Supporting Information

Radical Phosphinylation of α,α-Diaryl Allylic Alcohols with Concomitant 1,2-Aryl Migration

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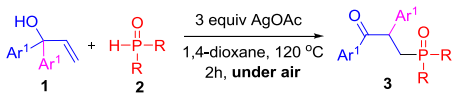
Experimental Section:

General

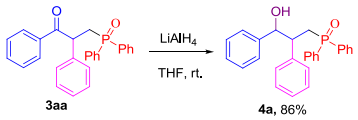
Unless otherwise stated, all reagents were purchased from commercial suppliers and used without further purification. All reactions were carried out in air and using undistilled solvent, without any precautions to exclude air and moisture unless otherwise noted. Melting points were recorded on an Electrothermal digital melting point apparatus. IR spectra were recorded on a FT-IR spectrophotometer using KBr optics. ^{1}H, ^{13}C and ^{31}P NMR spectra were recorded in $CDCl_3$ on 300 MHz, 400 MHz or 600 MHz spectometers. Tetramethylsilane (TMS) served as internal standard for ^{1}H NMR. High resolution mass spectra were obtained using a commercial apparatus (ESI or EI Source).

General procedure for phosphinylation of α,α-diaryl allylic alcohols

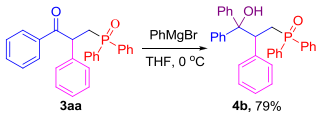


α,α-Diaryl allylic alcohol **1** (0.3 mmol), **2** (0.45 mmol) and AgOAc (0.9 mmol) in 2 mL 1,4-Dioxane was stirred at 120ºC under air for 2h. Upon completion of the reaction (indicated by TLC), the residue was directly purified by flash column chromatography by using ethyl acetate and petroleum ether as eluents to afford pure product **3**.

General procedure for further transformations of the α-aryl-β-phosphinyl ketone



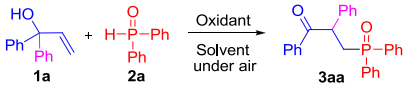
0.5 mmol (205.2 mg) of 3-(diphenylphosphoryl)-1,2-diphenylpropan-1-one was dissolved in 5.0 mL dry THF. Then 2 mmol of $LiAlH_4$ was added slowly at 0 ℃. After 10 minutes, the cold bath was removed and the reaction mixture was stirred at room temperature for 3h. The desired product was obtained after purification by flash chromatography on silica gel (white solid, 176.9 mg, 86 %).



3aa **4b**, 79%

0.5 mmol (205.2 mg) of 3-(diphenylphosphoryl)-1,2-diphenylpropan-1-one was dissolved in 2.0 mL dry THF. Then 1 mmol of phenylmagnesium bromide (2 equiv) was added slowly via syringe at 0 °C and after stirring at room temperature for 3 hours the solvent was evaporated. The desired product was obtained after purification by flash chromatography on silica gel (white solid, 193.1 mg, 79 %).

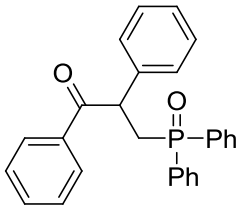
Table 1. Optimization of the Reaction Conditions[a]



Entry	Oxidant (equiv)	Solvent	Temp. (°C)	LC Yeild (%)[b]
1	AgOAc (2)	DMF	100	38
2	AgOAc (2)	MeCN	100	69
3	AgOAc (2)	EtOH	100	38
4	AgOAc (2)	Toluene	120	35
5	AgOAc (2)	CH_2ClCH_2Cl	120	Trace
6	AgOAc (2)	1,4-Dioxane	120	79
7	$AgNO_3$ (2)	1,4-Dioxane	120	36
8	$AgCO_3$(2)	1,4-Dioxane	120	Trace
9	AgOAc (1)	1,4-Dioxane	120	21
10	AgOAc (0.2)	1,4-Dioxane	120	15
11	AgOAc (0.2)+$Cu(OAc)_2$ (0.5)	1,4-Dioxane	120	16
12	AgOAc (0.2)+ $K_2S_2O_8$ (2)	1,4-Dioxane	120	messy
13	AgOAc (0.2)+ $PhI(OAc)_2$(2)	1,4-Dioxane	120	messy
14	AgOAc (0.2)+ DTBP[c] (3)	1,4-Dioxane	120	47
15	AgOAc (0.2)+ TBHP[d] (3)	1,4-Dioxane	120	24
16	AgOAc (0.2)+ TBPB[e] (3)	1,4-Dioxane	120	37
17	AgOAc (0.1)+ DTBP (3)	1,4-Dioxane	120	50(45)[f]
18	AgOAc (0.05)+ DTBP (3)	1,4-Dioxane	120	41
19	AgOAc (2.5)	1,4-Dioxane	120	80
20	AgOAc (3)	1,4-Dioxane	120	88 (86)[f]

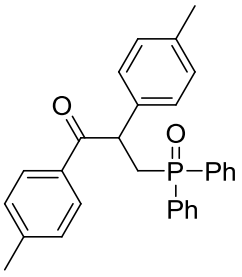
[a]All reactions were carried with **1a** (0.3 mmol), **2a** (0.45 mmol), and oxidant in solvent (2 mL) under Air for 2 h. [b]Yields were determined by LC with an internal standard (biphenyl) as the ratio between the formed products and the initial amount of limiting reactant. [c]DTBP = di-*tert*-butyl peroxide. [d]TBHP = *tert*-butyl hydroperoxide (70% in aqueous solution). [e]TBPB = *tert*-butylperoxybenzoate. [f]Isolated yields.

Analytical and spectral data for compounds



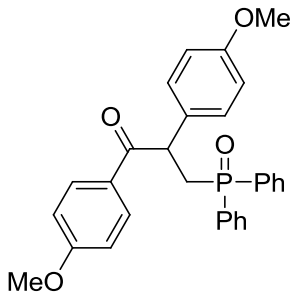
3aa

3-(Diphenylphosphoryl)-1,2-diphenylpropan-1-one (3aa): Yield = 86%. White solid. Mp = 160.0-161.1 ℃. IR (KBr) ν = 2947, 1676, 1455 ,1251, 1193, 1117, 1104, 993, 880, 847, 756, 714, 693 cm^{-1}. ^{1}H NMR (400MHz, $CDCl_3$): δ = 7.86–7.81 (m, 2H), 7.73–7.65 (m, 2H), 7.64–7.57 (m, 2H), 7.45–7.28 (m, 9H), 7.25–7.20 (m, 2H), 7.13–7.08 (m, 2H), 7.07–7.02 (m, 1H), 5.34–5.24 (m, 1H), 3.53–3.41 (m, 1H), 2.81–2.72 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 198.2 (d, *J* = 6.6 Hz), 138.9 (d, *J* = 7.4 Hz), 136.0, 133.6 (d, *J* = 51.4 Hz), 133.2, 132.6 (d, *J* = 51.3 Hz), 131.9 (d, *J* = 2.7 Hz), 131.6 (d, *J* = 2.7 Hz), 131.0 (dd, *J* = 15.5, 9.5 Hz), 129.1 (d, *J* = 8.7 Hz), 128.6 (dd, *J* = 11.7, 8.8 Hz), 128.6, 127.6, 46.9 (d, *J* = 1.3 Hz), 34.1 (d, *J* = 70.2 Hz) ppm. ^{31}P NMR (162 MHz, $CDCl_3$): δ = 30.17 ppm. HRMS m/z: calcd for $C_{27}H_{24}O_2P$ $[M+H]^+$ 411.1514, found: 411.1532.



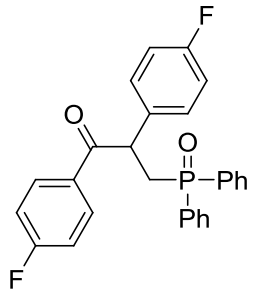
3ba

3-(Diphenylphosphoryl)-1,2-dip-tolylpropan-1-one (3ba): Yield = 88%. White solid. Mp = 139.9-141.2 ℃. IR (KBr) ν = 2959, 2933, 1731, 1671, 1599, 1574, 1509, 1439, 1248, 1168, 1119, 1027, 951, 890, 831, 783, 748, 695 cm^{-1}. ^{1}H NMR (400MHz, $CDCl_3$): δ = 7.75 (d, *J* = 8.2 Hz, 2H), 7.72–7.65 (m, 2H), 7.61–7.52 (m, 2H), 7.43–7.33 (m, 4H), 7.32–7.27 (m, 2H), 7.09 (dd, *J* = 8.1, 3.0 Hz, 4H), 6.87 (d, *J* = 7.9 Hz, 2H), 5.29–5.15 (m, 1H), 3.46–3.34 (m, 1H), 2.81–2.67 (m, 1H), 2.31 (s, 3H), 2.16 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ =197.7 (d, *J* = 7.2 Hz), 143.8, 137.0, 135.9 (d, *J* = 7.1 Hz), 133.7 (d, *J* = 65.3 Hz), 133.4, 132.7 (d, *J* = 65.3 Hz), 131.5 (dd, *J* = 69.6, 2.6 Hz), 130.9 (dd, *J* = 9.0, 8.5 Hz), 129.7, 129.2 (d, *J* = 3.3 Hz), 128.5 (dd, *J* = 18.5, 11.7 Hz), 128.4, 46.3, 34.1 (d, *J* = 70.5 Hz), 21.7, 21.5 ppm. ^{31}P NMR (162 MHz, $CDCl_3$): δ = 30.13 ppm. HRMS m/z: calcd for $C_{29}H_{28}O_2P$ $[M+H]^+$ 439.1827, found: 439.1815.



3ca

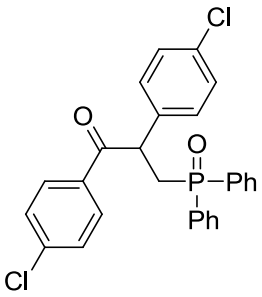
3-(Diphenylphosphoryl)-1,2-bis(4-methoxyphenyl)propan-1-one (3ca): Yield = 90%. White solid. Mp = 154.9-156.9 ℃. IR (KBr) ν = 2940, 2907, 1672, 1599, 1572, 1516, 1437, 1303, 1263, 1244, 1197, 1104, 1036, 1019, 883, 816, 794, 744, 690 cm^{-1}. ^{1}H NMR (400MHz, $CDCl_3$): δ = 7.85 (d, *J* = 8.8 Hz, 2H), 7.67 (dd, *J* = 11.0, 7.7 Hz, 2H), 7.56 (dd, *J* = 11.0, 7.8 Hz, 2H), 7.40–7.27 (m, 6H), 7.13 (d, *J* = 8.6 Hz, 2H), 6.78 (d, *J* = 8.7 Hz, 2H), 6.58 (d, *J* = 8.5 Hz, 2H), 5.27–5.19 (m, 1H), 3.78 (s, 3H), 3.66 (s, 3H), 3.38–3.28 (m, 1H), 2.81–2.71 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.8 (d, *J* = 7.7 Hz), 163.5, 158.8, 131.8 (d, *J* = 2.5 Hz), 131.4, 131.3 (d, *J* = 2.5 Hz), 131.1, 130.9 (dd, *J* = 6.4, 4.9 Hz), 129.7, 128.9, 128.5 (dd, *J* = 17.7, 11.8 Hz), 114.5, 113.8, 55.6, 55.3, 45.6, 34.1 (d, *J* = 71.1 Hz) ppm. ^{31}P NMR (162 MHz, $CDCl_3$): δ = 30.52 ppm. HRMS m/z: calcd for $C_{29}H_{28}O_4P$ $[M+H]^+$ 471.1725, found: 471,1723.



3da

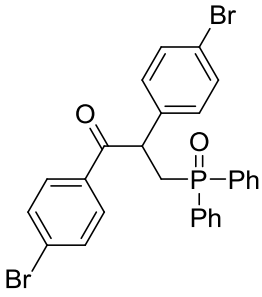
3-(Diphenylphosphoryl)-1,2-bis(4-fluorophenyl)propan-1-one (3da): Yield = 81%. White solid. Mp = 149.4-151.5 ℃. IR (KBr) ν = 2944, 2923, 1688, 1597, 1513, 1439, 1295, 1240, 1194, 1176, 1157, 999, 864, 803, 726, 694, 658 cm^{-1}. ^{1}H NMR (400MHz, $CDCl_3$): δ = 7.91–7.84 (m, 2H), 7.71–7.65 (m, 2H), 7.60–7.53 (m, 2H), 7.46–7.30 (m, 6H), 7.20–7.14 (m, 2H), 7.04–6.97 (m, 2H), 6.80–6.73 (m, 2H), 5.29–5.22 (m, 1H), 3.40–3.10 (m, 1H), 2.81–2.73 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.7 (d, *J* = 7.8 Hz), 165.8 (d, *J* = 254.0 Hz), 162.2 (d, *J* = 245.4 Hz), 134.2 (dd, *J* = 6.8, 3.3 Hz), 133.8, 132.9 (d, *J* = 20.7 Hz), 132.1 (dd, *J* = 10.4, 2.9 Hz), 131.8 (dd, *J* = 33.6, 2.7 Hz), 131.7 (d, *J* = 9.5 Hz), 130.9 (dd, *J* = 9.3, 8.5 Hz), 130.2 (d, *J* = 8.2 Hz), 128.7 (dd, *J* = 14.7, 11.8 Hz), 115.9 (dd, *J* = 23.0, 21.6 Hz), 46.0, 34.1 (d, *J* = 70.6 Hz) ppm. ^{31}P NMR (162 MHz, $CDCl_3$): δ =

29.86 ppm. HRMS m/z: calcd for $C_{27}H_{22}F_2O_2P$ $[M+H]^+$ 447.1325, found: 447.1343.



3ea

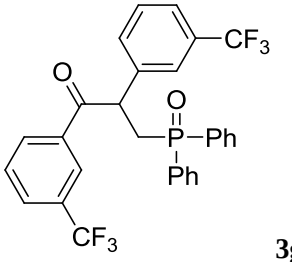
1,2-Dis(4-chlorophenyl)-3-(diphenylphosphoryl)propan-1-one (3ea): Yield = 84%. White solid. Mp = 145.7-147.5 ℃. IR (KBr) ν = 2945, 2924, 1678, 1588, 1490, 1436, 1398, 1253, 1183, 1117, 1093, 1013, 977, 890, 861, 817, 783, 742, 717, 691 cm^{-1}. ^1H NMR (400MHz, $CDCl_3$): δ = 7.78 (d, *J* = 8.6 Hz, 2H), 7.72–7.65 (m, 2H), 7.59–7.51 (m, 2H), 7.47–7.28 (m, 8H), 7.12 (d, *J* = 8.5 Hz, 2H), 7.03 (d, *J* = 8.5 Hz, 2H), 5.27–5.18 (m, 1H), 3.40–3.29 (m, 1H), 2.82–2.70 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.8 (d, *J* = 7.8 Hz), 139.4, 136.7 (d, *J* = 6.7 Hz), 133.9 (d, *J* = 24.8 Hz), 133.3 (d, *J* = 88.7 Hz), 132.3 (d, *J* = 88.4 Hz), 132.1 (d, *J* = 2.7 Hz), 131.6 (d, *J* = 2.7 Hz), 130.9 (dd, *J* = 9.5, 6.0 Hz), 130.4, 130.0, 129.2 (d, *J* = 27.7 Hz), 128.70 (dd, *J* = 15.7, 11.8 Hz), 46.30 (d, *J* = 1.5 Hz), 34.0 (d, *J* = 70.6 Hz) ppm. ^{31}P NMR (162 MHz, $CDCl_3$): δ = 29.87 ppm. HRMS m/z: calcd for $C_{27}H_{22}Cl_2O_2P$ $[M+H]^+$ 479.0734, found: 479.0738.



3fa

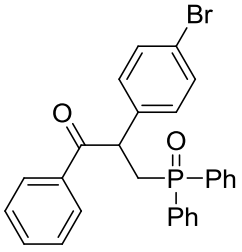
1,2-Dis(4-bromophenyl)-3-(diphenylphosphoryl)propan-1-one (3fa): Yield = 52%. White solid. Mp = 157.4-159.7 ℃. IR (KBr) ν = 2954, 1677, 1584, 1487, 1436, 1396, 1254, 1183, 1119, 1072, 1011, 977, 889, 861, 815, 779, 742, 693 cm^{-1}. ^1H NMR (400MHz, $CDCl_3$): δ = 7.73–7.63 (m, 4H), 7.57–7.51 (m, 2H), 7.50–7.41 (m, 4H), 7.41–7.36 (m, 2H), 7.35–7.29 (m, 2H), 7.18 (d, *J* = 8.4 Hz, 2H), 7.05 (d, *J* = 8.4 Hz, 2H), 5.25–5.16 (m, 1H), 3.39–3.28 (m, 1H), 2.81–2.70 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 197.0 (d, *J* = 7.9 Hz), 137.2 (d, *J* = 6.5 H), 134.4, 133.3 (d, *J* = 94.6 H), 132.3 (d, *J* = 94.1 H), 132.2, 132.1, 131.6 (d, *J* = 2.8 Hz), 130.9 (dd, *J* = 9.5, 4.9 Hz), 130.5, 130.3, 128.7 (dd, *J* = 15.1, 11.9 Hz), 122.0, 46.4, 33.9 (d, *J* = 70.6 Hz) ppm. ^{31}P NMR (162 MHz, $CDCl_3$): δ = 29.70

ppm. HRMS m/z: calcd for $C_{27}H_{22}Br_2O_2P$ $[M+H]^+$ 566.9724, found: 566.9729.



3ga

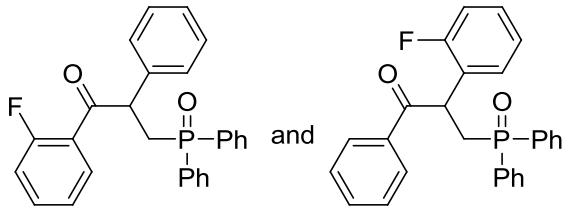
3-(Diphenylphosphoryl)-1,2-bis(3-(trifluoromethyl)phenyl)propan-1-one (3ga): Yield = 76%. White solid. Mp = 42.3-46.5 ℃. IR (KBr) ν = 2918, 1686, 1610, 1438, 1326, 1236, 1165, 1118, 1072, 815, 736, 717, 691 cm^{-1}. ^1H NMR (400MHz, $CDCl_3$): δ = 8.06 (d, *J* = 8.6 Hz, 2H), 7.75–7.65 (m, 3H), 7.61–7.54 (m, 2H), 7.53–7.35 (m, 7H), 7.34–7.27 (m, 3H), 7.24 (t, *J* = 7.7 Hz, 1H), 5.44–5.34 (m, 1H), 3.44–3.34 (m, 1H), 2.89–2.80 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.6 (d, *J* = 7.6 Hz), 138.8 (d, *J* = 6.9 Hz), 136.1, 133.5, 132.6 (d, *J* = 4.6 Hz), 133.2, 132.2, 132.1, 131.9 (d, *J* = 2.7 Hz), 131.7 (d, *J* = 17.3 Hz), 131.4 (d, *J* = 16.6 Hz), 130.9 (dd, *J* = 13.3, 9.5 Hz), 129.9 (d, *J* = 3.4 Hz), 129.8, 129.5, 128.8 (dd, *J* = 13.4, 12.0 Hz), 125.9 (d, *J* = 3.7 Hz), 125.3 (d, *J* = 3.9 Hz), 124.8 (d, *J* = 3.6 Hz), 46.8, 34.1 (d, *J* = 70.0 Hz) ppm. ^{31}P NMR (162 MHz, $CDCl_3$): δ = 29.34 ppm. HRMS m/z: calcd for $C_{29}H_{22}F_6O_2P$ $[M+H]^+$ 547.1262, found: 547.1262.



3ha

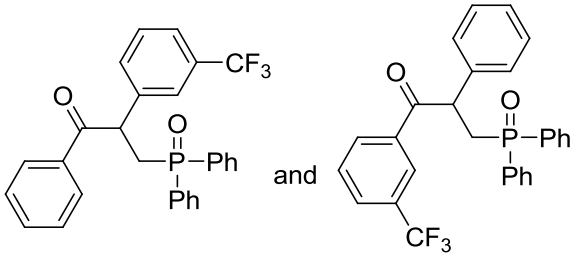
2-(4-Bromophenyl)-3-(diphenylphosphoryl)-1-phenylpropan-1-one (3ha): Yield = 34%. White solid. Mp = 153.9-155.2 ℃. IR (KBr) ν = 2906, 1688, 1487, 1438, 1406, 1245, 1175, 1118, 1102, 999, 971, 886, 816, 744, 716, 698 cm^{-1}. The ^1H NMR spectrum of the crude product showed a 1.8:1 mixture of **3ha** and its isomer **3ha'**, flash chromatography on silica gel afforded ketone **3ha**. ^1H NMR (400MHz, $CDCl_3$): δ = 7.86–7.81 (m, 2H), 7.72–7.66 (m, 2H), 7.57–7.51 (m, 2H), 7.46–7.40 (m, 3H), 7.40–7.34 (m, 3H), 7.32 (d, *J* = 7.4 Hz, 4H), 7.17 (d, *J* = 8.5 Hz, 2H), 7.08 (d, *J* = 8.5 Hz, 2H), 5.31–5.24 (m, 1H), 3.38–3.29 (m, 1H), 2.83–2.74 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 197.9 (d, *J* = 8.1 Hz), 137.5 (d, *J* = 6.3 Hz), 135.6, 133.4 (d, *J* = 95.3 Hz), 133.4, 132.5 (d, *J* = 95.3 Hz), 132.1, 131.7 (dd, *J* = 47.6, 2.5 Hz), 130.8 (dd, *J* = 9.5, 3.9 Hz), 130.4, 128.9 (d, *J* = 35.3

Hz), 128.7 (dd, *J* = 16.9, 11.4 Hz), 121.7, 46.3, 33.9 (d, *J* = 70.8 Hz) ppm. ^{31}P NMR (162 MHz, $CDCl_3$): δ = 29.78 ppm. HRMS m/z: calcd for $C_{27}H_{23}BrO_2P$ $[M+H]^+$ 489.0619, found: 498.0594.



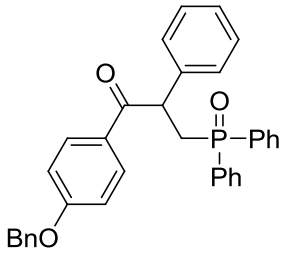
3ia **3ia'**

3-(Diphenylphosphoryl)-1-(2-fluorophenyl)-2-phenylpropan-1-one (3ia) and **3-(diphenylphosphoryl)-2-(2-fluorophenyl)-1-phenylpropan-1-one (3ia'):** Yield = 78% (2:1). White solid. IR (KBr) ν = 2953, 1680, 1607, 1494, 1481, 1276, 1234, 1181, 1117, 1105, 977, 871, 763, 733, 695 cm^{-1}. The ^{1}H NMR spectrum of the isolated product showed a 2:1 mixture of **3ia** and its isomer **3ia'**. ^{1}H NMR (400MHz, $CDCl_3$) **3ia**: δ = 7.87 (d, *J* = 7.3 Hz, 1H), 7.74–7.69 (m, 2H), 7.63–7.59 (m, 2H), 7.48–7.31 (m, 9H), 7.17 (d, *J* = 7.4 Hz, 2H), 7.10–7.06 (m, 2H), 6.96 (dd, *J* = 10.6, 8.4 Hz, 1H), 5.21–5.11 (m, 1H), 3.58–3.47 (m, 1H), 2.75–2.65 (m, 1H) ppm; **3ia'**: δ = 7.75 (d, *J* = 7.3 Hz, 1H), 7.68–7.64 (m, 2H), 7.60–7.57 (m, 2H), 7.31–7.26 (m, 9H), 7.06–6.99 (m, 2H), 6.92–6.85(m, 2H), 6.82–6.76 (m, 1H), 5.55–5.46 (m, 1H), 3.49–3.39 (m, 1H), 2.84–2.75 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) **3ia** and **3ia'**: δ = 196.8 (d, *J* = 6.4 Hz), 196.7 (d, *J* = 6.8 Hz), 162.4, 159.9, 138.0 (d, *J* = 7.6 Hz), 135.6, 134.6 (d, *J* = 9.1 Hz), 134.1 (d, *J* = 1.5 Hz), 133.4 (d, *J* = 3.4 Hz), 133.1 (d, *J* = 1.9 Hz), 132.4, 131.9 (d, *J* = 2.8 Hz), 131.9 (d, *J* = 2.8 Hz), 131.6 (d, *J* = 2.8 Hz), 131.6 (d, *J* = 2.8 Hz), 131.3 (d, *J* = 2.3 Hz), 131.0, 130.9 (dd, *J* = 16.5, 2.2 Hz), 129.8 (d, *J* = 3.2 Hz), 129.5 (d, *J* = 3.2 Hz), 129.0, 128.8 (d, *J* = 22.4 Hz), 128.7 (dd, *J* = 21.8, 11.7 Hz), 128.6 (dd, *J* = 14.6, 11.8 Hz), 127.6, 125.3 (d, *J* = 12.2 Hz), 124.8 (d, *J* = 3.6 Hz), 124.3 (d, *J* = 3.4 Hz), 116.8 (d, *J* = 23.5 Hz), 116.0 (d, *J* = 22.2 Hz), 50.6 (d, *J* = 1.3 Hz), 50.5 (d, *J* = 1.2 Hz), 39.6, 33.9 (d, *J* = 70.4 Hz) ppm. ^{31}P NMR (162 MHz, $CDCl_3$) **3ia** and **3ia'**: δ = 29.79, 29.49 ppm. HRMS m/z: calcd for $C_{27}H_{23}FO_2P$ $[M+H]^+$ 429.1420, found: 429.1400.



3ja **3ja'**

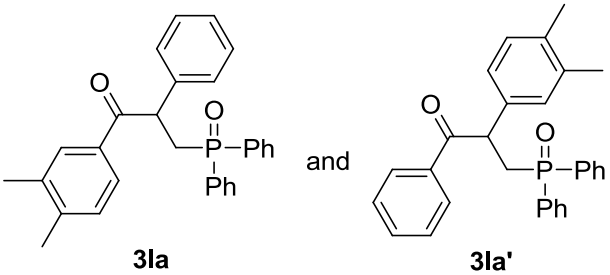
3-(Diphenylphosphoryl)-2-phenyl-1-(3-(trifluoromethyl)phenyl)propan-1-one (3ja) and **3-(diphenylphosphoryl)-1-phenyl-2-(3-(trifluoromethyl)phenyl)propan-1-one (3ja'):** Yield = 75% (2.51:1). Colourless oil. IR (KBr) ν = 3057, 1680, 1595, 1447, 1437, 1326, 1165, 1118, 1072, 976, 814, 735, 692 cm^{-1}. The ^{1}H NMR spectrum of the isolated product showed a 2.51:1 mixture of **3ja** and its isomer **3ja'**. ^{1}H NMR (400MHz, $CDCl_3$) **3ja**: δ = 7.87 (d, *J* = 7.6 Hz, 2H), 7.69 (dd, *J* = 11.5, 7.2 Hz, 3H), 7.54 (dd, *J* = 11.5, 7.3 Hz, 2H), 7.40–7.31 (m, 6H), 7.30–7.26 (m, 3H), 7.25–7.16 (m, 2H), 7.15–7.08 (m, 1H), 5.46–5.38 (m, 1H), 3.40–3.29 (m, 1H), 2.91–2.81 (m, 1H) ppm; **3ja'**: δ = 8.02 (d, *J* = 8.1 Hz, 2H), 7.62 (dd, *J* = 11.6, 7.4 Hz, 3H), 7.51–7.41 (m, 8H), 7.27–7.26 (m, 3H), 7.18–7.15 (m, 2H), 7.11–7.06 (m, 1H), 5.30–5.22 (m, 1H), 3.56–3.46 (m, 1H), 2.81–2.71 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) **3ja** and **3ja'**: δ = 197.8 (d, *J* = 8.4 Hz), 139.3 (d, *J* = 6.1 Hz), 135.6, 133.6, 132.3, 132.7, 132.1 (d, *J* = 2.9 Hz), 131.7, 130.9, 130.8, 130.7, 129.6, 129.4, 129.1, 128.9, 128.8, 128.7, 128.6, 128.5, 128.5, 127.9, 125.4 (d, *J* = 3.7 Hz), 124.5 (d, *J* = 3.8 Hz), 46.5 (d, *J* = 1.1 Hz), 34.0 (d, *J* = 70.6 Hz) ppm. ^{31}P NMR (162 MHz, $CDCl_3$) **3ja** and **3ja'**: δ = 30.04, 29.59 ppm. HRMS m/z: calcd for $C_{28}H_{23}F_3O_2P$ $[M+H]^+$ 479.1388, found: 479.1382.



3ka

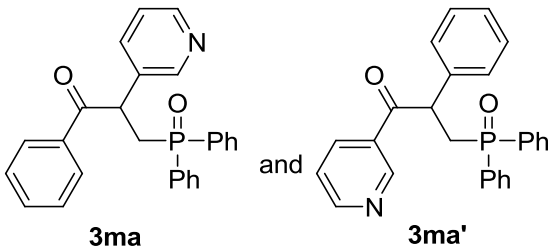
1-(4-(Benzyloxy)phenyl)-3-(diphenylphosphoryl)-2-phenylpropan-1-one (3ka): Yield = 73%. White solid. Mp = 203.3–204.3 ℃. IR (KBr) ν = 2925, 1725, 1667, 1598, 1572, 1454, 1436, 1241, 1171, 1117, 1002, 955, 843, 741, 691 cm^{-1}. The ^{1}H NMR spectrum of the crude product showed a 7.2:1 mixture of **3ka** and its isomer **3ka'**, flash chromatography on silica gel afforded ketone **3ka**. ^{1}H NMR (400MHz, $CDCl_3$): δ = 7.83 (d, *J* = 8.8 Hz, 2H), 7.71–7.64 (m, 2H), 7.62–7.55 (m, 2H), 7.42–7.28 (m, 11H), 7.23 (d, *J* = 7.2 Hz, 2H), 7.13–7.01 (m, 3H), 6.85 (d, *J* = 8.8 Hz, 2H), 5.29–5.19 (m, 1H), 5.05 (s, 2H), 3.51–3.39 (m, 1H), 2.80–2.70 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.6 (d, *J* = 6.7 Hz), 162.7, 139.3 (d, *J* = 7.4 Hz), 136.3, 133.7 (d, *J* = 47.7 Hz), 132.7 (d, *J* = 47.8 Hz), 131.7 (dd, *J* = 27.3, 2.7 Hz), 131.4, 131.0 (dd, *J* = 16.4, 9.5 Hz), 129.1 (d, *J* = 3.9 Hz), 129.1, 128.9, 128.6 (dd, *J* = 11.7, 7.0 Hz), 128.5 (d, *J* = 7.6 Hz), 127.7, 127.4, 114.6, 70.3, 46.4, 34.1 (d, *J* = 70.4 Hz) ppm. ^{31}P NMR (162 MHz, $CDCl_3$): δ = 30.24 ppm. HRMS m/z: calcd for $C_{34}H_{30}O_3P$ $[M+H]^+$

517.1933, found: 517.1932.



3la **3la'**

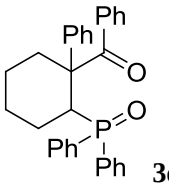
1-(3,4-Dimethylphenyl)-3-(diphenylphosphoryl)-2-phenylpropan-1-one (3la) and 2-(3,4-dimethylphenyl)-3-(diphenylphosphoryl)-1-phenylpropan-1-one (3la'): Yield = 80% (2.3:1). White solid. IR (KBr) ν = 2918, 1672, 1602, 1437, 1250, 1181, 1118, 1102, 1072, 980, 817, 784, 734, 692 cm^{-1}. The ^{1}H NMR spectrum of the isolated product showed a 2.3:1 mixture of **3la** and its isomer **3la'**. ^{1}H NMR (400MHz, $CDCl_3$) **3la**: δ = 7.88–7.84 (m, 1H), 7.73–7.69 (m, 2H), 7.62–7.54 (m, 3H), 7.42–7.34 (m, 7H), 7.24–7.20 (m, 2H), 7.07 (d, *J* = 7.9 Hz, 2H), 6.95 (d, *J* = 6.9 Hz, 1H), 5.32–5.25 (m, 1H), 3.48–3.40 (m, 1H), 2.82–2.76 (m, 1H) ppm; **3la'**: δ = 7.79–7.75 (m, 1H), 7.68–7.64 (m, 2H), 7.50–7.45 (m, 3H), 7.32–7.27 (m, 7H), 7.11–7.08 (m, 2H), 7.03–7.00 (m, 1H), 6.84 (d, *J* = 8.0 Hz, 1H), 5.25–5.19 (m, 1H), 3.40–3.34 (m, 1H), 2.75–2.72 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) **3la** and **3la'**: δ = 198.3 (d, *J* = 7.4 Hz), 198.0 (d, *J* = 7.0 Hz), 142.8, 139.1 (d, *J* = 7.0 Hz), 137.2, 136.8, 136.0, 135.8, 134.1 (d, *J* = 10.0 Hz), 133.8, 133.2 (dd, *J* = 25.1, 3.6 Hz), 133.1, 132.4 (d, *J* = 5.0 Hz), 131.8 (dd, *J* = 10.1, 2.7 Hz), 131.8 (d, *J* = 2.7 Hz), 131.5 (d, *J* = 2.7 Hz), 131.2 (d, *J* = 2.7 Hz), 130.9 (dd, *J* = 12.8, 9.5 Hz), 130.9, 130.4, 130.2, 129.8 (d, *J* = 2.7 Hz), 129.1 (d, *J* = 3.7 Hz), 128.5 (dd, *J* = 17.2, 11.8 Hz), 128.5, 128.3, 128.2, 127.4, 126.8, 125.9, 46.6(d, *J* = 1.5 Hz), 46.5(d, *J* = 1.5 Hz), 34.2 (d, *J* = 70.6 Hz), 34.0 (d, *J* = 70.4 Hz), 20.1, 19.9, 19.8, 19.4 ppm. ^{31}P NMR (162 MHz, $CDCl_3$) **3la** and **3la'**: 30.34, 30.23 ppm. HRMS m/z: calcd for $C_{29}H_{28}O_2P$ $[M+H]^+$ 439.1827, found: 439.1843.



3ma **3ma'**

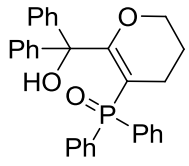
3-(Diphenylphosphoryl)-1-phenyl-2-(pyridin-3-yl)propan-1-one (3ma) and 3-(diphenylphosphoryl)-2-phenyl-1-(pyridin-3-yl)propan-1-one (3ma'): Yield = 73% (2:1).

