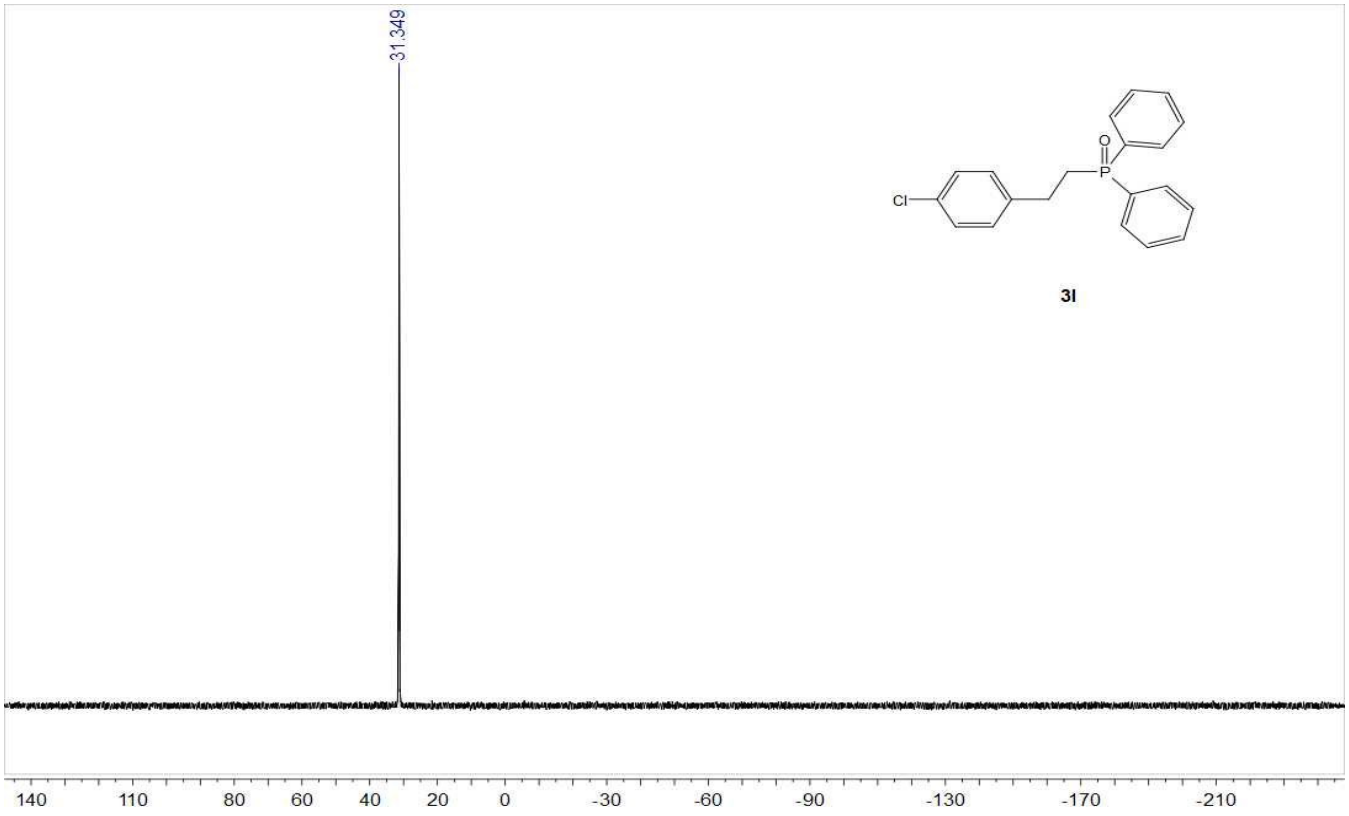
2.917
2.896
2.876
2.584
2.558
2.542
2.515

O
P
Cl

3l


4.15
6.00
2.01
2.06
2.11
2.10
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


31.349
O
P
Cl
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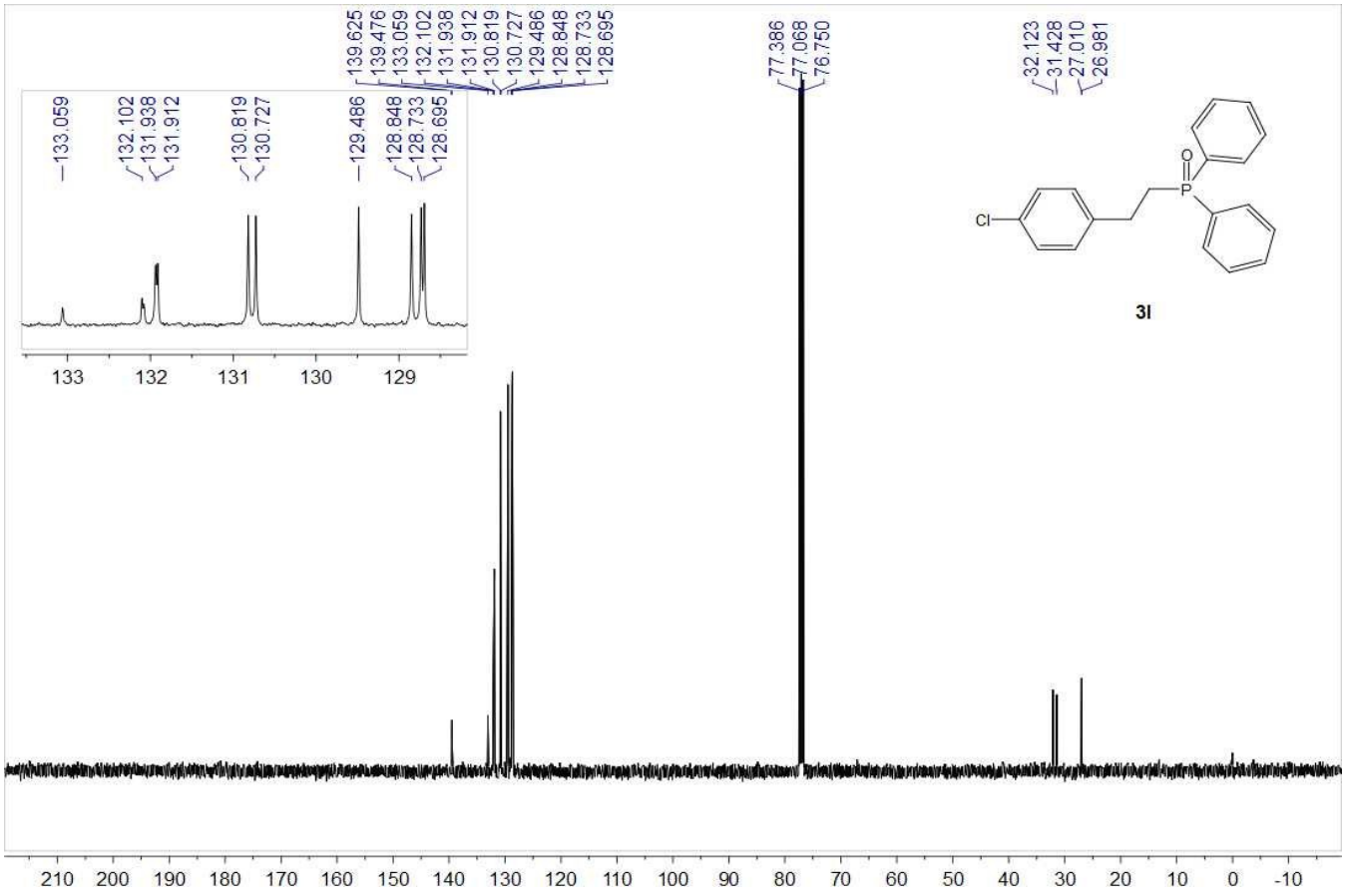
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210

