Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

$P(O)R_2$ Directed Pd(II)-Catalyzed $C(sp^2)$-H Acylation

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Supporting Information

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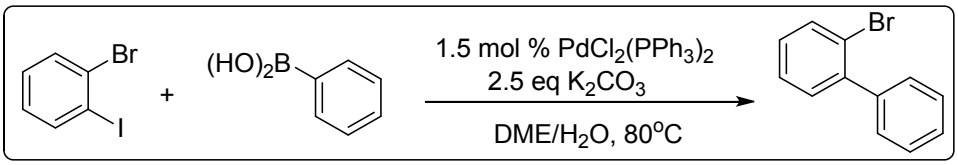
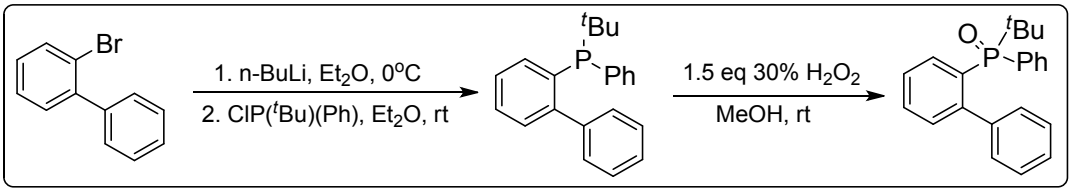
Ⅰ. General Methods and Materials

^{1}H and ^{13}C NMR spectra were recorded on a Bruker advance Ⅲ 400 spectrometer (400 MHz for ^{1}H and 100 MHz for ^{13}C) in $CDCl_3$ with TMS as internal standard. Chemical shifts (δ) were measured in ppm relative to TMS δ = 0 for ^{1}H, or to chloroform δ = 77.0 for ^{13}C as internal standard. ^{31}P NMR spectra and ^{19}F NMR were recorded on the same instrument. Data are reported as follows: Chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), Coupling constants, *J*, are reported in hertz. Mass data were measured with Thermo Scientific DSQ II mass spectrometer. IR spectra were recorded on a FT-IR spectrometer and only major peaks are reported in cm^{-1}. The starting materials were purchased from Aldrich, Acros Organics, J&K Chemicals Adamas-beta or TCI and used without further purification. Solvents were dried and purified according to the procedure from "Purification of Laboratory Chemicals book". Thin-layer chromatography (TLC) was performed using 60 mesh silica gel plates visualized with short-wavelength UV light (254 nm).

Ⅱ. Typical Procedures for the Synthesis of Substrates[1]



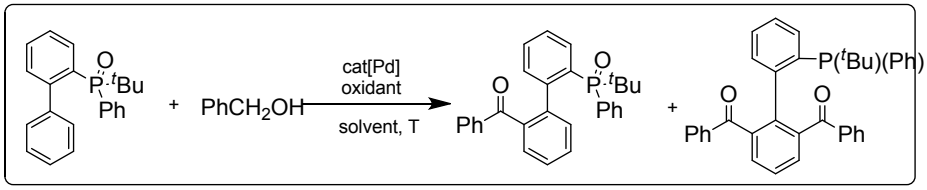
Water (4.0 mL) and DME (30.0 mL) were poured into a round-bottomed flask, fitted with a condenser and argon flow, and bubbled through with argon. Potassium carbonate (3.45 g, 25 mmol), 1-bromo-2-iodobenzene (2.8 g, 10.0 mmol), substituted phenylboronic acid (10.5 mmol), and bis(triphenylphosphine)palladium(II) chloride (105 mg, 0.15 mmol) were added to the mixture, which was stirred at 80 °C for 5 h in an oil bath until substrate disappeared as judged by TLC. The reaction mixture was allowed to cool to r.t., DME was evaporated, and water (40.0 mL) and ether (20.0 mL) were added. The layers were separated and the aqueous layer was extracted with diethylether (3 x 20.0 mL). The combined organic layers were washed with brine, dried over magnesium sulfate, filtered, and evaporated in vacuo to obtain a yellow oil, which was purified further using column chromatography on silica gel (eluent: heptane 30% EtOAc in heptane). The title compound was isolated as a white amorphous solid (2.10 g, 90%).



5.6 mL (14.0 mmol) of n-BuLi in n-hexane (2.50 M) were added dropwise to a suspension of

(11.5 mmol) of 2-bromo-1,1'-biphenyl in 24 mL of diethyl ether at 0 °C. The resulting beige-colored suspension was stirred for an additional 2 h at 0 °C. Then, ClP(*t*-Bu)(Ph) (2.0 g,10.0 mmol) was added dropwise in freshly distilled diethyl ether (20.0 mL). The mixture was then stirred at r.t. for 2 h, filtered and solvent was removed in vacuo to yield a residue, which was used without further purification. To the residue in MeOH (36.0 mL) was added dropwise at < 40 °C 30 % aq. H_2O_2 solution (1.7 mL, 15.0 mmol). The resulting clear solution was stirred at r.t. for 1 h, treated with sat. Na_2SO_3 solution (2.0 mL), and the mixture was concentrated at the rotavapor to remove the MeOH. The aqueous layer was extracted with CH_2C1_2 (3 × 20 mL). The extract was washed with brine and dried over $MgSO_4$. The organic layers were dried over Na_2SO_4, filtered and solvent was evaporated under reduced pressure. The desired product was obtained after purification by flash chromatography on silica gel.

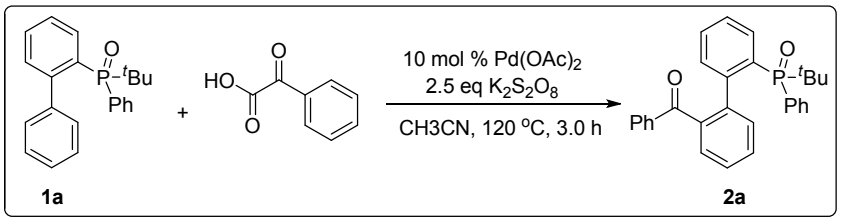
Ⅲ. Optimization of the reaction conditions for the palladium-catalyzed C(sp2)-H acylation with alcohols[a]



Entry	Cat	Oxidant	Solvent	Yield (%)[b]	Ratio[c]
1	$Pd(OAc)_2$	$K_2S_2O_8$	CH_3CN	trace	
2	$Pd(OAc)_2$	TBHP	CH_3CN	5	
3	$Pd(OAc)_2$	Ag_2O	CH_3CN	n.r.	
4	$Pd(OAc)_2$	$(NH_4)_2S_2O_8$	CH_3CN	trace	
5	$Pd(OAc)_2$	TBHP	DCE	58	10 : 1
6	$Pd(OAc)_2$	TBHP	PhCl	48	14:1
7	$Pd(OAc)_2$	TBHP	DME	n.r.	
8	$Pd(TFA)_2$	TBHP	DCE	70	10 : 1
9	$PdCl_2$	TBHP	DCE	trace	
10	$Pd(CH_3CN)_2Cl_2$	TBHP	DCE	trace	
11[d]	$Pd(TFA)_2$	TBHP	DCE	76	10 :1
12[e]	$Pd(TFA)_2$	TBHP	DCE	80	10 : 1

[a] Reaction conditions: **1a** (0.3 mmol), benzyl alcohol (0.6 mmol), Pd catalyst (10 mol %), oxidant (0.75 mmol), solvent (1.5 mL), 100 ℃ for 16 h under air atmosphere unless otherwise noted. [b] Isolated yield. [c] Ratio of **2a** : **3a**. [d] 80 ℃. [e] 60 ℃.

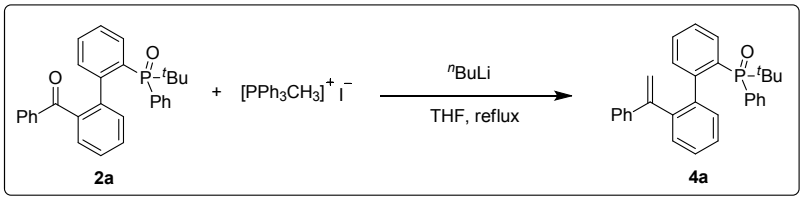
Ⅳ. General procedures for the palladium-catalyzed C(sp2)-H acylation.



Under air atmosphere, 2-(*tert*-butylphenylhosphoryl)biphenyl **1a** (100.2 mg, 0.30 mmol, 1.0 equiv), $Pd(OAc)_2$ **(**6.7 mg, 0.03 mmol, 10 mol %), $K_2S_2O_8$ (202.5 mg, 0.75 mmol, 2.5 equiv), and benzoyl formic acid **2a** (90 mg, 0.6 mmol, 2.0 equiv) were added to a sealed tube containing a magnetic stir bar. After which, 3.0 mL CH_3CN was added with a syringe. Then the mixture was stirred at 130 ℃ in an oil bath for 3.0 hours. After cooling to room temperature, the solution was removed in vacuo to yield a residue, which was purified by silica gel to afford pure **3a** as oil (101.1 mg, 72%).

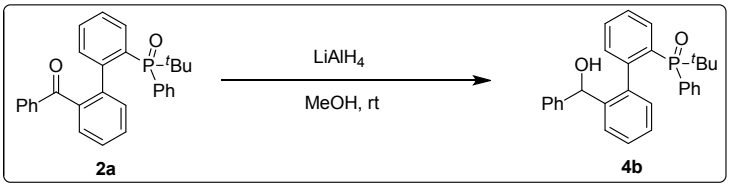
Under air atmosphere, 2-(tert-butylphenylhosphoryl)biphenyl **1a** (100.2 mg, 0.30 mmol, 1.0 equiv) and $Pd(TFA)_2$ (9.9 mg, 0.03 mmol, 10 mol %) were added to a tube containing a magnetic stir bar. After which, 1.5 mL DCE was added using a syringe. Then 70 % aq. TBHP solution (165uL, 1.20 mmol, 4.0 equiv) and benzyl alcohol (78 uL, 0.75 mmol, 2.5 eq) were added with microsyringes. The reaction mixture was stirred at 60 ℃ in an oil bath for 16 hours until substrate disappeared as judged by TLC. After cooling to room temperature, the solution was removed in vacuo to yield a residue, which was purified by silica gel to afford pure 2a and 3a
(80% 2a : 3a = 10:1).

Ⅴ. General procedures for the transformations of acylated products

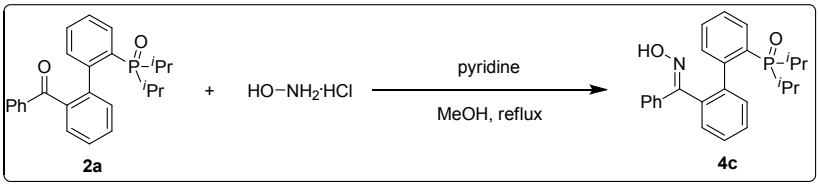


Under Ar atmosphere, 0.1 mL (0.25 mmol) of n-BuLi in n-hexane (2.50 M) were added dropwise to a suspension of triphenylmethyphosphonium iodide (0.3 mmol) in 2 mL of THF at 0

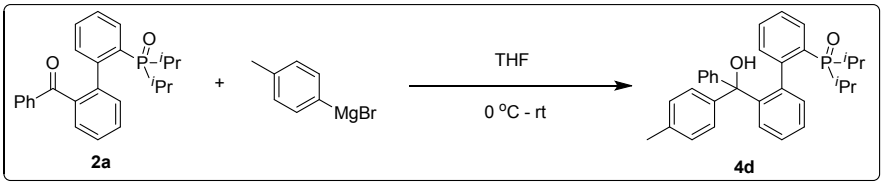
°C. The resulting suspension was stirred for an additional 2 h at rt. Then, 87.2 mg (0.2 mmol) of (2'-(t*ert*-butyl(phenyl)phosphoryl)biphenyl-2-yl)(phenyl)methanone was added dropwise in freshly distilled THF (1.0 mL). The mixture was then stirred for 10 h under reflux. The desired product was obtained after purification by flash chromatography on silica gel (white solid, 71 mg, 81%).[2]



87.2 mg (0.2 mmol) of (2'-(t*ert*-butyl(phenyl)phosphoryl)biphenyl-2-yl)(phenyl)methanone was dissolved in 2.0 mL MeOH. Then 0.24 mmol of $LiAlH_4$ was added slowly at 0 ℃. After 10 minutes, the reaction was completed. The desired product was obtained after purification by flash chromatography on silica gel (white solid, 45 mg, 51 %).



(2'-(diisopropylphosphoryl)biphenyl-2-yl)(phenyl)methanone (0.2 mmol) and hydroxylamine hydrochloride (0.8 mmol) were dissolved in 2 ml MeOH. Pyridine (1.0 mmol) was added *via* syringe and after stirring at room temperature overnight the solvent was evaporated. The product was obtained after purification by flash chromatography on silica gel (white solid, 58 mg, 71%).[3]



(2'-(diisopropylphosphoryl)biphenyl-2-yl)(phenyl)methanone (0.2 mmol) was dissolved in 2 ml THF, p-tolylmagnesium bromide (3.0 mmol, 1.5 equiv) was added slowly *via* syringe at 0 ℃ and after stirring at room temperature for 3 hours the solvent was evaporated. The product was obtained after purification by flash chromatography on silica gel (white solid, 59 mg, 61%)

Ⅵ. References

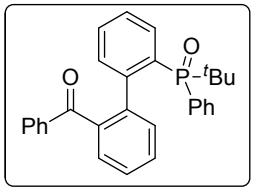
[1] H. L. Wang, R. B. Hu, H. Zhang, A. X. Zhou and S.-D.Yang, *Org. Lett*. 2013, **15**, 5302.

[2] X. Wang, Y. F. Chen, L. F. Niu and P. F. Xu, *Org. Lett.*, 2009, **11**, 3310.

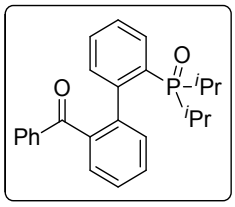
[3] S. B. Liu, Y. Yu and L. S. Liebeskind, *Org. Lett.*, 2007, **9**, 1947.

Ⅶ. Characterization of the products

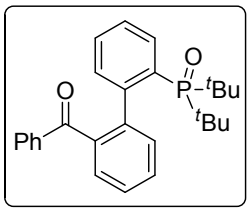
Note: when the directed group is -P(O)(tBu)(Ph), the products have two chiral centers, which determine they are a mixture of four diastereoisomers.



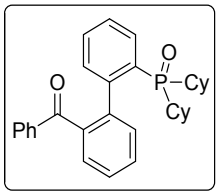
colorless oil; **^{31}P NMR** (162 MHz, $CDCl_{13}$) δ: 40.98, 39.67; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.98 (d, *J* = 7.3 Hz, 0.78H), 7.90-7.80 (m, 1.0H), 7.74-7.72 (m,1.0H), 7.64-7.60 (m, 1.0H), 7.57-7.53 (t, *J* = 7.2 Hz, 1.0H), 7.51-7.33 (m, 7.3H), 7.30-7.23 (m, 3.3H), 7.17-7.10 (m, 1.0H), 7.07-7.03 (m, 1.0H), 6.64 (d, *J* = 7.6 Hz, 0.4H), 1.23 (d, *J* = 14.5 Hz, 5.0H), 1.15 (d, *J* = 14.5 Hz, 4.0H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 198.22, 196.59, 146.77, 146.72, 146.22, 146.17, 141.76, 141.72, 140.56, 140.53, 138.20, 138.05, 137.87, 137.11, 134.05, 133.97, 133.26, 133.16, 133.11, 132.18, 132.05, 131.99, 131.94, 131.86, 131.78, 131.74, 131.00, 130.93, 130.89, 130.82, 130.79, 130.63, 130.49, 130.40, 130.37, 130.23, 130.21, 130.15, 130.12, 129.32, 129.24, 128.63, 128.52, 127.93, 127.82, 127.78, 127.64, 127.33, 127.22, 126.45, 126.11, 126.06, 125.94, 125.88, 125.77, 35.12 (d, *J* = 16.0 Hz), 34.42 (d, *J* = 16.0 Hz), 26.01, 25.92; **MS (ESI)**: found $[M+H]^+$ 439.4



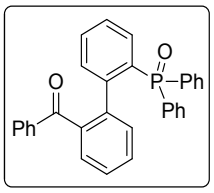
colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 52.17; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.97-7.95 (m, 2H), 7.53-7.45 (m, 3H), 7.43-7.35 (m, 5H), 7.33 (s, 1H), 7.32-7.27 (m, 2H), 2.20-2.00 (m, 2H), 1.05-0.99 (m, 9H), 0.58 (dd, *J* = 15.4 Hz, *J* = 7.2 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 197.04, 147.24 (d, *J* = 4.3Hz), 141.01 (d, *J* = 3.1 Hz), 138.28, 138.11, 133.22, 133.13, 132.00, 131.00, 130.61, 129.96 (d, *J* = 10.8 Hz), 129.85 (d, *J* = 2.5 Hz), 129.27 (d, *J* = 7.4 Hz), 127.59, 126.92 (d, *J* = 82.1 Hz), 126.26 (d, *J* = 11.1 Hz), 125.98, 27.96 (d, *J* = 65.1 Hz), 25.60 (d, *J* = 67.0 Hz), 16.72 (d, *J* = 1.4 Hz), 15.67 (d, *J* = 3.0 Hz), 15.60 (d, *J* = 3.2 Hz), 14.91 (d, *J* = 2.6 Hz); **MS (ESI)**: found $[M+H]^+$ 391.3



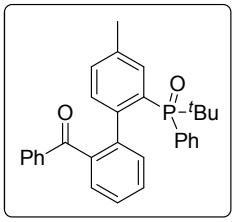
colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 54.54; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.00-7.98 (m, 2H), 7.55-7.29 (m, 10H), 7.24 (d, *J* = 7.6 Hz, 1H), 1.18 (d, *J* = 13.3 Hz, 9H), 0.98 (d, *J* = 13.4 Hz, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 197.46, 147.48 (d, *J* = 3.5 Hz）， 142.93 (d, *J* = 2.7 Hz), 138.47, 137.79, 134.33, 134.24 132.10, 131.16, 130.68 (d, *J* = 11.4 Hz), 130.44, 129.36 (d, *J* = 2.6 Hz), 129.19 (d, *J* = 4.7 Hz), 127.65, 127.50 (d, *J* = 67.7 Hz), 125.74, 125.32 (d, *J* = 11.1 Hz), 37.12 (d, *J* = 40.7 Hz), 36.54 (d, *J* = 39.8 Hz), 27.90, 26.91; **MS (ESI)**: found $[M+H]^+$ 419.2



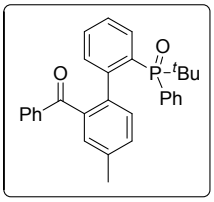
colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 47.51; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.98 (d, *J* = 7.6 Hz, 2H), 7.51-7.45 (m, 3H), 7.42-7.34 (m, 6H), 7.32-7.26 (m, 2H), 1.89-1.60 (m, 9H), 1.46-1.43 (m, 1H), 1.30-1.04 (m, 9H), 0.92-0.86 (m, 1H), 0.78-0.71 (m, 1H), 0.61-0.56 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 196.92, 147.06 (d, *J* = 4.4 Hz), 142.04 (d, *J* = 2.8 Hz), 137.99, 137.94, 133.14 (d, *J* = 9.0 Hz), 132.07, 131.25, 130.42, 129.78 (d, *J* = 11.0 Hz), 129.55 (d, *J* = 2.4 Hz), 129.20, 128.86, 127.52, 126.72, 126.22 (d, *J* = 2.1 Hz), 125.80, 37.96 (d, *J* = 65.2 Hz), 35.56 (d, *J* = 67.1 Hz), 26.51, 26.40 (d, *J* = 3.1 Hz), 26.29, 26.17, 26.05, 25.69, 25.57, 25.29 (d, *J* = 3.0 Hz), 24.63 (d, *J* = 2.5 Hz), 24.54 (d, *J* =3.1 Hz); **MS (ESI)**: found $[M+H]^+$ 471.2



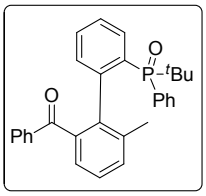
colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 28.55; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.85-7.83 (m, 2H), 7.75-7.71 (m, 2H), 7.60-7.7.37 (m, 11H), 7.33-7.29 (t, J = 6.9 Hz, 6H), 7.28-7.14 (m, 7H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 197.10, 145.51 (d, *J* = 7.7 Hz), 140.46 (d, *J* = 3.8 Hz), 138.17, 137.27, 134.45, 134.10 (d, *J* = 12.3 Hz), 133.39 (d, *J* = 8.0 Hz), 132.36 (d, *J* = 12.6 Hz), 132.17 (d, *J* = 9.7 Hz), 131.89, 131.79 (d, *J* = 3.1 Hz), 131.67, 131.34 (d, *J* = 2.3 Hz), 131.26, 131.01, 130.98, 130.41, 130.24, 129.59 (d, *J* = 10.0 Hz), 128.27 (d, *J* = 11.8 Hz), 128.10, 127.72 (d, *J* = 12.1 Hz), 126.69, 126.57 (d, *J* = 12.5 Hz); **MS (ESI)**: found $[M+H]^+$ 459.4



colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 40.88, 39.55; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.99 (d, *J* = 7.2 Hz, 0.8H), 7.73 (d, *J* = 7.2 Hz, 1.2H), 7.68-7.62 (m, 1.8H), 7.55-7.34 (m, 6.0H), 7.28-7.21 (m, 4.2H), 7.17-7.09 (m, 1.6H), 7.07-7.02 (m, 1.2H), 6.64 (d, *J* = 8.0 Hz, 0.4H), 2.40 (s, 3.0H), 1.26- 1.13 (m, 9.0H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 198.34, 196.71, 143.73, 143.68, 143.26, 143.21, 141.73, 141.70, 140.54, 140.51, 138.29, 138.01, 137.85, 137.13, 135.58, 135.47, 135.39, 135.28, 134.02, 133.93, 133.84, 133.14, 133.09, 132.99, 132.61, 132.50, 132.19, 132.03, 131.90, 131.86, 131.78, 131.75, 131.66, 131.42, 131.31, 131.17, 131.14, 131.09, 131.01, 130.96, 130.94, 130.78, 130.76, 130.69, 130.53, 130.33, 130.30, 129.54, 129.25, 129.13, 128.66, 128.49, 128.45, 127.90, 127.80, 127.64, 127.58, 127.28, 127.17, 126.29, 125.93, 35.22, 35.04, 34.52, 34.34, 26.07, 25.97, 21.30; **MS (ESI)**: found $[M+H]^+$ 453.2

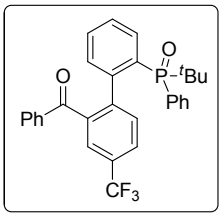


colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 40.67, 39.39; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.99 (d, *J* = 7.2 Hz, 0.8H), 7.88-7.78 (m, 1.0H), 7.73 (d, *J* = 7.0 Hz, 1.0H), 7.66-7.62 (m, 1.0Hz), 7.53-7.17 (m, 10.4H), 7.05-7.04 (m, 1.6H), 6.94 (d, *J* = 7.2 Hz, 0.5H), 7.53 (d, *J* = 7.6 Hz, 0.4H), 2.32-2.31 (d, 3.0 H), 1.25-1.14 (m, 9.0 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 198.69, 196.84, 146.84, 146.79, 146.23, 138.88, 138.23, 138.06, 137.97, 137.60, 137.12, 136.15, 135.70, 134.34, 134.25, 134.13, 133.45, 133.35, 133.25, 132.17, 132.09, 131.92, 131.83, 131.77, 131.68, 131.55, 131.17, 130.95, 130.84, 130.64, 130.49, 130.32, 130.19, 130.06, 129.81, 129.33, 129.10, 128.97, 128.77, 127.95, 127.84, 127.64, 127.48, 127.29, 129.18, 125.92, 125.76, 125.64, 35.18 (d, J =19 Hz), 34.48 (d, J = 19 Hz), 26.09, 25.98, 21.08, 21.02; **MS (ESI)**: found $[M+H]^+$ 453.2

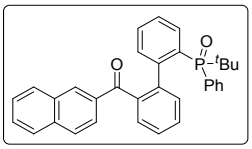


colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 38.57; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.98-7.97 (m, 2H), 7.92-7.88 (m, 1H), 7.70-7.66 (m, 2H), 7.58-7.54 (m, 1H), 7.51-7.36 (m, 8H), 7.24-7.17 (m,

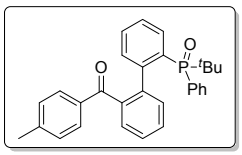
3H), 1.44 (s, 3H), 1.01 (d, J = 14.6 Hz, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 197.85, 145.69 (d, J = 4.8 Hz), 141.10 (d, J = 2.8 Hz), 138.72, 137.64, 136.49, 134.49, 134.39, 132.63, 132.12, 131.88, 131.80, 131.58, 131.04, 130.83, 130.80, 130.74, 130.63, 130.16 (d, J= 2.5 Hz), 127.94, 127.83, 127.69, 126.84, 126.13, 126.01, 125.84; **MS (ESI)**: found $[M+H]^+$ 453.2



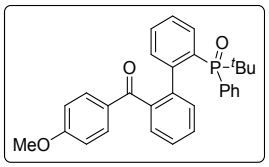
colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 41.19, 39.74; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.97 (d, J = 12.4 Hz, 0.8H), 7.93-7.88 (m, 0.5H), 7.87-7.82 (m, 0.6H), 7.72-7.60 (m, 3.6H), 7.53-7.32 (m, 7.7H), 7.29-7.24 (m, 2.0H), 7.21-7.17 (m, 0.6H), 7.08-7.04 (m, 1.0H), 6.73 (d, J = 8.0 Hz, 0.4H), 1.26-1.13 (m, 9.0H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 196.98, 195.32, 145.52, 145.37, 145.32, 144.76, 144.71, 144.24, 138.82, 137.81, 137.24, 136.94, 134.10, 134.01, 133.56, 133.18, 133.08, 132.86, 132.71, 132.17, 132.06, 131.88, 131.80, 131.74, 131.66, 131.48, 131.32, 131.24, 131.21, 130.99, 130.88, 130.80, 130.78, 130.72, 130.59, 130.51, 130.48, 130.42, 129.77, 128.95, 128.89, 128.62, 128.53, 128.29, 128.25, 128.16, 128.14, 128.02, 127.65, 127.60, 127.49, 126.83, 126.71, 126.64, 126.52, 125.96, 125.92, 125.59, 125.33, 125.12, 125.07, 125.03, 122.42, 35.29, 35.16, 34.59, 34.47, 25.98, 25.87; **^{19}F NMR** (376 Hz, $CDCl_3$) δ: -62.39, -62.50; **MS (ESI)**: found $[M+H]^+$ 507.2



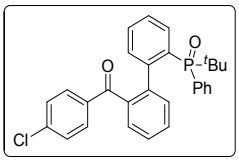
colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 40.65, 39.81; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.65 (s, 0.4H), 8.27 (s, 0.5H), 8.03-7.98 (m, 0.8H), 7.87-7.76 (m, 3.4H), 7.69-7.63 (m, 1.4H), 7.60-7.25 (m, 10.5H), 7.15 (t, J = 7.2 Hz 0.5H), 6.94-6.90 (m, 0.6H), 6.82-6.78 (m, 1.0H), 6.67 (d, J =8.0 Hz, 0.4H), 1.24- 1.02 (m, 9.0H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 198.74, 196.63, 146.81, 146.75, 146.07 146.02, 141.91, 141.88, 140.30, 140.26, 138.70, 137.42, 135.27, 135.23, 135.10, 134.97, 134.27, 134.18, 134.10, 133.54, 133.44, 133.33, 133.25, 132.29, 132.04, 131.95, 131.85, 131.74, 131.72, 131.66, 131.64, 131.55, 131.48, 131.02, 130.91, 130.87, 130.66, 130.25, 130.20, 130.17, 129.74, 129.68, 129.37, 129.07, 128.86, 128.54, 128.44, 128.28, 128.10, 128.05, 128.01, 127.94, 127.66, 127.58, 127.51, 127.44, 127.40, 127.16, 127.05, 126.74, 126.25, 126.21, 126.10, 126.00, 125.97, 125.86, 125.65, 125.14, 35.16, 34.47, 25.97, 25.91; **MS (ESI)**: found $[M+H]^+$ 489.2



colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 40.91, 39.71; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.89-7.82 (m, 1.7H), 7.63 (d, *J* = 8.0 Hz, 2.0H), 7.57-7.02 (m, 12.7H), 6.67 (d, *J* = 7.6Hz, 0.4H), 2.35-2.32 (m, 3.0H), 1.26-1.13 (m, 9.0H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 197.99, 196.41, 146.68, 146.63, 146.25, 146.20, 142.93, 142.76, 141.39, 141.36, 140.43, 140.40, 138.44, 137.36, 135.48, 135.23, 134.07, 133.98, 133.30, 133.21, 133.12, 132.11, 132.00, 131.91, 131.83, 131.77, 131.71, 131.69, 130.98, 130.84, 130.67, 130.32, 130.29, 130.24, 130.22, 130.14, 130.11, 129.77, 129.00, 128.93, 128.73, 128.58, 128.47, 128.40, 128.31, 127.95, 127.84, 127.28, 127.16, 126.43, 126.11, 126.04, 125.92, 125.89, 125.77, 35.24, 35.07, 34.54, 34.37, 26.02, 25.96, 21.53, 21.47; **MS (ESI)**: found $[M+H]^+$ 453.2

