Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Nickel-Catalyzed C-P Coupling of Aryl Mesylates and Tosylates with H(O)PR^{1}R^2

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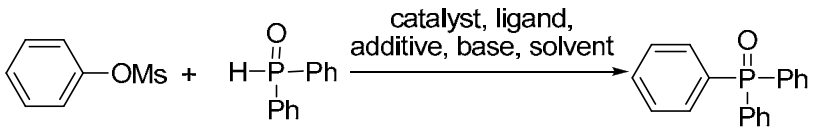
I. General Details

Unless otherwise noted, all reagents were purchased from commercial suppliers and used without purification. All reactions were performed in flame-dried glassware under an atmosphere of dry nitrogen, and the workup was carried out in air, unless otherwise stated. CH_2Cl_2, DMF, NMP, DMSO, HMPA, toluene, pyridine and NEt_3 were distilled at atmospheric or reduced pressure over CaH_2 prior to use. Solvents 1, 4-dioxane, DME and THF were distilled from sodium benzophenone ketyl prior to use. NaH (65% in mineral oil) was degreased with n-hexane under N_2 prior to use. Column chromatographic purification of products was carried out using silica gel 60 (200~300 mesh). NMR spectra were recorded on a Varian MERCURY plus-400 (400 MHz, ^1H; 100 MHz, ^{13}C; 162 MHz, ^{31}P) spectrometer with chemical shifts reported in ppm relative to the residual deuterated solvent, the internal standard tetramethylsilane, or external 85% H_3PO_4 for ^{31}P. Mass spectrometry analysis was carried out using an electrospray spectrometer Waters 4 micro quadrupole. Melting points were measured with SGW X-4 micro melting point apparatus.

II Experimental procedures and spectral data

Table S1. Reaction condition screening[a]



Entry	Catalyst	Ligand	Additive	Base	Temperature (°C)	Solvent	Isolated yield (%)
1	-	-	Zn	DIPEA	100	DMF	0
2	$Ni(acac)_2$	IPr·HCl	Zn	NaH	90	DMF	0
3	$Ni(acac)_2$	IPr·HCl	Zn	NaH	60	THF	0
4	$NiCl_2$	IPr·HCl	-	DABCO	100	DMF	0
5	$NiCl_2$	IPr·HCl	Zn	DABCO	100	DMF	0
6	$NiCl_2$	2,2'-bipy	Zn	-	100	DMF	0
7	$NiCl_2(PCy_3)_2$	-	Zn	NEt_3	100	Toluene	0
8	$NiCl_2(PCy_3)_2$	-	Zn	DABCO	100	DMF	0
9	$NiCl_2$(dppe)	-	-	DABCO	100	DMF	0
10	$NiCl_2$(dppp)	-	-	DABCO	100	DMF	0
11	$NiCl_2$(dppf)	-	Zn	DIPEA	100	DMF	17
12	$NiCl_2$	dppf	Zn	-	100	DMF	26
13	$NiCl_2$	dppf	-	DABCO	100	DMF	0
14	$NiCl_2$	dppf	Zn	-	100	1,4-dioxane	56[b]
15	$NiCl_2$(dppf)	dppf	Zn	-	100	DMF	74[c]
16	$NiCl_2$(dppf)	dppf	Zn	-	100	1,4-dioxane	30[c]
17	$NiCl_2$(dppp)	dppp	Zn	-	100	DMF	0[c]
18	$NiCl_2$(dppf)	dppf	Zn	-	100	DMA	30[c]
19	$NiCl_2$(dppf)	dppf	Zn	-	100	DMF	52[d]
20	$NiCl_2$(dppf)	dppf	Zn	-	80	DME	27[c]
21	$NiCl_2$(dppf)	dppf	Zn	-	100	NMP	50[c]

22	$NiCl_2$(dppf)	dppf	Zn	-	100	DMSO	0[c]
23	$NiCl_2$(dppf)	dppf	Zn	-	100	HMPA	31[c]
24	$NiCl_2$(dppf)	dppf	Zn	-	120	DMF	57[c]
25	$NiCl_2$(dppf)	dppf	Zn	-	100	DMF	74[e]
26	$NiCl_2$(dppf)	dppf	Zn	-	80	DMF	69[c]
27	$NiCl_2$(dppf)	dppf	Zn	DIPEA	100	DMF	87[f]
28	$NiCl_2$(dppf)	dppf	Zn	NEt_3	100	DMF	78[f]
29	$NiCl_2$(dppf)	dppf	Zn	DABCO	100	DMF	84[f]
30	$NiCl_2$(dppf)	dppf	-	DIPEA	100	DMF	0[g]

[a] Unless otherwise stated, reaction conditions: phenyl mesylate (0.50 mmol), $Ph_2P(O)H$ (0.60 mmol), catalyst (0.05 mmol), ligand (0.05 mmol), zinc dust (0.50 mmol), base (0.50 mmol), solvent (3 mL) under N_2. [b] ligand (0.15 mmol). [c] ligand (0.10 mmol). [d] ligand (0.05 mmol). [e] zinc dust (1.0 mmol). [f] base (1.0 mmol). [g] no zinc dust.

Preparation of Ni complex and ligand

$NiCl_2$(dppf) was prepared from $NiCl_2 \cdot 6H_2O$ and bisphosphine ligand dppf according to a literature procedure.[1a] $NiCl_2$(dppp) was synthesized from $NiCl_2 \cdot 6H_2O$ and bisphosphine ligand dppp according to a literature procedure.[1b] $NiCl_2(PCy_3)_2$ was prepared from $NiCl_2 \cdot 6H_2O$ and monophosphine ligand PCy_3 according to a literature procedure.[1c] IPr·HCl was prepared according to a literature procedure.[1d]

Preparation of Aryl Mesylates or Aryl Tosylates

Phenyl mesylate (**1a**, CAS number: 16156-59-5)[2], phenyl tosylate (**1a**, CAS number: 640-60-8)[2], *p*-tolyl tosylate (**1b**, CAS number: 3988-96-5)[2], *m*-tolyl tosylate (**1c**, CAS number: 3955-72-4)[2], *o*-tolyl tosylate (**1d**, CAS number: 599-75-5)[2], 4-methoxyphenyl mesylate (**1e**, CAS number: 19013-30-0)[2], methyl 4-(tosyloxy)benzoate (**1f**, CAS number: 51207-43-3)[2], 3-methoxyphenyl tosylate (**1g**, CAS number: 3988-92-1)[3], 2-naphthyl mesylate (**1h**, CAS number: 10290-91-2)[2], 1-naphthyl tosylate (**1i**, CAS number: 68211-49-4)[3], 1,3-benzodioxol-5-ol mesylate (**1j**, CAS number: 128612-45-3)[4] were prepared from their corresponding phenols with MsCl or TsCl in the presence of pyridine in CH_2Cl_2 according to known procedures.

Preparation of diarylphosphine oxide

Bis(4-methylphenyl)phosphine oxide (**2k**, CAS number: 2409-61-2)[5],
bis(4-methoxyphenyl)phosphine oxide (**2l**, CAS number: 15754-51-5)[5],
bis(4-(trifluoromethyl)phenyl)phosphine oxide (**2m**, CAS number: 15929-43-8)[6] and
bis(3,5-dimethylphenyl)phosphine oxide (**2n**, CAS number: 187344-92-9)[7] were prepared from their corresponding Grignard reagents with diethyl phosphate in THF according to known procedures and their spectral data are in agreement with literature values.

Preparation of ethyl phenylphosphinate

Ethyl phenylphosphinate (CAS number: 2511-09-3) was prepared from dichloro(phenyl)-phosphine (DCPP) with ethanol according to a literature procedure.[8]

Procedure for activation of zinc dust

Acticvated zinc dust was prepared prior to use according to a literature procedure.[9]

General procedure for Ni-catalyzed cross-coupling of aryl mesylates or tosylate with H(O)PR^{1}R^2

In a typical reaction, to an oven-dried 25 mL Schlenk tube was added $NiCl_2$(dppf) (50 μmol), dppf (0.10 mmol) and activated Zn dust (0.50 mmol). The tube was sealed with a rubber septum and then degassed by pumping and backfilling with nitrogen three times. DMF (1 mL) was added *via* a syringe. The reaction mixture was stirred at 80 °C for 0.5 h. During this time, the solution of mixture turned from yellow to red. DMF (2 mL) solution containing aryl mesylates or tosylates (0.50 mol), H(O)PR^{1}R^2 (0.60 mmol), and DIPEA (95 μL, 1.0 mmol) was added *via* a syringe through the rubber septum. The reaction mixture was stirred at 100 °C under a nitrogen atmosphere for 36 h. The reaction mixture was allowed to cool to room temperature and the DMF was evaporated *in vacuo*. The residue was dissolved in CH_2Cl_2 (10 mL) and then washed with 5% HCl (3 × 5 mL) and H_2O (3 × 3 mL), and dried by Na_2SO_4. Solvent was evaporated *in vacuo* and the residue was purified by column chromatography (silica gel, petroleum ether/ethyl acetate/EtOH).

Phosphoryl triphenyl (3a, CAS Number: 791-28-6)[10]

White solid, ^{31}P NMR ($CDCl_3$, 162 MHz): δ = 29.45; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.64-7.70 (m, 6H), 7.52-7.56 (m, 3H), 7.43-7.48 (m, 6H).

(4-Methylphenyl)diphenyl phosphine oxide (**3b**, CAS Number: 23081-74-5)[10]

White solid, ^{31}P NMR ($CDCl_3$, 162 MHz): δ = 27.73; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.64-7.69 (m, 4H), 7.49-7.58 (m, 4H), 7.41-7.46 (m, 4H), 7.25-7.28 (m, 2H), 2.39 (s, 3H).

(3-Methylphenyl)diphenyl phosphine oxide (**3c,** CAS number: 6840-27-3)

White solid, mp: 123.7-124.2 °C; ^{31}P NMR ($CDCl_3$, 162 MHz): δ = 30.53; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.68-7.63 (m, 4H), 7.56-7.52 (m, 3H), 7.48-7.43 (m, 4H), 7.37-7.33 (m, 3H) 2.36 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 138.7 (d, *J* = 12.7 Hz), 132.8 (d, *J* = 142.1 Hz), 133.0 (d, *J* = 3.6 Hz), 132.4 (d, *J* = 102.5 Hz), 132.7 (d, *J* = 9.5 Hz), 132.3 (d, *J* = 10.0 Hz), 132.1 (d, *J* = 1.9 Hz), 129.4 (d, *J* = 11.3 Hz), 128.7 (d, *J* = 12.4 Hz), 128.5 (d, *J* =12.9 Hz), 21.66; HR-ESI-MS: $[M+H]^+$ m/z calcd for : 293.1095, found: 293.1108.

(2-Methylphenyl)diphenyl phosphine oxide (**3d**, CAS number: 6840-26-2)[11]

White solid, ^{31}P NMR ($CDCl_3$, 162 MHz): δ = 31.71; ^{1}H NMR ($CDCl_3$, 400 MHz): 7.68-7.61 (m, 4H), 7.56-7.38 (m, 7H), 7.27 (ddd, *J* = 14.0, 7.6, 0.4 Hz, 1H), 7.11 (m, 1H), 7.02 (ddd, *J* = 14.0, 7.6, 1.2 Hz, 1H), 2.45 (s, 3H).

(4-Methoxyphenyl)diphenyl phosphine oxide (**3e**, CAS Number: 795-44-8)[10]

White solid, ^{31}P NMR ($CDCl_3$, 162 MHz): δ = 32.91; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.64-7.70 (m, 4H), 7.50-7.54 (m, 2H), 7.42-7.46 (m, 4H), 7.28-7.37 (m, 2H), 7.12-7.17 (m, 1H), 7.06 (dd, *J* = 8.9, 2.6 Hz), 3.77 (s, 3H).

(4-Carbomethoxyphenyl)diphenyl phosphine oxide (3f, CAS Number: 5032-55-3)[12]

White solid, ^{31}P NMR (100MHz, $CDCl_3$): δ = 28.9; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.12 (d, *J* = 8.2 Hz, 2H), 7.77 (dd, *J* = 11.4, 8.4 Hz, 2H), 7.64–7.69 (m, 4H), 7.46–7.60 (m, 6H), 3.93 (s, 3H).

(3-Methoxyphenyl)diphenyl phosphine oxide (**3g**, CAS Number: 95278-09-4)[10]

White solid, ^{31}P NMR ($CDCl_3$, 162 MHz): δ = 32.91; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.64-7.70 (m, 4 H), 7.50-7.54 (m, 2H), 7.42-7.46 (m, 4H), 7.28-7.37 (m, 2H), 7.12-7.17 (m, 1H), 7.06 (dd, *J* = 8.9, 2.6 Hz), 3.77 (s, 3 H).

2-Naphthalenyldiphenyl phosphine oxide (**3h**, CAS Number: 28402-08-6)[13]

White solid, ^{31}P NMR ($CDCl_3$, 162 MHz): δ = 31.32; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.27 (d, *J* = 13.6 Hz, 1H), 7.88 (d, *J* = 9.6 Hz, 2H), 7.73-7.68 (m, 4H), 7.64-7.46 (m, 10H).

1-Naphthalenyldiphenyl phosphine oxide (**3i**, CAS Number: 3095-33-8)[14]

White solid, ^{31}P NMR ($CDCl_3$, 162 MHz): δ = 33.12; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.58 (d, *J* = 8.20 Hz, 1H), 8.01 (d, *J* = 8.08 Hz, 1H), 7.88 (d, *J* = 8.18 Hz, 1H), 7.66-7.71 (m, 4H), 7.52-7.56 (m, 2H), 7.43-7.50 (m, 6H), 7.35-7.39 (m, 1H), 7.26-7.32 (m, 1H).

1,3-Benzodioxol-5-yldiphenyl phosphine oxide (**3j**, CAS number: 209981-66-8)[15]

White solid, ^{31}P NMR ($CDCl_3$, 162 MHz): δ = 34.53; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.66 (dd, *J* = 11.6, 7.5 Hz, 4H), 7.54 (d, *J* = 7.1 Hz, 2H), 7.46 (t, *J* = 7.1 Hz, 4H), 7.18 (dd, *J* = 12.6, 8.0 Hz, 1H), 7.07 (d, *J* = 11.4 Hz, 1H), 6.88 (dd, *J* = 7.9, 2.1 Hz, 1H), 6.02 (s, 2H).

Bis(4-methylphenyl)phenyl phosphine oxide (**3k**, CAS number: 18957-70-5)[16]

Colorless slurry, ^{31}P NMR ($CDCl_3$, 162 MHz): δ = 30.53; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.68-7.62 (m, 2H), 7.53 (dd, *J* = 11.8, 8.0 Hz, 4H), 7.48 (m, 1H), 7.24 (dd, *J* = 8.4, 2.4 Hz, 4H), 2.38 (s, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 142.6 (d, *J* = 2.9 Hz), 133.0 (d, *J* = 102.5 Hz), 132.2 (d, *J* = 10.2 Hz), 132.0 (d, *J* = 8.7 Hz),131.9 (d, *J* = 3.2 Hz), 129.4 (d, *J* = 106.9 Hz), 129.4 (d, *J* = 12.6 Hz), 128.6 (d, *J* = 11.8 Hz), 21.7.

Bis(4-methoxypheny1)phenylphosphine oxide (3l, CAS number: 799-55-3)[17]

White solid, mp: 96.5-97.4 °C; ^{31}P NMR ($CDCl_3$, 162 MHz): δ = 30.36; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.61-7.54 (m, 2H), 7.56 (dd, *J* = 11.2, 8.4 Hz, 4H), 7.51-7.49 (m, 1H), 7.45-7.41 (m, 2H), 6.94 (dd, *J* = 6.4, 1.8 Hz, 4H), 3.83 (s, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 162.6 (d, *J* = 1.8 Hz), 134.1 (d, *J* = 11.7 Hz), 133.3 (d, *J* = 104.3 Hz), 132.2 (d, *J* = 9.8 Hz), 131.9 (d, *J* = 2.7 Hz), 128.6 (d, *J* = 12.4 Hz), 124.0 (d, *J* = 110.2 Hz), 114.2 (d, *J* = 12.9 Hz), 55.5.

Bis(3, 5-bismethylphenyl)phenylphosphine oxide (3n)

White solid, mp: 158.6-159.2 °C; ^{31}P NMR ($CDCl_3$, 162 MHz): δ = 30.89; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.68-7.63 (m, 2H), 7.55-7.51 (m, 1H), 7.47-7.42 (m, 2H), 7.26 (d, *J* = 12.4 Hz, 4H), 7.15 (s, 2H), 2.31 (s, 12H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 138.3 (d, *J* = 12.2 Hz), 133.9 (d, *J* = 2.3 Hz), 133.1 (d, *J* = 102.7 Hz), 132.4 (d, *J* = 102.6 Hz), 132.3 (d, *J* = 9.7 Hz), 131.9 (d, *J* = 2.2 Hz), 129.8(d, *J* = 10.0 Hz), 128.6 (d, *J* = 11.7 Hz), 21.56; HR-ESI-MS: $[M+H]^+$ m/z calcd for :335.1565, found: 335.1574.

Diethyl phenylphosphonate (5a, CAS number: 1754-49-0)[18]

Oil, ^{1}H NMR (400 MHz,$CDCl_3$): δ = 7.82 (m, 2H), 7.55 (tq, *J* = 7.5, 1.4 Hz, 1H), 7.47 (m, 2H), 4.12 (m, 4H), 1.32 (td, *J* = 7.0, 0.5 Hz, 6H).

Diethyl *p*-tolylphosphonate (5b, CAS number: 1754-46-7**)**[18]
Oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.68 (d, *J* = 8.9 Hz, 2H), 7.26 (d, *J* = 8.9 Hz, 2H), 4.04-4.18 (m, 4H), 2.41 (s, 3H), 1.31 (t, *J* = 6.9 Hz, 6H).