3l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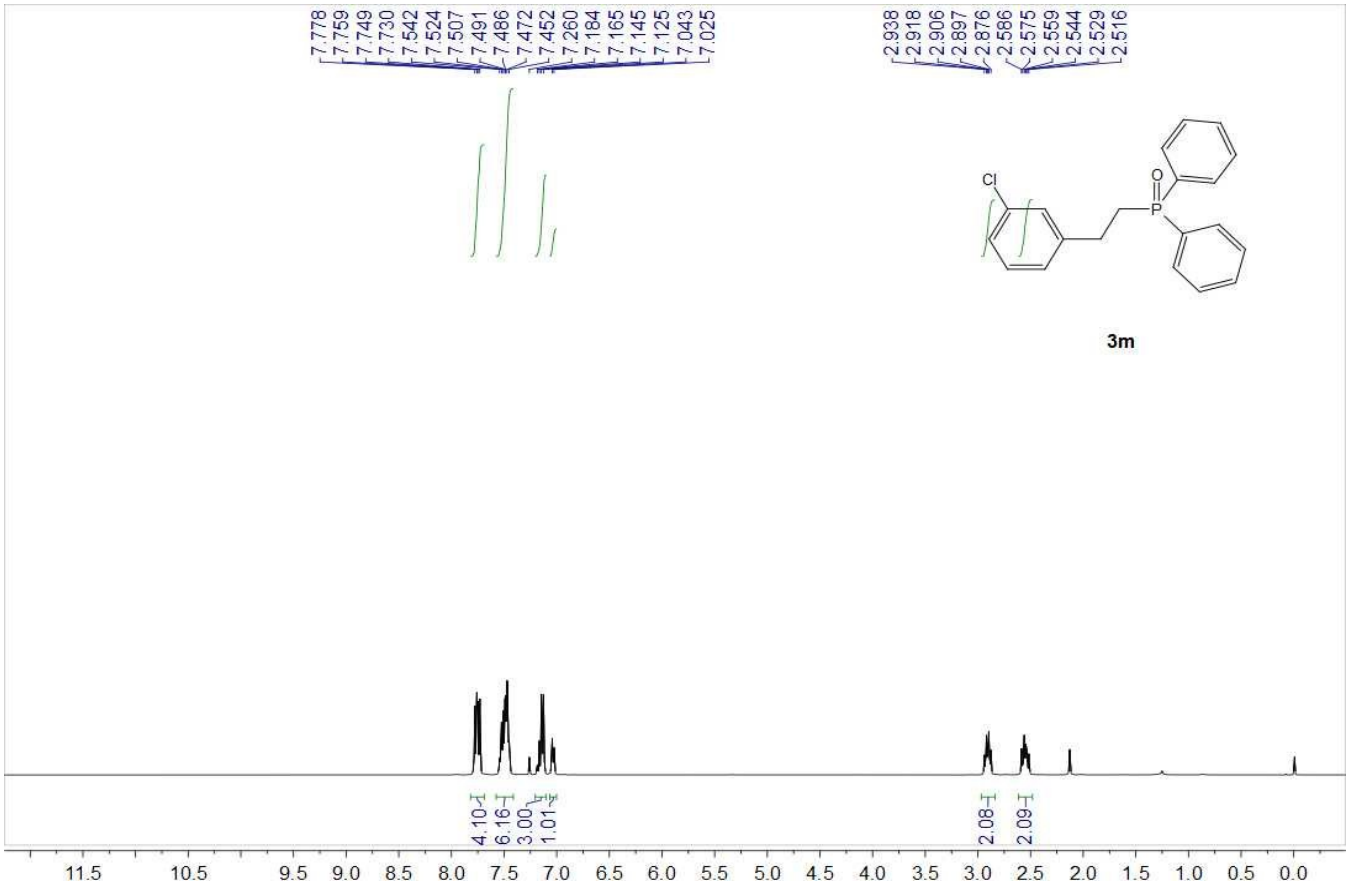
139.476
133.059
132.102
131.938
131.912
130.819
130.727
129.486
128.848
128.733
128.695
77.386
77.068
76.750
32.123
31.428
27.010
26.981
133.059
132.102
131.938
131.912
130.819
130.727
129.486
128.848
128.733
128.695
133
132
131
130
129
Cl
O
P
3l
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10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7.759
7.749
7.730
7.542
7.524
7.507
7.491
7.486
7.472
7.452
7.260
7.184
7.165
7.145
7.125
7.043
7.025
2.938
2.918
2.906
2.897
2.876
2.586
2.575
2.559
2.544
2.529
2.516
Cl
O
P
3m
4.10
6.16
3.00
1.01
2.08
2.09
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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5.0
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4.0
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3.0
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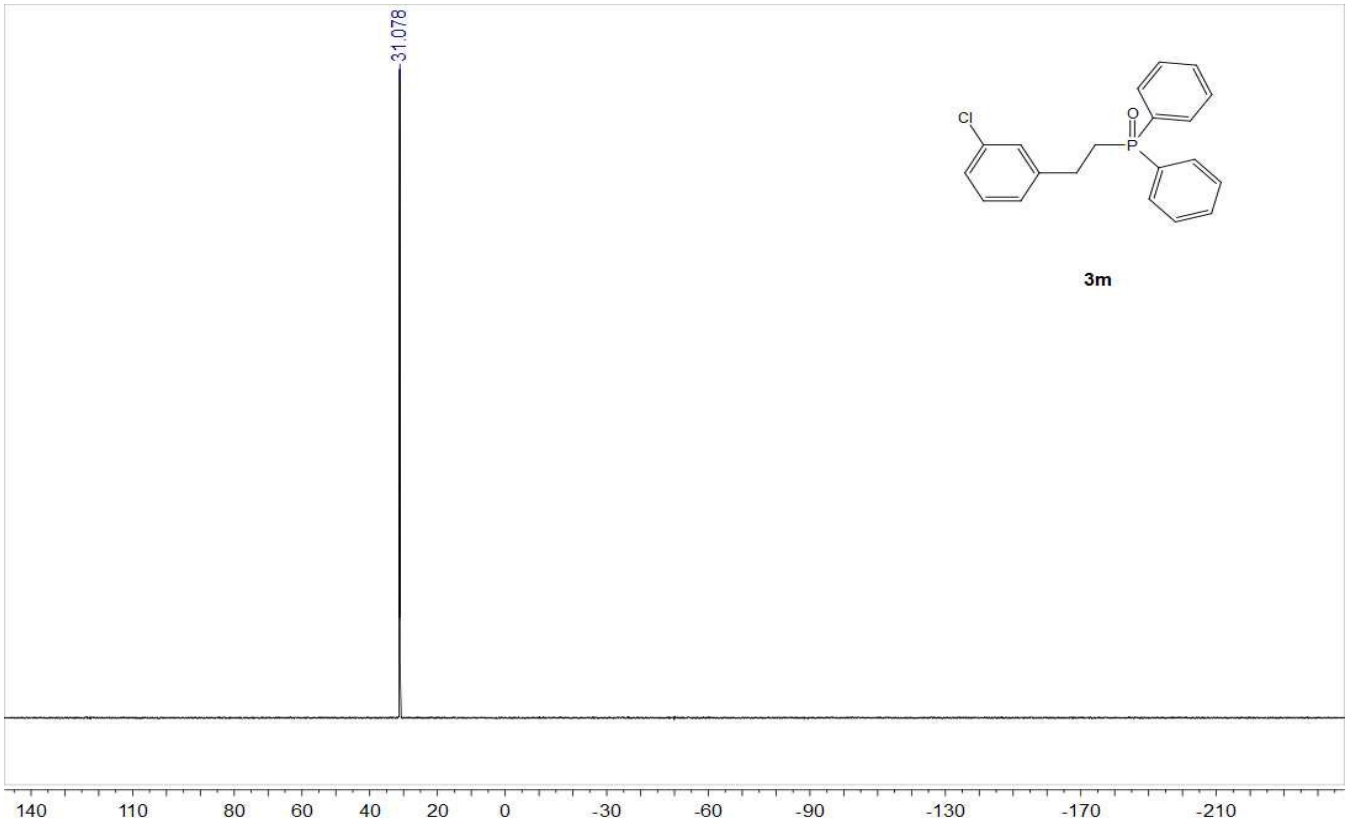
2.0
1.5
1.0
0.5
0.0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Cl
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3m
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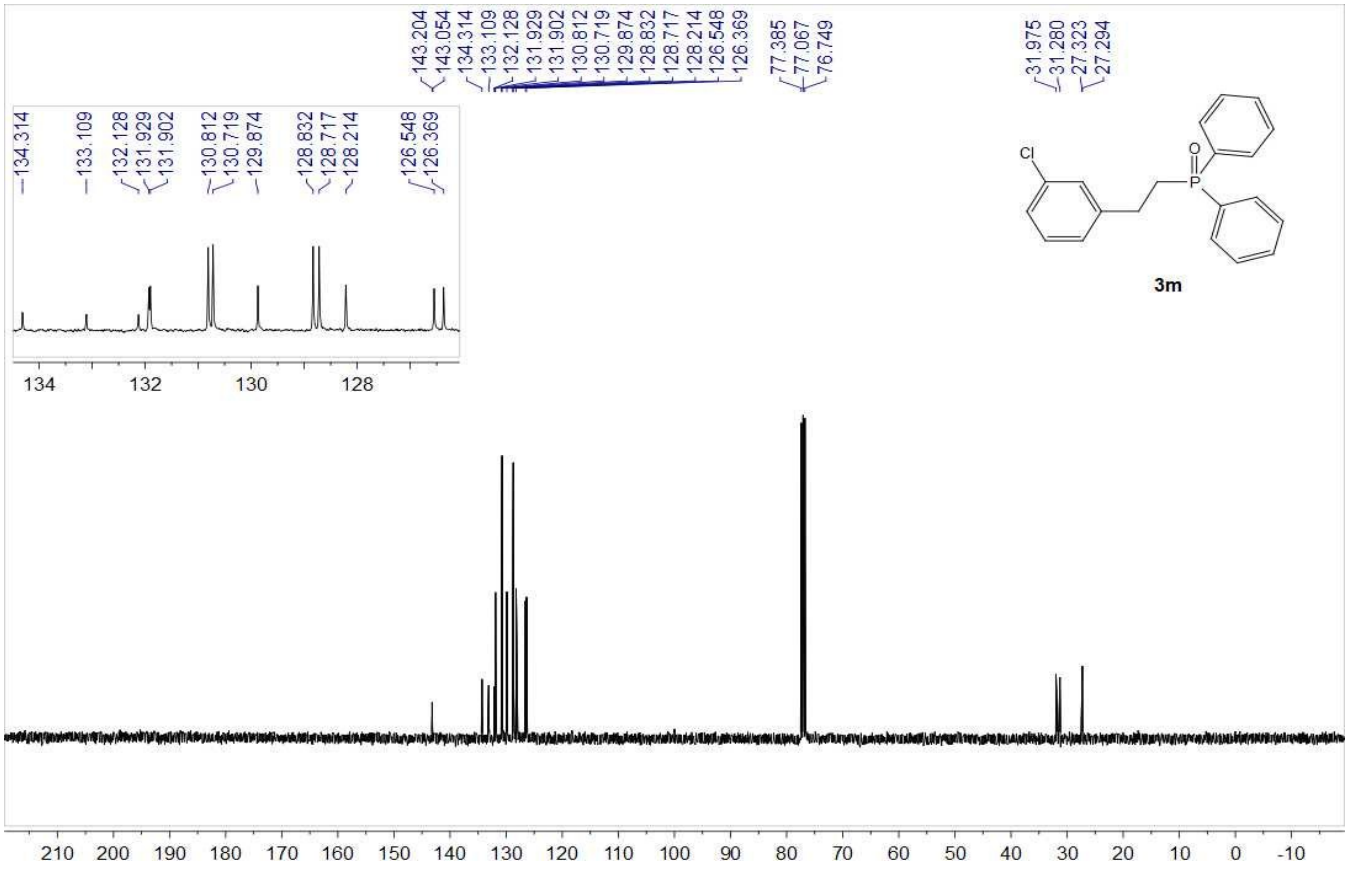
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210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131.929
131.902
130.812
130.719
129.874
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128.717
128.214
126.548
126.369
77.385
77.067
76.749
31.975
31.280
27.323
27.294
Cl
O
P
3m
134
132
130
128
210
200
190
180
170
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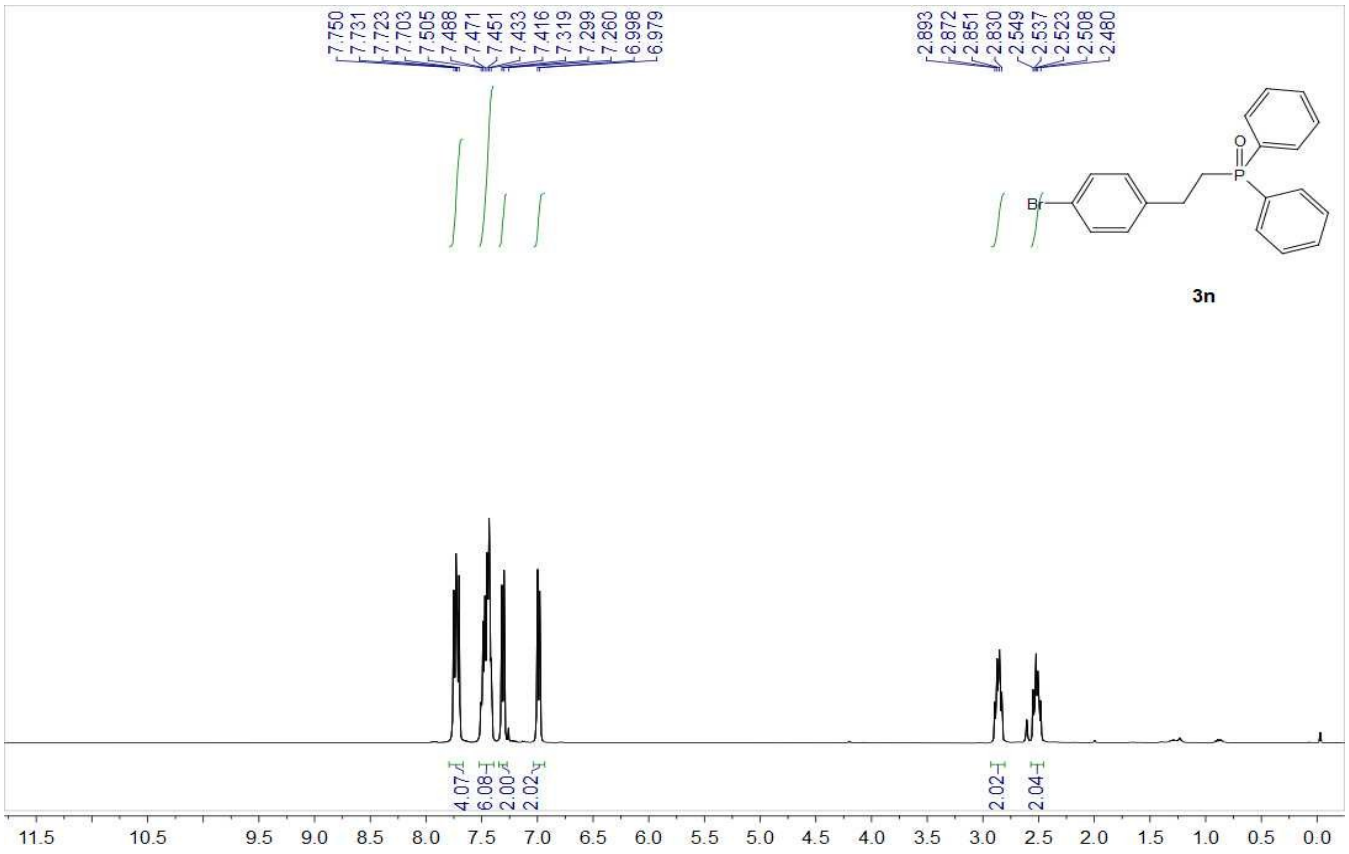
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10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7.723
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7.505
7.488
7.471
7.451
7.433
7.416
7.319
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7.260
6.998
6.979
2.893
2.872
2.851
2.830
2.549
2.537
2.523
2.508
2.480
Br
O
P
3n
4.07
6.08
2.00
2.02
2.02
2.04
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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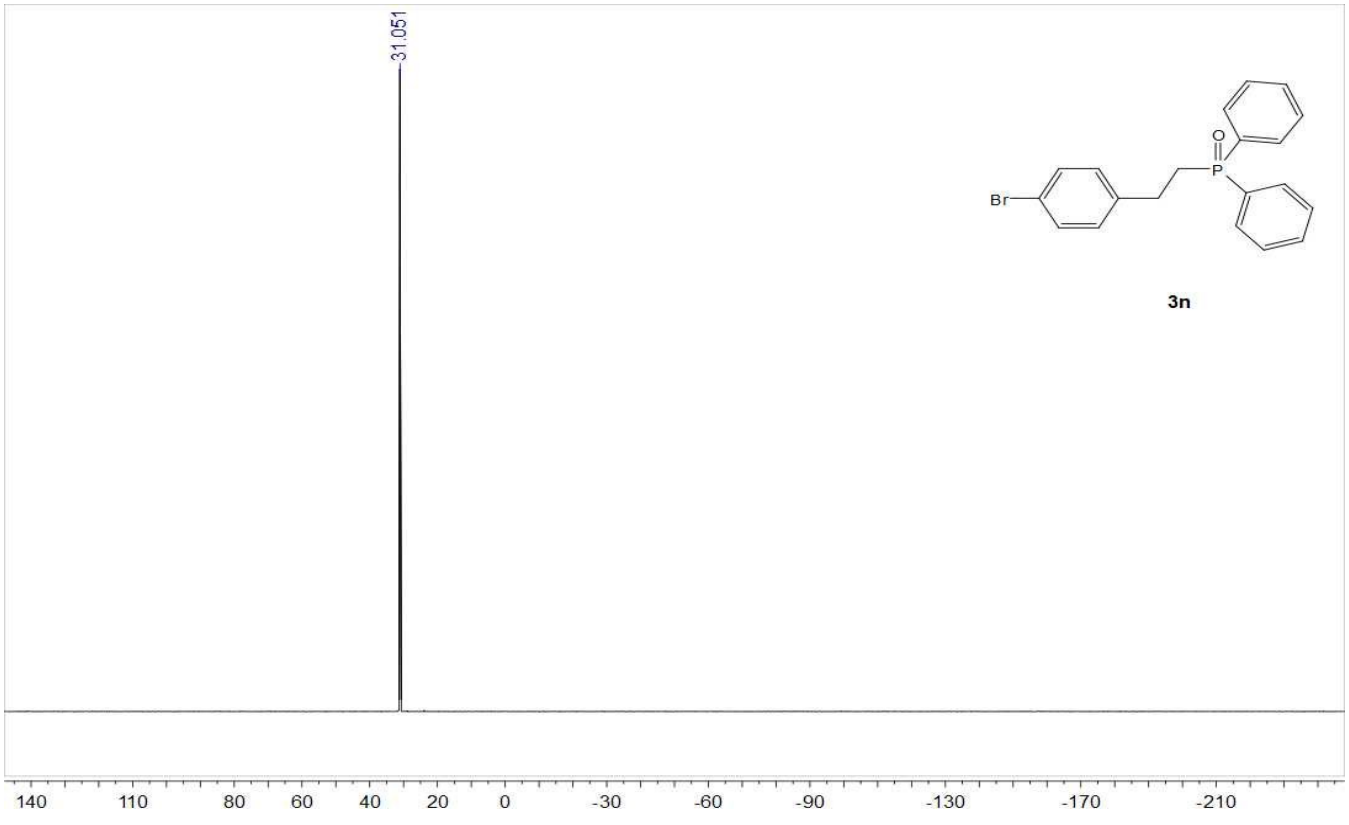
2.0
1.5
1.0
0.5
0.0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Br
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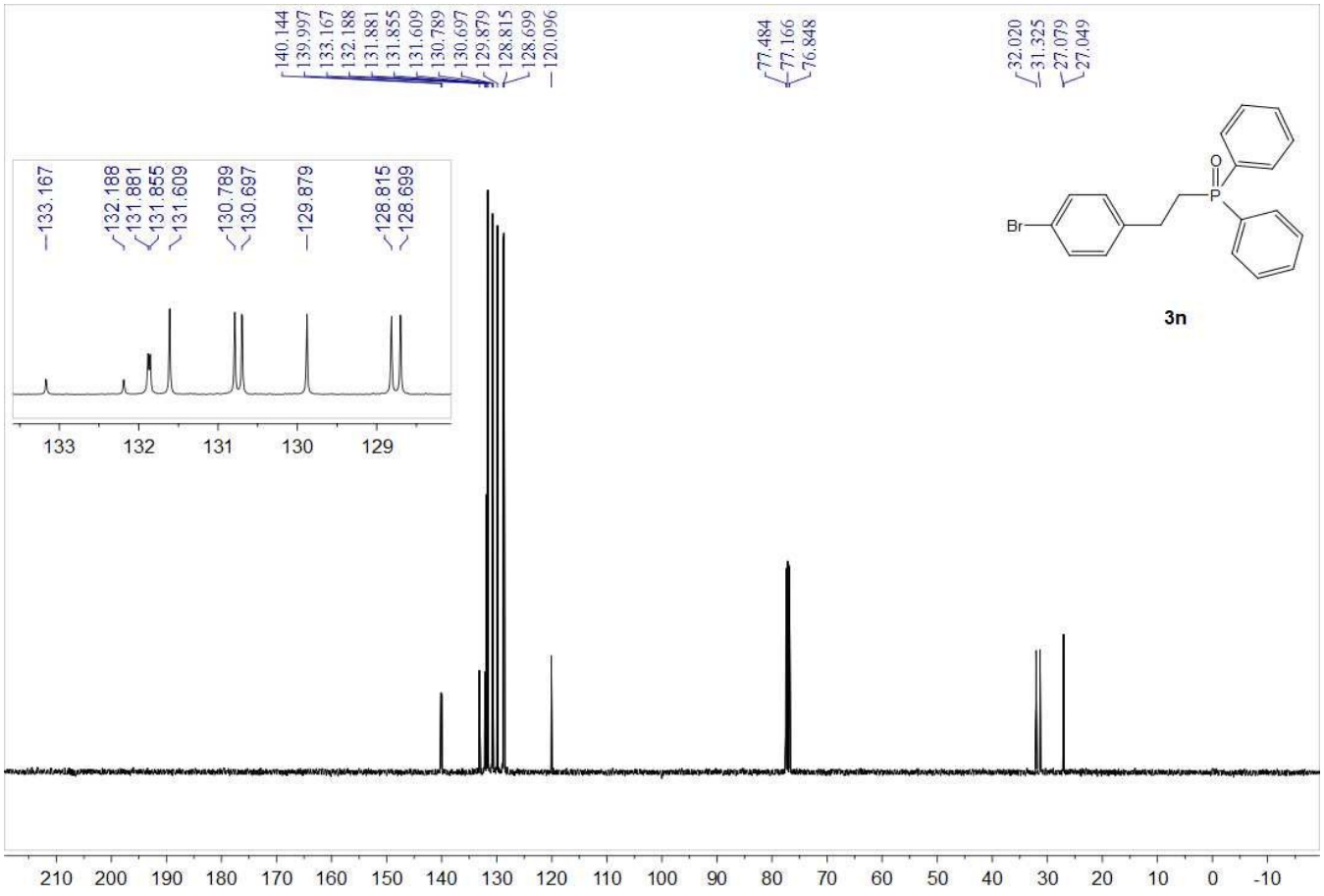
80
60
40
20
0
-30
-60
-90
-130
-170
-210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128.699
120.096
77.484
77.166
76.848
32.020
31.325
27.079
27.049
133.167
132.188
131.881
131.855
131.609
130.789
130.697
129.879
128.815
128.699
133
132
131
130
129
Br
O
P
3n
210
200
190
180
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140
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120
110
100
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80
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60
50
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30
20
10
0
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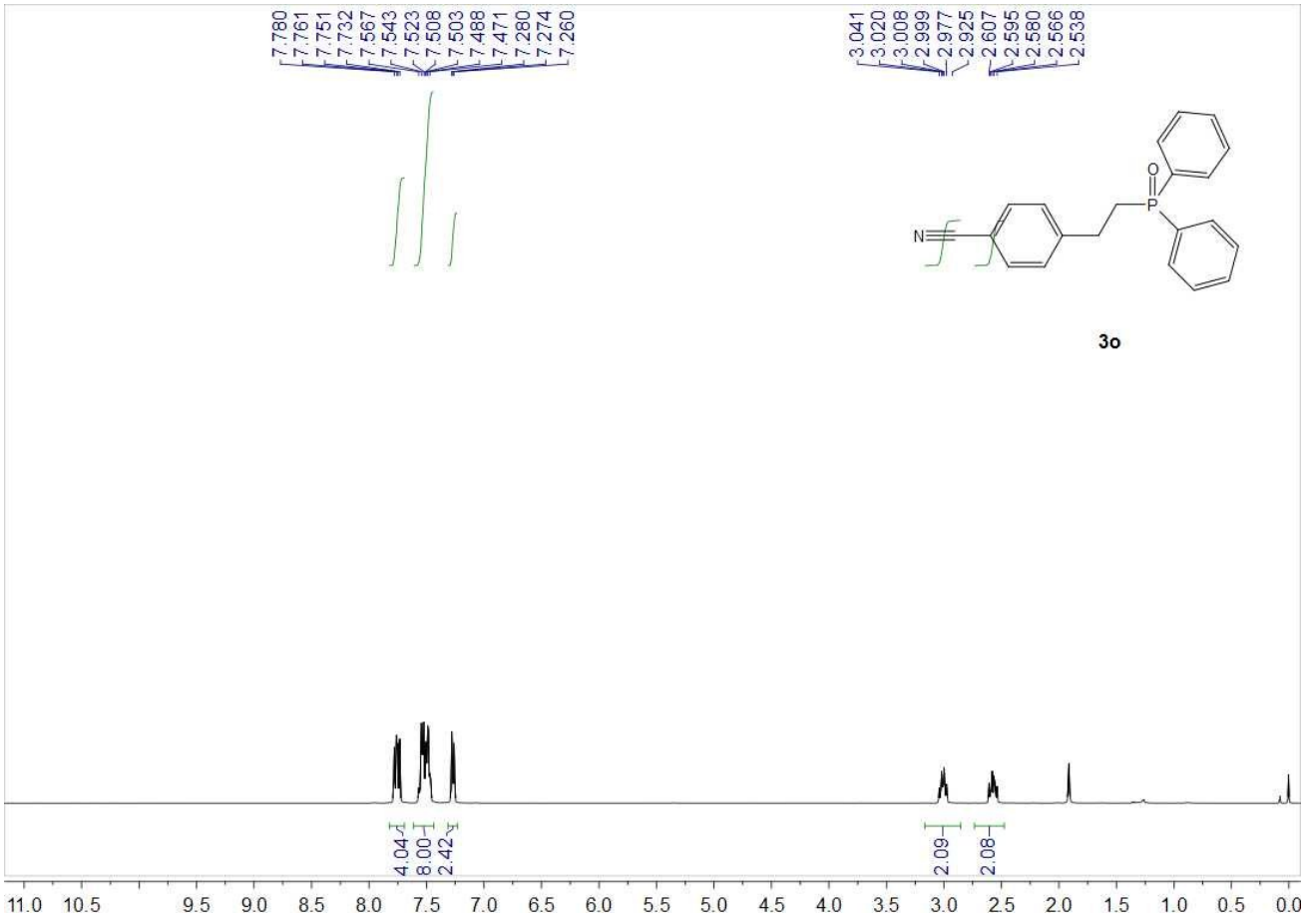
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7.761
7.751
7.732
7.567
7.543
7.523
7.508
7.503
7.488
7.471
7.280
7.274
7.260
3.041
3.020
3.008
2.999
2.977
2.925
2.607
2.595
2.580
2.566
2.538
O
P
N
3o
4.04
8.00
2.42
2.09
2.08
11.0
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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4.0
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3.0
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1.0
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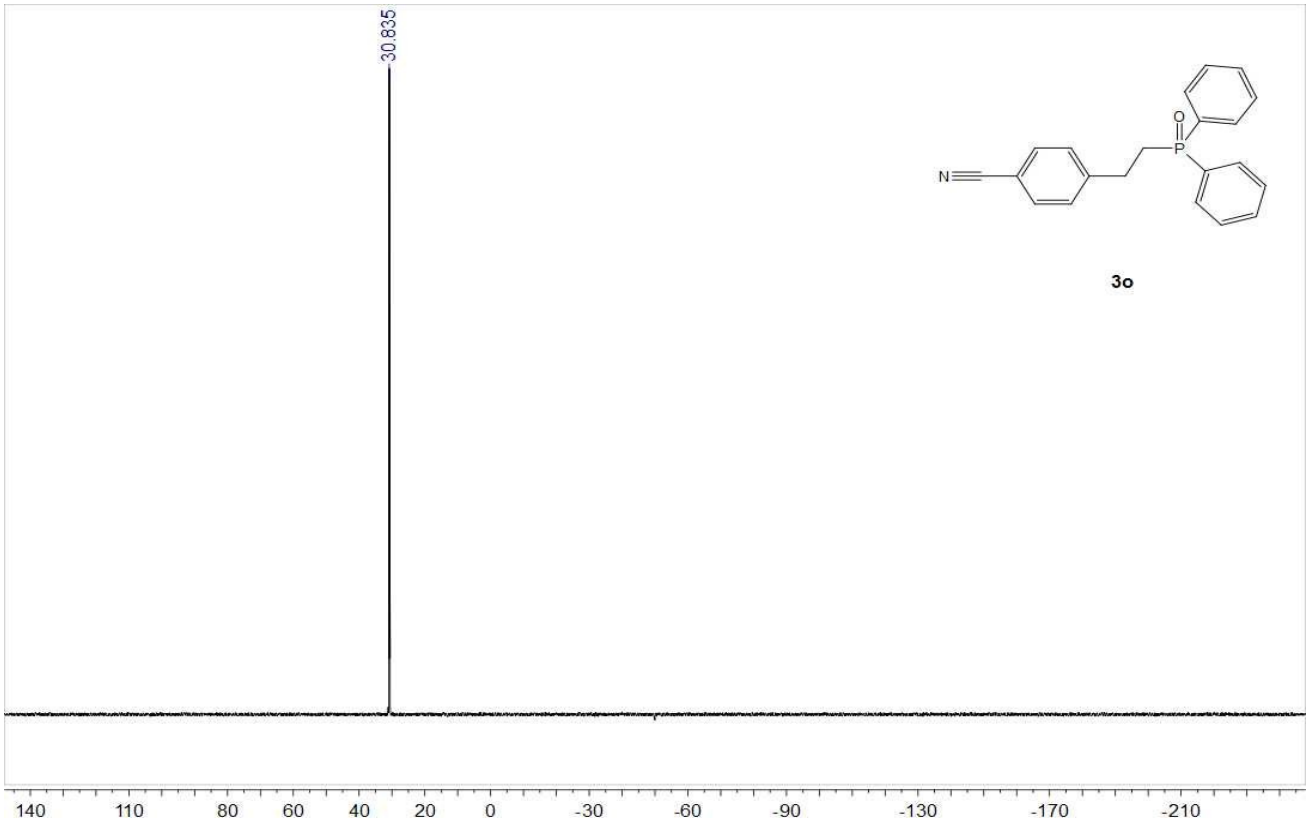
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N
O
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3o
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80
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0
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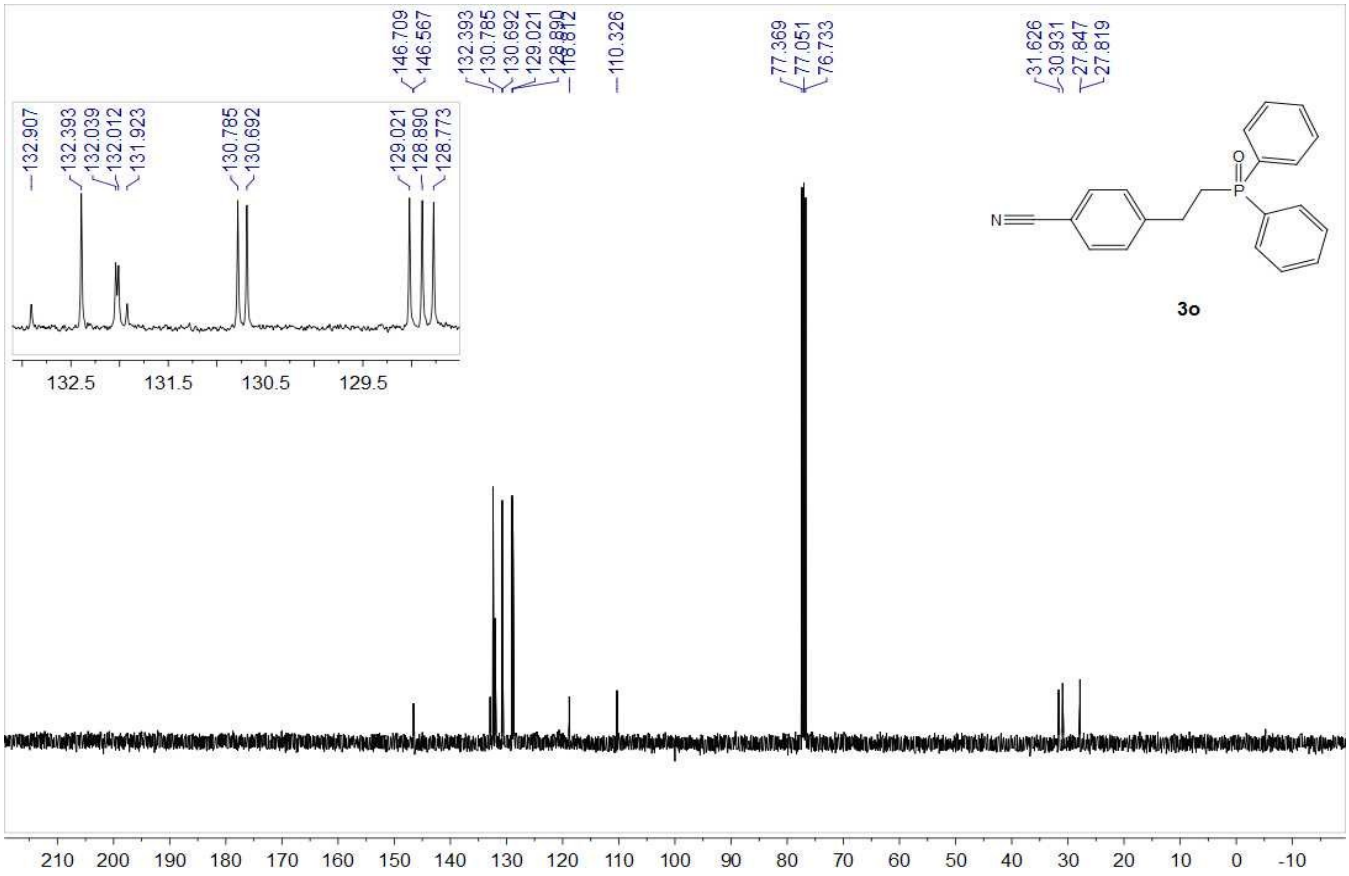
-60
-90
-130
-170
-210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146.567
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77.369
77.051
76.733
31.626
30.931
27.847
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131.923
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130.692
129.021
128.890
128.773
132.5
131.5
130.5
129.5
N
O
P
3o
210
200
190
180
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100
90
80
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60
50
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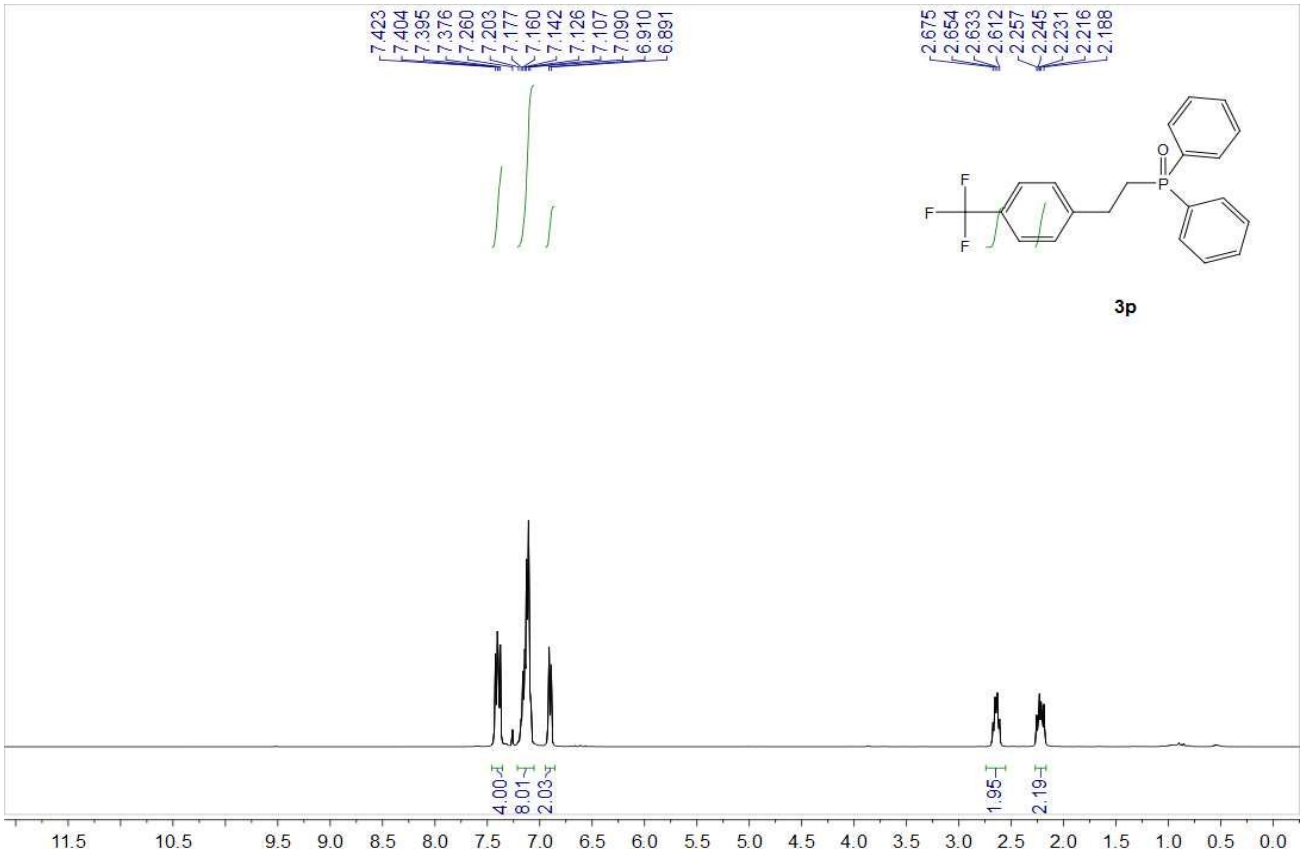
20
10
0
-10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7.090
6.910
6.891
2.675
2.654
2.633
2.612
2.257
2.245
2.231
2.216
2.188
F
F
F
O
P
3p
4.00
8.01
2.03
1.95
2.19
11.5
10.5
9.5
9.0
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8.0
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7.0
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6.0
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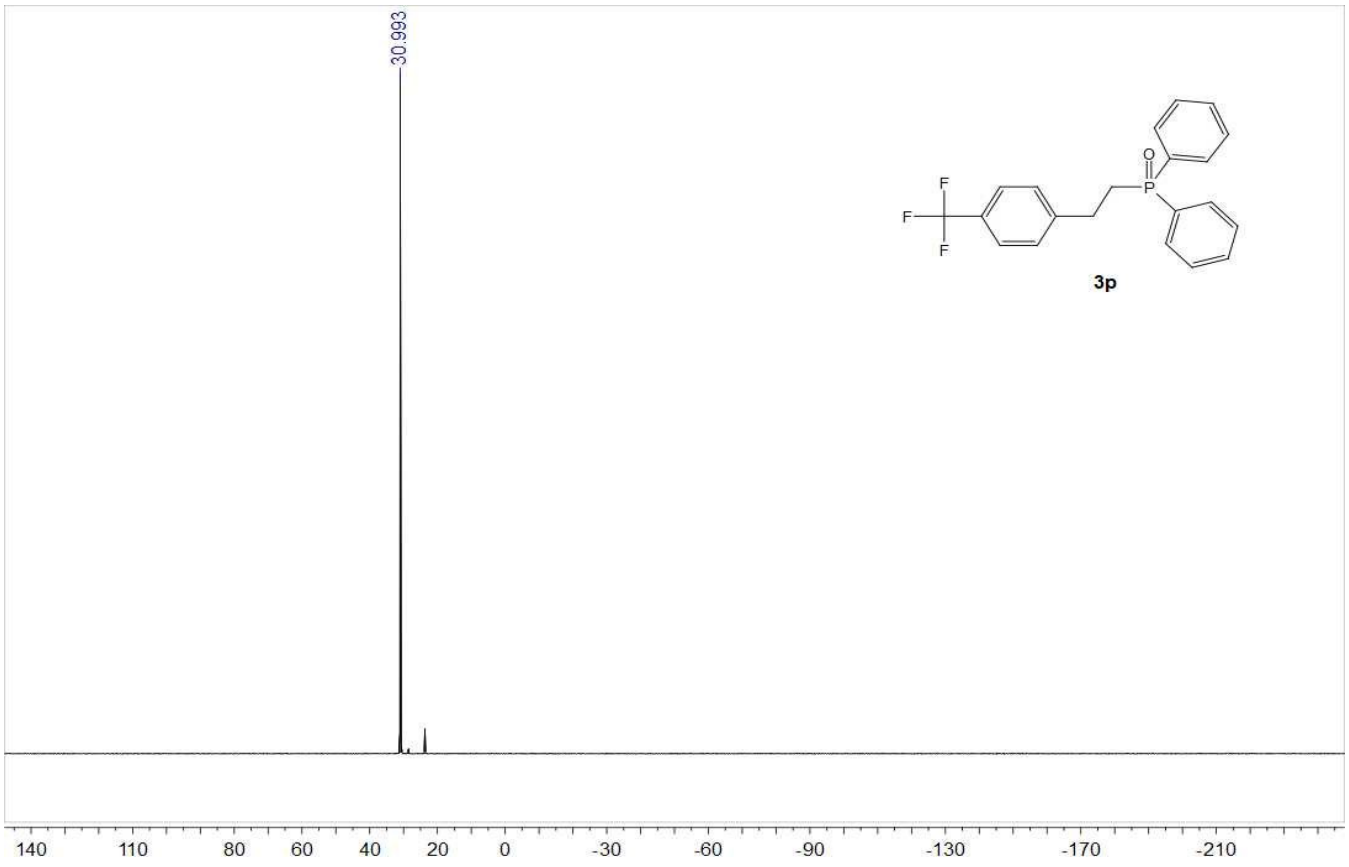
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F
F
F
O
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3p
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60
40
20
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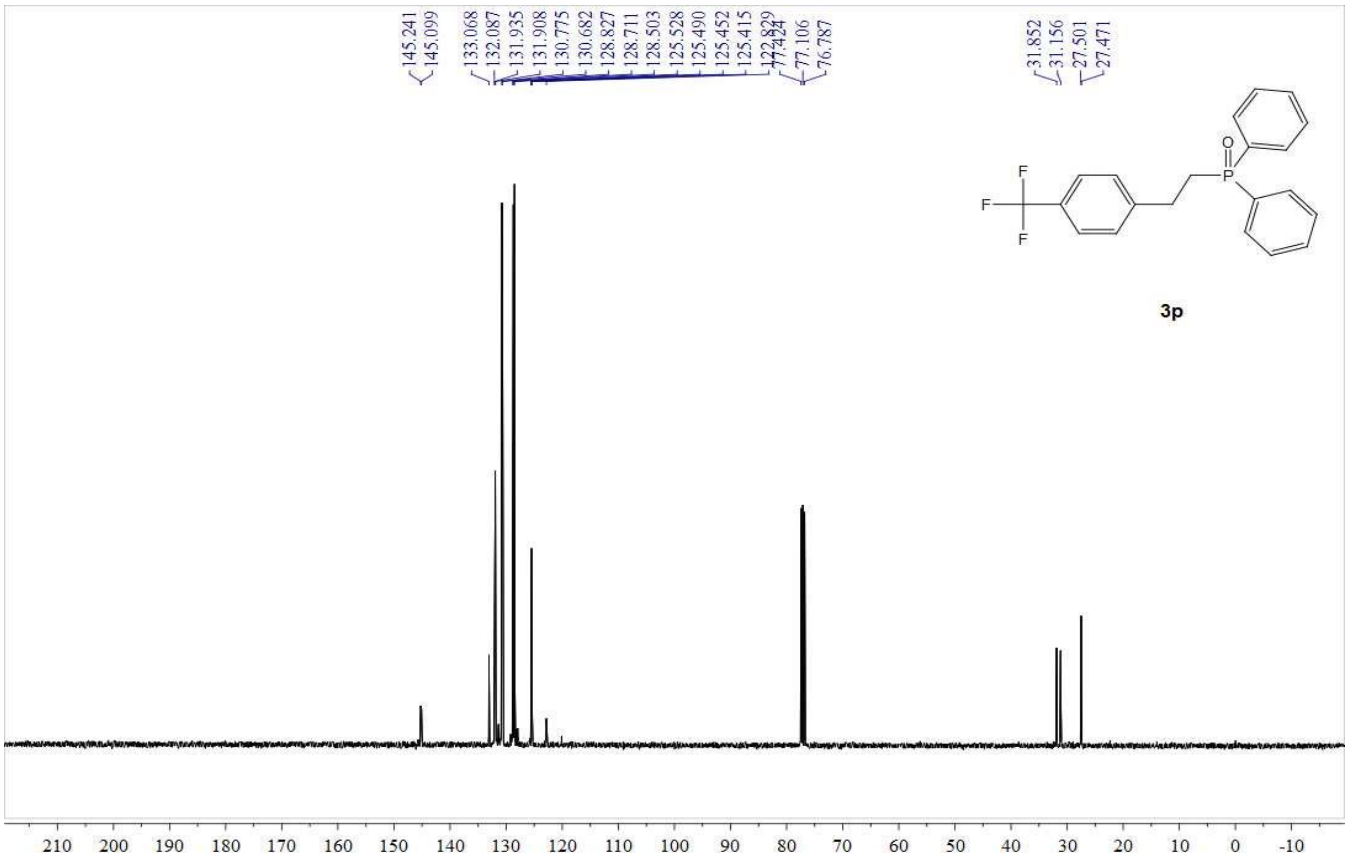
-30
-60
-90
-130
-170
-210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128.711
128.503
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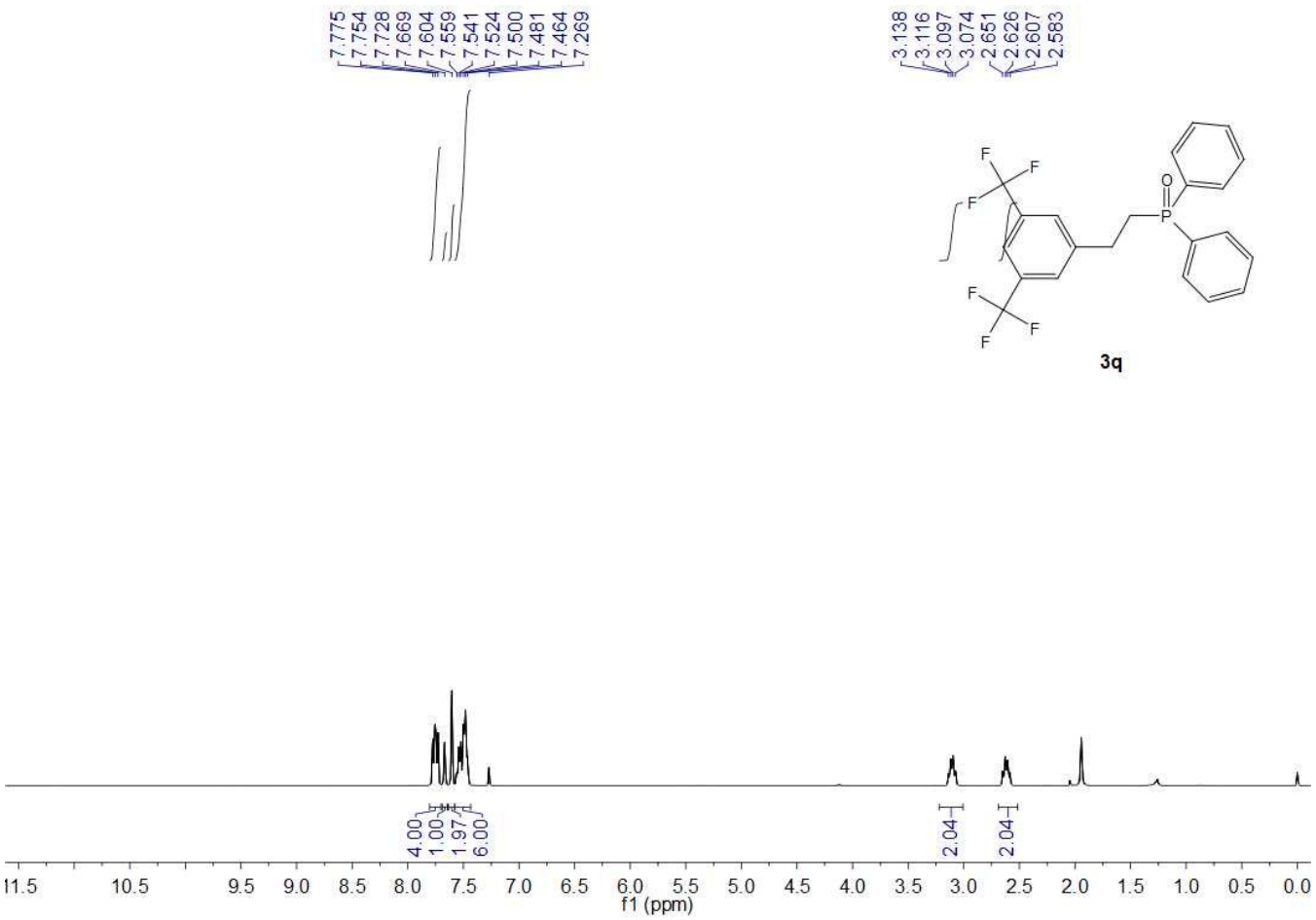
125.490
125.452
125.415
122.829
77.424
77.106
76.787
31.852
31.156
27.501
27.471
F
F
F
O
P
3p
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10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7.728
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7.604
7.559
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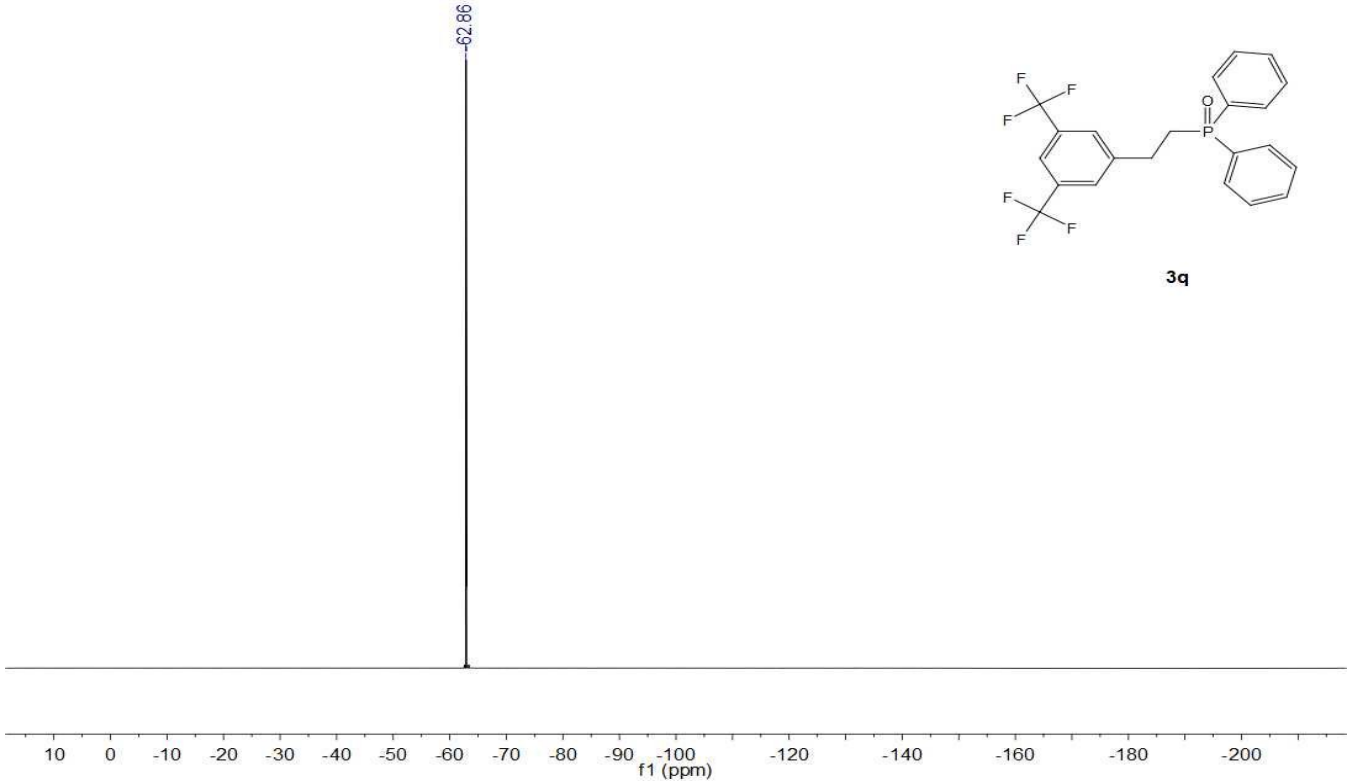
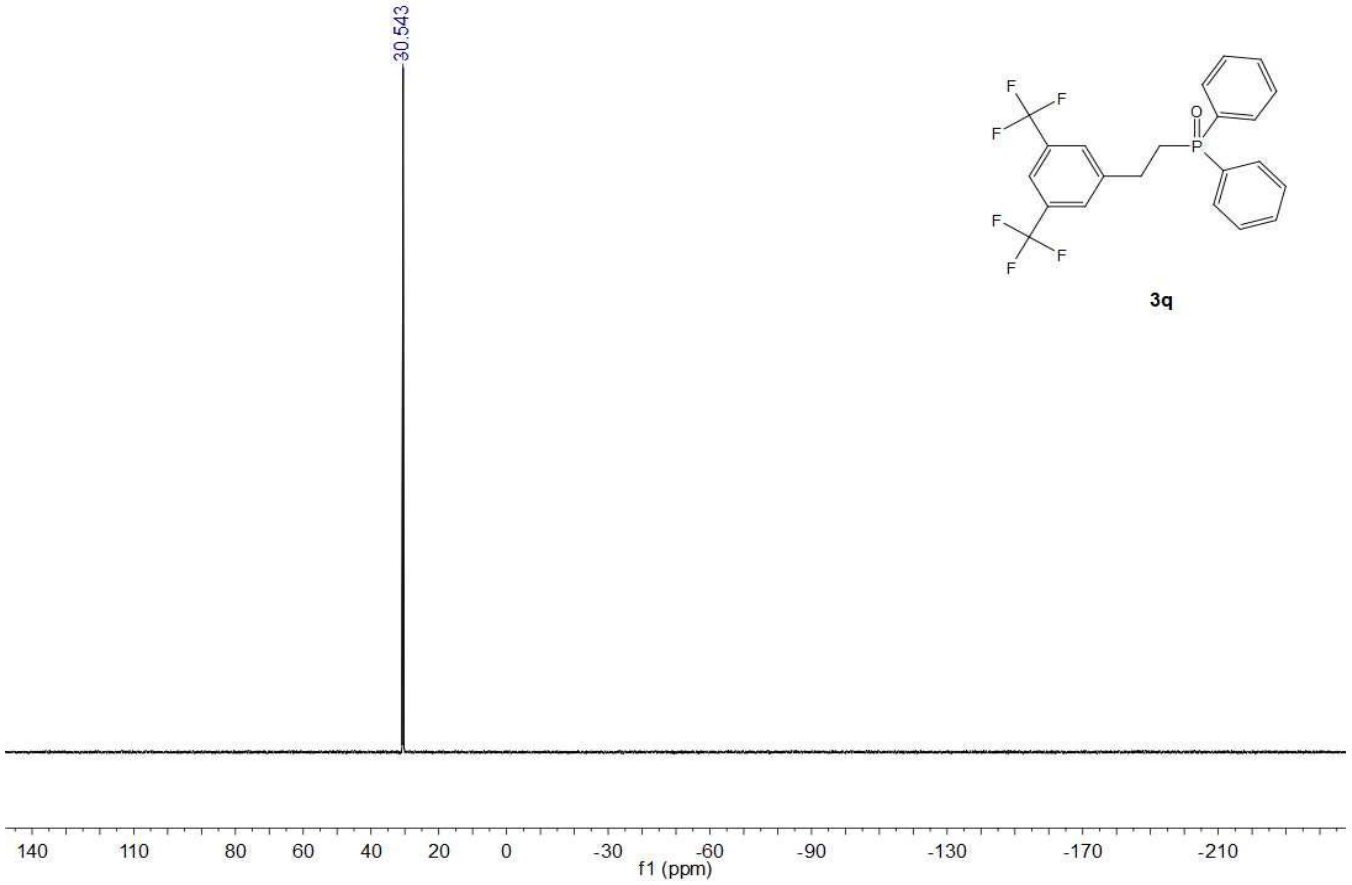
7.524
7.500
7.481
7.464
7.269
3.138
3.116
3.097
3.074
2.651
2.626
2.607
2.583
F
F
F
O
P
F
F
F
4.00
1.00
1.97
6.00
2.04
2.04
f1 (ppm)