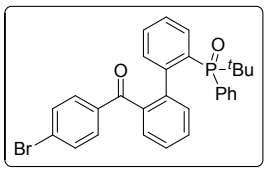


colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ:40.67, 39.41; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.02 (d, *J* = 8.8 Hz, 1.0H), 7.89-7.85 (m, 0.5H), 7.82-7.78 (m, 0.6H), 7.74 (d, *J* = 8.8 Hz, 1.0H), 7.67-7.63 (m, 1.0H), 7.54-7.46 (m, 2.2H), 7.44-7.22 (m, 7.0H), 7.19-7.15 (m, 0.6H), 7.12-7.04 (m, 1.5H), 6.83 (d, *J* = 8.8 Hz, 1.0H), 6.71 (d, *J* = 8.8 Hz, 1.0H), 6.64 (d, *J* = 7.6 Hz, 0.4H), 3.79-3.77(m, 3.0H), 1.23-1.15 (m, 9.0H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 197.23, 195.56, 163.01, 162.85, 146.66, 146.60, 146.10, 141.28, 141.25, 140.00, 139.97, 138.81, 137.78, 134.25, 134.16, 134.10, 133.48, 133.38, 133.17, 132.97, 132.12, 132.09, 132.01, 131.90, 131.81, 131.76, 131.68, 131.49, 131.19, 130.95, 130.88, 130.64, 130.37, 130.35, 130.17, 130.15, 129.84, 128.96, 128.78, 128.58, 128.50, 128.07, 128.03, 128.00, 127.89, 127.70, 127.34, 127.23, 126.56, 126.09, 125.97, 113.12, 112.93, 55.25, 55.23, 35.28, 35.12, 34.58, 34.42, 26.08, 25.97; **MS (ESI)**: found $[M+H]^+$ 469.2

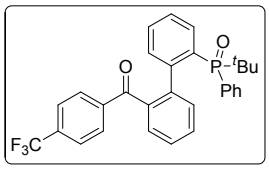


colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 40.62, 39.42; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.94 (d, *J* = 8.5 Hz, 0.8H), 7.91-7.87 (m, 0.4H), 7.77-7.72 (m, 0.6H), 7.67 (d, *J* = 8.5 Hz, 1.0H), 7.59-7.55 (m, 1.0H), 7.51-7.45 (m, 3.6H), 7.43-7.21 (m, 6.7H), 7.14-7.07 (m ,2.7H), 6.55 (d, *J* = 7.7 Hz, 0.4H), 1.22-1.14 (m, 9.0H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 197.31, 195.75, 146.84, 146.79,

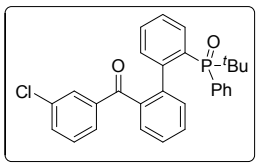
146.04, 146.00, 141.79, 141.76, 140.46, 140.43, 138.59, 138.45, 138.08, 137.31, 136.46, 135.97, 134.25, 134.16, 133.97, 133.66, 133.57, 133.10, 132.16, 132.13, 132.07, 131.96, 131.84, 131.76, 131.74, 131.65, 131.33, 131.19, 130.94, 130.91, 130.84, 130.74, 130.68, 130.66, 130.31, 130.28, 130.25, 129.82, 129.48, 128.94, 128.78, 128.63, 128.43, 128.16, 128.01, 127.92, 127.51, 127.46, 127.13, 126.67, 126.30, 126.26, 126.15, 125.99, 125.88, 35.23, 34.53, 26.05, 25.87; **MS (ESI)**: found $[M+H]^+$ 473.2,475.2



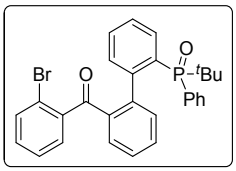
colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 41.13, 39.90; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.92-7.84 (m, 1.3H), 7.78-7.73 (m, 0.6H), 7.63-7.22 (m, 13.4H), 7.12-7.07 (m, 1.5H), 6.54 (d, *J* = 7.6 Hz, 0.4H), 1.22-1.15(m, 9.0H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 197.37, 195.85, 146.80, 146.75, 146.05, 146.00, 141.74, 141.71, 140.46, 140.42, 137.89, 137.12, 136.83, 136.28, 134.18, 134.09, 133.71, 133.62, 133.53, 132.83, 132.24, 132.03, 131.92, 131.81, 131.77, 131.62, 131.33, 131.13, 130.90, 130.85, 130.74, 130.72, 130.36, 130.34, 129.54, 128.86, 128.66, 128.54, 128.00, 127.89, 127.81, 127.58, 127.47, 127.36, 127.28, 126.92, 126.63, 126.27, 126.15, 126.01, 125.89, 35.21, 35.16, 34.51, 34.46, 25.99, 25.82; **MS (ESI)**: found $[M+H]^+$ 517.2, 519.2



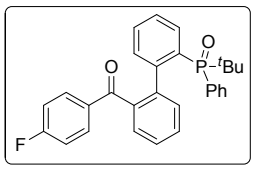
colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 40.83, 39.50; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.06 (d, *J* = 8.1 Hz, 0.8H), 7.92-7.87 (m, 0.4H), 7.83 (d, *J* = 8.0 Hz, 1.0H), 7.76-7.71 (m, 0.6H), 7.60 (d, *J* = 8.4 Hz, 0.9H), 7.55-7.26 (m, 11.0H), 7.20-7.16 (m, 0.6H), 7.10-7.04 (m, 1.6H), 6.51 (d, *J* = 7.6 Hz, 0.4H), 1.21-1.14 (m, 9.0H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 197.40, 195.74, 146.97, 146.92, 146.10, 146.05, 142.20, 142.17, 141.25, 140.72, 140.69, 140.52, 137.71, 136.99, 134.14, 134.05, 133.85, 133.61, 133.52, 133.32, 132.99, 132.05, 131.97, 131.81, 131.73, 131.63, 131.30, 131.08, 130.99, 130.90, 130.82, 130.77, 130.44, 130.41, 130.00, 129.79, 129.26, 128.95, 128.84, 128.37, 128.00, 127.89, 127.82, 127.60, 127.49, 126.93, 126.82, 126.76, 126.38, 126.32, 126.21, 126.05, 125.94, 125.11, 125.05, 124.84, 124.81, 124.65, 124.61, 124.58, 35.25, 35.10, 34.56, 34.40, 25.99, 25.81; **^{19}F NMR** (376 Hz, $CDCl_3$) δ: -62.93, -62.98; **MS (ESI)**: found $[M+H]^+$ 507.2



colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 40.73, 39.51; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.92-7.87 (m, 1.2H), 7.84-7.79 (m, 0.6H), 7.64 (s, 0.6H),7.60-7.07 (m, 11.4H), 6.58 (d, *J* = 7.6 Hz, 0.4H), 1.22-1.14 (m, 9.0H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 196.92, 195.24, 146.87, 146.81, 146.13, 146.08, 142.18, 142.15, 140.66, 140.63, 139.92, 139.44, 137.72, 136.69, 134.09, 134.03, 134.00, 133.90, 133.49, 133.40, 132.97, 132.14, 131.95, 131.90, 131.86, 131.81, 131.78, 131.70, 131.53, 130.96, 130.90, 130.85, 130.79, 130.60, 130.58, 130.38, 130.36, 130.32, 130.12, 129.95, 129.91, 129.29, 129.12, 129.07, 129.01, 128.57, 128.41, 128.31, 127.99, 127.88, 127.48, 127.43, 127.37, 126.66, 126.32, 126.28, 126.16, 126.05, 125.94, 35.20, 35.18, 34.50, 34.48, 26.04, 25.87; **MS (ESI)**: found $[M+H]^+$ 473.2, 475.2

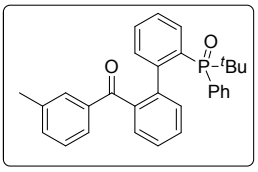


colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 41.32, 39.17; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.92-7.87 (m, 0.9H), 7.80 (dd, *J* = 7.6 Hz, 1.6 Hz, 0.4H), 7.60-7.53 (m, 2.0 H), 7.51-7.46 (m, 1.6H), 7.44-7.07 (m, 11.1H), 7.01 (dt, J = 7.6 Hz, 1.2 Hz, 0.5H), 6.39 (d, *J* = 7.6 Hz), 1.26 (t, *J* = 14.6 Hz, 9.0H), **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 196.07, 195.17, 147.45, 147.39, 147.02, 146.97, 141.98, 141.94, 141.5, 141.21, 141.18, 141.11, 137.02, 135.24, 133.61, 132.90, 132.83, 132.77, 132.73, 132.49, 132.27, 132.18, 132.03, 131.99, 131.90, 131.73, 131.65, 131.56, 131.28, 130.99, 130.86, 130.71, 130.68, 130.65, 130.61, 130.53, 130.51, 130.46, 130.41, 130.36, 130.15, 129.75, 129.12, 128.86, 128.23, 127.74, 127.63, 127.41, 127.30, 126.93, 126.86, 125.95, 125.83, 125.79, 125.68, 119.82, 119.66, 34.90, 34.20, 26.07, 26.02; **MS (ESI)**: found $[M+H]^+$ 517.2, 519.2

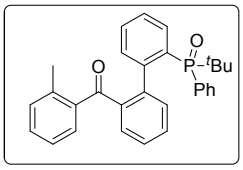


colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 40.54, 39.31; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.06 (dd, *J* = 8.6 Hz, 5.6 Hz, 0.8H), 7.91-7.87 (m, 0.4H), 7.79-7.72 (m, 1.7H), 7.62-7.57 (m, 1.0H), 7.52-7.19 (m, 9.3H), 7.11-7.07 (m, 1.6H), 7.01 (t, *J* = 8.7 Hz, 1.0H), 6.84 (t, *J* = 8.7 Hz, 1.0H), 6.56 (d, *J* = 7.7 Hz, 0.4H), 1.23-1.13 (m, 9.0H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 197.06, 195.47, 166.51, 166.30, 163.99, 163.79, 146.76, 146.71, 145.90, 145.86, 141.66, 141.63, 140.18, 140.15,

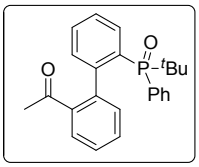
138.26, 137.44, 134.26, 133.94, 133.92, 133.59, 133.49, 133.47, 133.38, 133.33, 133.24, 133.06, 132.11, 132.03, 131.92, 131.79, 131.71, 131.67, 131.59, 131.24, 130.90, 130.87, 130.79, 130.74, 130.69, 130.57, 130.54, 130.20, 129.81, 129.30, 128.93, 128.54, 128.52, 128.18, 127.97, 127.86, 127.48, 127.37, 127.12, 126.64, 126.22, 126.09, 125.93, 125.81, 114.98, 114.77, 114.71, 114.49, 35.18, 35.15, 34.48, 34.45, 26.00, 25.82; **^{19}F NMR** (376 Hz, $CDCl_3$) δ: -106.29, -106.61; **MS (ESI)**: found $[M+H]^+$ 457.1



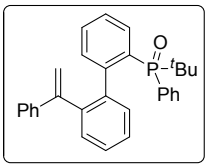
colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 40.93, 39.72; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.91-7.84 (m, 1.0H), 7.79-7.77 (m, 0.7H), 7.65 (d, *J* = 8.8 Hz, 0.8H), 7.58 (d, *J* = 7.6 Hz, 0.6H), 7.53-7.38 (m, 6.6H), 7.32-7.20 (m, 3.6H), 7.18-7.13 (m, 1.5H), 7.07-7.03 (m, 1.2H), 7.69 (d, *J* = 7.7 Hz, 0.4H), 2.35-2.19 (m, 3.0H), 1.26-1.12 (m, 9.0H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 198.57, 196.75, 146.79,146.73, 146.30, 146.26 ,141.75, 141.72, 140.59, 138.36, 138.18, 138.00, 137.56, 137.04, 134.12, 134.03, 133.24, 133.20, 133.15, 132.95, 132.09, 131.98, 131.94, 131.85, 131.76, 131.67, 131.06, 131.02, 130.95, 130.89, 130.74, 130.39, 130.37, 130.34, 130.20, 129.83, 129.37, 128.94, 128.55, 128.52, 128.19, 128.02, 127.99, 127.91, 127.83, 127.67, 127.26, 127.15, 126.52, 126.17, 126.10, 125.97, 125.86, 35.28, 35.11, 34.58, 34.41, 26.06, 26.02, 21.21, 21.19; **MS (ESI)**: found $[M+H]^+$ 453.2



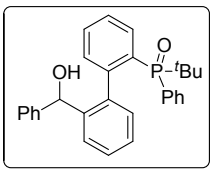
colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 40.39, 39.14; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.96-7.88 (m, 1.0H), 7.73 (d, *J* = 7.2 Hz, 0.4H), 7.61-7.52 (m, 2.6H), 7.48-7.34 (m, 4.8H), 7.30-7.19 (m, 3.4H), 7.17-7.03 (m, 4.6H), 6.53 (d, *J* = 7.5 Hz, 0.4H), 2.26-2.23 (m, 3.0H), 1.30-1.17 (m, 9.0H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 199.53, 198.20, 147.44, 147.39, 146.96, 146.91, 141.88, 141.84, 141.12, 139.13, 139.06, 138.76, 137.56, 137.42, 137.13, 133.90, 133.45, 133.35, 133.02, 132.51, 132.42, 132.36, 132.01, 131.92, 131.83, 131.74, 131.22, 131.06, 130.92, 130.83, 130.78, 130.68, 130.58, 130.52, 130.50, 130.46, 130.42, 130.40, 130.24, 130.18, 130.08, 129.73, 129.31, 129.23, 128.84, 128.34, 127.88, 127.77, 127.33, 127.21, 126.73, 126.63, 125.93, 125.83, 125.72, 125.13, 124.87, 35.16, 35.00, 34.46, 34.30, 26.09, 26.07, 20.24, 20.13; **MS (ESI)**: found $[M+H]^+$ 453.2



colorless oil; **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 40.54, 39.31; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.10-8.06 (m, 0.5H), 7.77-7.72 (m, 0.5H), 7.67-7.25 (m, 9.5H), 7.22-7.18 (m, 1.5H), 6.86-6.82 (m, 0.5H), 6.18 (d, *J* = 7.0 Hz, 0.5H), 2.33 (s, 1.6H), 2.22 (s, 1.3H), 1.30-1.19 (m, 9.0H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 201.51, 199.58, 148.28, 148.23, 148.01, 147.96, 140.55, 139.79, 139.17, 137.39, 133.41, 132.86, 132.77, 132.51, 132.44, 132.41, 132.14, 132.02, 131.98, 131.90, 131.66, 131.57, 130.88, 130.85, 130.74, 130.71, 130.67, 130.64, 130.58, 130.55, 130.52, 130.12, 129.89, 129.21, 129.00, 128.21, 128.16, 127.77, 127.56, 127.32, 127.22, 126.97, 126.19, 126.08, 125.68, 125.56, 34.87, 34.68, 34.17, 33.98, 29.54, 28.71, 25.92, 25.90; **MS (ESI)**: found $[M+H]^+$ 377.1

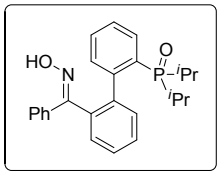


White solid: **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 38.14, 37.74; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.99-7.95 (m, 0.6H), 7.85-7.81 (m, 0.7H), 7.69 (dd, *J* = 8.1 Hz, 10.3 Hz, 0.4H), 7 .55-7.15 (m, 11.7H), 7.06-7.02 (m, 1.0H), 7.00-6.91 (m, 1.2H), 6.86-6.80 (m, 1.7H), 6.25 (d, *J* = 7.5 Hz, 0.6H), 5.45 (dd, *J* = 28.6 Hz, 1.5 Hz, 1.2H), 4.93 (dd, *J* = 1.1 Hz, 12.2 Hz, 0.7H), 1.24-1.14 (m, 9.0H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 148.56, 147.83, 147.56, 147.51, 147.44, 142.09, 140.74, 140.71, 140.68, 139.82, 134.06, 133.38, 133.29, 133.18, 133.01, 132.67, 132.58, 132.13, 132.09, 132.01, 131.96, 131.87, 131.37, 131.26, 130.76, 130.70, 130.68, 130.58, 130.47, 130.11, 130.09, 129.97, 129.88, 129.68, 129.63, 129.40, 129.38, 129.30, 128.66, 127.75, 127.65, 127.34, 127.21, 127.04, 126.83, 126.82, 126.74, 126.51, 125.98, 125.55, 125.51, 125.40, 125.17, 125.06, 117.92, 117.39, 35.45, 35.07, 34.75, 34.37, 25.97, 25.74; **MS (ESI)**: found $[M+H]^+$ 437.2

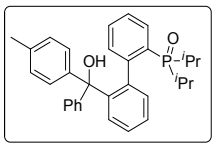


White solid: **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 43.23; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 8.02-7.97 (m, 1H), 7.44-7.40 (m, 2H), 7.33-7.14 (m, 7H), 7.06-6.98 (m, 6H), 6.55 (dt, *J* = 7.6 Hz, 0.3 Hz, 1H), 6.19-6.16 (m, 1H), 5.87 (s, 1H), 5.72 (dd, *J* = 7.9 Hz, 0.8 Hz, 1H), 1.36 (d, *J* = 14.5 Hz, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 146.75 (d, *J* = 5.3 Hz), 144.96, 141.91, 138.38 (d, J = 3.5 Hz), 133.08 (d, *J* = 9.1 Hz), 131.91, 131.68 (d, *J* = 8.9 Hz), 130.95, 130.87 (d, *J* = 22.3 Hz), 129.99 (d, *J* = 2.3

Hz), 129.58, 129.45 (d, *J* = 11.0 Hz), 129.28, 128.83 (d, *J* = 89.4 Hz), 127.56 (d, *J* = 11.5 Hz), 127.16, 126.96, 125.52, 125.48, 125.36 (d, *J* = 11.5 Hz), 76.95, 33.98 (d, *J* = 69.7 Hz), 25.60; **MS (ESI)**: found $[M+H]^+$ 441.2

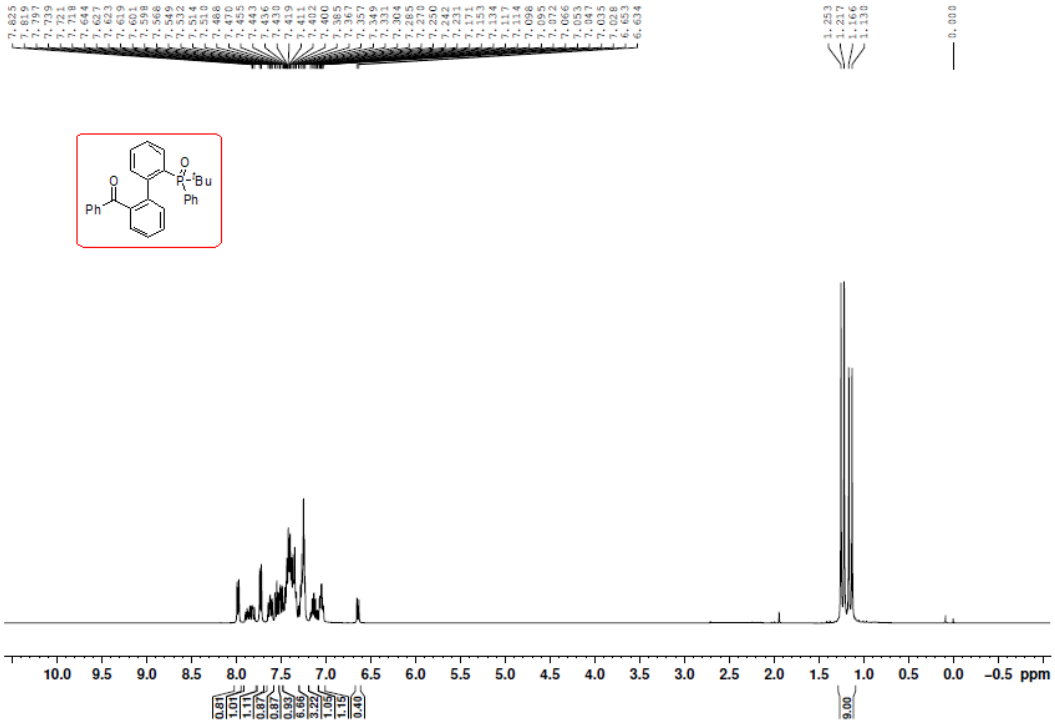


White solid: **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 43.23; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 10.56 (d, *J* = 2.1 Hz, 1H), 7.55-7.43 (m, 3H), 7.21-7.16 (m, 4H), 7.09 (t, *J* = 7.6 Hz, 2Hz), 7.03-6.97 (m, 3H), 6.65 (dd, *J* = 7.6 Hz, 2.5 Hz, 1H), 2.47-2.42 (m, 1H), 2.12-2.02 (m, 1H), 1.29-1.23 (m, 6H), 0.95 (dt, *J* = 8.1 Hz, 7.1 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 161.90, 145.95 (d, *J* = 4.3 Hz), 140.84, 137.65, 133.77, 132.56 (d, *J* = 9.0 Hz), 129.95, 129.88, 129.85, 129.78, 128.88, 128.52, 128.08, 127.57, 127.49, 127.09, 126.58, 126.47, 125.70, 28.00 (d, *J* = 67.5 Hz), 24.23 (d, *J* = 66.5 Hz), 16.66, 15.67 (d, *J* = 3.3 Hz), 15.53 (d, *J* = 3.3 Hz), 14.58 (d, *J* = 2.4 Hz); **MS (ESI)**: found $[M+H]^+$ 406.2



White solid: **^{31}P NMR** (162 MHz, $CDCl_3$) δ: 57.60; **^{1}H NMR** (400 MHz, $CDCl_3$) δ: 7.68 (s, 1H), 7.29-7.06 (m, 13H), 6.89-6.82 (m, 3H), 6.00 (dd, *J* = 7.7 Hz, 3.0 Hz, 1H), 2.56-2.46 (m, 1H), 2.30 (s, 1H), 2.26-2.15 (m, 1H), 1.34-1.22 (m, 6H), 1.13-1.00 (m, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ: 148.66 (d, *J* = 4.5 Hz), 146.15 (d, *J* = 2.5 Hz), 145.81, 141.42 (d, *J* = 3.0 Hz), 135.89, 132.57 (d, *J* = 9.5 Hz), 131.84, 130.00 (d, *J* = 10.8 Hz), 129.26, 128.88 (d, *J* = 2.4 Hz), 128.43, 128.23, 127.58, 127.16, 126.86, 126.36, 126.33, 125.69 (d, *J* = 13.9 Hz), 124.88 (d, *J* = 11.2 Hz), 80.90, 28.41 (d, *J* = 65.2 Hz), 24.30 (d, *J* = 66.4 Hz), 20.95, 16.49, 16.24 (d, *J* = 3.6 Hz), 16.12 (d, *J* = 1.8 Hz), 15.33 (d, J = 2.4 Hz); **MS (ESI)**: found $[M+H]^+$ 483.2

Ⅷ. NMR charts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Ph
O
Ph
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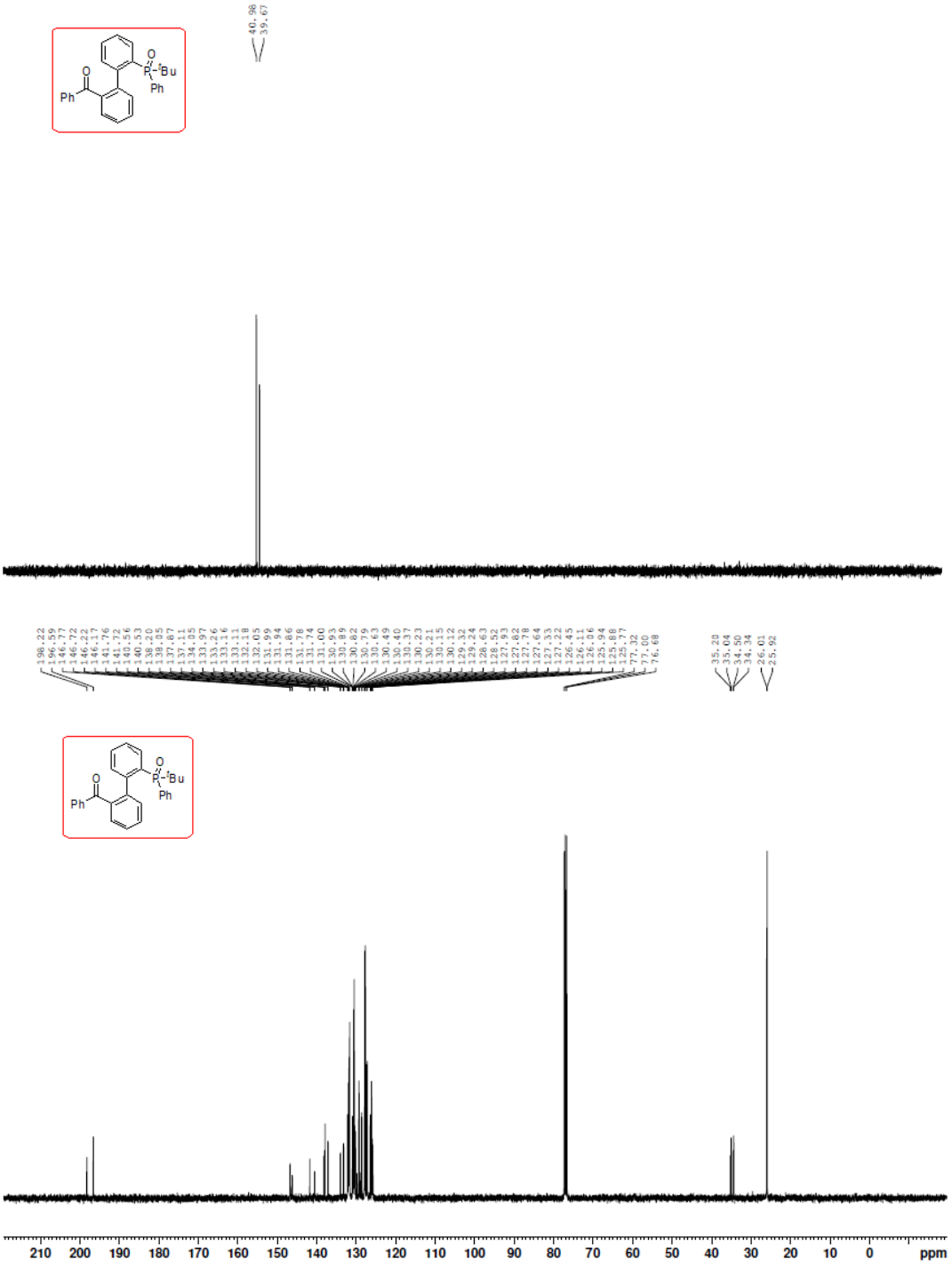
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140.56
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138.20
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137.87
137.11
134.05
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133.16
133.11
132.18
132.05
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131.66
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76.68
35.20
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26.01
25.92
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P
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Ph
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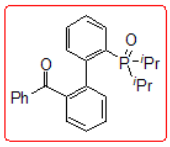
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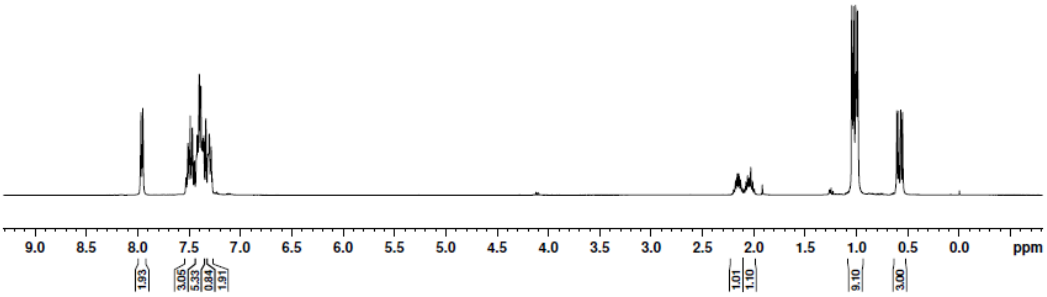
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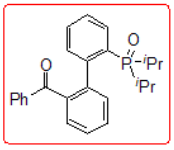
9.10
3.00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O
Ph