Diethyl *m*-tolylphosphonate (5c, CAS number: 15286-13-2**)**[18]
Oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.57-7.67 (m, 2H), 7.27-7.36 (m, 2H**),** 4.12-4.20 (m, 4H), 2.37 (s, 3H), 1.32 (t, *J* = 7.0 Hz, 6H).

Diethyl *o*-tolylphosphonate (5d, CAS number: 15286-11-0**)**[18]
Oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.88-7.96 (m, 1H), 7.42-7.46 (m, 1H), 7.23-7.28 (m, 2H), 4.12 (m, 4H), 2.57 (s, 3H), 1.33 (t, *J* = 6.9 Hz, 6H).

Diethyl *p*-methoxyphenylphosphonate (5e, CAS number: 3762-33-2**)**[18]
Oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.72 (dd, *J* = 13.0, 9.0 Hz, 2H), 6.97 (dd, *J* = 9.0, 3.0 Hz, 2H), 4.04-4.13 (m, 4H), 3.83 (s, 3H), 1.30 (t, *J* = 7.3 Hz, 6H).

Diethyl 3-methoxyphenylphosphonate (**5g**, CAS number: 65442-22-0)[18]
Oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.32-7.40 (m, 3H), 7.07-7.10 (m, 1H), 4.05-4.18 (m, 4H), 3.85 (s, 3H), 1.33 (t, *J* = 6.9 Hz, 6H).

Diethyl naphthalen-2-yl phosphonate (5h, CAS number: 17067-93-5**)**[18]
Oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.41 (d, *J* = 16.4 Hz, 1H), 7.84-7.93 (m, 3H), 7.71-7.76 (m, 1H), 7.51-7.59 (m, 2H), 4.17-4.23 (m, 4H), 1.32 (t, *J* = 7.3 Hz, 6H).

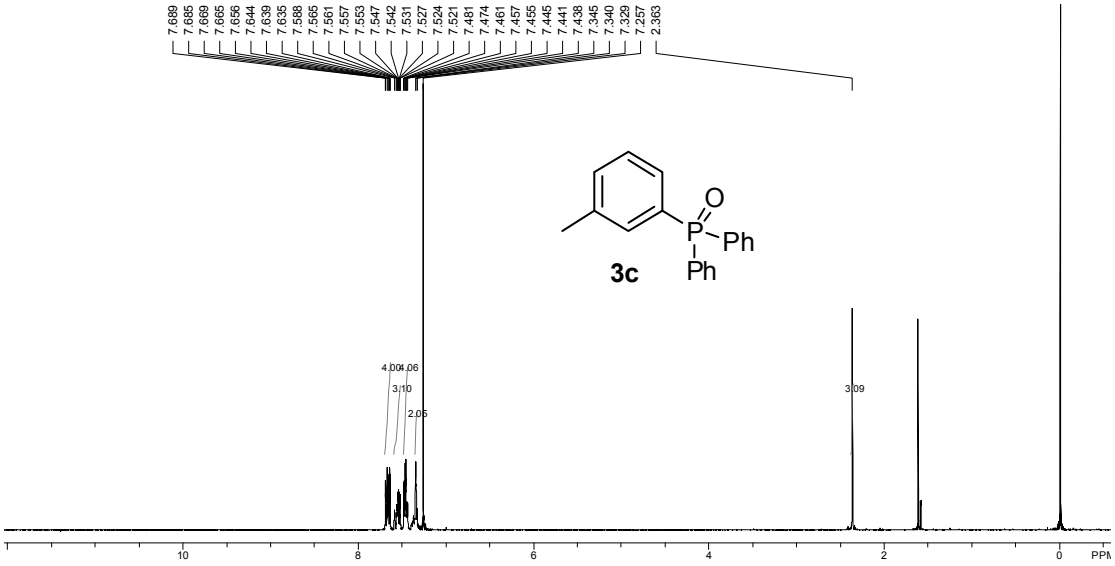
Diethyl naphthalen-1-yl phosphonate (5i, CAS number: 25944-75-6**)**[18]
Oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.47 (d, *J* = 8.0 Hz, 1H), 8.22 (dd, *J* = 16.2, 7.0 Hz, 1H), 8.01 (d, *J* = 8.0 Hz, 1H), 7.87 (d, *J* = 7.2 Hz, 1H), 7.491-7.61 (m, 3H), 4.02-4.24 (m, 2H), 1.31 (t, *J* = 7.1 Hz, 6H).