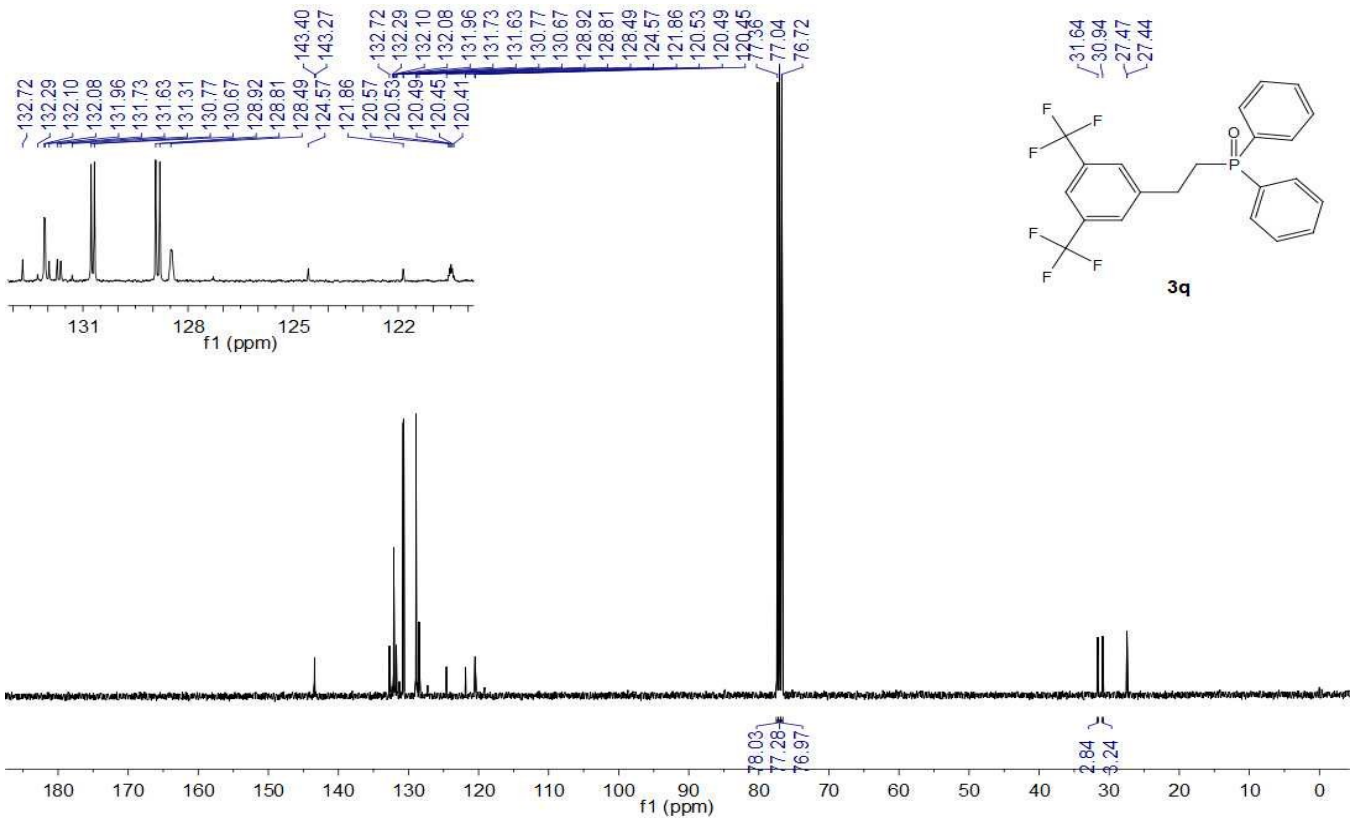
3q


-62.86
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -120 -140 -160 -180 -200
f1 (ppm)

3q


30.543
140 110 80 60 40 20 0 -30 -60 -90 -130 -170 -210
f1 (ppm)