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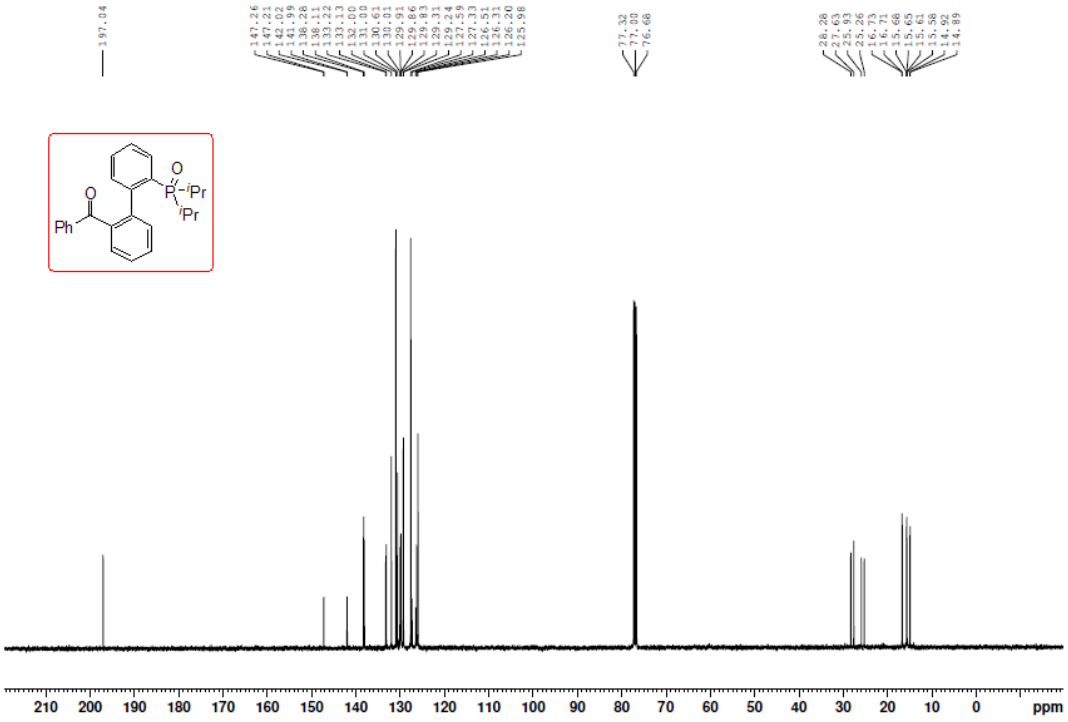
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126.20
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77.00
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27.63
25.93
25.26
16.73
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15.68
15.65
15.61
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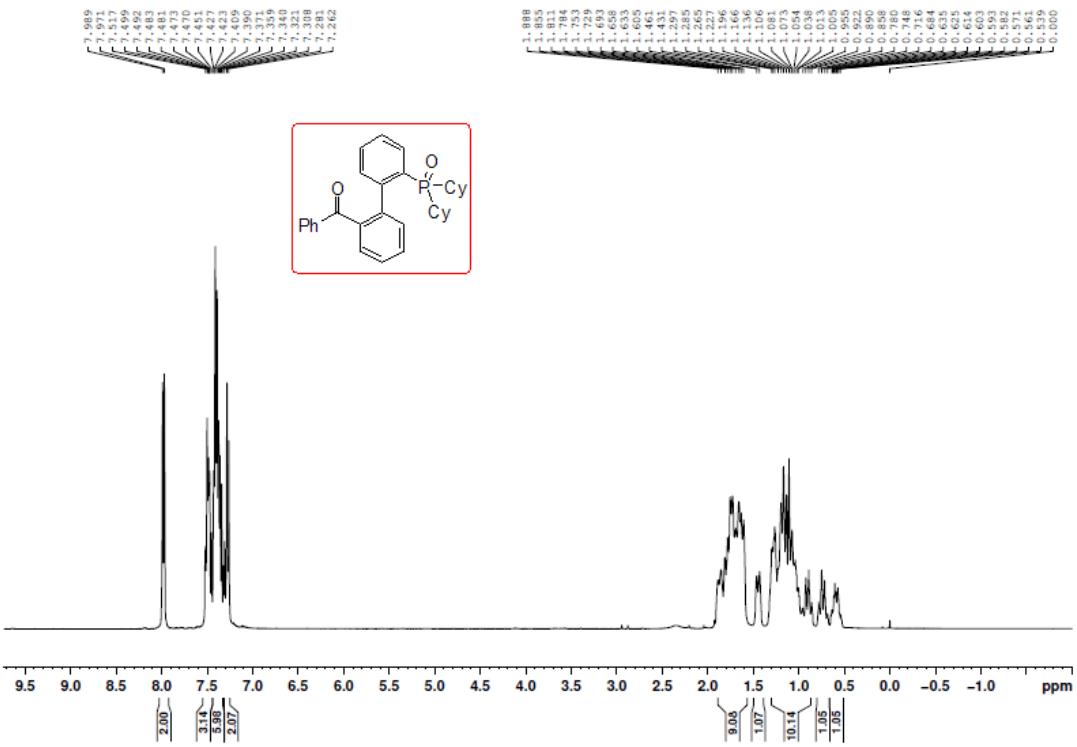
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0.593
0.582
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0.561
0.539
0.000
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P
Cy
Cy
O
Ph
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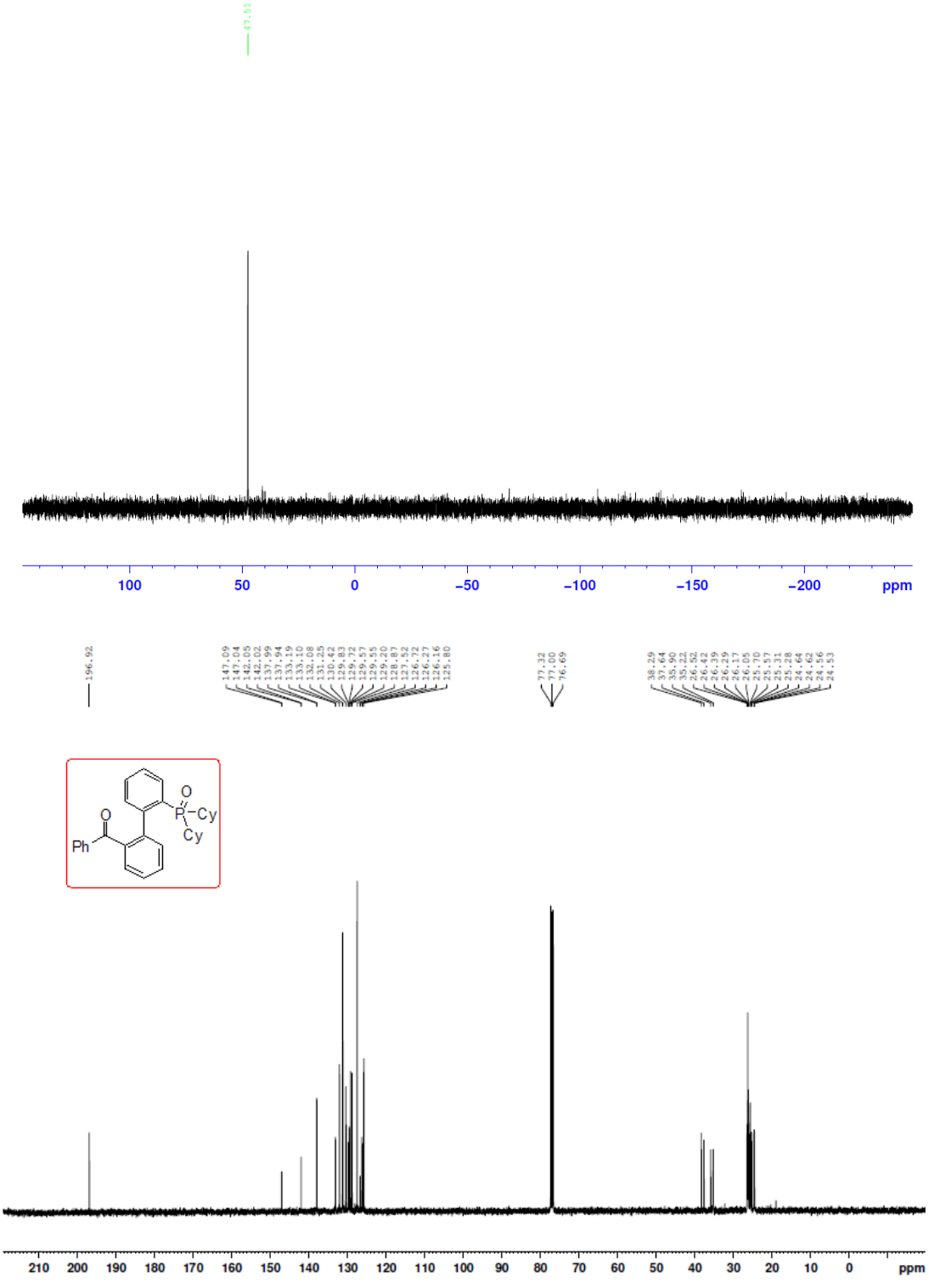
9.08
1.07
10.14
1.05
1.05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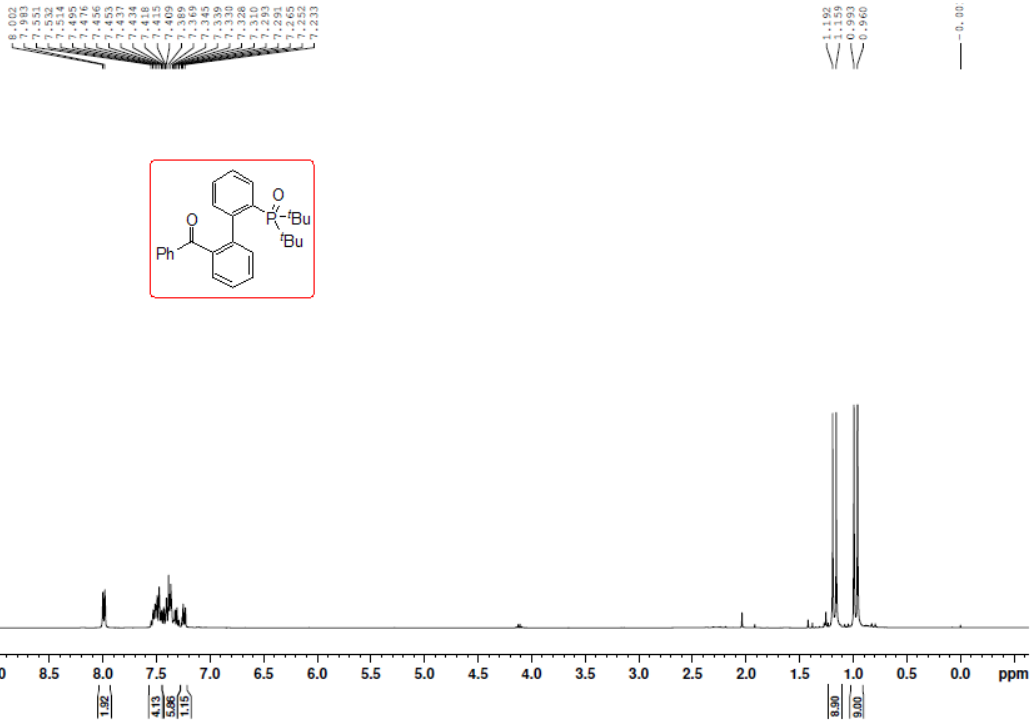
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tBu
O
Ph
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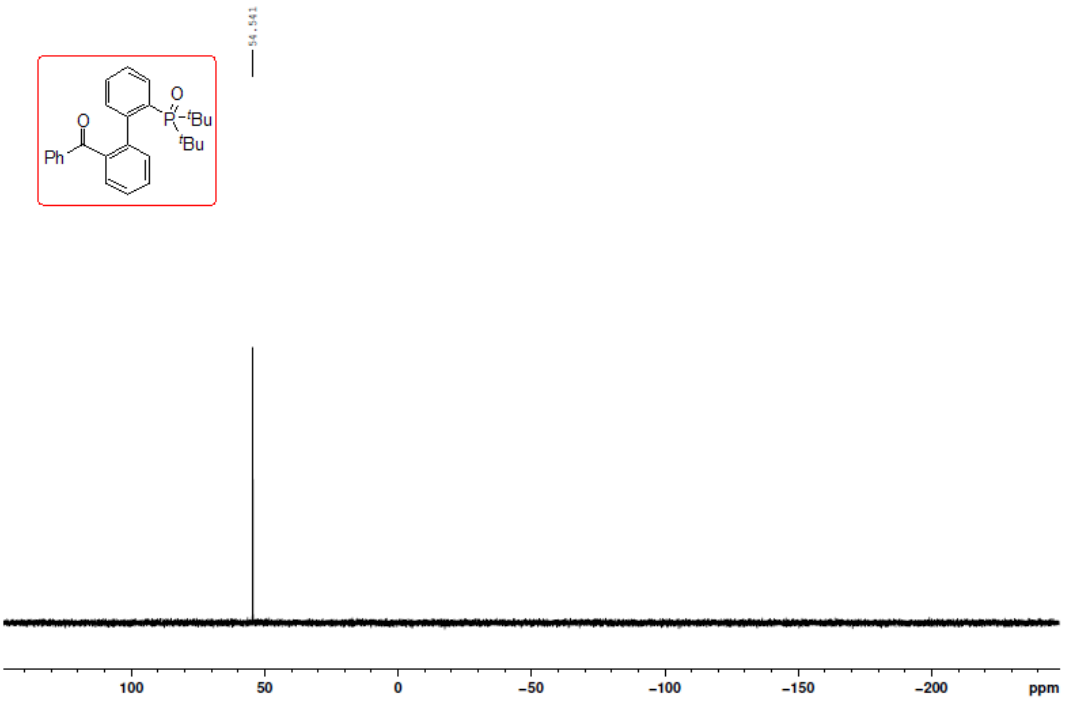
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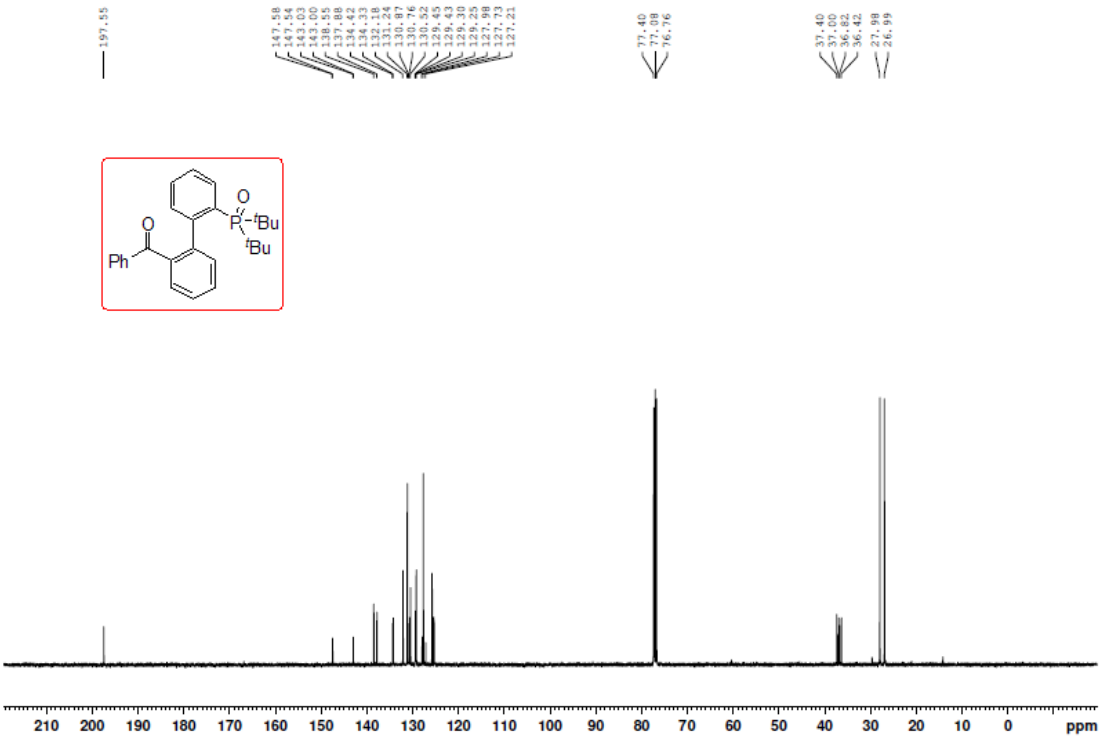
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129.45
129.43
129.30
129.25
127.98
127.73
127.21
77.40
77.08
76.76
37.40
37.00
36.82
36.42
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26.99
O
O
P
tBu
tBu
Ph
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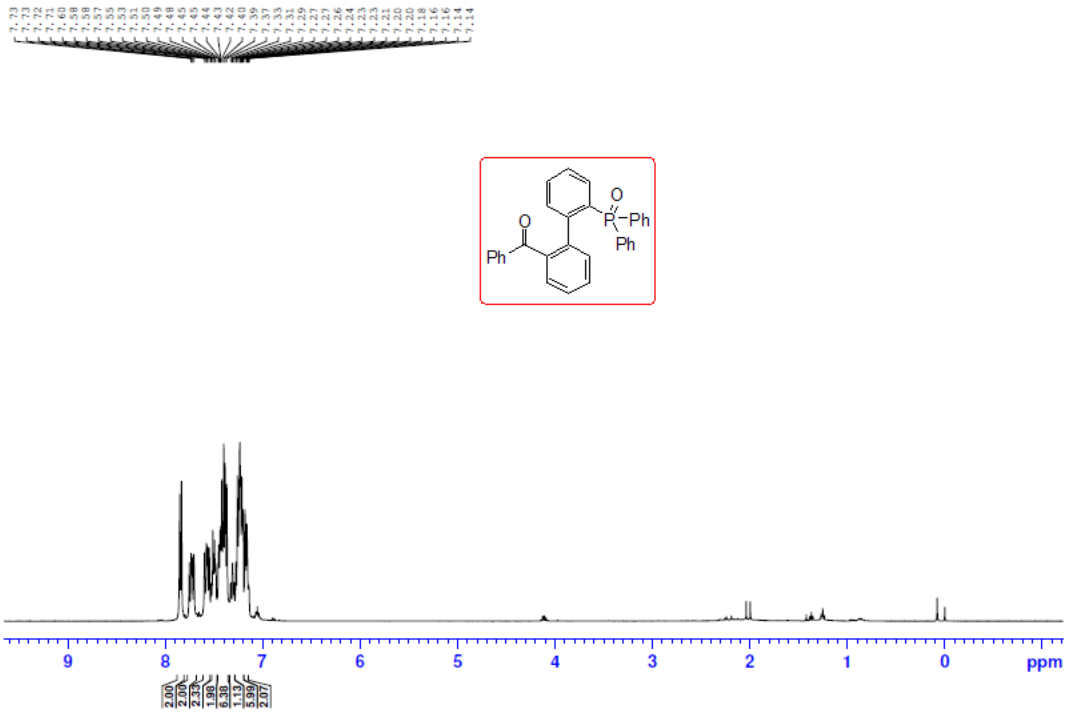
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7.33
7.31
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7.16
7.14
7.14
O
O
P
Ph
Ph
Ph
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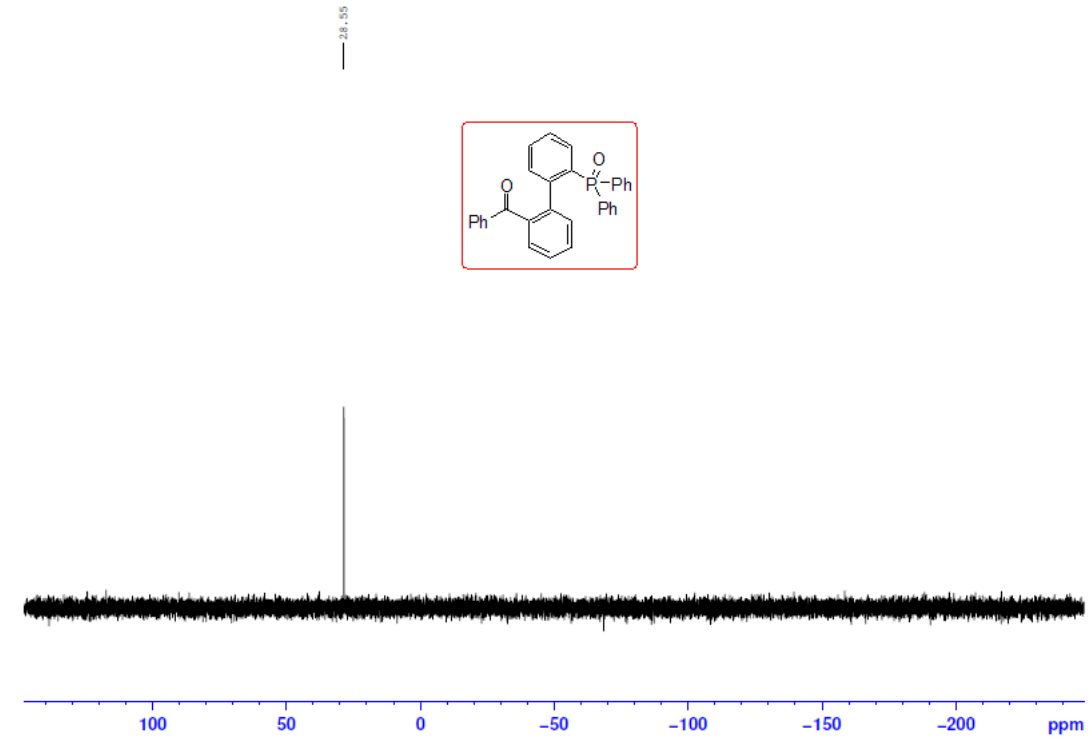
2.07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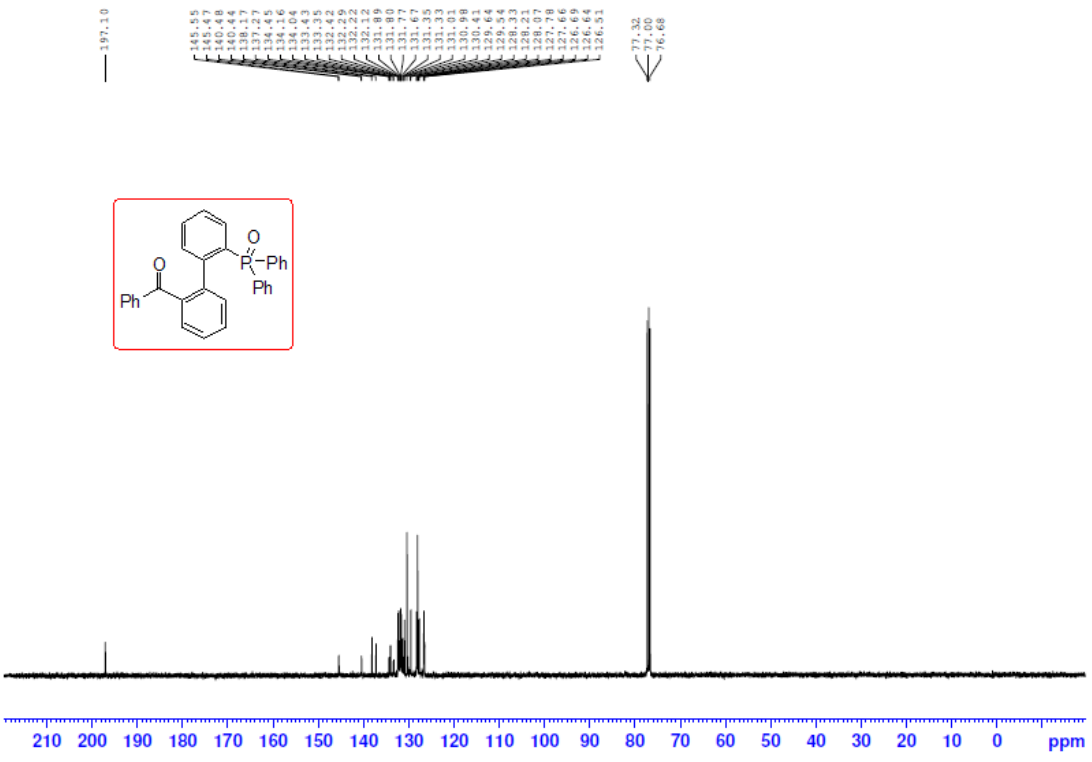
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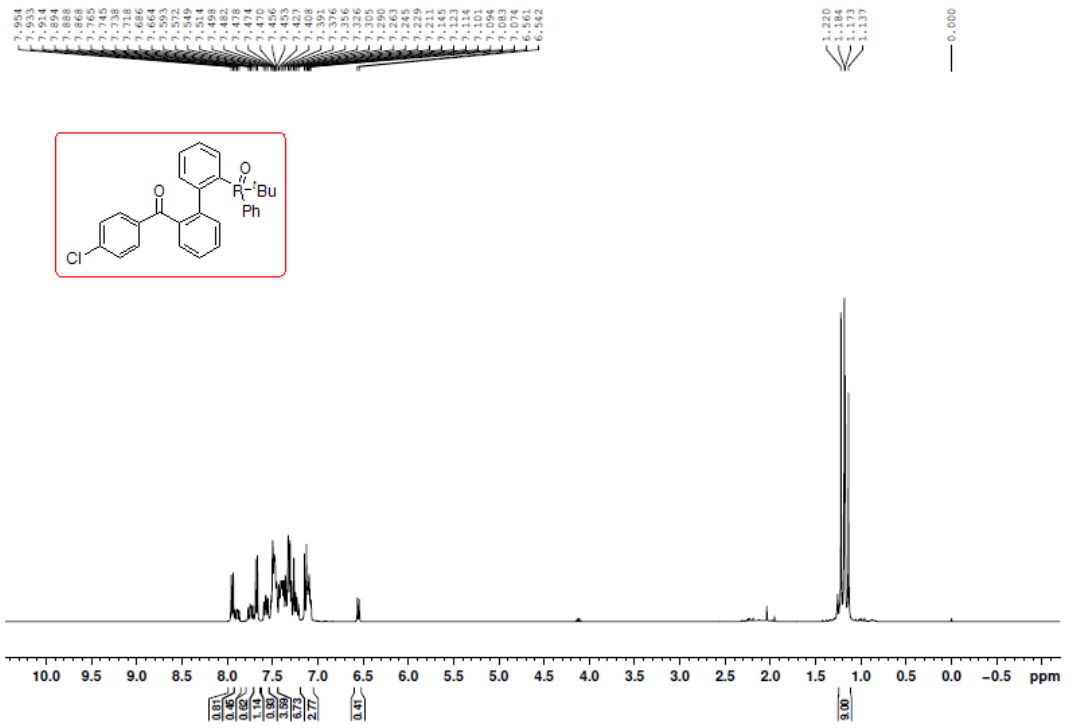
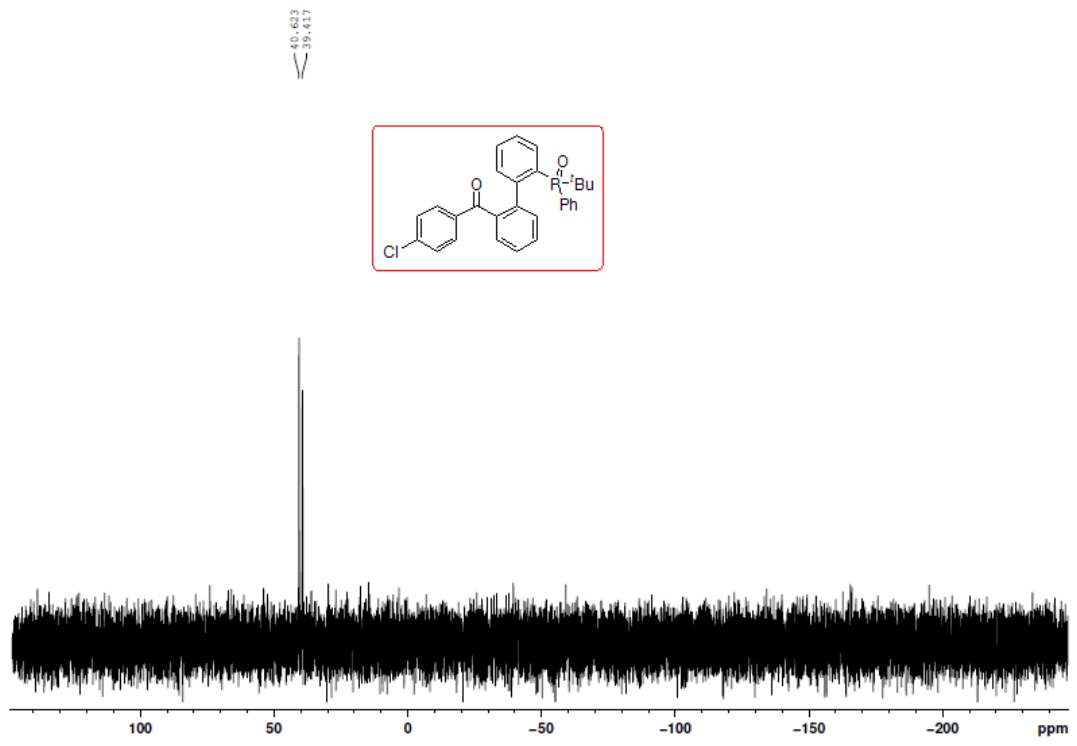
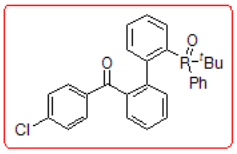
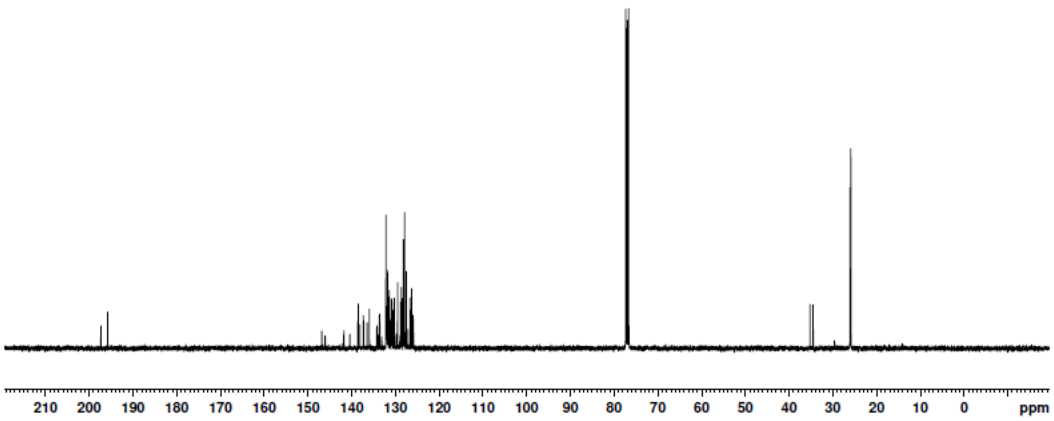
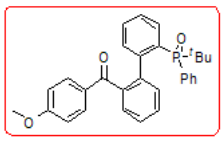
134.16
133.64
133.43
133.35
132.42
132.29
132.22
131.89
131.80
131.77
131.67
131.35
131.33
131.01
130.98
130.41
130.04
129.54
128.33
128.21
128.07
127.78
127.66
126.69
126.64
126.51
77.32
77.00
76.68
O
P
Ph
Ph
O
Ph
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