White solid. IR (KBr) ν = 2922, 1681, 1582, 1437, 1256, 1195, 1117, 1103, 1024, 811, 853, 813, 741, 716, 694 cm^{-1}. The ^{1}H NMR spectrum of the isolated product showed a 2:1 mixture of **3ma** and its isomer **3ma'**. ^{1}H NMR (400MHz, $CDCl_3$) **3ma**: δ = 8.44 (s, 1H), 8.20 (d, *J* = 3.1 Hz, 1H), 7.79 (d, *J* = 7.5 Hz, 2H), 7.66–7.58 (m, 3H), 7.47 (d, *J* = 8.7 Hz, 1H), 7.31–7.21 (m, 8H), 7.14 (d, *J* = 7.2 Hz, 1H), 7.06 (t, *J* = 7.2 Hz, 1H), 6.90 (dd, *J* = 7.6, 4.8 Hz, 1H), 5.34–5.24 (m, 1H), 3.26–3.37 (m, 1H), 2.79–2.70 (m, 1H) ppm; **3ma'**: 8.94 (s, 1H), 8.55 (d, *J* = 3.9 Hz, 1H), 8.03 (d, *J* = 7.9 Hz, 1H), 7.58–7.52 (m, 3H), 7.50 (d, *J* = 4.1 Hz, 2H), 7.40–7.31 (m, 8H), 7.22–7.21 (m, 1H), 7.19–7.16 (m, 1H), 7.02–6.98 (m, 1H), 5.19–5.10 (m, 1H), 3.39–3.50 (m, 1H), 2.69–2.61 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) **3ma** and **3ma'**: δ = 197.6 (d, *J* = 7.7 Hz), 197.1 (d, *J* = 6.3 Hz), 153.3, 150.3, 150.1, 148.8, 136.3, 135.9, 135.4, 134.3 (dd, *J* = 5.5, 2.3 Hz), 133.6, 133.5 (d, *J* = 9.9 Hz), 132.6 (d, *J* = 25.1 Hz), 132.1 (d, *J* = 2.6 Hz), 132.0 (d, *J* = 2.3 Hz), 131.8 (d, *J* = 1.9 Hz), 131.0, 130.8 (dd, *J* = 14.6, 5.0 Hz), 130.8 (dd, *J* = 9.3, 8.6 Hz), 129.4, 129.0, 128.8, 128.7 (dd, *J* = 11.7, 6.0 Hz), 128.6, 128.5, 127.9, 123.8, 123.5, 47.4 (d, *J* = 1.8 Hz), 44.2 (d, *J* = 1.2 Hz), 33.9 (d, *J* = 69.8 Hz), 33.8 (d, *J* = 70.3 Hz) ppm. ^{31}P NMR (162 MHz, $CDCl_3$) **3ma** and **3ma'**: 30.05, 29.57 ppm. HRMS m/z: calcd for $C_{26}H_{23}NO_2P$ $[M+H]^+$ 412.1466, found: 412.1452.



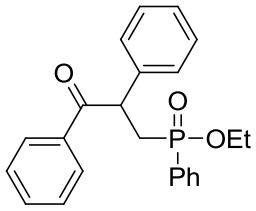
3oa

(2-(diphenylphosphoryl)-1-phenylcyclohexyl)(phenyl)methanone (3oa): Yield = 38%. White soild. Mp = 208.9–209.8 ℃. IR (KBr) ν = 2927, 2846, 1672, 1449, 1438, 1248, 1174, 1106, 1073, 975, 781, cm^{-1}. ^{1}H NMR (400MHz, $CDCl_3$): δ = 7.89–7.81 (m, 2H), 7.82–7.74 (m, 2H), 7.47–7.27 (m, 11H), 7.25–7.12 (m, 5H), 3.78–3.66 (m, 1H), 2.94–2.82 (m, 1H), 2.49–2.37 (m, 1H), 1.95–1.82 (m, 1H), 1.73–1.47 (m, 3H), 1.47–1.34 (m, 2H), 1.22–1.12 (m, 1H), 0.95–0.80 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 205.6, 205.6, 140.1, 140.1, 139.1, 137.2, 126.2, 125.4, 134.5, 132.0, 131.9, 131.0, 130.0, 130.5, 130.5, 130.5, 130.4, 130.4, 128.7, 128.7, 128.6, 128.3, 128.3, 128.2, 128.2, 127.7, 127.3, 59.4, 59.3, 45.6, 44.9, 32.8, 31.6, 30.4, 29.9, 25.0, 23.5, 23.4, 21.7 ppm. ^{31}P NMR (162 MHz, $CDCl_3$): δ = 32.97 ppm. HRMS m/z: calcd for $C_{31}H_{30}O_2P$ $[M+H]^+$ 465.1983, found: 465.2030.



3pa

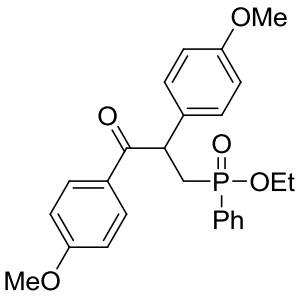
(5-(diphenylphosphoryl)-3,4-dihydro-2H-pyran-6-yl)diphenylmethanol (3pa): Yield = 36%. White soild. Mp = 172.8–174.1 ℃. IR (KBr) ν = 3165, 3053, 2925, 1583, 1435, 1238, 1167, 1117, 1068, 905, 750, 695 cm^{-1}. ^{1}H NMR (400MHz, $CDCl_3$): δ = 8.35 (s, 1H), 7.63–7.49 (m, 6H), 7.48–7.40 (m, 4H), 7.35–7.29 (m, 4H), 7.29–7.23 (m, 6H), 4.03–3.95 (m, 2H), 1.88–1.77 (m, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 172.2, 172.0, 146.2, 133.4, 132.3, 132.2, 132.2, 131.9, 131.8, 128.6, 128.5, 128.1, 127.6, 127.1, 97.0, 95.9, 82.0, 82.0, 66.3, 26.3, 26.2, 22.0, 21.9 ppm. ^{31}P NMR (162 MHz, $CDCl_3$): δ = 36.55 ppm. HRMS m/z: calcd for $C_{30}H_{28}O_3P$ $[M+H]^+$ 467.1776, found: 467.1816.



3ab

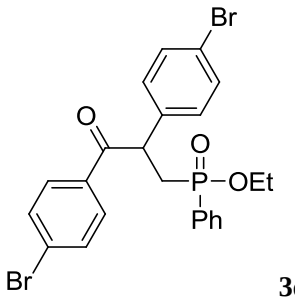
Ethyl 3-oxo-2,3-diphenylpropyl(phenyl)phosphinate (3ab): Yield = 88% (1.3:1). Colourless oil. IR (KBr) ν = 2978, 1725, 1680, 1596, 1448, 1439, 1230, 1207, 1177, 1120, 1026, 955, 913, 756, 694 cm^{-1}. The ^{1}H NMR spectrum of the isolated product characterized as a 1.3:1 diastereomeric mixture. ^{1}H NMR (400MHz, $CDCl_3$): δ = 8.02–7.98 (m, 2H), 7.69–7.63 (m, 2H), 7.40–7.31 (m, 7H), 7.25–7.17 (m, 4H), 5.24–5.17 (m, 1H), 3.80–3.65 (m, 2H), 3.22–3.17 (m, 1H), 2.40–2.32 (m, 1H), 1.06 (t, *J* = 7.0 Hz, 3H) and isomer: δ = 7.87–7.82 (m, 2H), 7.74–7.68 (m, 2H), 7.50–7.41 (m, 7H), 7.22 (ddd, *J* = 8.8, 5.6, 2.3 Hz, 8H), 7.18–7.03 (m, 4H), 5.17–5.10 (m, 1H), 3.98–3.84 (m, 2H), 3.04–2.94 (m, 1H), 2.51–2.40 (m, 1H), 1.11 (t, *J* = 7.0 Hz, 6H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 198.2 (d, *J* = 6.7 Hz), 139.2 (d, *J* = 9.8 Hz), 138.5 (d, *J* = 9.8 Hz), 136.3, 136.1, 133.2, 133.1, 132.4 (d, *J* = 2.8 Hz), 132.3 (d, *J* = 2.7 Hz), 132.0 (d, *J* = 10.2 Hz), 131.8, 131.7, 131.6 (d, *J* = 24.7 Hz), 130.4 (d, *J* = 14.2 Hz), 130.1, 129.3, 129.2, 129.1, 129.0, 128.6 (dd, *J* = 12.5, 9.1 Hz), 128.5 (dd, *J* = 22.6, 12.3 Hz), 127.5, 127.5, 60.8 (d, *J* = 6.3 Hz), 60.7 (d, *J* = 6.3 Hz), 47.3 (d, *J* = 1.2 Hz), 46.8 (d, *J* = 1.6 Hz), 34.3 (d, *J* = 98.9 Hz), 34.2 (d, *J* = 99.4 Hz), 16.4 (d, *J* = 6.8 Hz), 16.3 (d, *J* = 6.6 Hz) ppm. ^{31}P NMR (162 MHz, $CDCl_3$): δ = 42.26, 42.16 ppm. HRMS m/z: calcd for $C_{23}H_{24}O_3P$ $[M+H]^+$ 379.1463, found:

379.1457.



3cb

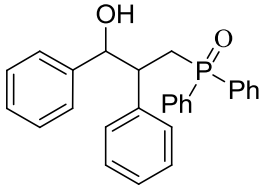
Ethyl 2,3-bis(4-methoxyphenyl)-3-oxopropyl(phenyl)phosphinate (3cb): Yield = 93% (1.1:1). Colourless oil. IR (KBr) ν = 2953, 1673, 1606, 1513, 1435, 1254, 1184, 1116, 971, 888, 862, 810, 796, 766, 731, 693 cm^{-1}. The ^{1}H NMR spectrum of the isolated product characterized as a 1.1:1 diastereomeric mixture. ^{1}H NMR (400MHz, $CDCl_3$): δ = 7.98 (d, *J* = 8.9 Hz, 2H), 7.74–7.66 (m, 2H), 7.51–5.40 (m, 3H), 7.23 (d, *J* = 8.7 Hz, 2H), 6.86 (d, *J* = 8.9 Hz, 2H), 6.83–6.80 (m, 2H), 5.15–5.06 (m, 1H), 3.95–3.86 (m, 2H), 3.82 (s, 3H), 3.80 (s, 3H), 3.11–3.01 (m, 1H), 2.47–2.07 (m, 1H), 1.12 (t, *J* = 7.1 Hz, 3H) and isomer: δ = 7.84 (d, *J* = 8.9 Hz, 2H), 7.67–7.59 (m, 2H), 7.40–7.31 (m, 3H), 7.11 (d, *J* = 8.7 Hz, 2H), 6.80–6.77 (m, 2H), 6.62 (d, *J* = 8.7 Hz, 2H), 5.06–4.98 (m, 1H), 3.74–3.72 (m, 3H), 3.74 (s, 3H), 3.68 (s, 3H), 2.98–2.87 (m, 1H), 2.40–2.33 (m, 1H), 1.08 (t, *J* = 7.1 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.8 (d, *J* = 7.6 Hz), 196.5 (d, *J* = 7.4 Hz), 163.6, 163.5, 158.9, 158.8, 132.4 (d, *J* = 2.6 Hz), 132.1 (d, *J* = 2.6 Hz), 131.9, 131.8 (d, *J* = 10.0 Hz), 131.6, 131.3 (d, *J* = 7.0 Hz), 131.0 (d, *J* = 24.4 Hz), 130.6 (d, *J* = 25.8 Hz), 129.5, 129.2, 129.0, 128.6 (dd, *J* = 15.2, 12.6 Hz), 114.5, 114.4, 113.9, 113.8, 60.7, 60.6, 55.6, 55.6, 55.4, 55.3, 46.0, 45.7, 34.3 (d, *J* = 98.7 Hz), 34.1 (d, *J* = 99.9 Hz), 16.5 (d, *J* = 6.3 Hz), 16.4 (d, *J* = 6.3 Hz) ppm. ^{31}P NMR (162 MHz, $CDCl_3$): δ = 42.77, 42.58 ppm. HRMS m/z: calcd for $C_{25}H_{28}O_5P$ $[M+H]^+$ 439.1674, found: 439.1677.



3db

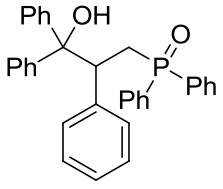
Ethyl 2,3-bis(4-bromophenyl)-3-oxopropyl(phenyl)phosphinate (3db): Yield = 50% (1:1). Colourless oil. IR (KBr) ν = 3421, 3058, 2978, 2925, 1682, 1584, 1485, 1438, 1396, 1249, 1120,

1070, 1031, 1009, 952, 889, 819, 785, 745, 694 cm^{-1}. The ^{1}H NMR spectrum of the isolated product characterized as a 1:1 diastereomeric mixture. ^{1}H NMR (400MHz, $CDCl_3$) a mixture: δ = 7.83 (d, *J* = 8.4 Hz, 2H), 7.69 (dd, *J* = 13.5, 5.3 Hz, 4H), 7.63–7.58 (m, 2H), 7.57–7.52 (m, 3H), 7.52–7.47 (d, *J* = 8.2 Hz, 3H), 7.47–7.38 (m, 4H), 7.38–7.33 (m, 2H), 7.20 (t, *J* = 8.0 Hz, 4H), 7.04 (d, *J* = 8.3 Hz, 2H), 5.13–5.08 (m, 1H), 5.07–5.02 (m, 1H), 3.98–3.86 (m, 2H), 3.78–3.65 (m, 2H), 3.09–2.97 (m, 1H), 2.84–2.83 (m, 1H), 2.47–2.40 (m, 1H), 2.40–2.33 (m, 1H), 1.13 (t, *J* = 7.0 Hz, 3H), 1.10 (t, *J* = 7.0 Hz, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) a mixture: δ = 196.9 (d, *J* = 8.2 Hz), 196.7 (d, *J* = 7.5 Hz), 137.9, 137.8, 137.0, 136.9, 134.6, 134.5, 132.5 (dd, *J* = 32.1, 2.8 Hz), 132.4, 132.2, 132.2, 132.1, 131.7 (dd, *J* = 10.0, 2.7 Hz), 131.3 (d, *J* = 31.1 Hz), 130.5, 130.4, 130.2, 130.1, 130.1 (d, *J* = 30.8 Hz), 128.9, 128.7, 128.7, 128.6, 121.9, 121.9, 60.9 (d, *J* = 2.0 Hz), 60.8 (d, *J* = 2.0 Hz), 46.7, 46.4, 34.0 (d, *J* = 99.3 Hz), 33.8 (d, *J* = 100.3 Hz), 16.5, 16.4 ppm. ^{31}P NMR (162 MHz, $CDCl_3$) a mixture: δ = 42.01, 41.80 ppm. HRMS m/z: calcd for $C_{23}H_{22}Br_2O_3P$ $[M+H]^+$ 534.9673, found: 534.9682.



4a

3-(diphenylphosphoryl)-1,2-diphenylpropan-1-ol (4a): Yield = 86%. White soild. Mp = 204.7–205.7 ℃. IR (KBr) ν = 3353, 2926, 1437, 1173, 1147, 1119, 1087, 1058, 870, 769, 761, 744, 693 cm^{-1}. ^{1}H NMR (400 MHz, DMSO-d_6) δ = 7.77–7.69 (m, 2H), 7.62–7.46 (m, 5H), 7.44–7.38 (m, 1H), 7.37–7.30 (m, 2H), 7.19–7.09 (m, 3H), 7.00–6.93 (m, 5H), 6.90–6.83 (m, 2H), 5.45 (d, *J* = 4.6 Hz, 1H), 4.98 (t, *J* = 4.6 Hz, 1H), 3.23 (td, *J* = 11.7, 6.5 Hz, 1H), 2.82 (dd, *J* = 10.7, 7.1 Hz, 2H) ppm. ^{13}C NMR (100 MHz, DMSO-d_6): δ = 144.3, 140.8, 140.7, 135.7, 134.8, 134.6, 133.6, 131.9, 131.9, 131.5, 131.5, 130.9, 130.8, 130.7, 130.6, 129.7, 129.1, 129.0, 128.8, 128.7, 127.9, 127.5, 127.1, 126.7, 126.4, 76.0, 75.9, 47.4, 47.3, 32.5, 31.8 ppm. ^{31}P NMR (162 MHz, DMSO-d_6): δ = 29.09 ppm. HRMS m/z: calcd for $C_{27}H_{26}O_2P$ $[M+H]^+$ 413.1670, found: 413.1666.

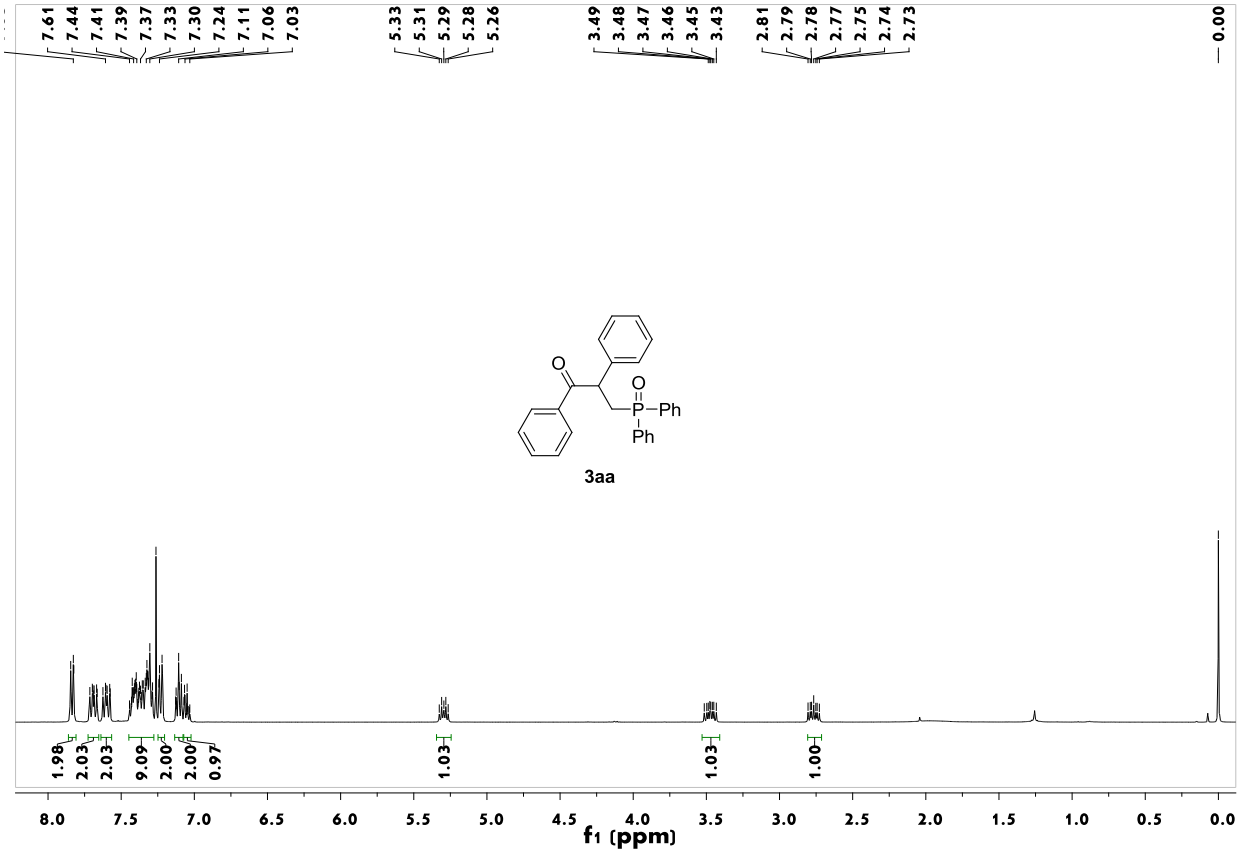


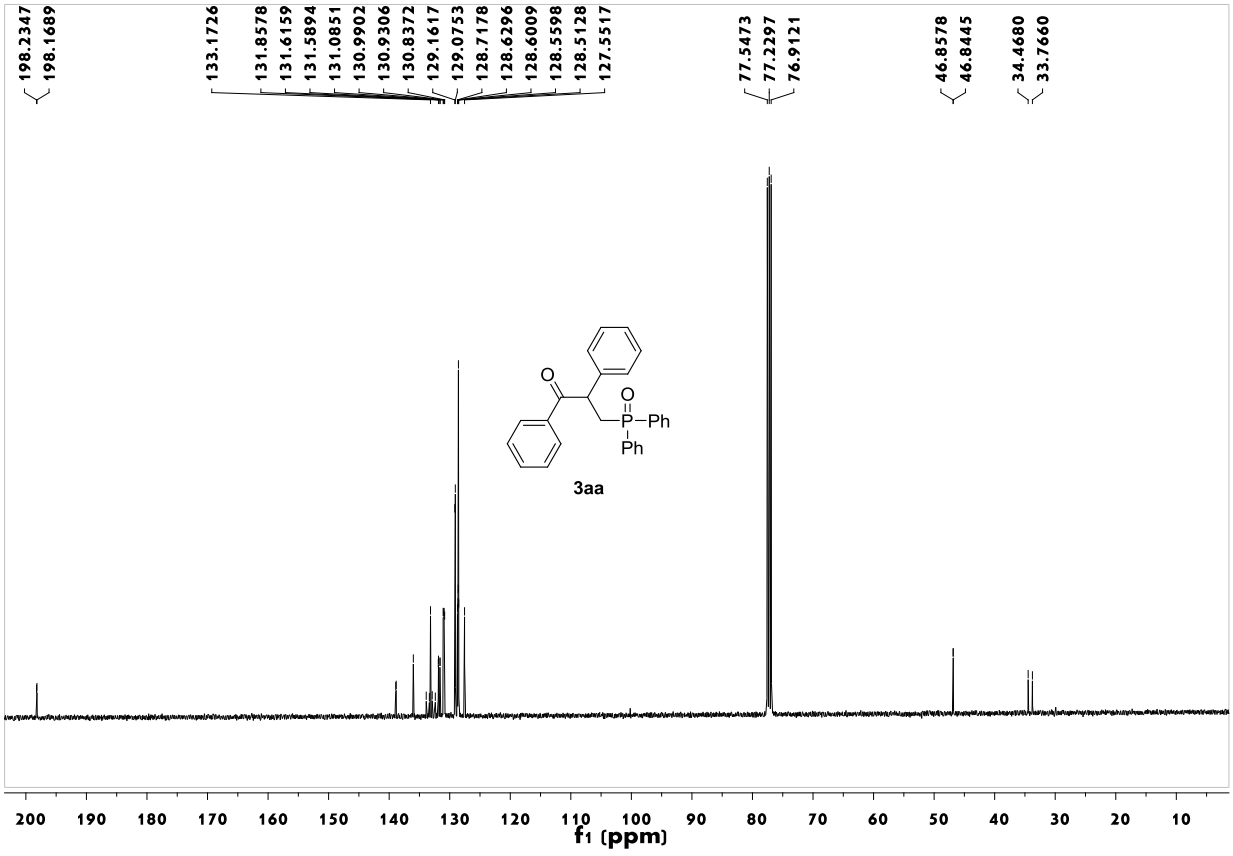
4b

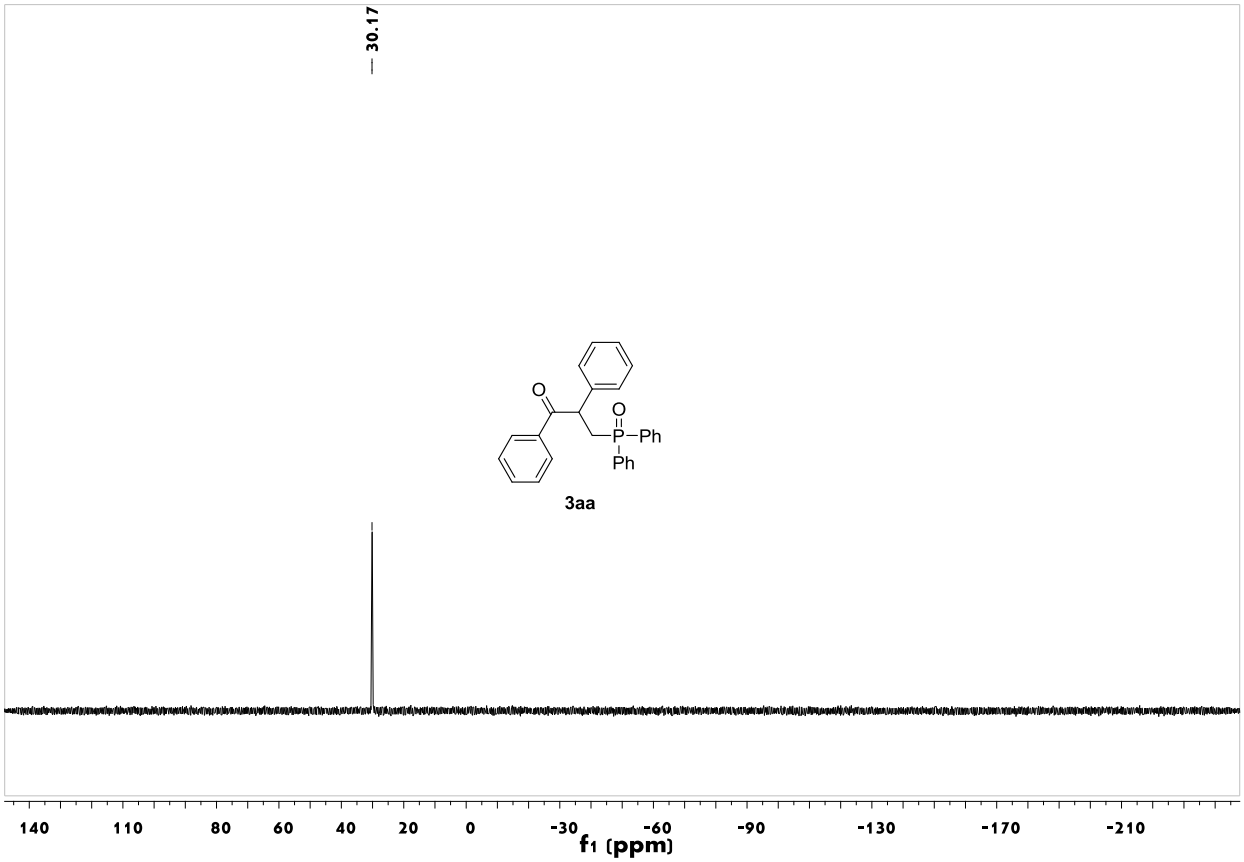
3-(diphenylphosphoryl)-1,1,2-triphenylpropan-1-ol (4a): Yield = 79%. White soild. Mp>

300 ℃. IR (KBr) ν = 3402, 3060, 1492, 1448, 1436, 1173, 1143, 1118, 1098, 912, 897, 868, 780, 746, 692 cm^{-1}. ^{1}H NMR (400 MHz, DMSO-d_6) δ = 7.74–7.65 (m, 2H), 7.63–7.55 (m, 2H), 7.52–7.40 (m, 3H), 7.38–7.17 (m, 6H), 7.12 (d, *J* = 7.2 Hz, 4H), 6.95 (dd, *J* = 15.1, 5.5 Hz, 4H), 6.88–6.79 (m, 1H), 6.75–6.59 (m, 3H), 5.99 (s, 1H), 4.35 (t, *J* = 12.1 Hz, 1H), 3.15–3.04 (m, 1H), 2.62 (d, *J* = 3.6 Hz, 1H) ppm. ^{13}C NMR (150 MHz, CF_3COOD): *δ* = 50.5, 150.4, 144.9, 144.5, 142.5, 137.2, 134.6, 134.6, 133.11, 133.0, 132.3, 132.2, 132.0, 131.9, 131.2, 130.8, 130.8, 130.7, 130.5, 129.8, 129.0, 128.4, 66.3, 39.8, 39.3, 22.2, 22.0 ppm. ^{31}P NMR (162 MHz, DMSO-d_6): *δ* = 28.75 ppm. HRMS m/z: calcd for $C_{33}H_{30}O_2P$ $[M+H]^+$ 489.1983, found: 489.2008.

The ^{1}H , ^{13}C and ^{31}P NMR spectra of compounds:



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P–Ph
Ph
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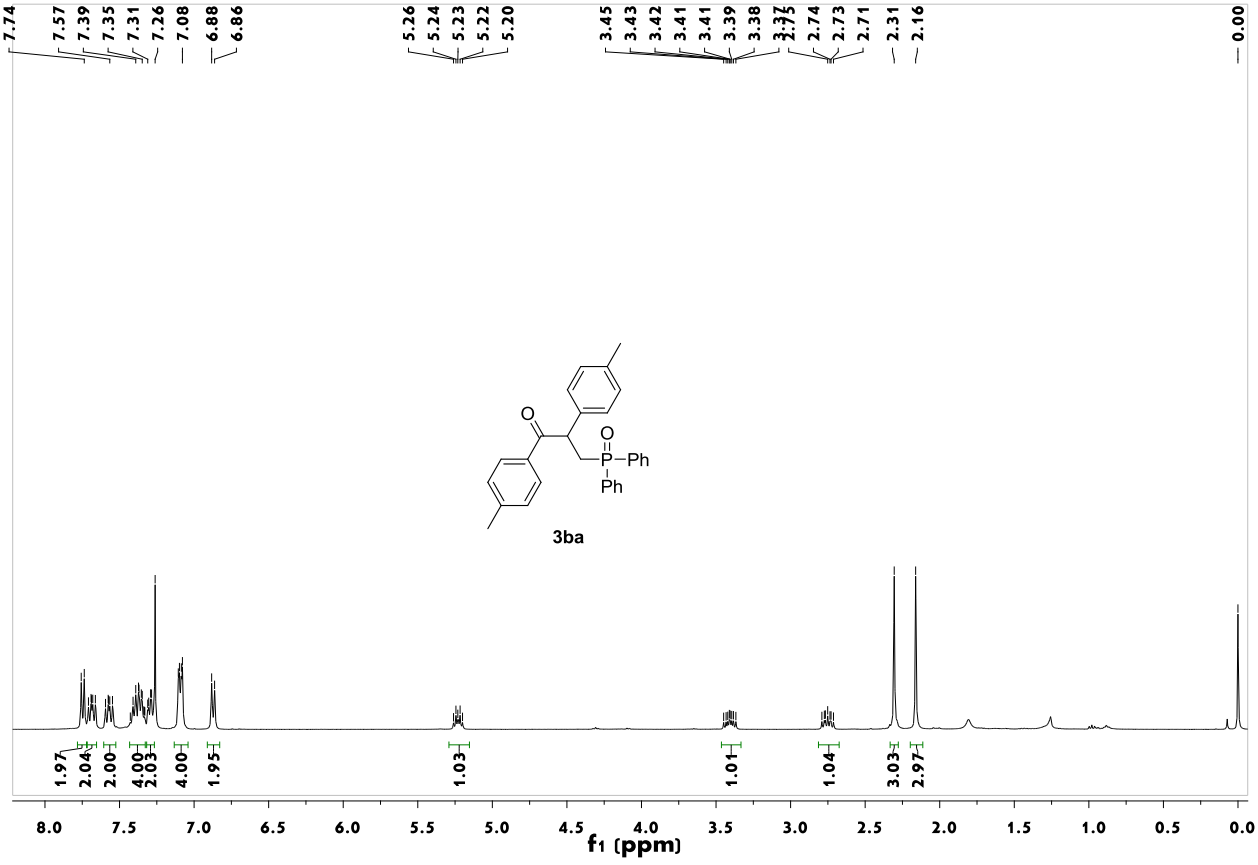
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210
f1 (ppm)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7.08
6.88
6.86
5.26
5.24
5.23
5.22
5.20
3.45
3.43
3.42
3.41
3.41
3.39
3.38
2.74
2.73
2.71
2.31
2.16
0.00
O
O
P–Ph
Ph
3ba
1.97
2.04
2.00
4.00
2.03
4.00
1.95
1.03
1.01
1.04
3.03
2.97
8.0
7.5
7.0
6.5
6.0
5.5
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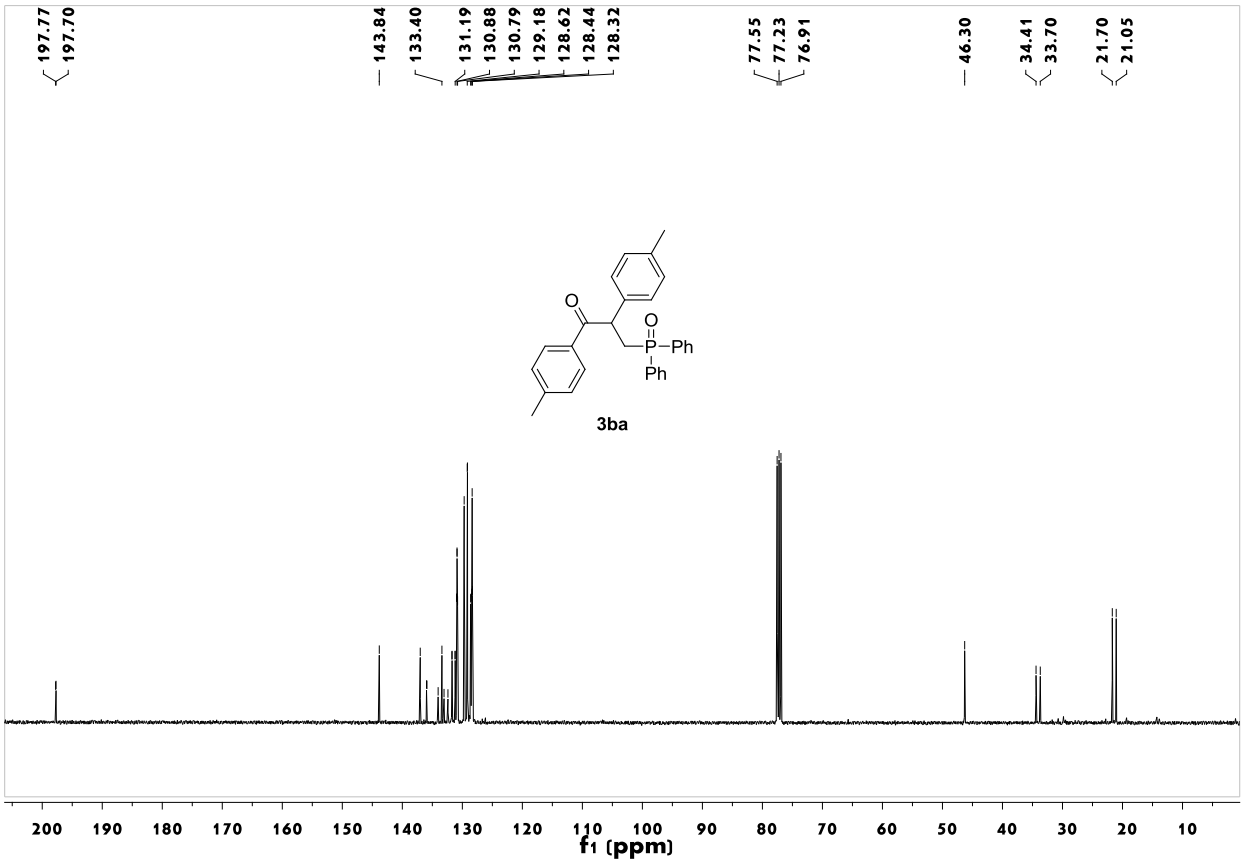
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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133.40
131.19
130.88
130.79
129.18
128.62
128.44
128.32
77.55
77.23
76.91
46.30
34.41
33.70
21.70
21.05
O
O
P–Ph
Ph
3ba
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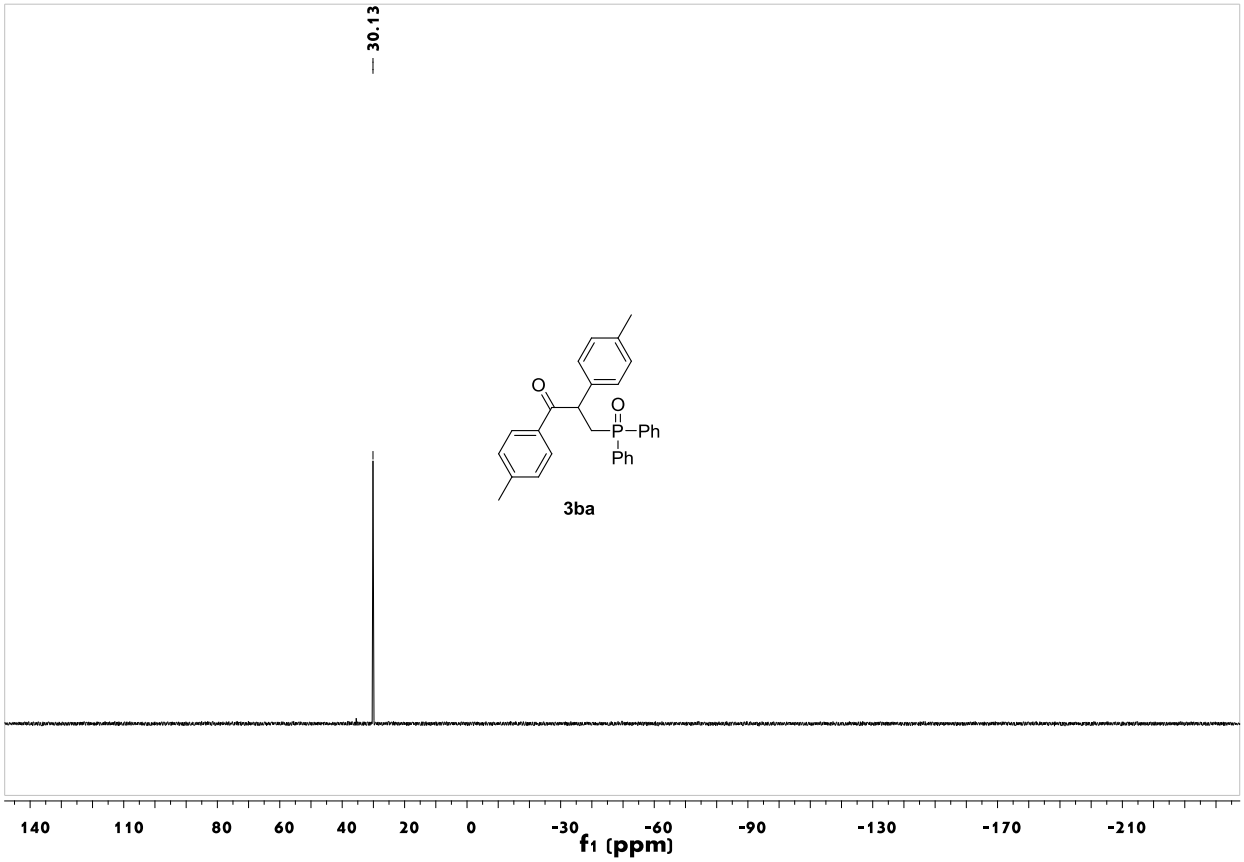
f1 (ppm)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O
P–Ph
Ph
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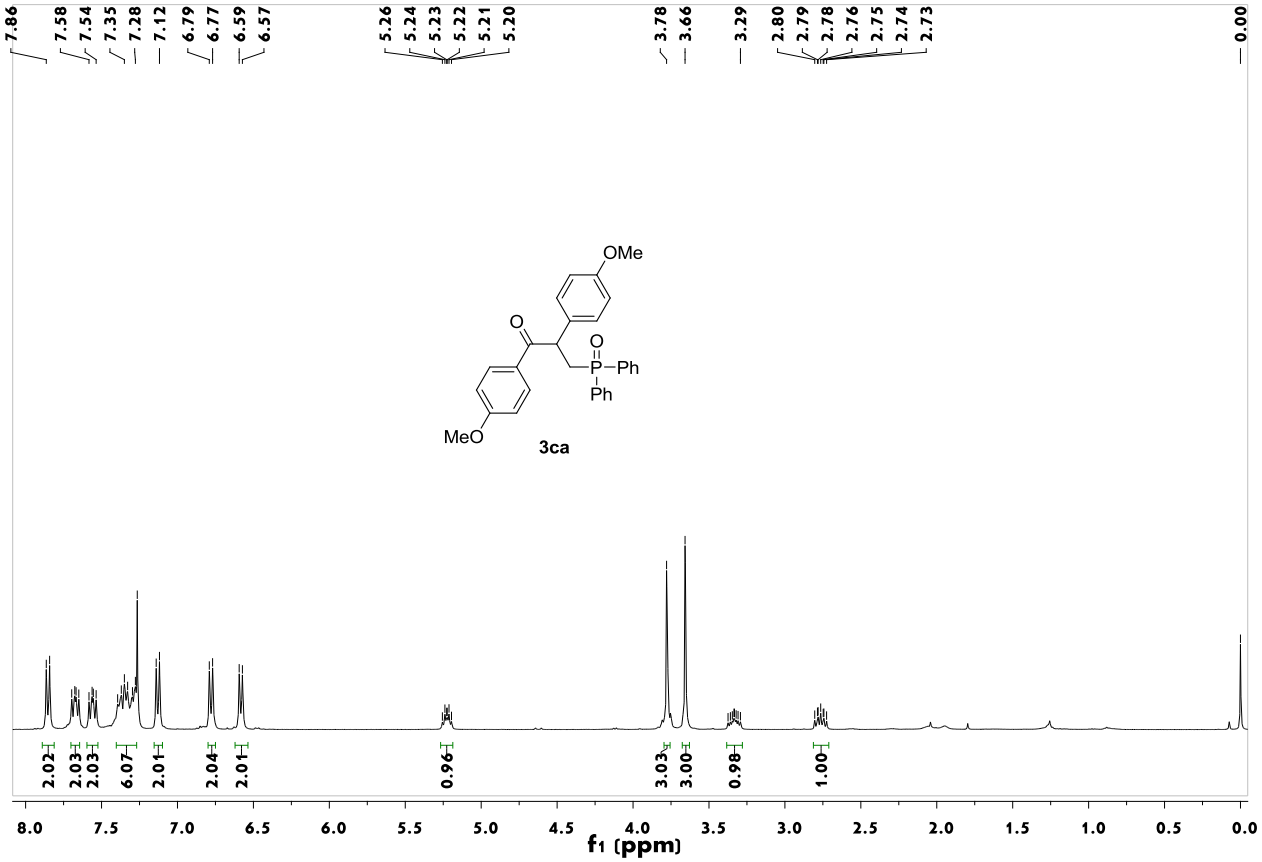
f1 (ppm)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6.77
6.59
6.57
5.26
5.24
5.23
5.22
5.21
5.20
3.78
3.66
3.29
2.80
2.79
2.78
2.76
2.75
2.74
2.73
0.00
OMe
O
O
P–Ph
Ph
MeO
3ca
2.02
2.03
2.03
6.07
2.01
2.04
2.01
0.96
3.03
3.00
0.98
1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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3.0
2.5
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1.0
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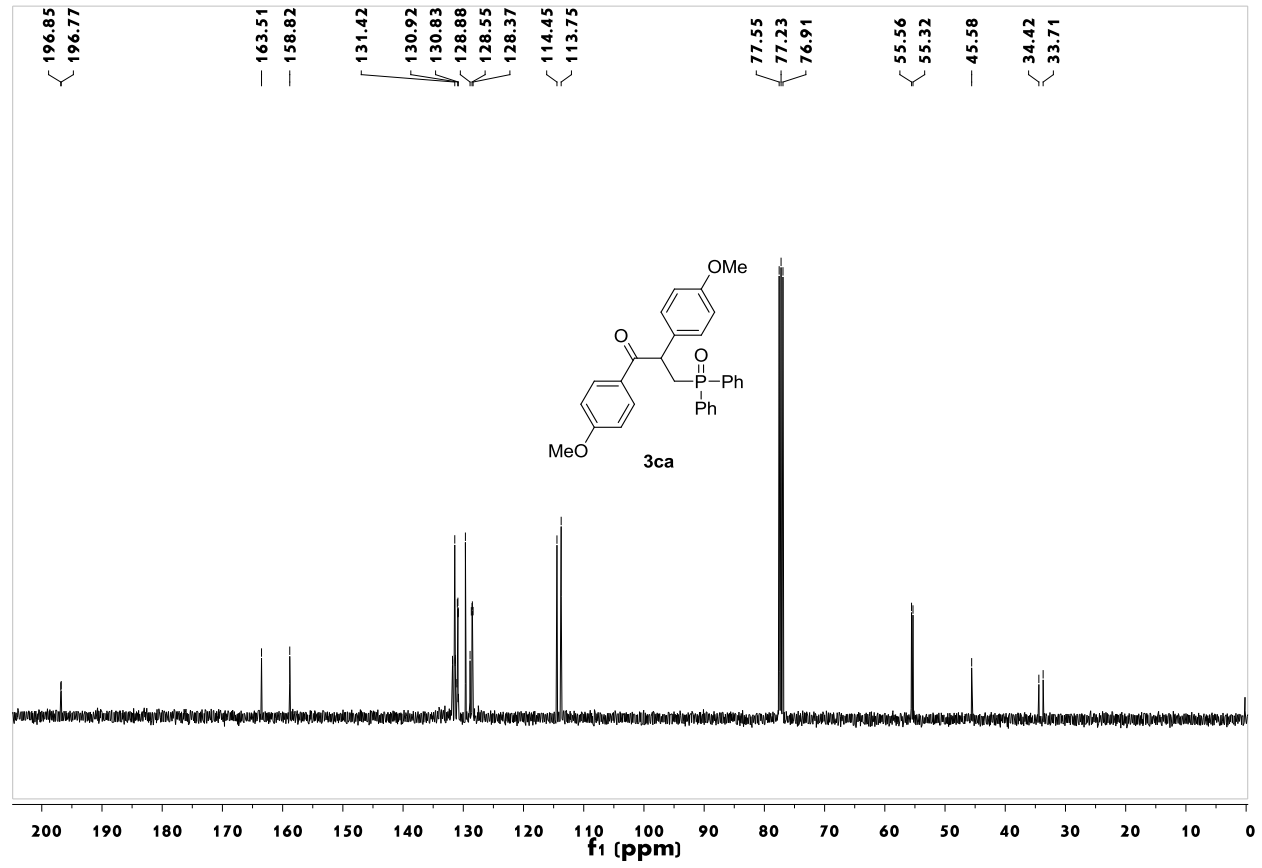
0.0
f1 (ppm)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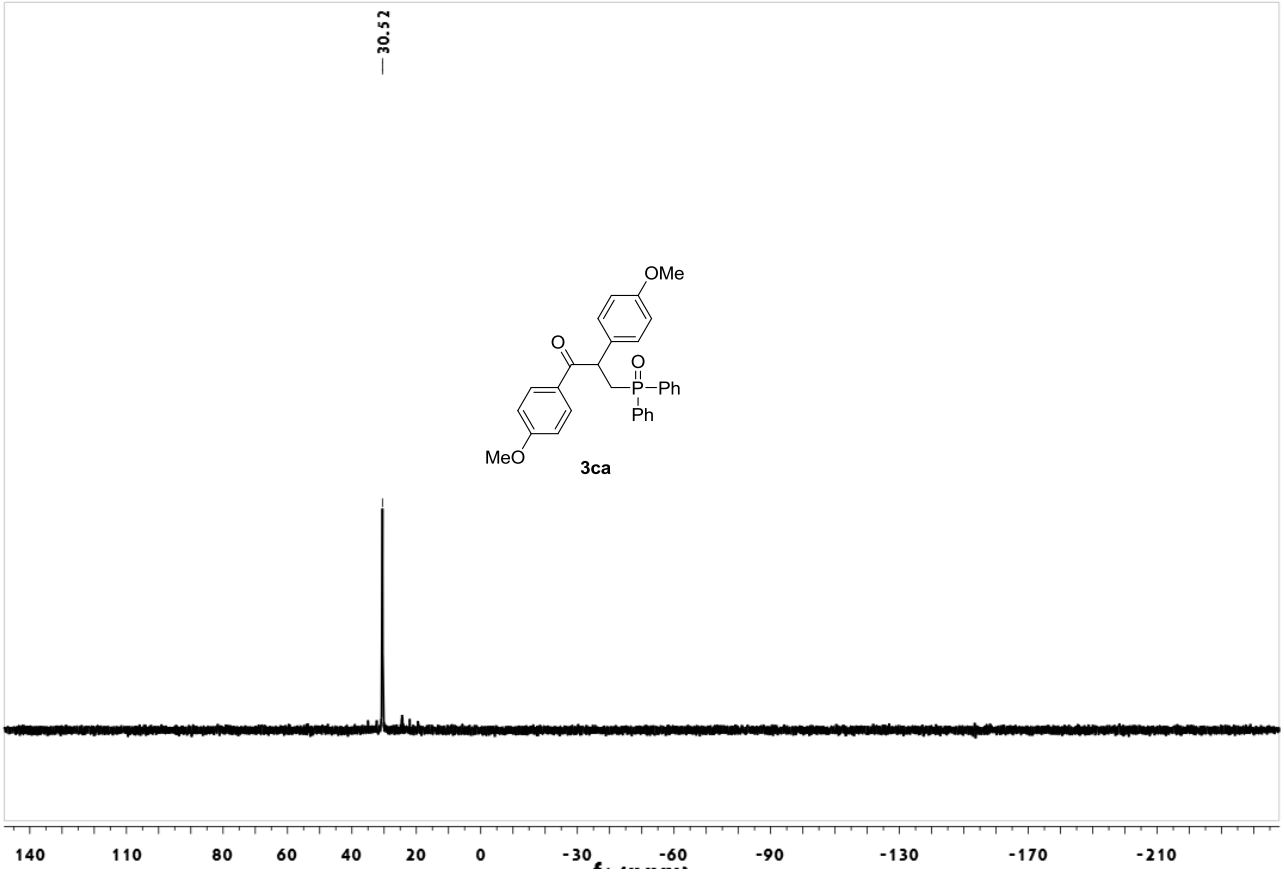
128.88
128.55
128.37
114.45
113.75
77.55
77.23
76.91
55.56
55.32
45.58
34.42
33.71
OMe
O
O
P–Ph
Ph
MeO
3ca
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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Ph
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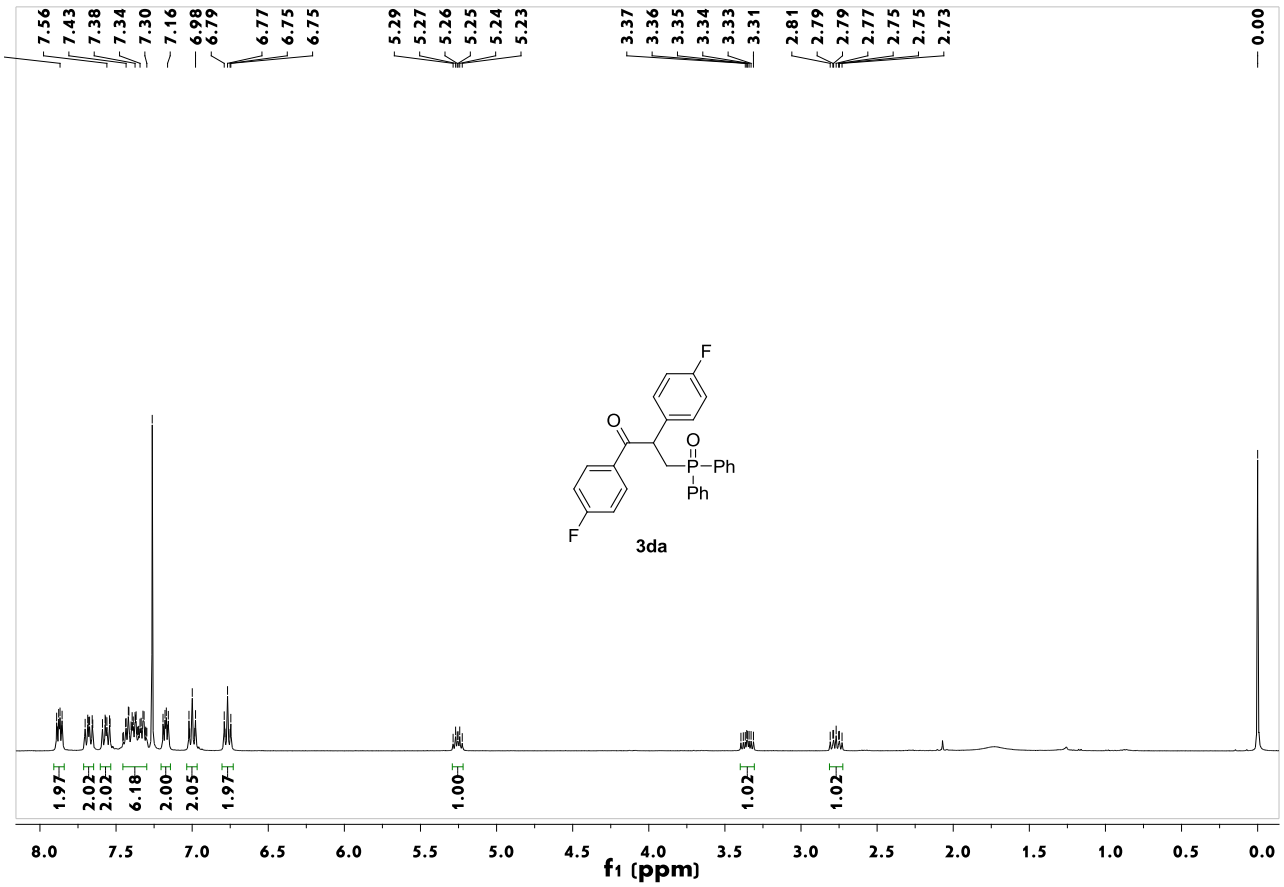
-130
-170
-210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7.16
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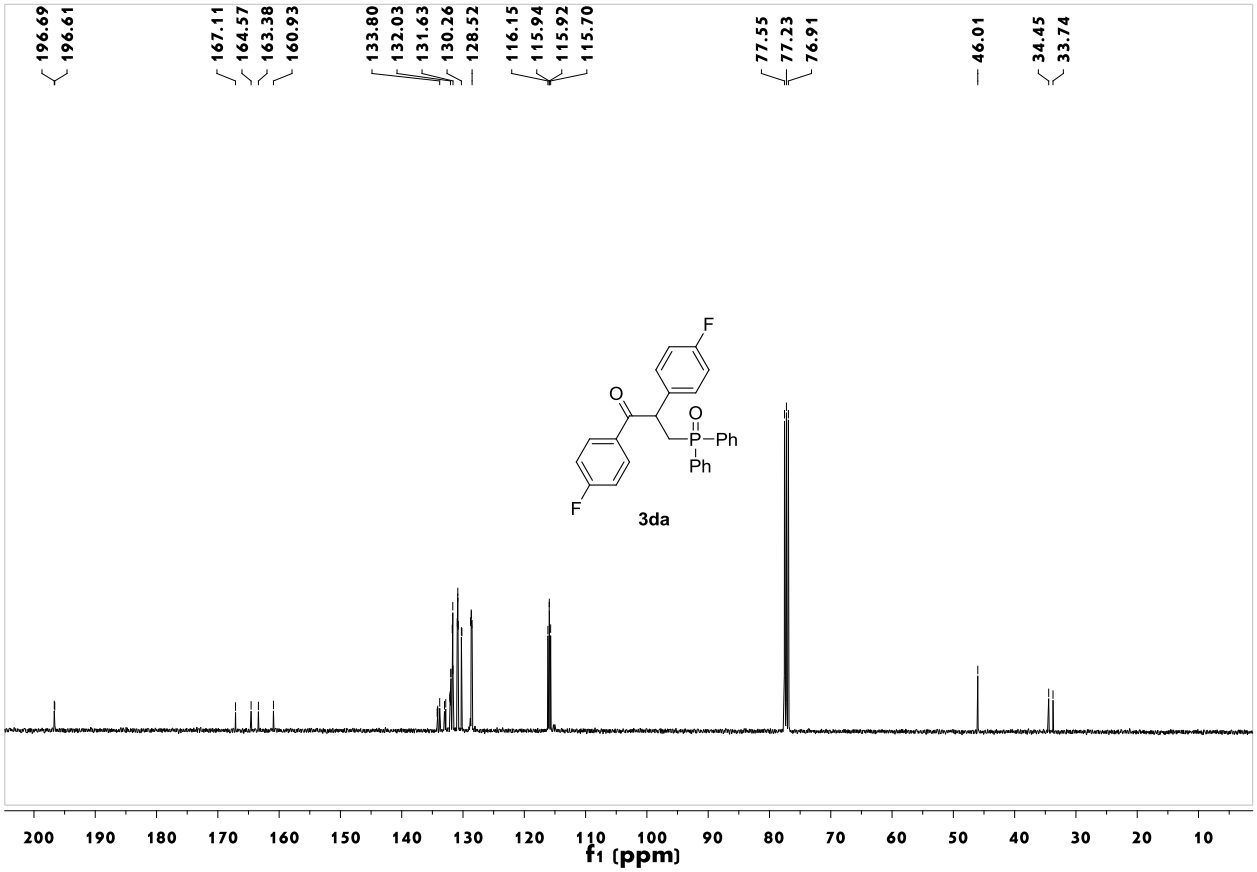
6.79
6.77
6.75
6.75
5.29
5.27
5.26
5.25
5.24
5.23
3.37
3.36
3.35
3.34
3.33
3.31
2.81
2.79
2.79
2.77
2.75
2.75
2.73
0.00
F
O
O
P–Ph
Ph
F
3da
1.97
2.02
2.02
6.18
2.00
2.05
1.97
1.00
1.02
1.02
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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130.26
128.52
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115.94
115.92
115.70
77.55
77.23
76.91
46.01
34.45
33.74
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O
O
P–Ph
Ph
F
3da
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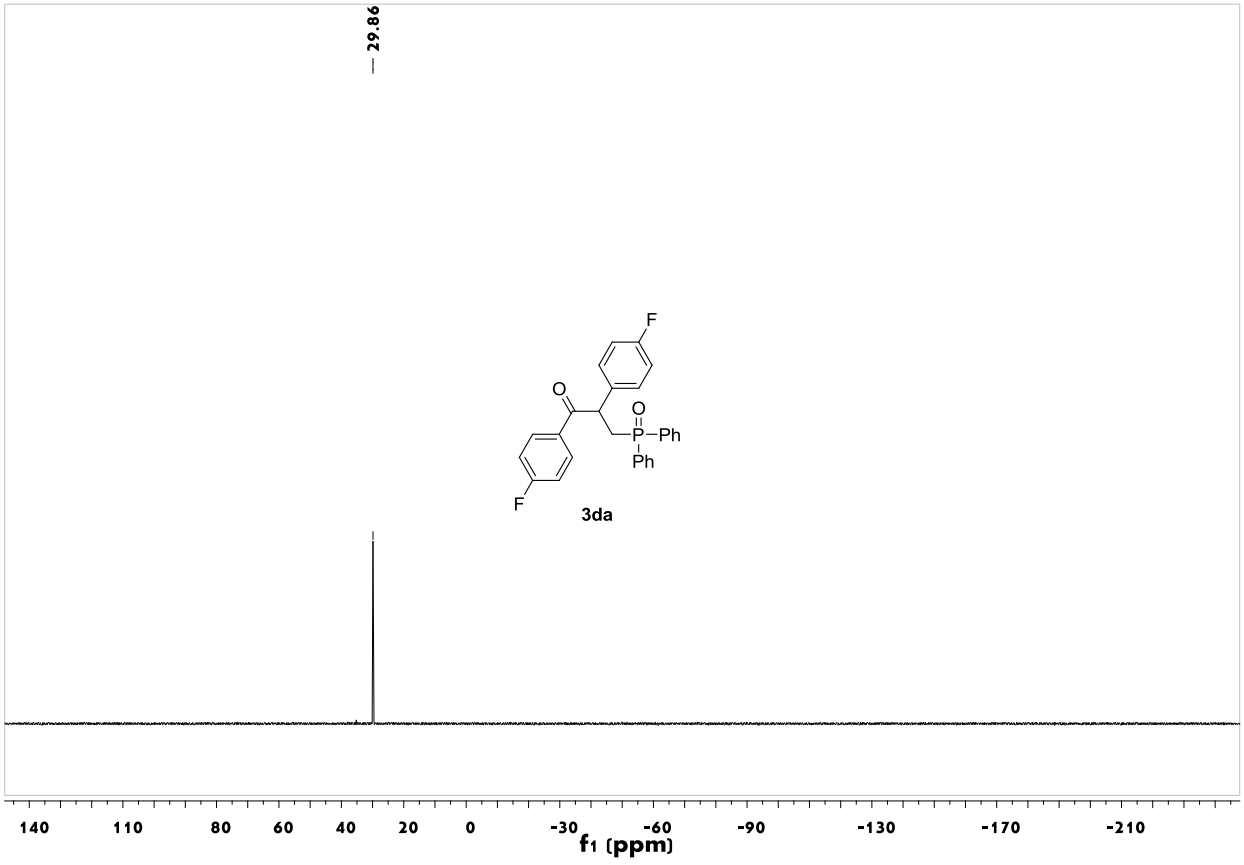
f1 (ppm)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P–Ph
Ph
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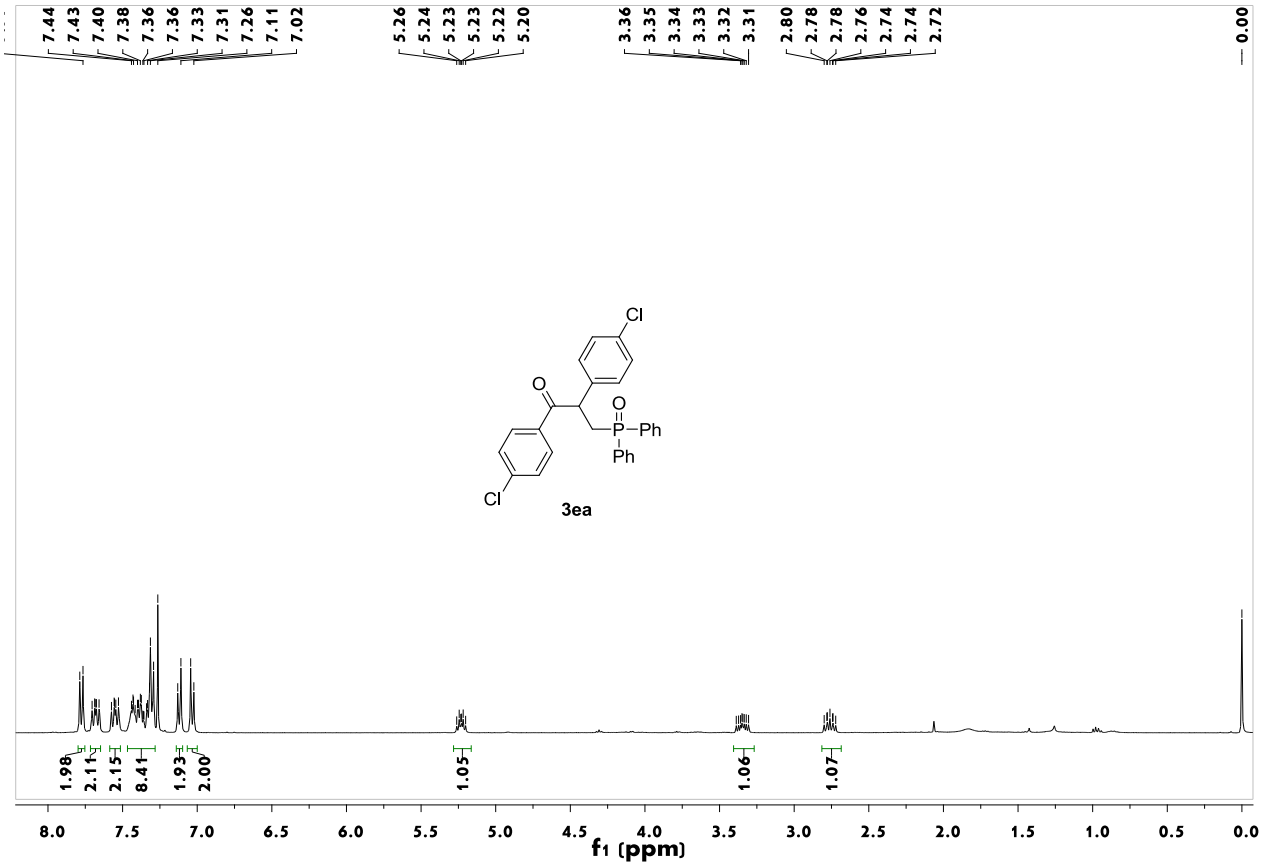
f1 (ppm)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5.23
5.23
5.22
5.20
3.36
3.35
3.34
3.33
3.32
3.31
2.80
2.78
2.78
2.76
2.74
2.74
2.72
0.00
Cl
O
O
P–Ph
Ph
Cl
3ea
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2.11
2.15
8.41
1.93
2.00
1.05
1.06
1.07
8.0
7.5
7.0
6.5
6.0
5.5
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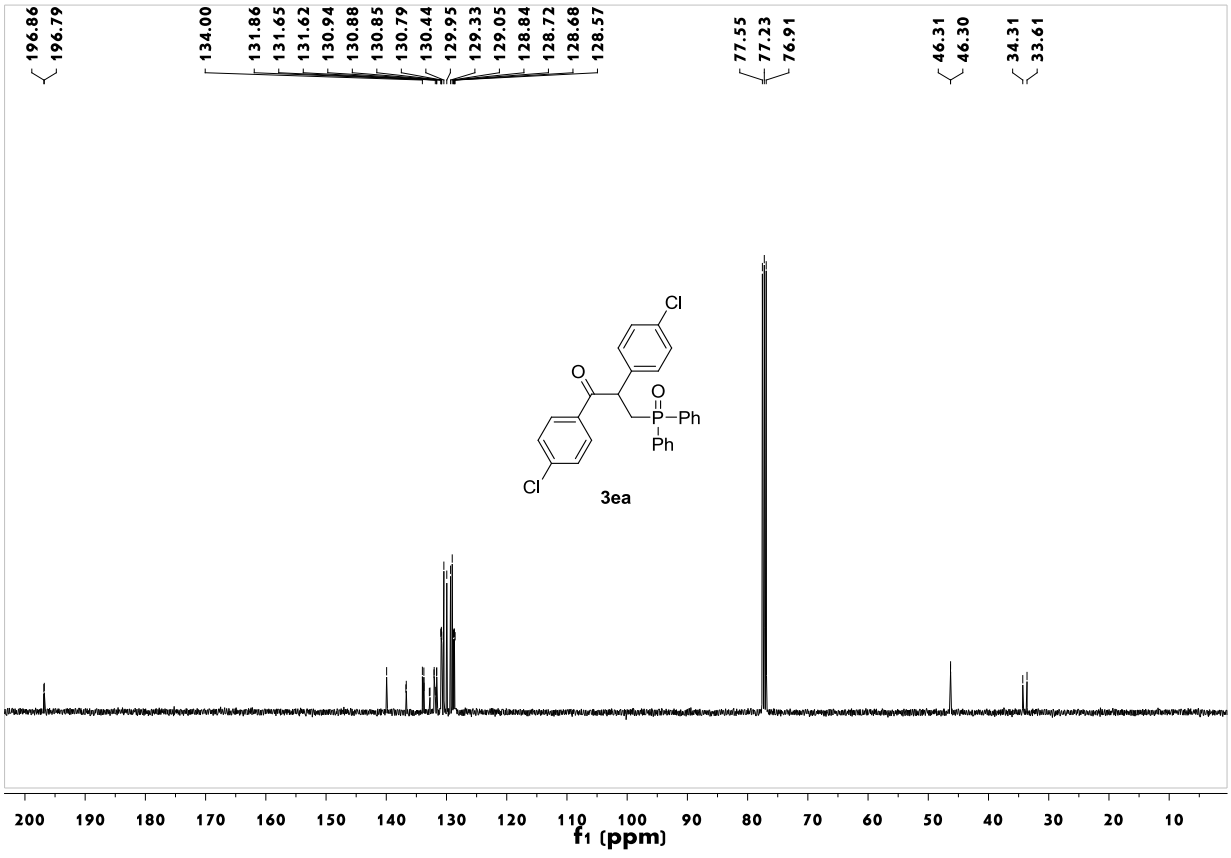
f1 (ppm)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129.33
129.05
128.84
128.72
128.68
128.57
77.55
77.23
76.91
46.31
46.30
34.31
33.61
Cl
O
O
P–Ph
Ph
Cl
3ea
200
190
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140
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120
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100
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80
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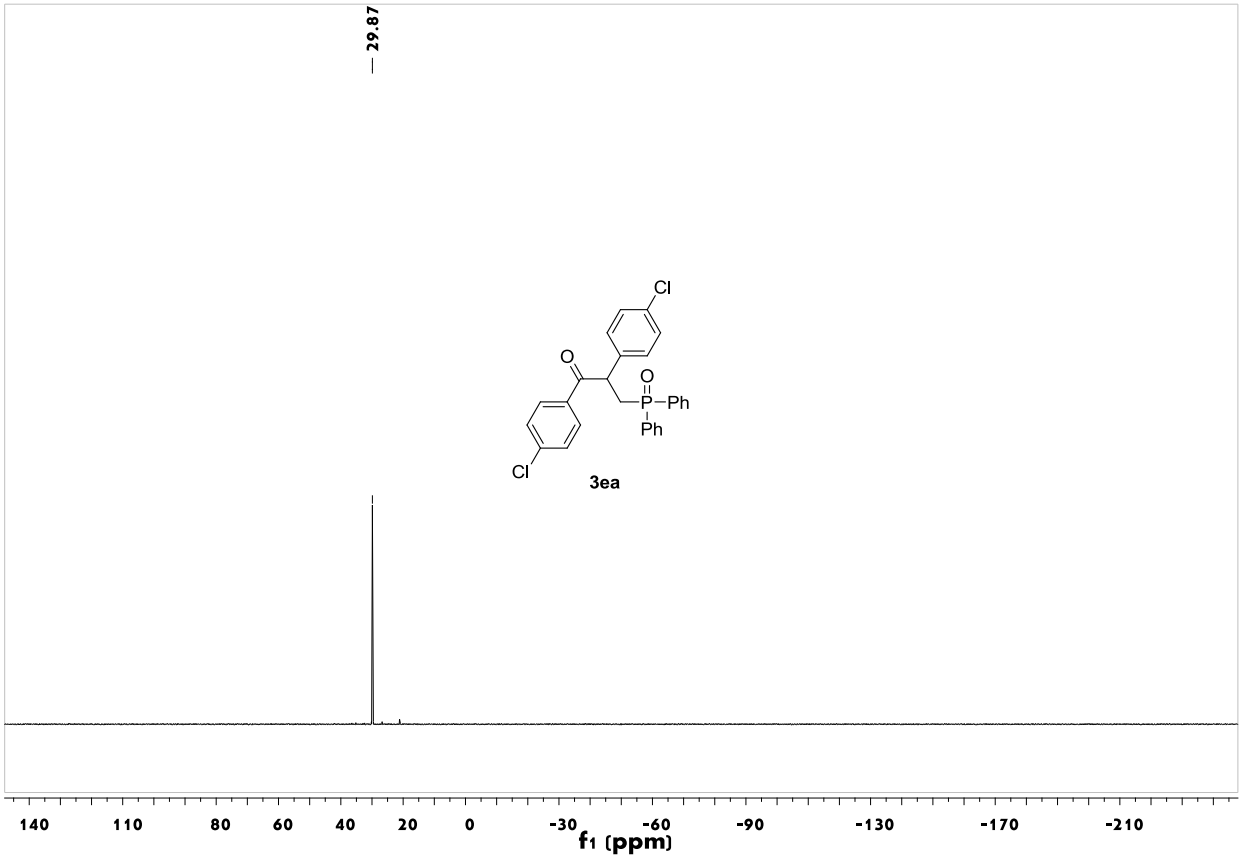
60
50
40
30
20
10
f1 (ppm)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O
P
Ph
Ph
Cl
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110
80
60
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20
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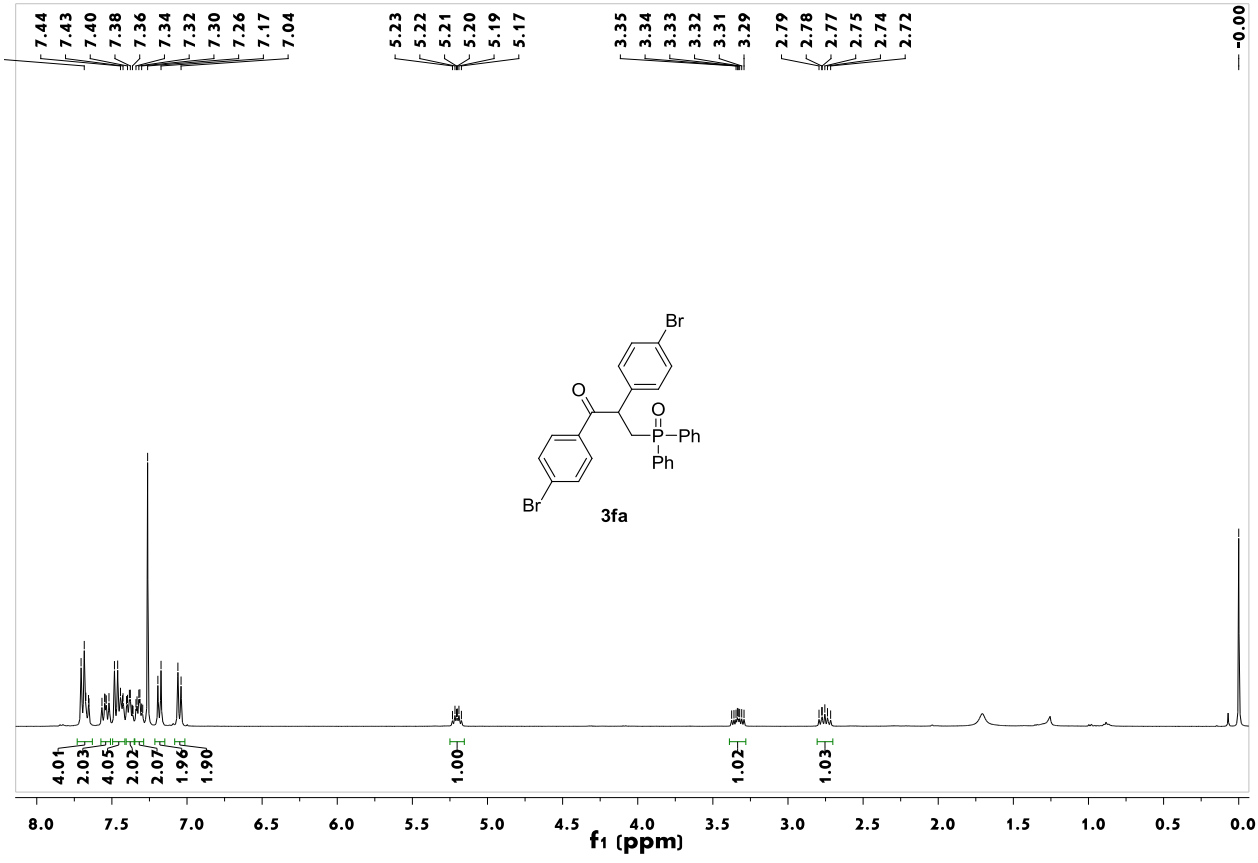
-60
-90
-130
-170
-210
f1 (ppm)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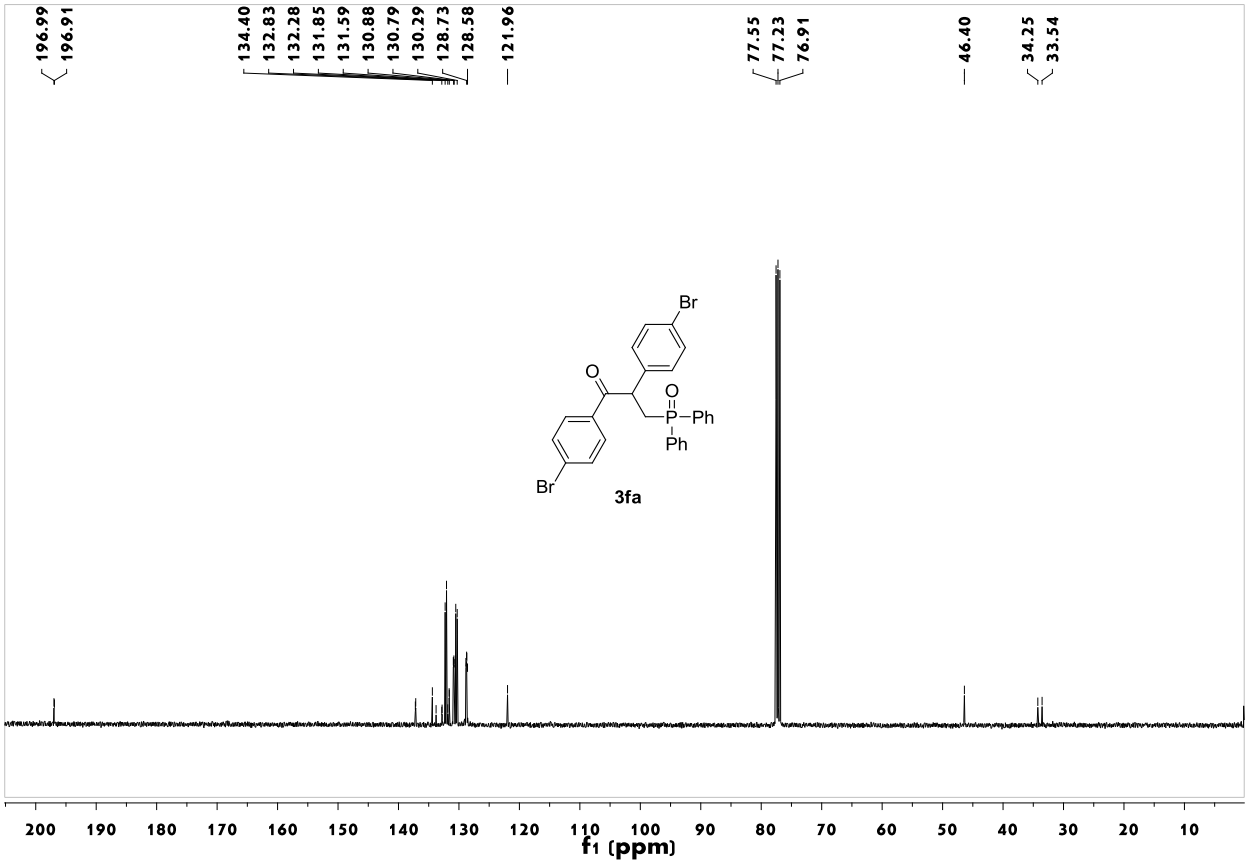
5.22
5.21
5.20
5.19
5.17
3.35
3.34
3.33
3.32
3.31
3.29
2.79
2.78
2.77
2.75
2.74
2.72
-0.00
Br
O
O
P
Ph
Ph
Br
3fa
4.01
2.03
4.05
2.02
2.07
1.96
1.90
1.00
1.02
1.03
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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131.85
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130.29
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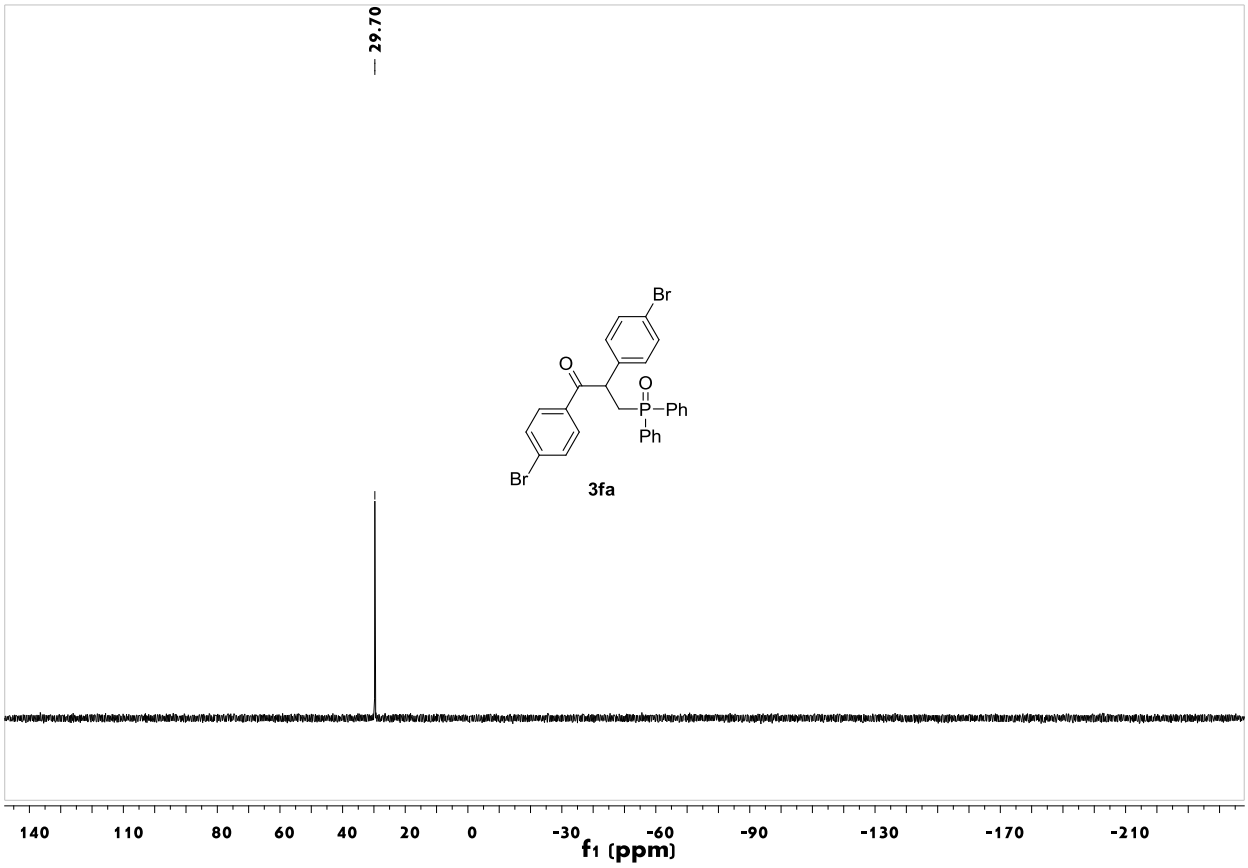
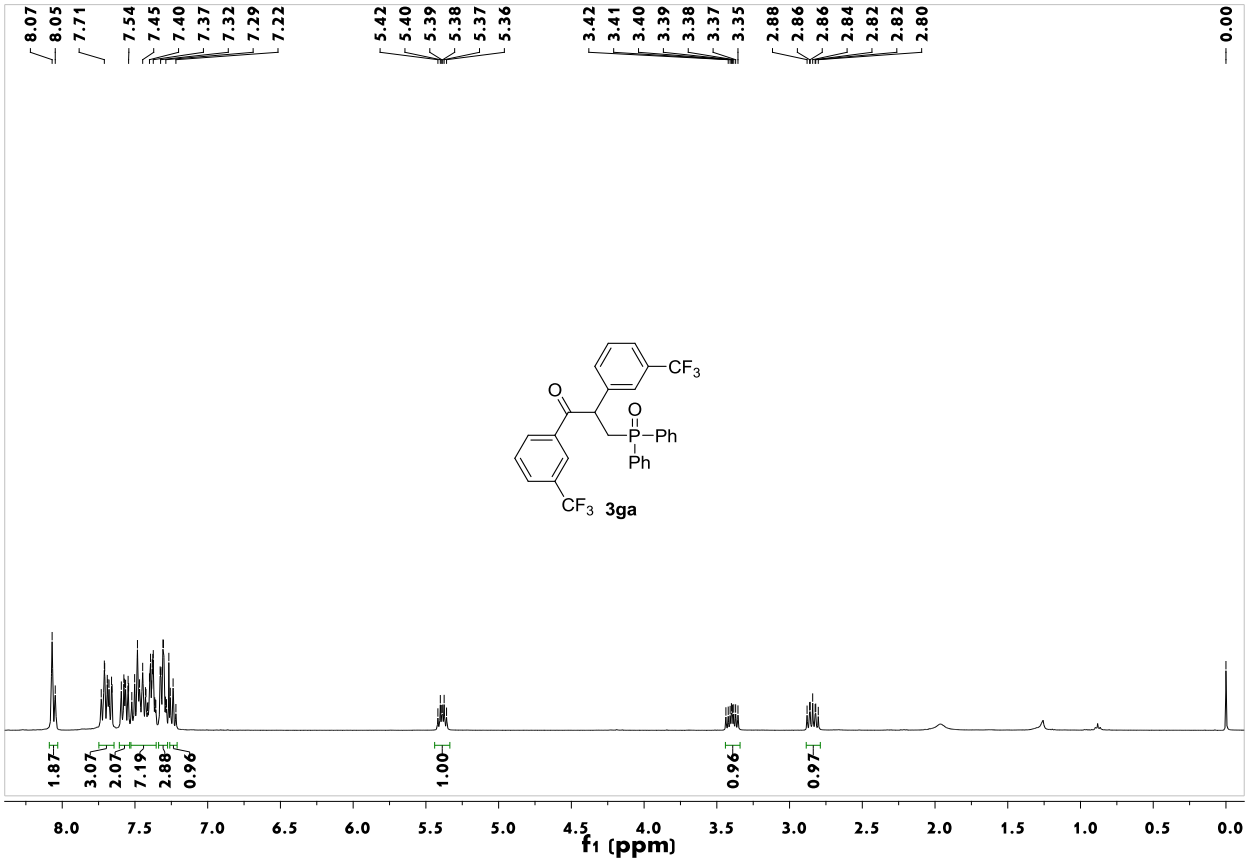
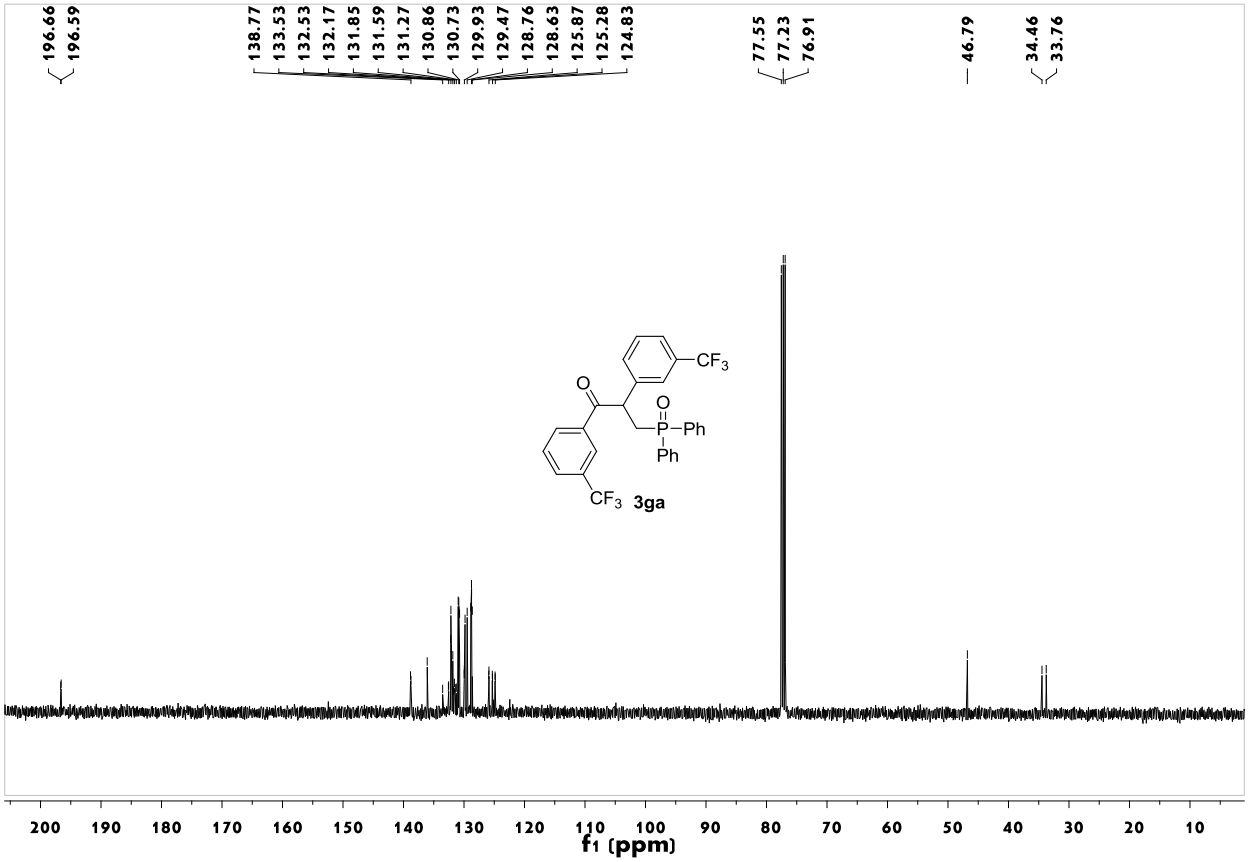
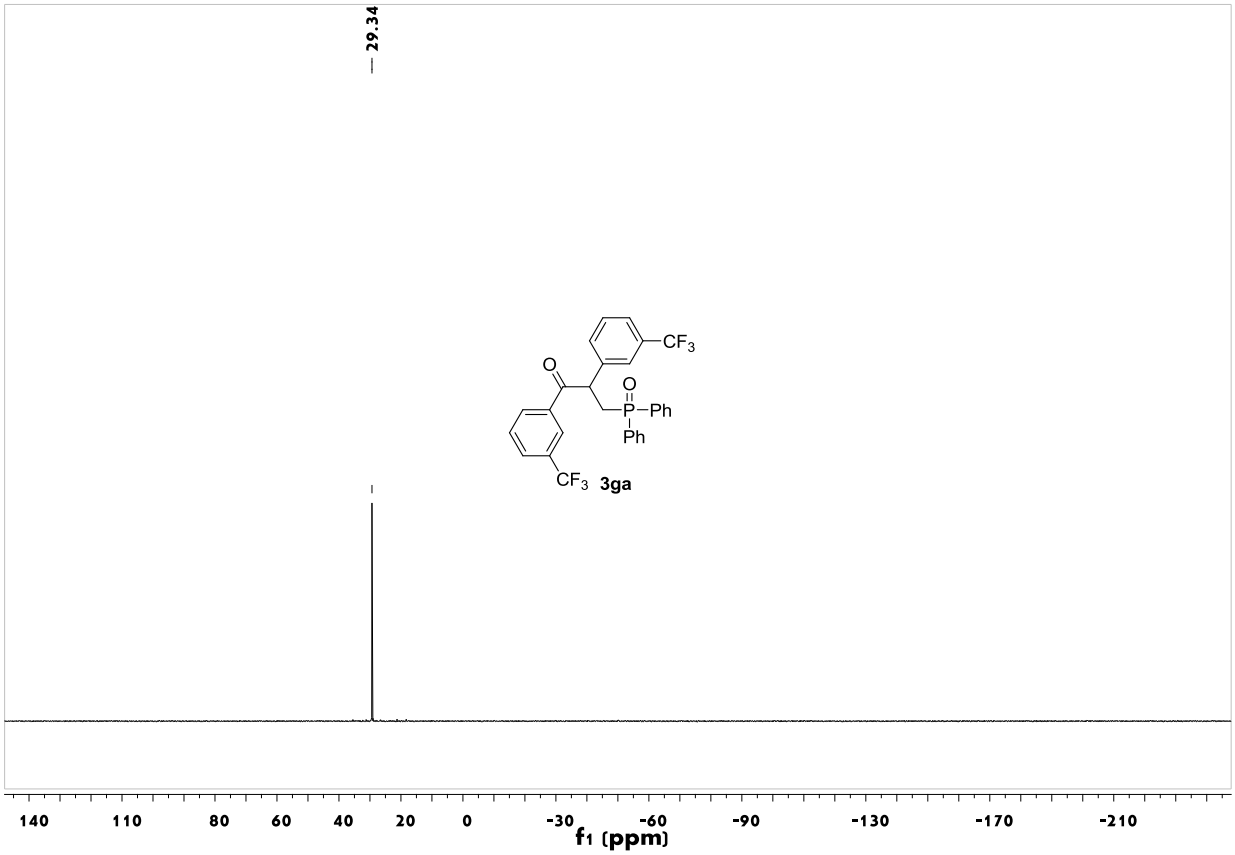
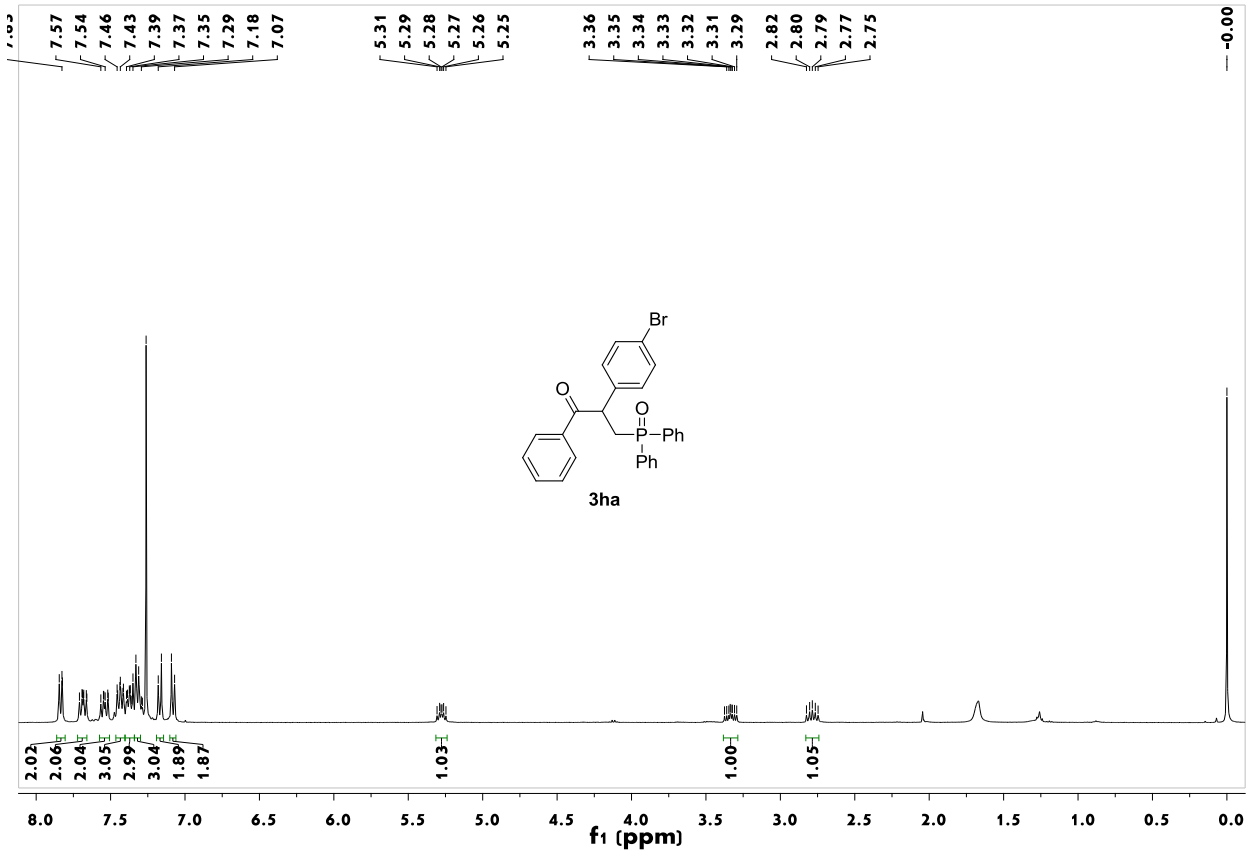
128.58
121.96
77.55
77.23
76.91
46.40
34.25
33.54
Br
O
O
P–Ph
Ph
Br
3fa
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