3q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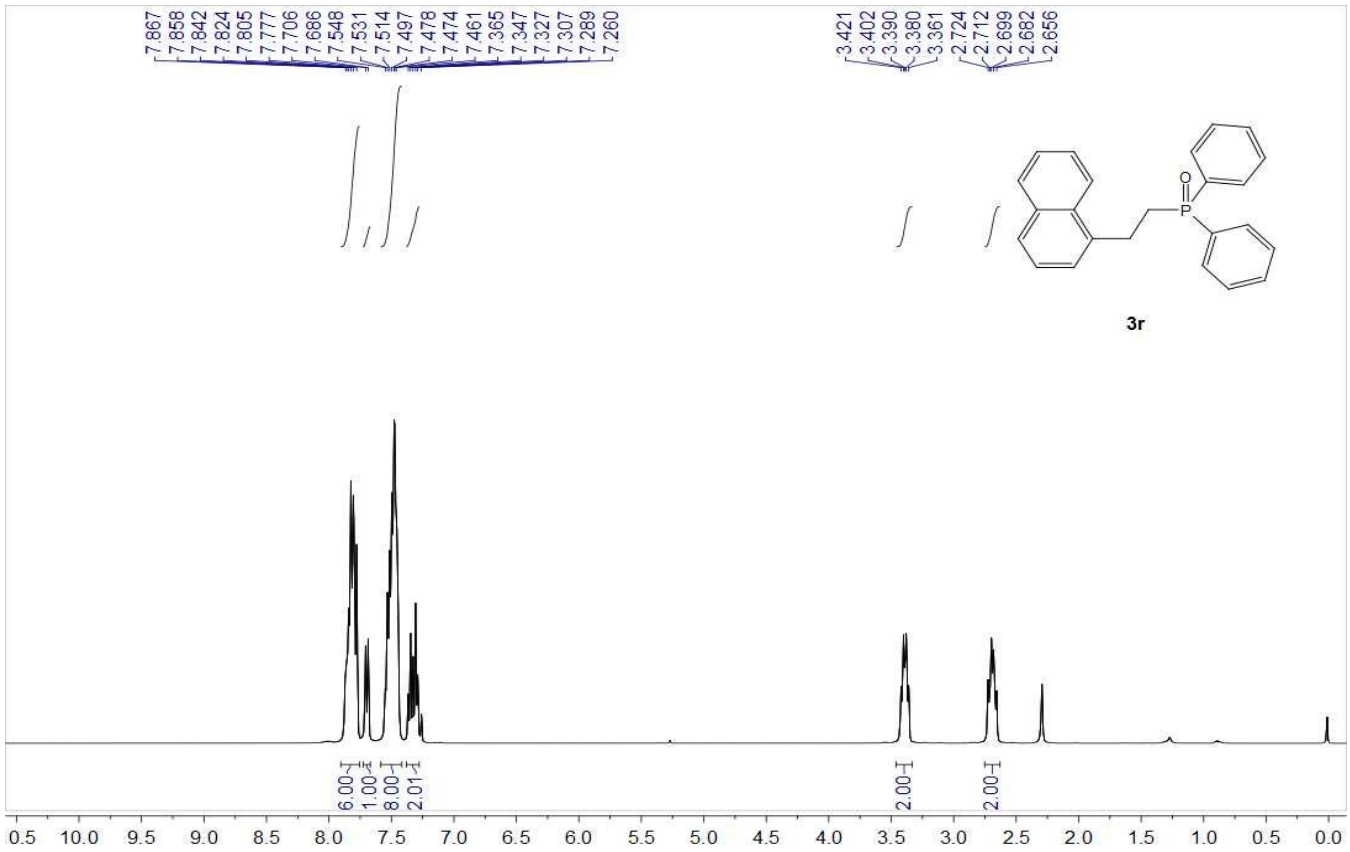
132.08
131.96
131.73
131.63
131.31
130.77
130.67
128.92
128.81
128.49
124.57
121.86
120.57
120.53
120.49
120.45
120.41
77.36
77.04
76.72
31.64
30.94
27.47
27.44
f1 (ppm)
78.03
77.28
76.97
2.84
3.24
3q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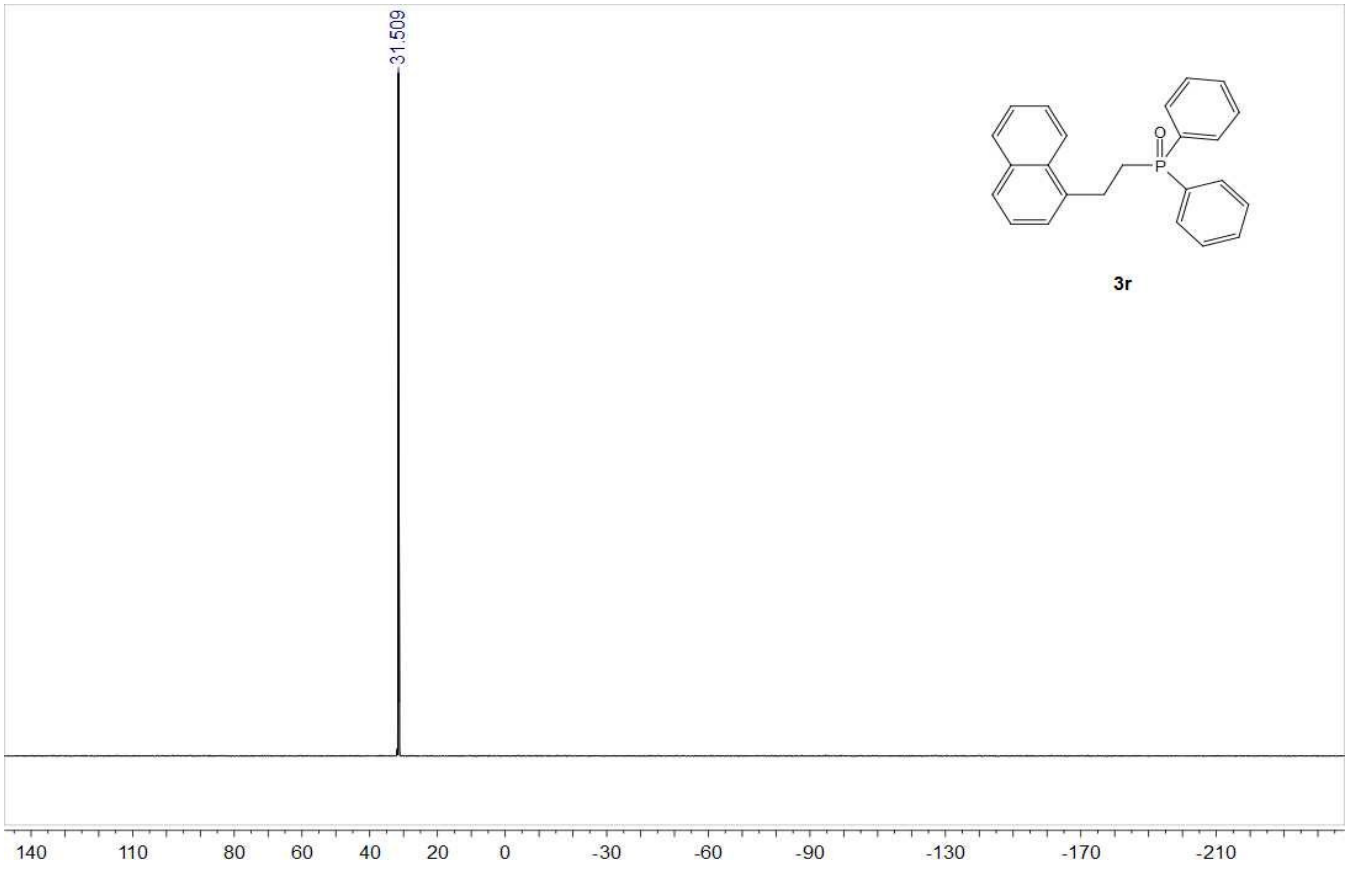
7.548
7.531
7.514
7.497
7.478
7.474
7.461
7.365
7.347
7.327
7.307
7.289
7.260
3.421
3.402
3.390
3.380
3.361
2.724
2.712
2.699
2.682
2.656
6.00
1.00
8.00
2.01
2.00
2.00
3r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P
3r
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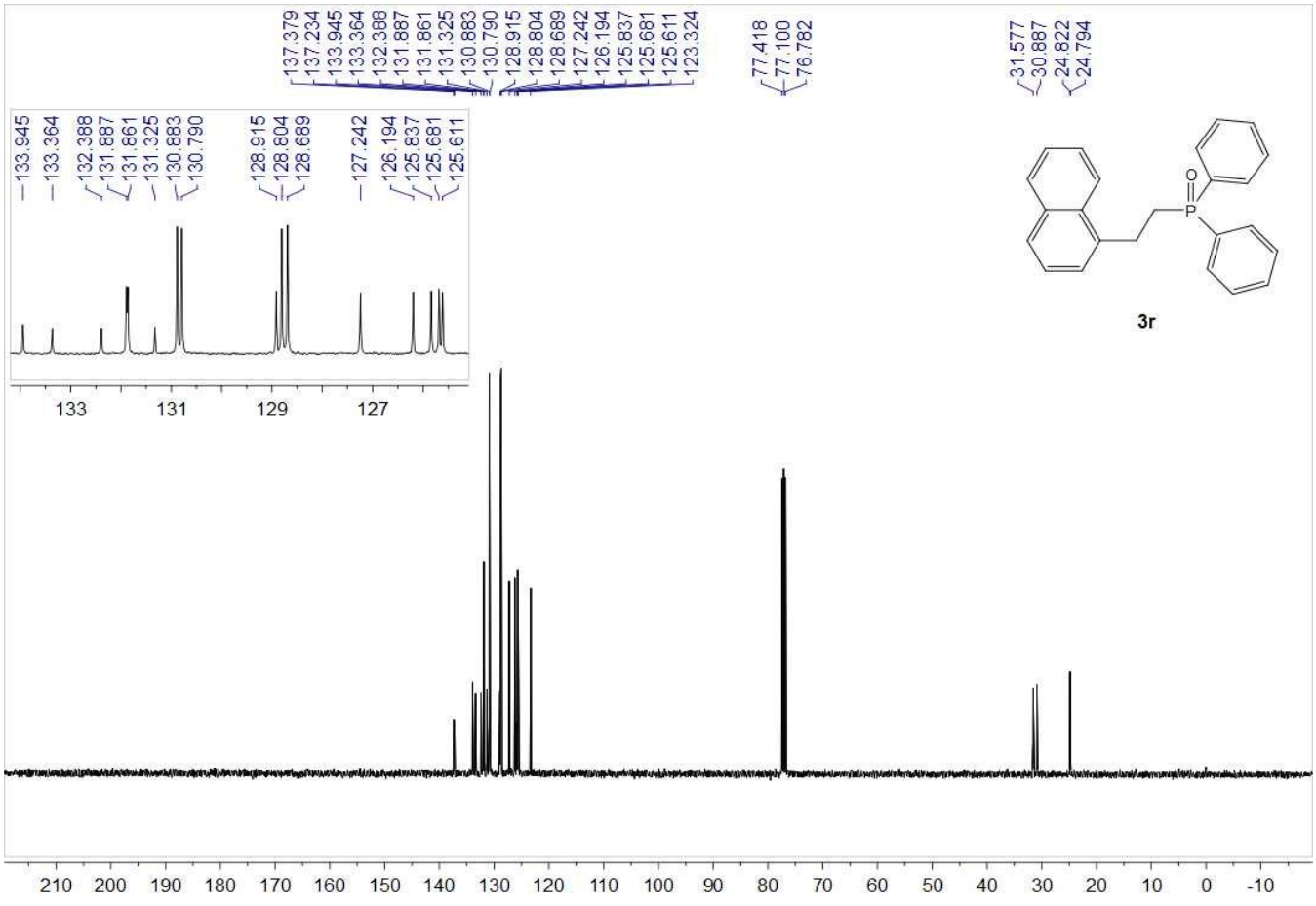
60
40
20
0
-30
-60
-90
-130
-170
-210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137.234
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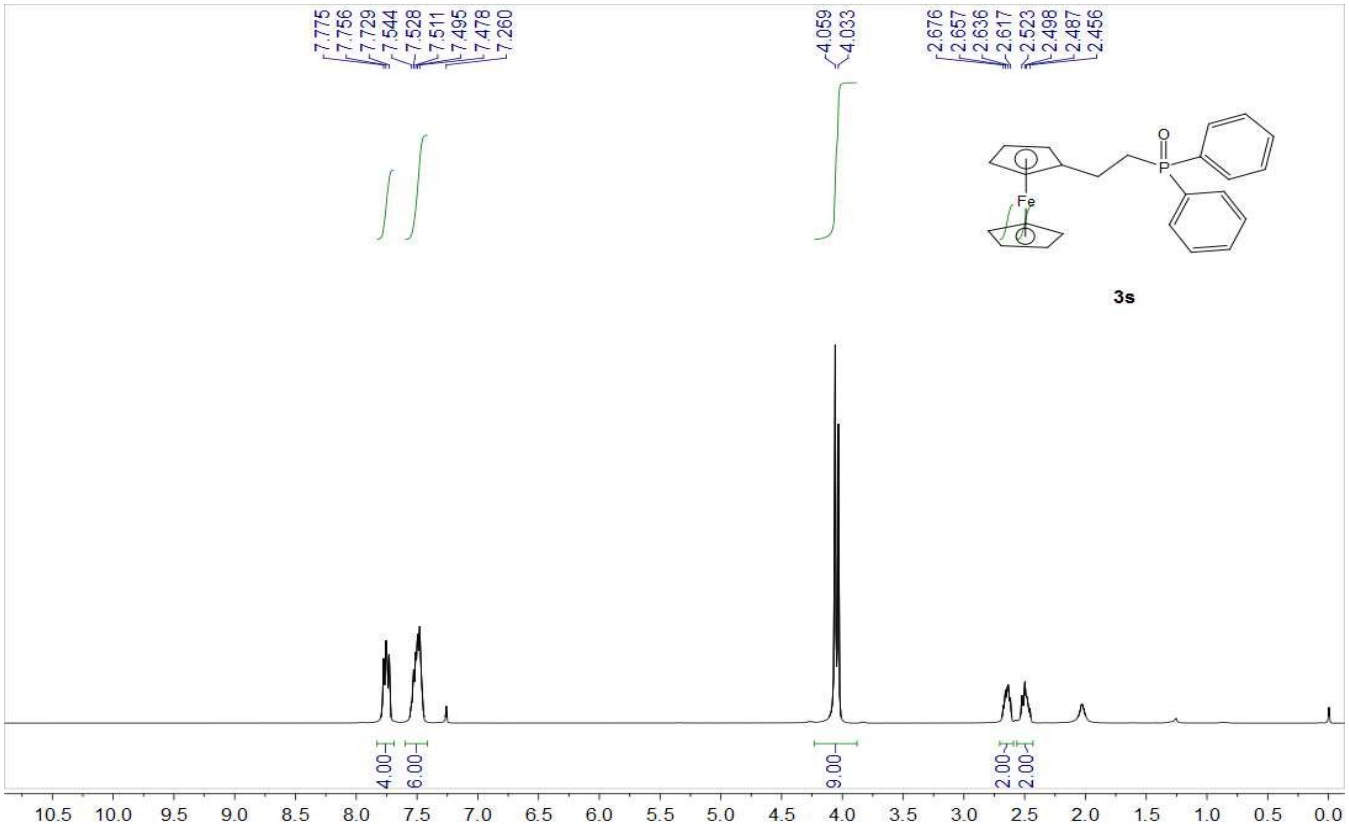
132.388
131.887
131.861
131.325
130.883
130.790
128.915
128.804
128.689
127.242
126.194
125.837
125.681
125.611
123.324
77.418
77.100
76.782
31.577
30.887
24.822
24.794
O
P
3r
133
131
129
127
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10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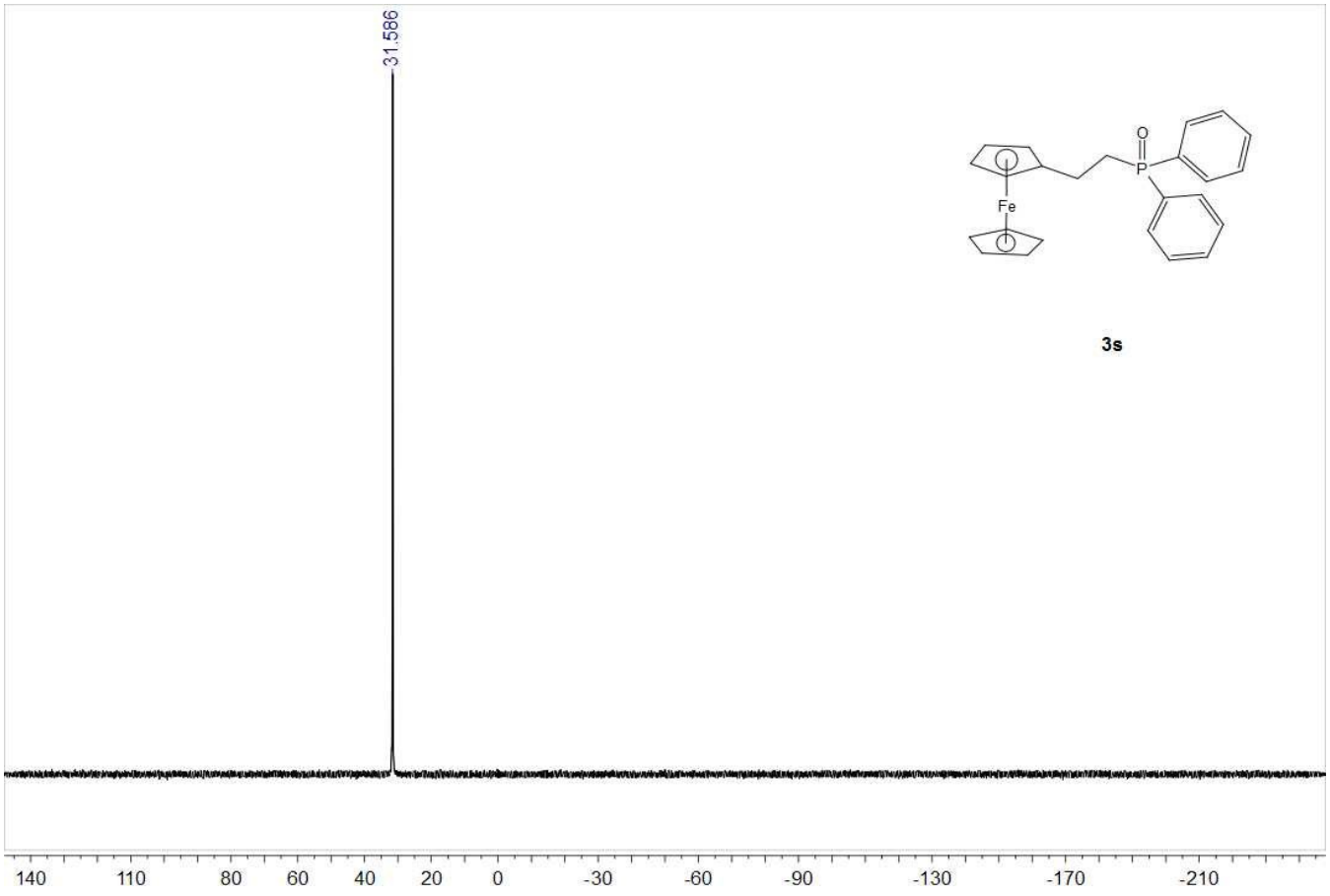
7.756
7.729
7.544
7.528
7.511
7.495
7.478
7.260
4.059
4.033
2.676
2.657
2.636
2.617
2.523
2.498
2.487
2.456
O
P
Fe
3s
4.00
6.00
9.00
2.00
2.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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P
Fe
3s
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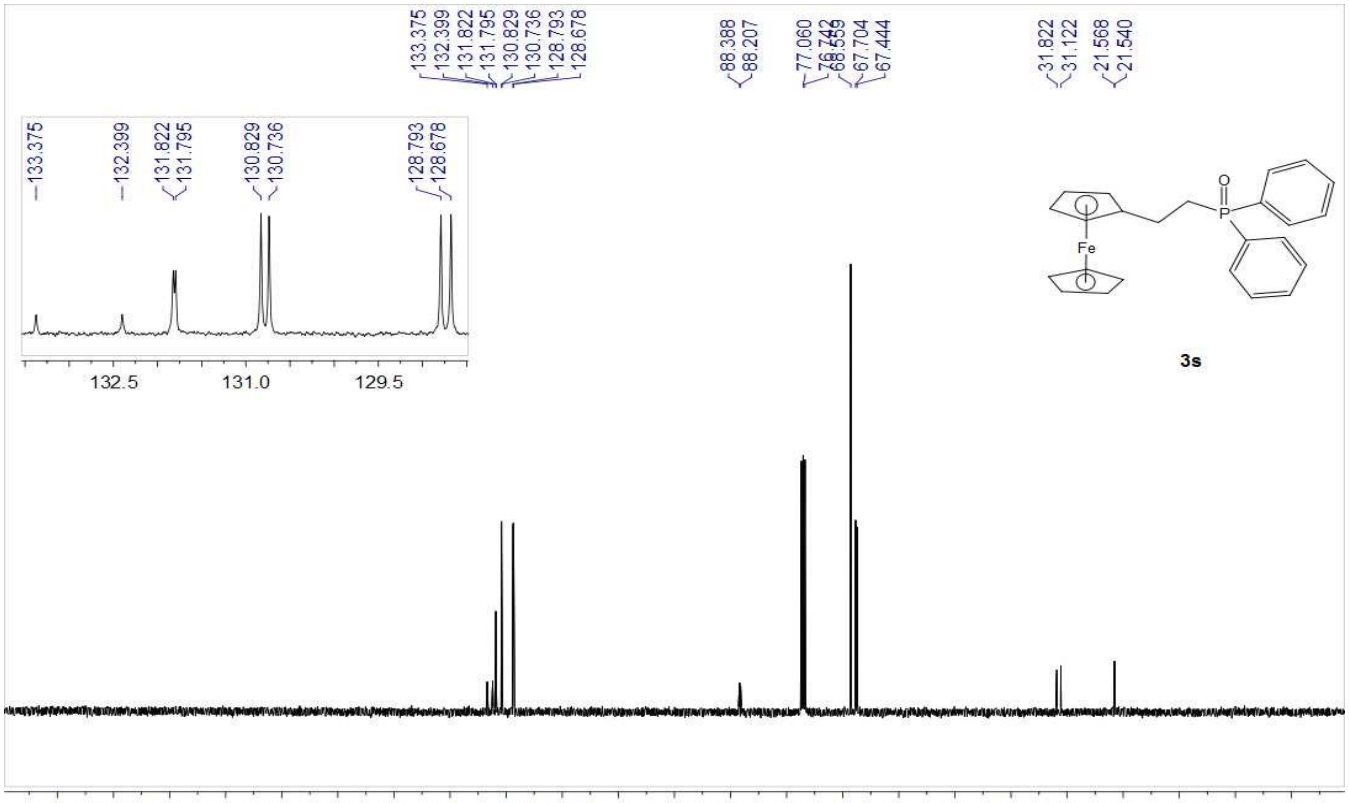
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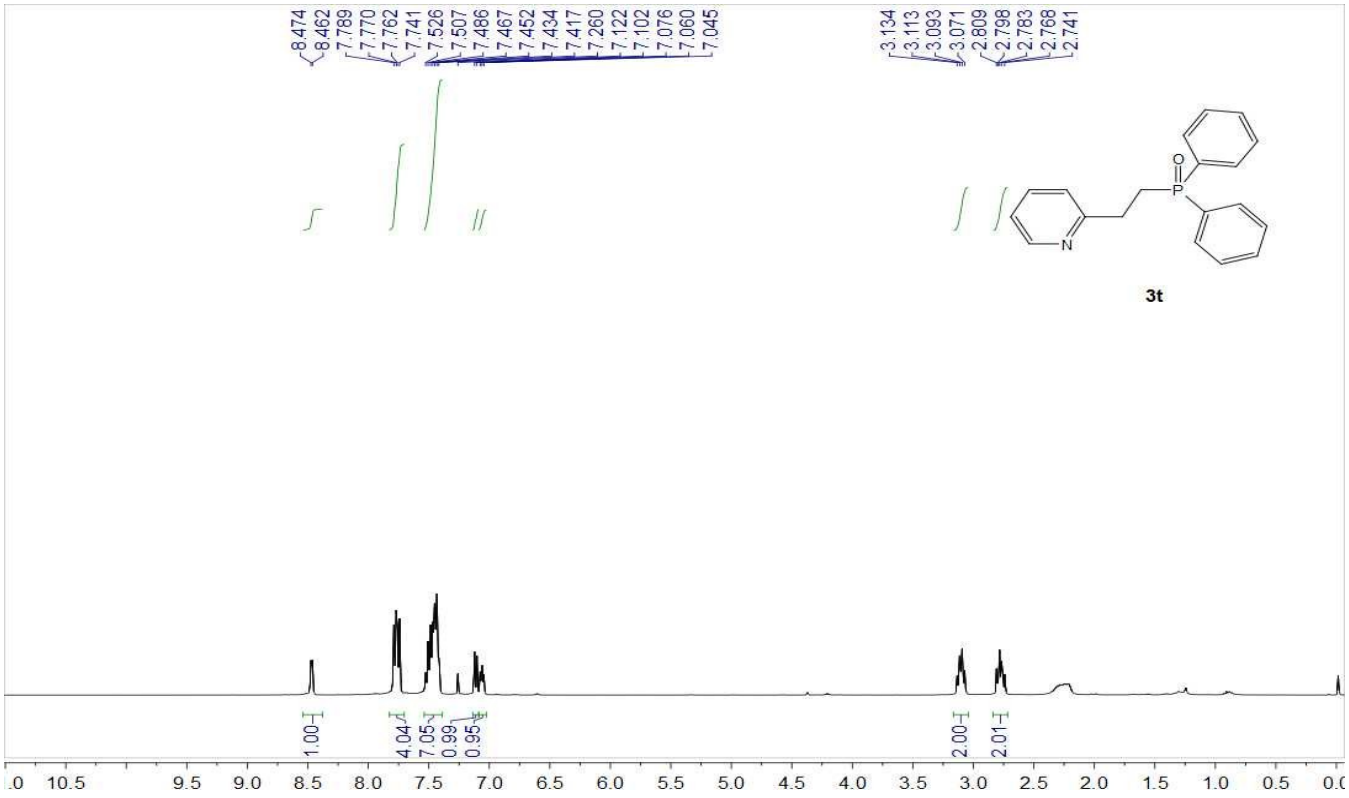
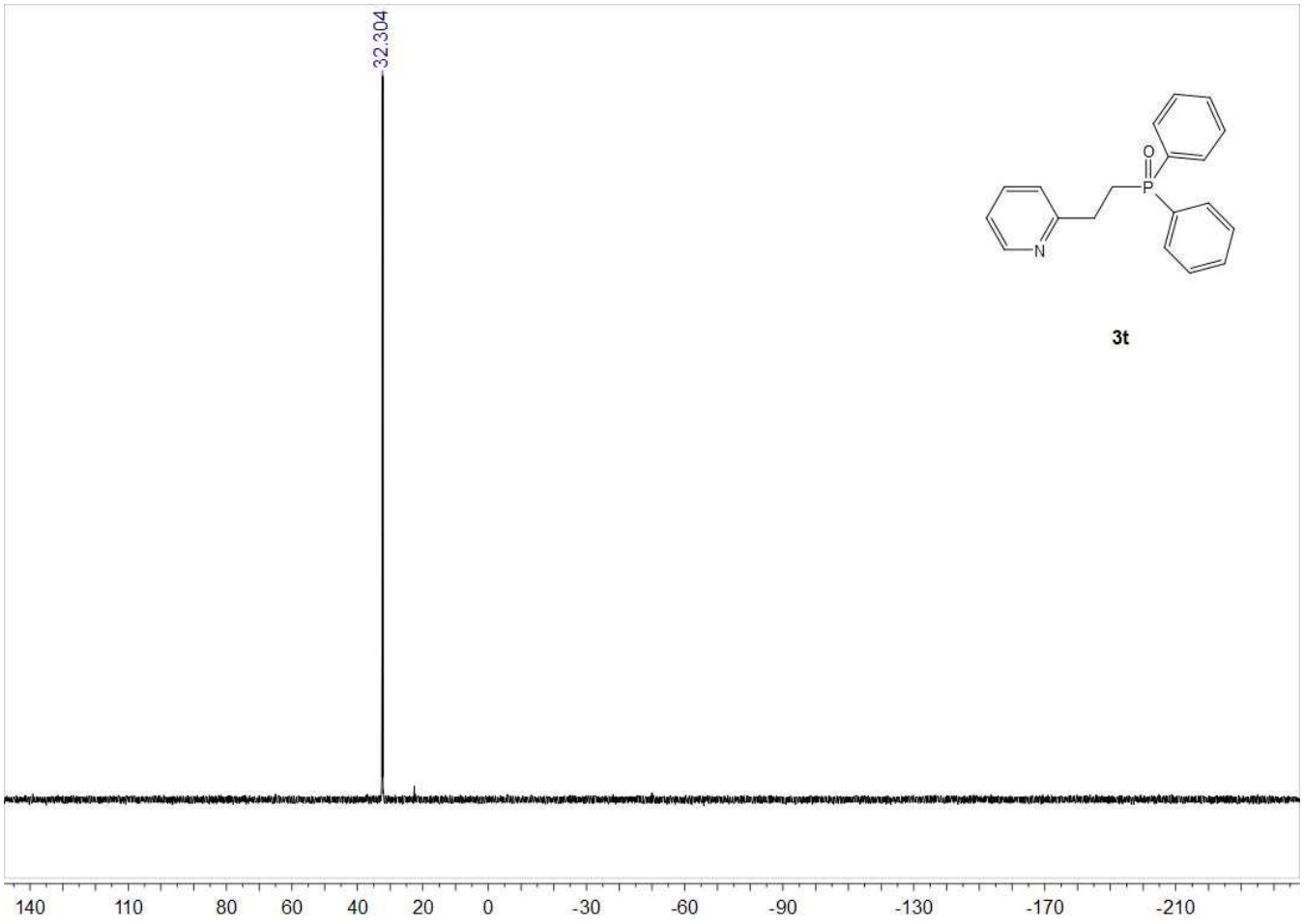
132.399
131.822
131.795
130.829
130.736
128.793
128.678
88.388
88.207
77.060
67.704
67.444
31.822
31.122
21.568
21.540
Fe
O
P
3s
132.5
131.0
129.5
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10


8.474
8.462
7.789
7.770
7.762
7.741
7.526
7.507
7.486
7.467
7.452
7.434
7.417
7.260
7.122
7.102
7.076
7.060
7.045
3.134
3.113
3.093
3.071
2.809
2.798
2.783
2.768
2.741
O
P
N
3t
1.00
4.04
7.05
0.99
0.95
2.00
2.01
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0


32.304
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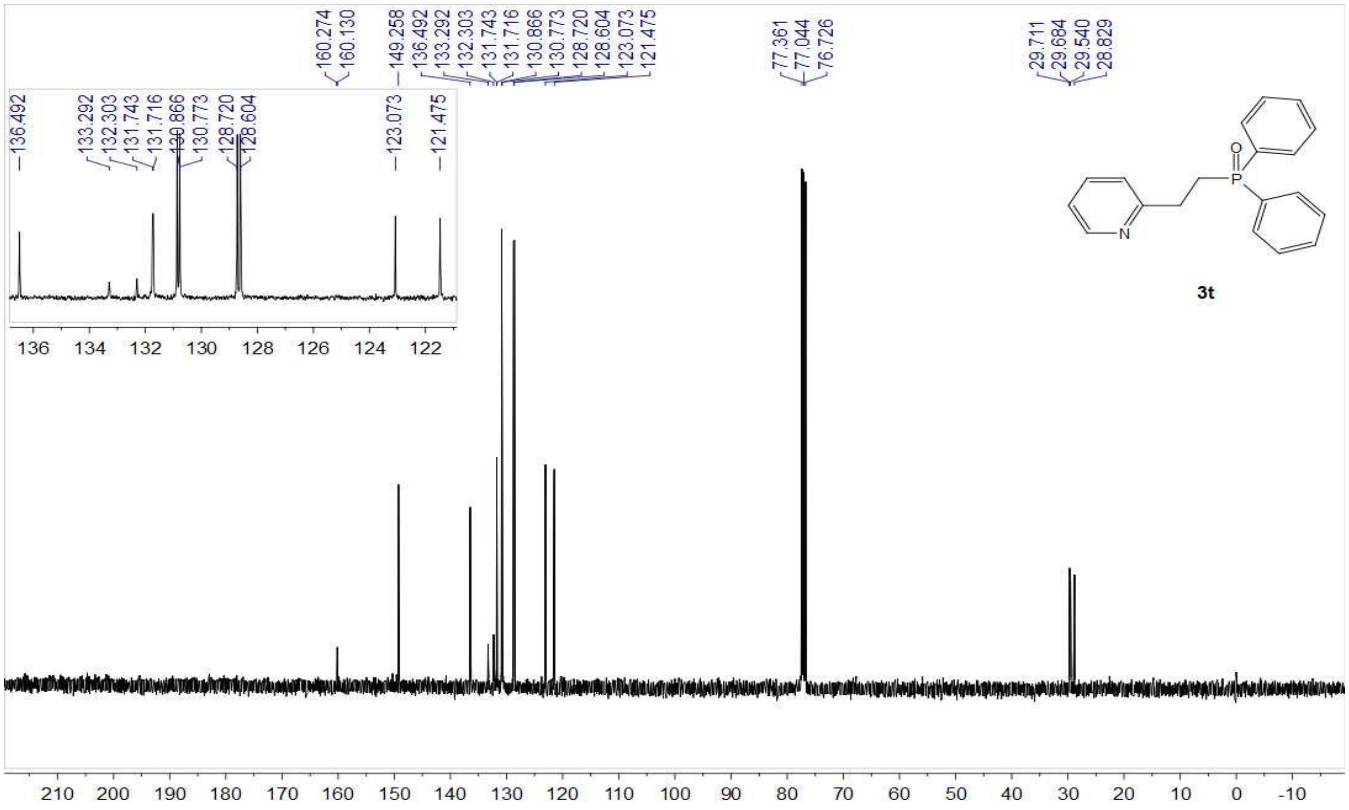
P
N
3t
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210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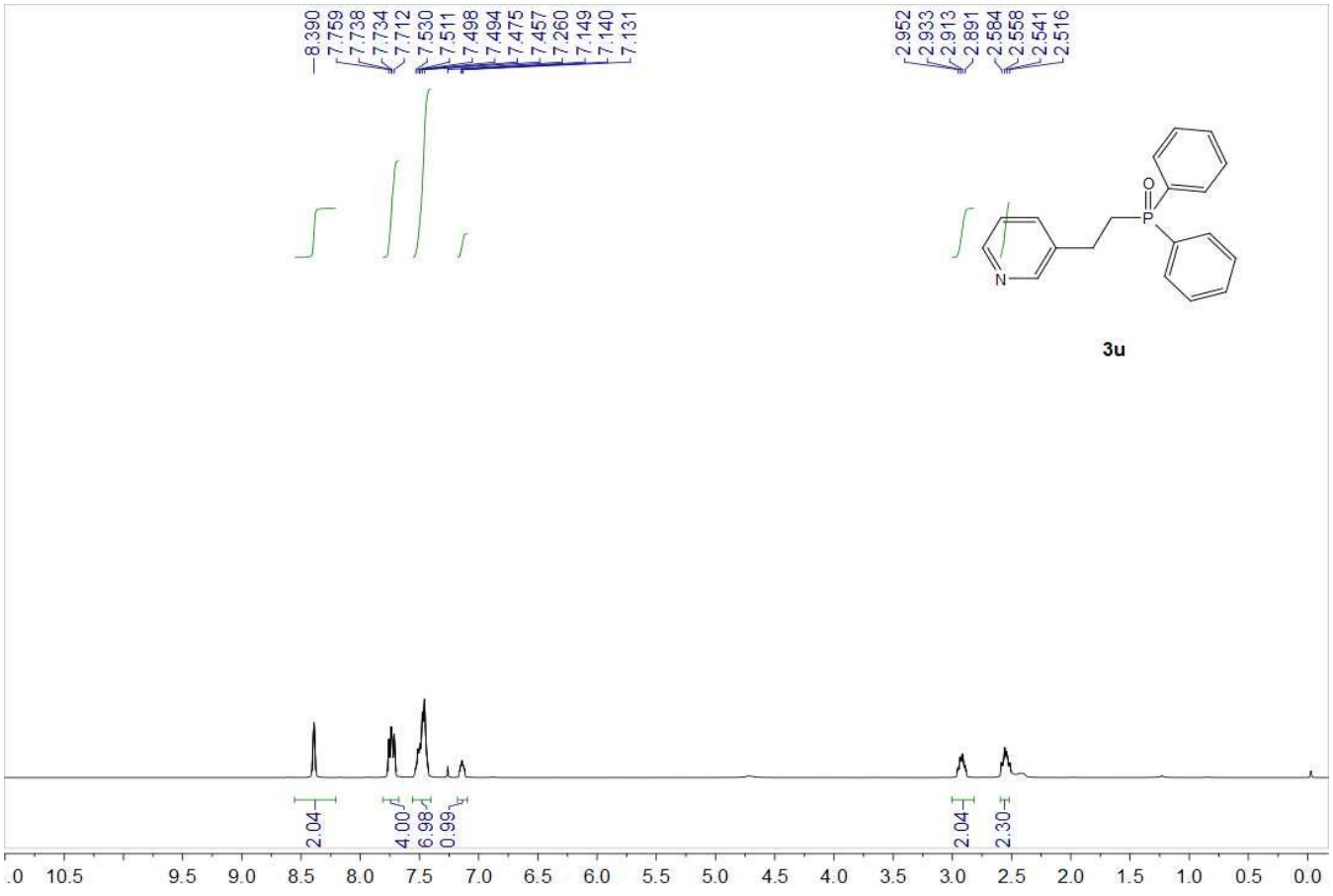
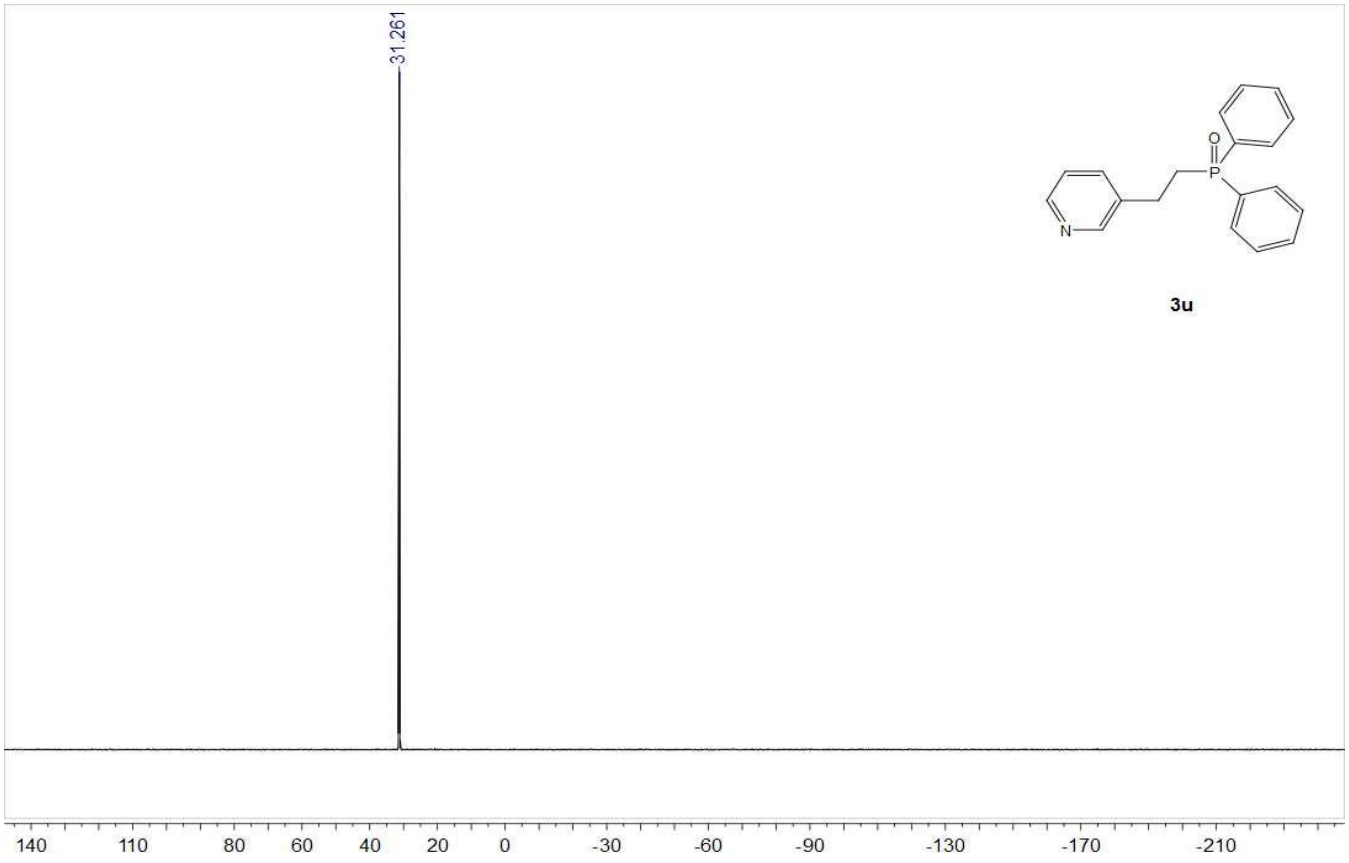
160.130
149.258
136.492
133.292
132.303
131.743
131.716
130.866
130.773
128.720
128.604
123.073
121.475
77.361
77.044
76.726
29.711
29.684
29.540
28.829
O
P
N
3t