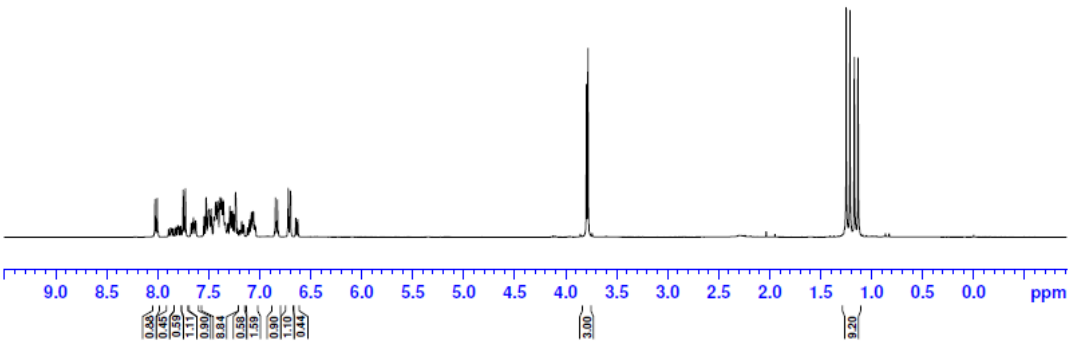

7.954
7.933
7.914
7.894
7.888
7.868
7.765
7.745
7.738
7.718
7.686
7.664
7.595
7.572
7.549
7.514
7.498
7.482
7.478
7.474
7.470
7.456
7.453
7.427
7.408
7.391
7.356
7.326
7.305
7.290
7.263
7.245
7.219
7.211
7.145
7.123
7.114
7.101
7.094
7.083
7.064
6.561
6.542
1.220
1.184
1.153
1.137
0.000
O
R-tBu
Ph
Cl
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 −0.5 ppm
0.81
0.46
0.62
1.14
0.98
3.98
0.73
2.77
0.41
9.00

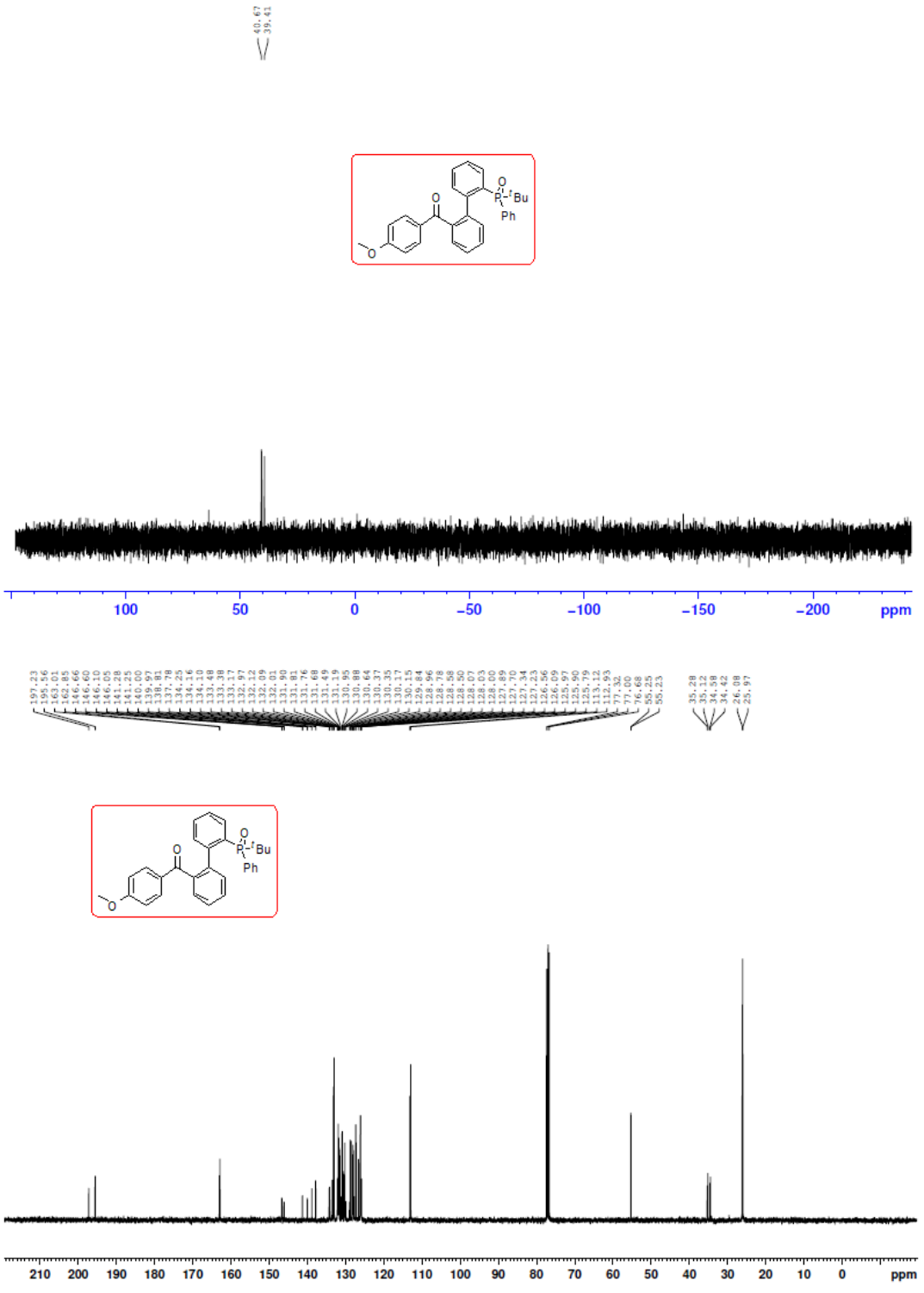

40.623
39.417
O
R-tBu
Ph
Cl
100 50 0 −50 −100 −150 −200 ppm

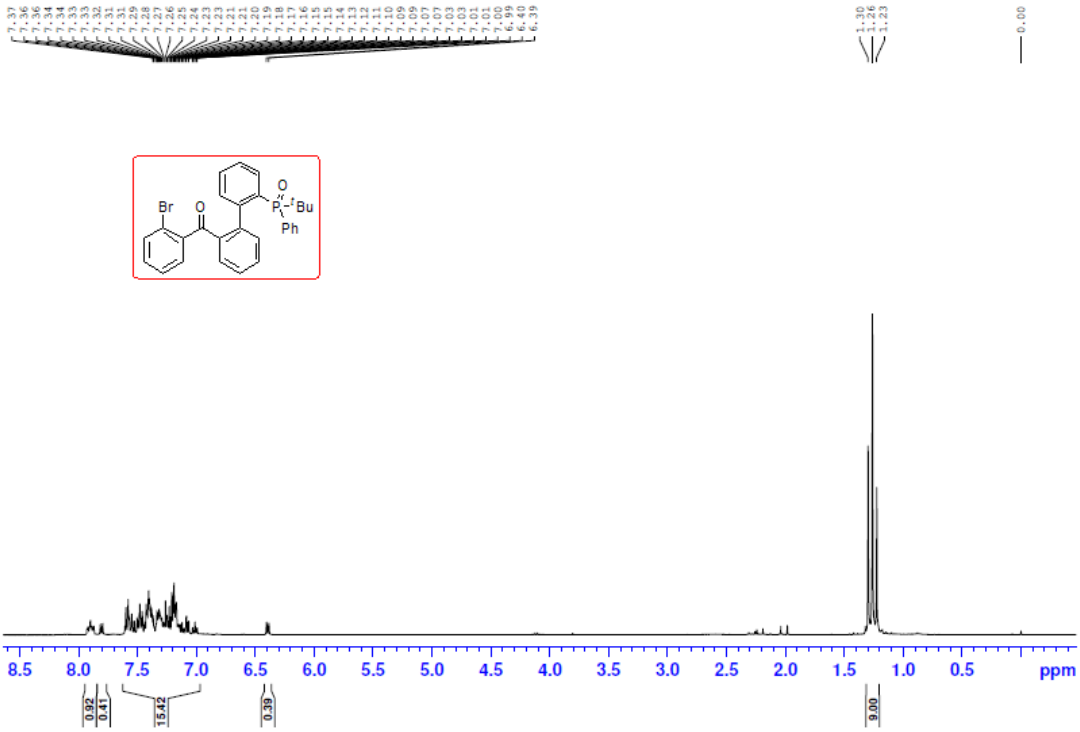

O
P
tBu
Ph
O
Cl

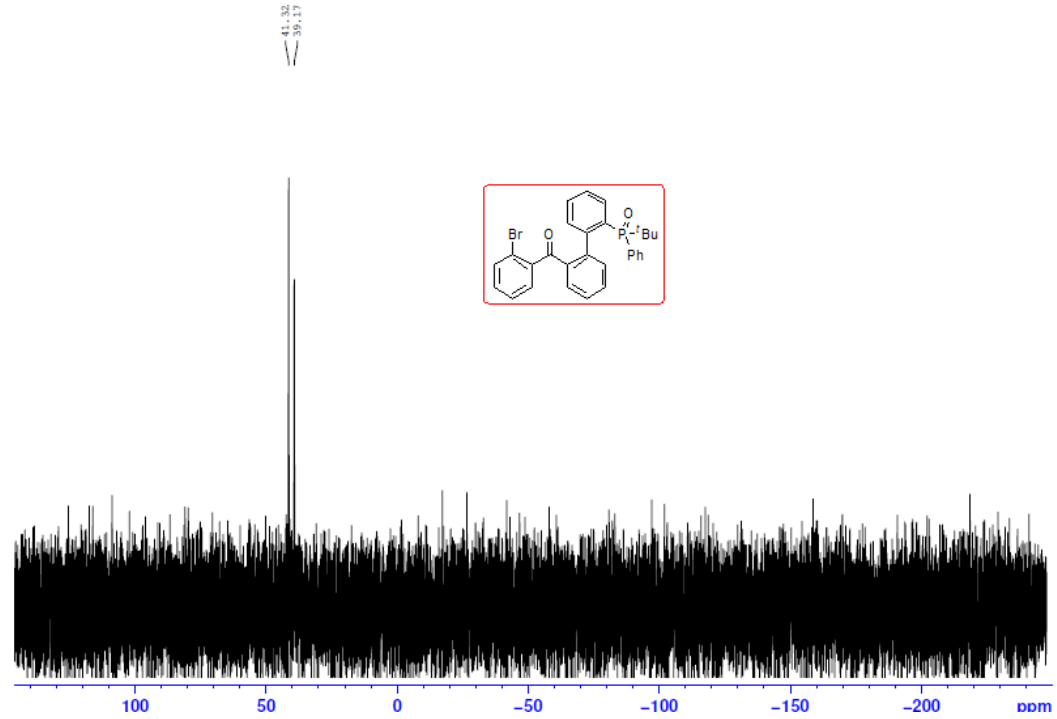

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

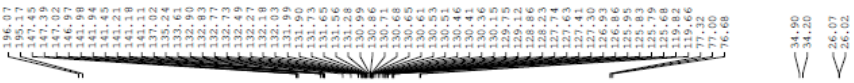

O
P
tBu
Ph
O
O

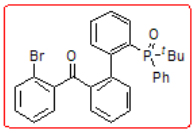

9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.60
0.45
0.59
1.11
0.99
9.84
0.58
1.59
0.90
1.10
0.44
3.00
9.20

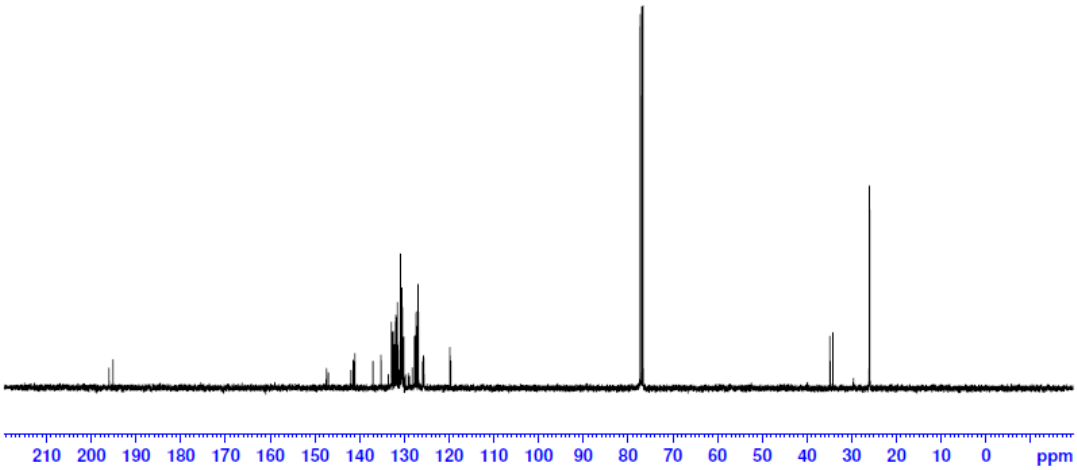

40.67
39.41
O
P
tBu
Ph
O
100
50
0
−50
−100
−150
−200
ppm
197.23
195.56
163.01
162.85
146.66
146.60
146.10
146.05
141.28
141.25
140.00
139.97
138.81
137.78
134.25
134.16
134.10
133.48
133.38
133.17
132.97
132.12
132.09
132.01
131.90
131.81
131.76
131.68
131.49
131.19
130.95
130.88
130.64
130.37
130.35
130.17
130.15
129.84
128.96
128.78
128.58
128.50
128.07
128.03
128.00
127.89
127.70
127.34
127.23
126.56
126.09
125.97
125.90
125.79
123.12
112.93
77.32
77.00
76.68
55.25
55.23
35.28
35.12
34.58
34.42
26.08
25.97
O
P
tBu
Ph
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