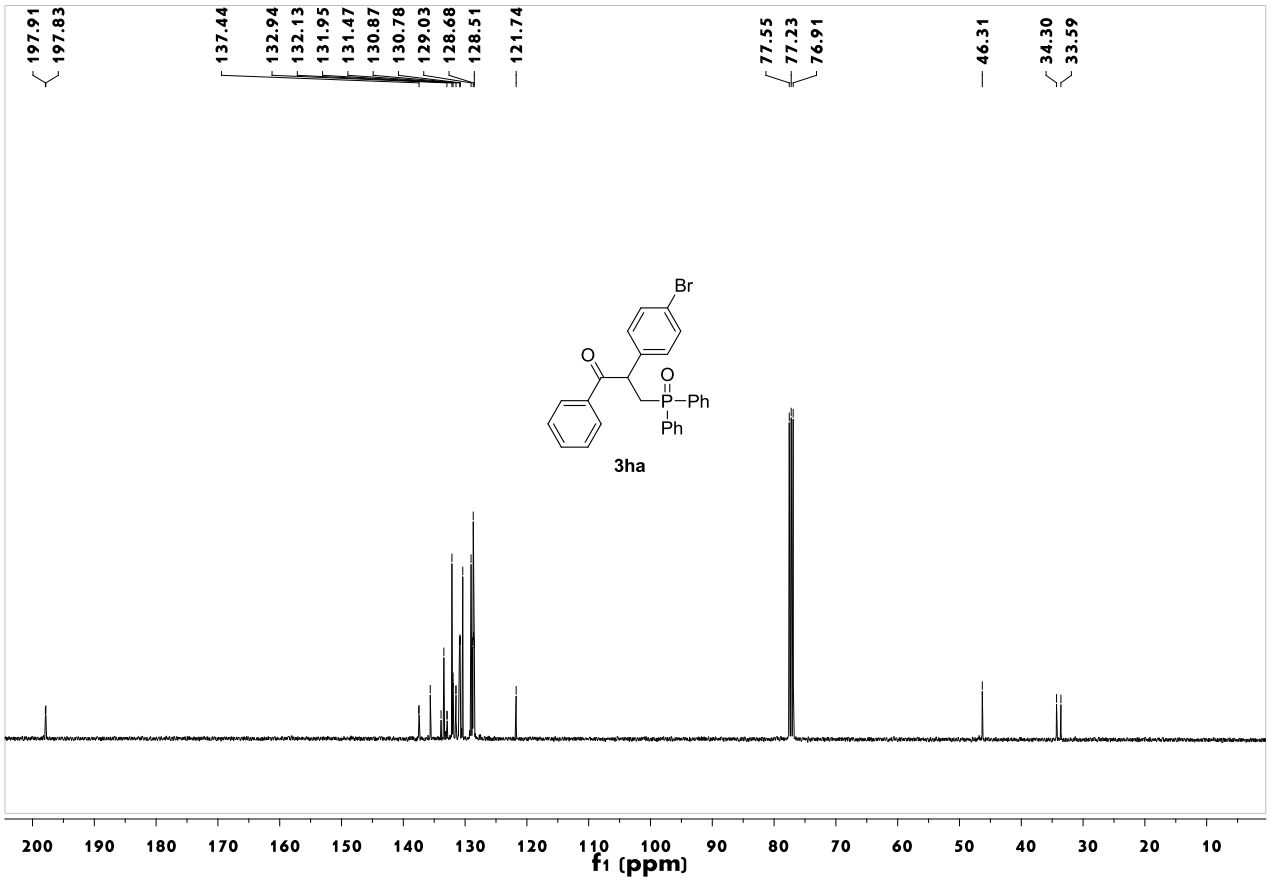

29.70
Br
O
O
P–Ph
Ph
Br
3fa
140 110 80 60 40 20 0 -30 -60 -90 -130 -170 -210
f1 (ppm)

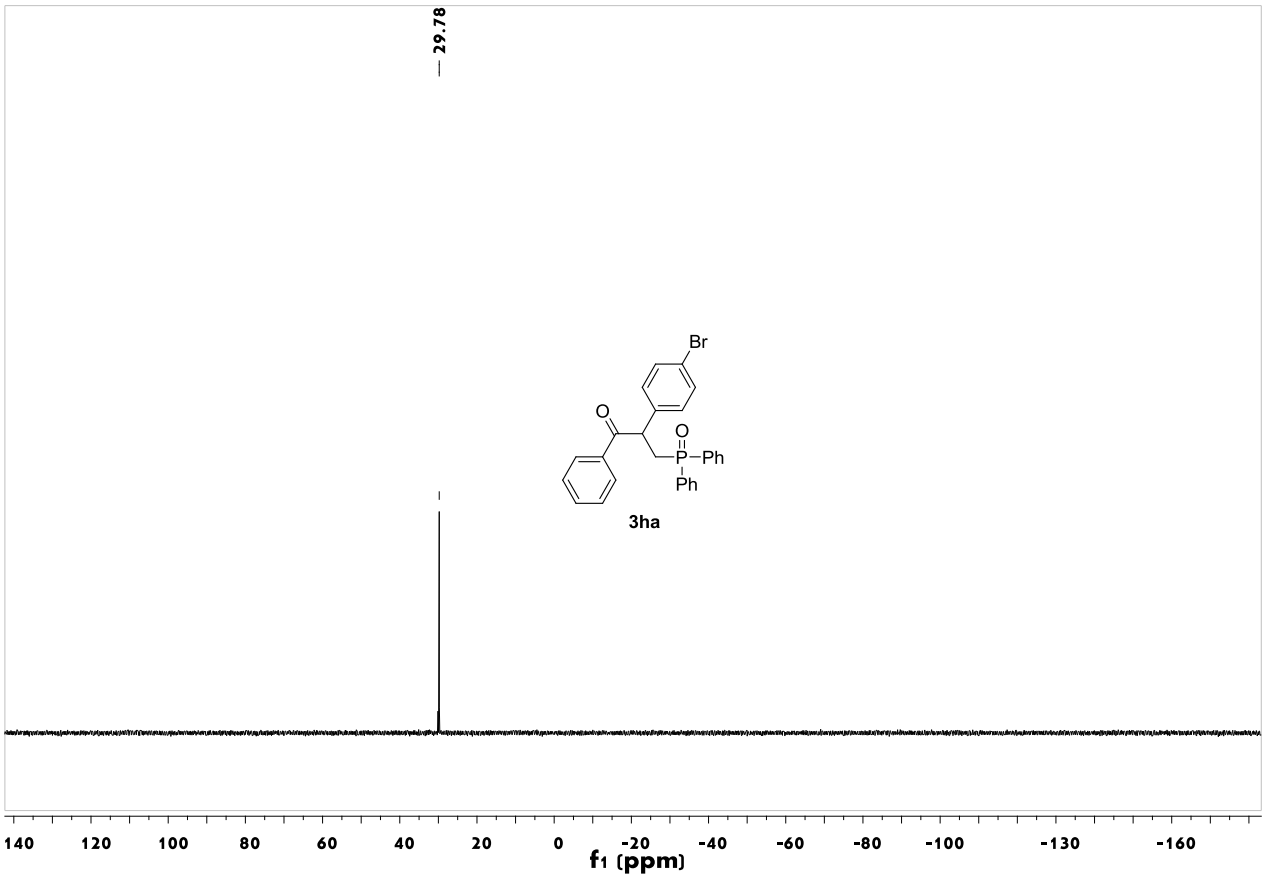

8.07
8.05
7.71
7.54
7.45
7.40
7.37
7.32
7.29
7.22
5.42
5.40
5.39
5.38
5.37
5.36
3.42
3.41
3.40
3.39
3.38
3.37
3.35
2.88
2.86
2.86
2.84
2.82
2.82
2.80
0.00
CF3
O
O
P
Ph
Ph
CF3
3ga
1.87
3.07
2.07
7.19
2.88
0.96
1.00
0.96
0.97
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

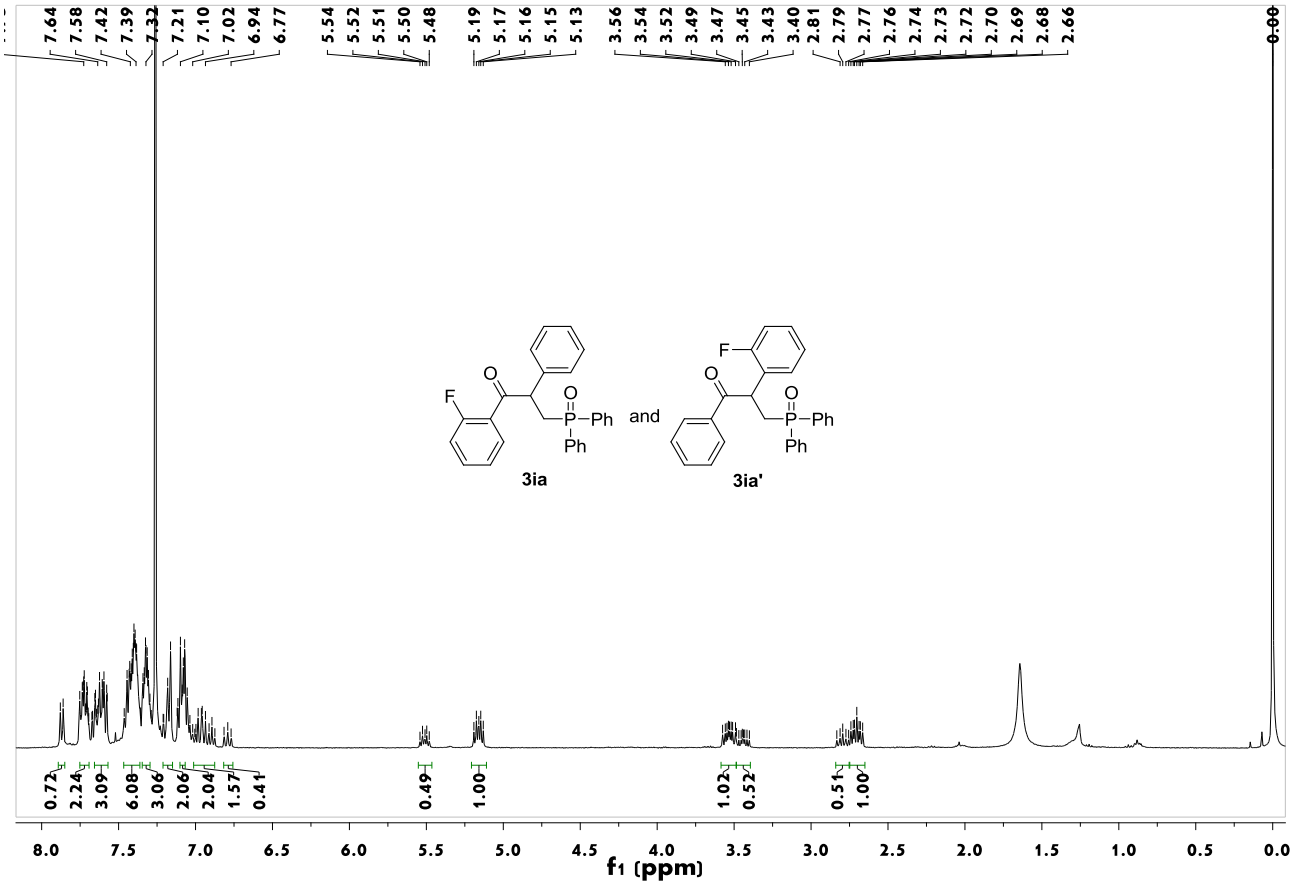

196.66
196.59
138.77
133.53
132.53
132.17
131.85
131.59
131.27
130.86
130.73
129.93
129.47
128.76
128.63
125.87
125.28
124.83
77.55
77.23
76.91
46.79
34.46
33.76
CF3
O
O
P
Ph
Ph
CF3
3ga
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

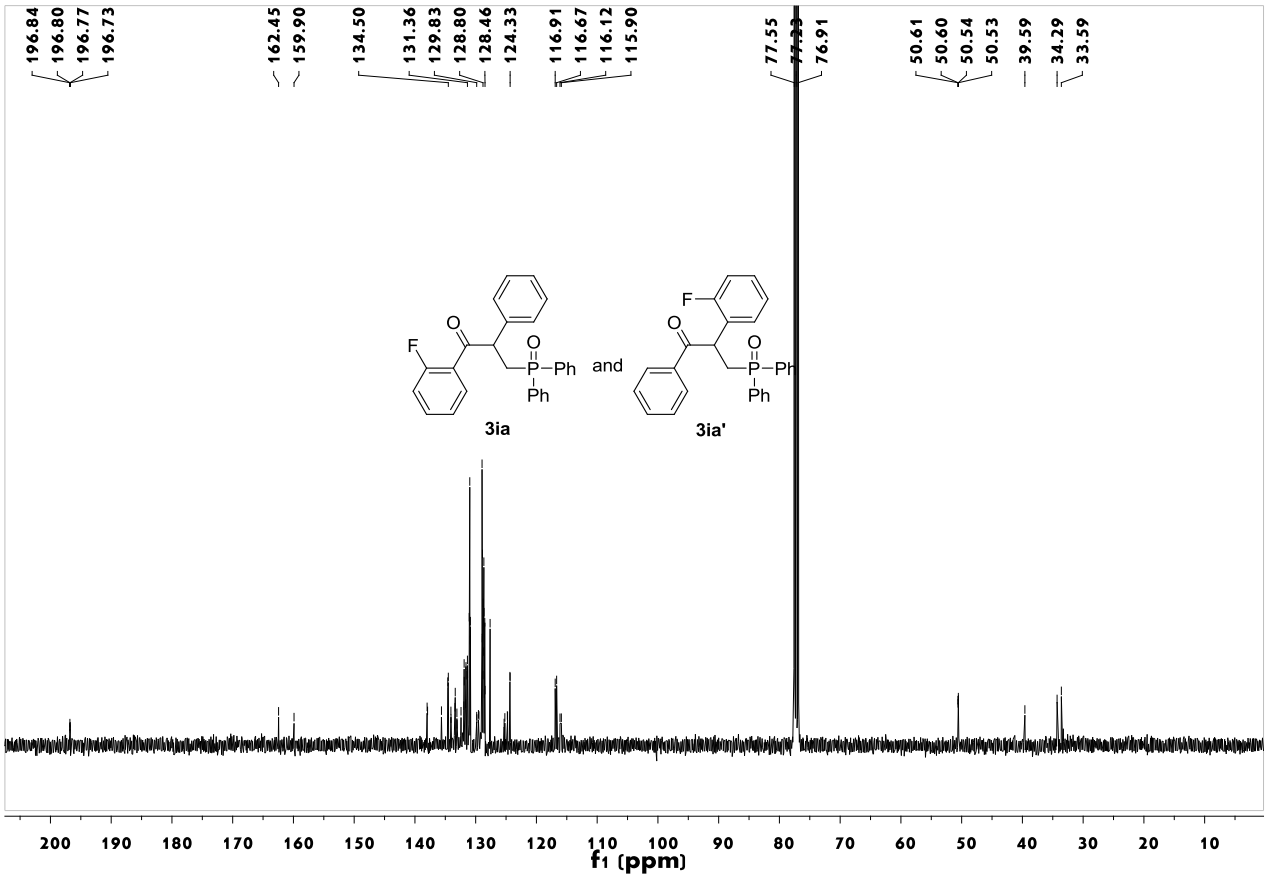

29.34
CF3
O
O
P
Ph
Ph
CF3
3ga
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210
f1 (ppm)

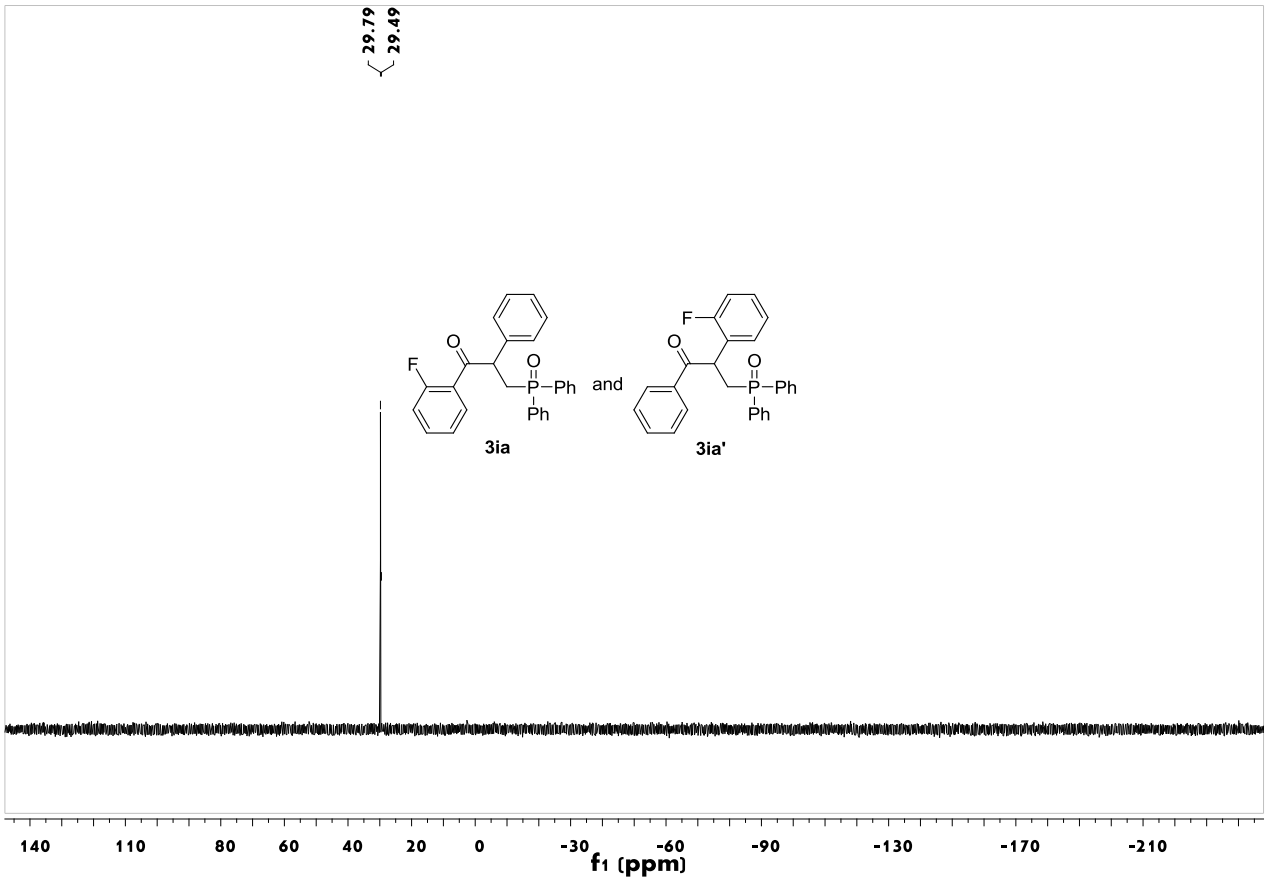

7.57
7.54
7.46
7.43
7.39
7.37
7.35
7.29
7.18
7.07
5.31
5.29
5.28
5.27
5.26
5.25
3.36
3.35
3.34
3.33
3.32
3.31
3.29
2.82
2.80
2.79
2.77
2.75
0.00
Br
O
O
P
Ph
Ph
3ha
2.02
2.06
2.04
3.05
2.99
3.04
1.89
1.87
1.03
1.00
1.05
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

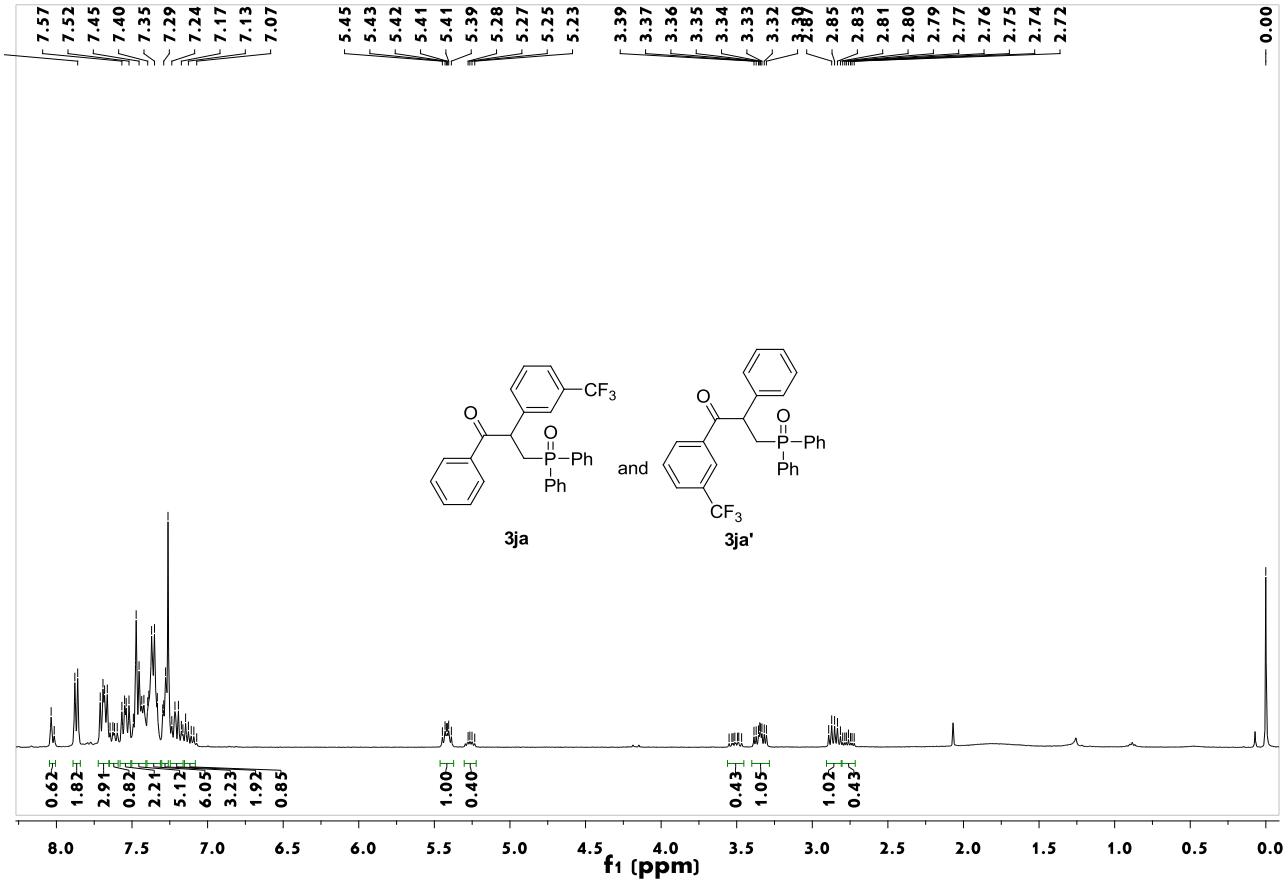

197.91
197.83
137.44
132.94
132.13
131.95
131.47
130.87
130.78
129.03
128.68
128.51
121.74
77.55
77.23
76.91
46.31
34.30
33.59
Br
O
O
P
Ph
Ph
3ha
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

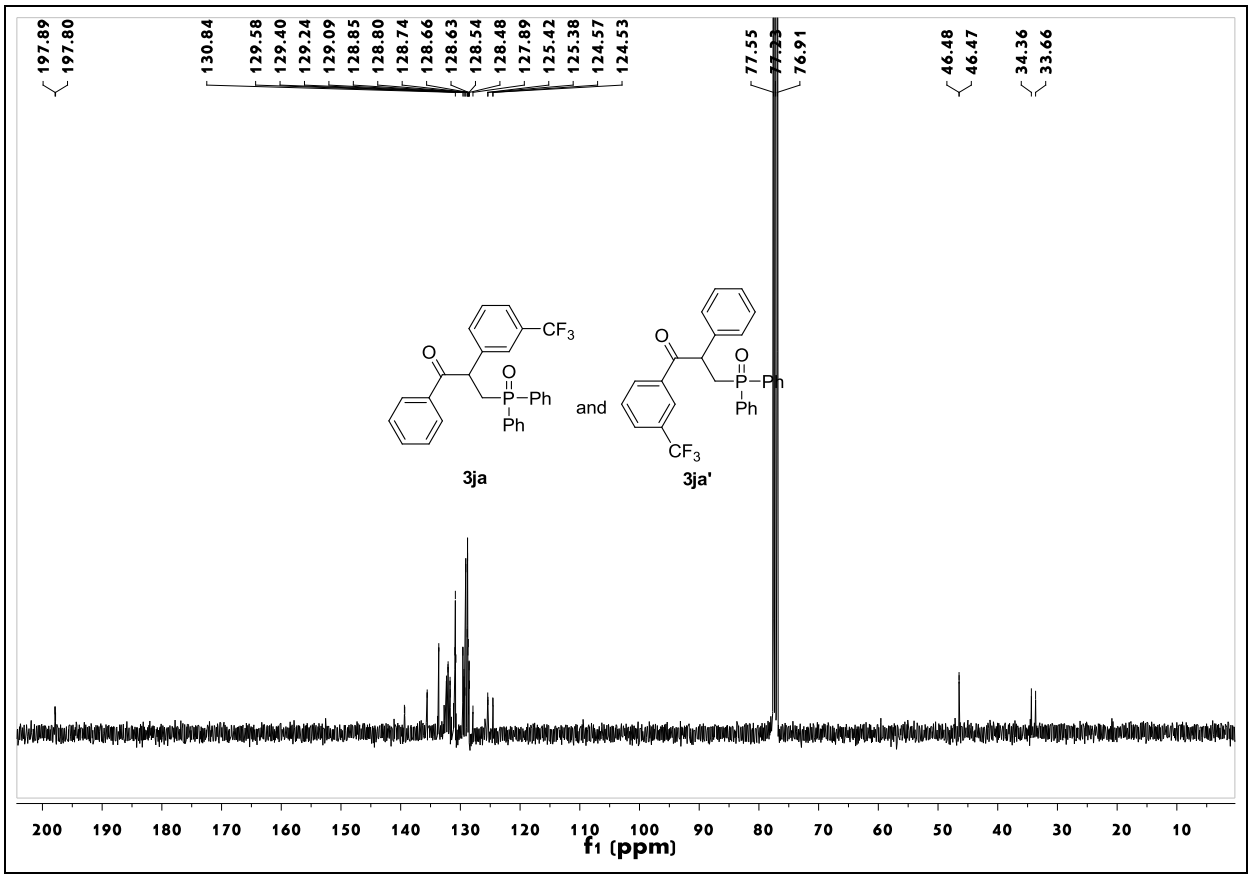

29.78
Br
O
O
P
Ph
Ph
3ha
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160
f1 (ppm)

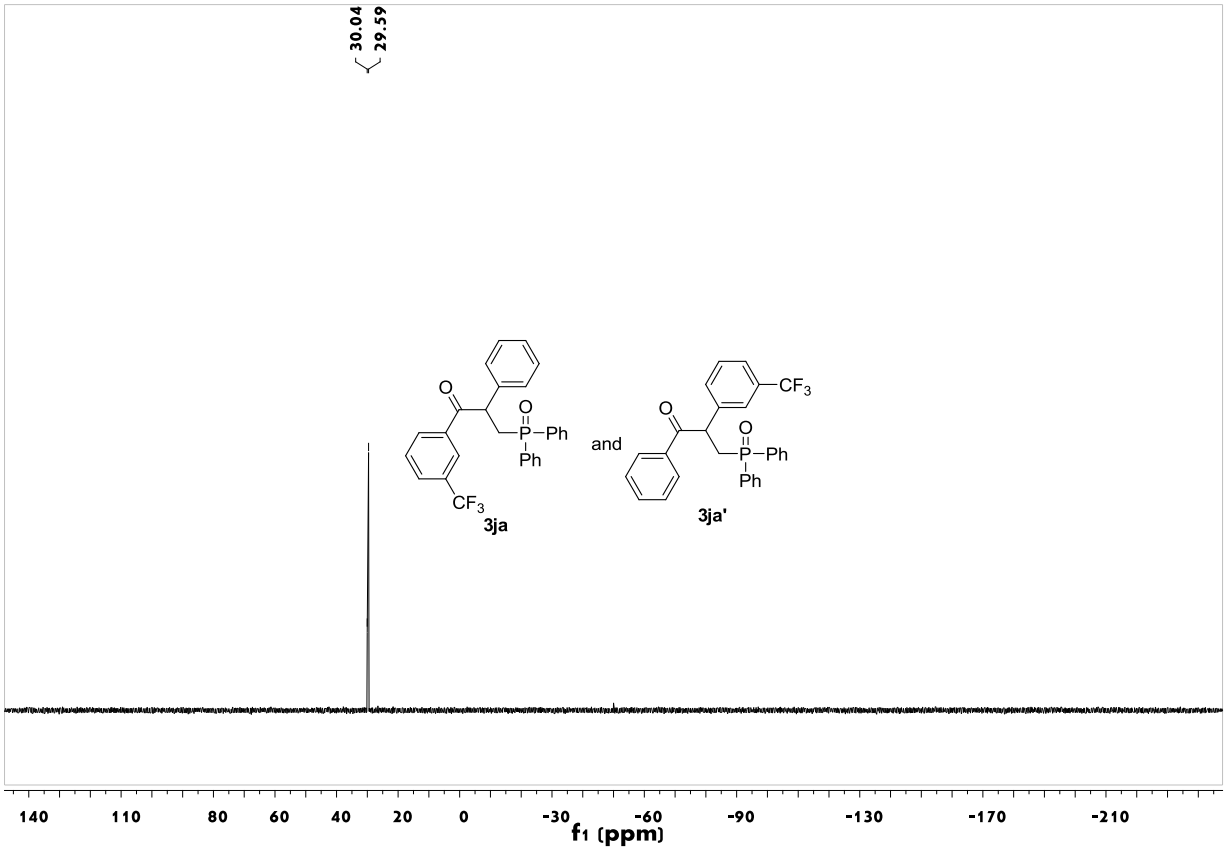

7.64 7.58 7.42 7.39 7.32 7.21 7.10 7.02 6.94 6.77
5.54 5.52 5.51 5.50 5.48
5.19 5.17 5.16 5.15 5.13
3.56 3.54 3.52 3.49 3.47 3.45 3.43 3.40
2.81 2.79 2.77 2.76 2.74 2.73 2.72 2.70 2.69 2.68 2.66
0.00
0.72 2.24 3.09 6.08 3.06 2.06 2.04 1.57 0.41 0.49 1.00 1.02 0.52 0.51 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
and
3ia
3ia'

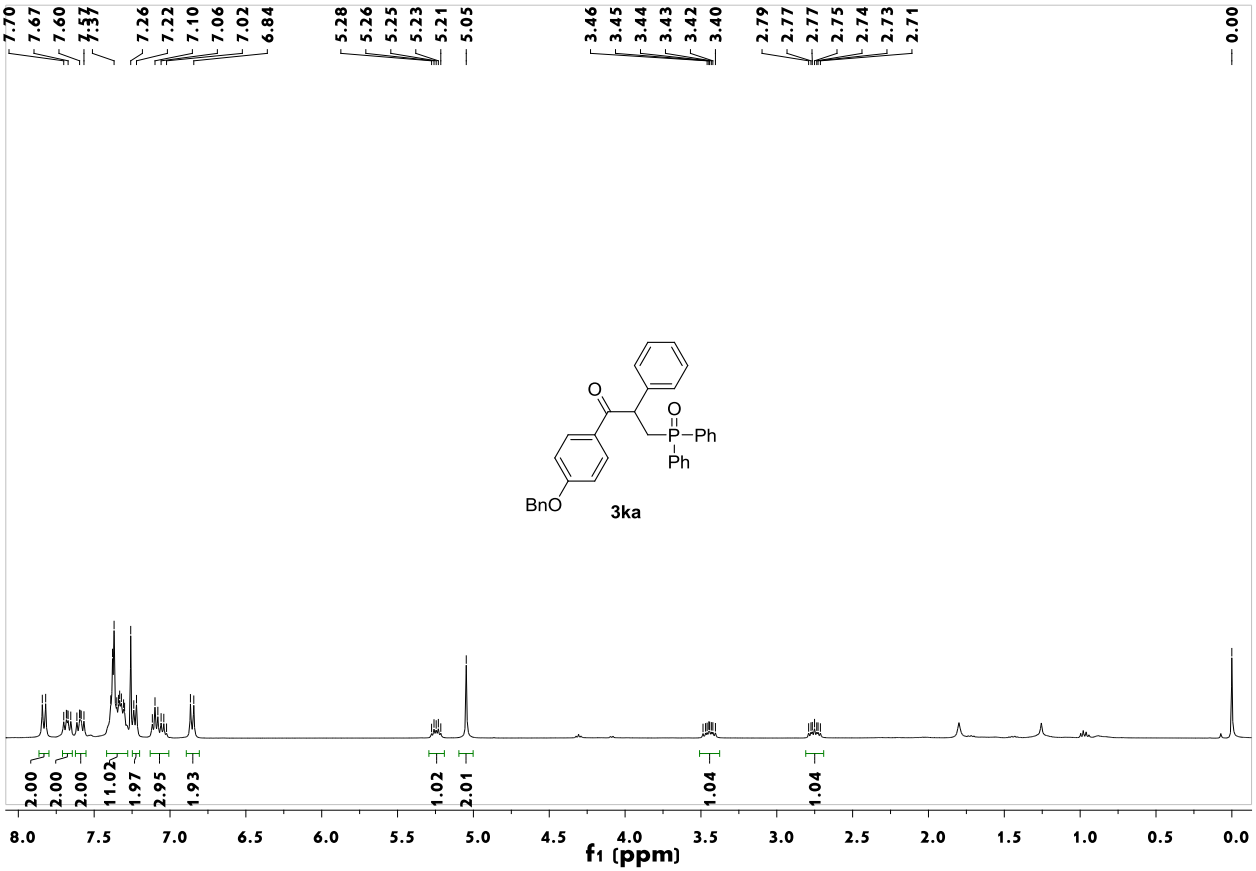

196.84 196.80 196.77 196.73
162.45 159.90
134.50
131.36 129.83 128.80 128.46 124.33
116.91 116.67 116.12 115.90
77.55 77.23 76.91
50.61 50.60 50.54 50.53
39.59
34.29 33.59
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)
and
3ia
3ia'

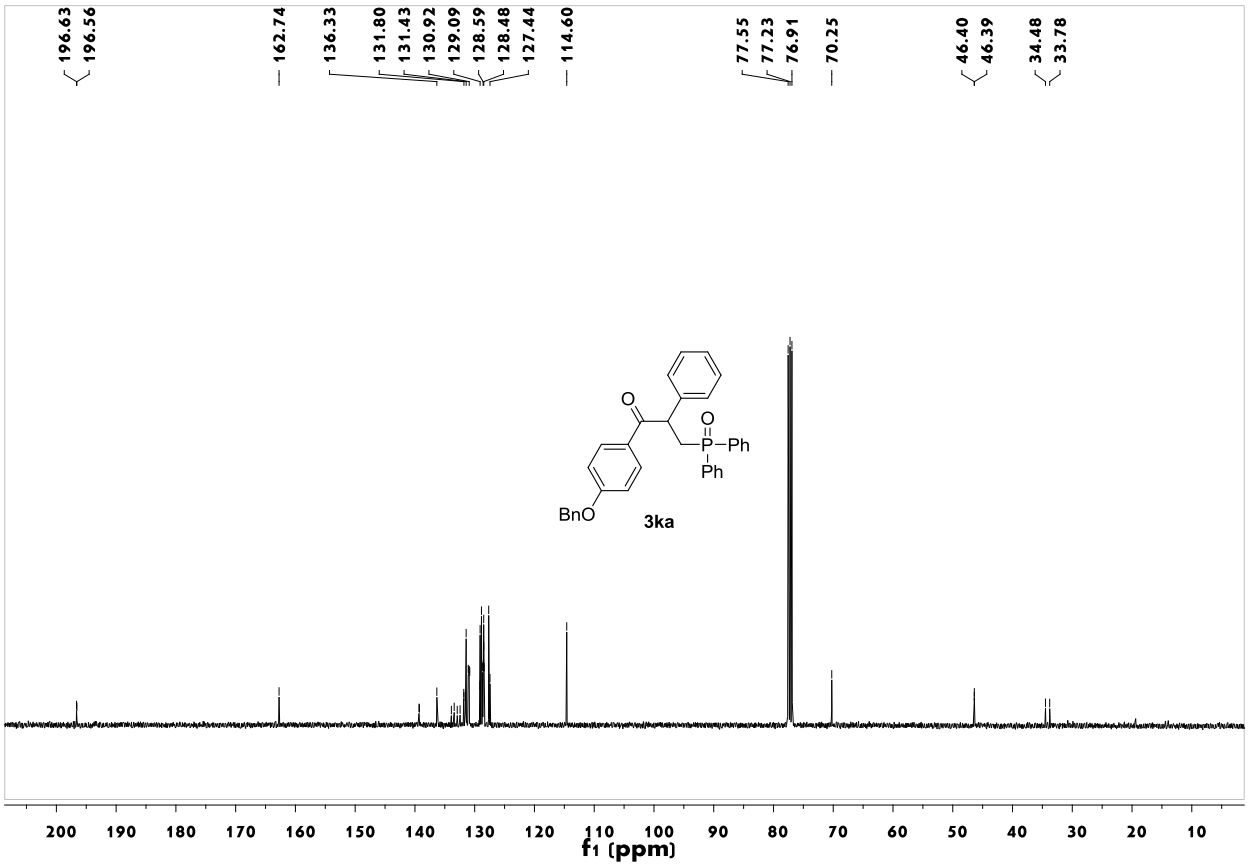

29.79
29.49
F
O
O
P–Ph
Ph
3ia
and
3ia'
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210
f1 (ppm)

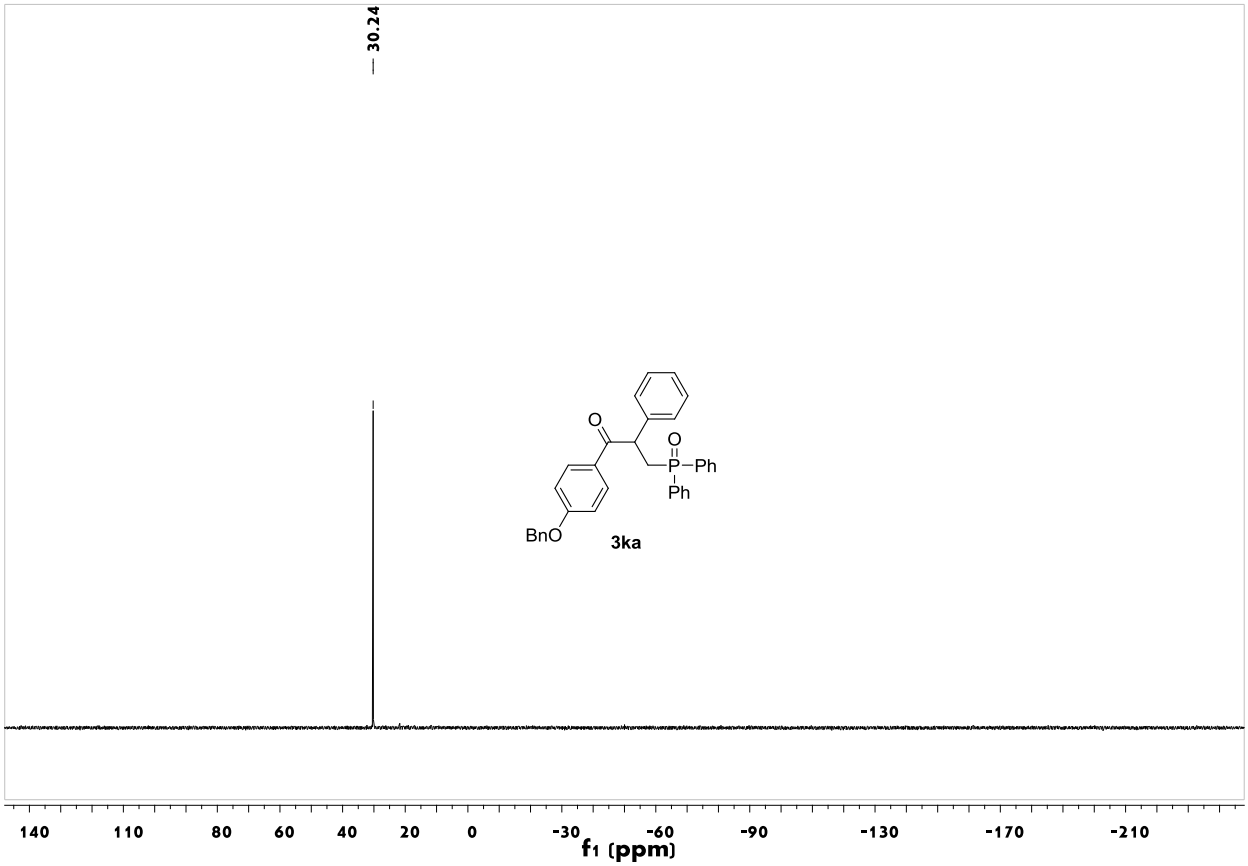

7.57
7.52
7.45
7.40
7.35
7.29
7.24
7.17
7.13
7.07
5.45
5.43
5.42
5.41
5.41
5.39
5.28
5.27
5.25
5.23
3.39
3.37
3.36
3.35
3.34
3.33
3.32
2.85
2.83
2.81
2.80
2.79
2.77
2.76
2.75
2.74
2.72
0.00
CF3
O
P–Ph
Ph
3ja
and
3ja'
0.62
1.82
2.91
0.82
2.21
5.12
6.05
3.23
1.92
0.85
1.00
0.40
0.43
1.05
1.02
0.43
8.0
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)

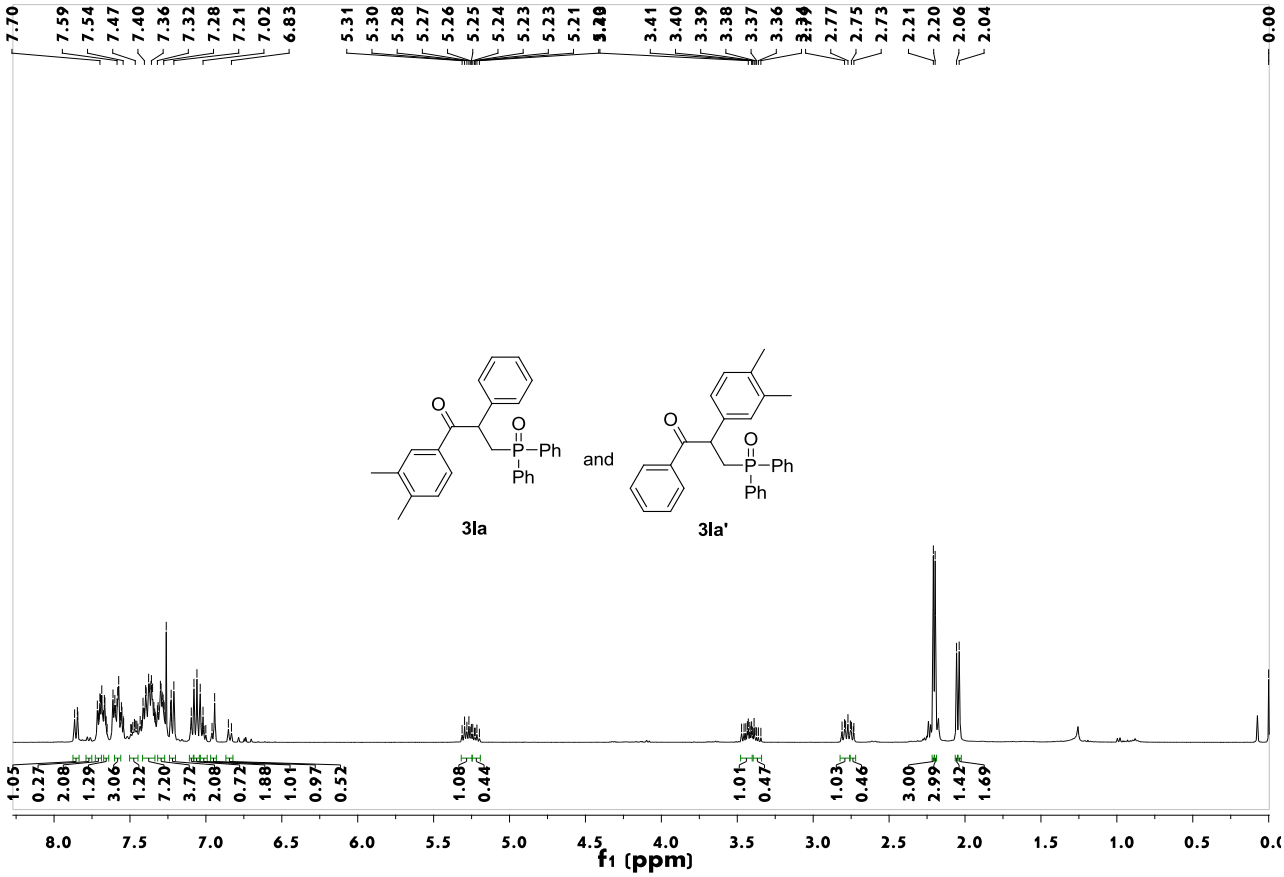

197.89
197.80
130.84
129.58
129.40
129.24
129.09
128.85
128.80
128.74
128.66
128.63
128.54
128.48
127.89
125.42
125.38
124.57
124.53
77.55
76.91
46.48
46.47
34.36
33.66
CF3
O
O
P
Ph
Ph
and
3ja
3ja'
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

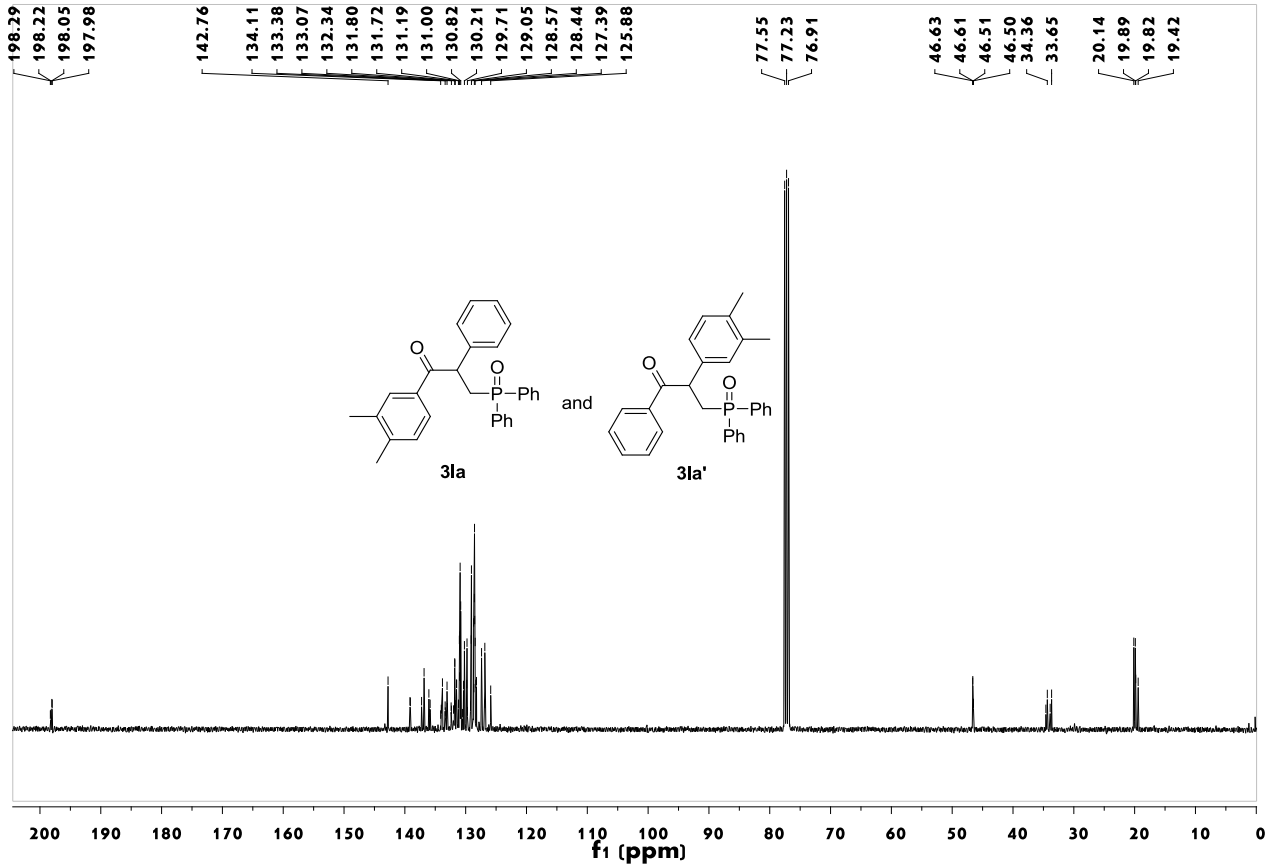

30.04
29.59
O
O
P
Ph
Ph
CF3
and
3ja
3ja'
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210
f1 (ppm)

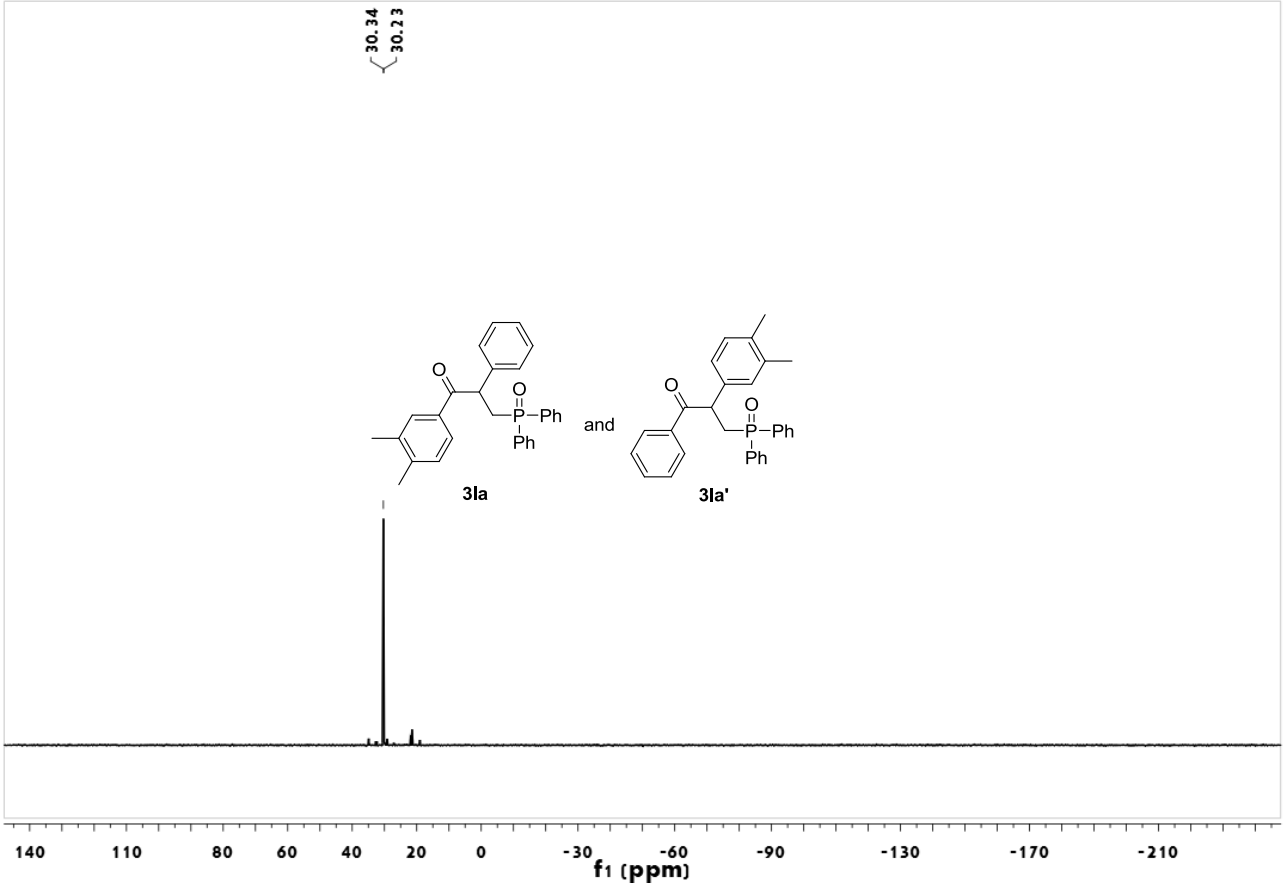

7.70
7.67
7.60
7.57
7.37
7.26
7.22
7.10
7.06
7.02
6.84
5.28
5.26
5.25
5.23
5.21
5.05
3.46
3.45
3.44
3.43
3.42
3.40
2.79
2.77
2.77
2.75
2.74
2.73
2.71
0.00
O
O
P–Ph
Ph
BnO
3ka
2.00
2.00
2.00
11.02
1.97
2.95
1.93
1.02
2.01
1.04
1.04
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

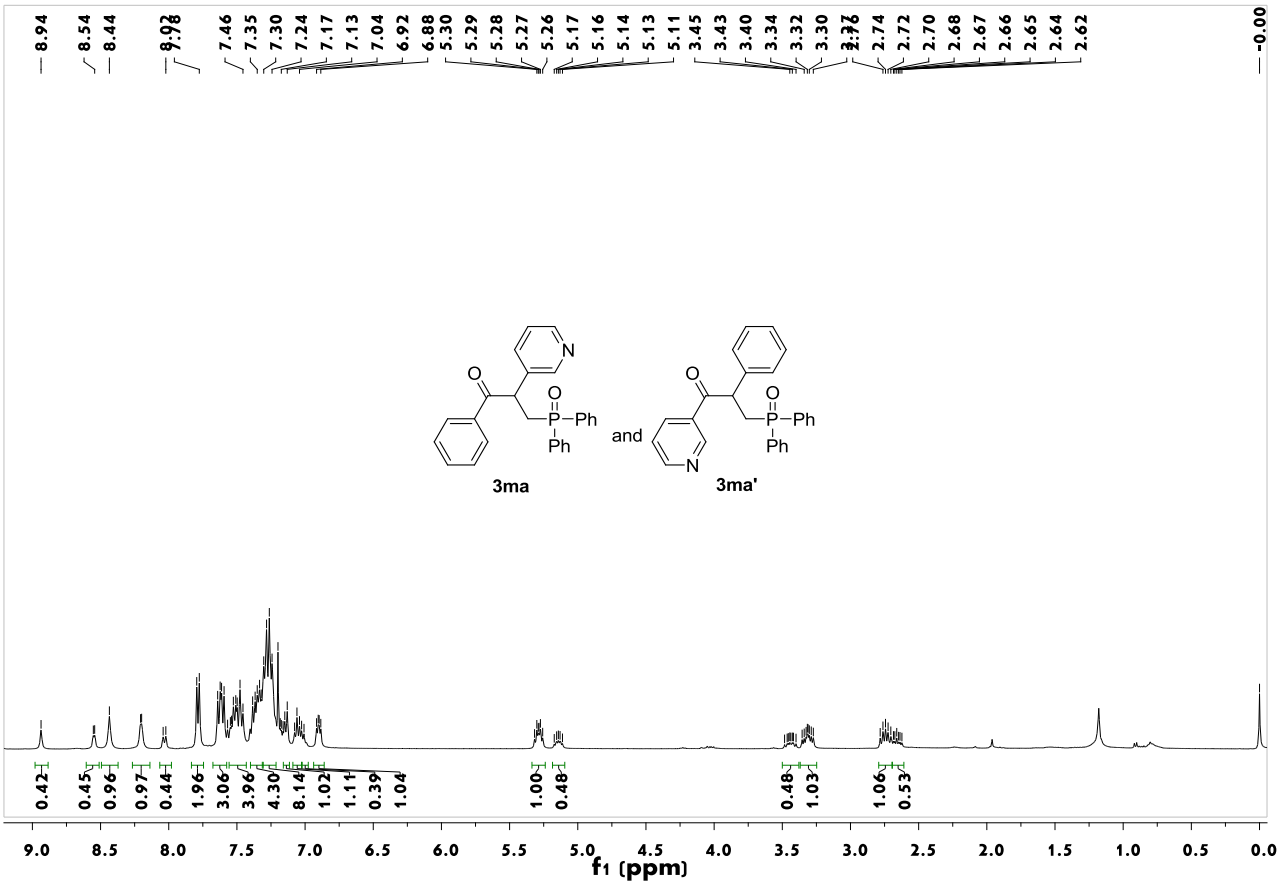

196.63
196.56
162.74
136.33
131.80
131.43
130.92
129.09
128.59
128.48
127.44
114.60
77.55
77.23
76.91
70.25
46.40
46.39
34.48
33.78
O
O
P–Ph
Ph
BnO
3ka
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

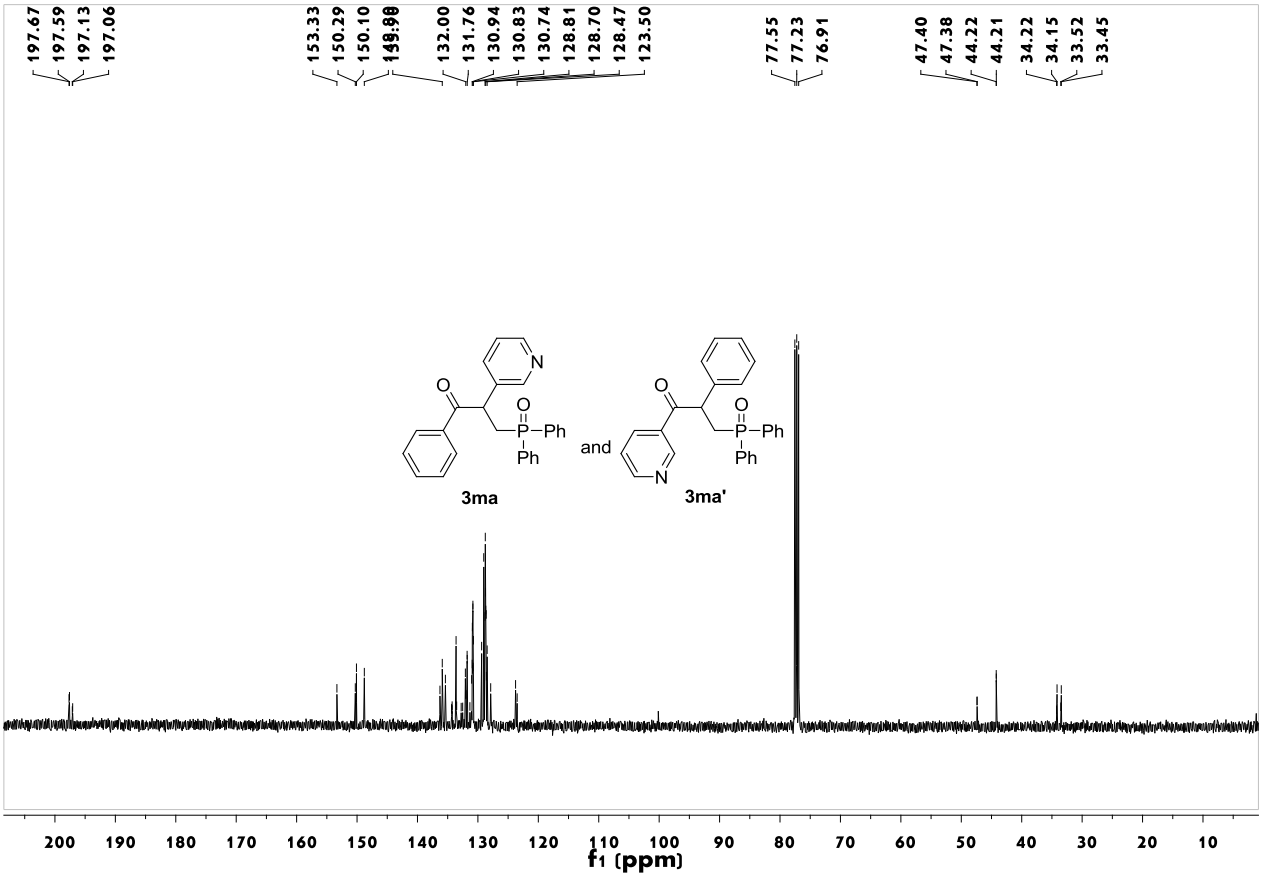

30.24
O
O
P–Ph
Ph
BnO
3ka
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210
f1 (ppm)


7.70
7.59
7.54
7.47
7.40
7.36
7.32
7.28
7.21
7.02
6.83
5.31
5.30
5.28
5.27
5.26
5.25
5.24
5.23
5.21
3.41
3.40
3.39
3.38
3.37
3.36
2.77
2.75
2.73
2.21
2.20
2.06
2.04
0.00
O
O
P–Ph
Ph
3la
and
3la'
1.05
0.27
2.08
1.29
3.06
1.22
7.20
3.72
2.08
0.72
1.88
1.01
0.97
0.52
1.08
0.44
1.01
0.47
1.03
0.46
3.00
2.99
1.42
1.69
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
f1 (ppm)


198.29
198.22
198.05
197.98
142.76
134.11
133.38
133.07
132.34
131.80
131.72
131.19
131.00
130.82
130.21
129.71
129.05
128.57
128.44
127.39
125.88
77.55
77.23
76.91
46.63
46.61
46.51
46.50
34.36
33.65
20.14
19.89
19.82
19.42
and
3la
3la'
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


30.34
30.23
and
3la
3la'
140 110 80 60 40 20 0 -30 -60 -90 -130 -170 -210
f1 (ppm)


8.94
8.54
8.44
7.46
7.35
7.30
7.24
7.17
7.13
7.04
6.92
6.88
5.30
5.29
5.28
5.27
5.26
5.17
5.16
5.14
5.13
5.11
3.45
3.43
3.40
3.34
3.32
3.30
2.74
2.72
2.70
2.68
2.67
2.66
2.65
2.64
2.62
-0.00
3ma
and
3ma'
0.42
0.45
0.96
0.97
0.44
1.96
3.06
3.96
4.30
8.14
1.02
1.11
0.39
1.04
1.00
0.48
0.48
1.03
1.06
0.53
f1 (ppm)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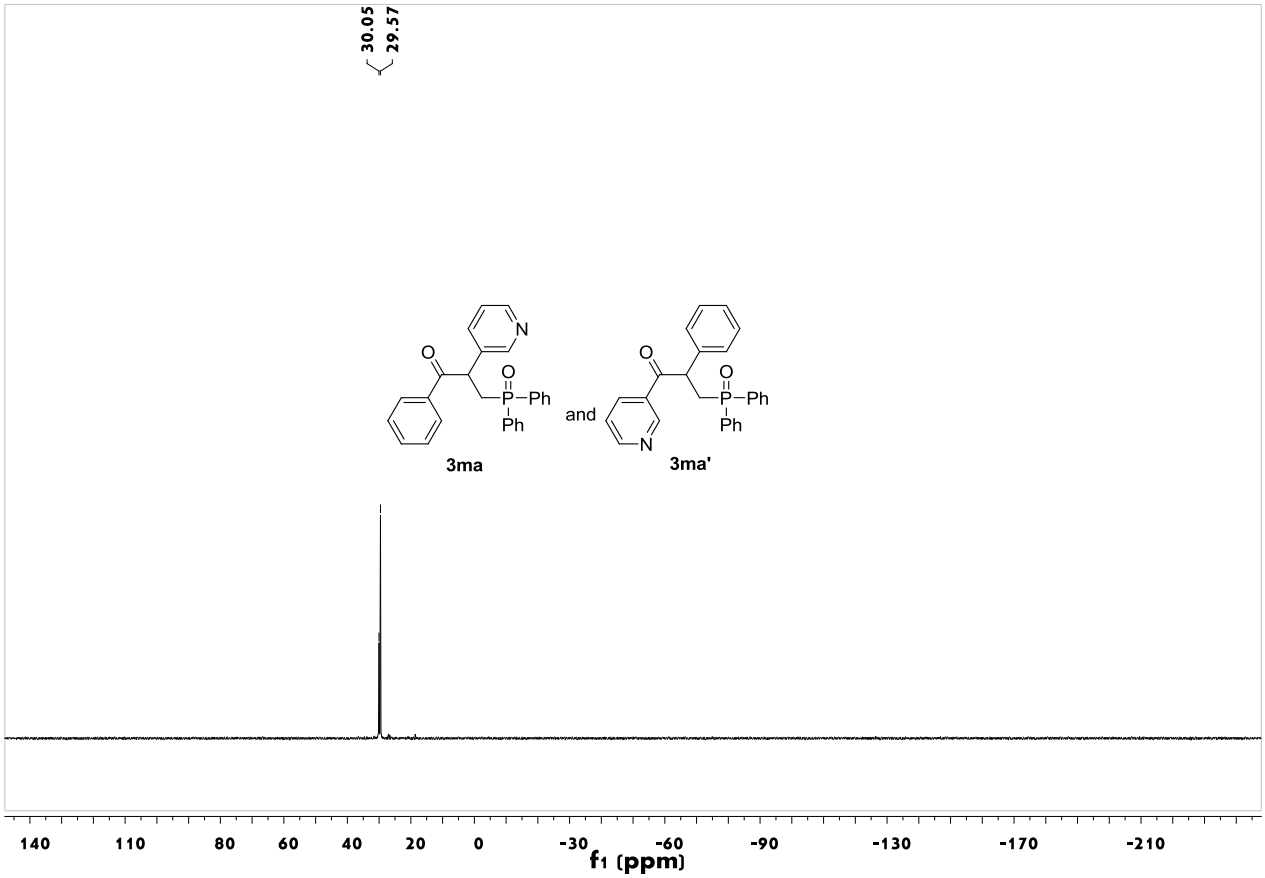
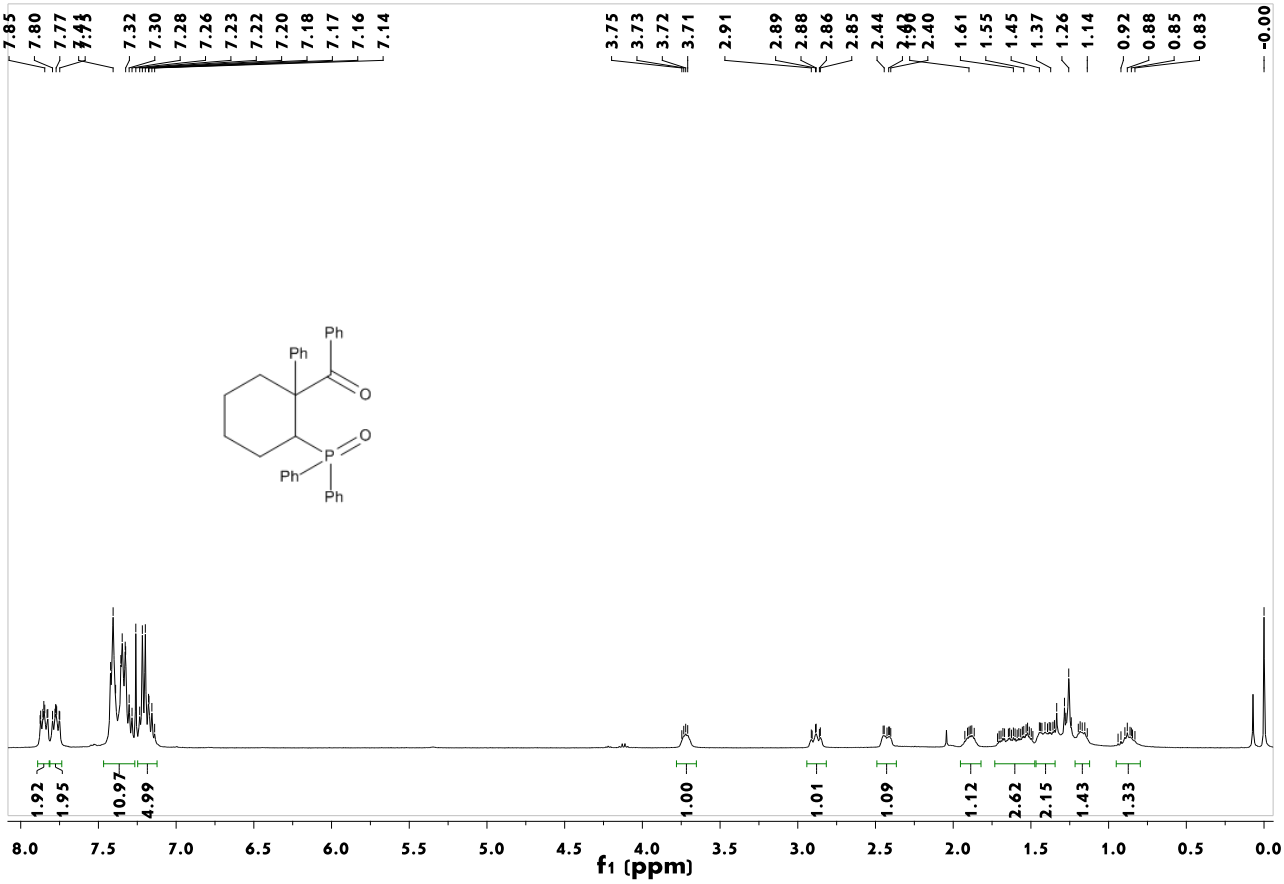
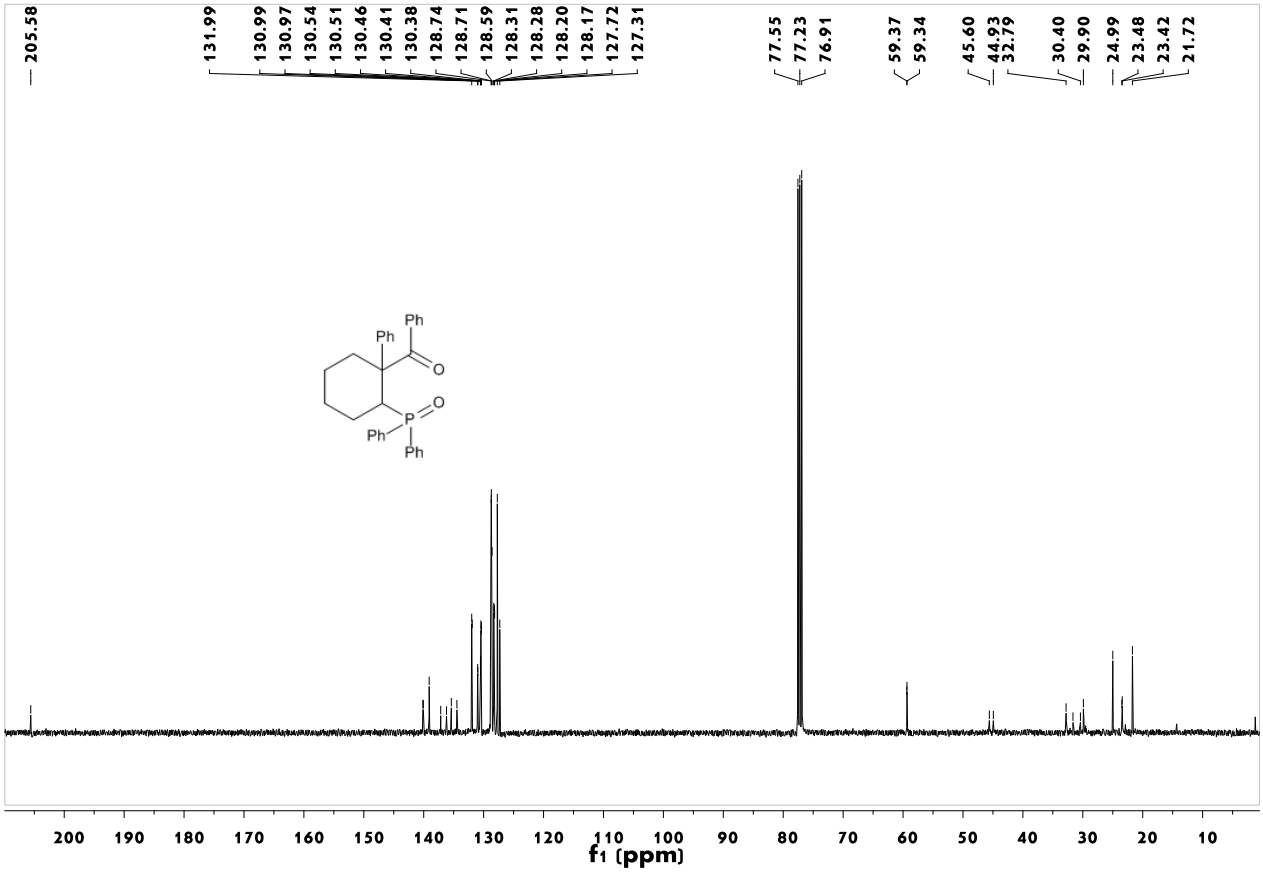
197.59
197.13
197.06
153.33
150.29
150.10
132.00
131.76
130.94
130.83
130.74
128.81
128.70
128.47
123.50
77.55
77.23
76.91
47.40
47.38
44.22
44.21
34.22
34.15
33.52
33.45
3ma
and
3ma'
f1 (ppm)


30.05
29.57
3ma
and
3ma'
f1 (ppm)


7.85
7.80
7.77
7.32
7.30
7.28
7.26
7.23
7.22
7.20
7.18
7.17
7.16
7.14
3.75
3.73
3.72
3.71
2.91
2.89
2.88
2.86
2.85
2.44
2.40
1.61
1.55
1.45
1.37
1.26
1.14
0.92
0.88
0.85
0.83
-0.00
1.92
1.95
10.97
4.99
1.00
1.01
1.09
1.12
2.62
2.15
1.43
1.33
f1 (ppm)


205.58
131.99
130.99
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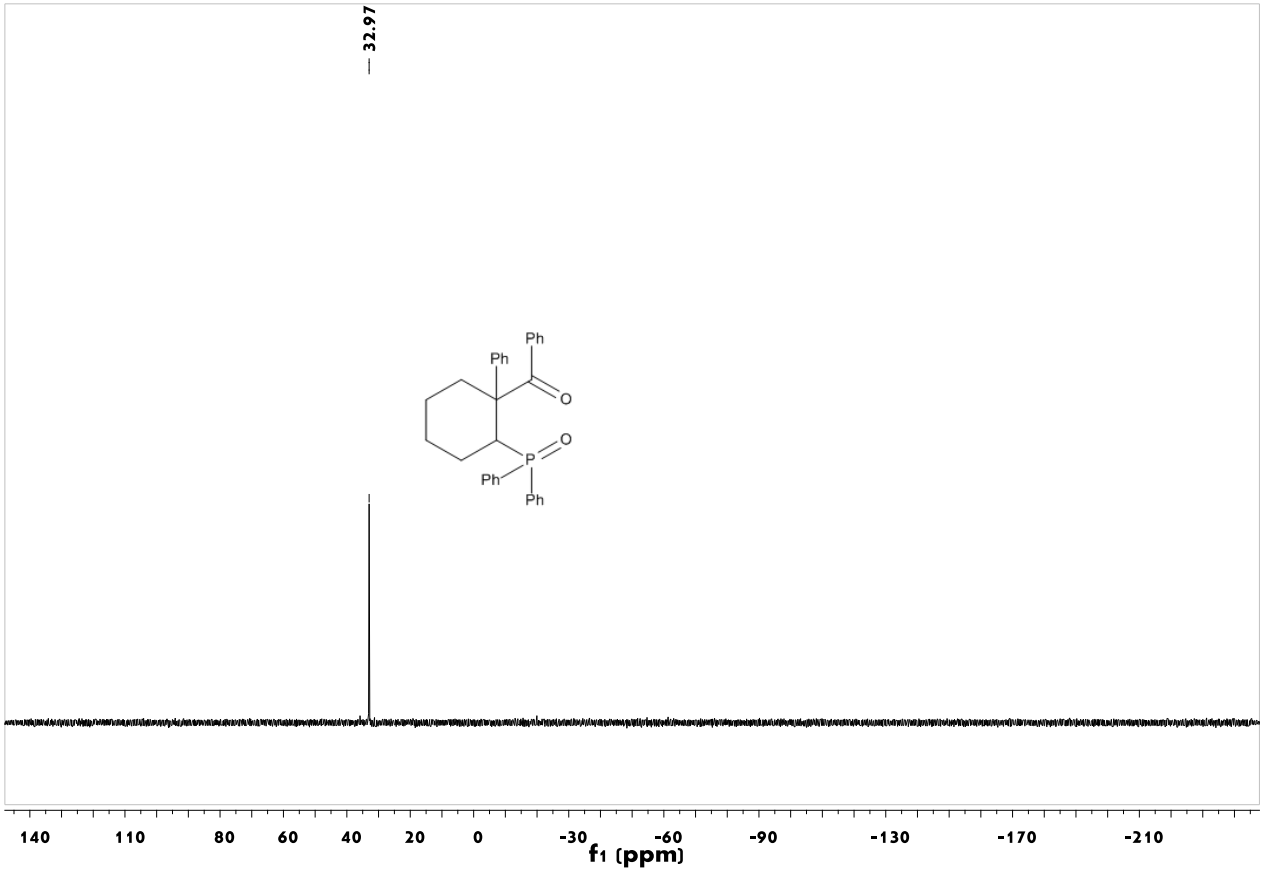
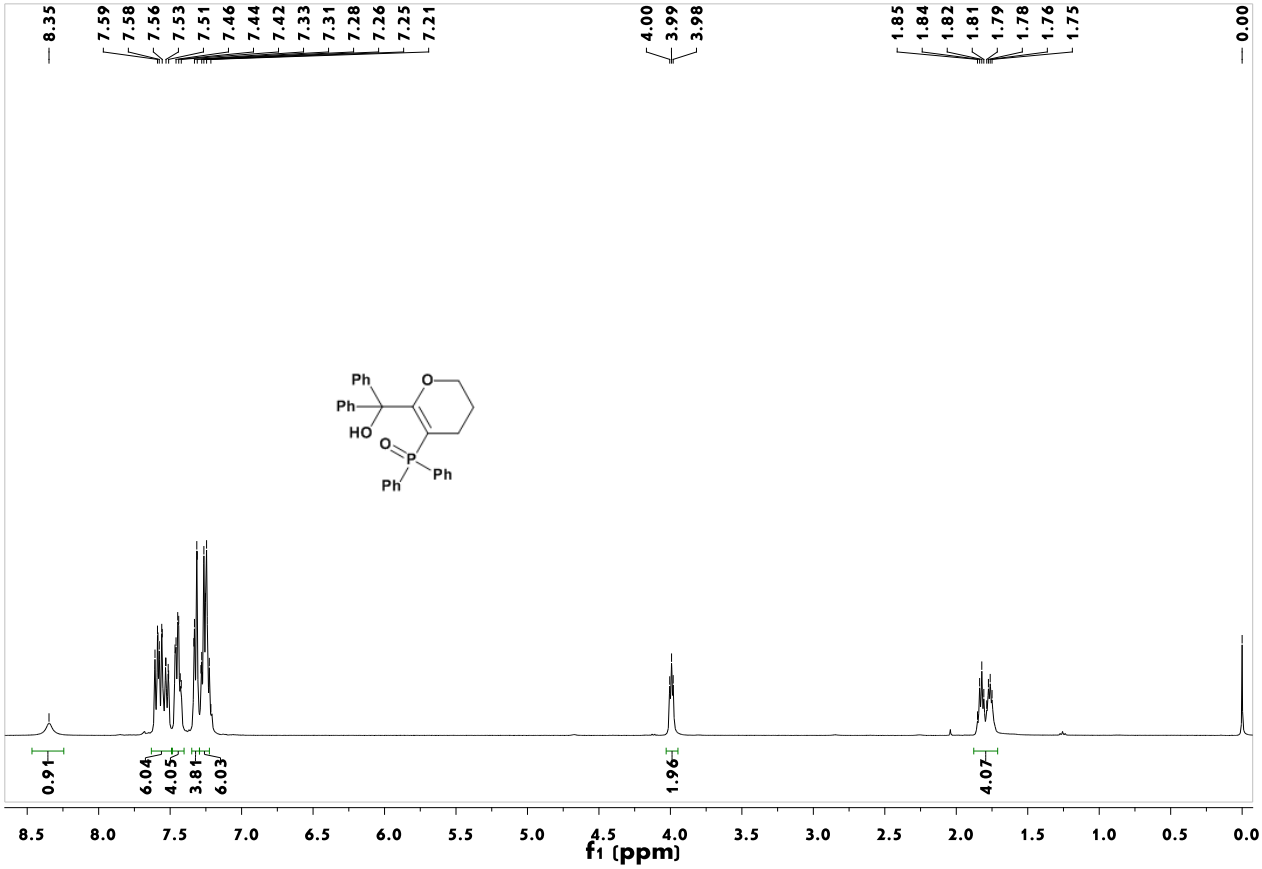
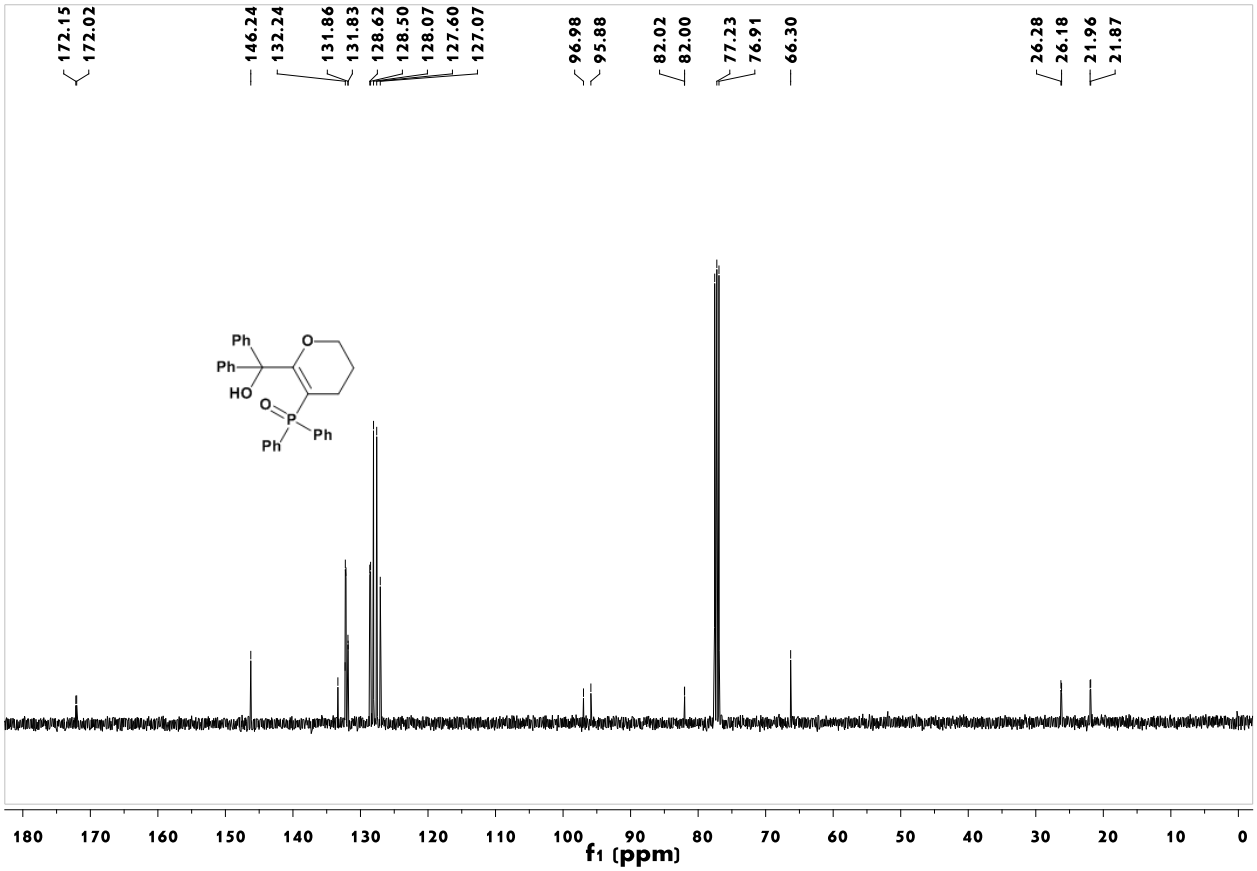
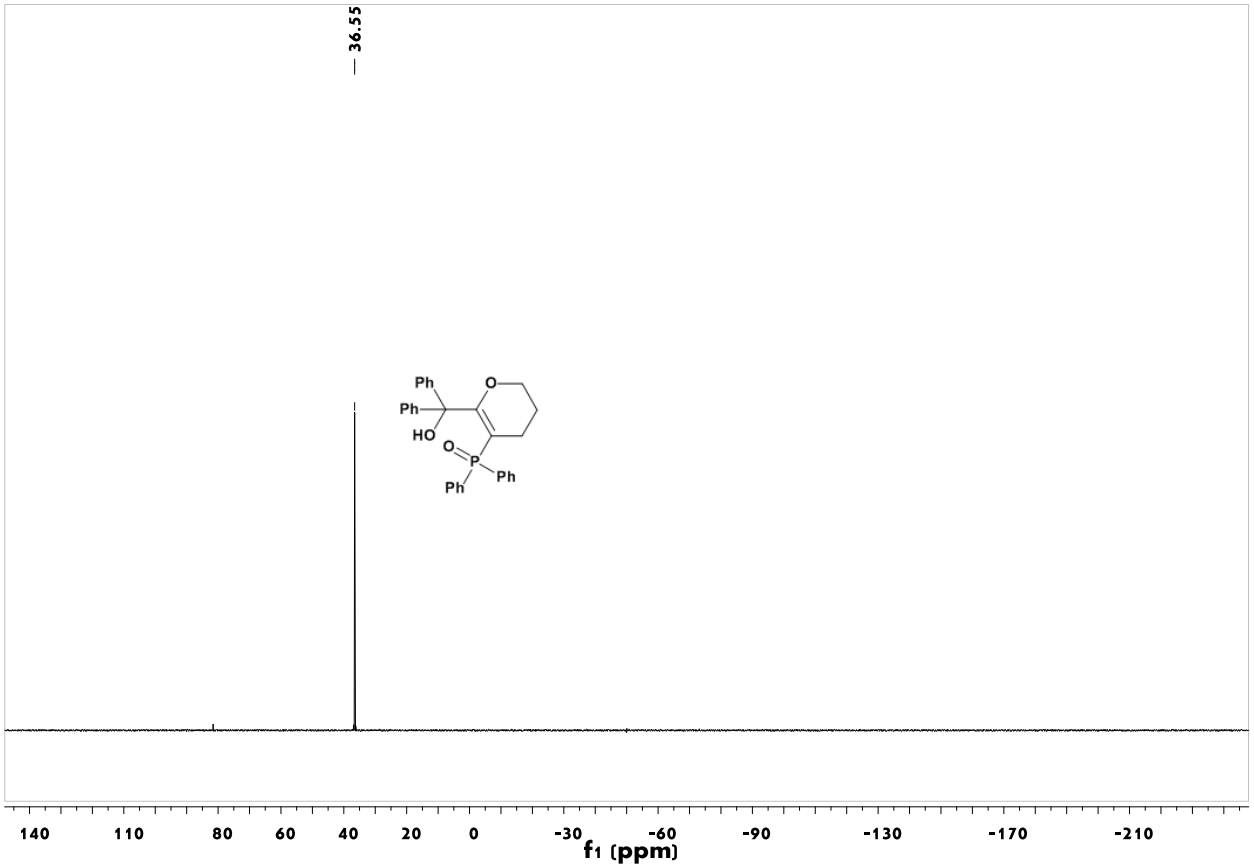
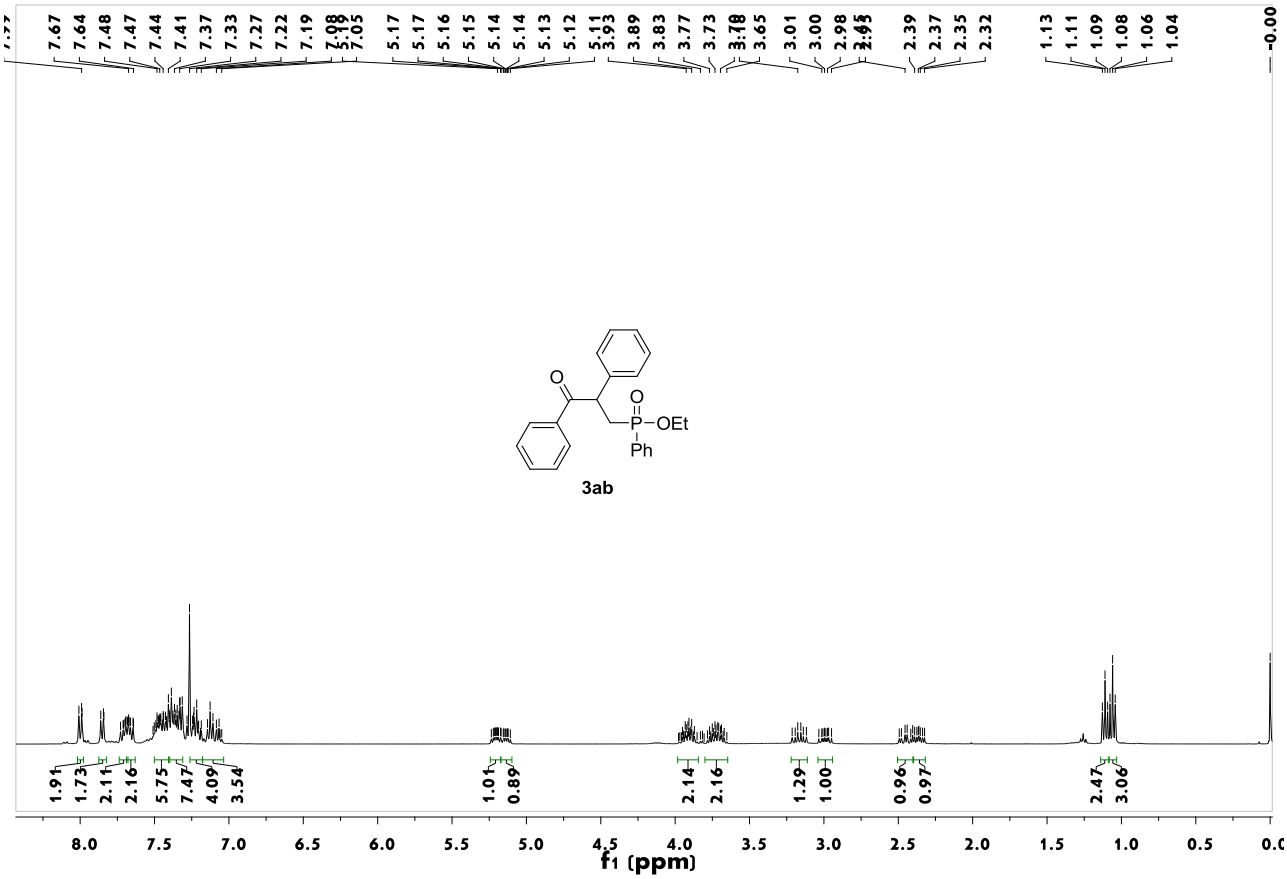
130.51
130.46
130.41
130.38
128.74
128.71
128.59
128.31
128.28
128.20
128.17
127.72
127.31
77.55
77.23
76.91
59.37
59.34
45.60
44.93
32.79
30.40
29.90
24.99
23.48
23.42
21.72
Ph
Ph
O
P
O
Ph
Ph
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

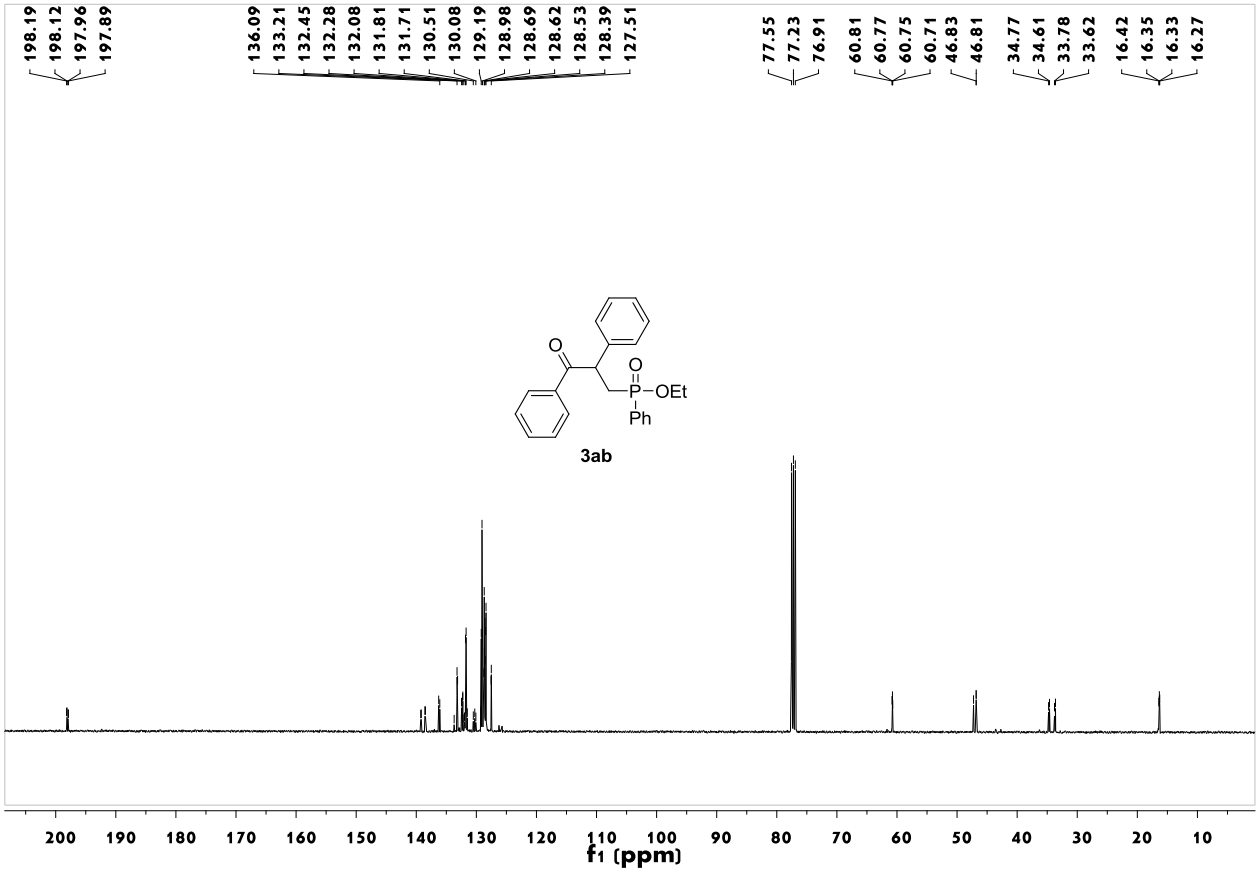

32.97
Ph
Ph
O
P
O
Ph
Ph
140 110 80 60 40 20 0 -30 -60 -90 -130 -170 -210
f1 (ppm)

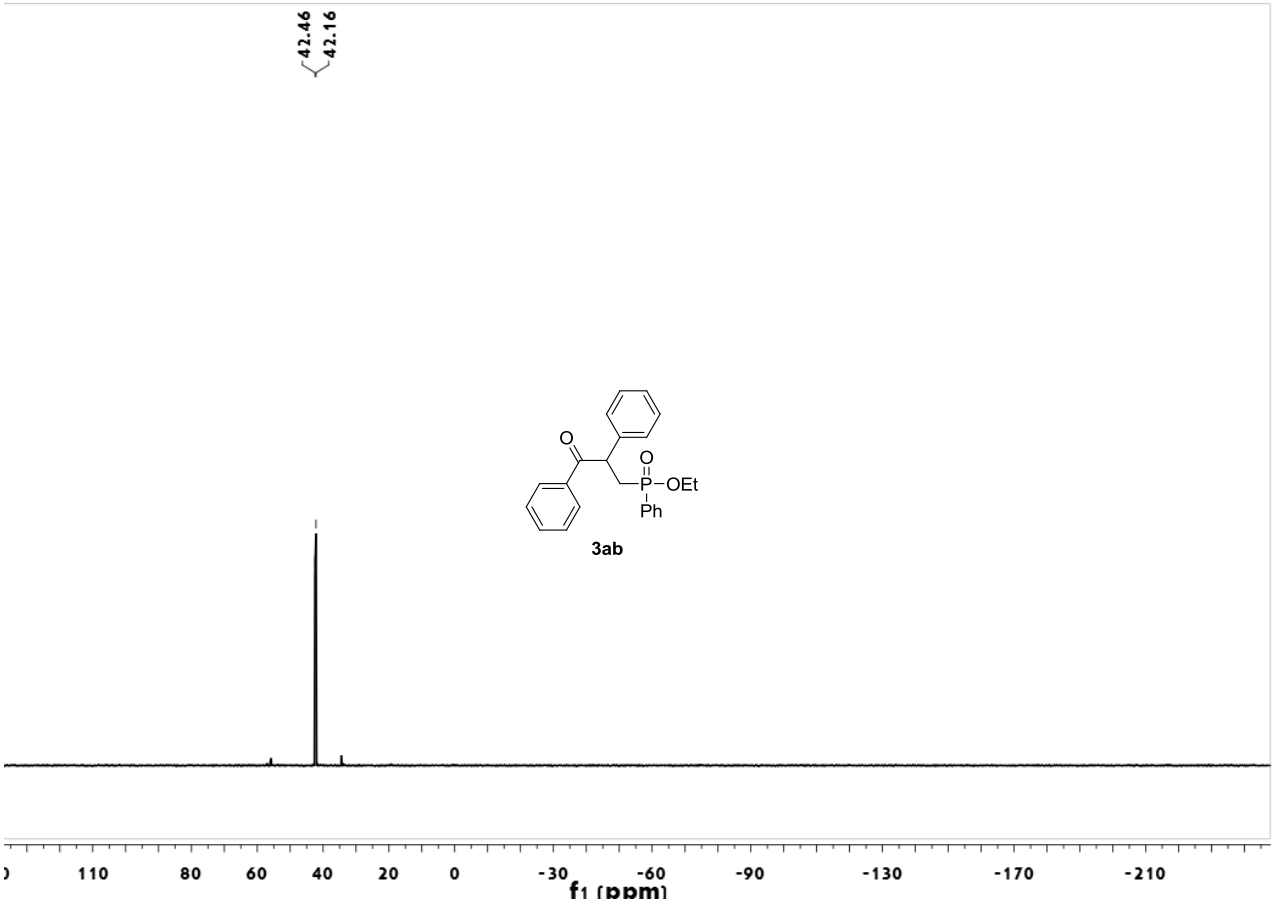

8.35
7.59
7.58
7.56
7.53
7.51
7.46
7.44
7.42
7.33
7.31
7.28
7.26
7.25
7.21
4.00
3.99
3.98
1.85
1.84
1.82
1.81
1.79
1.78
1.76
1.75
0.00
Ph
Ph
HO
O
P
Ph
Ph
O
0.91
6.04
4.05
3.81
6.03
1.96
4.07
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

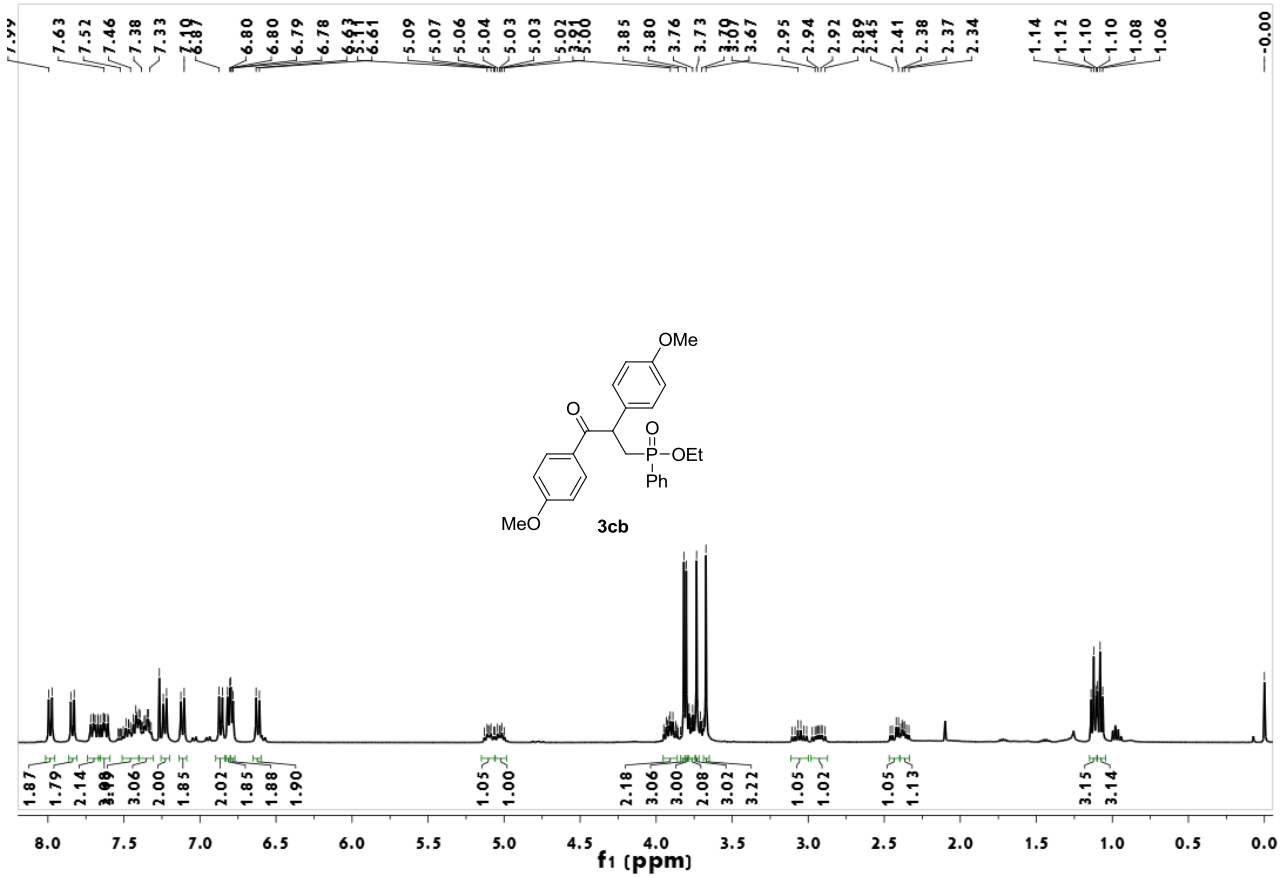

172.15
172.02
146.24
132.24
131.86
131.83
128.62
128.50
128.07
127.60
127.07
96.98
95.88
82.02
82.00
77.23
76.91
66.30
26.28
26.18
21.96
21.87
Ph
Ph
HO
O
P
Ph
Ph
O
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

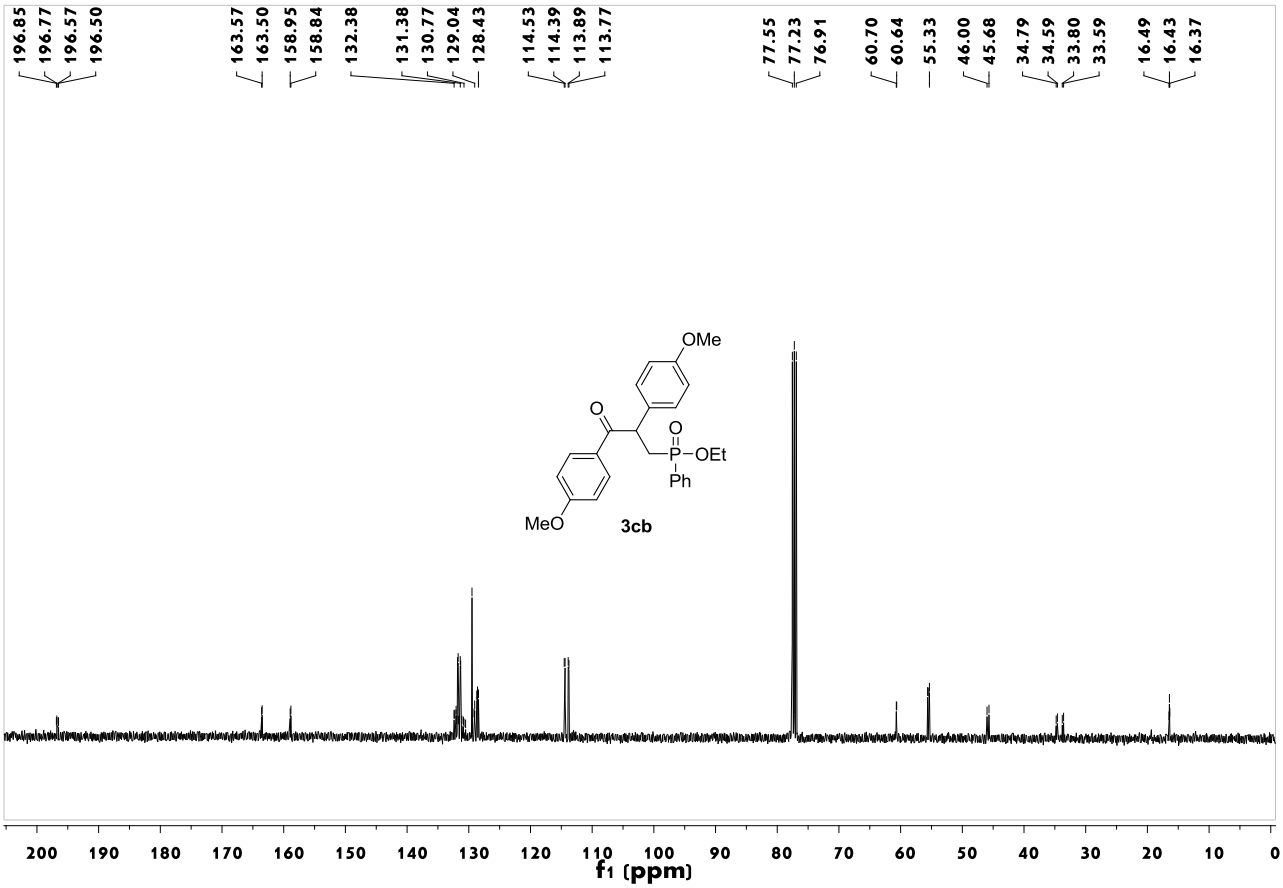

36.55
Ph
Ph
HO
O
O
P
Ph
Ph
Ph
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210
f1 (ppm)

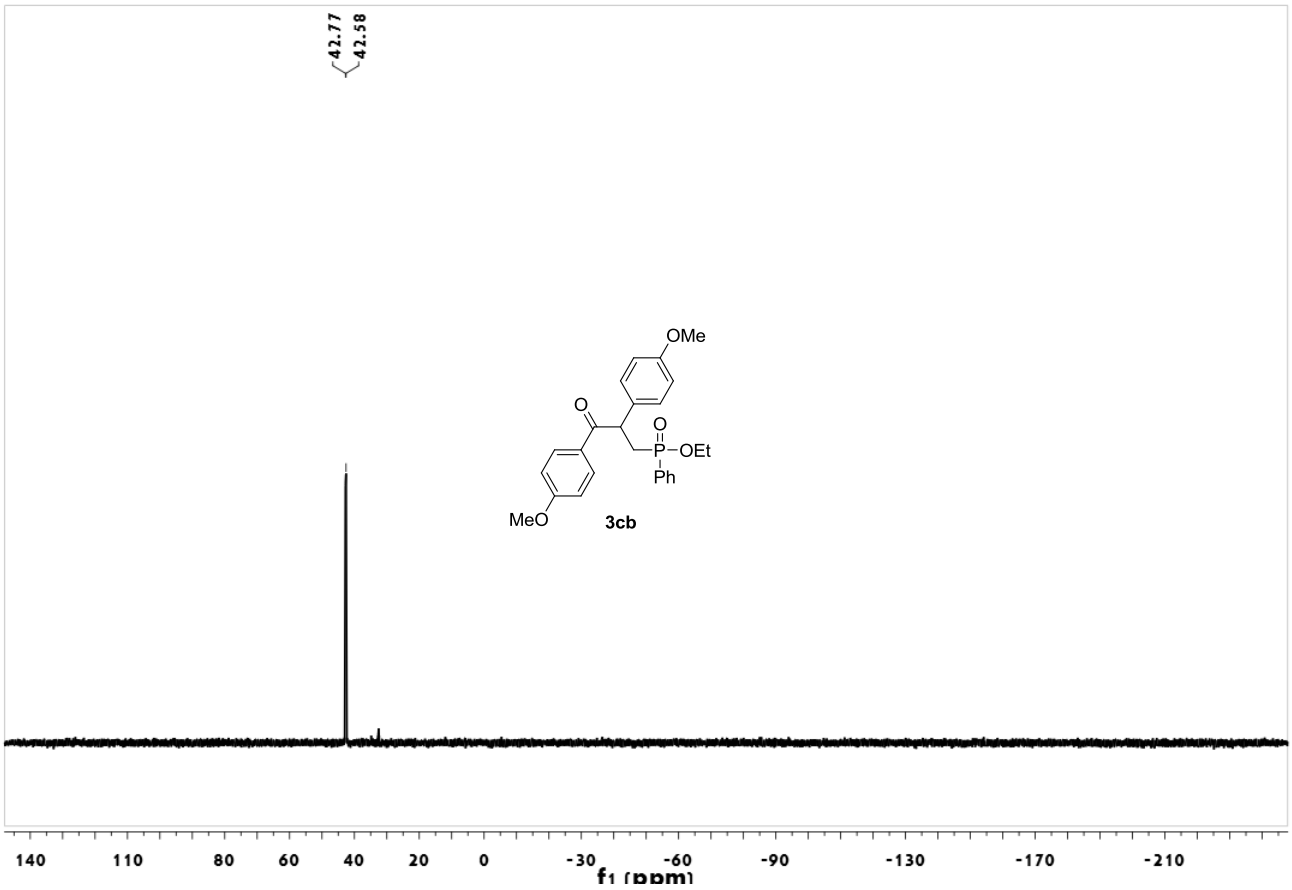

7.67
7.64
7.48
7.47
7.44
7.41
7.37
7.33
7.27
7.22
7.19
5.17
5.17
5.16
5.15
5.14
5.14
5.13
5.12
3.89
3.83
3.77
3.73
3.65
3.01
3.00
2.39
2.37
2.35
2.32
1.13
1.11
1.09
1.08
1.06
1.04
0.00
O
O
P
OEt
Ph
3ab
1.91
1.73
2.11
2.16
5.75
7.47
4.09
3.54
1.01
0.89
2.14
2.16
1.29
1.00
0.96
0.97
2.47
3.06
8.0
7.5
7.0
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)

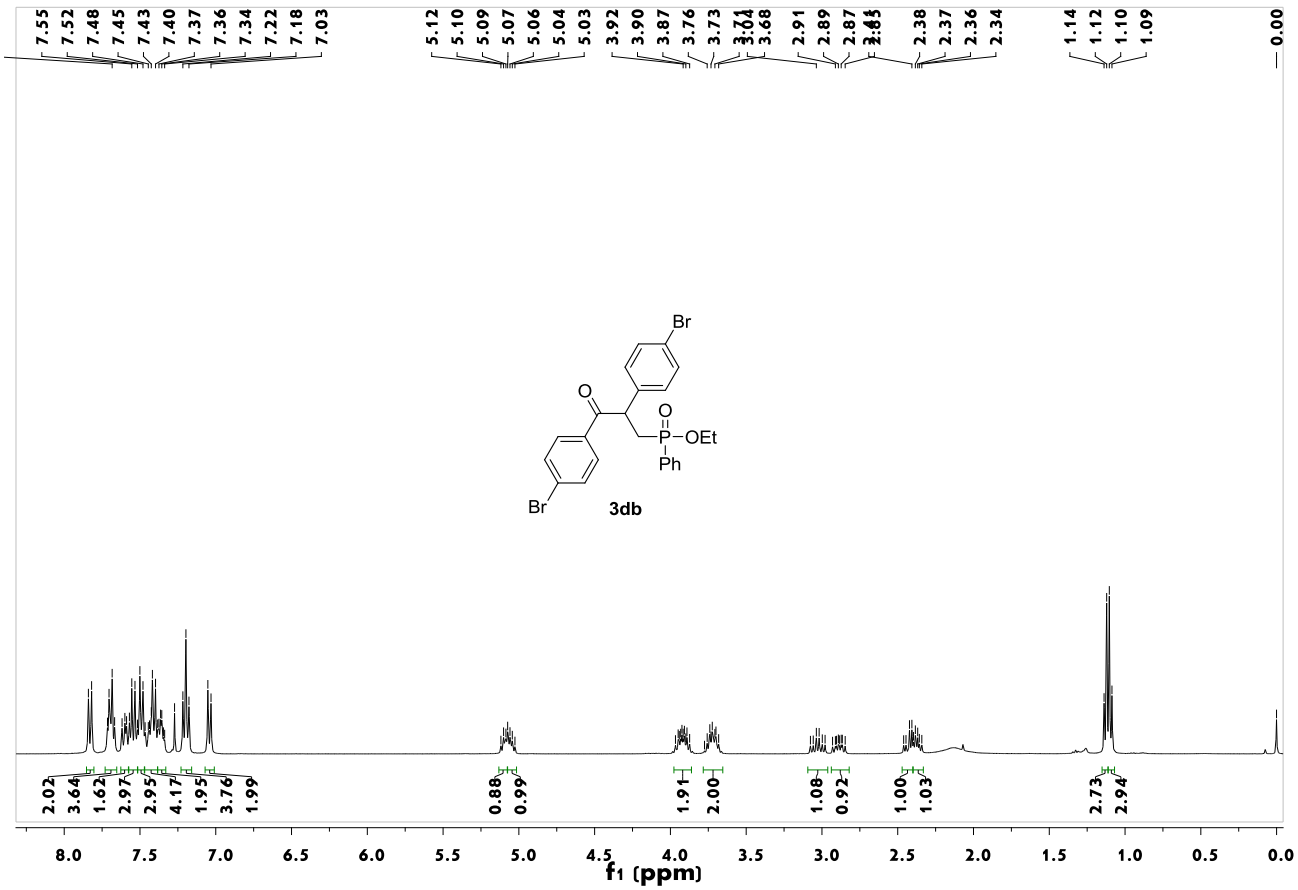

198.19
198.12
197.96
197.89
136.09
133.21
132.45
132.28
132.08
131.81
131.71
130.51
130.08
129.19
128.98
128.69
128.62
128.53
128.39
127.51
77.55
77.23
76.91
60.81
60.77
60.75
60.71
46.83
46.81
34.77
34.61
33.78
33.62
16.42
16.35
16.33
16.27
O
O
P–OEt
Ph
3ab
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

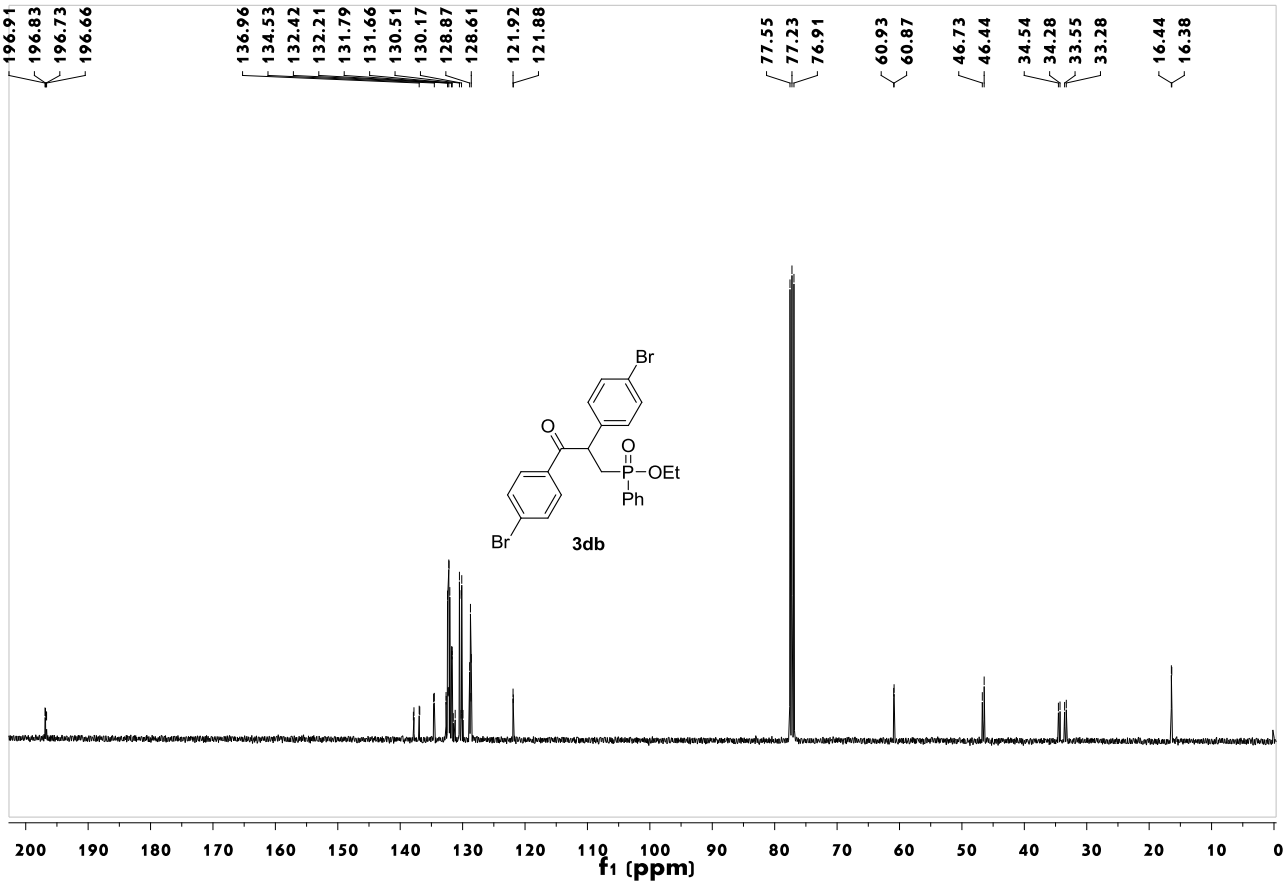

42.46
42.16
O
O
P–OEt
Ph
3ab
110 80 60 40 20 0 -30 -60 -90 -130 -170 -210
f1 (ppm)

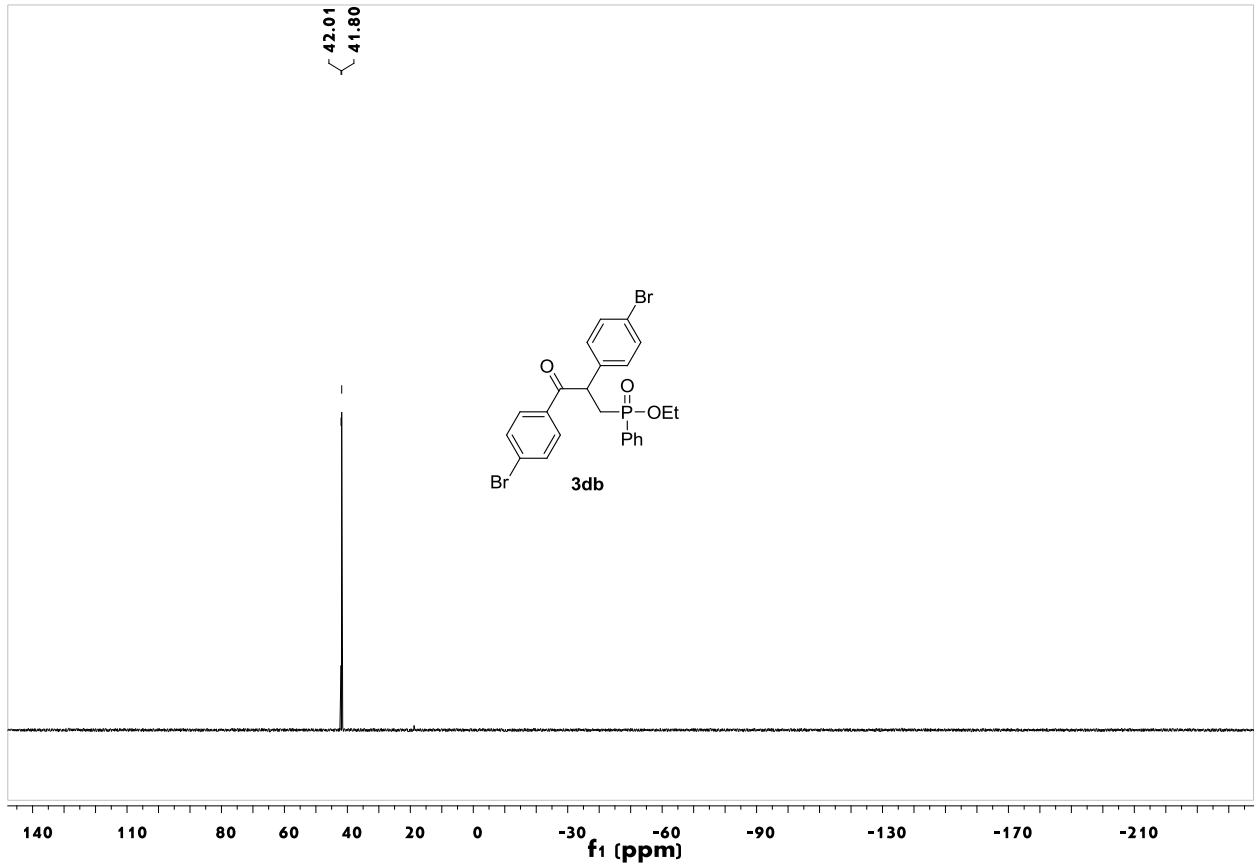

7.63
7.52
7.46
7.38
7.33
6.87
6.80
6.80
6.79
6.78
6.63
6.61
5.09
5.07
5.06
5.04
5.03
5.03
5.02
3.85
3.80
3.76
3.73
3.67
2.95
2.94
2.92
2.45
2.41
2.38
2.37
2.34
1.14
1.12
1.10
1.10
1.08
1.06
0.00
OMe
O
O
P
OEt
Ph
MeO
3cb
1.87
1.79
2.14
3.06
2.00
1.85
2.02
1.85
1.88
1.90
1.05
1.00
2.18
3.06
3.00
2.08
3.02
3.22
1.05
1.02
1.05
1.13
3.15
3.14
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)

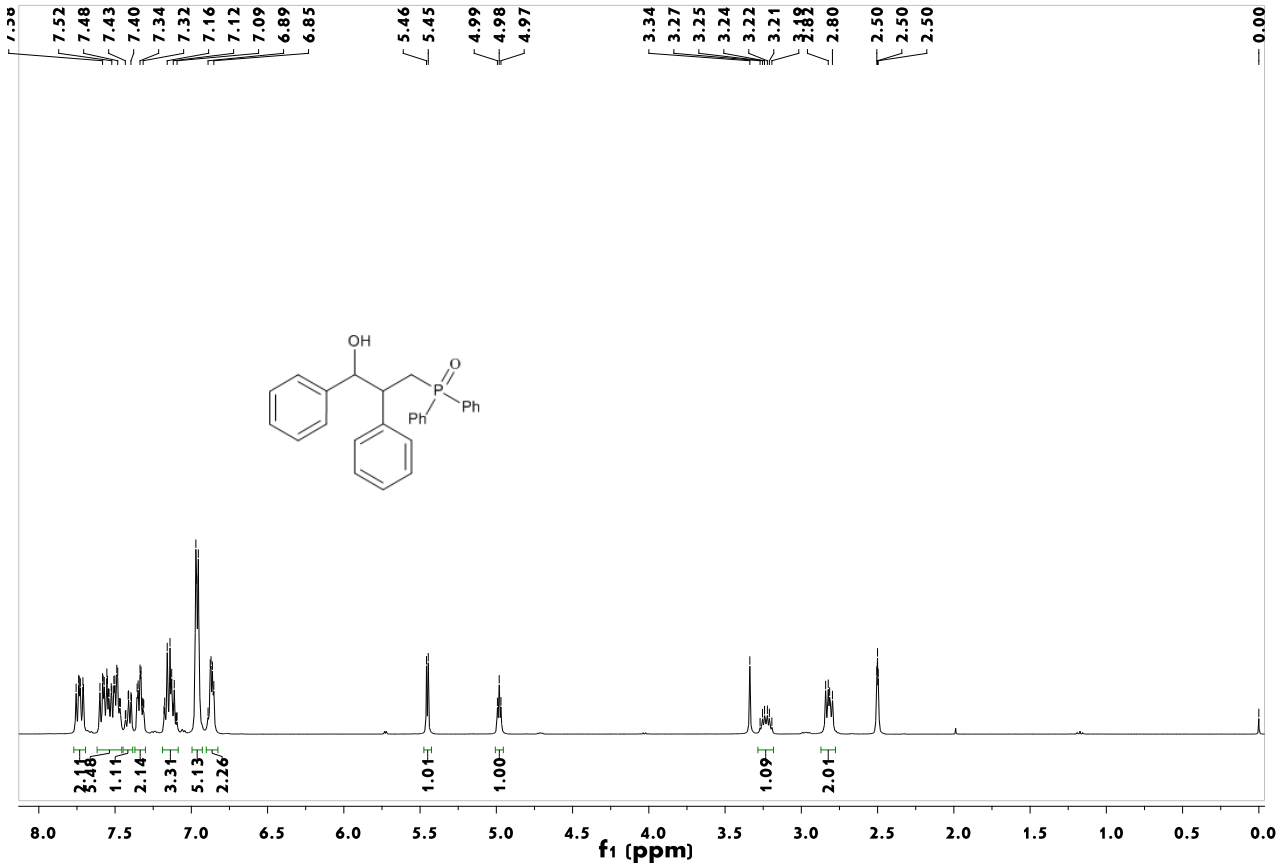

196.85
196.77
196.57
196.50
163.57
163.50
158.95
158.84
132.38
131.38
130.77
129.04
128.43
114.53
114.39
113.89
113.77
77.55
77.23
76.91
60.70
60.64
55.33
46.00
45.68
34.79
34.59
33.80
33.59
16.49
16.43
16.37
OMe
O
O
P
OEt
Ph
MeO
3cb
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

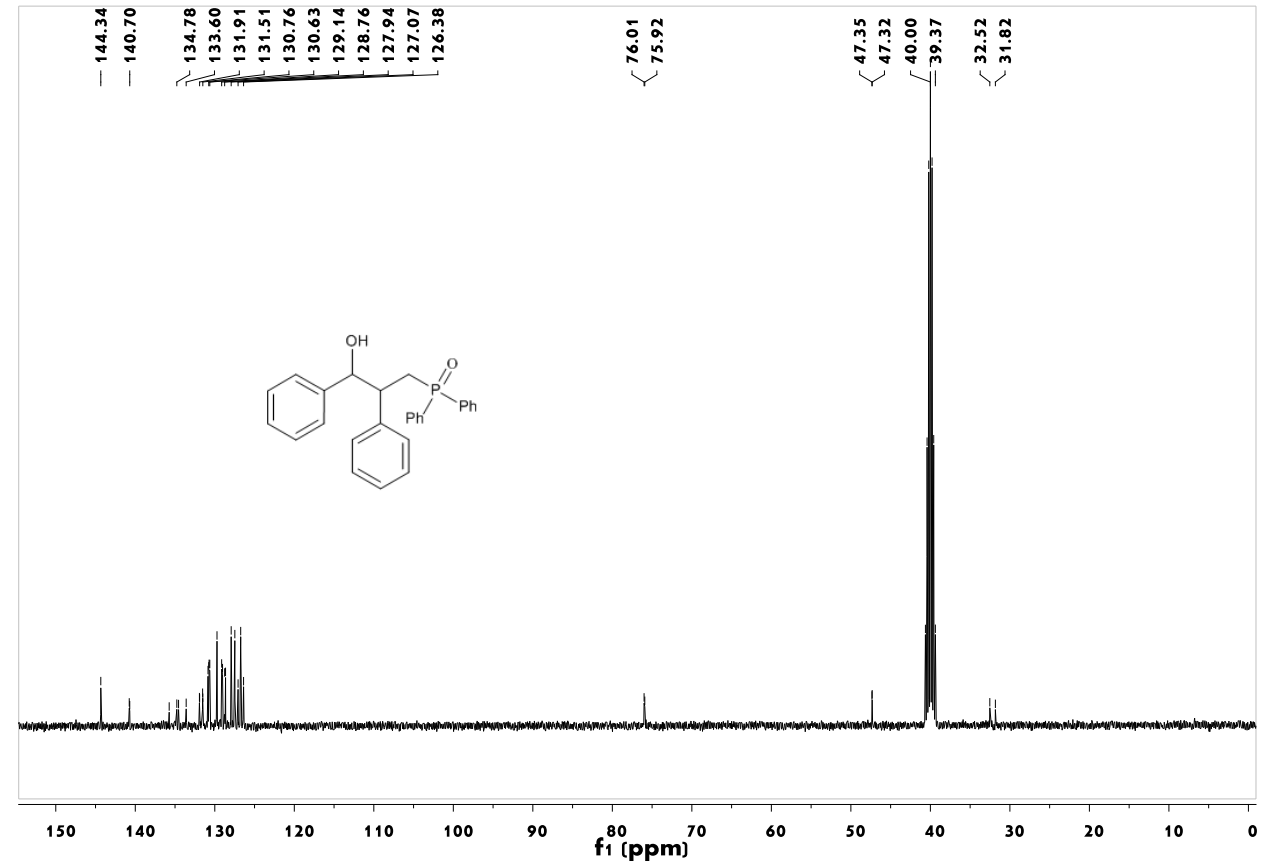

42.77
42.58
OMe
O
O
P
OEt
Ph
MeO
3cb
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210
f1 (ppm)

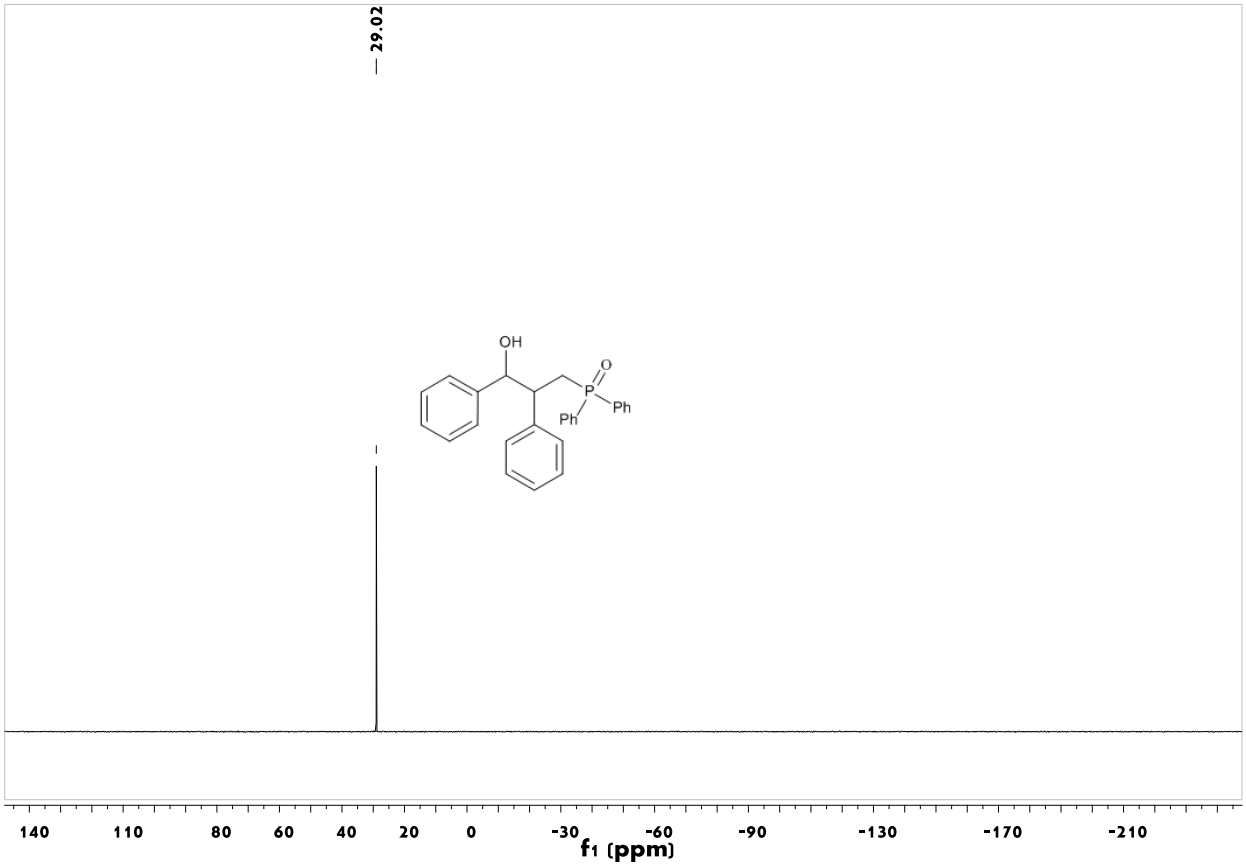

7.55
7.52
7.48
7.45
7.43
7.40
7.37
7.36
7.34
7.22
7.18
7.03
5.12
5.10
5.09
5.07
5.06
5.04
5.03
3.92
3.90
3.87
3.76
3.73
3.68
2.91
2.89
2.87
2.38
2.37
2.36
2.34
1.14
1.12
1.10
1.09
0.00
Br
O
O
P
OEt
Ph
Br
3db
2.02
3.64
1.62
2.97
2.95
4.17
1.95
3.76
1.99
0.88
0.99
1.91
2.00
1.08
0.92
1.00
1.03
2.73
2.94
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)

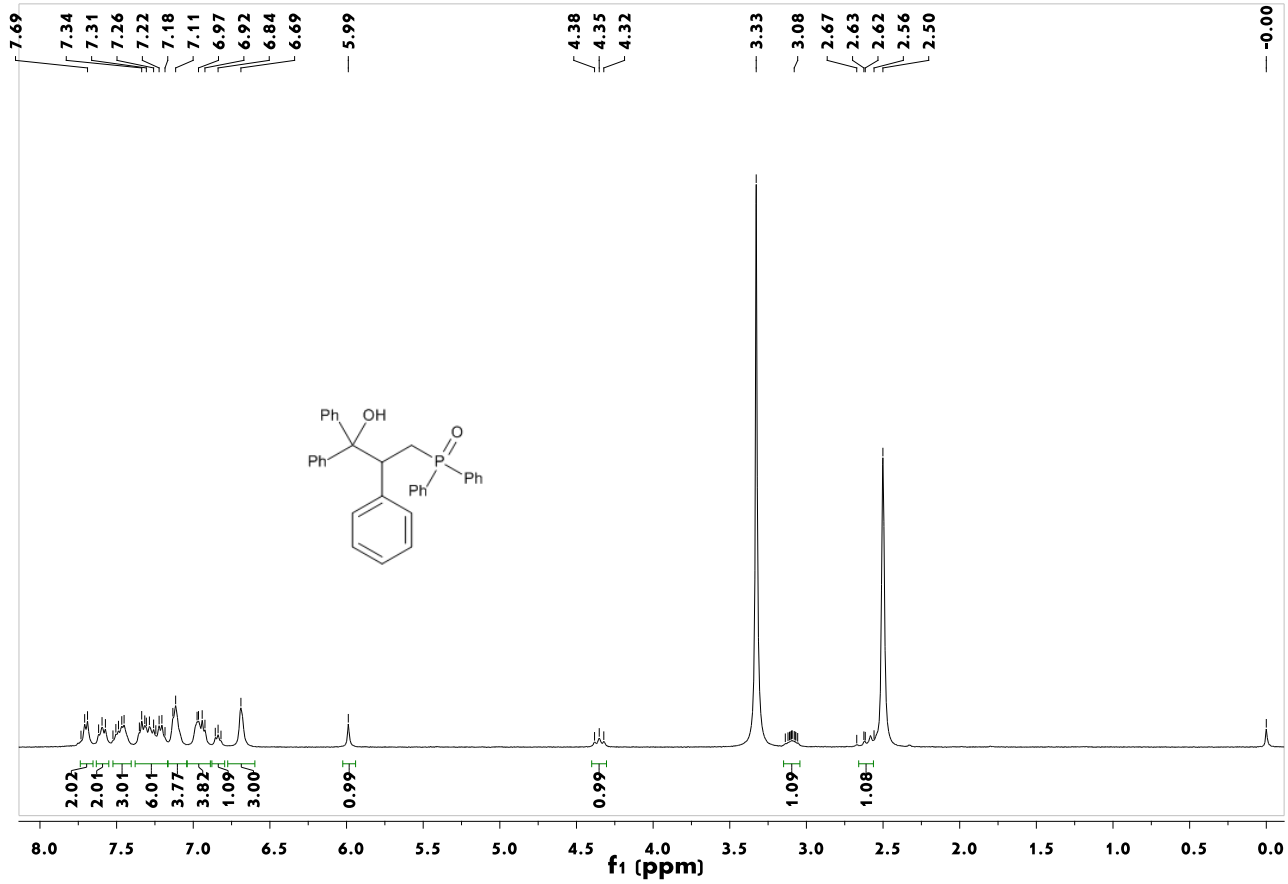

196.91
196.83
196.73
196.66
136.96
134.53
132.42
132.21
131.79
131.66
130.51
130.17
128.87
128.61
121.92
121.88
77.55
77.23
76.91
60.93
60.87
46.73
46.44
34.54
34.28
33.55
33.28
16.44
16.38
Br
O
O
P–OEt
Ph
Br
3db
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

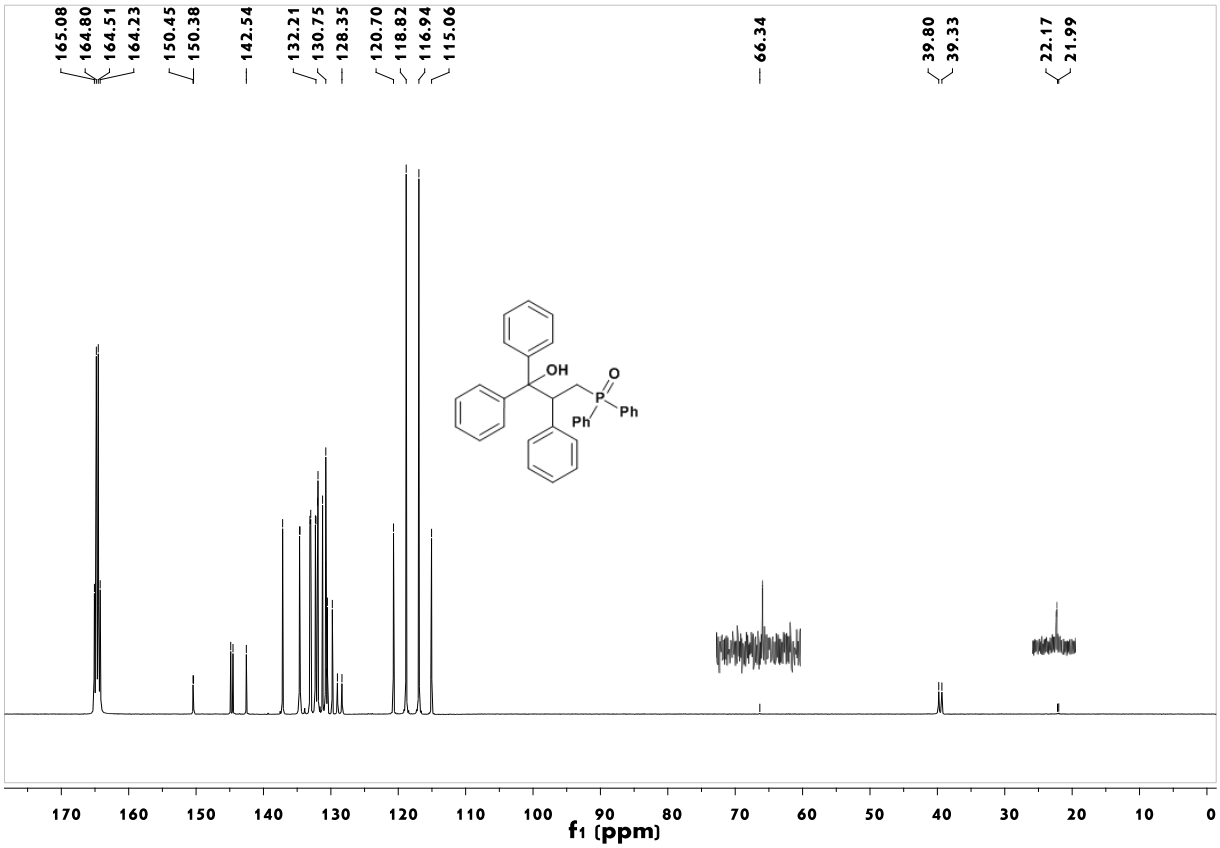

42.01
41.80
Br
O
O
P–OEt
Ph
Br
3db
140 110 80 60 40 20 0 -30 -60 -90 -130 -170 -210
f1 (ppm)

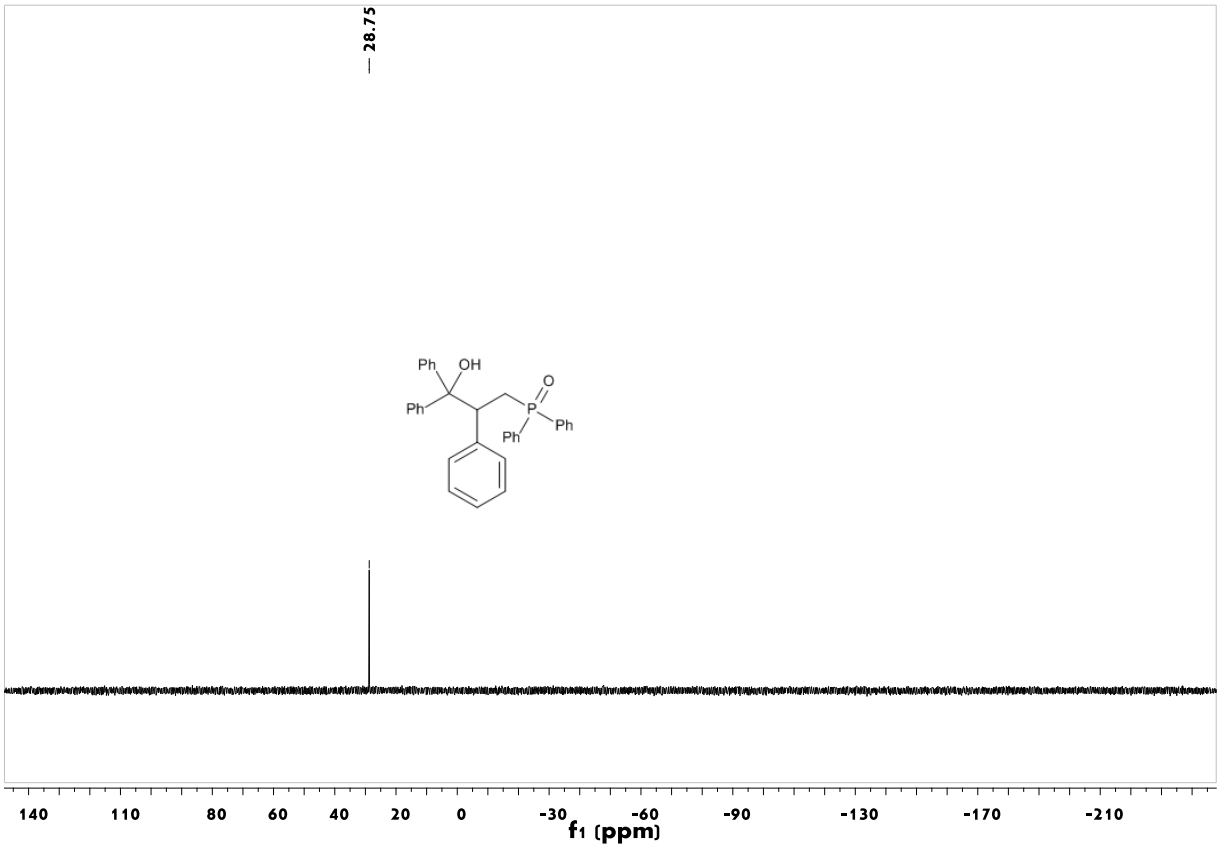

7.52
7.48
7.43
7.40
7.34
7.32
7.16
7.12
7.09
6.89
6.85
5.46
5.45
4.99
4.98
4.97
3.34
3.27
3.25
3.24
3.22
3.21
2.80
2.50
2.50
2.50
0.00
OH
O
P
Ph
Ph
2.11
5.48
1.11
2.14
3.31
5.13
2.26
1.01
1.00
1.09
2.01
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


144.34
140.70
134.78
133.60
131.91
131.51
130.76
130.63
129.14
128.76
127.94
127.07
126.38
76.01
75.92
47.35
47.32
40.00
39.37
32.52
31.82
OH
O
P
Ph
Ph
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


29.02
OH
O
P
Ph
Ph
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210
f1 (ppm)


7.69
7.34
7.31
7.26
7.22
7.18
7.11
6.97
6.92
6.84
6.69
5.99
4.38
4.35
4.32
3.33
3.08
2.67
2.63
2.62
2.56
2.50
-0.00
Ph
OH
Ph
O
P
Ph
Ph
2.02
2.01
3.01
6.01
3.77
3.82
1.09
3.00
0.99
0.99
1.09
1.08
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)


165.08
164.80
164.51
164.23
150.45
150.38
142.54
132.21
130.75
128.35
120.70
118.82
116.94
115.06
66.34
39.80
39.33
22.17
21.99
OH
O
P
Ph
Ph
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


28.75
Ph
OH
Ph
O
P
Ph
Ph
140 110 80 60 40 20 0 -30 -60 -90 -130 -170 -210
f1 (ppm)