8.390
7.759
7.738
7.734
7.712
7.530
7.511
7.498
7.494
7.475
7.457
7.260
7.149
7.140
7.131
2.952
2.933
2.913
2.891
2.584
2.558
2.541
2.516
O
P
N
3u
2.04
4.00
6.98
0.99
2.04
2.30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O
P
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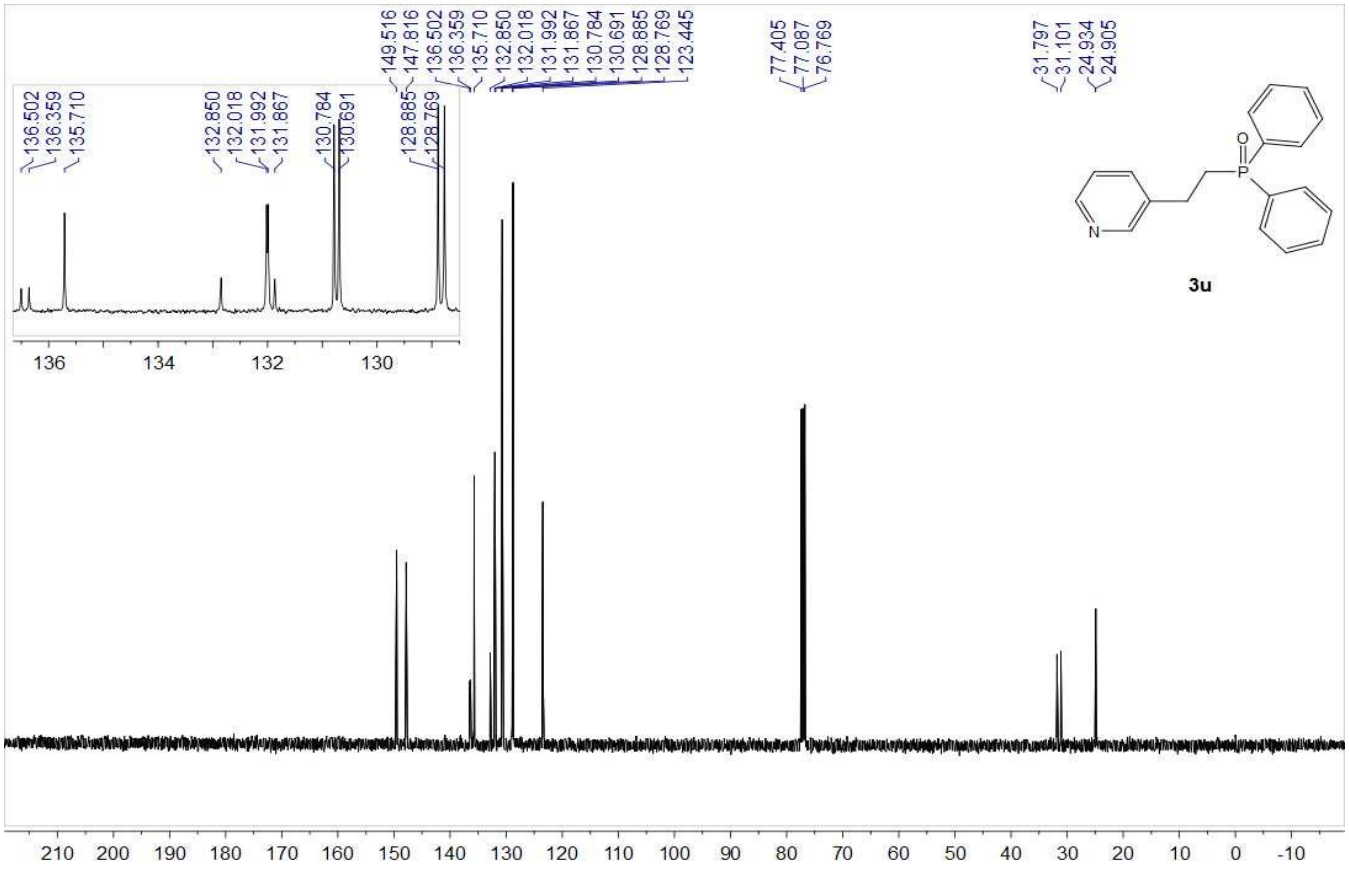
3u
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210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136.502
136.359
135.710
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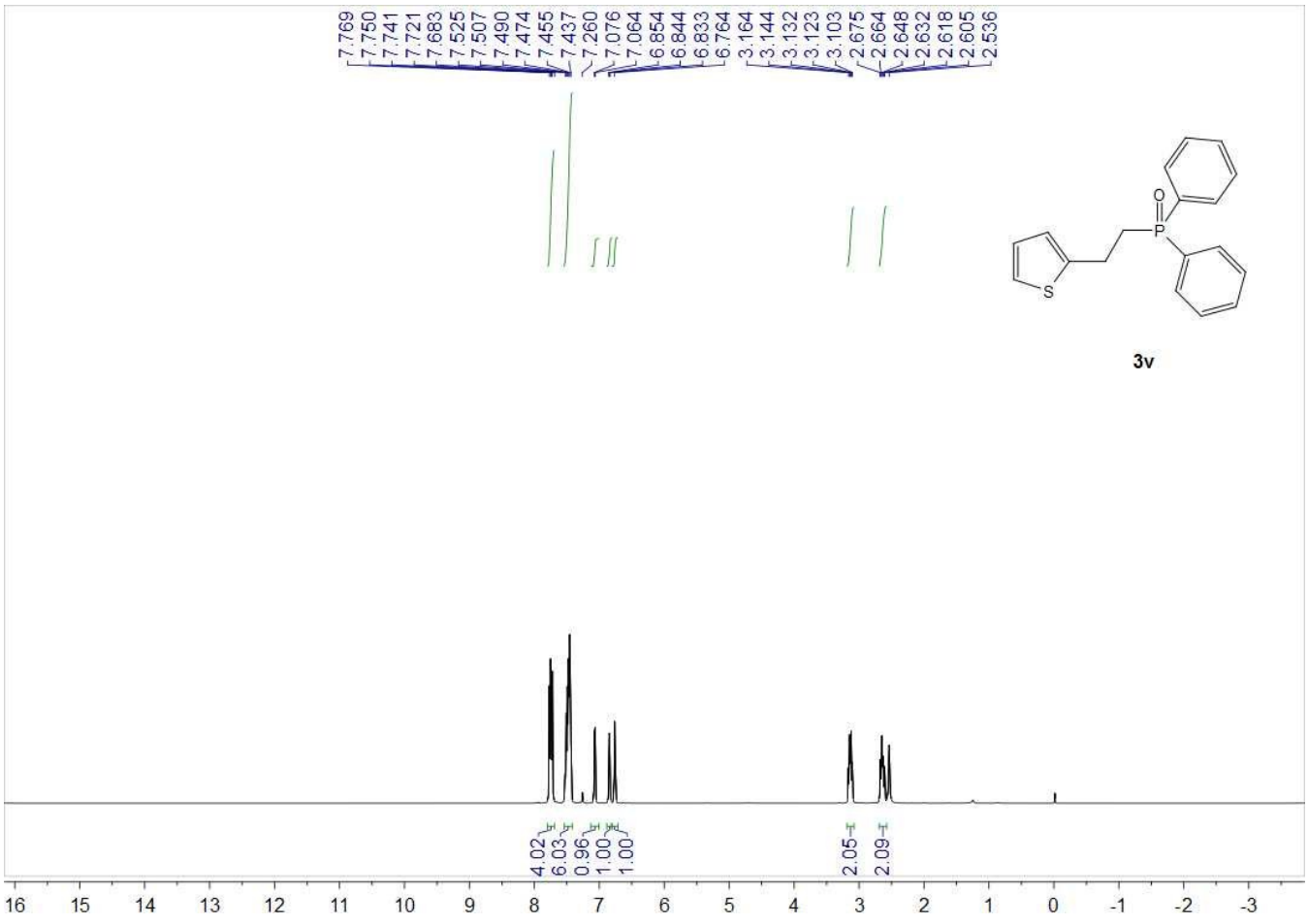
132.018
131.992
131.867
130.784
130.691
128.885
128.769
123.445
77.405
77.087
76.769
31.797
31.101
24.934
24.905
O
P
N
3u
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10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7.750
7.741
7.721
7.683
7.525
7.507
7.490
7.474
7.455
7.437
7.260
7.076
7.064
6.854
6.844
6.833
6.764
3.164
3.144
3.132
3.123
3.103
2.675
2.664
2.648
2.632
2.618
2.605
2.536
O
P
S
3v
4.02
6.03
0.96
1.00
1.00
2.05
2.09
16
15
14
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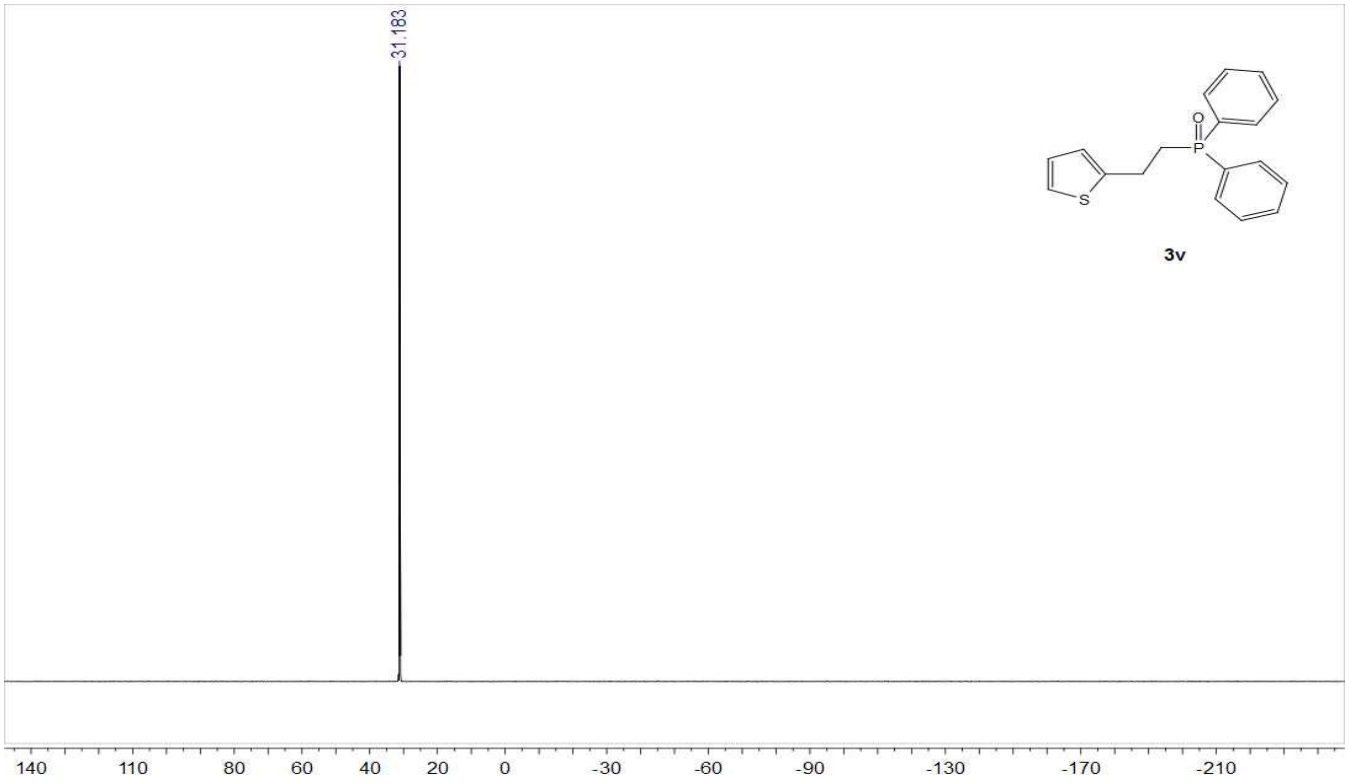
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3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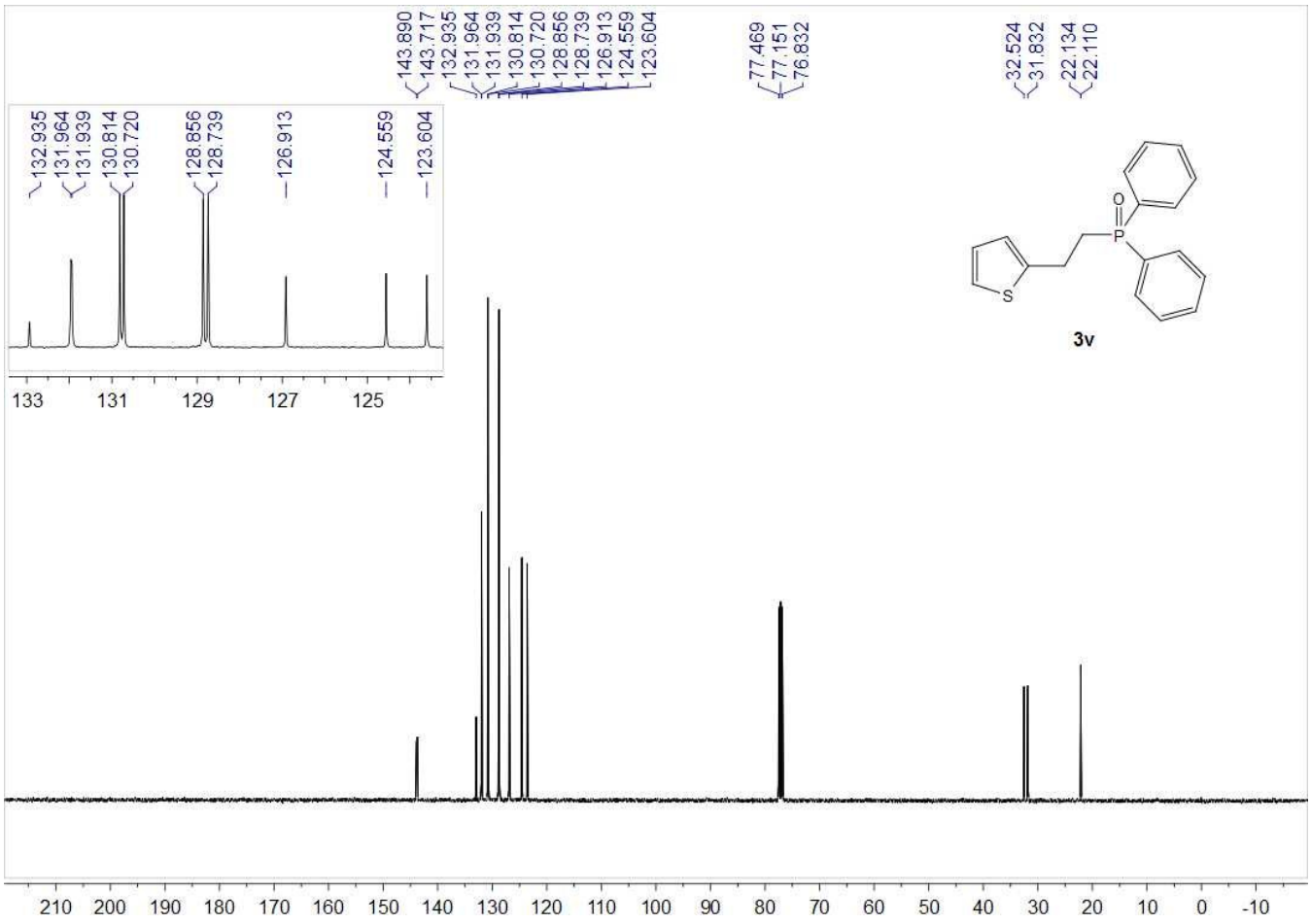
3v
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210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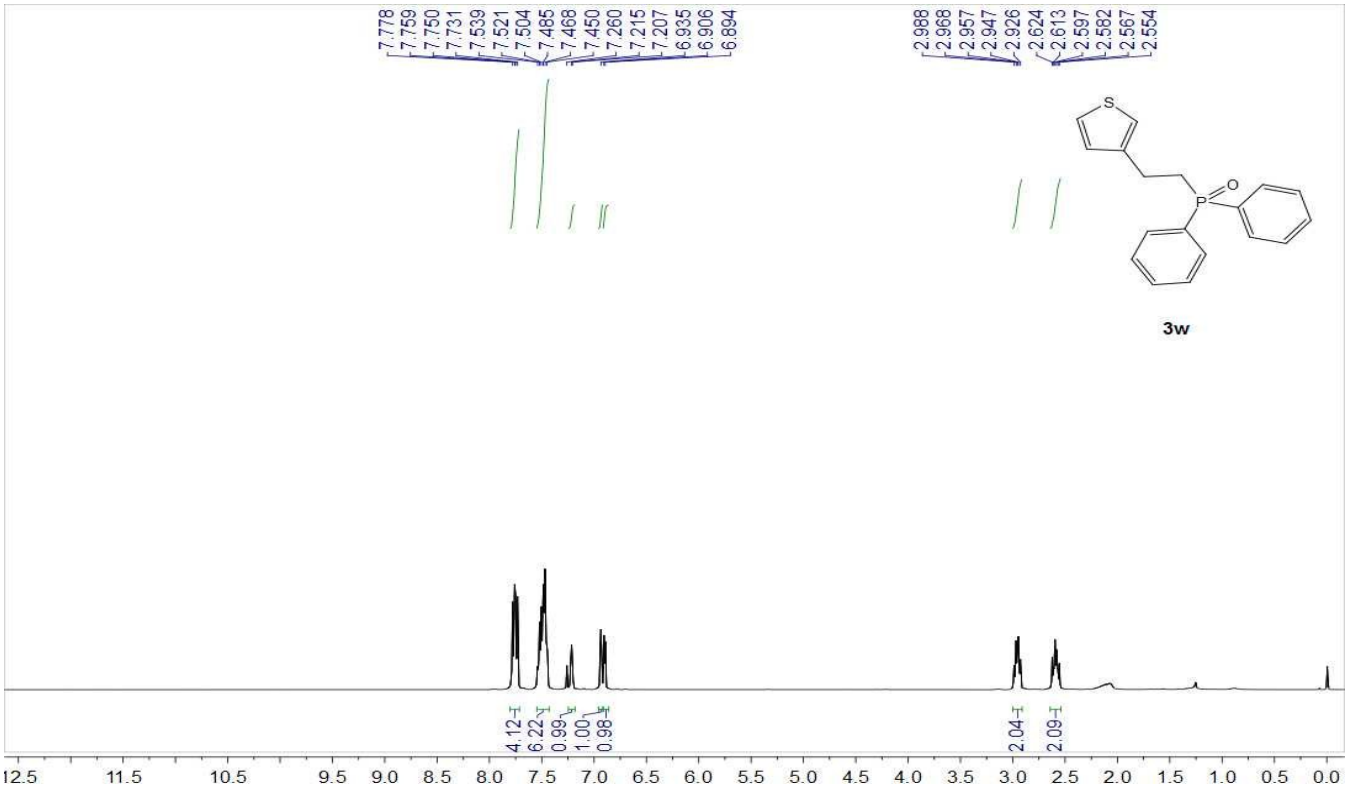
131.939
130.814
130.720
128.856
128.739
126.913
124.559
123.604
77.469
77.151
76.832
32.524
31.832
22.134
22.110
3v
133
131
129
127
125
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10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7.759
7.750
7.731
7.539
7.521
7.504
7.485
7.468
7.450
7.260
7.215
7.207
6.935
6.906
6.894
2.988
2.968
2.957
2.947
2.926
2.624
2.613
2.597
2.582
2.567
2.554
S
P
O
3w
4.12
6.22
0.99
1.00
0.98
2.04
2.09
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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4.0
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3.0
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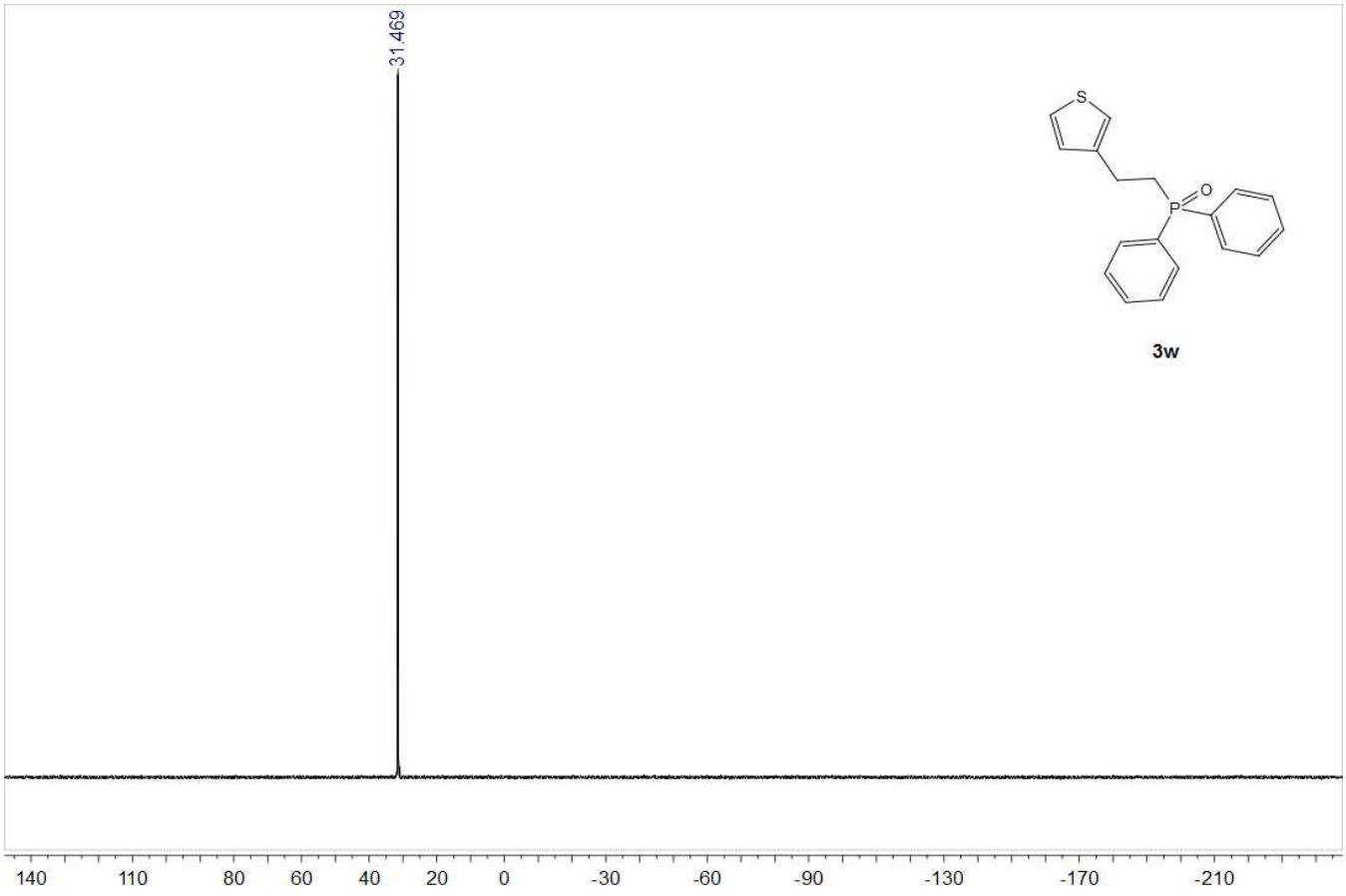
2.0
1.5
1.0
0.5
0.0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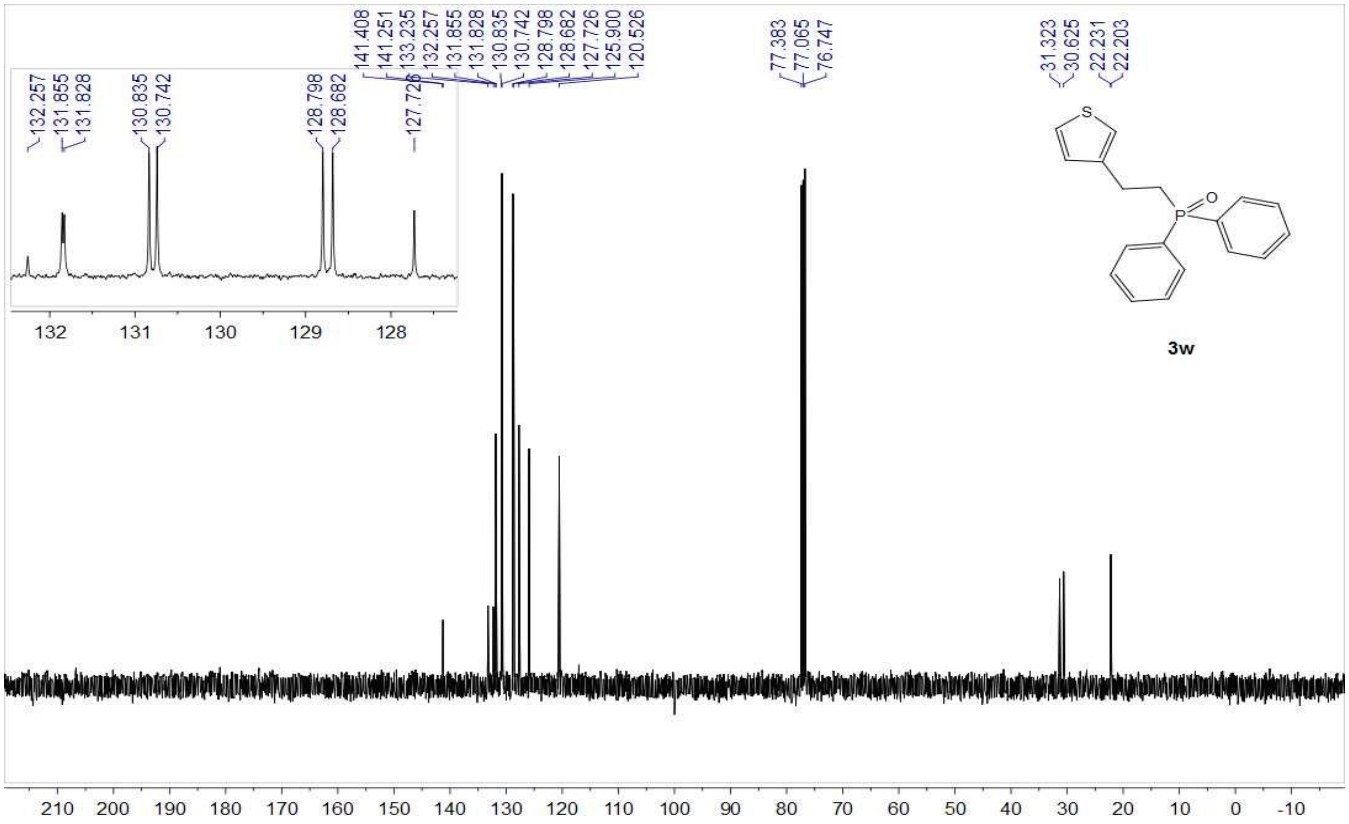
S
P
O
3w
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210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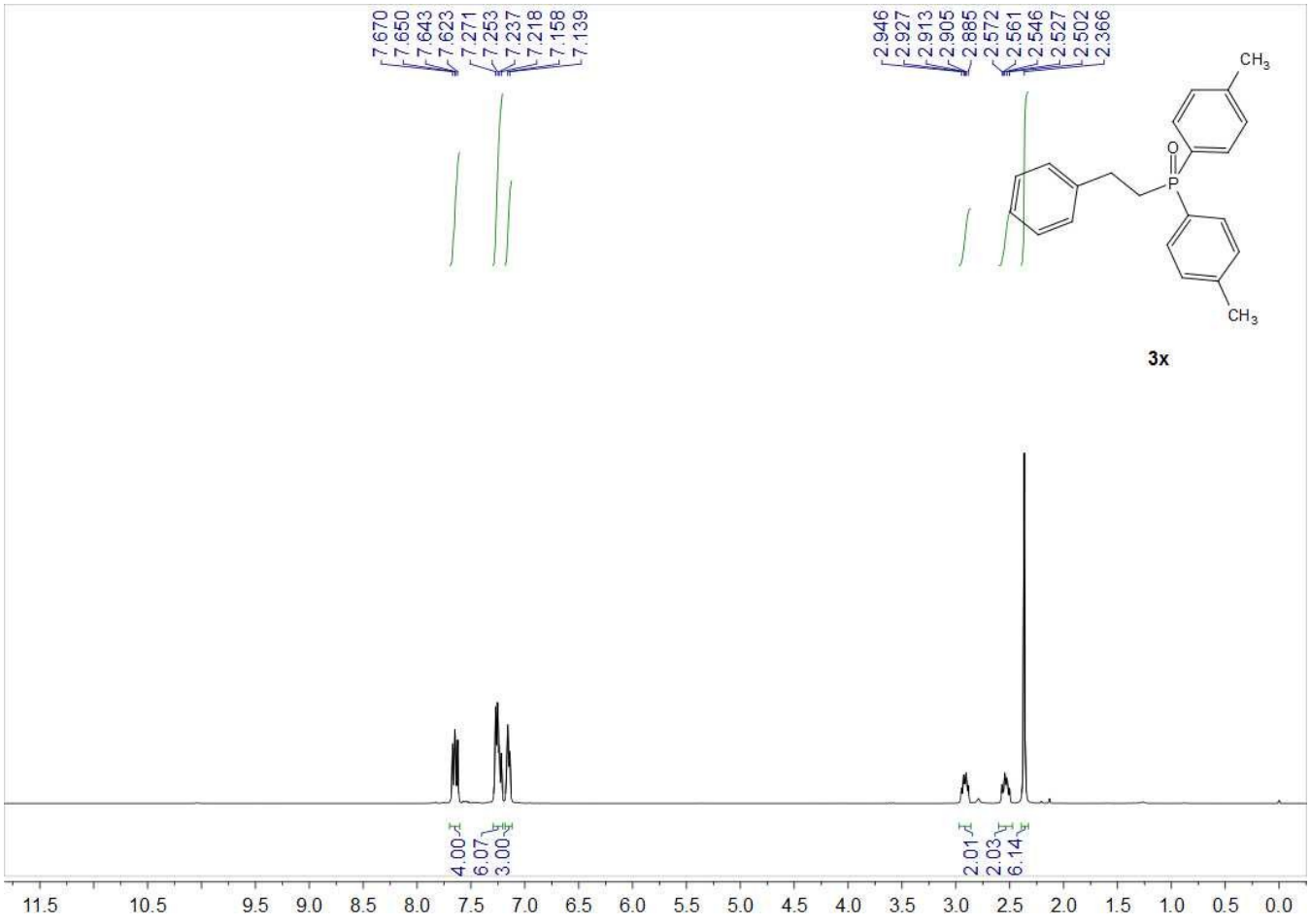
131.828
130.835
130.742
128.798
128.682
127.726
125.900
120.526
77.383
77.065
76.747
31.323
30.625
22.231
22.203
132.257
131.855
131.828
130.835
130.742
128.798
128.682
127.726
132
131
130
129
128
S
P
O
3w
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10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7.271
7.253
7.237
7.218
7.158
7.139
2.946
2.927
2.913
2.905
2.885
2.572
2.561
2.546
2.527
2.502
2.366
CH3
O
P
CH3
3x
4.00
6.07
3.00
2.01
2.03
6.14
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
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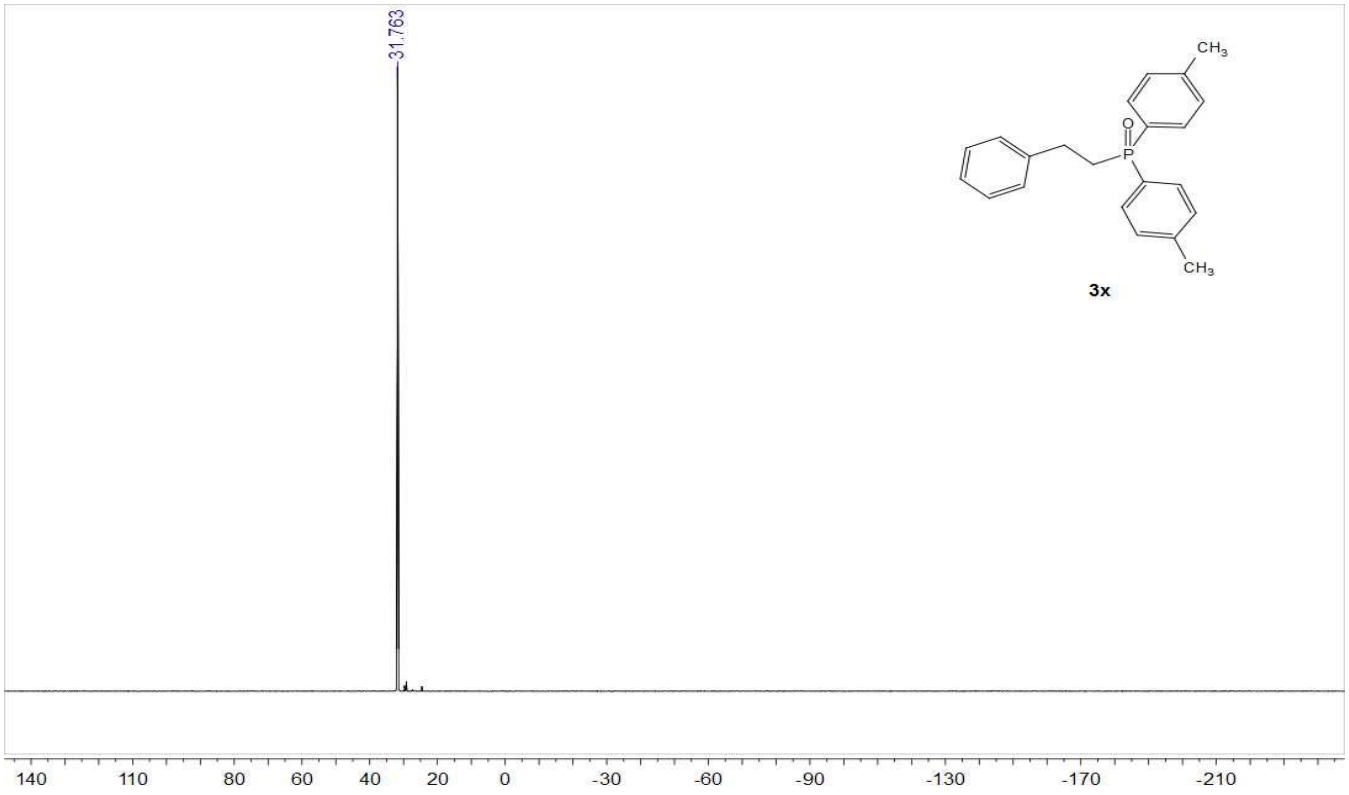
1.0
0.5
0.0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CH3
O
P
CH3
3x
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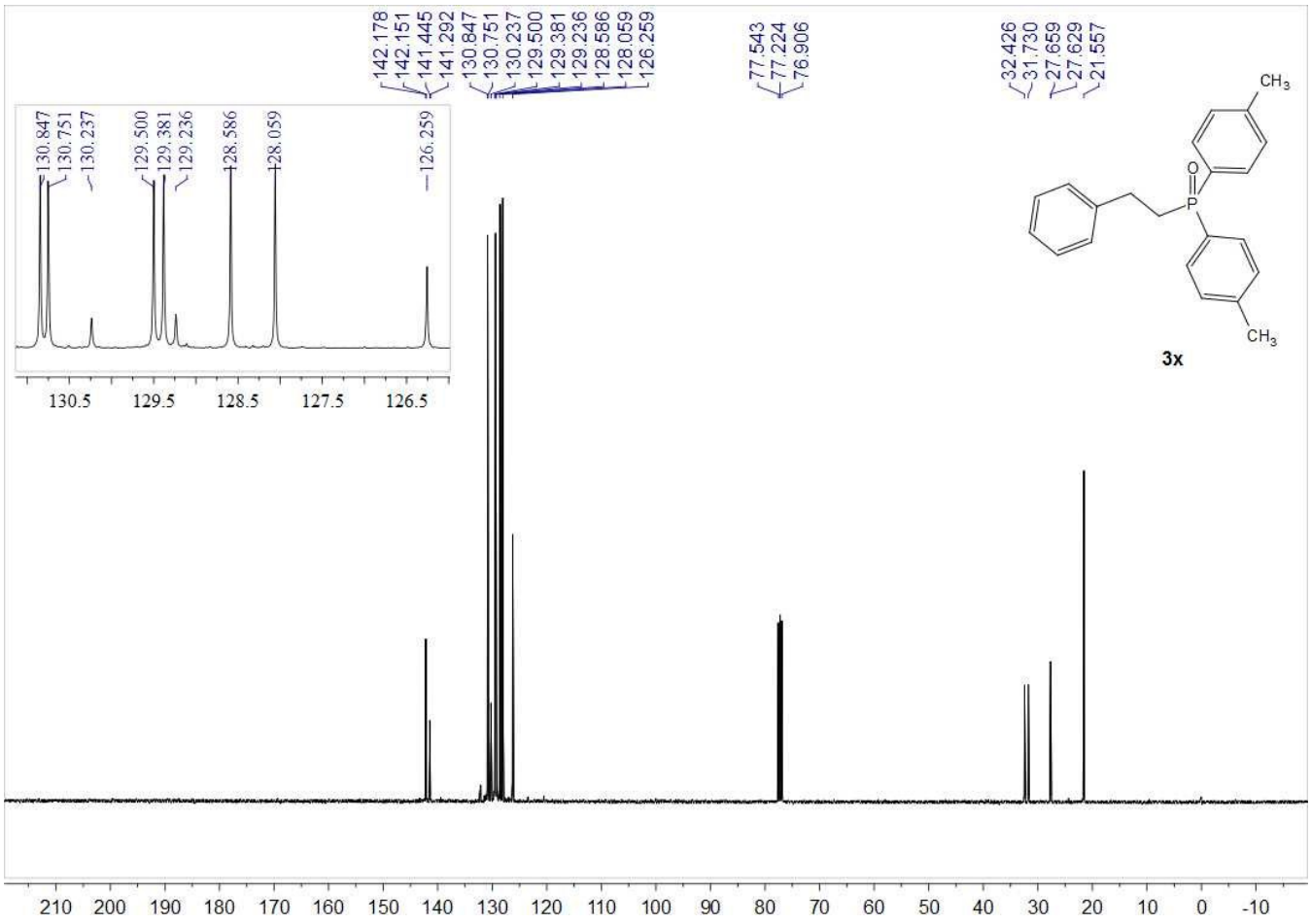
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210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141.445
141.292
130.847
130.751
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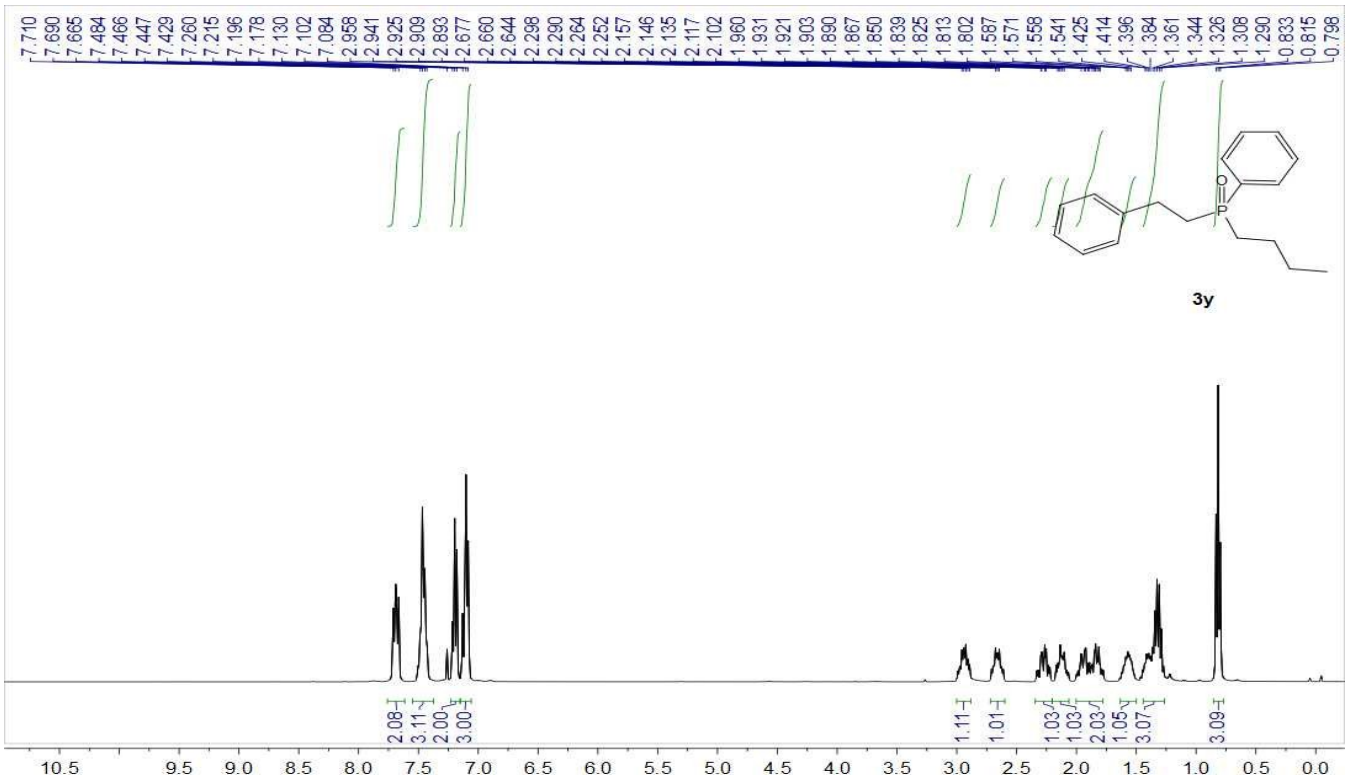
129.500
129.381
129.236
128.586
128.059
126.259
77.543
77.224
76.906
32.426
31.730
27.659
27.629
21.557
130.847
130.751
130.237
129.500
129.381
129.236
128.586
128.059
126.259
130.5
129.5
128.5
127.5
126.5
CH3
O
P
CH3
3x
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10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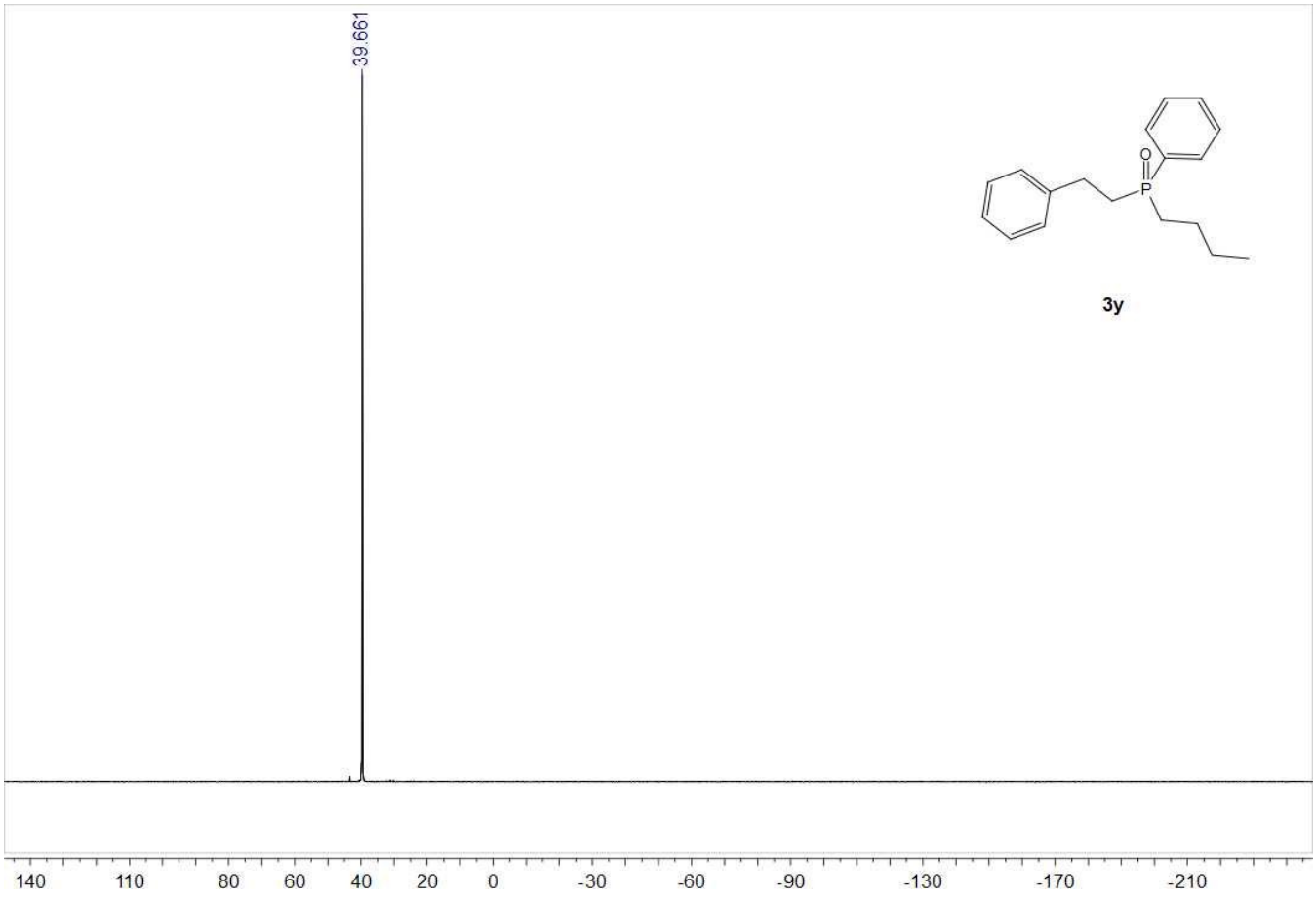
7.665
7.484
7.466
7.447
7.429
7.260
7.215
7.196
7.178
7.130
7.102
7.084
2.958
2.941
2.925
2.909
2.893
2.677
2.660
2.644
2.298
2.290
2.264
2.252
2.157
2.146
2.135
2.117
2.102
1.960
1.931
1.921
1.903
1.890
1.867
1.850
1.839
1.825
1.813
1.802
1.587
1.571
1.558
1.541
1.425
1.414
1.396
1.384
1.361
1.344
1.326
1.308
1.290
0.833
0.815
0.798
O
P
3y
2.08
3.11
2.00
3.00
1.11
1.01
1.03
1.03
2.03
1.05
3.07
3.09
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0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P
3y
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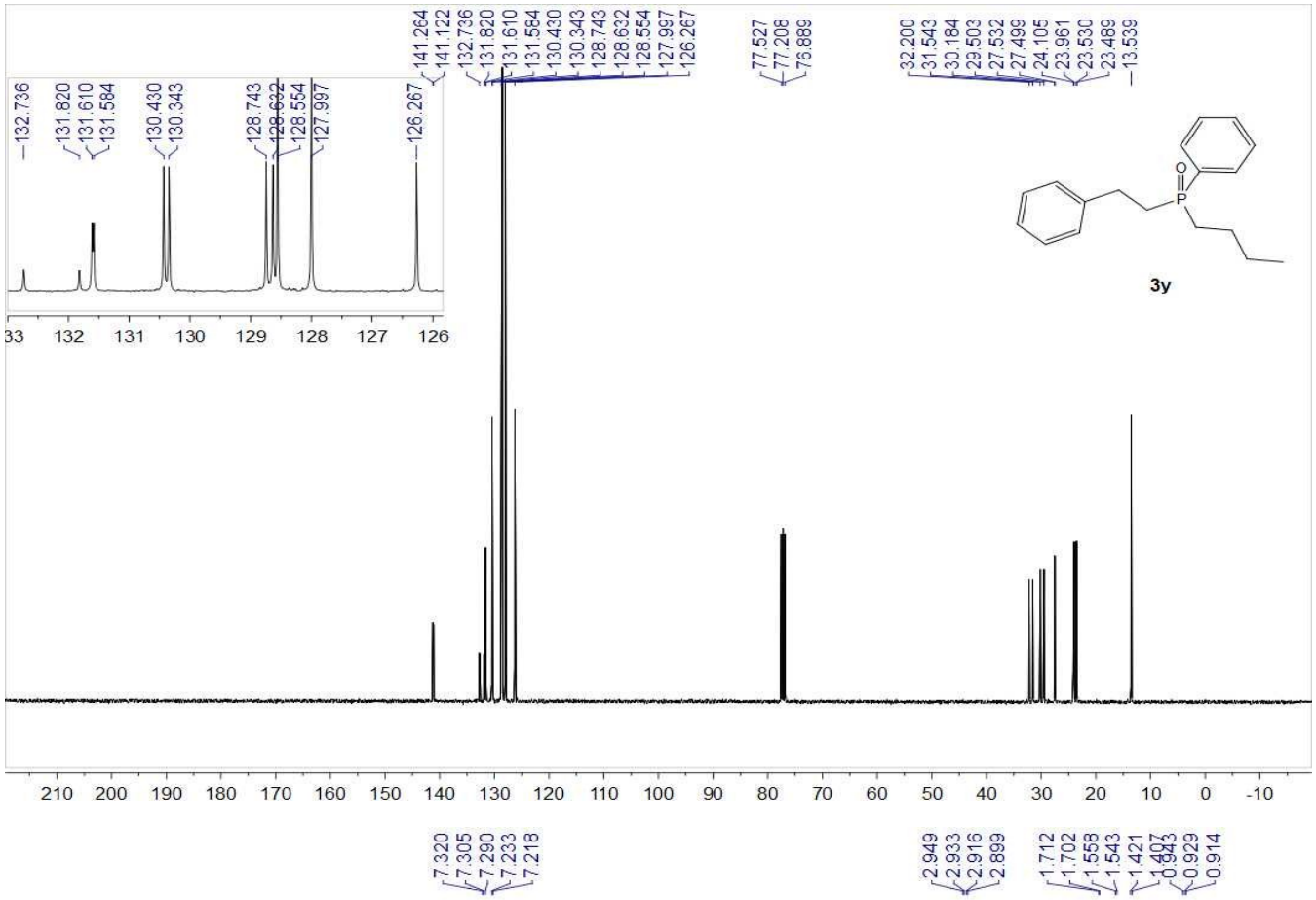
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210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141.122
132.736
131.820
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131.584
130.430
130.343
128.743
128.632
128.554
127.997
126.267
77.527
77.208
76.889
32.200
31.543
30.184
29.503
27.532
27.499
24.105
23.961
23.530
23.489
13.539
O
P
3y