Br
O
O
P
tBu
Ph
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
ppm

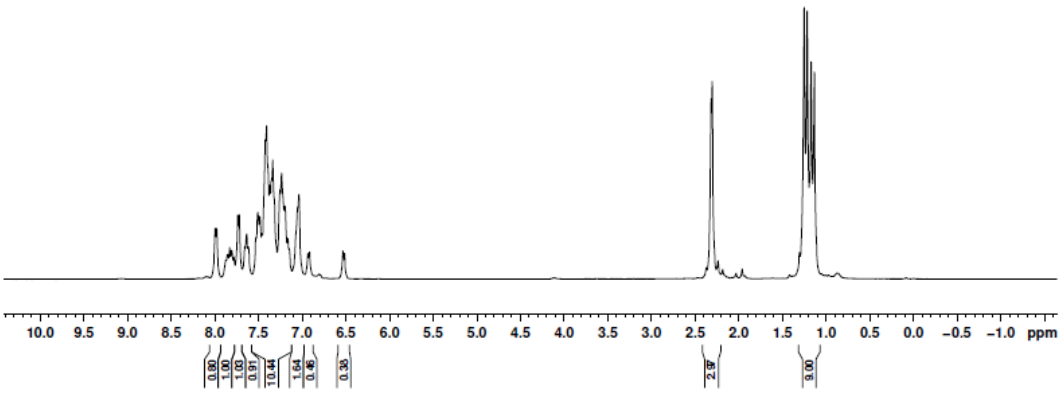

Br
O
O
P
tBu
Ph
100
50
0
−50
−100
−150
−200
ppm

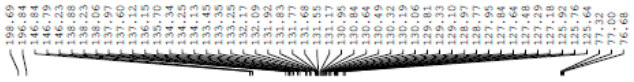


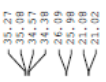

Br
O
O
P
tBu
Ph

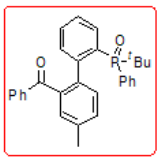

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

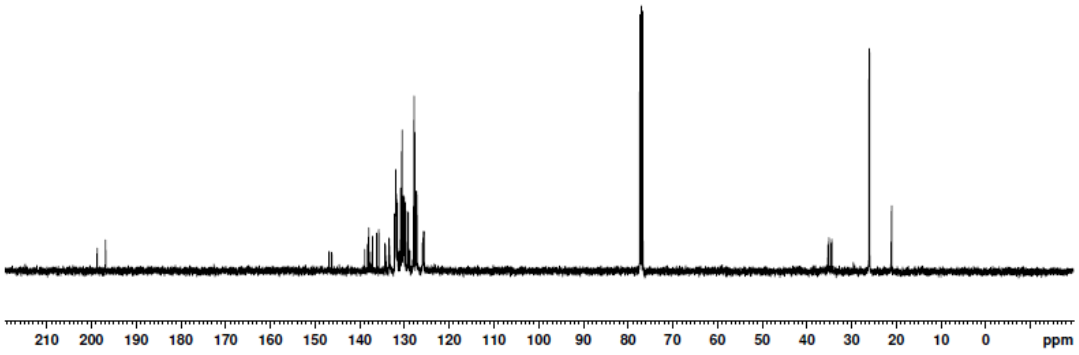

O
P
tBu
Ph
O
Ph

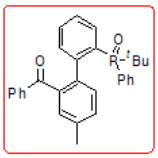

10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0 ppm

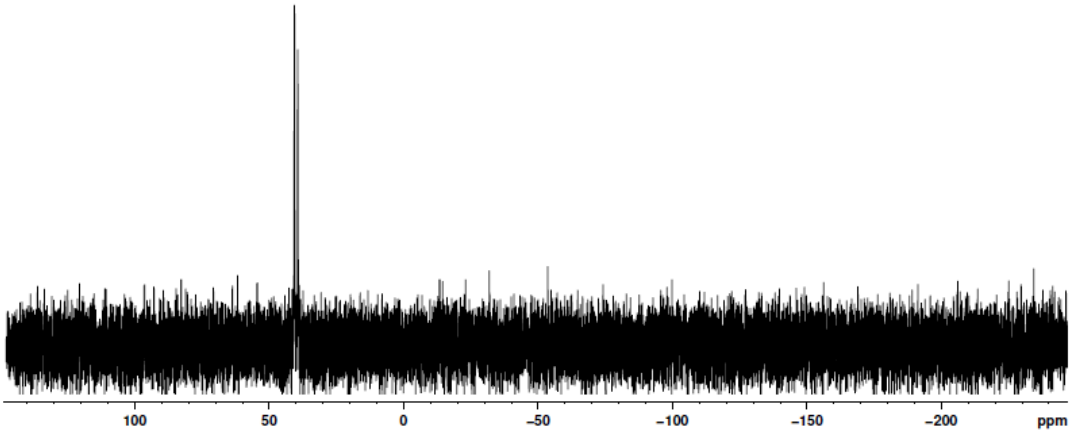


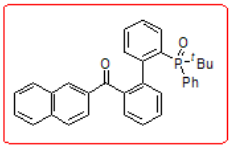


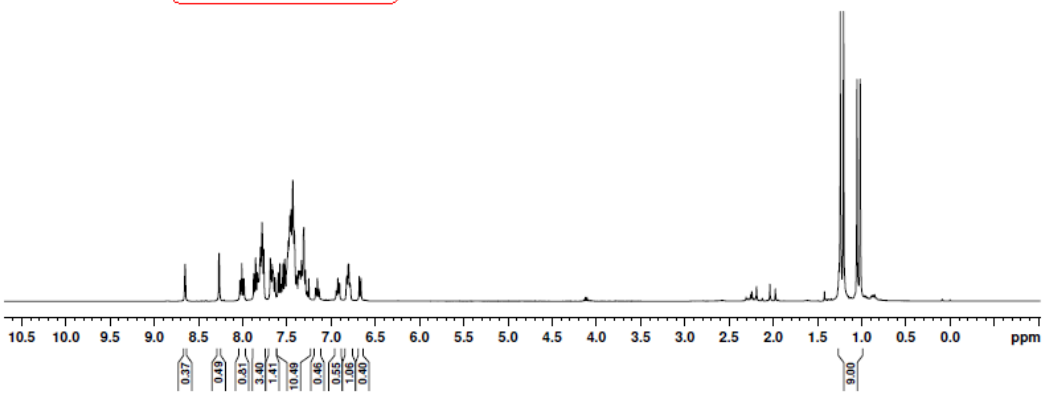

O
Ph
O
S
tBu
Ph

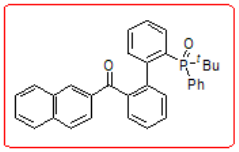

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

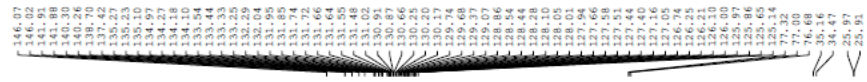

O
Ph
O
S
tBu
Ph

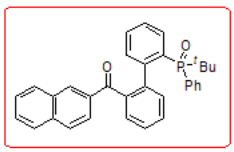

100
50
0
−50
−100
−150
−200
ppm



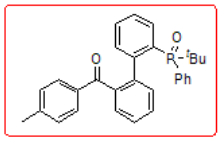


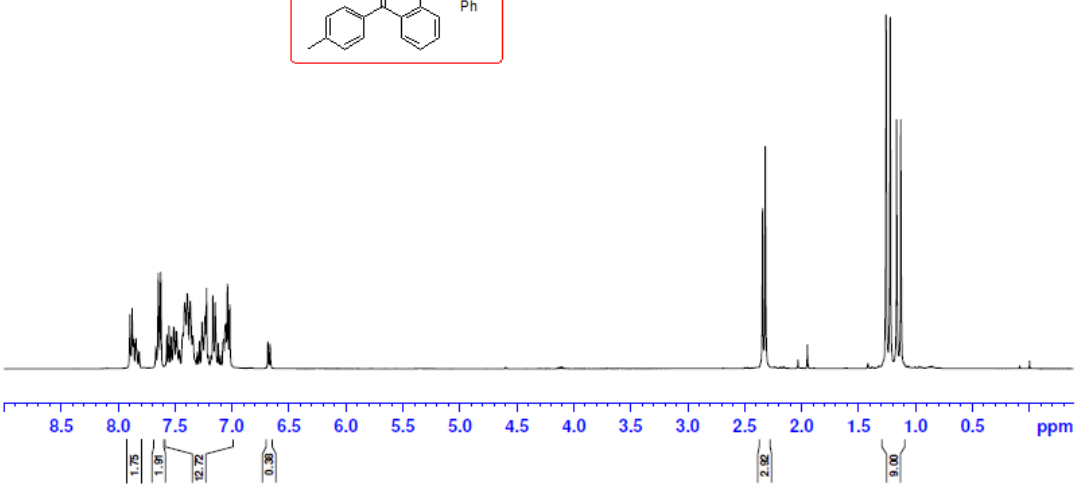


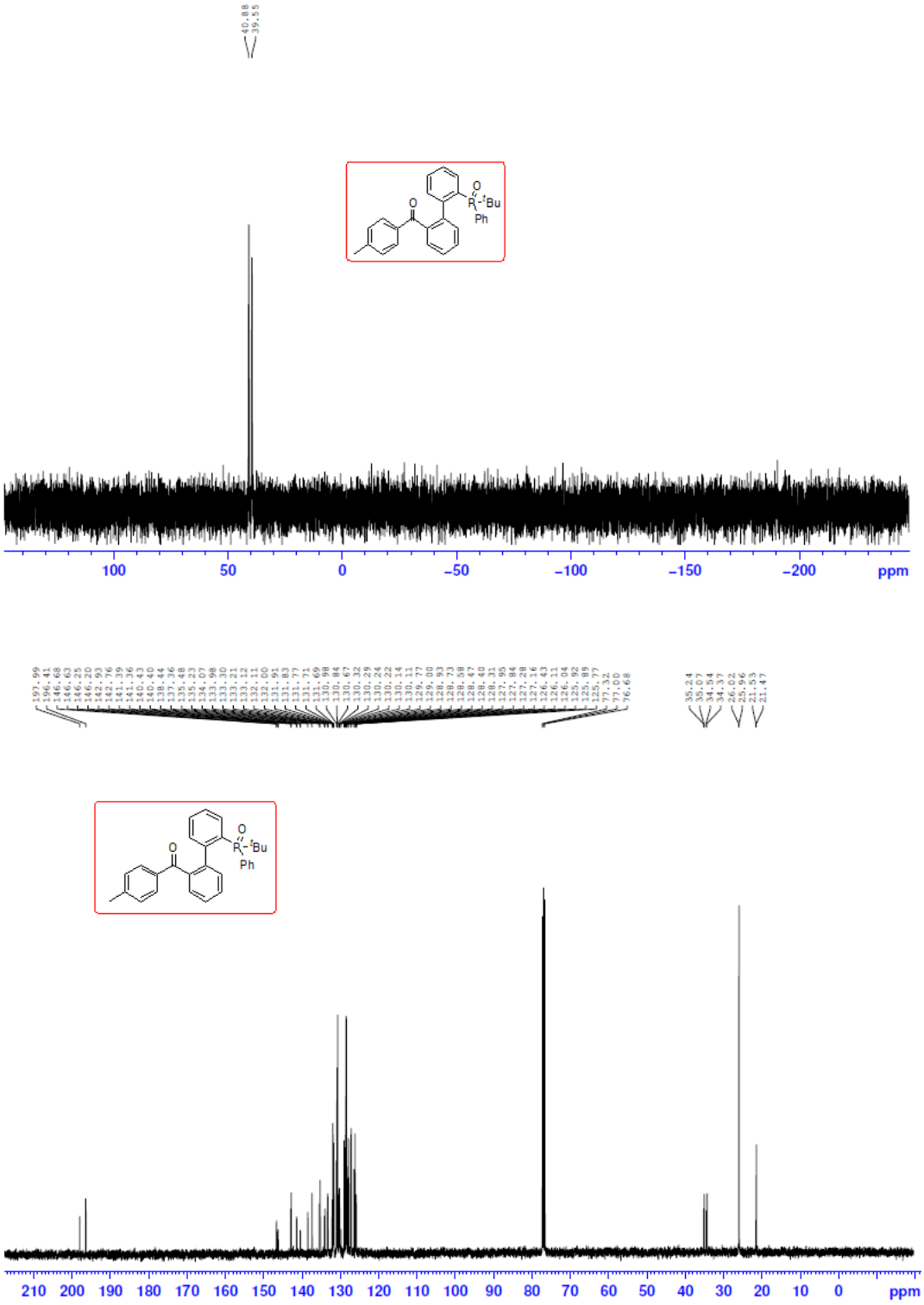


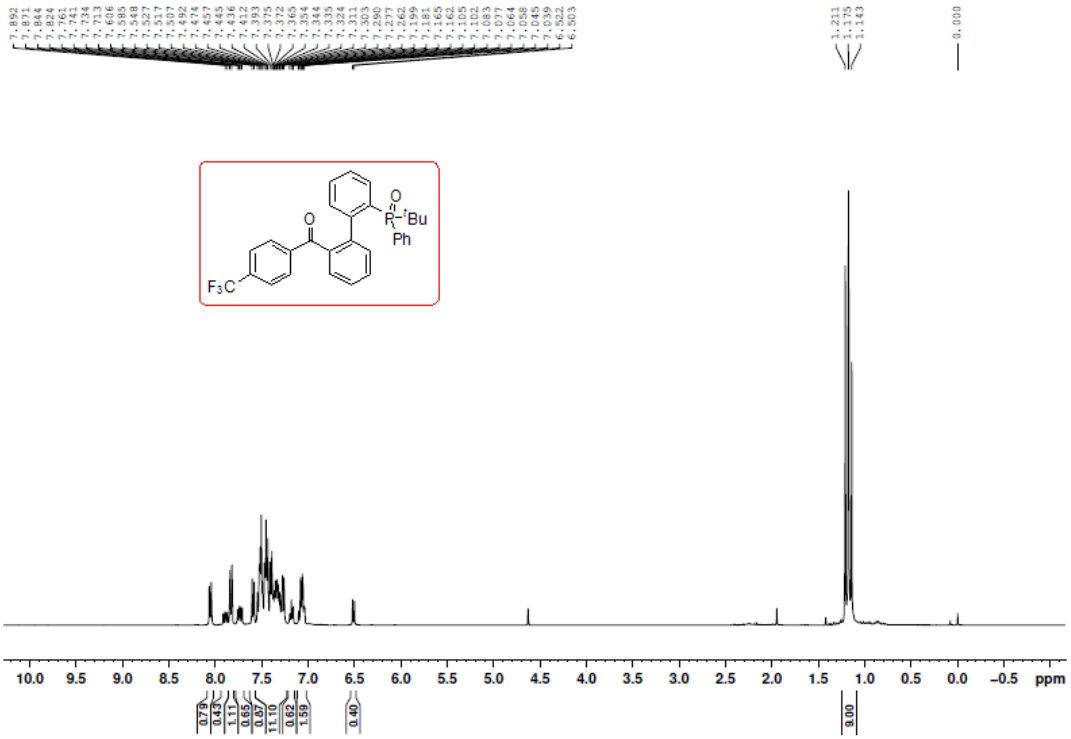

O
P
tBu
Ph
O

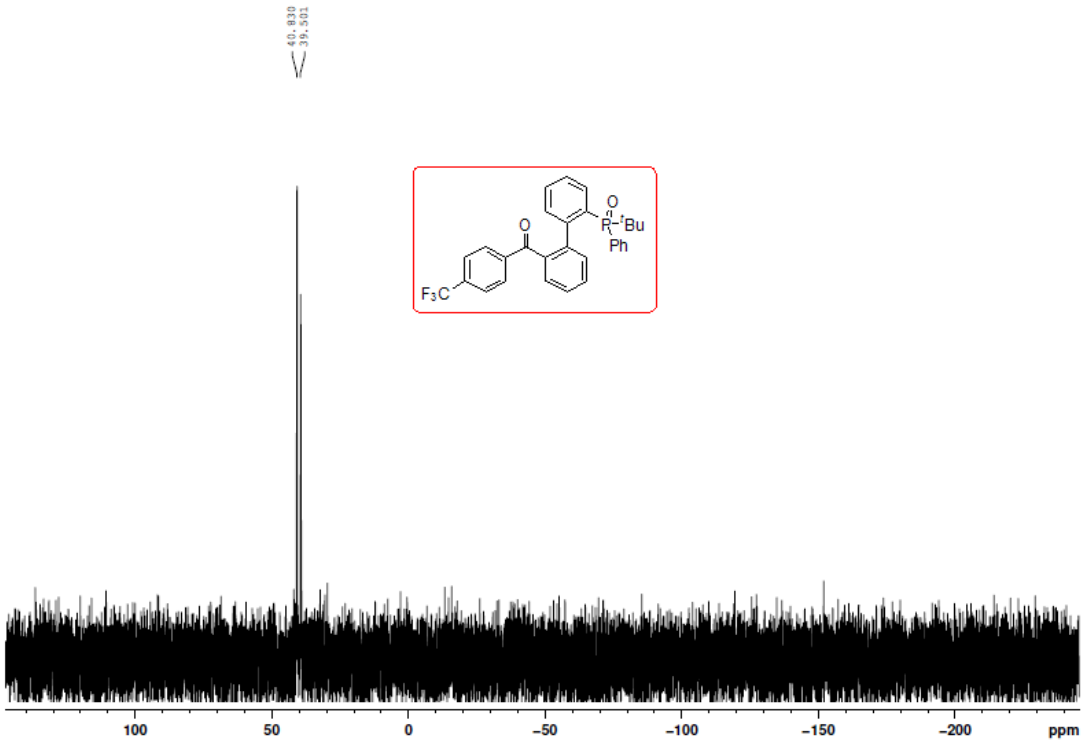
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

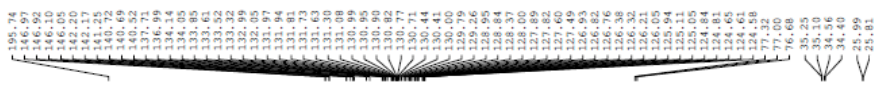

O
P
tBu
Ph
O

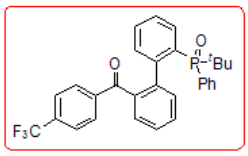

8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.75
1.91
12.72
0.38
2.02
9.00

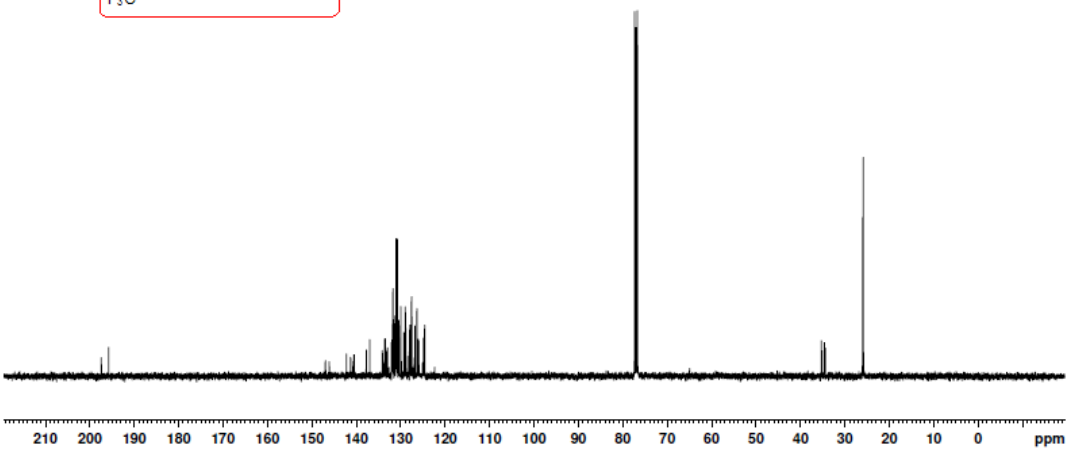

40.88
39.55
O
O
P
tBu
Ph
100
50
0
-50
-100
-150
-200
ppm
197.99
196.41
146.68
146.63
146.25
146.20
142.93
142.76
141.39
141.36
140.43
140.40
138.44
138.16
137.48
135.23
134.07
133.98
133.30
133.21
133.12
132.11
132.00
131.91
131.83
131.77
131.71
131.66
130.98
130.84
130.67
130.32
130.29
130.24
130.22
130.14
130.11
129.77
129.08
128.93
128.73
128.58
128.47
128.40
128.31
127.95
127.84
127.28
127.16
126.43
126.11
126.04
125.92
125.89
125.77
77.32
77.00
76.68
35.24
35.07
34.54
34.37
26.02
25.96
21.53
21.47
O
O
P
tBu
Ph
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

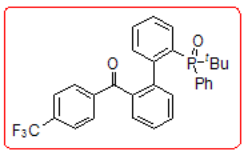

7.892
7.871
7.844
7.824
7.761
7.741
7.734
7.713
7.606
7.585
7.548
7.527
7.517
7.507
7.492
7.472
7.457
7.445
7.436
7.412
7.393
7.375
7.372
7.362
7.354
7.344
7.335
7.324
7.311
7.303
7.290
7.277
7.262
7.199
7.181
7.165
7.162
7.105
7.102
7.083
7.077
7.064
7.058
7.045
7.039
6.522
6.503
1.211
1.175
1.143
0.000
O
P
tBu
Ph
F3C
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
−0.5
ppm
0.79
0.43
1.11
0.65
0.87
11.10
0.62
1.59
0.40
9.00