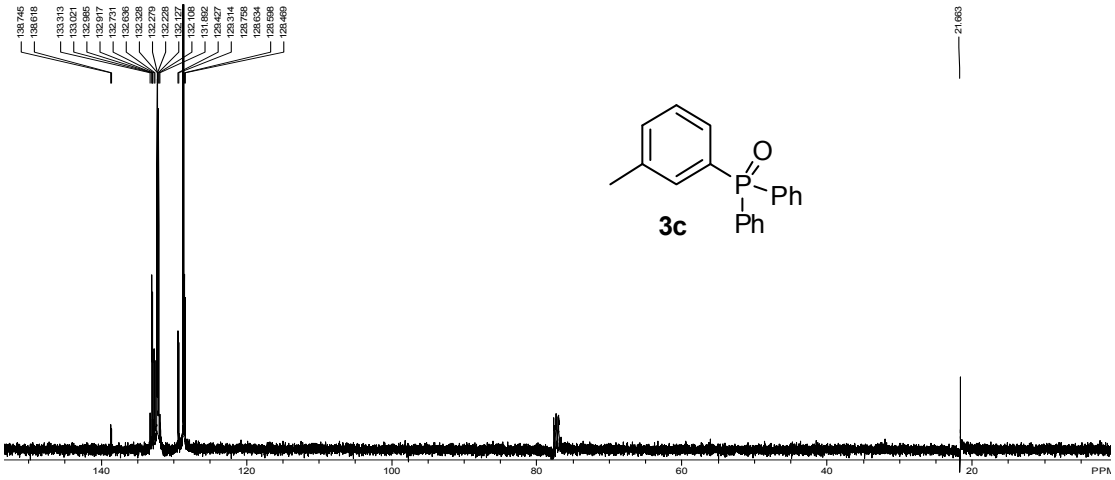
Ethyl diphenylphosphinate (5j, CAS number: 1262219-69-1**)**[19]
Oil, ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.64-7.66 (m, 4H), 7.47 (d, *J* = 7.56 Hz, 4H), 4.16 (m, 2H), 1.38 (m, 3H).

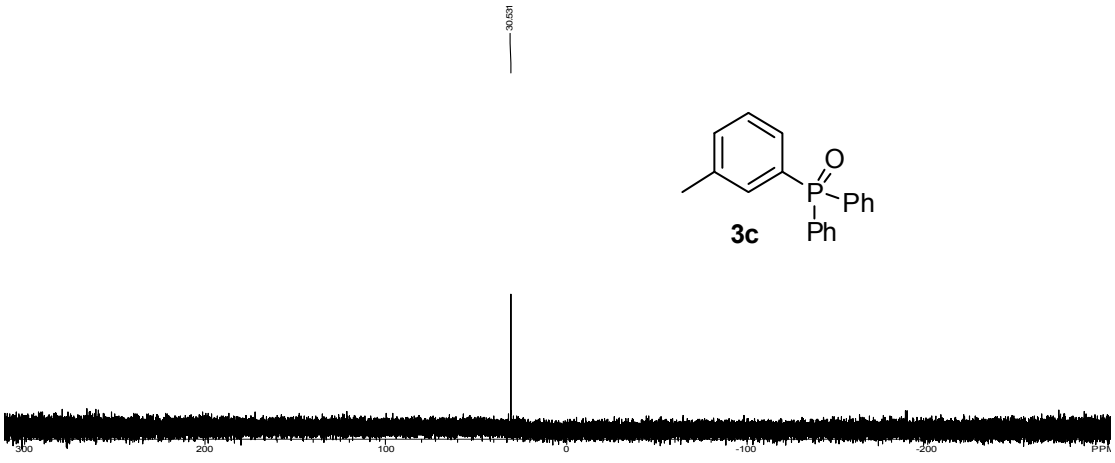
Ethyl (4-methylphenyl)phenylphosphinate (5k, CAS number: 26926-25-0**)**[19]
Oil, ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.66 (d, *J* = 7.2 Hz, 2H), 7.48-7.51 (m, 3H), 7.39-7.45 (m, 1H), 6.96-6.99 (m, 4H), 4.15 (m, 2H), 2.18 (s, 3H), 1.28 (m, 3H).

III. ^{1}H NMR, ^{13}C NMR and ^{31}P NMR spectra copes of new compounds

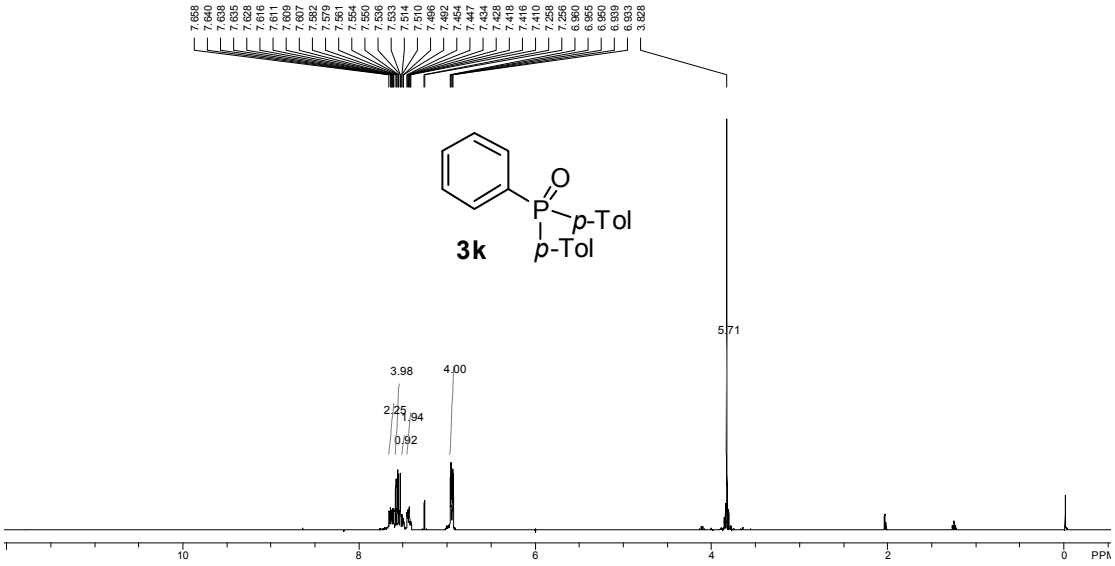
(3-Methylphenyl)diphenyl phosphine oxide (3c)

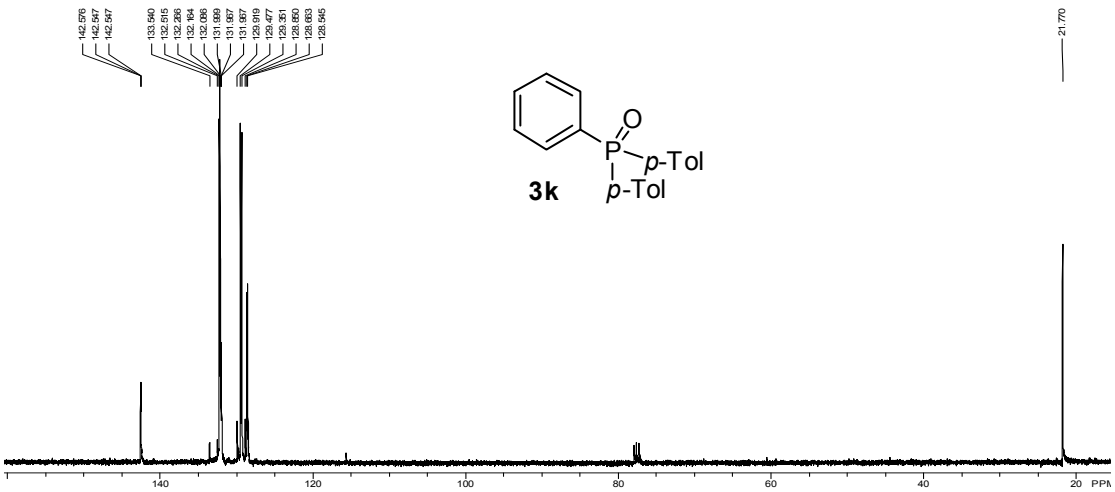


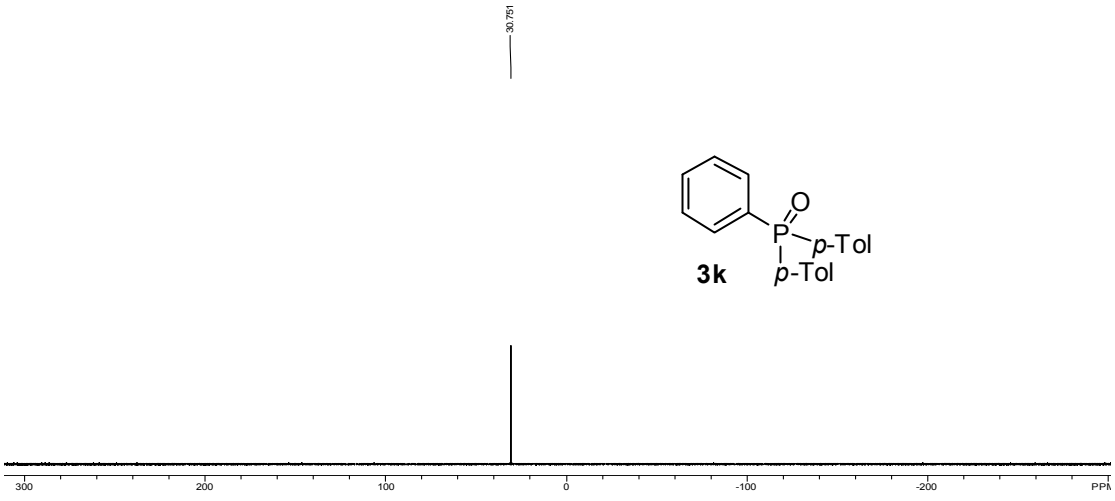




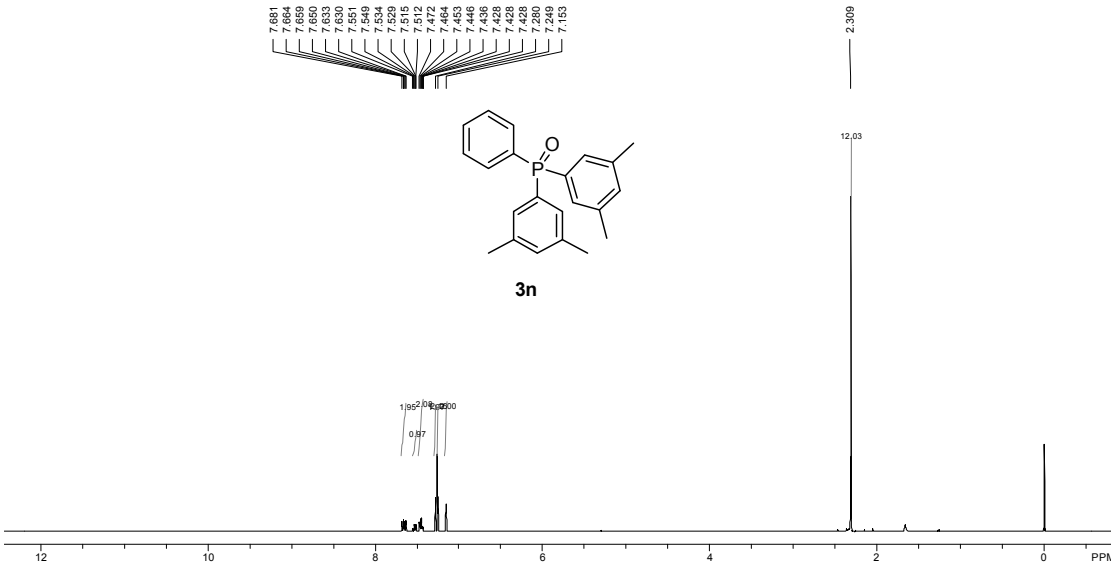
Bis(4-methylphenyl)phenyl phosphine oxide (3k)

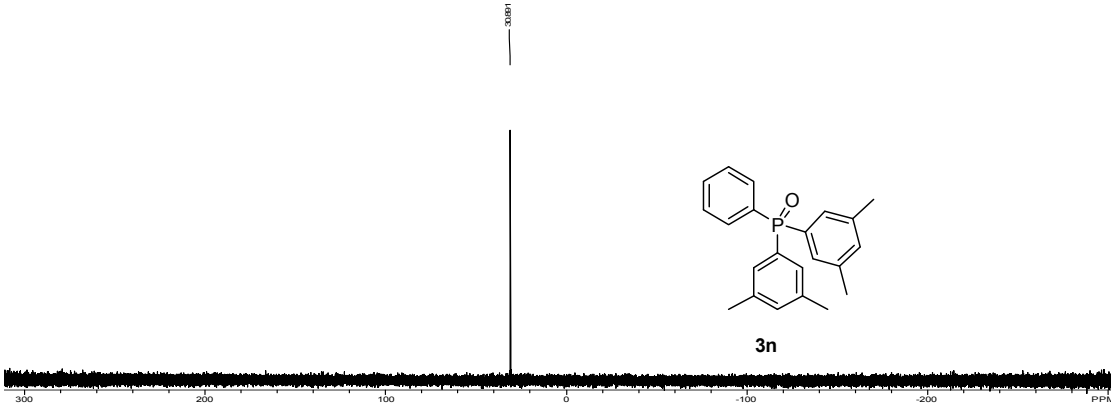






Bis(3, 5-bismethylphenyl)phenylphosphine oxide (3n)





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