7.320
7.305
7.290
7.233
7.218
2.949
2.933
2.916
2.899
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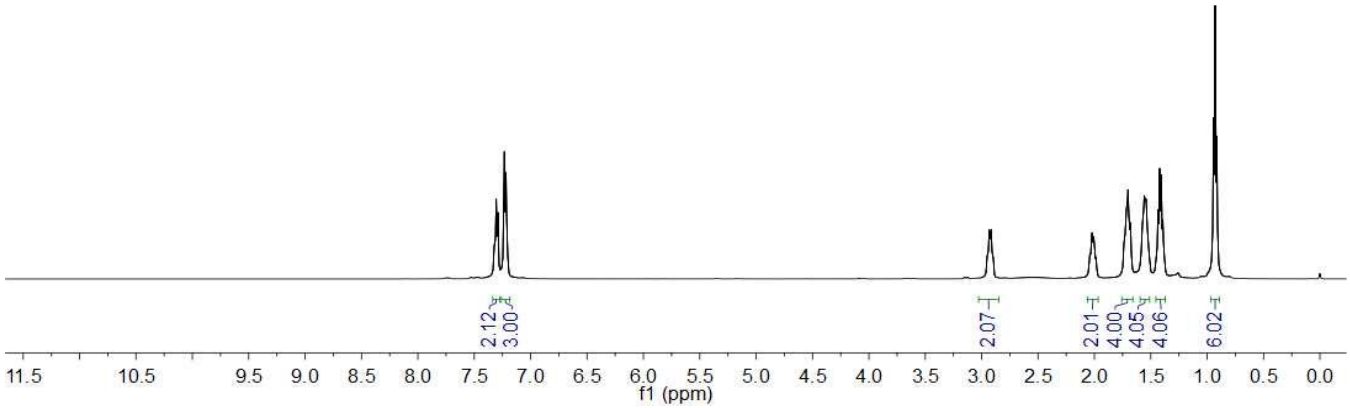
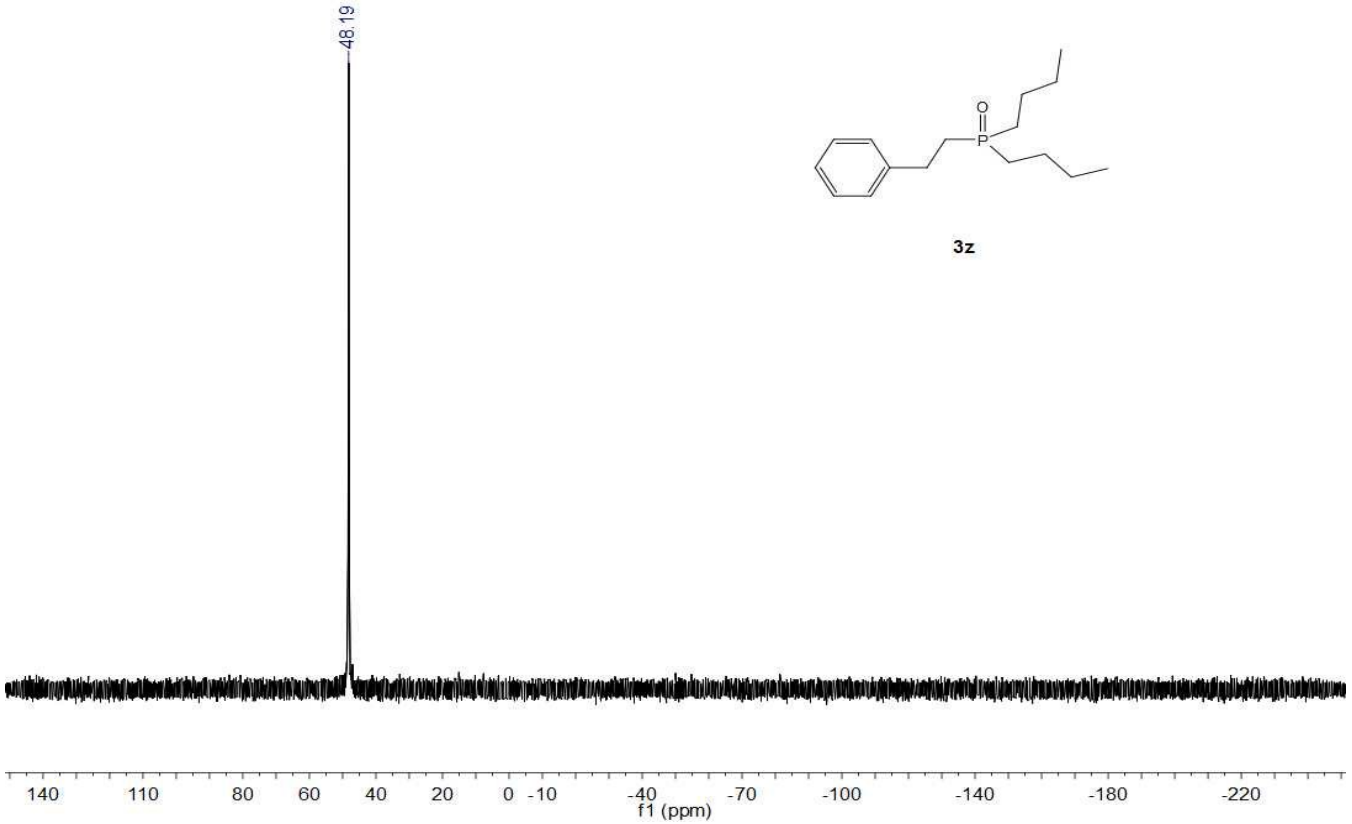
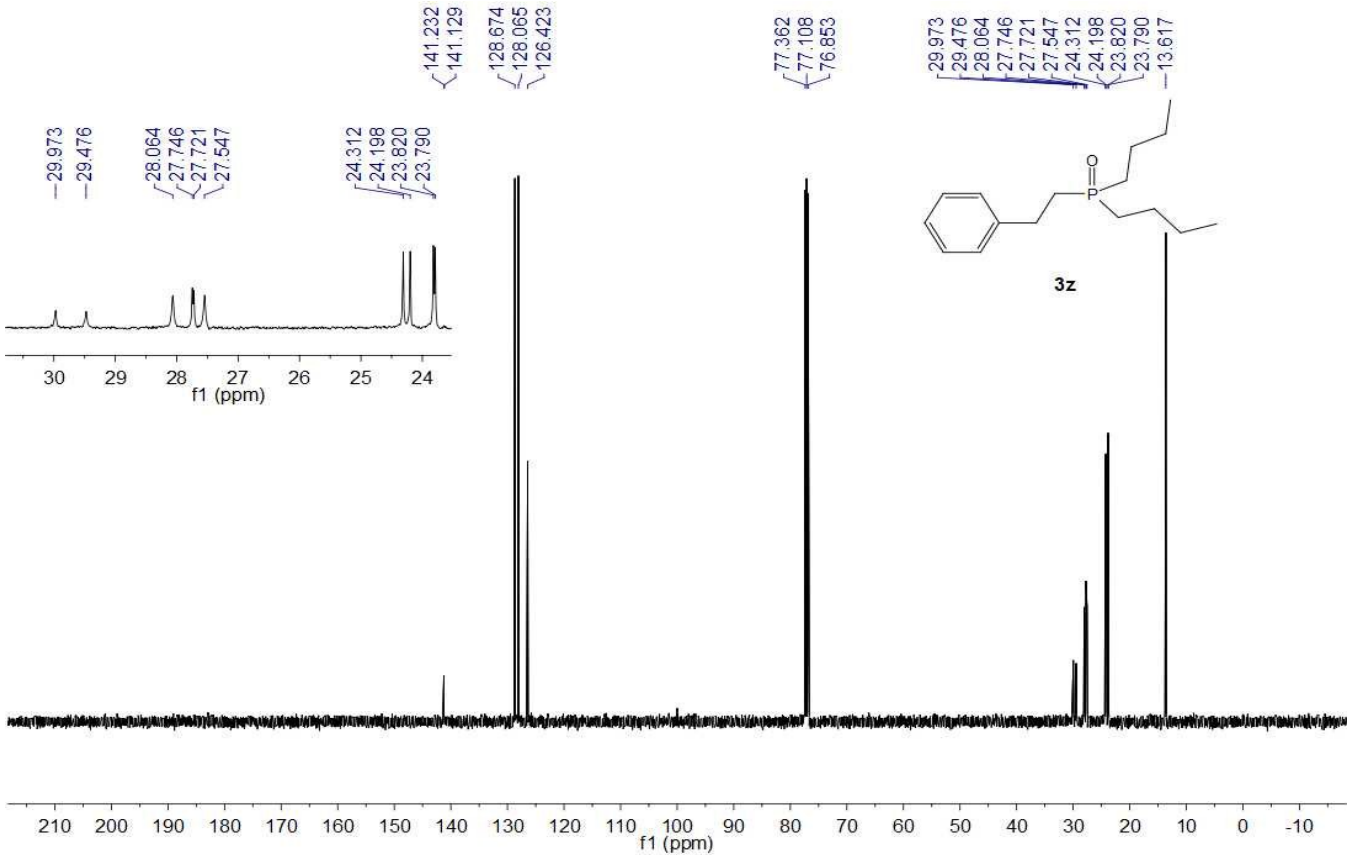
1.558
1.543
1.421
1.407
0.943
0.929
0.914
O
P
3z


2.12
3.00
2.07
2.01
4.00
4.05
4.06
6.02
f1 (ppm)


48.19
f1 (ppm)

3z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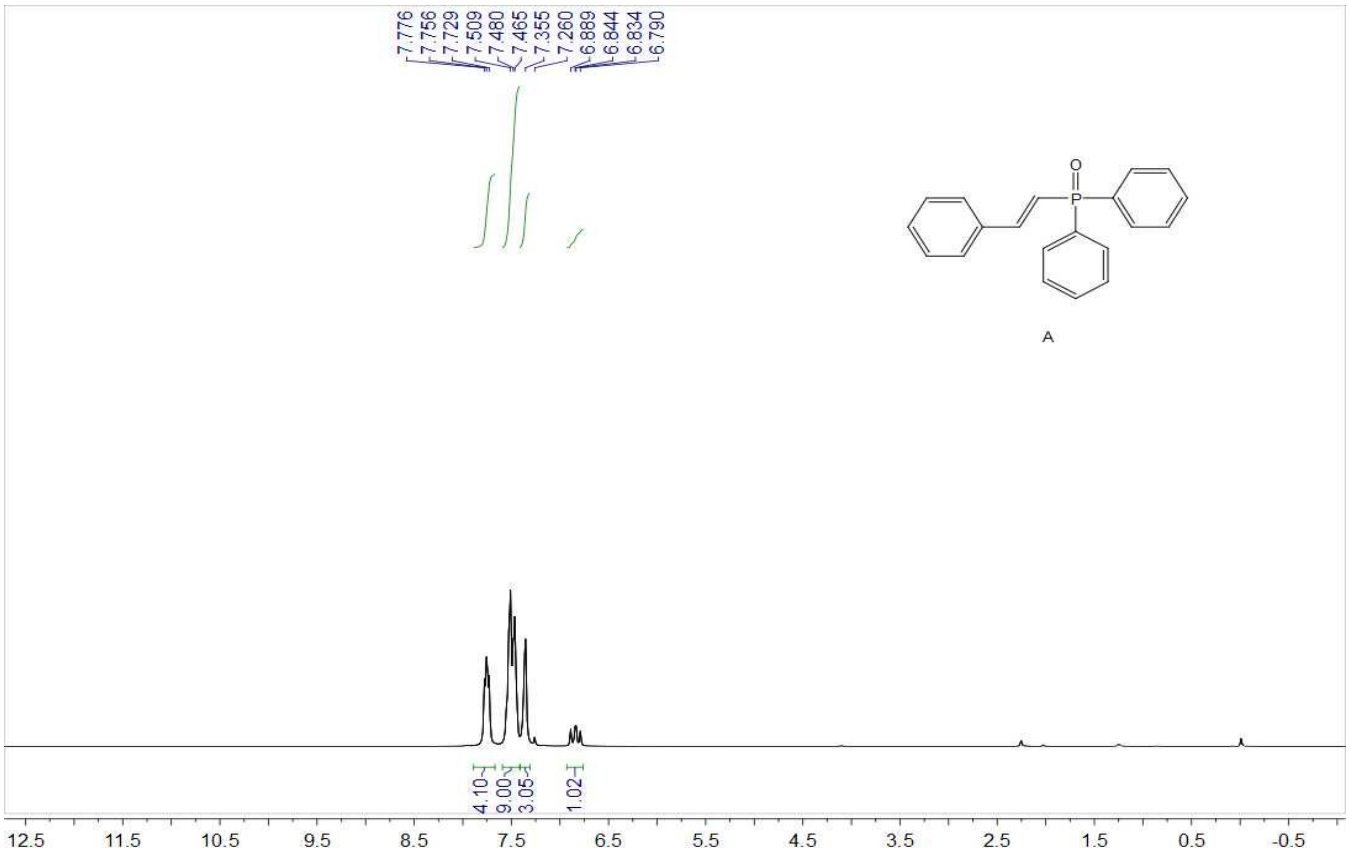
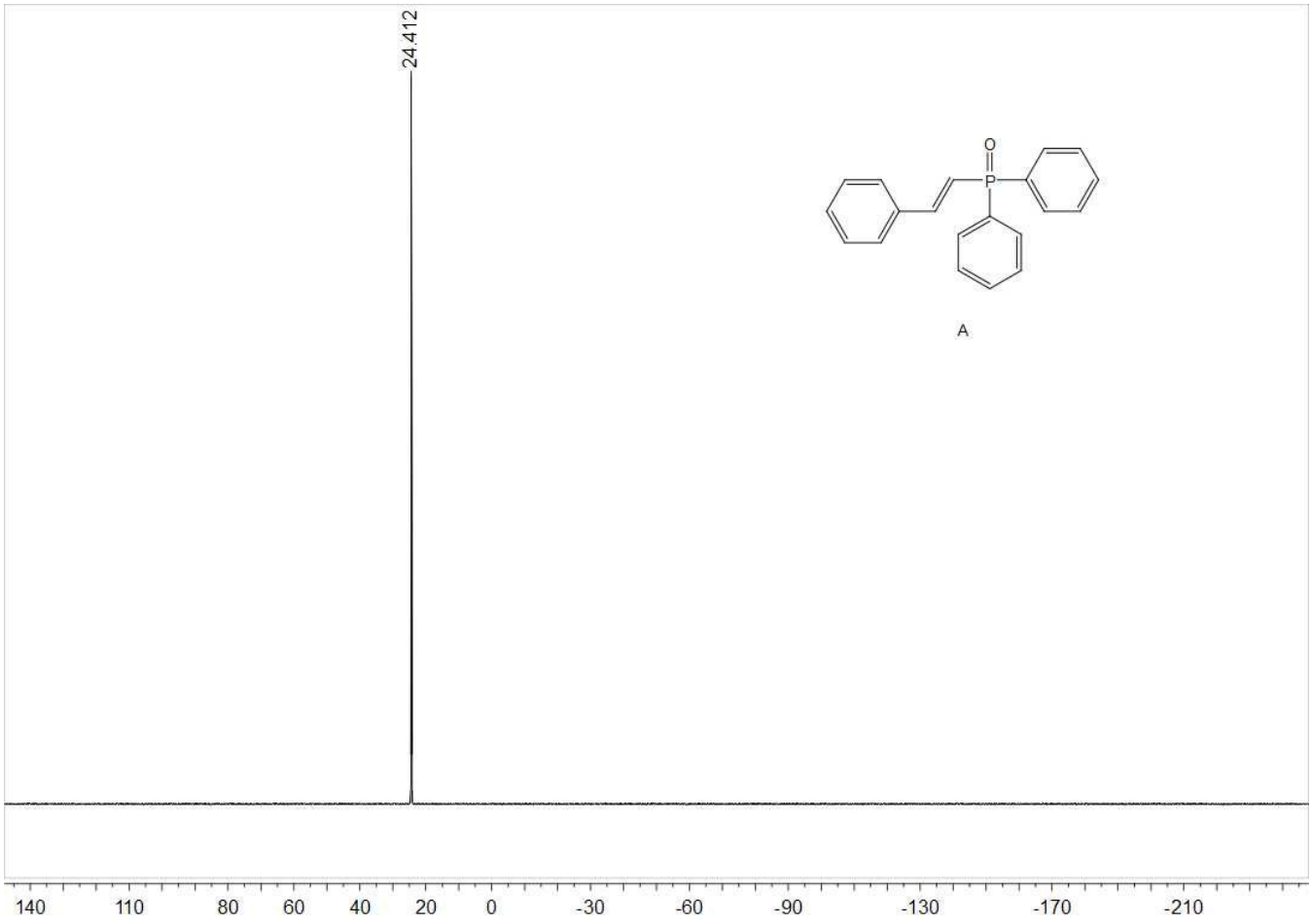
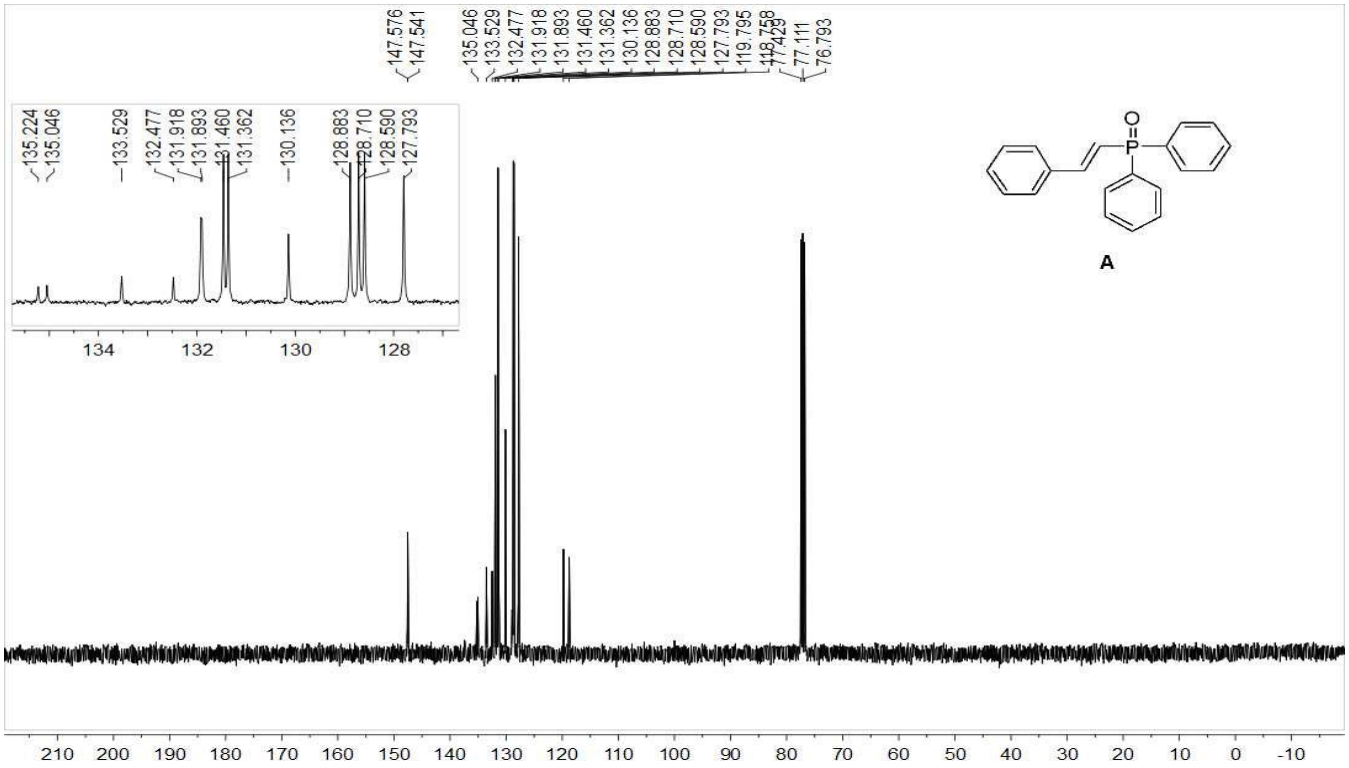
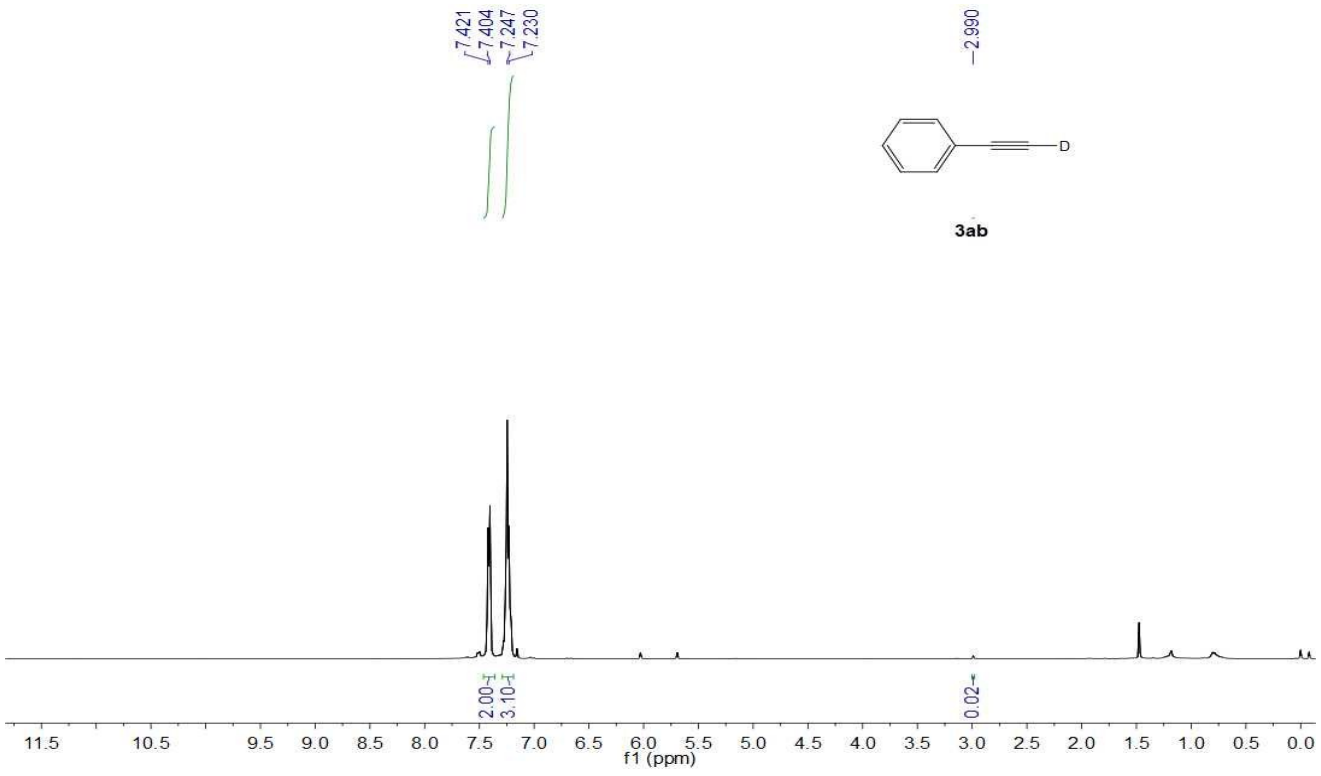
128.065
126.423
77.362
77.108
76.853
29.973
29.476
28.064
27.746
27.721
27.547
24.312
24.198
23.820
23.790
13.617
f1 (ppm)

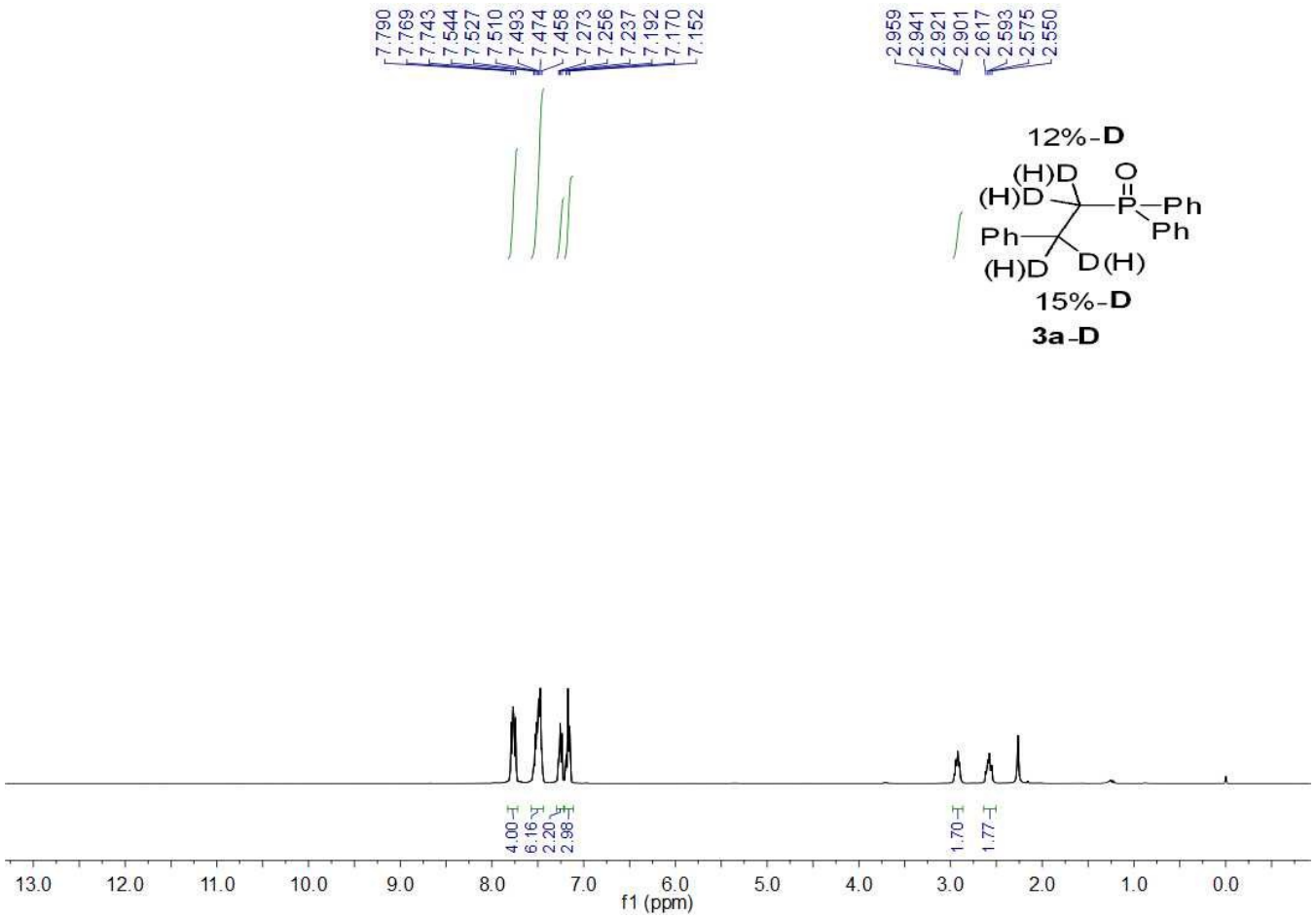
3z

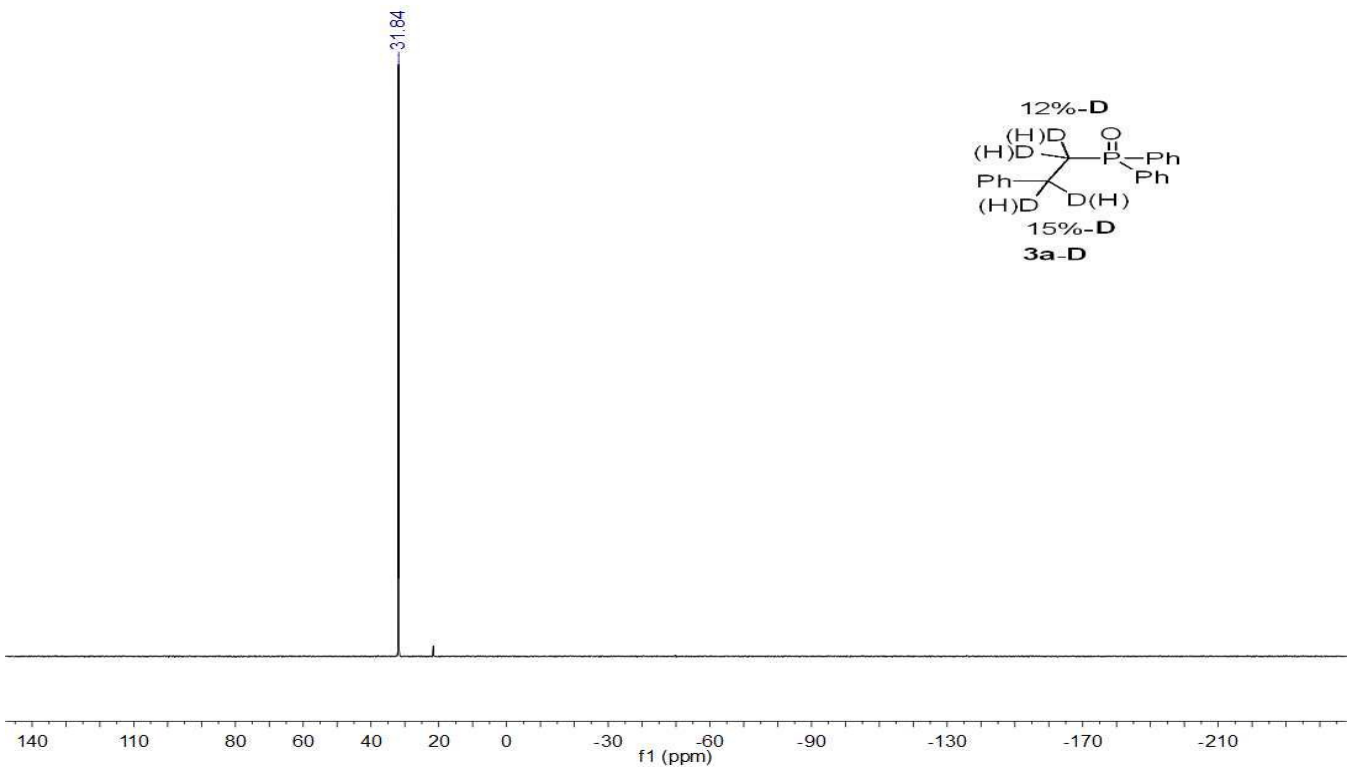

7.776
7.756
7.729
7.509
7.480
7.465
7.355
7.260
6.889
6.844
6.834
6.790
O
P
A
4.10
9.00
3.05
1.02
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5

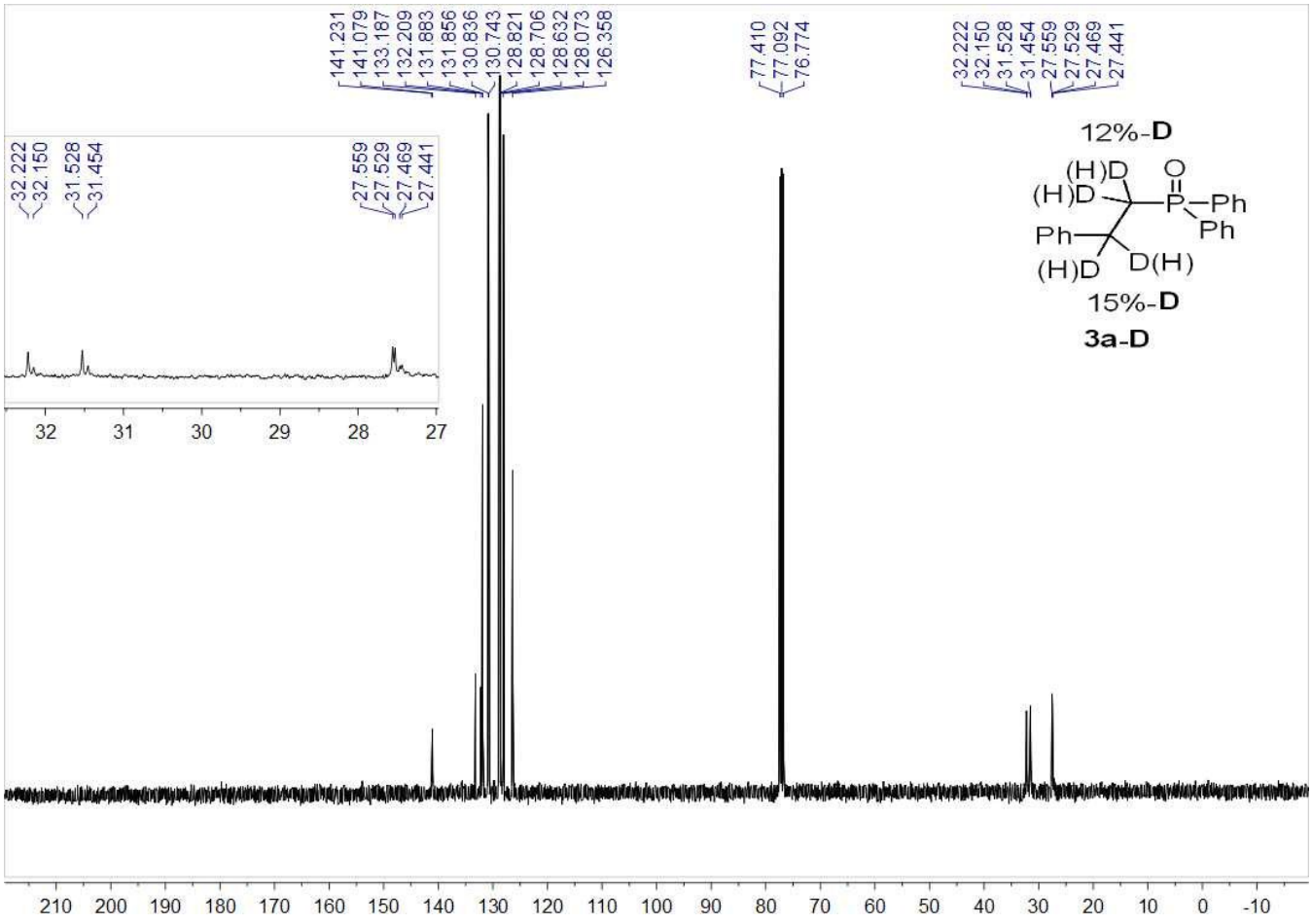

24.412
O
P
A
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210


147.576
147.541
135.046
133.529
132.477
131.918
131.893
131.460
131.362
130.136
128.883
128.710
128.590
127.793
119.795
118.758
77.111
76.793
135.224
135.046
133.529
132.477
131.918
131.893
131.460
131.362
130.136
128.883
128.710
128.590
127.793
134
132
130
128
O
P
A
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

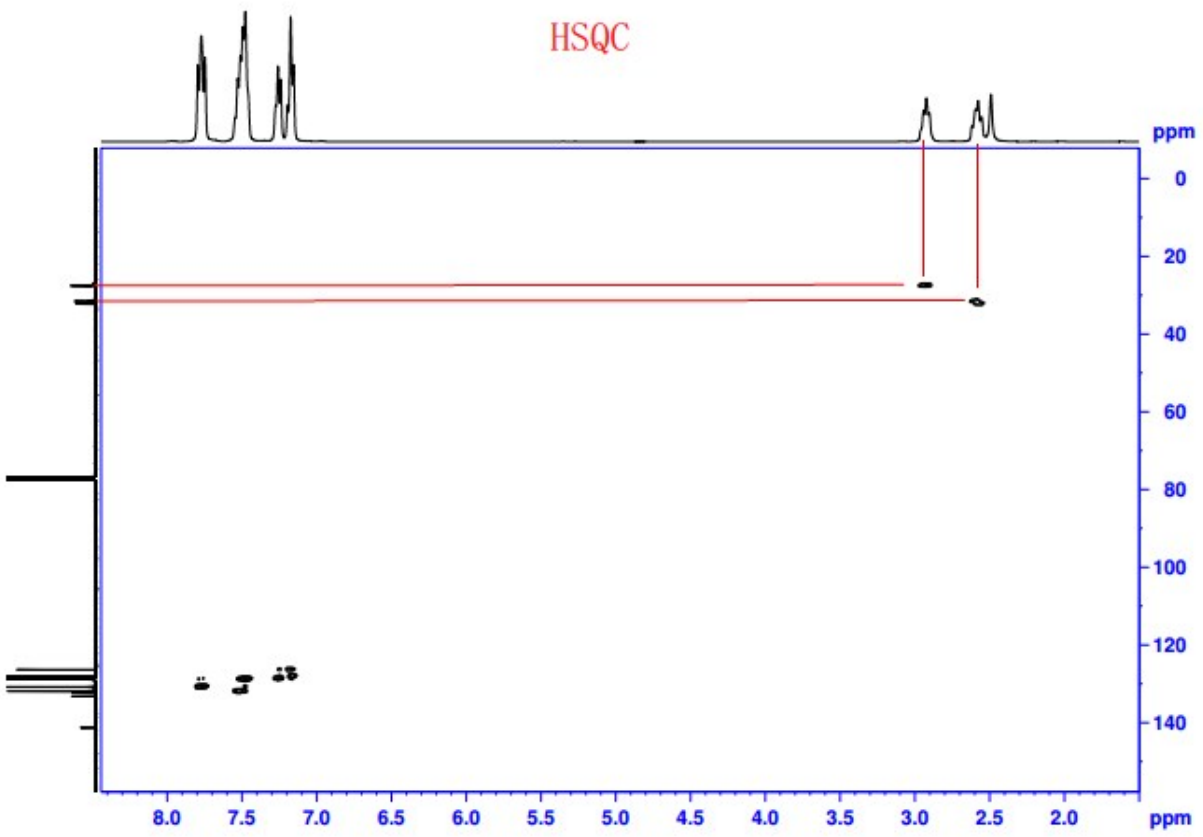

7.421
7.404
7.247
7.230
2.990
D
3ab
2.00
3.10
0.02
f1 (ppm)


7.790
7.769
7.743
7.544
7.527
7.510
7.493
7.474
7.458
7.273
7.256
7.237
7.192
7.170
7.152
2.959
2.941
2.921
2.901
2.617
2.593
2.575
2.550
12%-D
(H)D
(H)D
O
P
Ph
Ph
Ph
(H)D
D(H)
15%-D
3a-D
4.00
6.16
2.20
2.98
1.70
1.77
f1 (ppm)

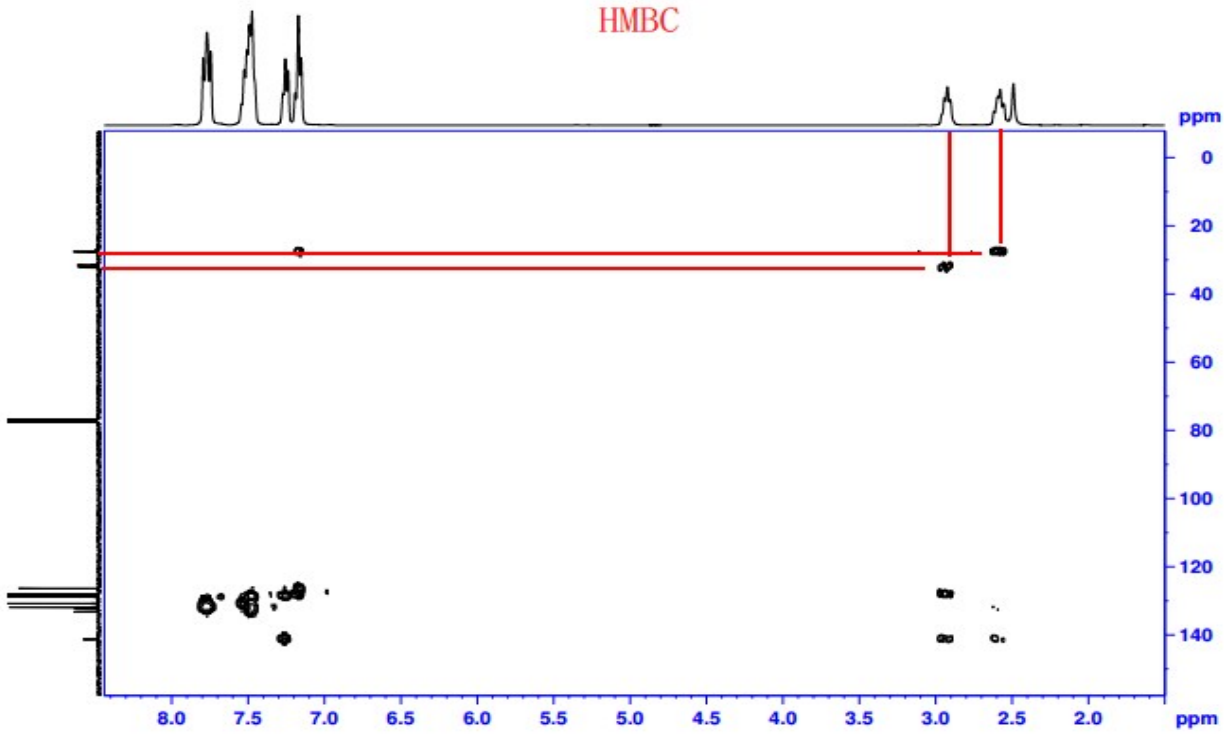

31.84
12%-D
(H)D
(H)D
O
P
Ph
Ph
Ph
(H)D
D(H)
15%-D
3a-D
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210
f1 (ppm)

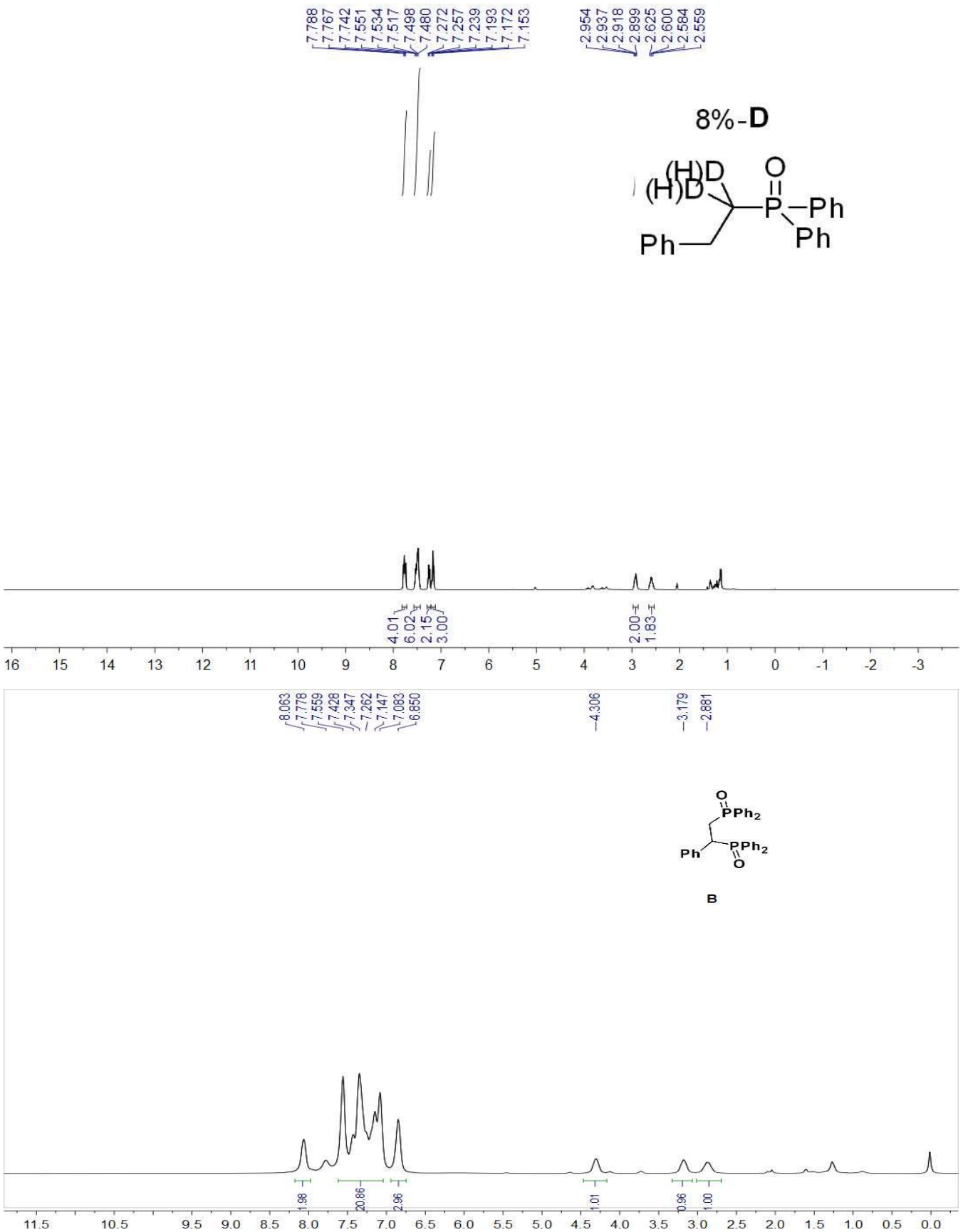

141.231
141.079
133.187
132.209
131.883
131.856
130.836
130.743
128.821
128.706
128.632
128.073
126.358
77.410
77.092
76.774
32.222
32.150
31.528
31.454
27.559
27.529
27.469
27.441
12%-D
(H)D
(H)D
O
P
Ph
Ph
Ph
(H)D
D(H)
15%-D
3a-D
32
31
30
29
28
27
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

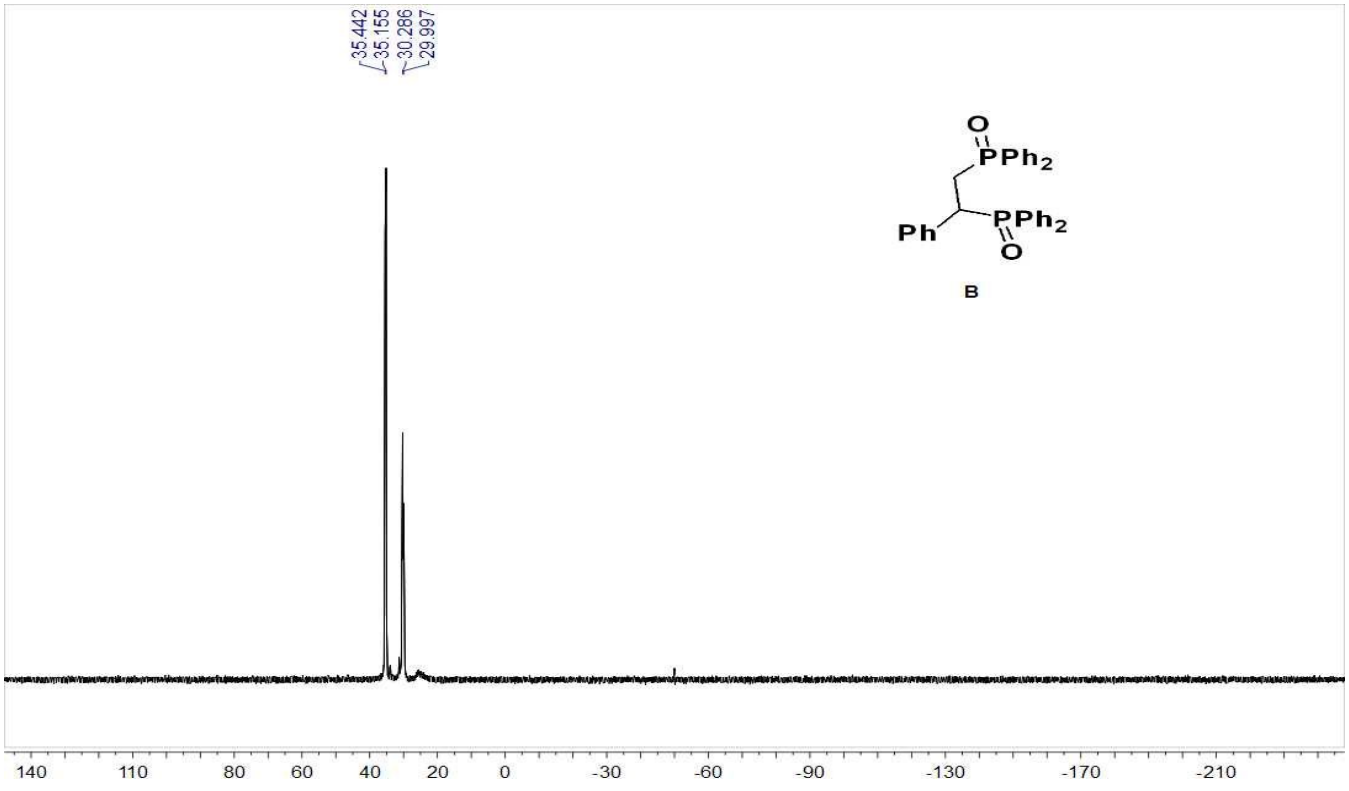
2305

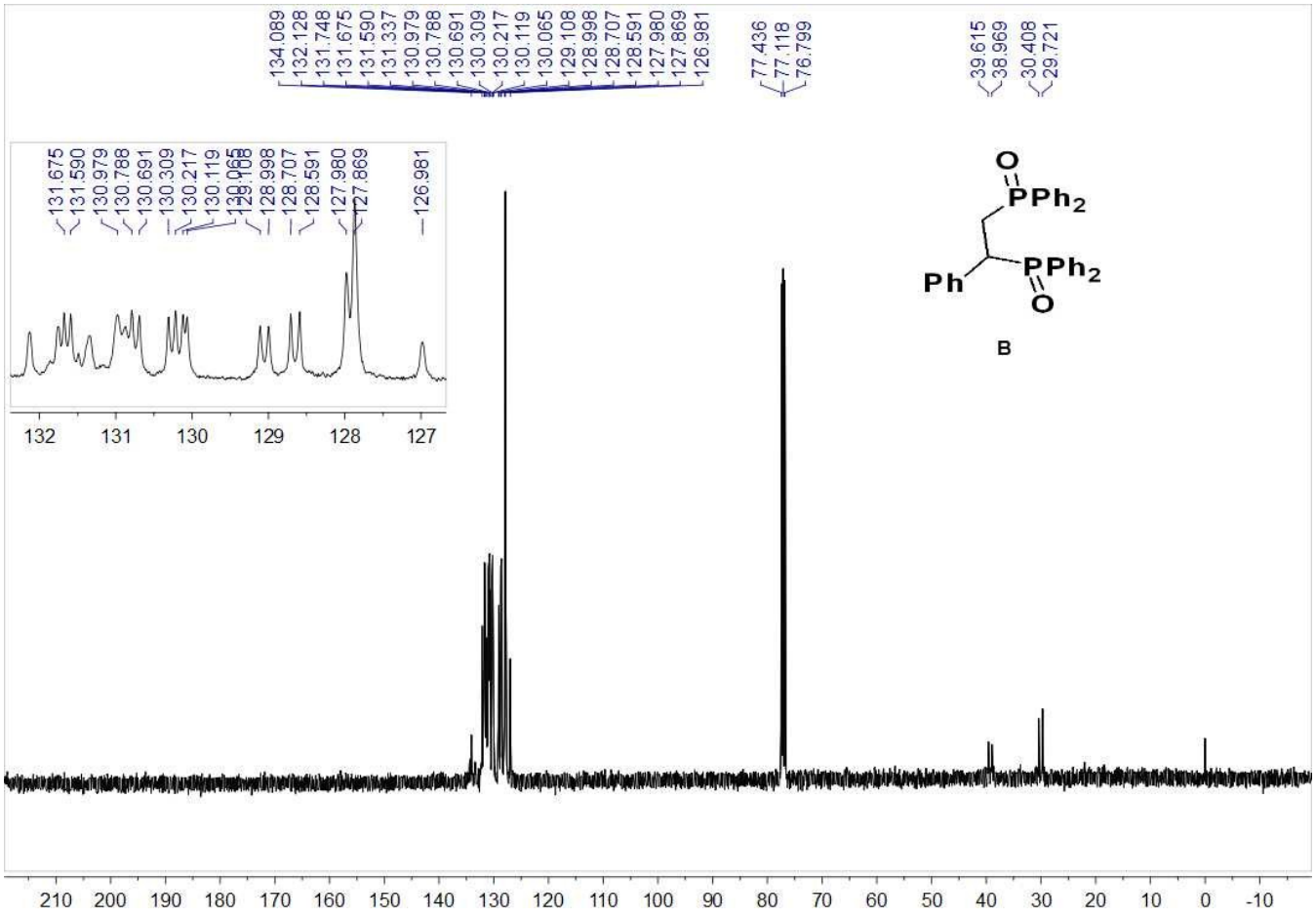

HSQC
ppm
0
20
40
60
80
100
120
140
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm

2305


HMBC
ppm
0
20
40
60
80
100
120
140
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm


7.788
7.767
7.742
7.551
7.534
7.517
7.498
7.480
7.272
7.257
7.239
7.193
7.172
7.153
2.954
2.937
2.918
2.899
2.625
2.600
2.584
2.559
8%-D
(H)D
(H)D
O
P
Ph
Ph
Ph
4.01
6.02
2.15
3.00
2.00
1.83
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
8.063
7.778
7.559
7.428
7.347
7.262
7.147
7.083
6.850
4.306
3.179
2.881
PPh2
Ph
PPh2
O
O
B
1.98
20.86
2.96
1.01
0.96
1.00
11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0


35.442
35.155
30.286
29.997
O
PPh2
Ph
PPh2
O
B
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210


134.089
132.128
131.748
131.675
131.590
131.337
130.979
130.788
130.691
130.309
130.217
130.119
130.065
129.108
128.998
128.707
128.591
127.980
127.869
126.981
77.436
77.118
76.799
39.615
38.969
30.408
29.721
O
PPh2
Ph
PPh2
O
B
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
132 131 130 129 128 127