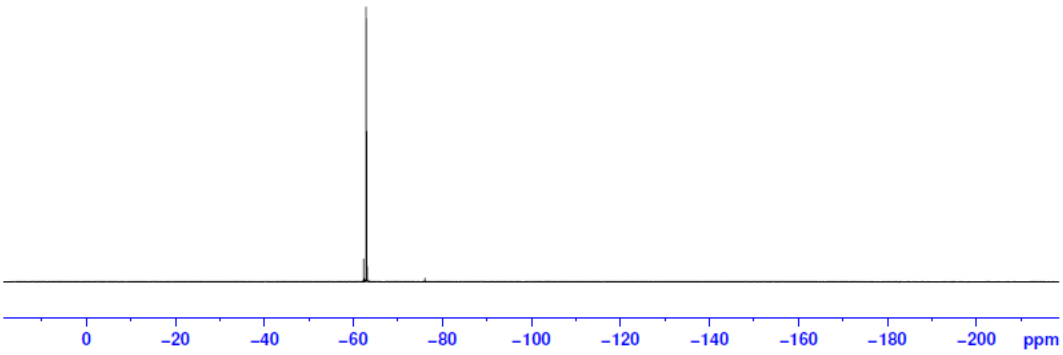

40.830
39.501
O
P
tBu
Ph
F3C
100
50
0
−50
−100
−150
−200
ppm

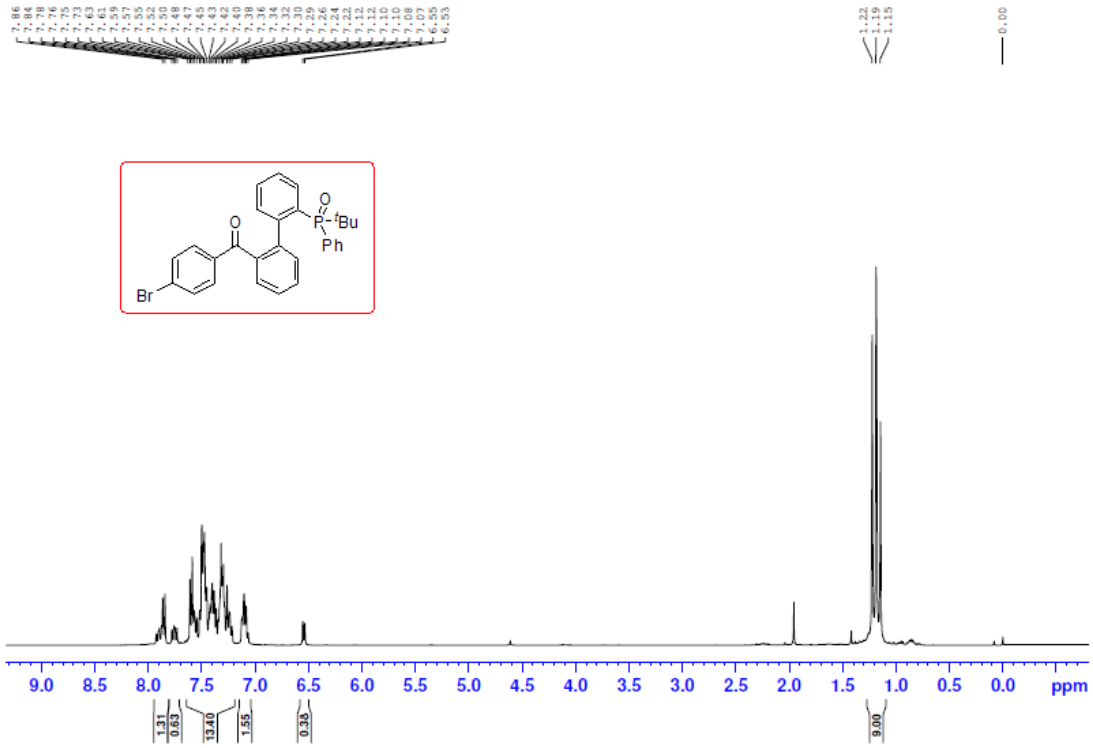


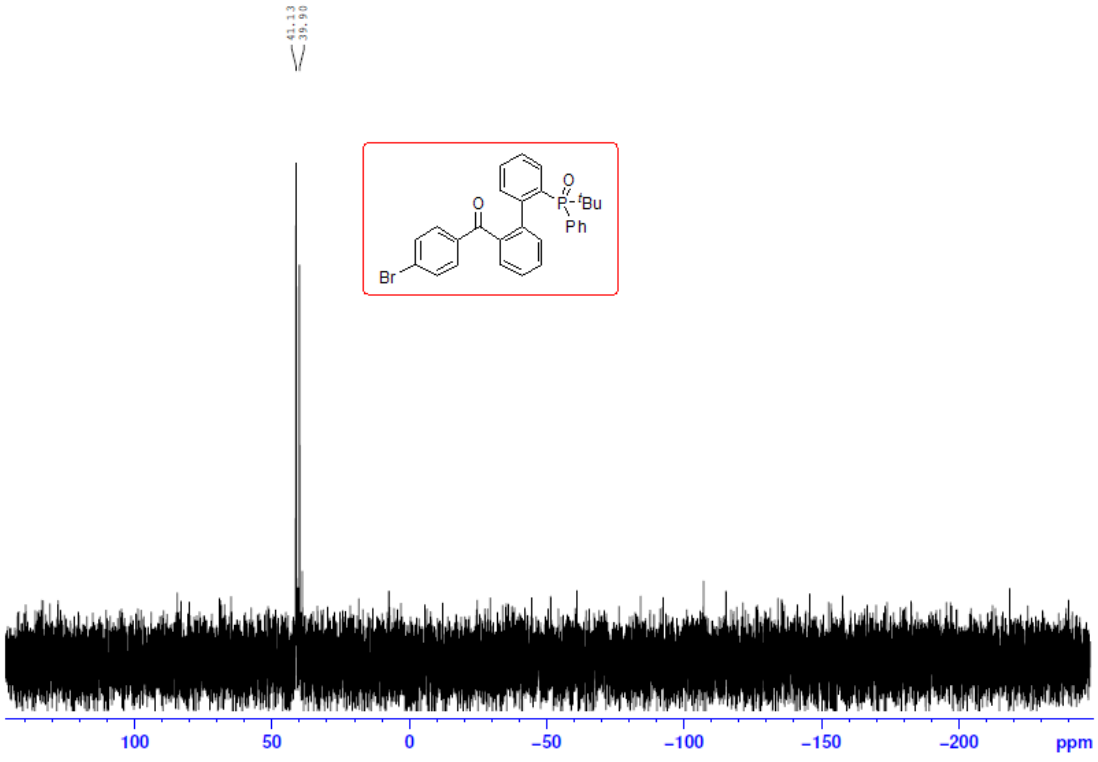

O
P
tBu
Ph
O
F3C

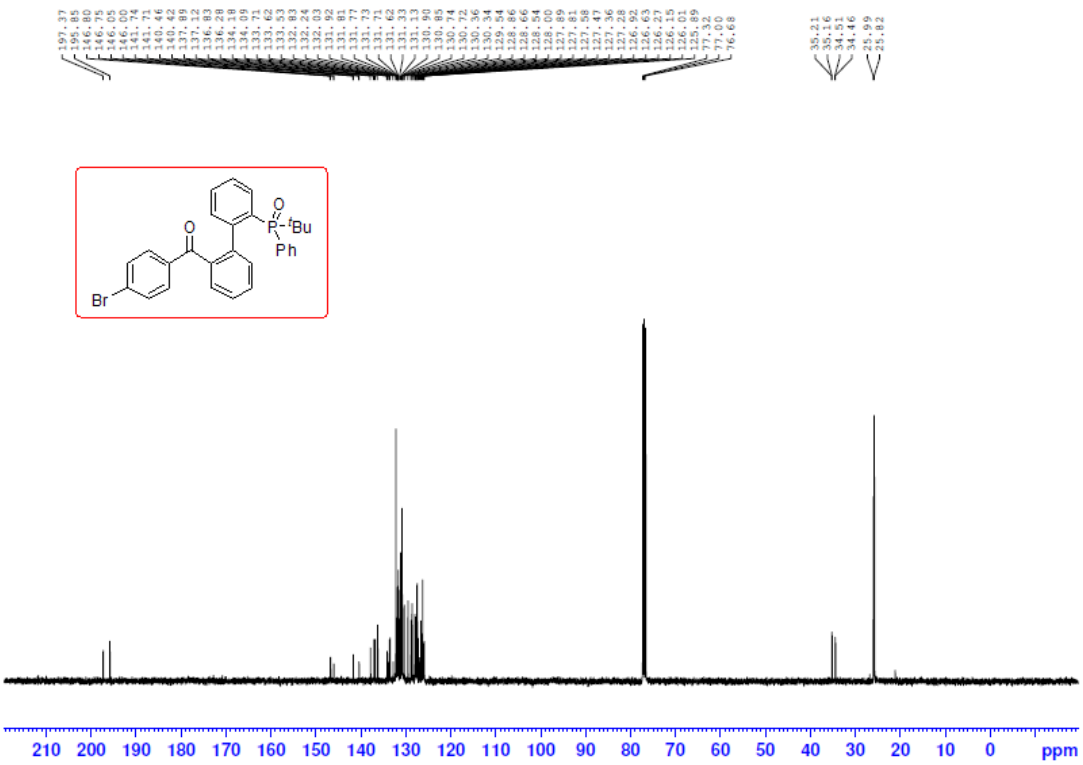

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

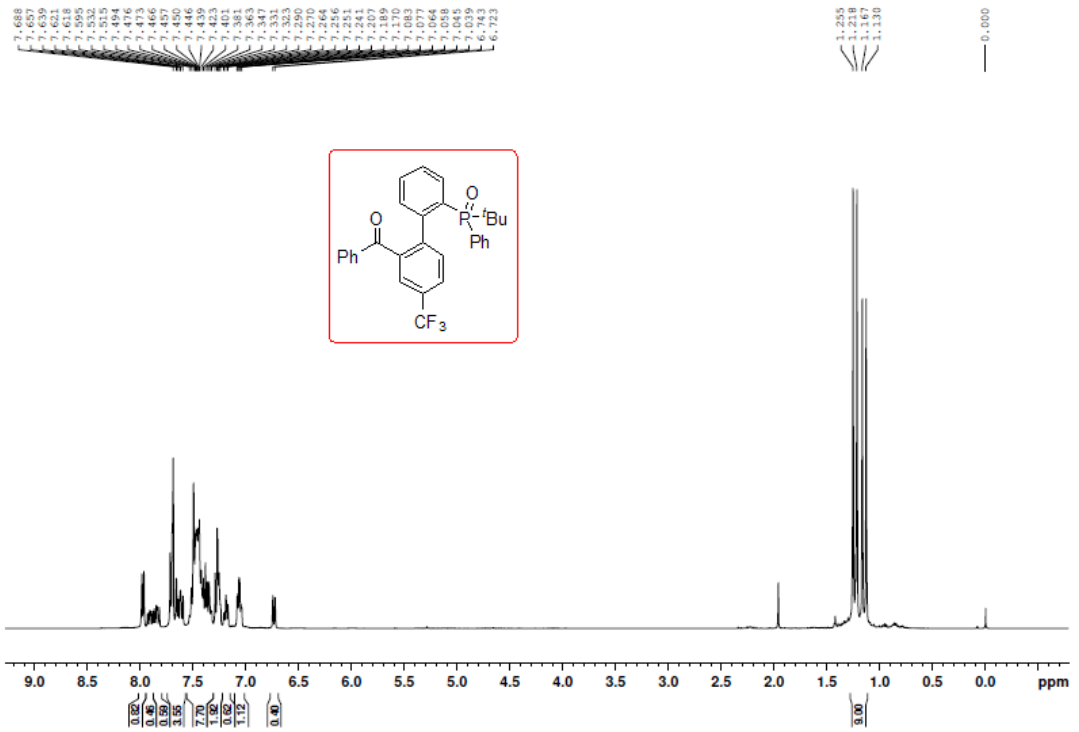

O
P
tBu
Ph
O
F3C

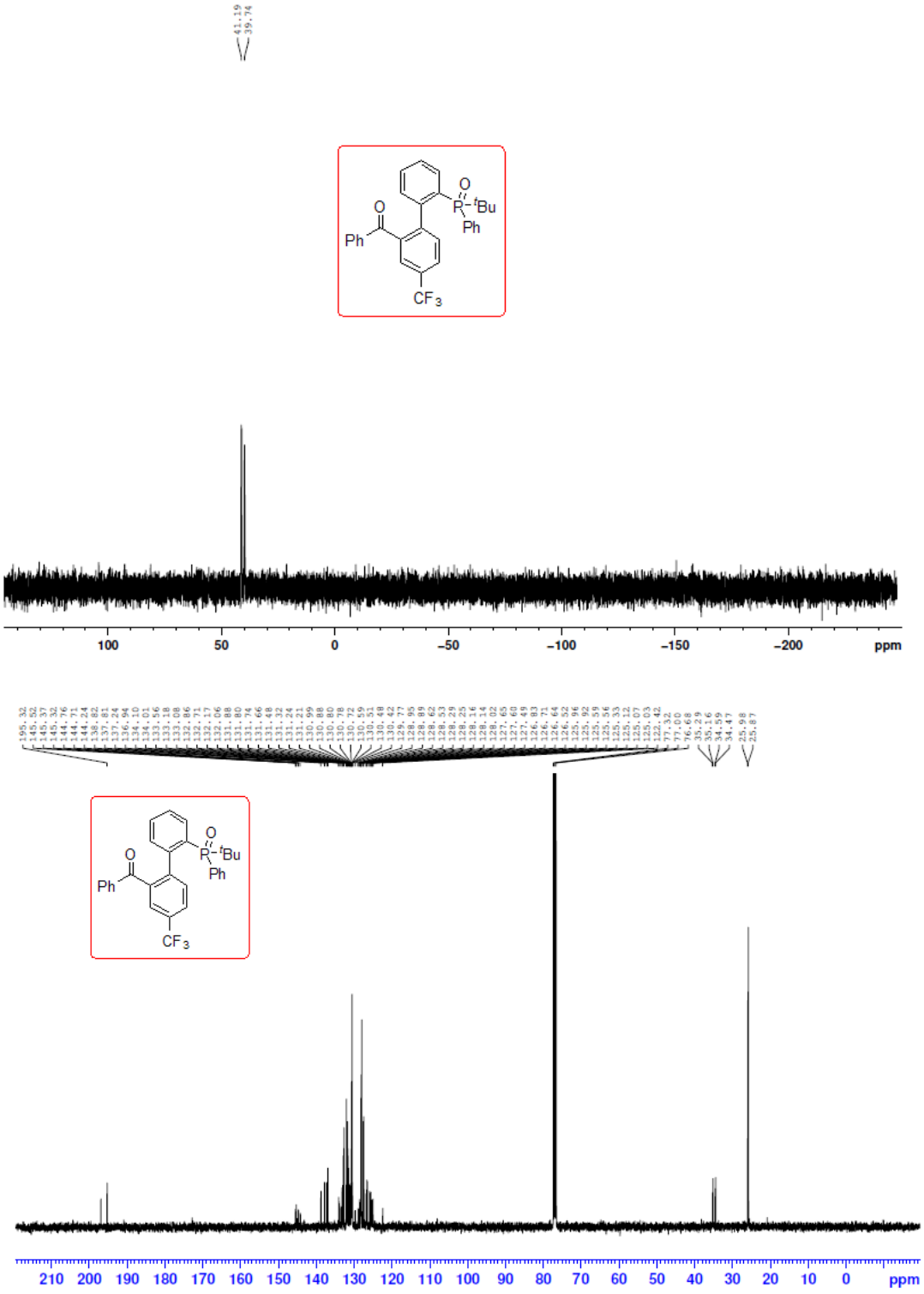

0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 ppm

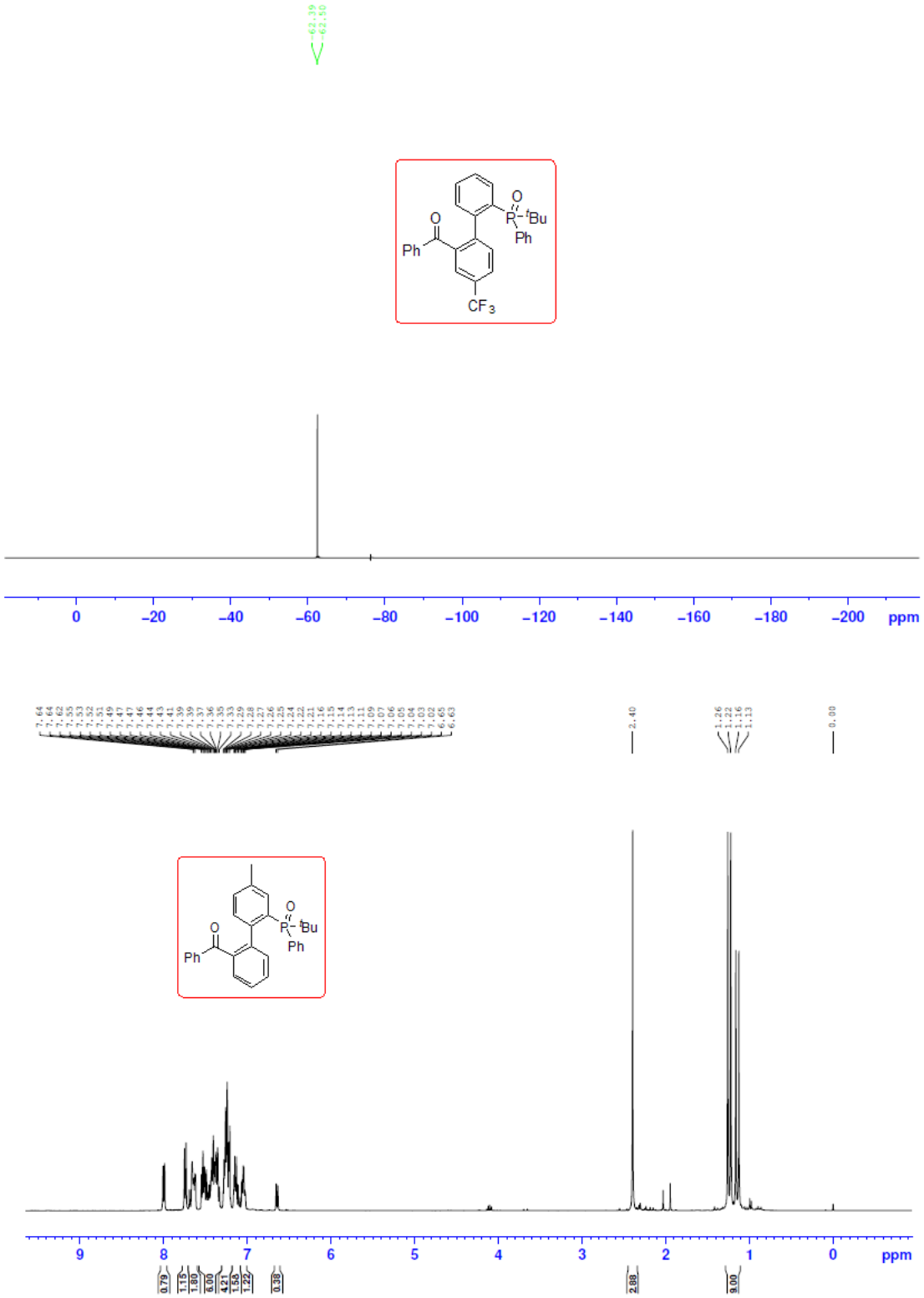

Br
O
P
tBu
Ph
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

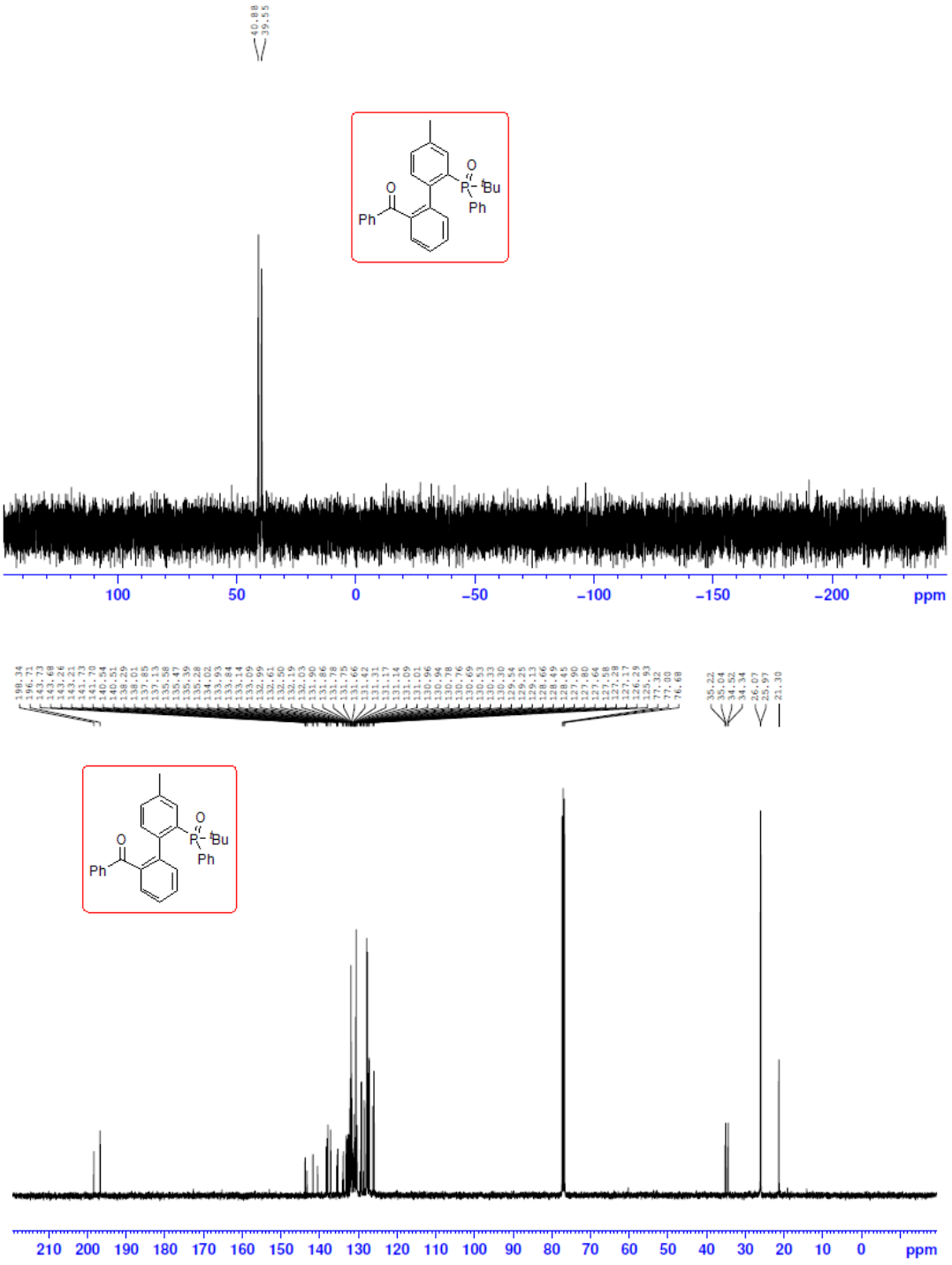

Br
O
P
tBu
Ph
100
50
0
−50
−100
−150
−200
ppm

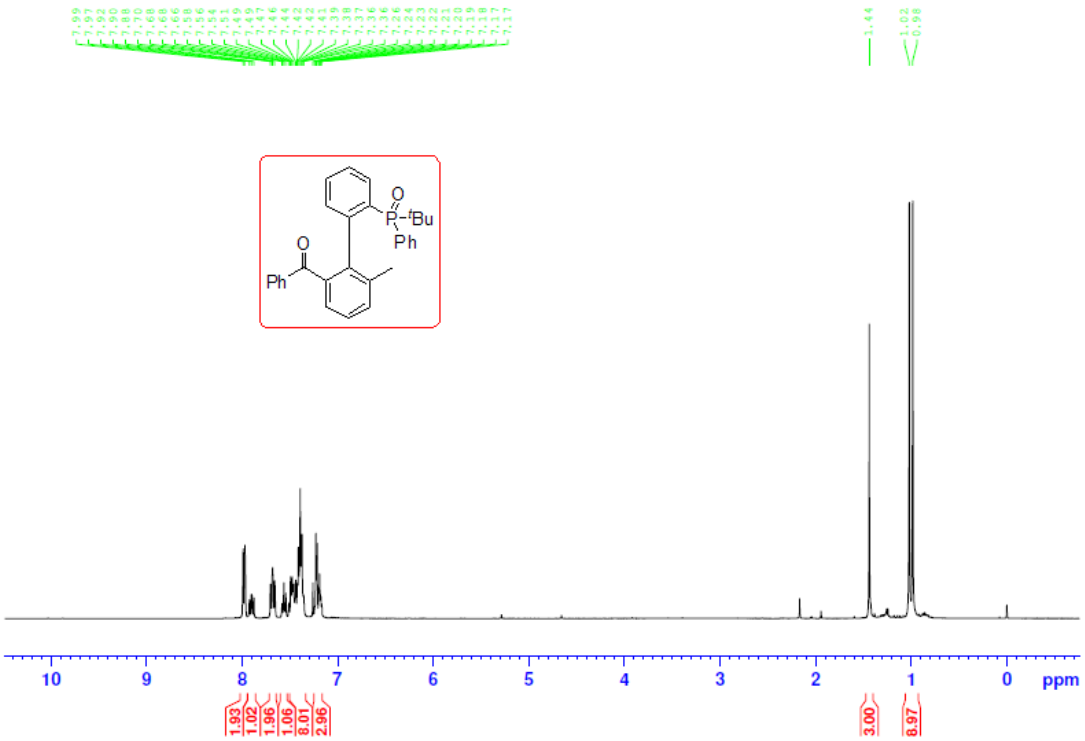

O
P
tBu
Ph
O
Br
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

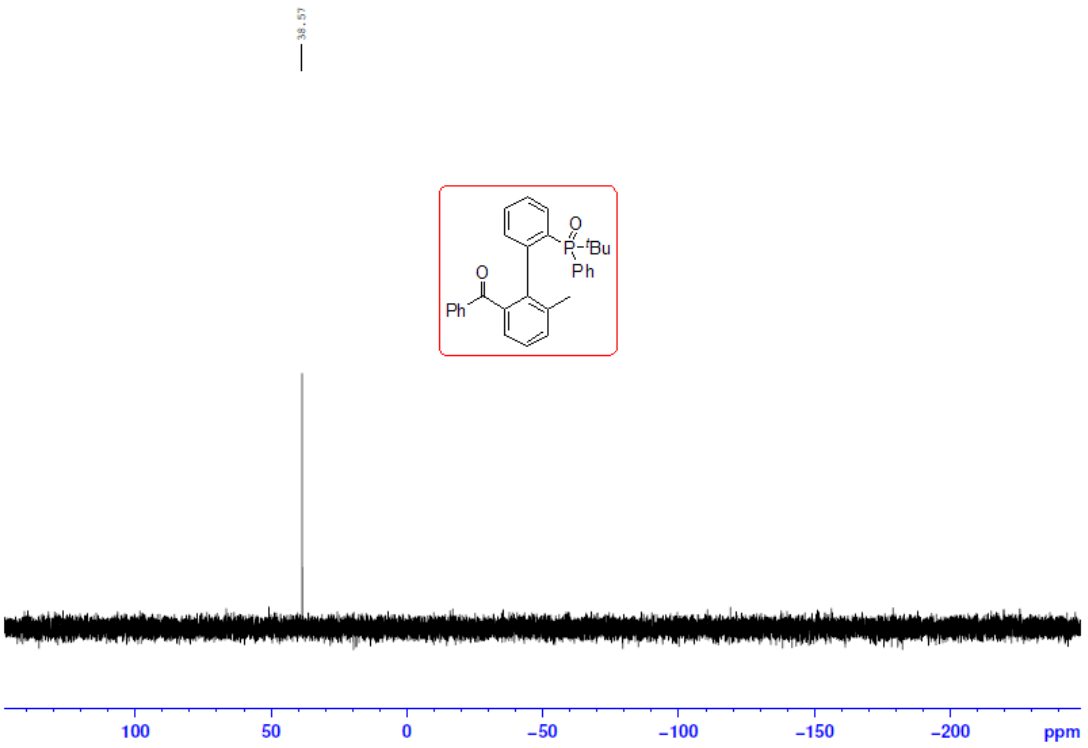

O
P
tBu
Ph
O
Ph
CF3
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

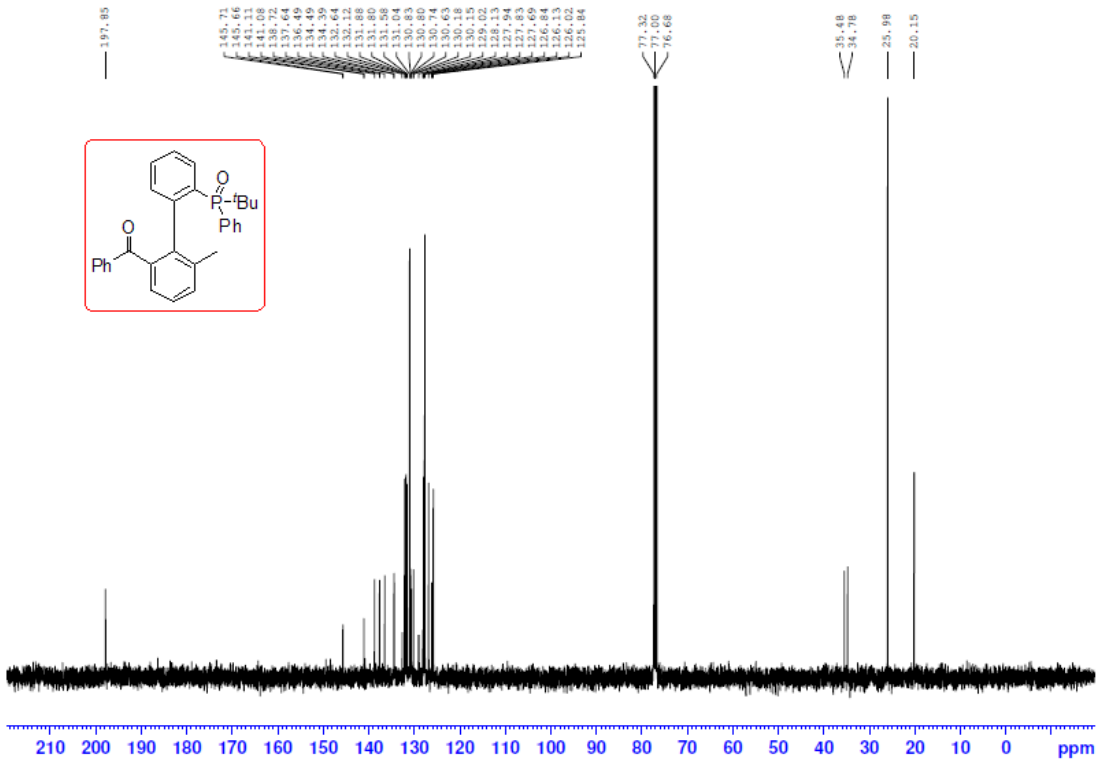

41.19
39.74
O
P
tBu
Ph
O
Ph
CF3
100
50
0
−50
−100
−150
−200
ppm
195.32
145.52
145.37
145.32
144.76
144.71
144.24
138.82
137.81
137.24
136.94
134.10
134.01
133.56
133.18
133.08
132.86
132.71
132.17
132.06
131.88
131.80
131.74
131.66
131.48
131.32
131.24
131.21
130.99
130.88
130.80
130.78
130.72
130.59
130.51
130.48
130.42
129.77
128.95
128.89
128.62
128.53
128.29
128.25
128.14
128.02
127.65
127.60
127.49
127.31
126.71
126.64
126.52
125.96
125.92
125.59
125.56
125.33
125.12
125.07
125.03
122.42
77.32
77.00
76.68
35.29
35.16
34.59
34.47
25.98
25.87
O
P
tBu
Ph
O
Ph
CF3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

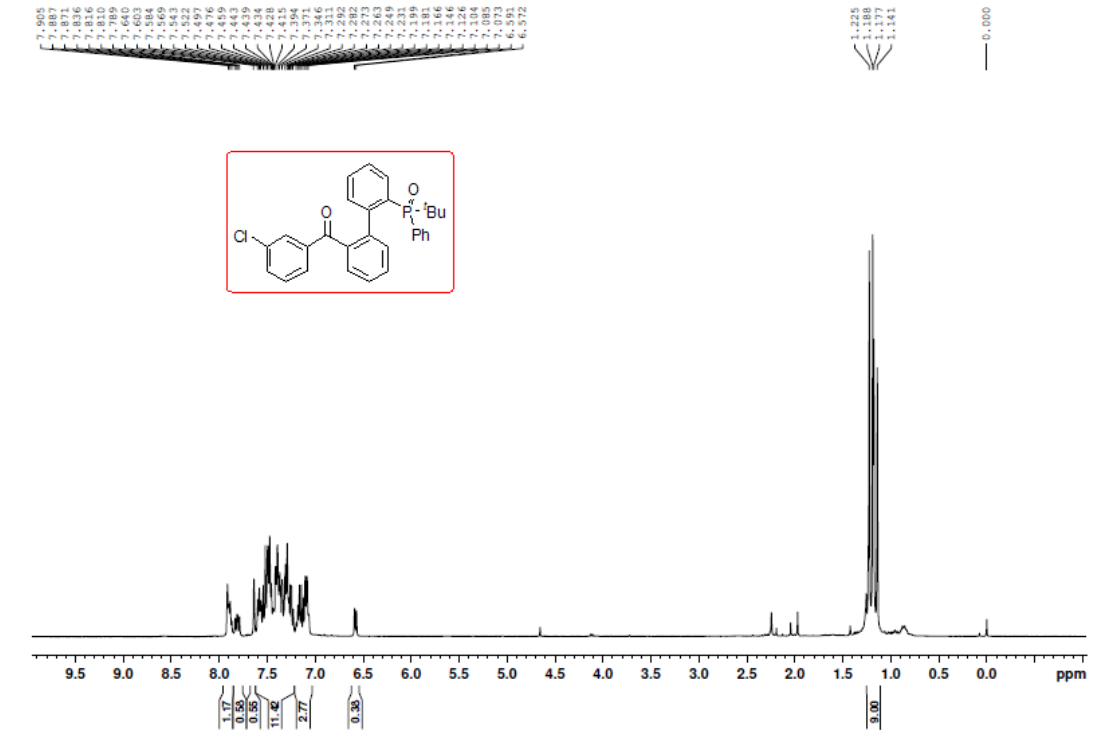

-62.39
-62.50
O
P
tBu
Ph
O
Ph
CF3
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
ppm
2.40
1.26
1.22
1.16
1.13
0.00
9
8
7
6
5
4
3
2
1
0
ppm
0.79
1.15
1.80
8.00
4.21
1.58
1.22
0.38
2.08
9.00

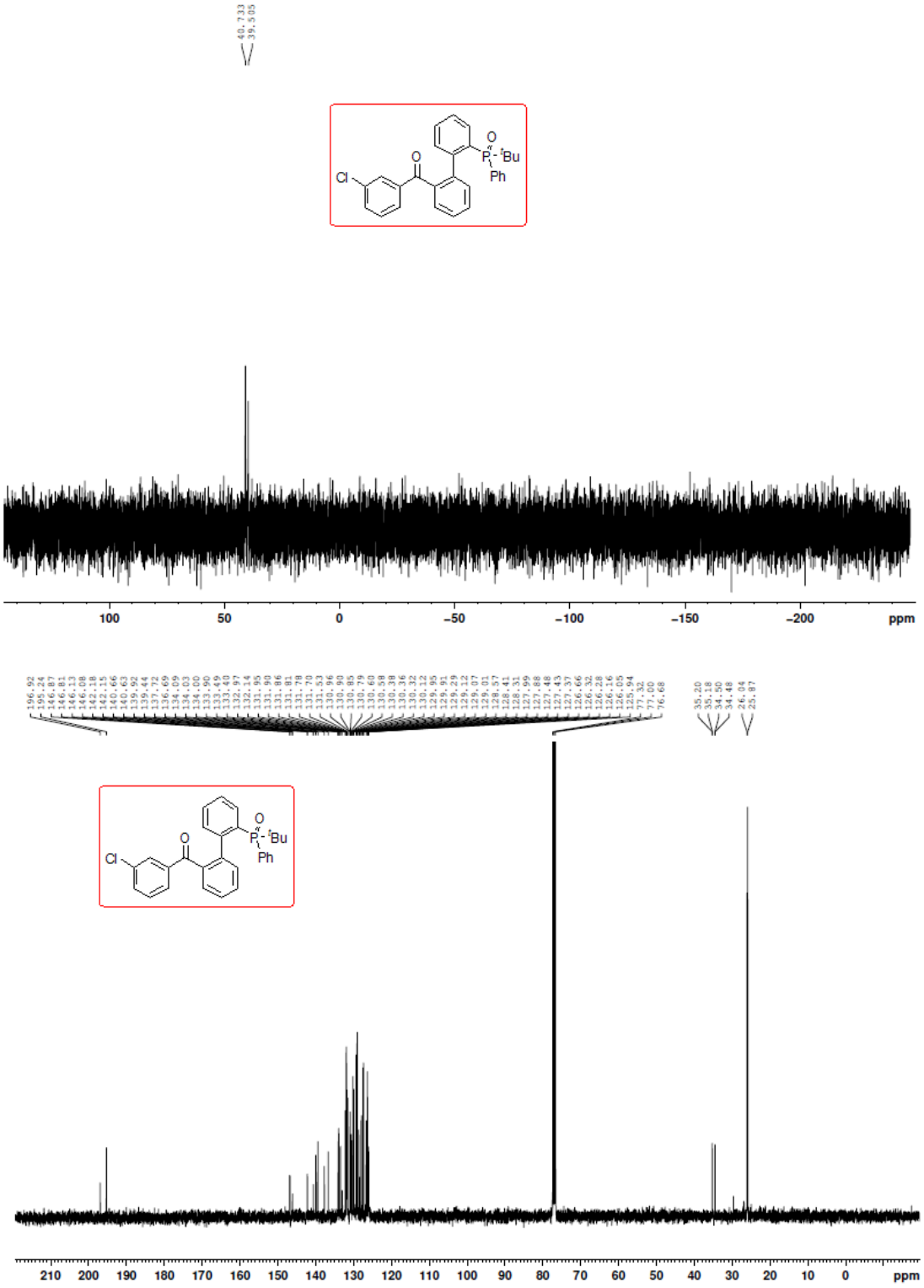

40.88
39.55
O
P
tBu
Ph
O
Ph
100
50
0
−50
−100
−150
−200
ppm
198.34
196.71
143.73
143.68
143.26
142.21
141.73
141.70
140.54
140.51
138.29
138.01
137.85
137.13
135.58
135.47
135.39
135.28
134.02
133.95
133.84
133.14
133.09
132.99
132.61
132.50
132.19
132.03
131.90
131.86
131.78
131.75
131.66
131.42
131.31
131.17
131.14
131.09
131.01
130.96
130.94
130.78
130.76
130.69
130.53
130.33
130.30
129.54
129.25
129.13
128.66
128.49
128.45
127.90
127.80
127.64
127.58
127.28
127.17
126.29
125.93
77.32
77.00
76.68
35.22
35.04
34.52
34.34
26.07
25.97
21.30
O
P
tBu
Ph
O
Ph
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

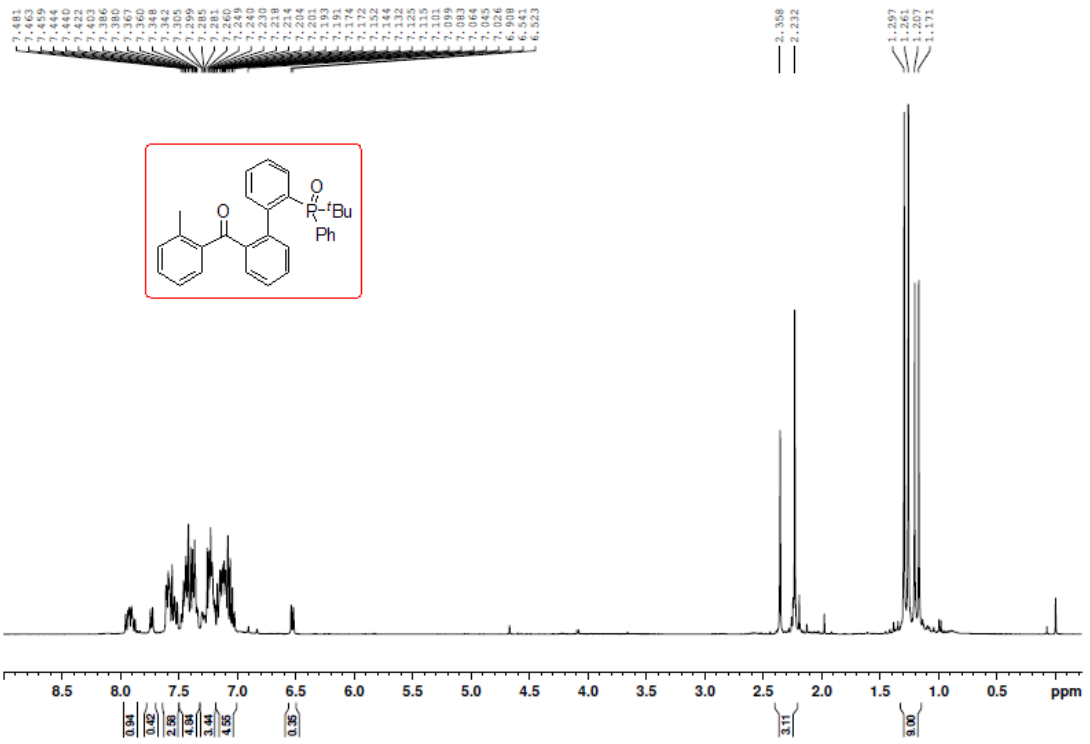

O
P
tBu
Ph
O
Ph
10
9
8
7
6
5
4
3
2
1
0
ppm
1.93
1.02
1.96
1.06
8.01
2.96
3.00
8.97

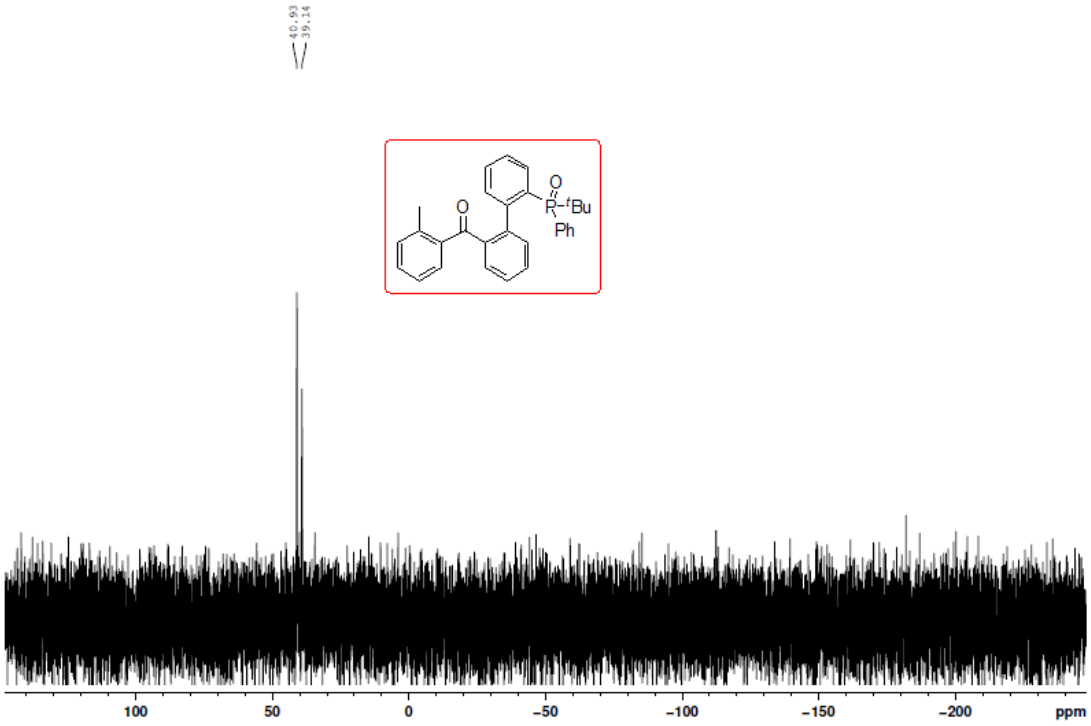

38.57
O
P
tBu
Ph
O
Ph
100
50
0
−50
−100
−150
−200
ppm

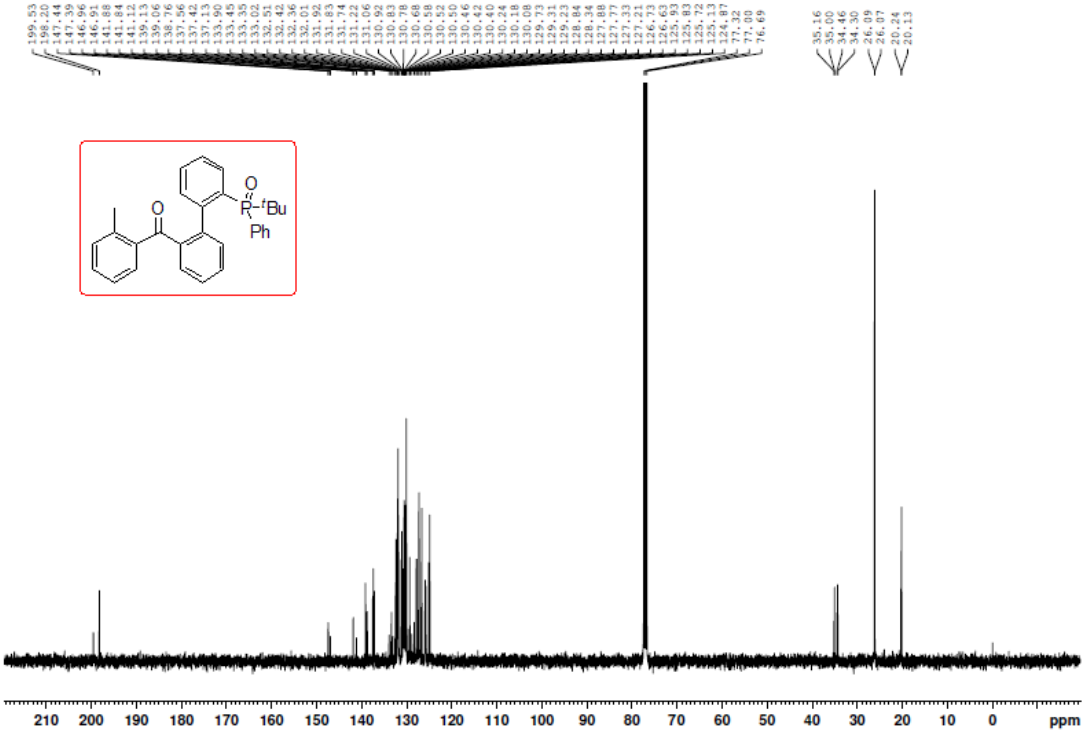

197.85
145.72
145.46
143.11
141.08
138.72
137.64
136.49
134.49
134.39
132.64
132.22
131.88
131.80
131.58
131.04
130.83
130.80
130.74
130.65
130.18
130.15
129.02
128.13
127.94
127.83
127.69
126.84
126.13
126.02
125.84
77.32
77.00
76.68
35.48
34.78
25.98
20.15
O
P
tBu
Ph
O
Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

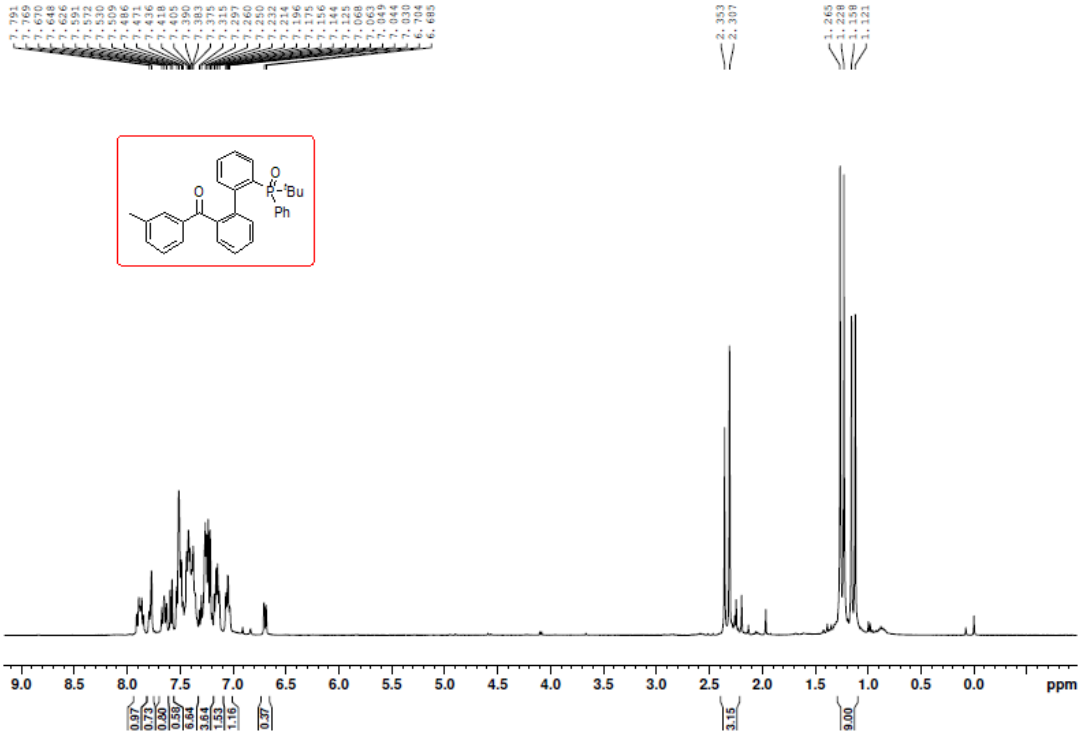

7.905
7.887
7.871
7.836
7.816
7.810
7.789
7.663
7.603
7.584
7.569
7.543
7.522
7.497
7.476
7.459
7.443
7.439
7.434
7.428
7.415
7.394
7.371
7.346
7.311
7.292
7.282
7.273
7.263
7.249
7.231
7.199
7.181
7.166
7.146
7.126
7.104
7.085
7.073
6.591
6.572
1.225
1.208
1.177
1.141
0.000
O
P
tBu
Ph
Cl
O
9.5 9.0 8.5 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 ppm
1.17
0.58
0.55
11.42
2.77
0.38
9.00

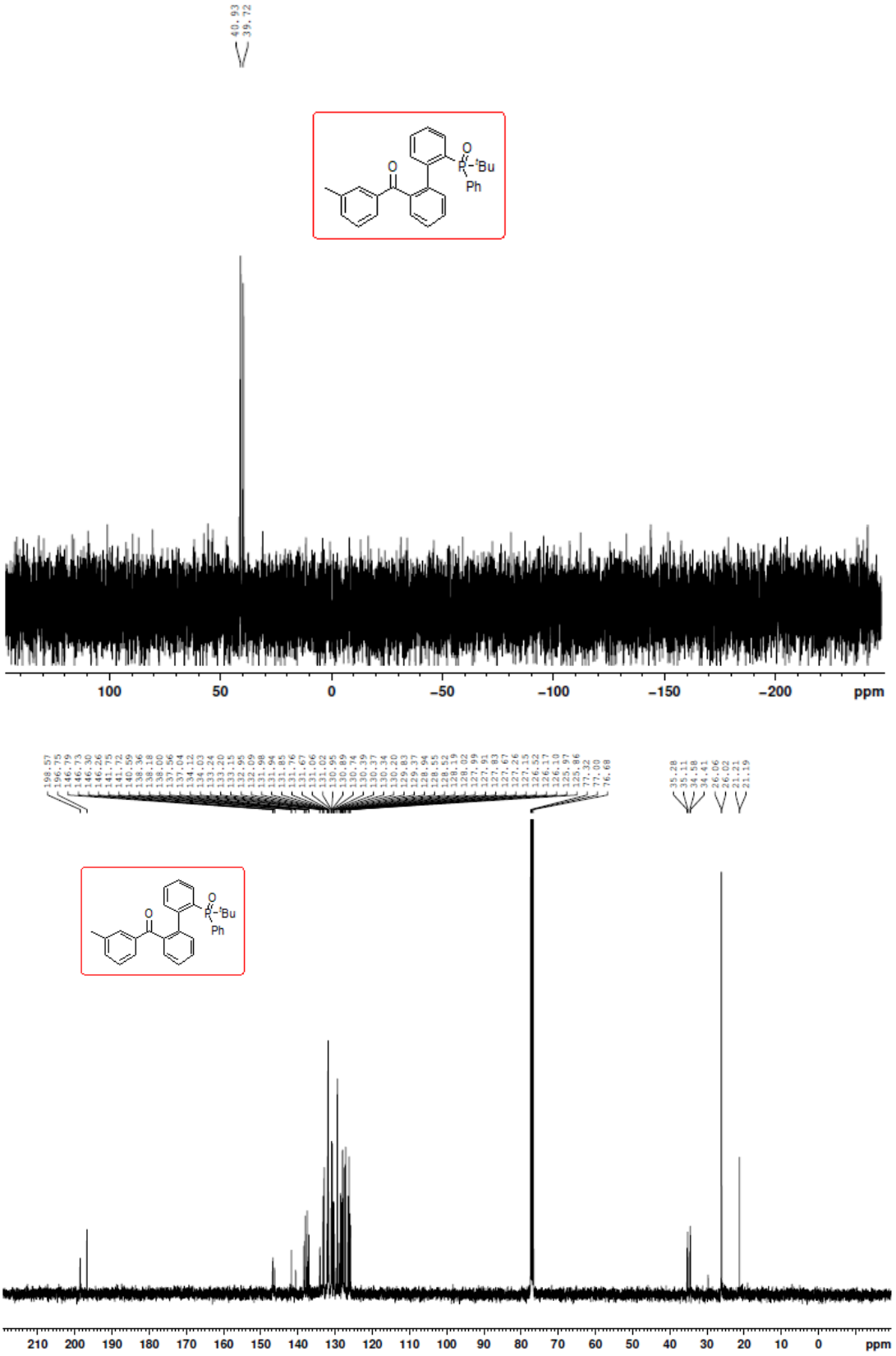

40.733
39.505
Cl
O
O
P
tBu
Ph
100
50
0
−50
−100
−150
−200
ppm
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

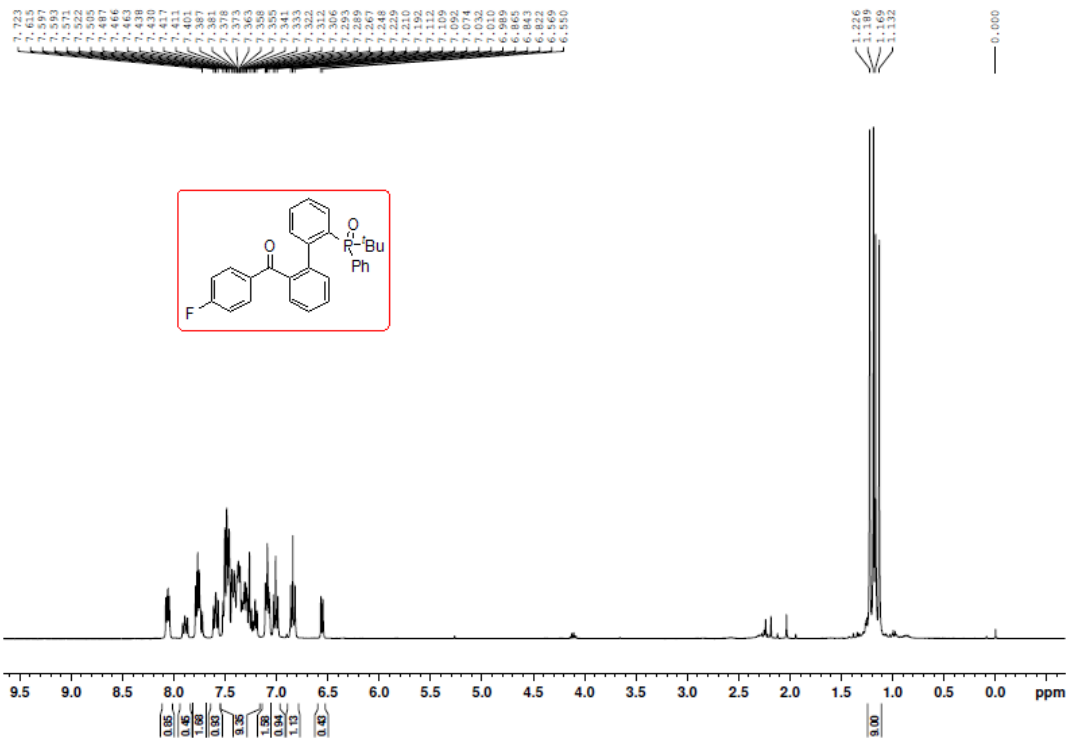

7.481
7.463
7.459
7.444
7.440
7.422
7.403
7.386
7.380
7.367
7.360
7.348
7.342
7.305
7.299
7.285
7.281
7.266
7.249
7.240
7.230
7.218
7.214
7.204
7.201
7.193
7.191
7.174
7.172
7.152
7.144
7.132
7.125
7.115
7.101
7.099
7.083
7.064
7.045
7.026
6.908
6.541
6.523
2.358
2.232
1.297
1.261
1.207
1.171
O
O
P
tBu
Ph
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
ppm
0.84
0.42
2.58
4.04
3.44
4.55
0.35
3.11
9.00

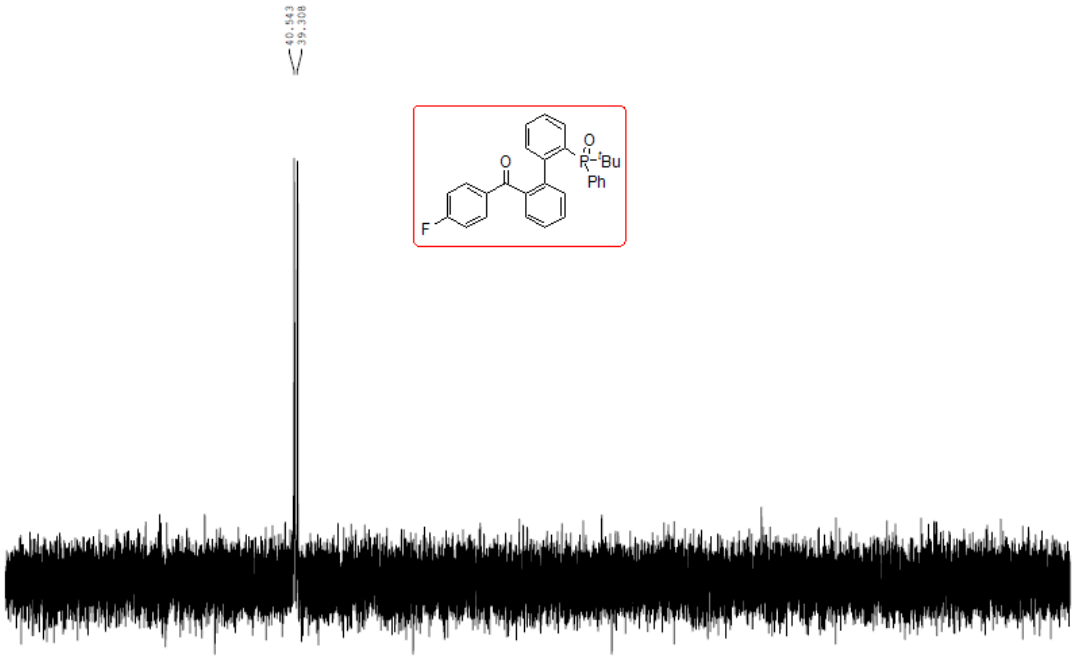

40.93
39.14
O
O
P
tBu
Ph
100
50
0
−50
−100
−150
−200
ppm

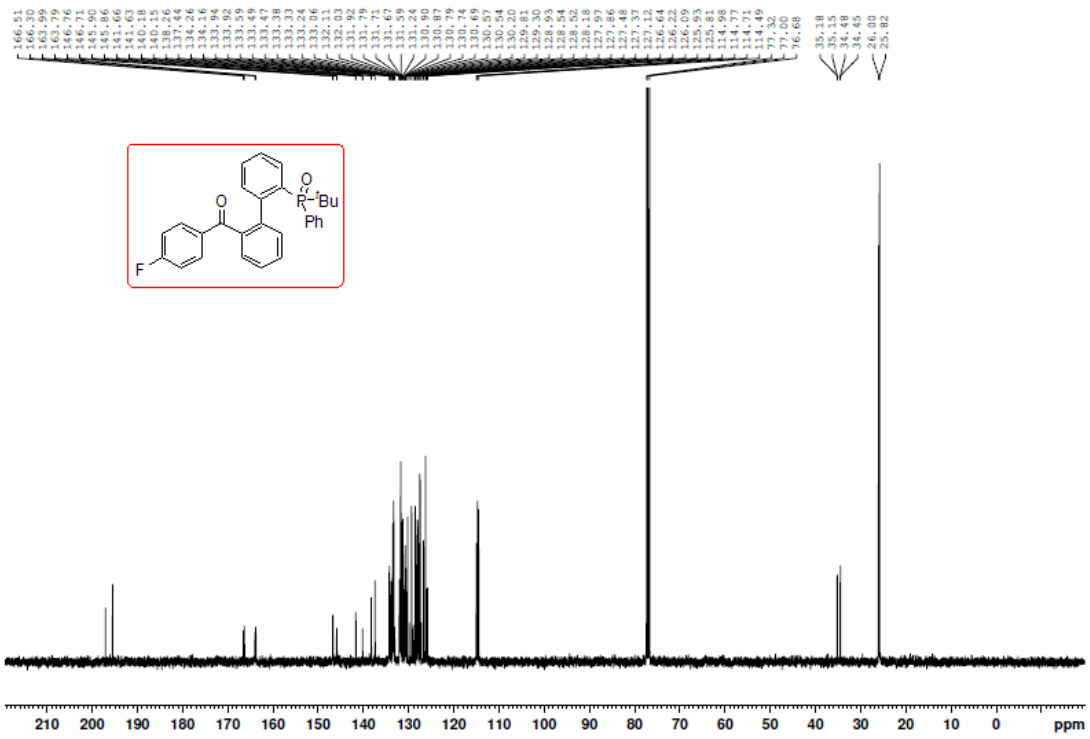

O
O
P
tBu
Ph
ppm

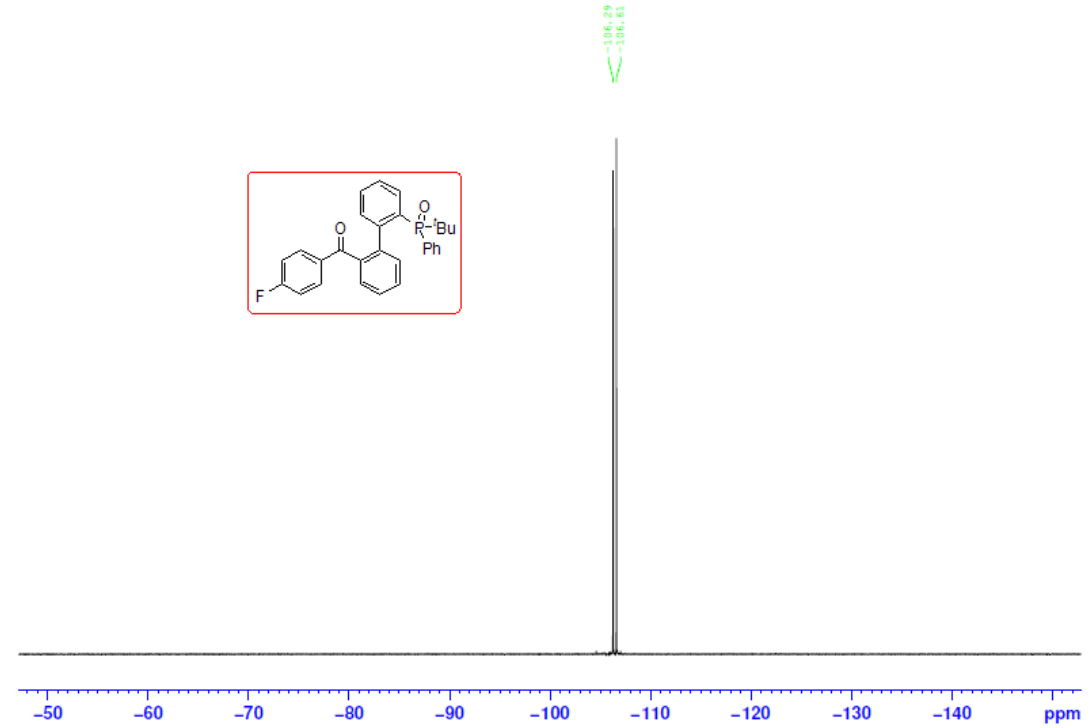

O
O
P
tBu
Ph
ppm

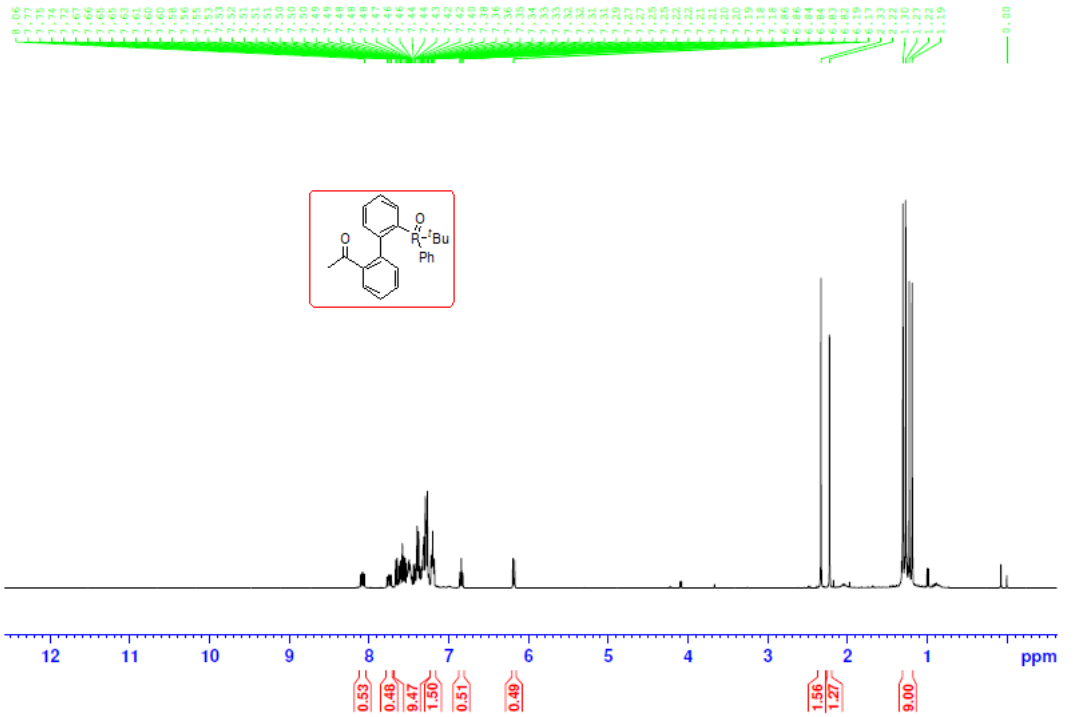

60.83
39.72
O
P
tBu
Ph
100
50
0
−50
−100
−150
−200
ppm
198.57
196.75
146.79
146.73
146.30
146.26
141.75
141.72
140.59
138.36
138.18
138.00
137.96
137.04
134.22
134.03
133.24
133.20
133.15
132.95
132.09
131.98
131.94
131.85
131.76
131.67
131.06
131.02
130.95
130.89
130.74
130.39
130.37
130.34
130.20
129.83
129.37
128.94
128.55
128.52
128.19
128.12
127.99
127.91
127.83
127.67
127.26
127.15
126.52
126.37
126.10
125.97
125.86
77.32
77.00
76.68
35.28
35.11
34.58
34.41
26.06
26.02
21.21
21.19
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

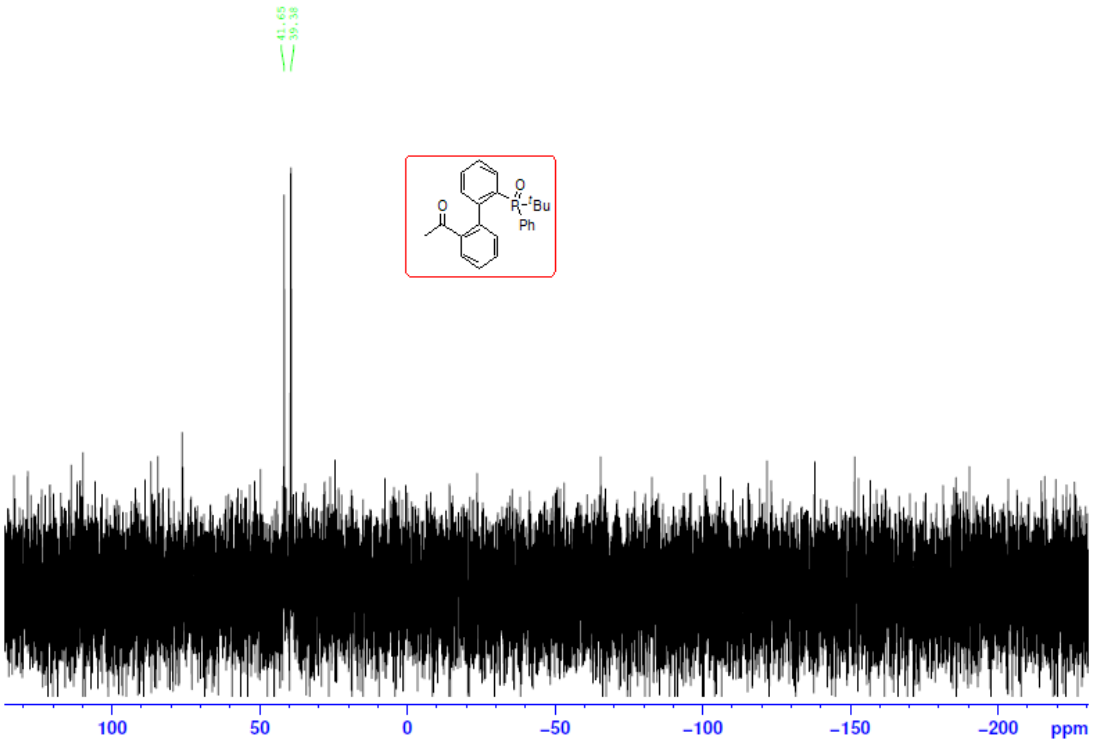

7.723
7.615
7.597
7.593
7.571
7.522
7.505
7.487
7.466
7.463
7.438
7.430
7.417
7.411
7.401
7.387
7.381
7.378
7.373
7.363
7.358
7.355
7.341
7.333
7.322
7.312
7.306
7.293
7.289
7.267
7.248
7.229
7.210
7.192
7.112
7.109
7.092
7.074
7.032
7.010
6.989
6.989
6.865
6.843
6.822
6.569
6.550
1.226
1.198
1.169
1.132
0.000
O
R
tBu
Ph
F
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
ppm
0.85
0.45
1.68
0.93
0.35
1.86
0.94
1.13
0.49
9.00

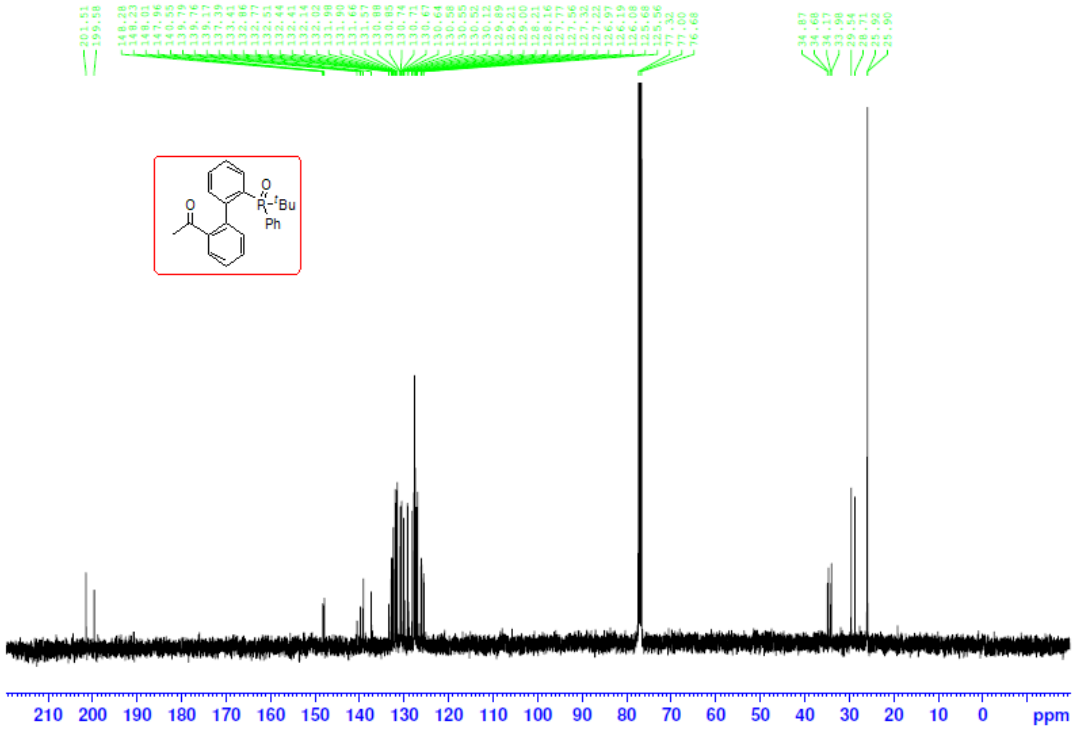

40.543
39.308
O
R
tBu
Ph
F

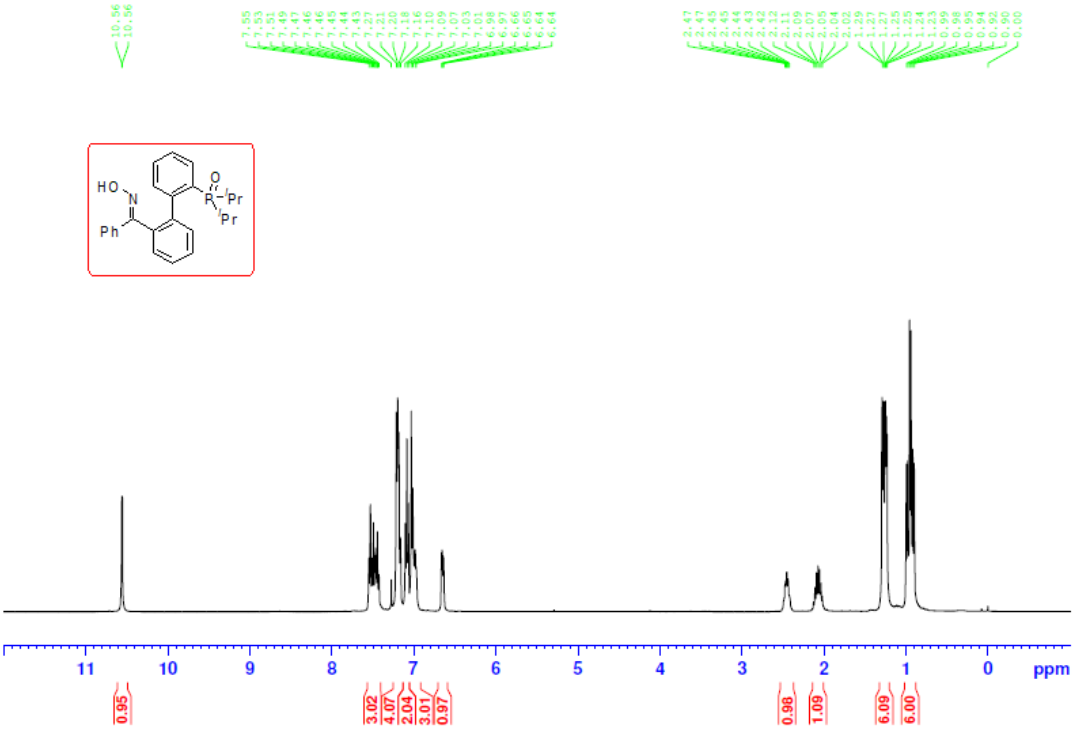

O
P
tBu
Ph
O
F
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

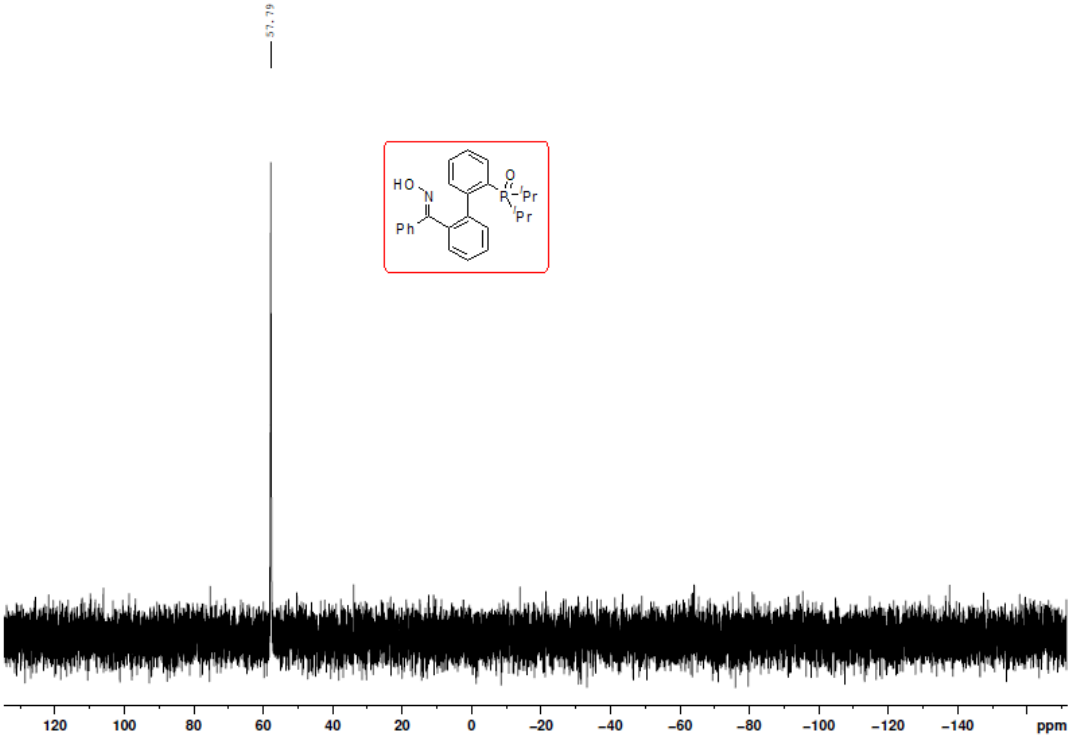

-106.29
-106.61
O
P
tBu
Ph
O
F
-50 -60 -70 -80 -90 -100 -110 -120 -130 -140 ppm

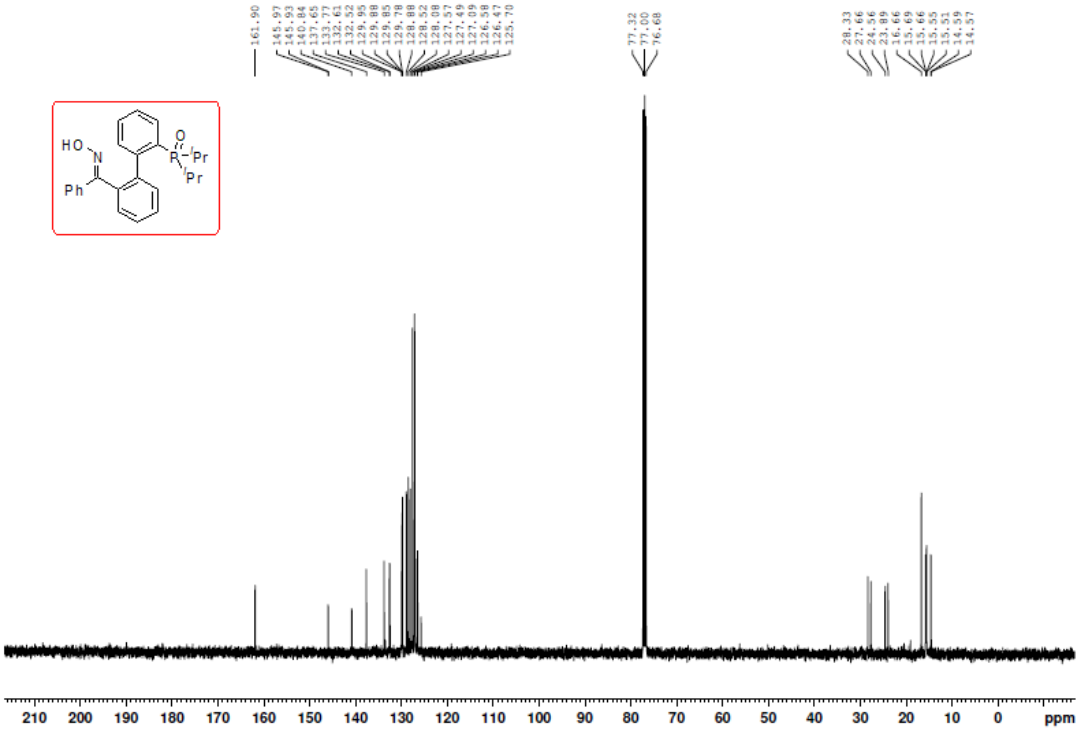

O
P
tBu
Ph
O
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.53
0.48
9.47
1.50
0.51
0.49
1.56
1.27
9.00

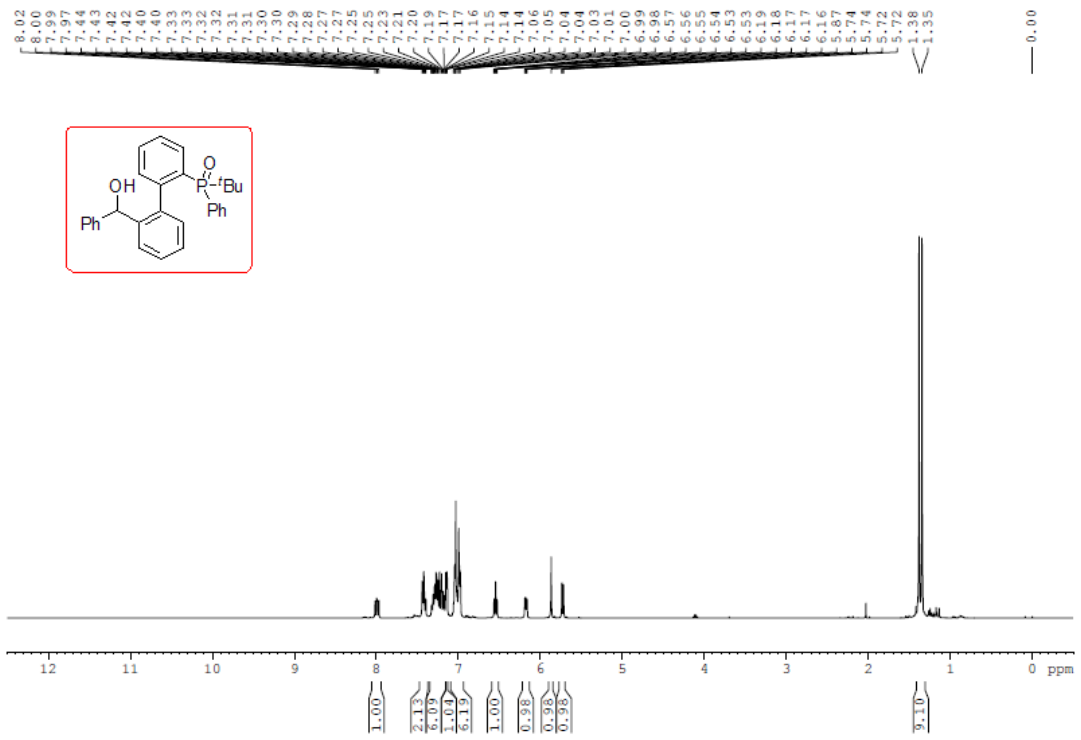

41.65
39.38
O
P
tBu
Ph
O
100
50
0
−50
−100
−150
−200
ppm

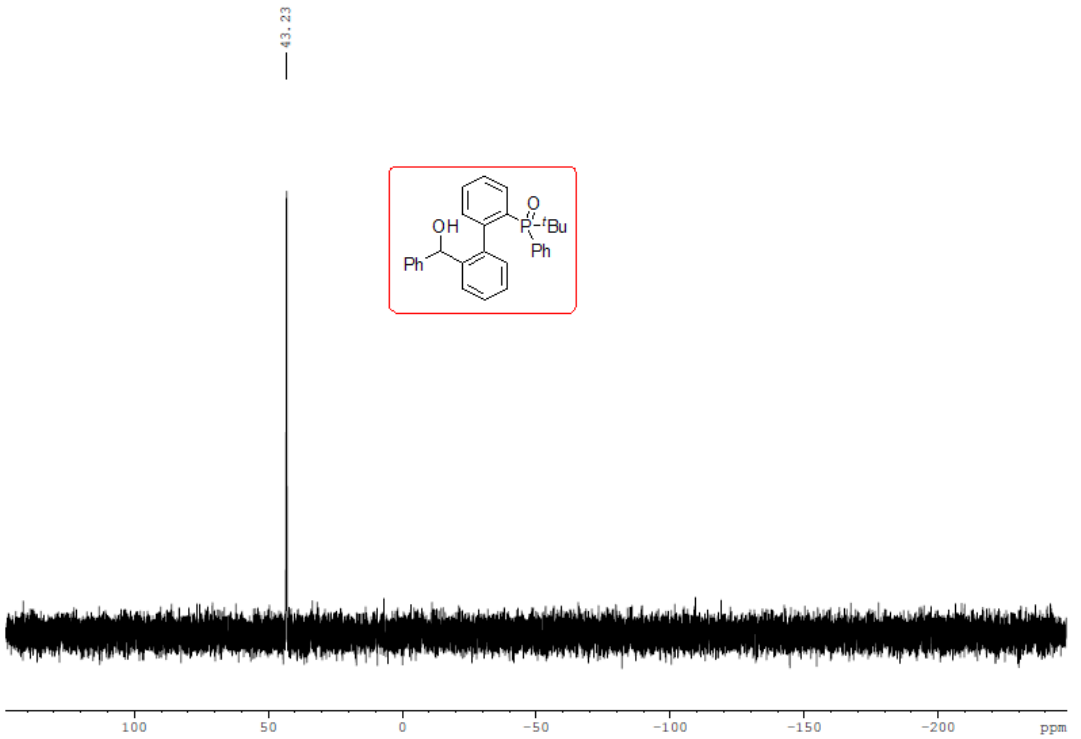

O
O
P
tBu
Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

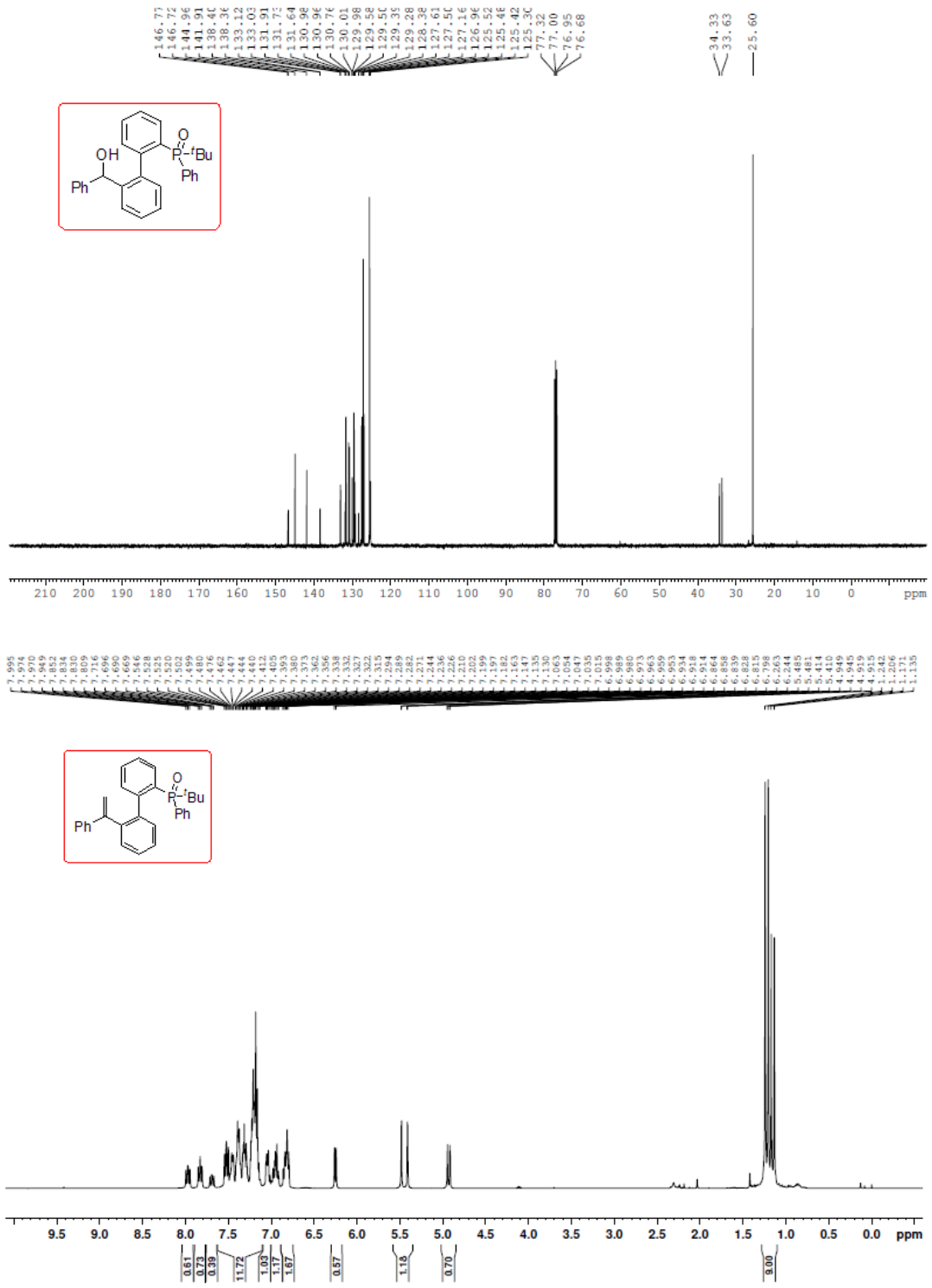

HO
N
Ph
O
P
iPr
iPr
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.95
3.02
4.07
2.04
3.01
0.97
0.98
1.09
6.09
6.00


57.79
HO
N
Ph
O
P
iPr
iPr
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
ppm


161.90
145.97
145.93
140.84
137.65
133.77
132.61
132.52
129.95
129.88
129.85
129.78
128.88
128.52
128.08
127.57
127.49
127.39
126.58
126.47
125.70
77.32
77.00
76.68
28.33
27.66
24.56
23.89
16.66
15.69
15.66
15.55
15.51
14.59
14.57
HO
N
Ph
O
P
iPr
iPr
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.02
8.00
7.99
7.97
7.44
7.43
7.42
7.42
7.40
7.40
7.33
7.33
7.32
7.32
7.31
7.31
7.30
7.30
7.29
7.28
7.27
7.27
7.25
7.25
7.23
7.21
7.20
7.19
7.17
7.17
7.16
7.15
7.14
7.14
7.06
7.05
7.04
7.04
7.03
7.01
7.00
6.99
6.98
6.57
6.56
6.55
6.54
6.53
6.53
6.19
6.18
6.17
6.17
6.16
5.87
5.74
5.74
5.72
5.72
1.38
1.35
0.00
OH
Ph
O
P
tBu
Ph
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.13
6.09
1.04
6.19
1.00
0.98
0.98
0.98
9.10


43.23
OH
Ph
O
P
tBu
Ph
100
50
0
-50
-100
-150
-200
ppm


OH
Ph
O
P
tBu
Ph
34.33
33.63
25.60
77.32
77.00
76.95
76.68
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
Ph
O
P
tBu
Ph
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 ppm
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0.89
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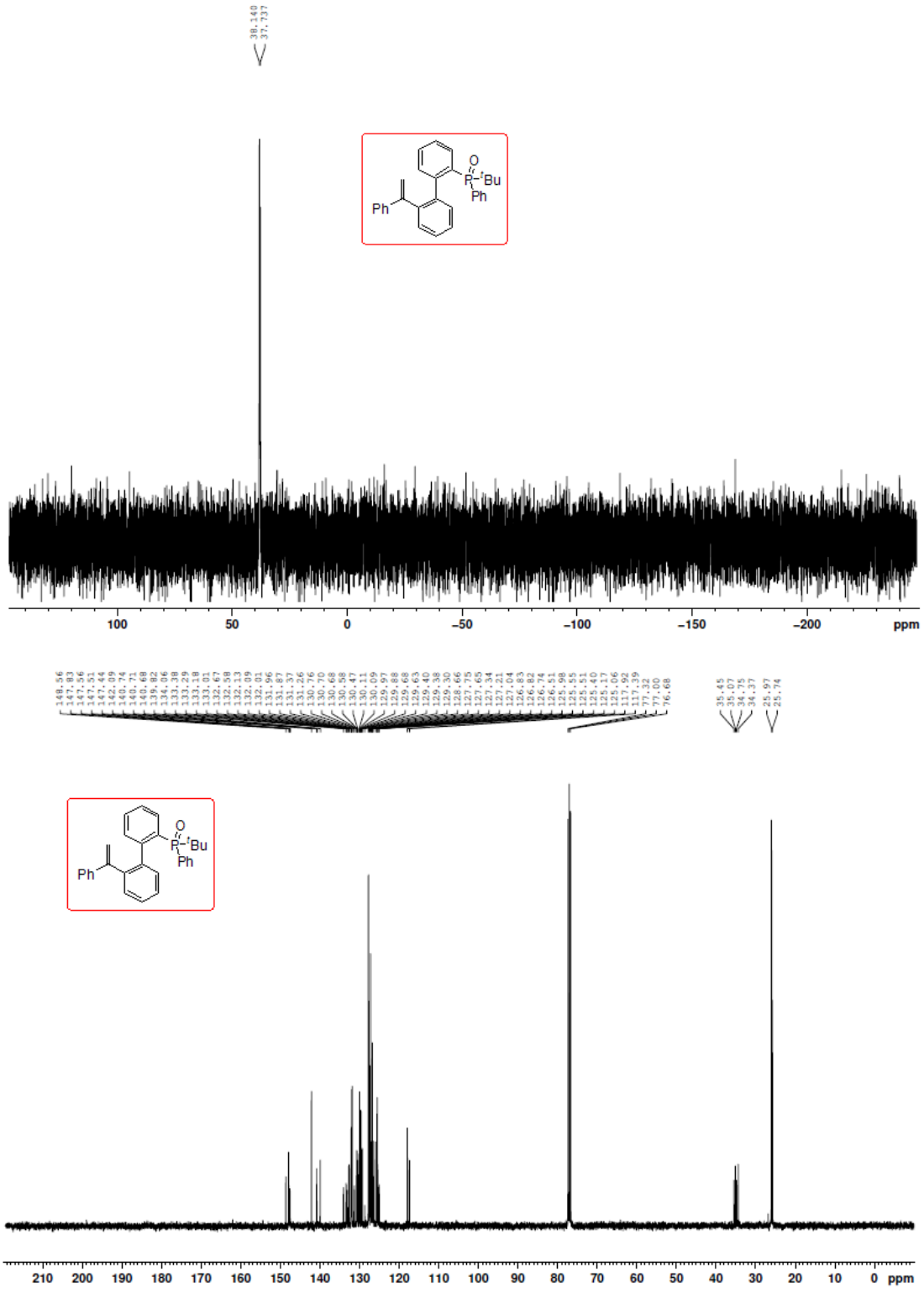
1.03
1.17
1.67
0.57
1.18
0.70
9.00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P
tBu
Ph
Ph
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147.56
147.21
142.44
142.09
140.74
140.71
140.68
139.82
134.06
133.38
133.29
133.18
133.01
132.67
132.58
132.13
132.09
132.01
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131.87
131.37
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130.58
130.47
130.11
130.09
130.07
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129.68
129.63
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129.38
129.30
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127.66
127.34
127.21
127.04
126.83
126.82
126.74
126.51
126.38
125.98
125.55
125.51
125.40
125.17
125.06
117.92
117.39
77.32
77.00
76.68
35.45
35.07
34.75
34.37
25.97
25.74
O
P
tBu
Ph
Ph
210
200
190
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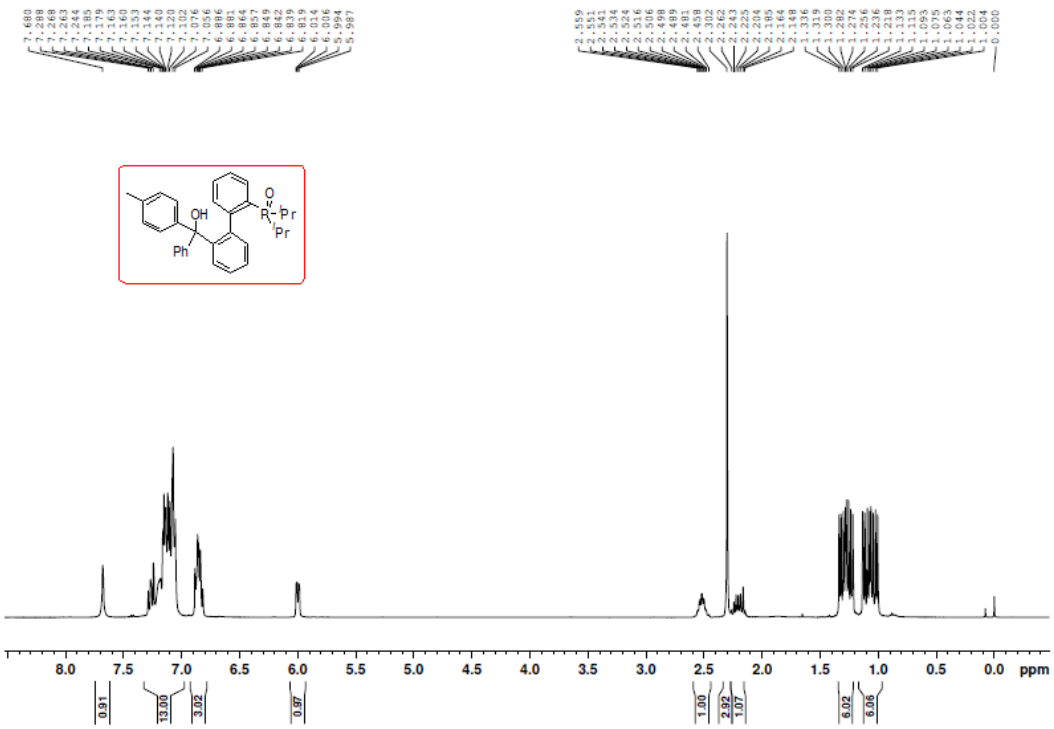
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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O
P
iPr
iPr
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7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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ppm
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3.02
0.97
1.00
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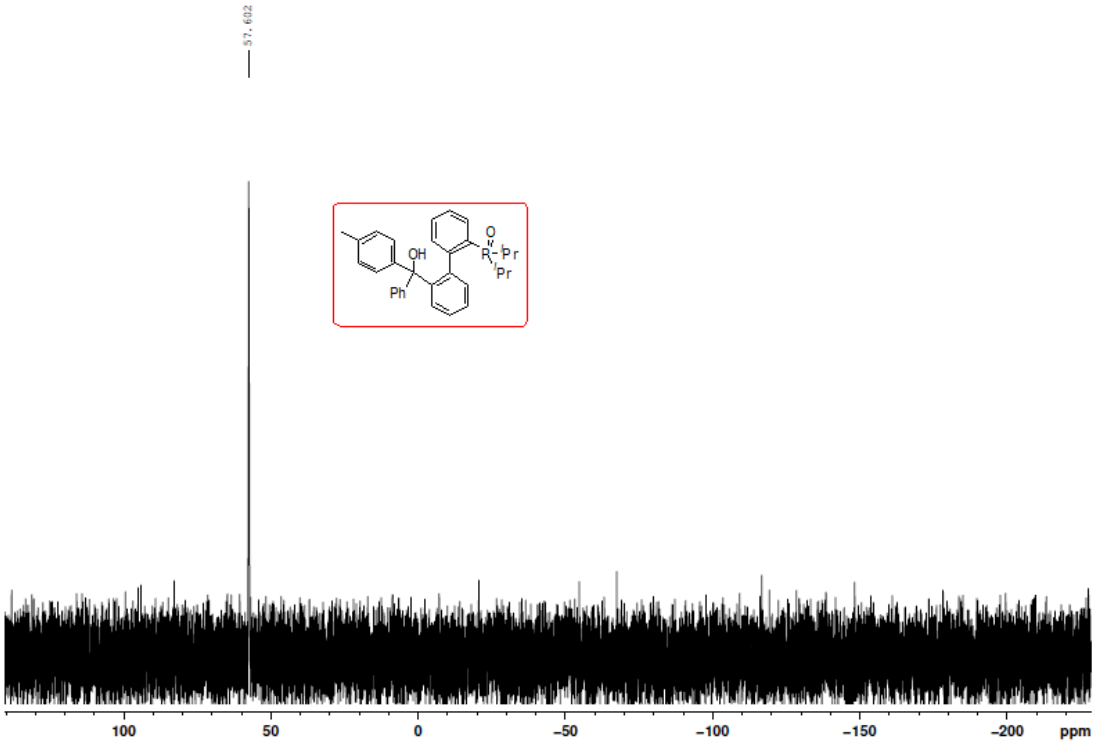
1.07
6.02
6.06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O
P
Pr
Pr
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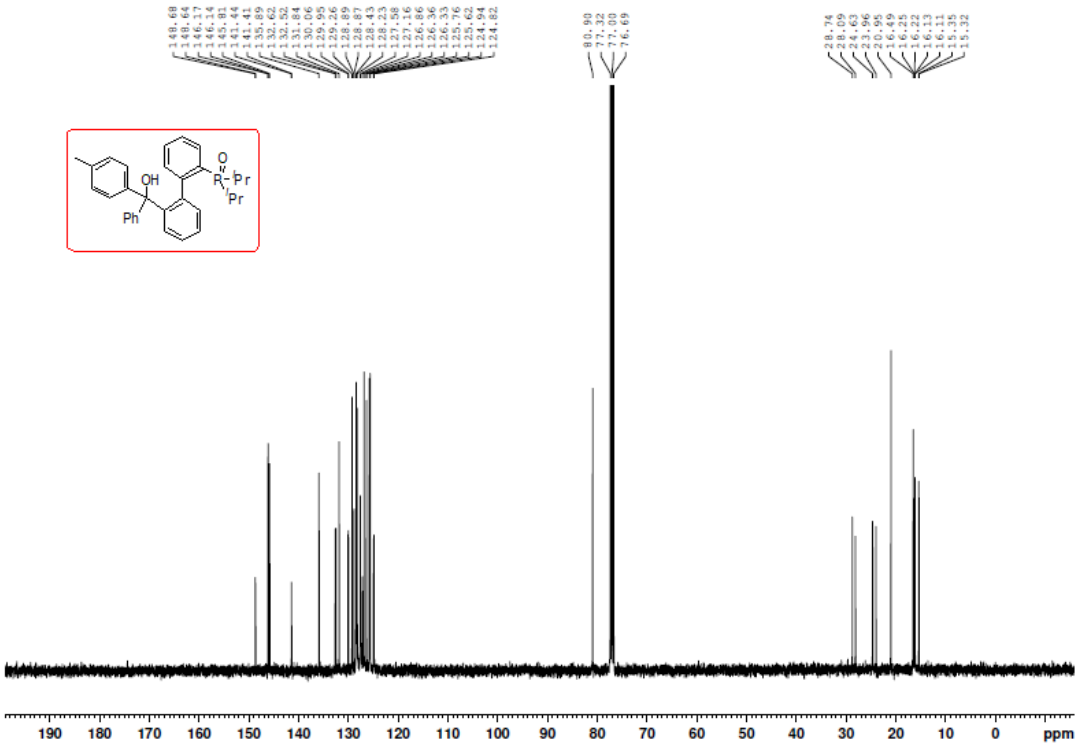
ppm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129.26
128.89
128.87
128.43
128.23
127.58
127.16
126.86
126.36
126.33
125.76
125.62
124.94
124.82
80.90
77.32
77.00
76.69
28.74
28.09
24.63
23.96
20.95
16.49
16.25
16.22
16.13
16.11
15.35
15.32
OH
Ph
O
P
Pr
Pr
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm