Eosin Y-Catalyzed Photooxidation of Triarylphosphines under Visible Light Irradiation and Aerobic Conditions

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Electronic Supplementary Information

Experimental Section	S2
NMR Spectra	S10
References	S35

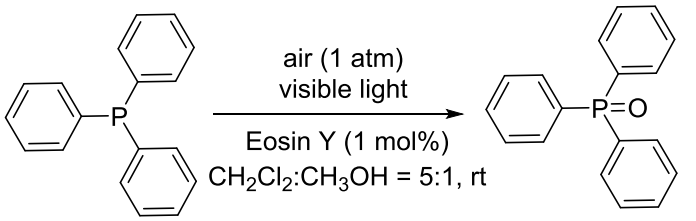
Experimental Section

General information

All reactions were carried out using a 23 W household lamp at a distance of 3-5 cm with the reactor. ^{1}H (400 MHz), ^{13}C (100 MHz), ^{31}P (162 MHz), ^{19}F (376 MHz) NMR spectra of samples in $CDCl_3$ were recorded on an AVANCE III 400 spectrometer. Melting points were determined on a SGWX-4 Micro Melting Point Apparatus. Compounds **1a**, **1b**, **1d**, **1e**, **1f**, **1j**, **1k**, and **1o** were commercial available. Compounds **1c**,[1] **1g**,[1] **1h**,[1] **1i**,[2] **1l**,[3] **1m**,[4] **1n**,[5] and **1p**[6] were prepared according to literature procedures. Quantum yield was determined using the phenylglyoxylic acid chemical actinometer system.[7]

Typical Procedure I for the photoreaction

Synthesis of triphenylphosphine oxide (2a)[8]

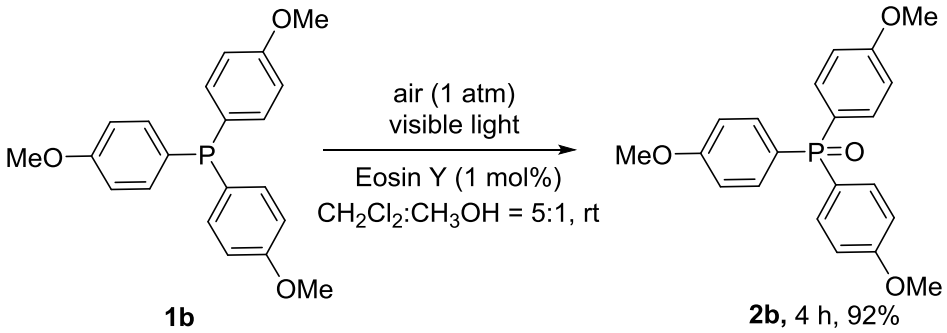


1a **2a**, 3.5 h, 95%

1a (54 mg, 0.21 mmol), Eosin Y (2 mg, 0.0031 mmol), CH_2Cl_2 (2.5 mL), and CH_3OH (0.5 mL) were added to a pyrex reaction flash which was equipped with a magnetic stirrer. The mixture was irradiated by a 23 W household lamp at rt under air atmosphere. The photoreaction was completed after 3.5 hours as monitored by TLC (eluent: petroleum ether). The solvent was removed and the residue was purified by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 2/1→1/1→EA) to afford **2a** as a solid (52 mg, 95%); mp 157.3-157.8 ºC (petroleum ether/ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71-7.63 (m, 6 H), 7.58-7.51 (m, 3 H), 7.50-7.42 (m, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 132.5 (d, J_{PC} = 104.1 Hz), 132.1 (d, J_{PC} = 9.8 Hz), 131.9 (d, J_{PC} = 1.9 Hz), 128.5 (d, J_{PC} = 12.1 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.8 ppm.

The following compounds were prepared according to Typical Procedure I.

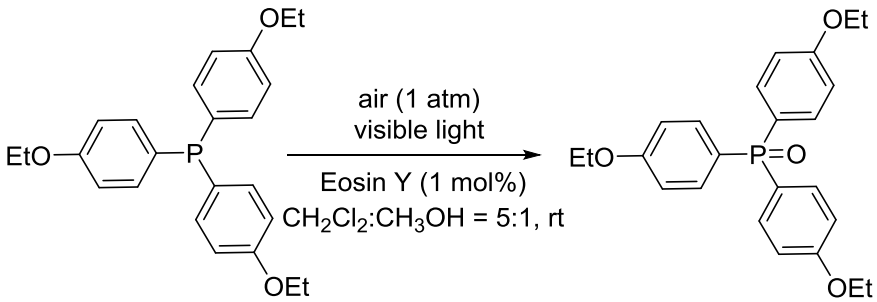
(1) Tris(4-methoxyphenyl)phosphine oxide (2b)[9]



1b **2b,** 4 h, 92%

The reaction of **1b** (72 mg, 0.20 mmol), Eosin Y (2 mg, 0.0031 mmol), CH_2Cl_2 (2.5 mL), and CH_3OH (0.5 mL) afforded **2b** as a solid (68 mg, 92%); mp 141.7-142.4 ºC (petroleum ether/ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (dd, J = 11.6, 8.8 Hz, 6 H), 6.96 (dd, J = 6.8, 2.0 Hz, 6 H), 3.84 (s, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.2 (d, J_{PC} = 2.8 Hz), 133.7 (d, J_{PC} = 11.2 Hz), 124.1 (d, J_{PC} = 110.7 Hz), 113.9 (d, J_{PC} = 13.1 Hz), 55.2; ^{31}P NMR (162 MHz, $CDCl_3$) δ 32.0 ppm.

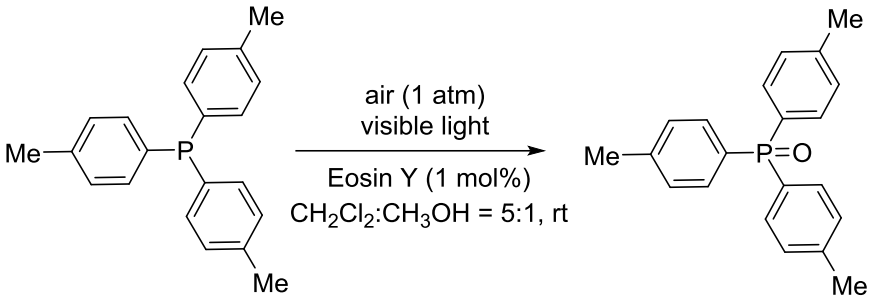
(2) Tris(4-ethoxyphenyl)phosphine oxide (2c)[1]



1c **2c**, 2 h, 99%

The reaction of **1c** (79 mg, 0.20 mmol), Eosin Y (2 mg, 0.0031 mmol), CH_2Cl_2 (2.5 mL), and CH_3OH (0.5 mL) afforded **2c** as a solid (81 mg, 99%); mp 151.2-151.5 ºC (petroleum ether/ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (dd, J = 11.6, 8.8 Hz, 6 H), 6.93 (dd, J = 8.8, 2.0 Hz, 6 H), 4.06 (q, J = 7.0 Hz, 6 H), 1.42 (t, J = 7.0 Hz, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.6, 133.7 (d, J_{PC} = 11.1 Hz), 124.1 (d, J_{PC} = 111.0 Hz), 114.3 (d, J_{PC} = 13.1 Hz), 63.5, 14.6; ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.3 ppm.

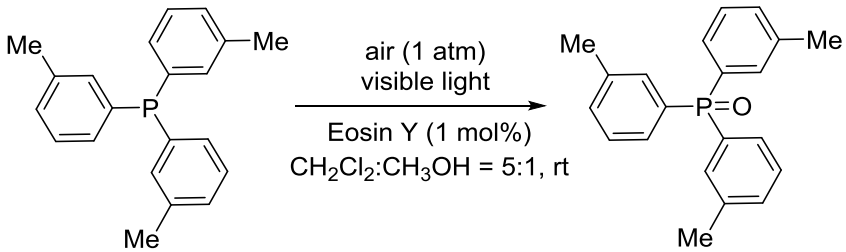
(3) Tris(4-methylphenyl)phosphine oxide (2d)[9]



1d **2d**, 4 h, 89%

The reaction of **1d** (61 mg, 0.20 mmol), Eosin Y (2 mg, 0.0031 mmol), CH_2Cl_2 (2.5 mL), and CH_3OH (0.5 mL) afforded **2d** as a solid (57 mg, 89%); mp 142.4-142.9 ºC (petroleum ether/ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (dd, J = 11.6, 8.0 Hz, 6 H), 7.25 (d, J = 7.2 Hz, 6 H), 2.39 (s, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.1, 132.0 (d, J_{PC} = 10.2 Hz), 129.6 (d, J_{PC} = 107.5 Hz), 129.1 (d, J_{PC} = 12.4 Hz), 21.5; ^{31}P NMR (162 MHz, $CDCl_3$) δ 30.2 ppm.

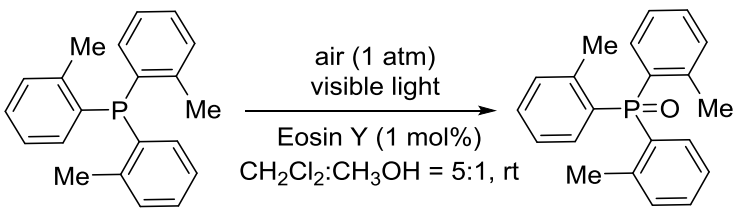
(4) Tris(3-methylphenyl)phosphine oxide (2e)[1]



1e **2e**, 4 h, 95%

The reaction of **1e** (61 mg, 0.20 mmol), Eosin Y (2 mg, 0.0031 mmol), CH_2Cl_2 (2.5 mL), and CH_3OH (0.5 mL) afforded **2e** as a solid (61 mg, 95%); mp 112.3-112.8 ºC (petroleum ether/ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, J = 12.4 Hz, 3 H), 7.40-7.31 (m, 9 H), 2.36 (s, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.3 (d, J_{PC} = 11.8 Hz), 132.5, 132.45 (d, J_{PC} = 102.8 Hz), 132.37 (d, J_{PC} = 9.4 Hz) 129.1 (d, J_{PC} = 10.2 Hz), 128.1 (d, J_{PC} = 12.8 Hz), 21.3; ^{31}P NMR (162 MHz, $CDCl_3$) δ 30.0 ppm.

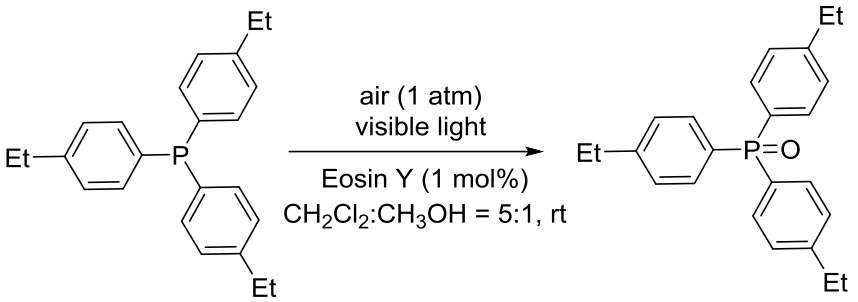
(5) Tris(2-methylphenyl)phosphine oxide (2f)[10]



1f **2f**, 6 h, 91%

The reaction of **1f** (61 mg, 0.20 mmol), Eosin Y (2 mg, 0.0031 mmol), CH_2Cl_2 (2.5 mL), and CH_3OH (0.5 mL) afforded **2f** as a solid (58 mg, 91%); mp 153.6-154.1 °C (petroleum ether/ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45-7.40 (m, 3 H), 7.34-7.28 (m, 3 H), 7.17-7.07 (m, 6 H), 2.50 (s, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.5 (d, J_{PC} = 7.5 Hz), 132.8 (d, J_{PC} = 12.6 Hz), 131.9 (d, J_{PC} = 10.3 Hz), 131.8 (d, J_{PC} = 1.6 Hz), 130.6 (d, J_{PC} = 100.7 Hz), 125.4 (d, J_{PC} = 12.6 Hz), 21.9 (d, J_{PC} = 3.7 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 30.0 ppm.

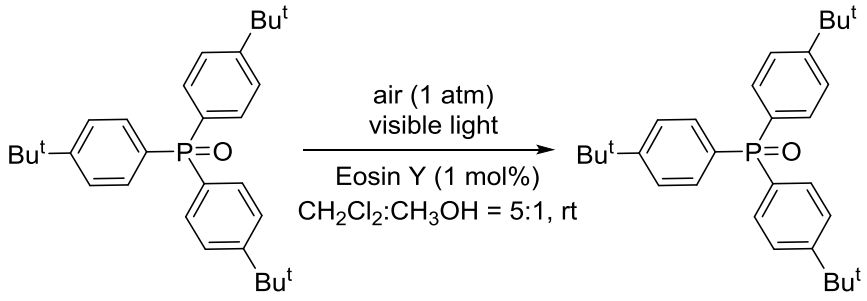
(6) Tris(4-ethylpheny)phosphine oxide (2g)[1]



1g **2g**, 2.5 h, 83%

The reaction of **1g** (71 mg, 0.21 mmol), Eosin Y (2 mg, 0.0031 mmol), CH_2Cl_2 (2.5 mL), and CH_3OH (0.5 mL) afforded **2g** as a solid (60 mg, 83%); mp 154.6-155.0 °C (petroleum ether/ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (dd, J = 11.8, 8.2 Hz, 6 H), 7.31-7.23 (m, 6 H), 2.68 (q, J = 7.6 Hz, 6 H), 1.24 (t, J = 7.6 Hz, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.3, 132.2 (d, J_{PC} = 10.1 Hz), 130.0 (d, J_{PC} = 105.7 Hz), 127.9 (d, J_{PC} = 12.3 Hz), 28.9, 15.2; ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.3 ppm.

(7) Tris(4-(*tert*-butyl)phenyl)phosphine oxide (2h)[1]

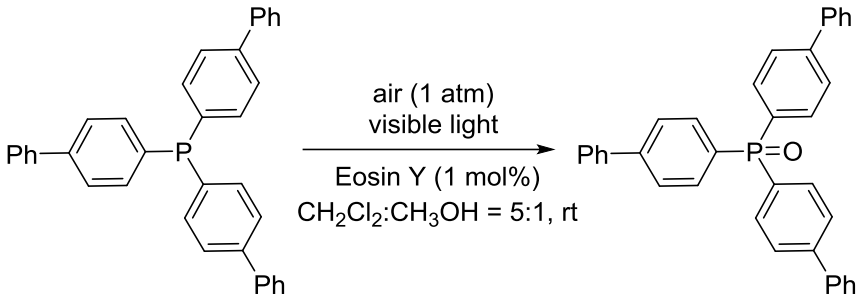


1h **2h,** 3.5 h, 87%

The reaction of **1h** (88 mg, 0.20 mmol), Eosin Y (2 mg, 0.0031 mmol), CH_2Cl_2 (2.5 mL), and CH_3OH (0.5 mL) afforded **2h** as a solid (77 mg, 87%); mp 282.5-282.9 °C (petroleum ether/ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (dd, J = 11.6, 8.4

Hz, 6 H), 7.46 (dd, J = 8.4, 2.4 Hz, 6 H), 1.32 (s, 27 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.1, 131.9 (d, J_{PC} = 10.2 Hz), 129.7 (d, J_{PC} = 105.9 Hz), 125.4 (d, J_{PC} = 12.3 Hz), 35.0, 31.1; ^{31}P NMR (162 MHz, $CDCl_3$) δ 31.1 ppm.

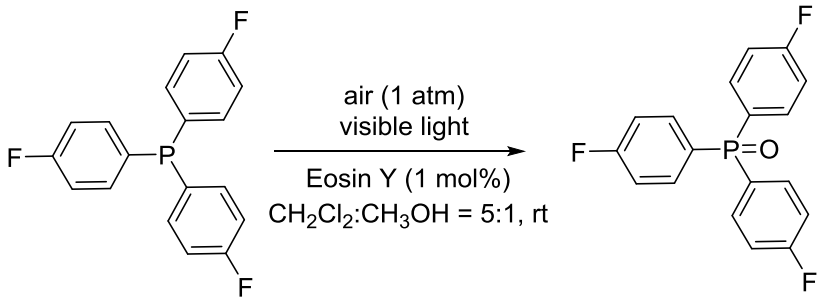
(8) Tris(4-phenylphenyl)phosphine oxide (2i)[11]



1i **2i,** 3 h, 95%

The reaction of **1i** (97 mg, 0.20 mmol), Eosin Y (2 mg, 0.0031 mmol), CH_2Cl_2 (2.5 mL), and CH_3OH (0.5 mL) afforded **2i** as a solid (96 mg, 95%); mp 231.4-231.8 °C (petroleum ether/ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91-7.77 (m, 6 H), 7.76-7.68 (m, 6 H), 7.67-7.57 (m, 6 H), 7.52-7.35 (m, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.8, 139.9, 132.6 (d, J_{PC} = 10.2 Hz), 131.2 (d, J_{PC} = 105.0 Hz), 128.9, 128.1, 127.3, 127.2; ^{31}P NMR (162 MHz, $CDCl_3$) δ 28.8 ppm.

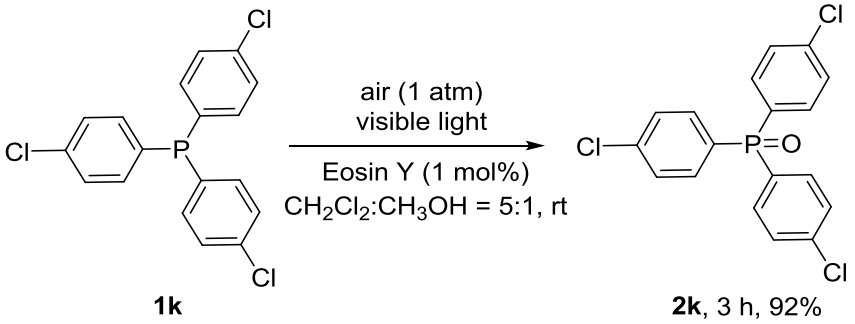
(9) Tris(4-fluorophenyl)phosphine oxide (2j)[12]



1j **2j**, 3 h, 95%

The reaction of **1j** (63 mg, 0.20 mmol), Eosin Y (2 mg, 0.0031 mmol), CH_2Cl_2 (2.5 mL), and CH_3OH (0.5 mL) afforded **2j** as a solid (63 mg, 95%); mp 139.5-140.0 °C (petroleum ether/ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70-7.59 (m, 6 H), 7.22-7.14 (m, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.2 (d, J_{FC} = 253.0 Hz), 134.4 (dd, J = 11.0, 9.3 Hz), 128.1 (d, J_{PC} = 107.7 Hz), 116.1 (dd, J = 21.3, 13.4 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -105.9; ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.3 ppm.

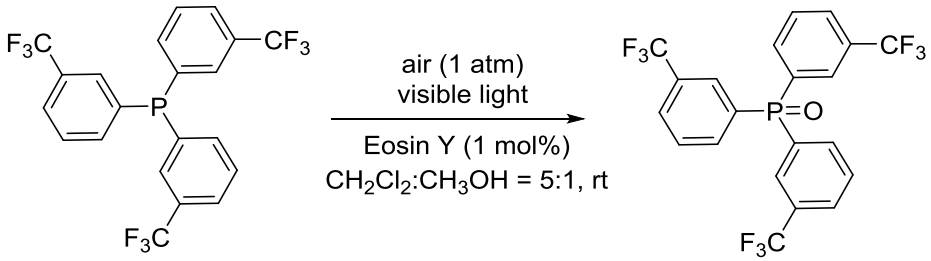
(10) Tris(4-chlorophenyl)phosphine oxide (2k)[13]



1k **2k**, 3 h, 92%

The reaction of **1k** (73 mg, 0.20 mmol), Eosin Y (2 mg, 0.0031 mmol), CH_2Cl_2 (2.5 mL), and CH_3OH (0.5 mL) afforded **2k** as a solid (70 mg, 92%); mp 174.3-174.7 °C (petroleum ether/ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62-7.54 (m, 6 H), 7.50-7.44 (m, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.1 (d, J_{PC} = 2.9 Hz), 133.3 (d, J_{PC} = 10.8 Hz), 130.2 (d, J_{PC} = 106.0 Hz), 129.1 (d, J_{PC} = 12.8 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 30.5 ppm.

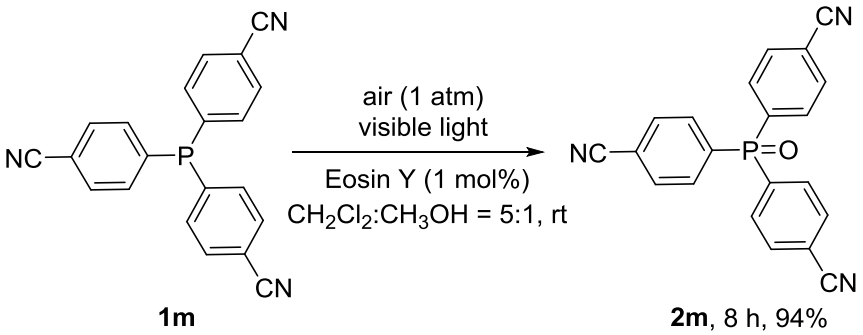
(11) Tris(3-(trifluoromethyl)phenyl)phosphine oxide (2l)[14]



1l **2l**, 8 h, 99%

The reaction of **1l** (95 mg, 0.20 mmol), Eosin Y (2 mg, 0.0031 mmol), CH_2Cl_2 (2.5 mL), and CH_3OH (0.5 mL) afforded **2l** as a solid (98 mg, 99%); mp 103.4-103.9 °C (petroleum ether/ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, J = 12.4 Hz, 3 H), 7.92-7.78 (m, 6 H), 7.73-7.65 (m, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.0 (d, J_{PC} = 10.0 Hz), 132.5 (d, J_{PC} = 104.6 Hz), 131.7 (dd, J = 33.3, 12.8 Hz), 129.6, 129.5, 128.8-128.5 (m), 123.3 (q, J_{FC} = 272.9 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -63.0; ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.6 ppm.

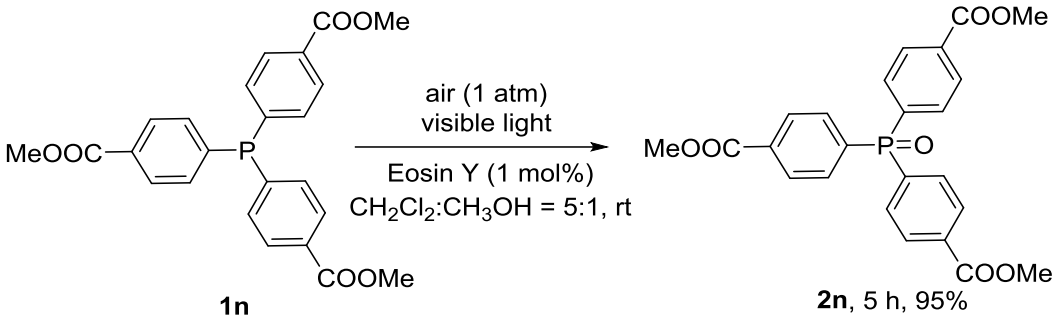
(12) Tris(4-cyanophenyl)phosphine oxide (2m)[15]



1m **2m**, 8 h, 94%

The reaction of **1m** (67 mg, 0.20 mmol), Eosin Y (2 mg, 0.0031 mmol), CH_2Cl_2 (2.5 mL), and CH_3OH (0.5 mL) afforded **2m** as a solid (66 mg, 94%); mp 198.3-198.7 °C (petroleum ether/ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90-7.73 (m, 12 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.4 (d, J_{PC} = 101.8 Hz), 132.5 (d, J_{PC} = 12.1 Hz), 132.4 (d, J_{PC} = 9.9 Hz), 117.3, 116.9; ^{31}P NMR (162 MHz, $CDCl_3$) δ 24.8 ppm.

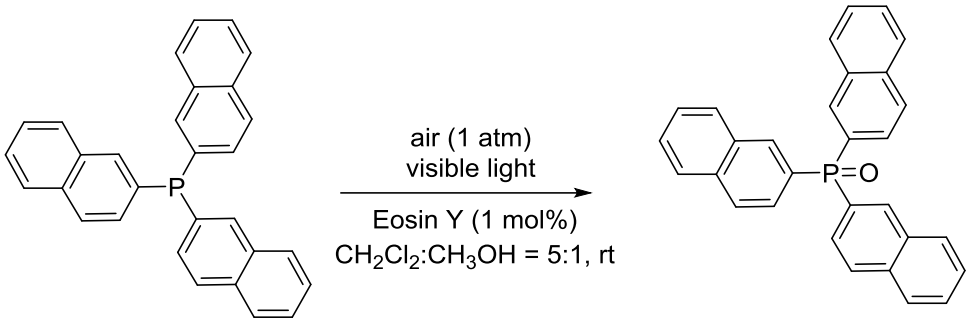
(13) Tris(4-(methoxycarbonyl)phenyl)phosphine oxide (2n)[16]



1n **2n**, 5 h, 95%

The reaction of **1n** (87 mg, 0.20 mmol), Eosin Y (2 mg, 0.0031 mmol), CH_2Cl_2 (2.5 mL), and CH_3OH (0.5 mL) afforded **2n** as a solid (86 mg, 95%); mp 173.6-174.1 °C (petroleum ether/ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (dd, J = 8.4, 2.8 Hz, 6 H), 7.75 (dd, J = 12.0, 8.4 Hz, 6 H), 3.95 (s, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.0, 136.1 (d, J_{PC} = 101.6 Hz), 133.7 (d, J_{PC} = 2.8 Hz), 132.0 (d, J_{PC} = 10.1 Hz), 129.7 (d, J_{PC} = 12.3 Hz), 52.6; ^{31}P NMR (162 MHz, $CDCl_3$) δ 26.9 ppm.

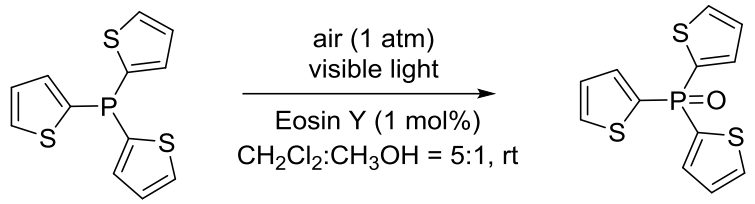
(14) Tris(naphthalen-2-yl)phosphine oxide (2o)[1]



1o **2o**, 3.5 h, 90%

The reaction of **1o** (83 mg, 0.20 mmol), Eosin Y (2 mg, 0.0031 mmol), CH_2Cl_2 (2.5 mL), and CH_3OH (0.5 mL) afforded **2o** as a solid (77 mg, 90%); mp 248.3-248.7 °C (petroleum ether/ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.37 (d, J = 14.0 Hz, 3 H), 7.96-7.84 (m, 9 H), 7.78-7.71 (m, 3 H), 7.63-7.51 (m, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.7, 134.1 (d, J_{PC} = 9.4 Hz), 132.4 (d, J_{PC} = 13.3 Hz), 129.6 (d, J_{PC} = 104.1 Hz), 128.9, 128.4 (d, J_{PC} = 1.2 Hz), 128.2, 127.8, 126.9, 126.8; ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.8 ppm.

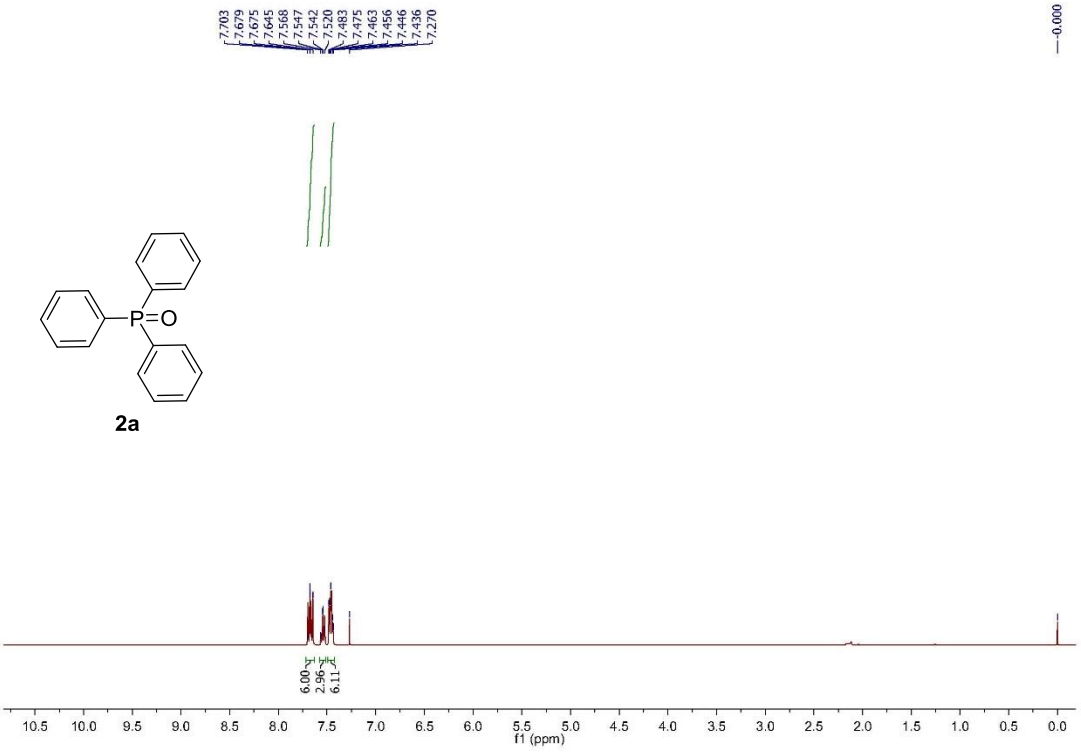
(15) Tris(thiophen-2-yl)phosphine oxide (2p)[17]



1p **2p**, 3.5 h, 90%

The reaction of **1p** (56 mg, 0.20 mmol), Eosin Y (2 mg, 0.0031 mmol), CH_2Cl_2 (2.5 mL), and CH_3OH (0.5 mL) afforded **2p** as a solid (53 mg, 90%); mp 128.4-128.8 °C (petroleum ether/ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79-7.74 (m, 3 H), 7.63-7.57 (m, 3 H), 7.23-7.18 (m, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.7 (d, J_{PC} = 11.3 Hz), 134.6 (d, J_{PC} = 127.2 Hz), 134.2 (d, J_{PC} = 5.6 Hz), 128.2 (d, J_{PC} = 15.0 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 6.6 ppm.

NMR Spectra


7.703
7.679
7.675
7.645
7.568
7.547
7.542
7.520
7.483
7.475
7.463
7.456
7.446
7.436
7.270
0.000
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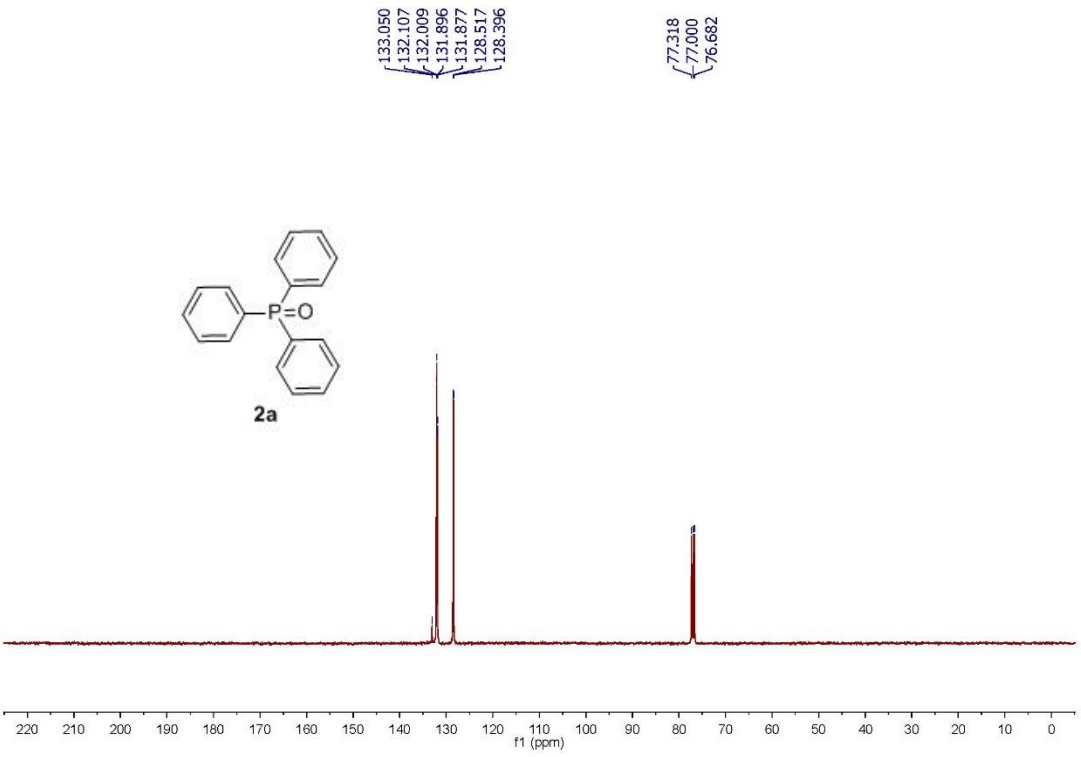
2a
6.00
2.96
6.11
f1 (ppm)


133.050
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128.396
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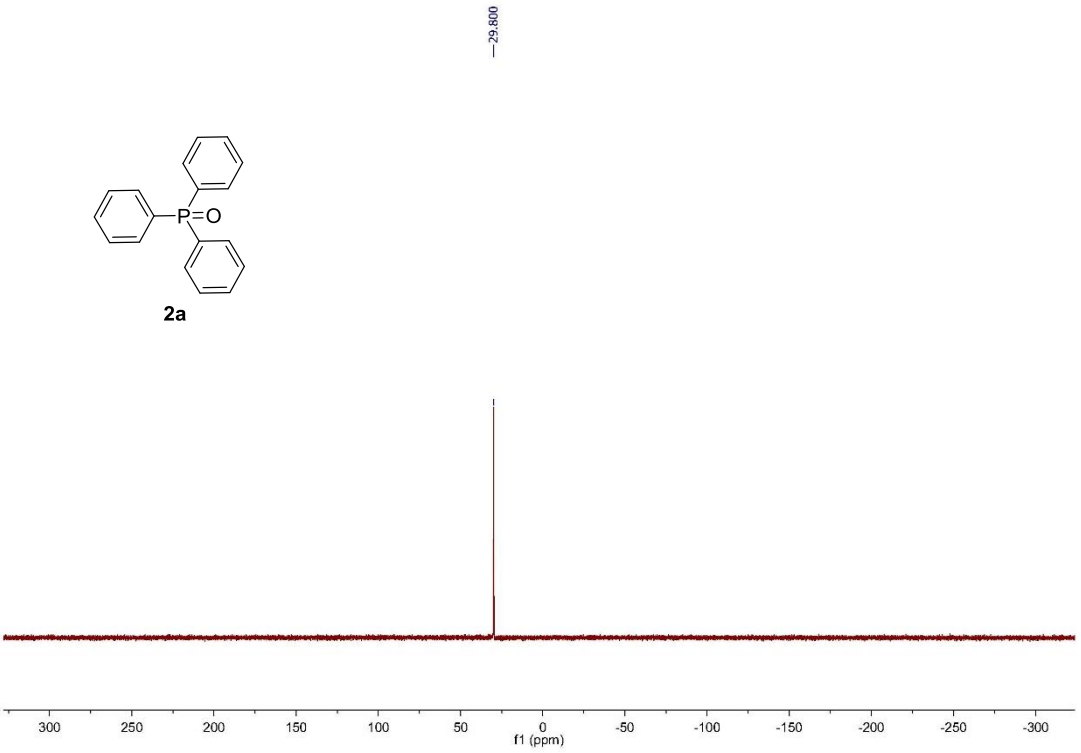
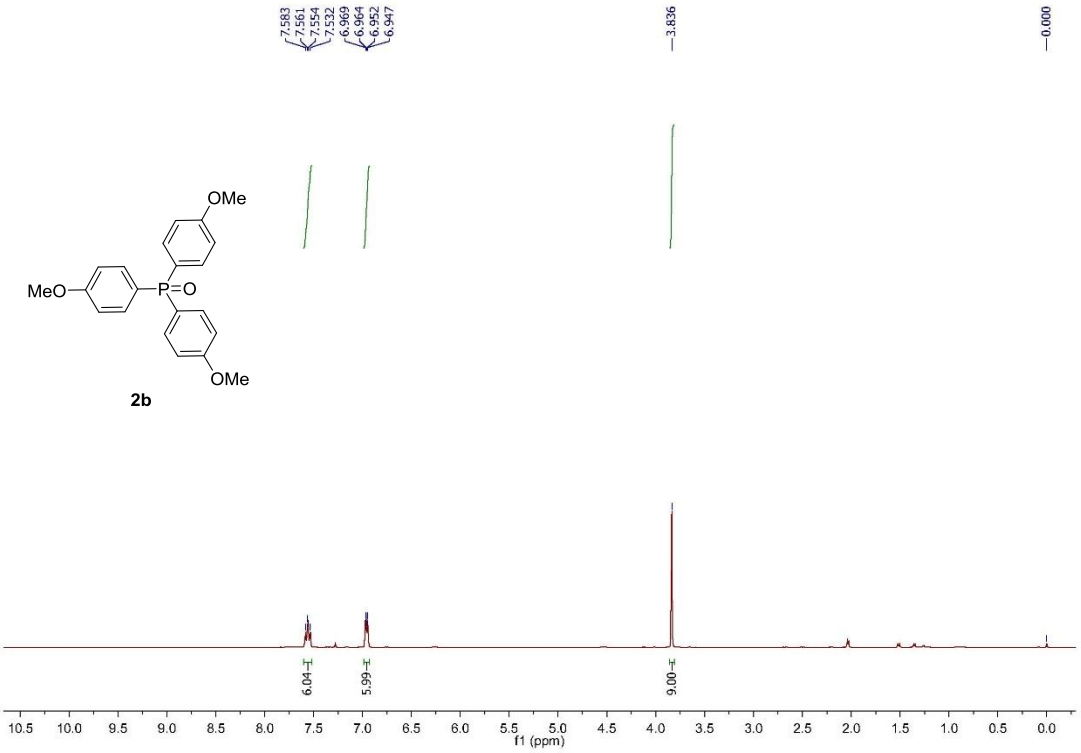
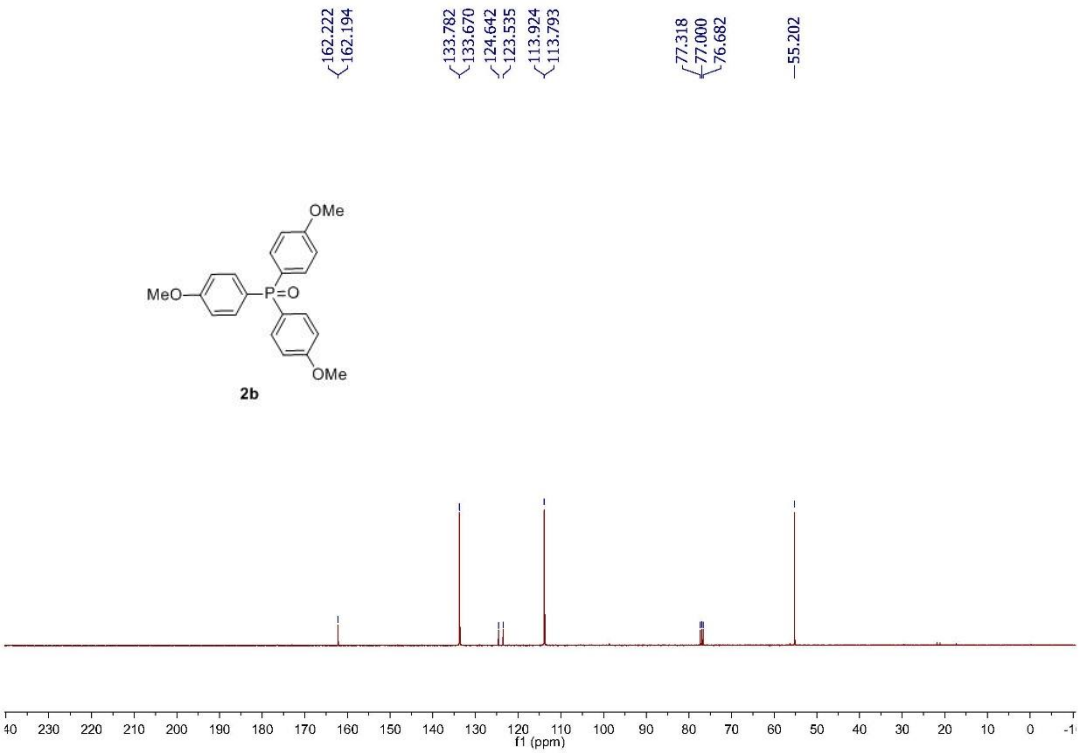
77.000
76.682
P=O
2a
f1 (ppm)

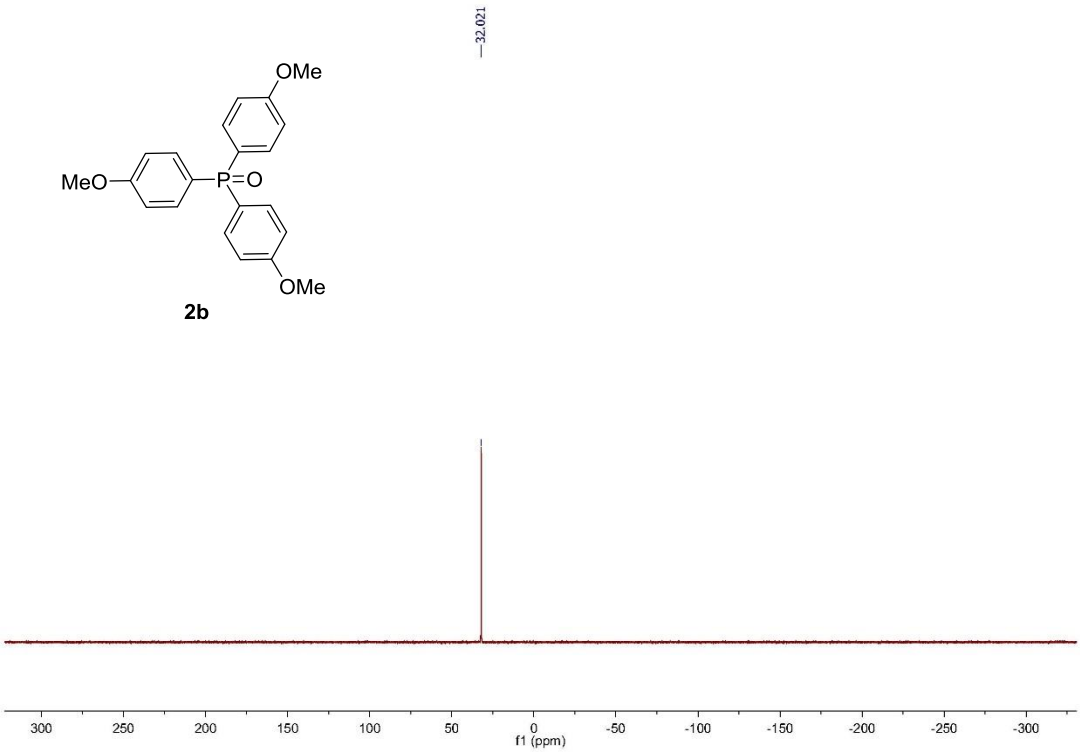

29.800
P=O
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)

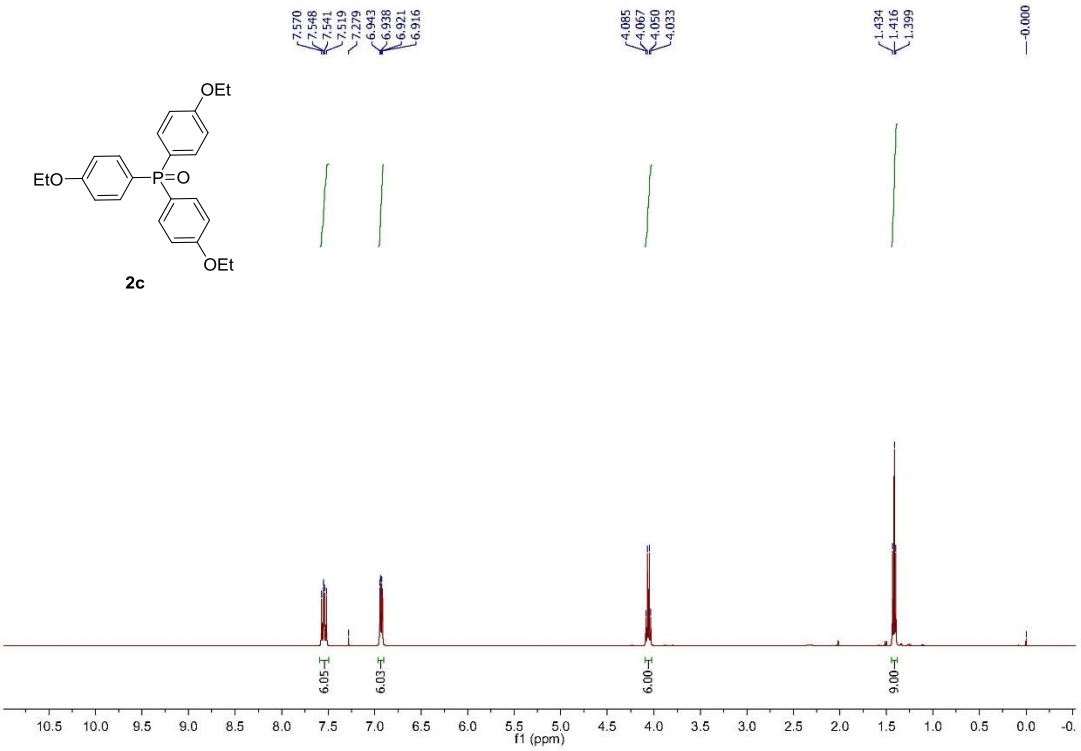
2a

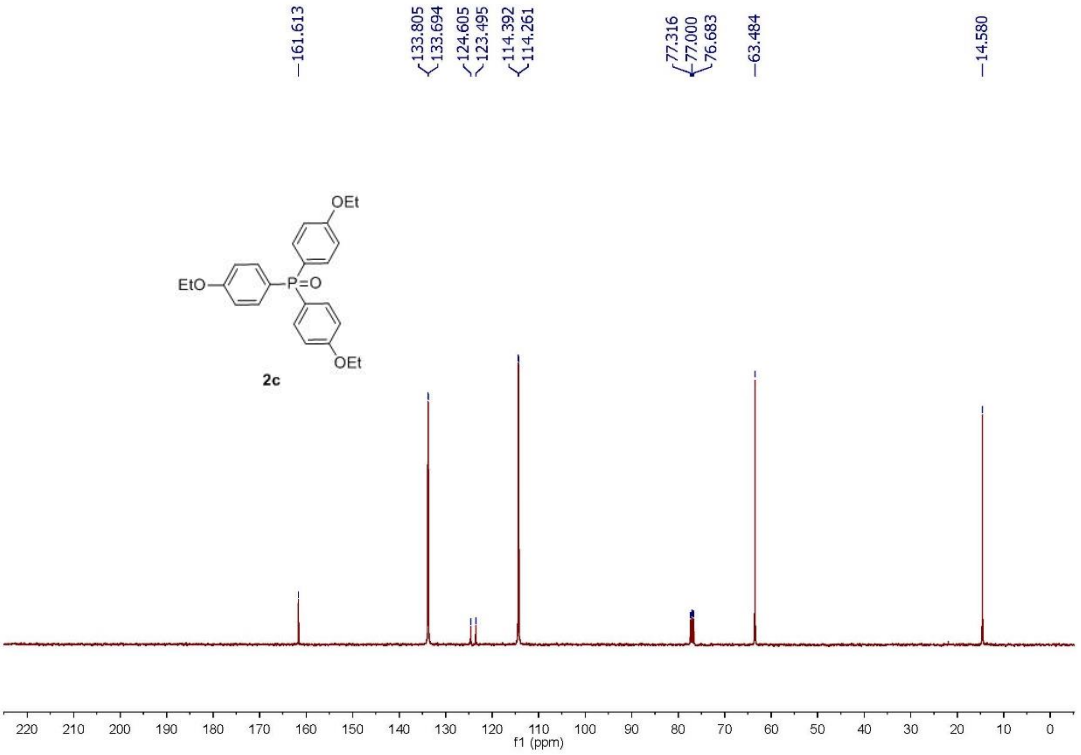

7.583
7.561
7.554
7.532
6.969
6.964
6.952
6.947
3.836
0.000
OMe
MeO
P=O
OMe
6.04
5.99
9.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

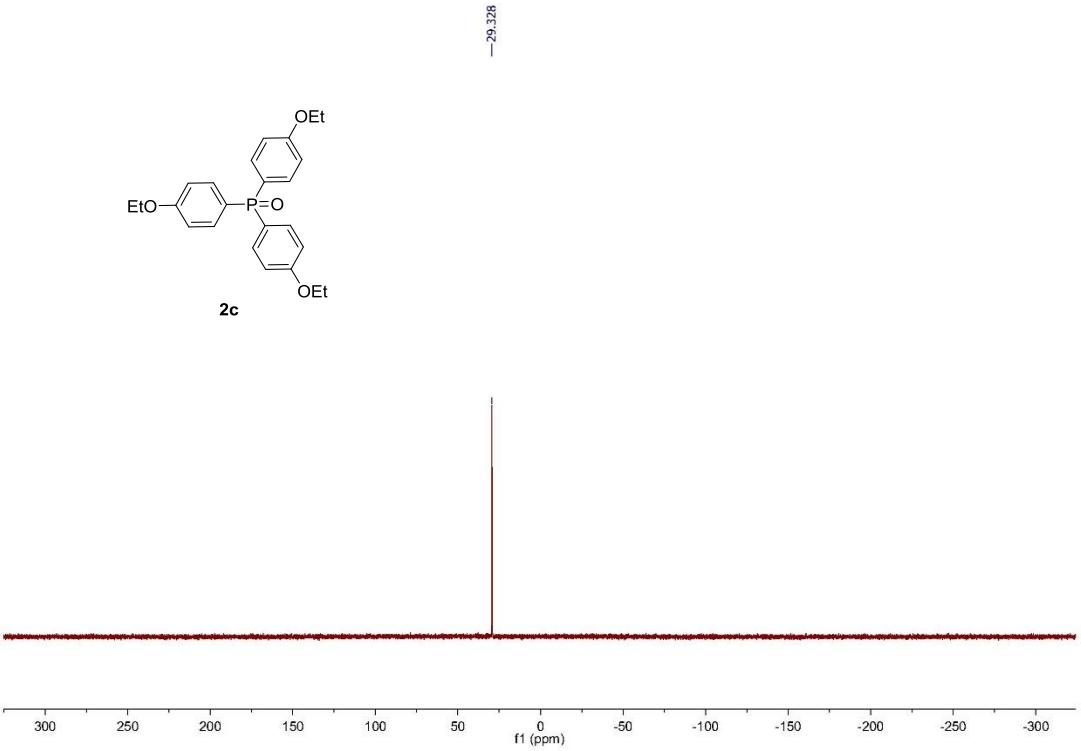
2b

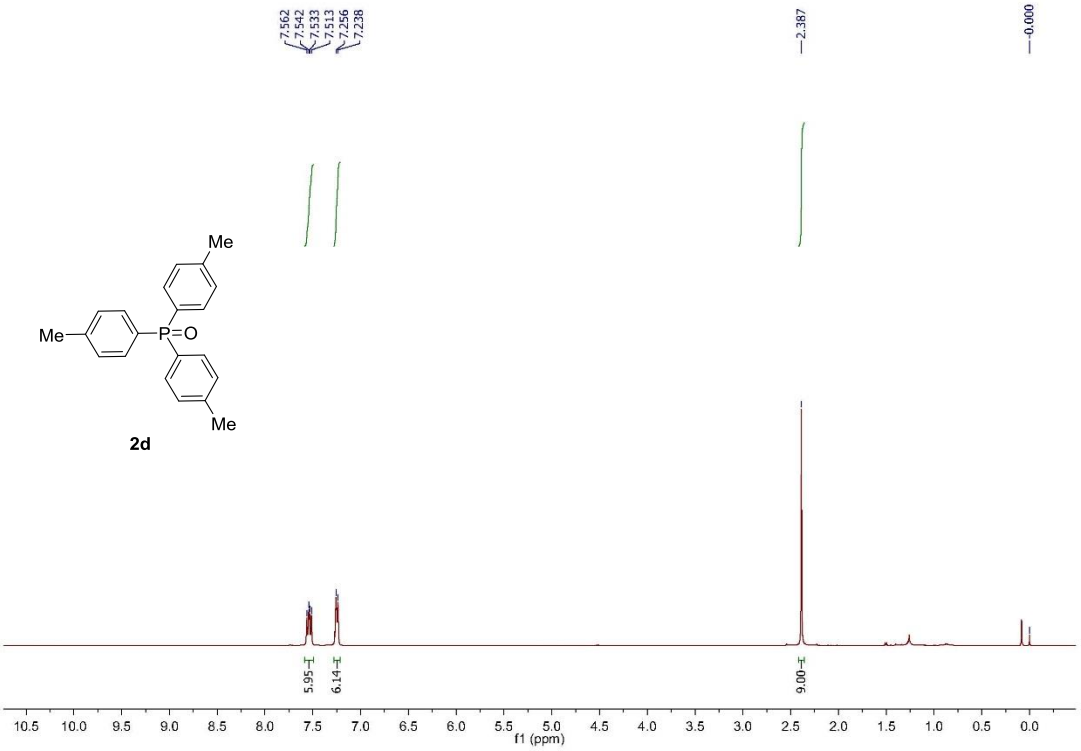

162.222
162.194
133.782
133.670
124.642
123.535
113.924
113.793
77.318
77.000
76.682
55.202
OMe
MeO
P=O
OMe
2b
f1 (ppm)

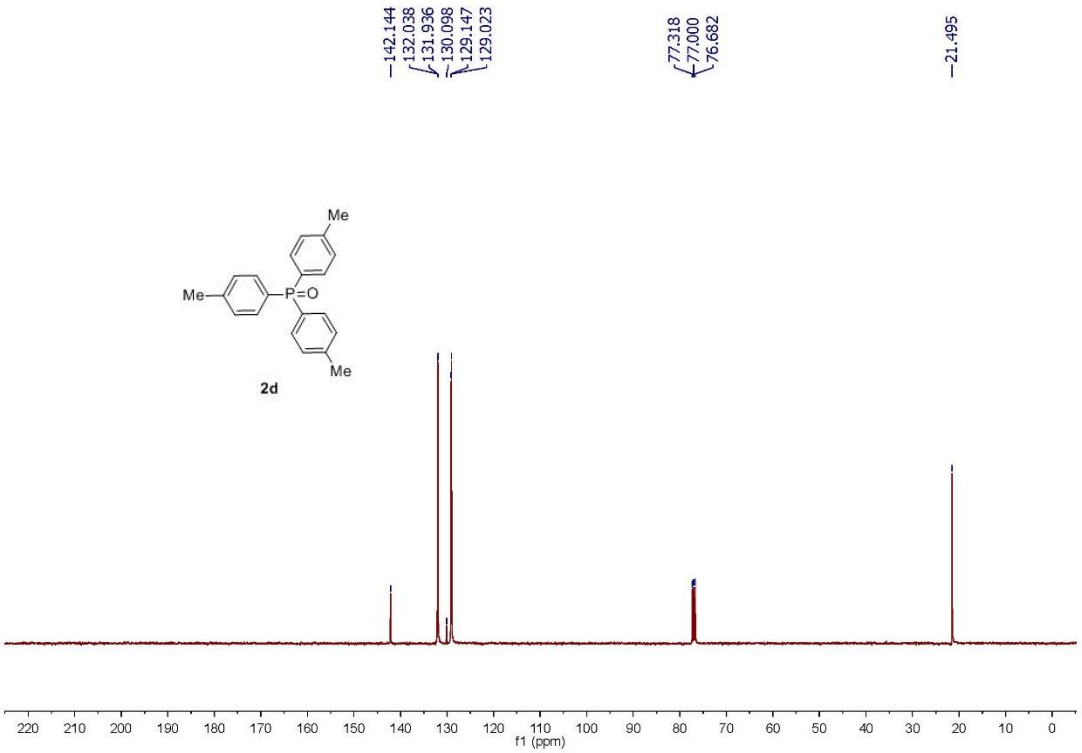

32.021
OMe
MeO
P=O
OMe
2b
f1 (ppm)

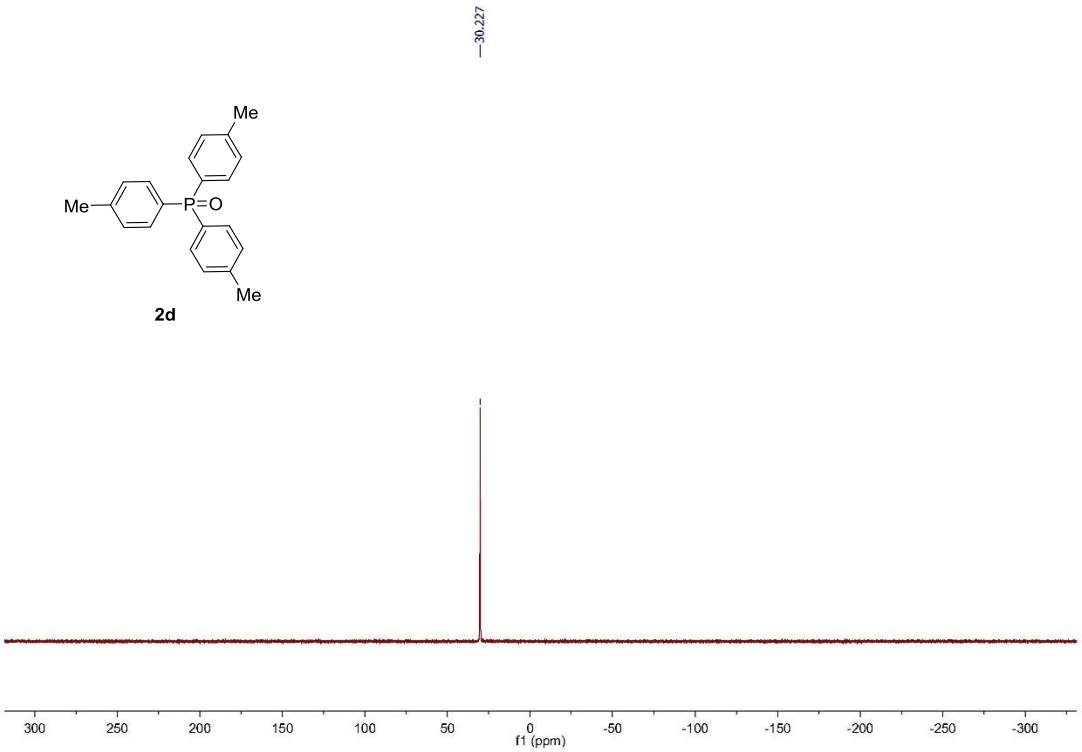

7.570
7.548
7.541
7.519
7.279
6.943
6.938
6.921
6.916
4.085
4.067
4.050
4.033
1.434
1.416
1.399
0.000
OEt
EtO
P=O
OEt
2c
6.05
6.03
6.00
9.00
f1 (ppm)

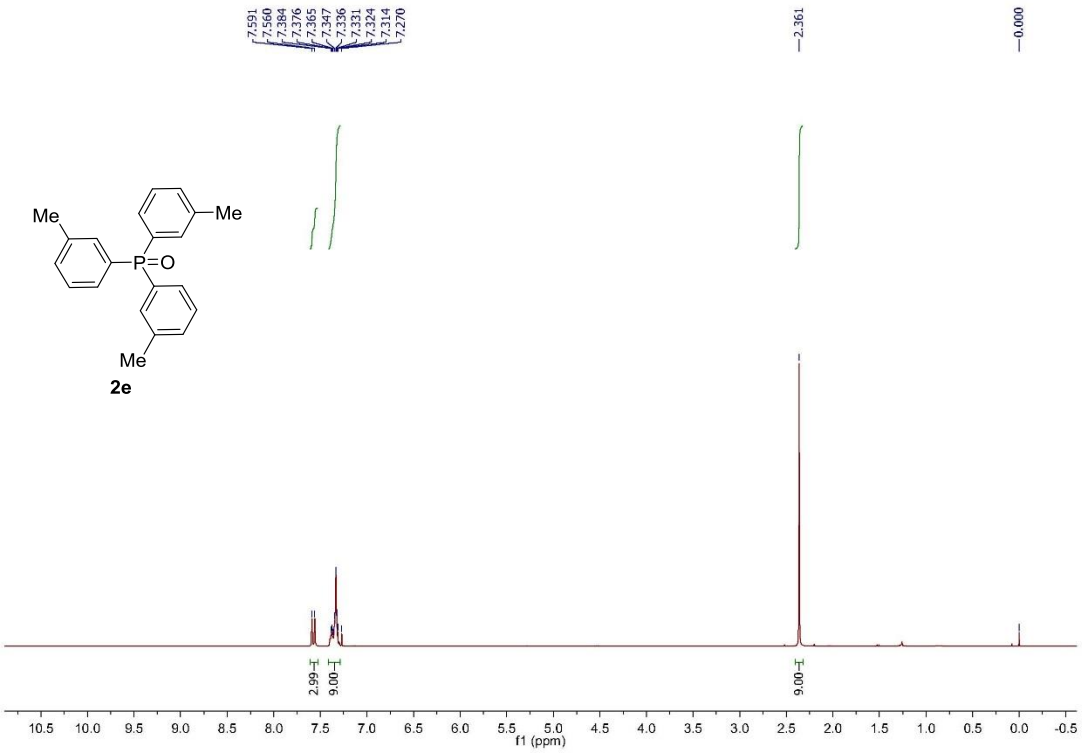

161.613
133.805
133.694
124.605
123.495
114.392
114.261
77.316
77.000
76.683
63.484
14.580
OEt
EtO
P=O
OEt
2c
f1 (ppm)

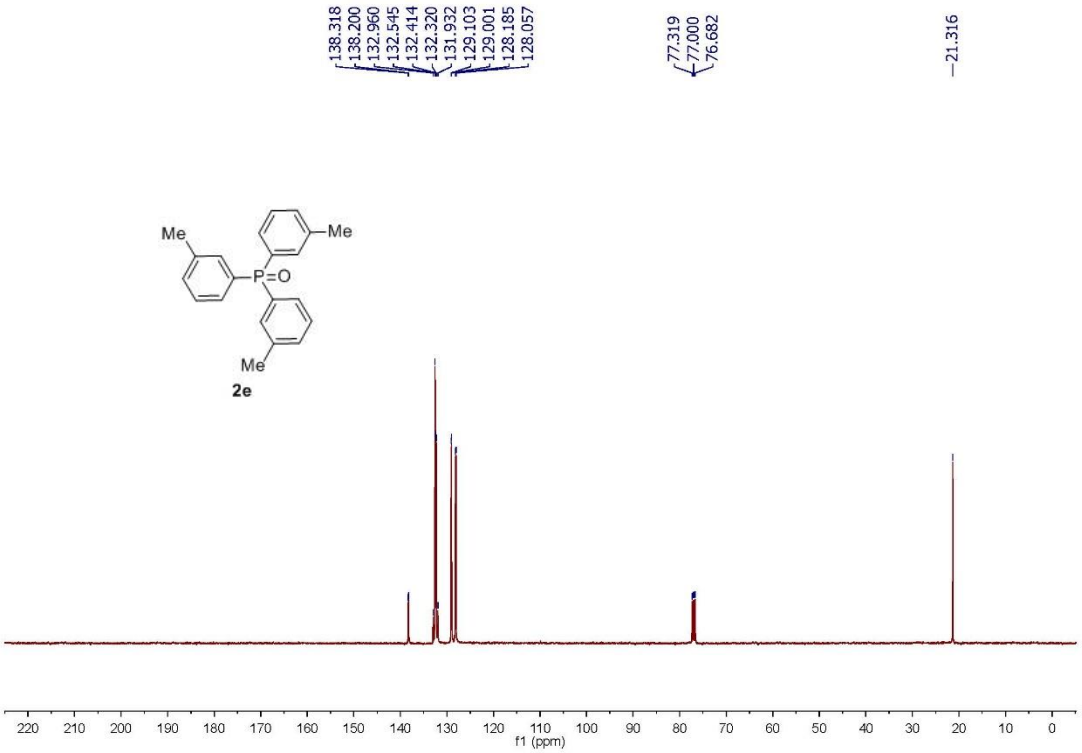

29.328
OEt
EtO
P=O
OEt
2c
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)

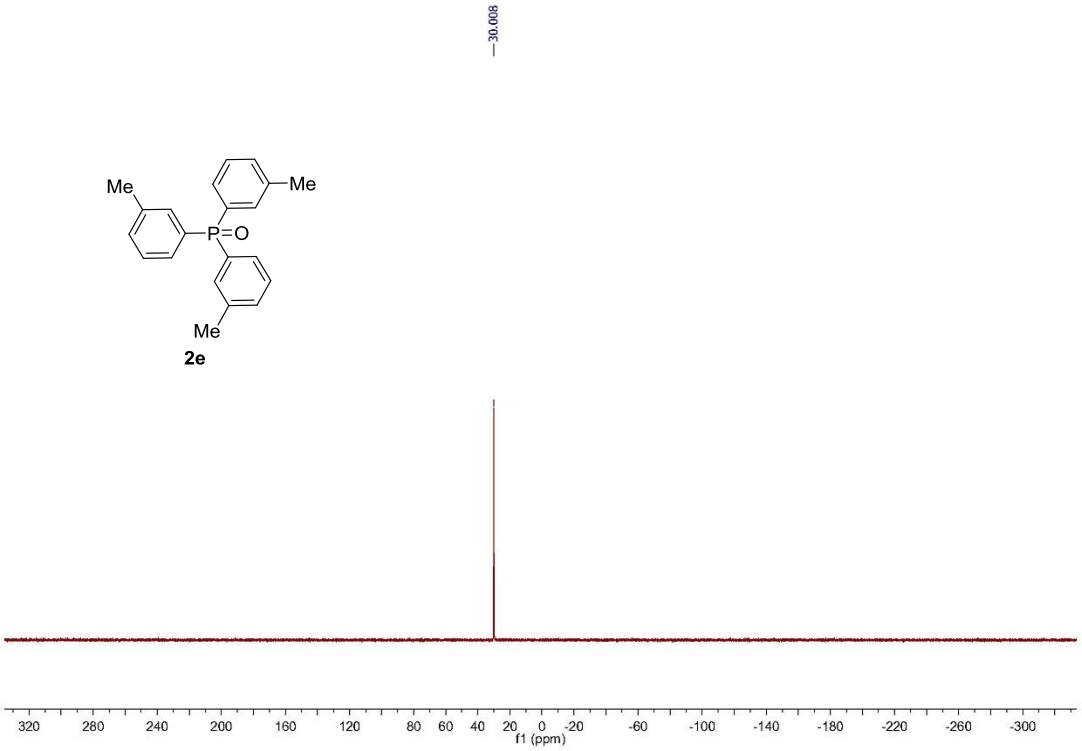

7.562
7.542
7.533
7.513
7.256
7.238
2.387
0.000
Me
Me
P=O
Me
2d
5.95
6.14
9.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

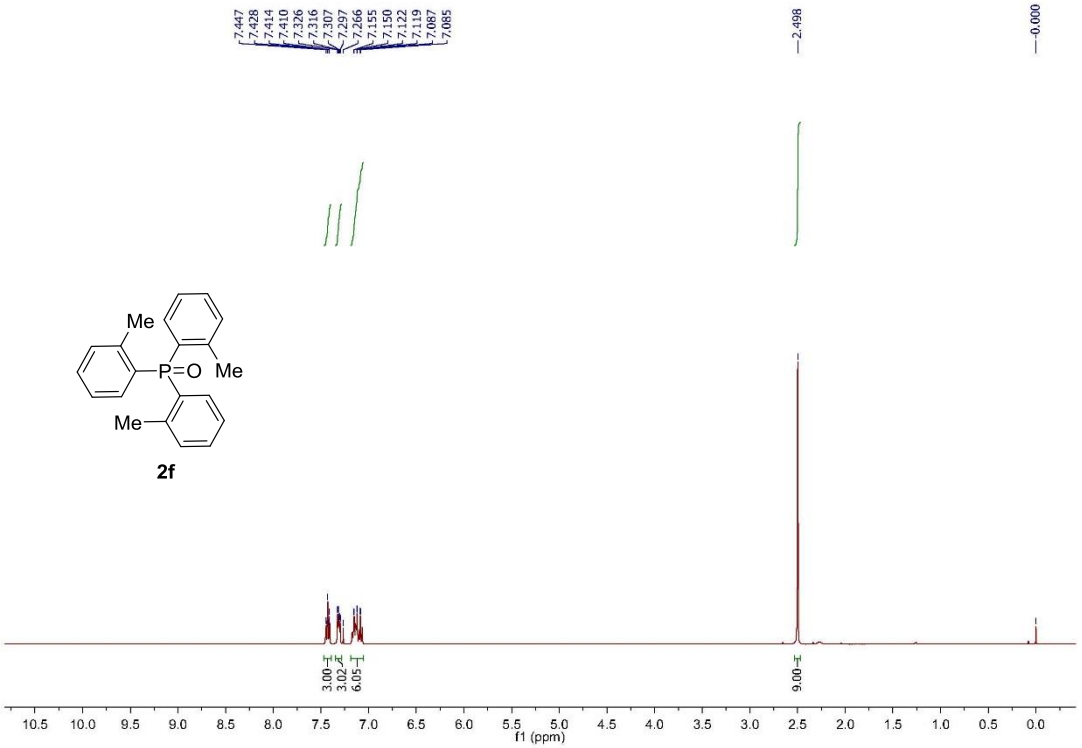

142.144
132.038
131.936
130.098
129.147
129.023
77.318
77.000
76.682
21.495
Me
Me
P=O
Me
2d
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

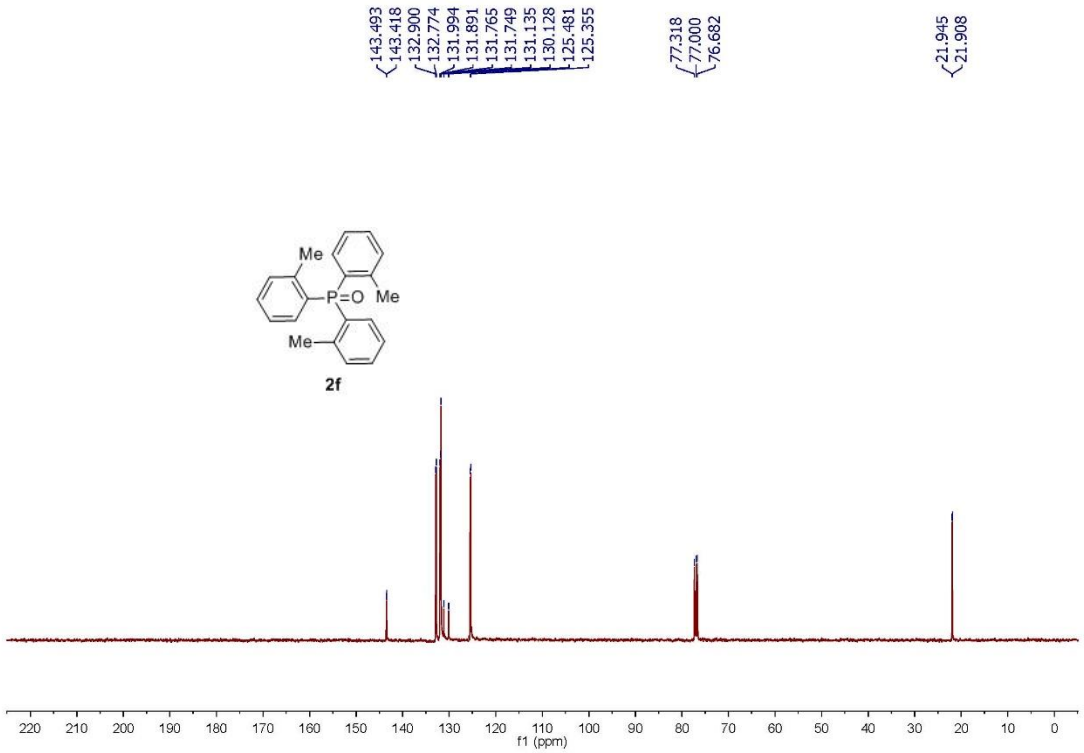

30.227
Me
Me
P=O
Me
2d
300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300
f1 (ppm)

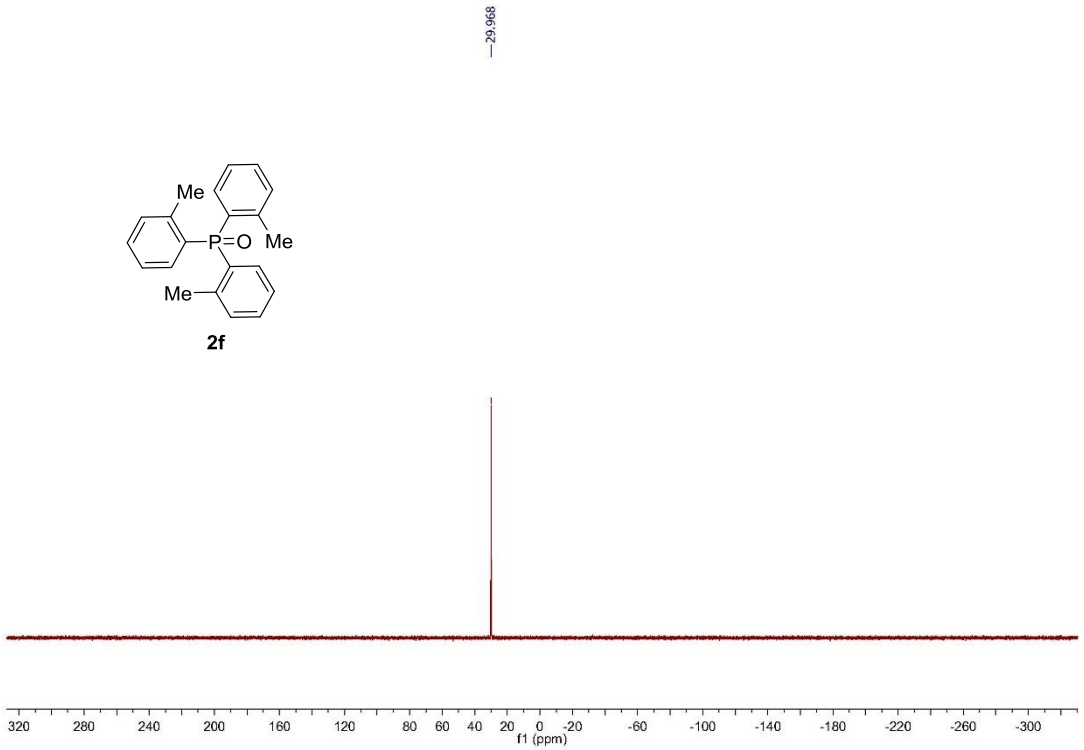

7.591
7.560
7.384
7.376
7.365
7.347
7.336
7.331
7.324
7.314
7.270
2.361
0.000
Me
Me
P=O
Me
2e
2.99
9.00
9.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

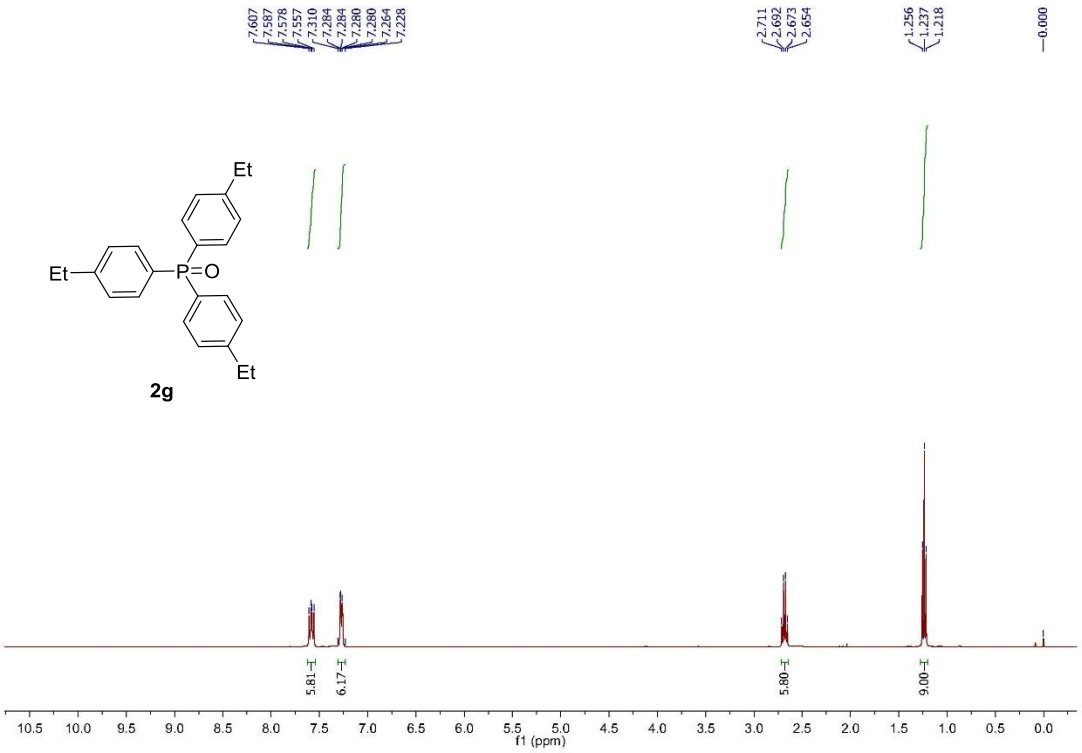

138.318
138.200
132.960
132.545
132.414
132.320
131.932
129.103
129.001
128.185
128.057
77.319
77.000
76.682
21.316
Me
Me
P=O
Me
2e
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

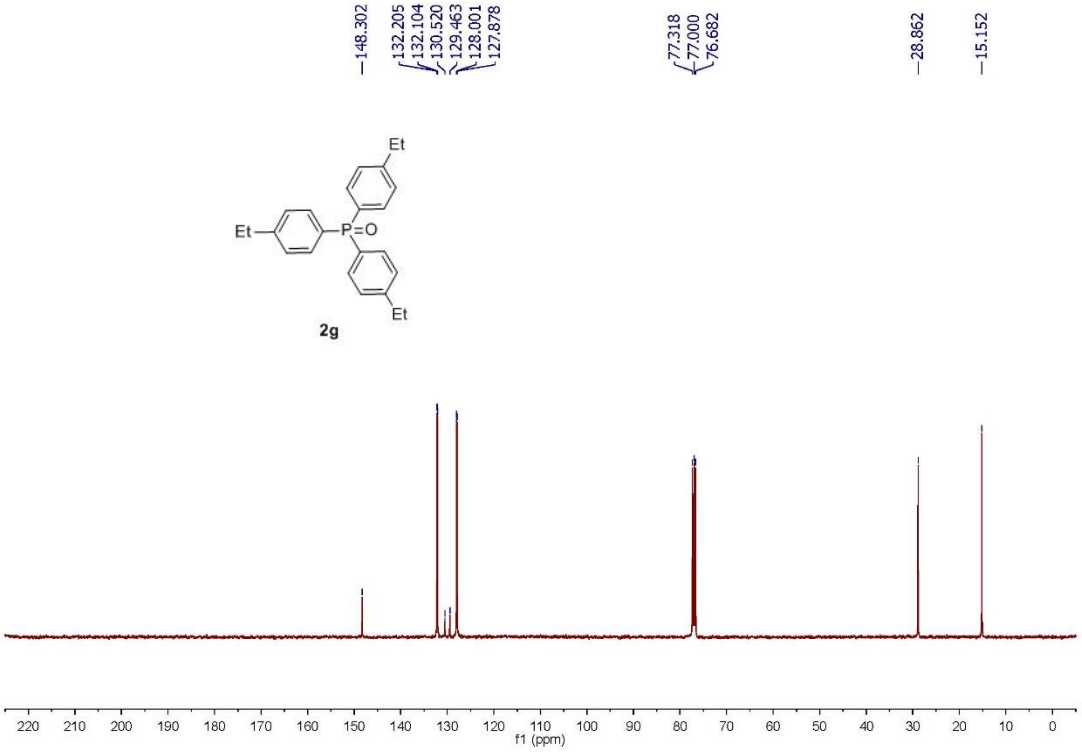

30.008
Me
Me
P=O
Me
2e
320
280
240
200
160
120
80
60
40
20
0
-20
-60
-100
-140
-180
-220
-260
-300
f1 (ppm)

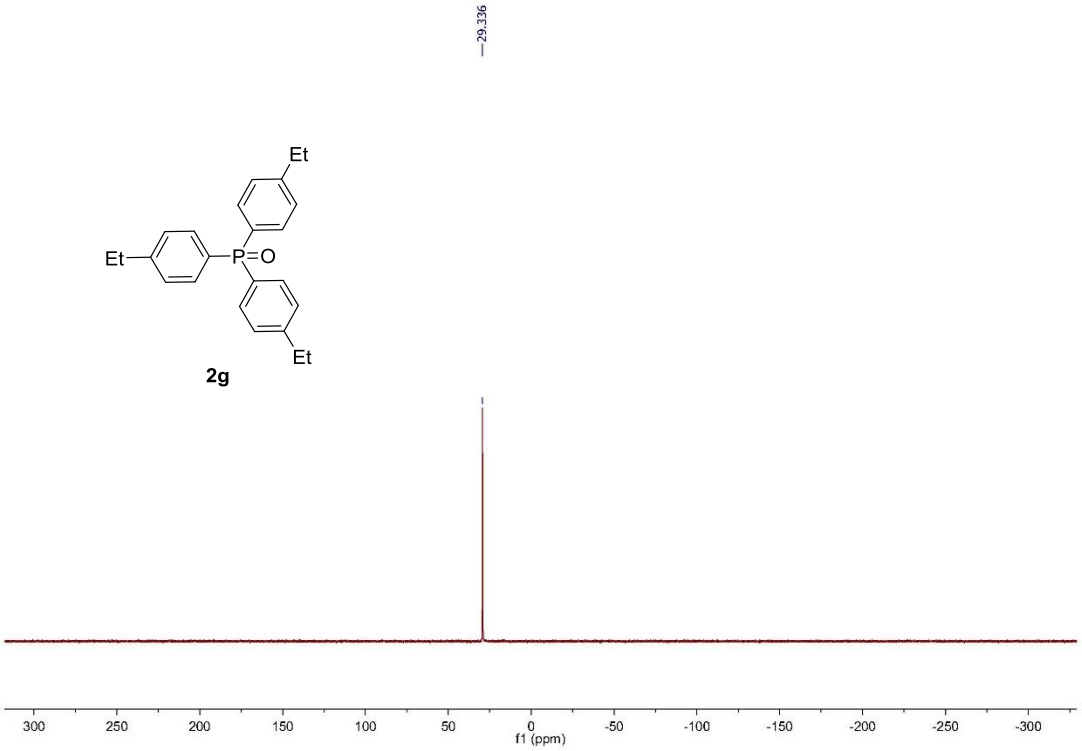

7.447
7.428
7.414
7.410
7.326
7.316
7.307
7.297
7.266
7.155
7.150
7.122
7.119
7.087
7.085
2.498
0.000
Me
P=O
Me
Me
2f
3.00
3.02
6.05
9.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


143.493
143.418
132.900
132.774
131.994
131.891
131.765
131.749
131.135
130.128
125.481
125.355
77.318
77.000
76.682
21.945
21.908
Me
P=O
Me
Me
2f
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

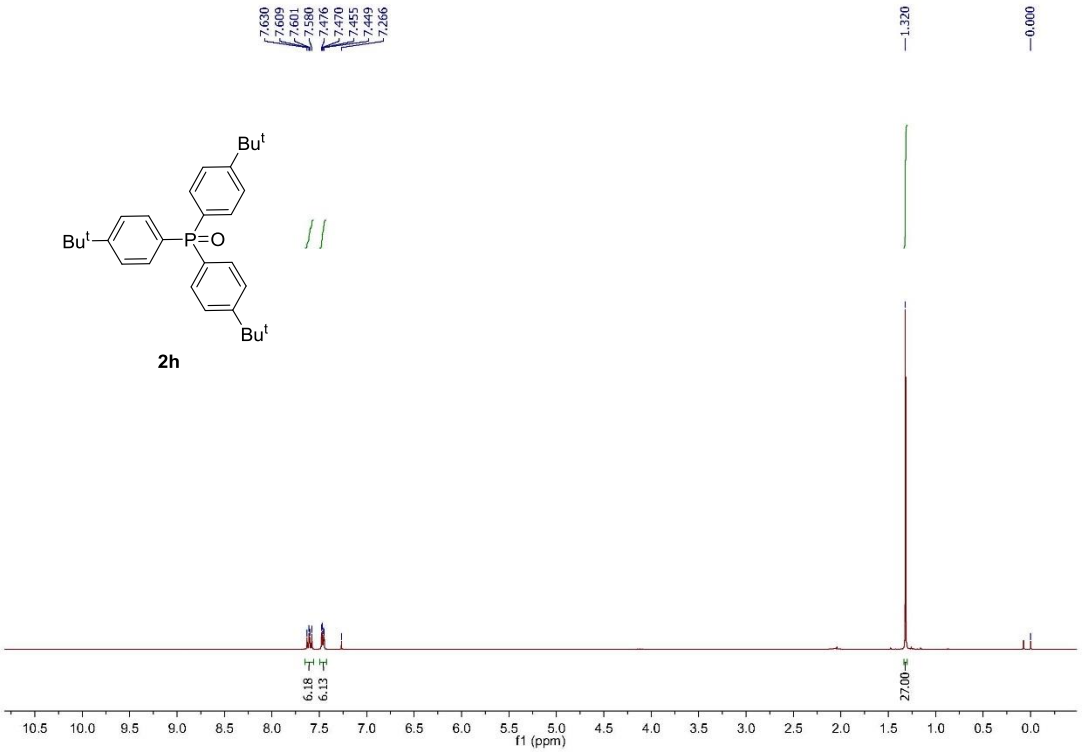

29.968
Me
P=O
Me
Me
2f
320 280 240 200 160 120 80 60 40 20 0 -20 -60 -100 -140 -180 -220 -260 -300
f1 (ppm)


7.607
7.587
7.578
7.557
7.310
7.284
7.284
7.280
7.280
7.264
7.228
2.711
2.692
2.673
2.654
1.256
1.237
1.218
0.000
Et
Et
P=O
Et
2g
5.81
6.17
5.80
9.00
f1 (ppm)

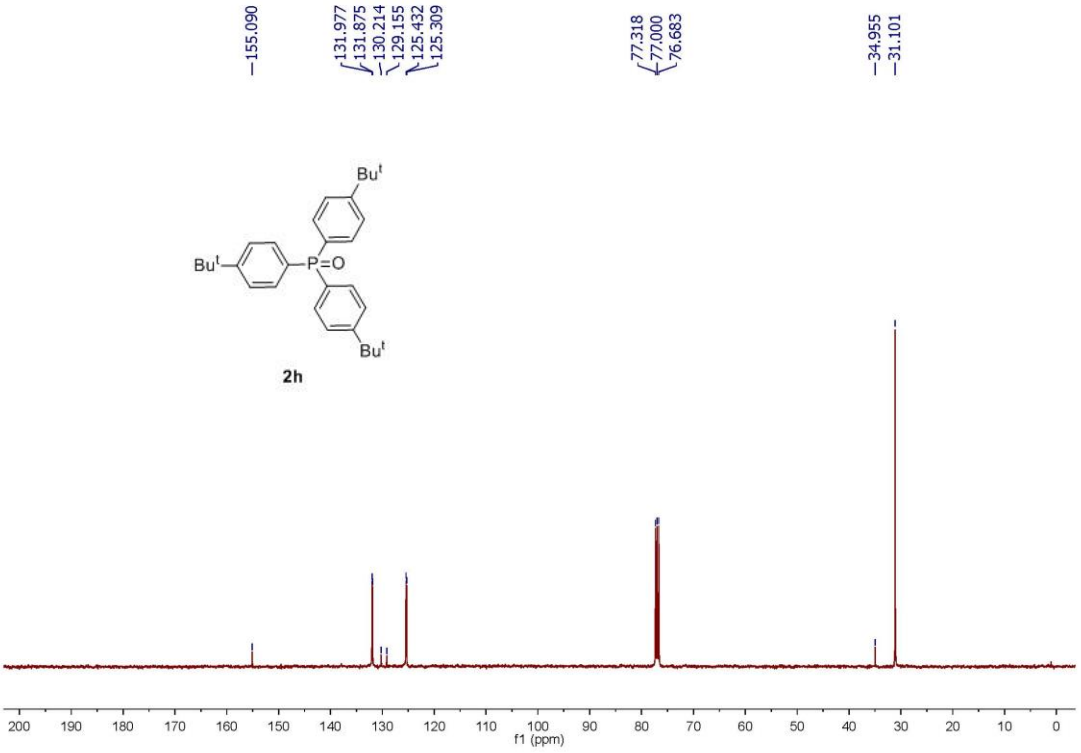

148.302
132.205
132.104
130.520
129.463
128.001
127.878
77.318
77.000
76.682
28.862
15.152
Et
Et
P=O
Et
2g
f1 (ppm)

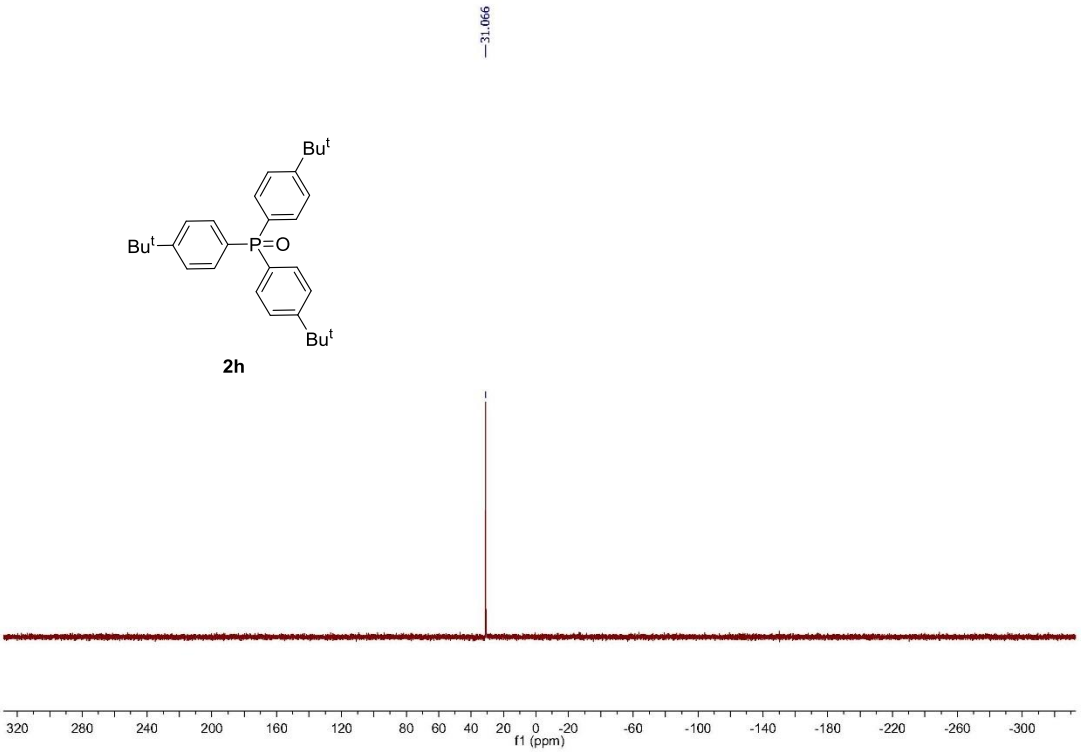

29.336
Et
Et
P=O
Et
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)

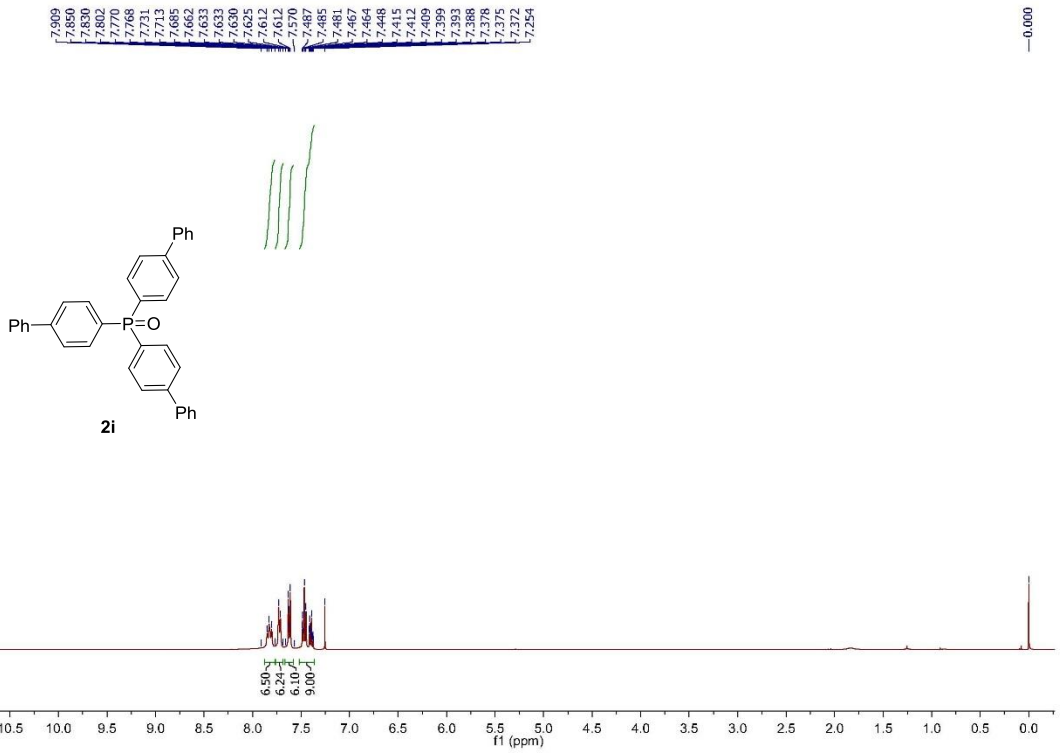
2g

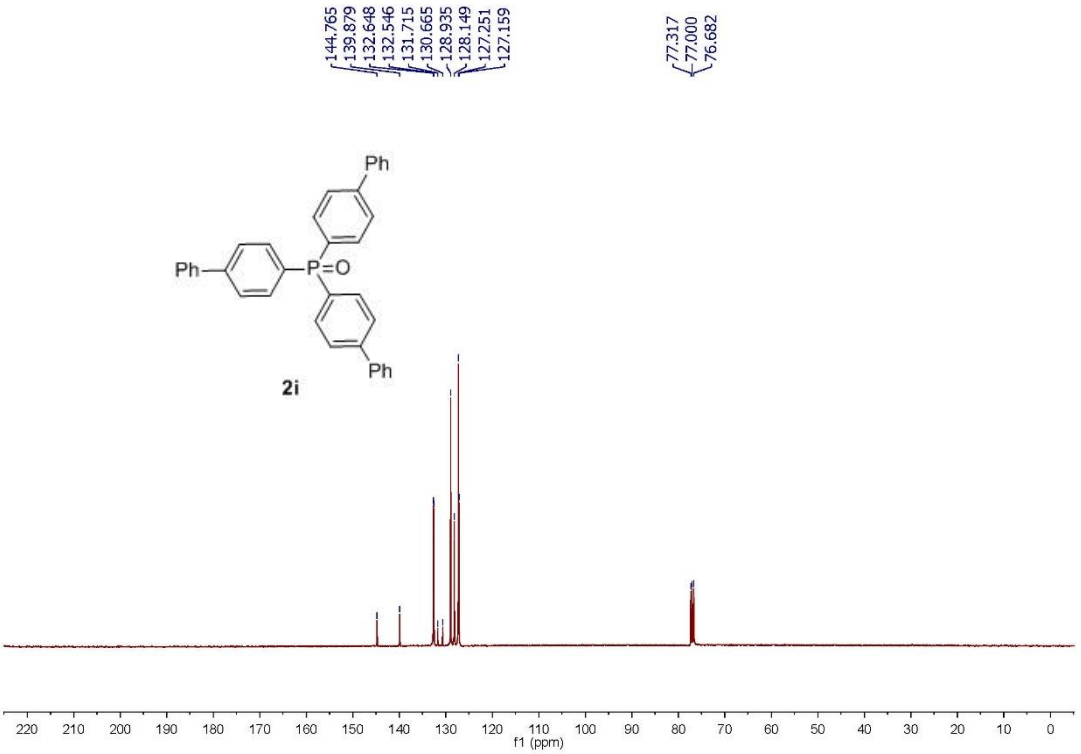

7.630
7.609
7.601
7.580
7.476
7.470
7.455
7.449
7.266
1.320
0.000
Bu^t
Bu^t
P=O
Bu^t
6.18
6.13
27.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

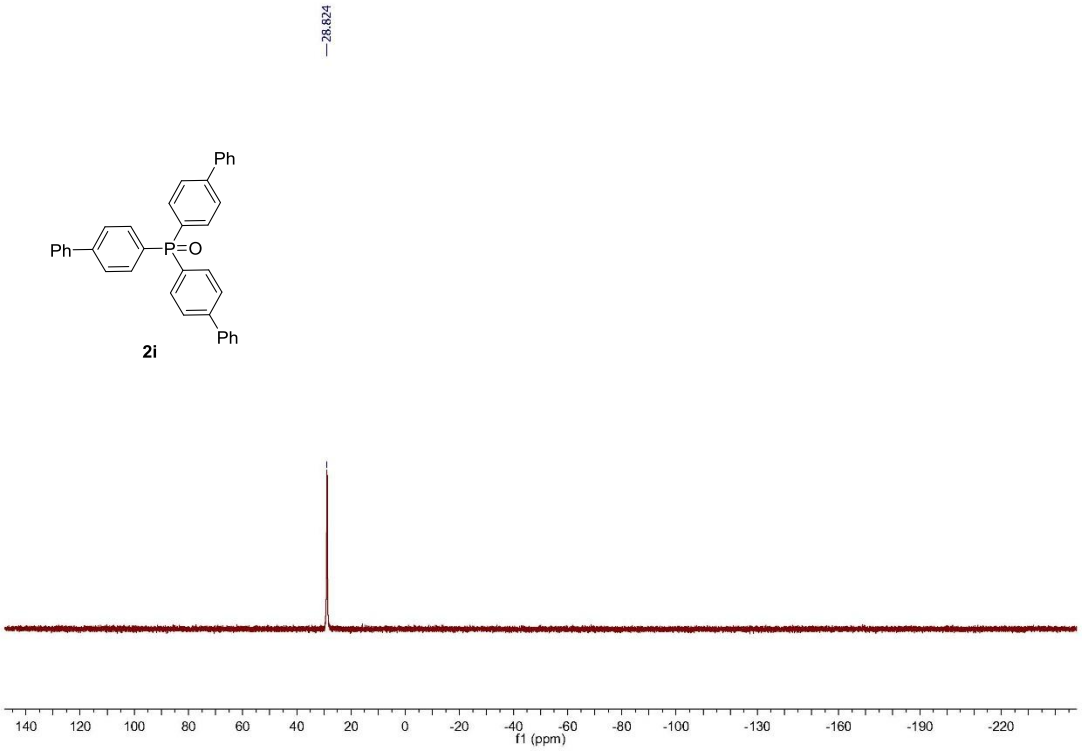
2h

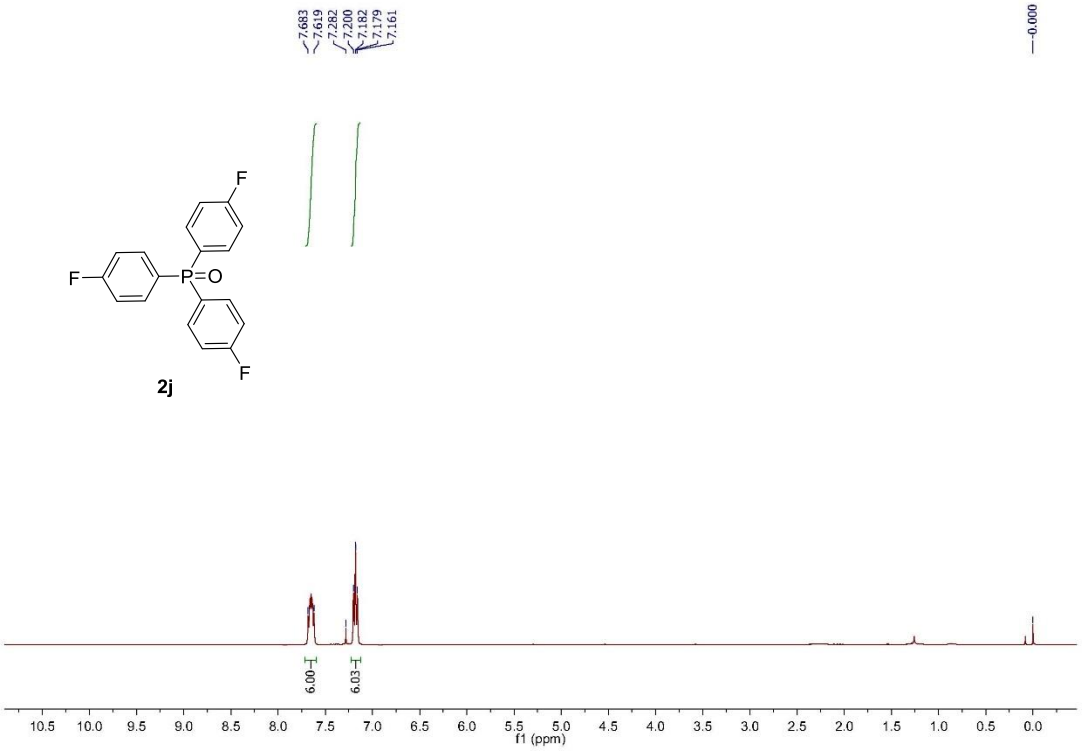

155.090
131.977
131.875
130.214
129.155
125.432
125.309
77.318
77.000
76.683
34.955
31.101
Bu^t
Bu^t
P=O
Bu^t
2h
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

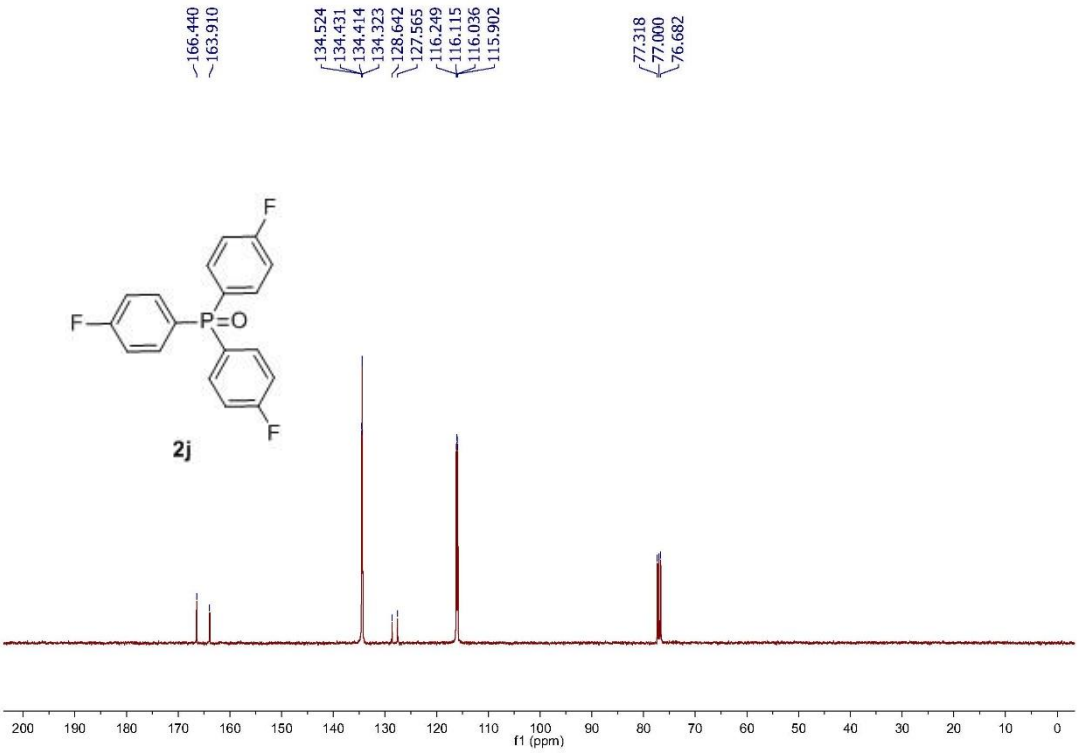

31.066
Bu^t
Bu^t
P=O
Bu^t
2h
320 280 240 200 160 120 80 60 40 20 0 -20 -60 -100 -140 -180 -220 -260 -300
f1 (ppm)

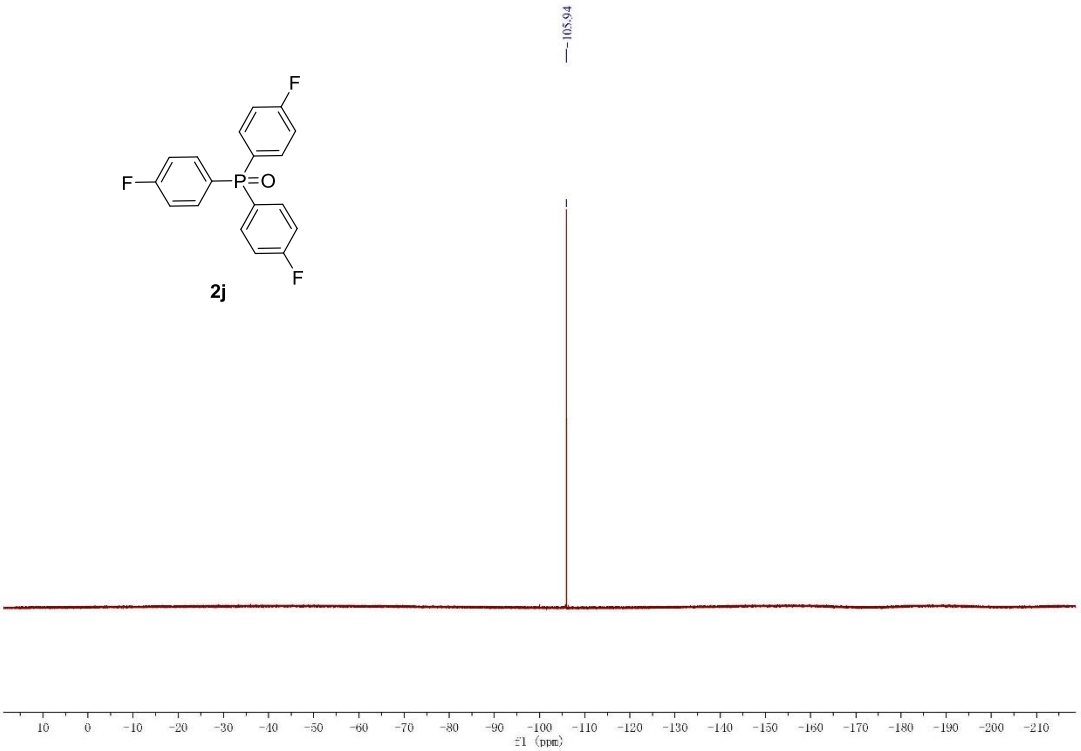

7.909
7.850
7.830
7.802
7.770
7.768
7.731
7.713
7.685
7.662
7.633
7.630
7.625
7.612
7.612
7.570
7.487
7.485
7.481
7.467
7.464
7.448
7.415
7.412
7.409
7.399
7.393
7.388
7.378
7.375
7.372
7.254
0.000
Ph
Ph
P=O
Ph
2i
6.50
6.24
6.10
9.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

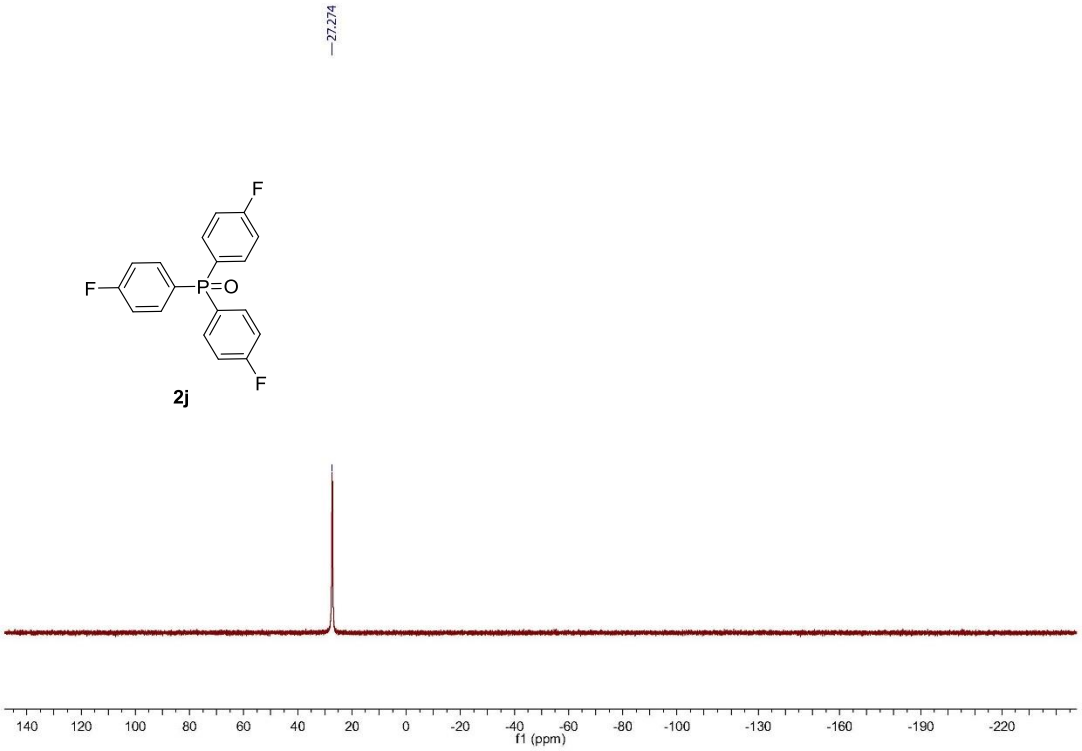

144.765
139.879
132.648
132.546
131.715
130.665
128.935
128.149
127.251
127.159
77.317
77.000
76.682
Ph
Ph
P=O
Ph
2i
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

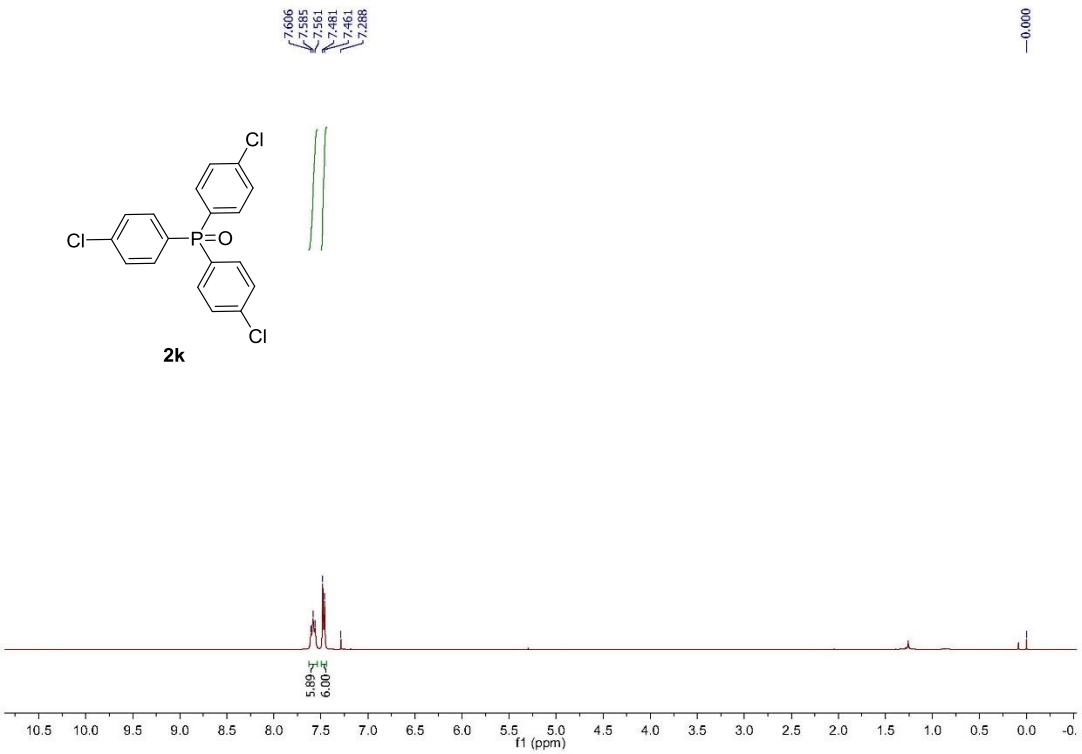

28.824
Ph
Ph
P=O
Ph
2i
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160 -190 -220
f1 (ppm)

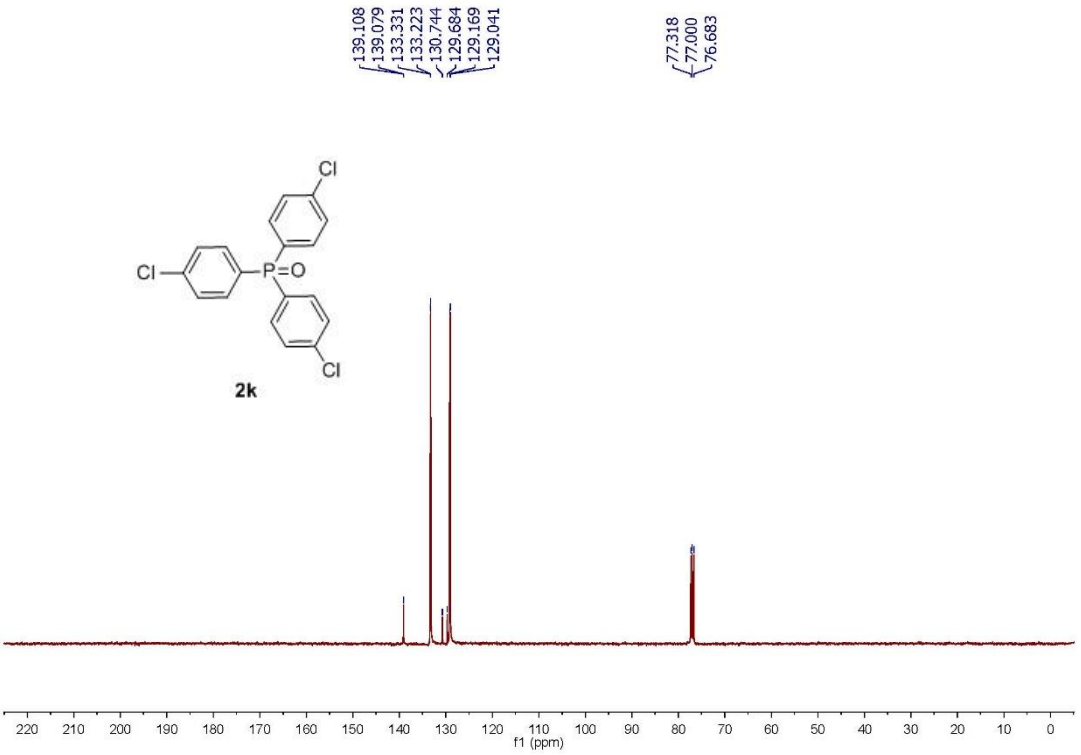

7.683
7.619
7.282
7.200
7.182
7.179
7.161
0.000
F
F
P=O
F
2j
6.00
6.03
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

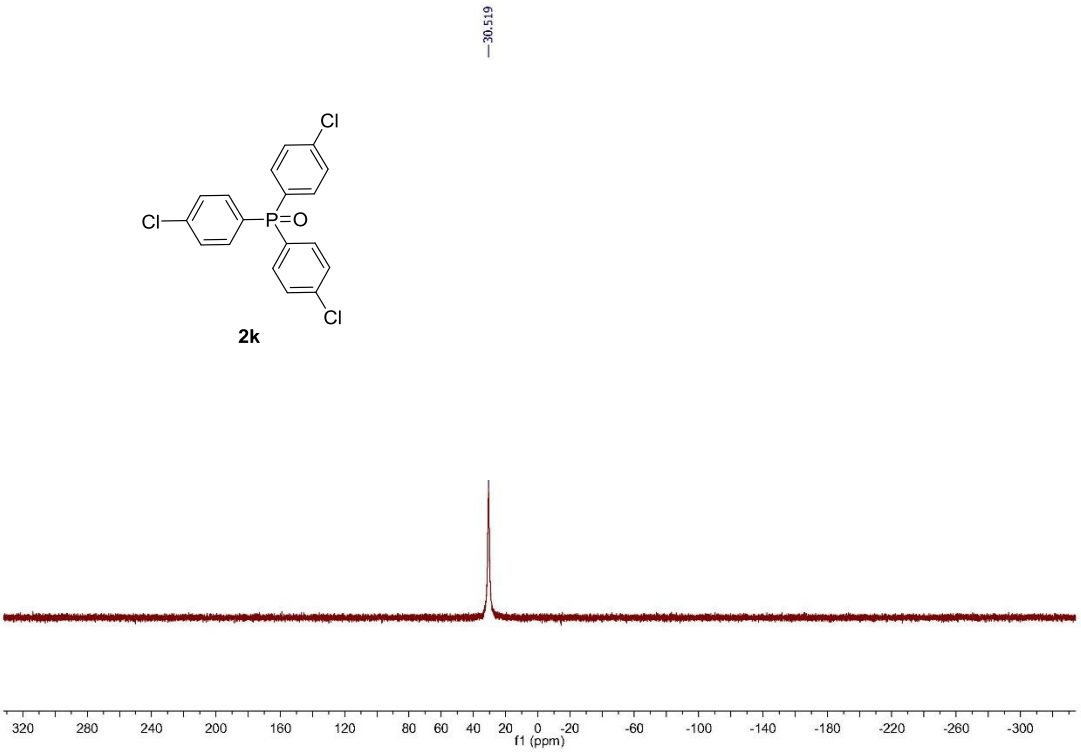

166.440
163.910
134.524
134.431
134.414
134.323
128.642
127.565
116.249
116.115
116.036
115.902
77.318
77.000
76.682
F
F
P=O
F
2j
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

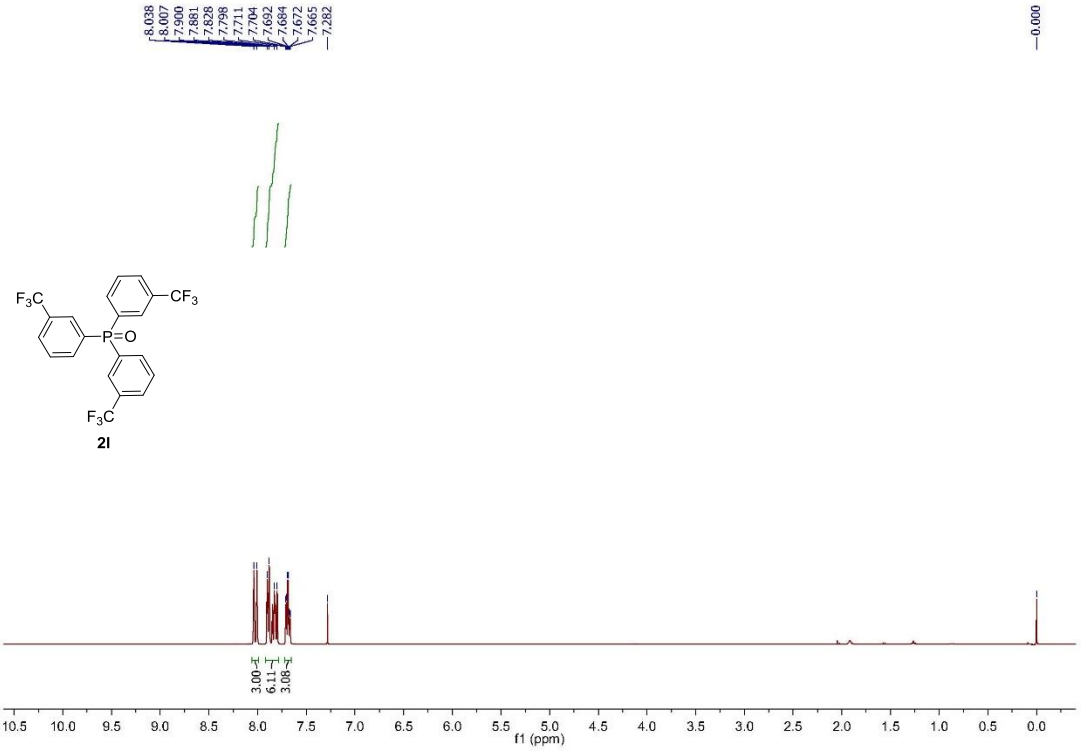

105.94
F
F
P=O
F
2j
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

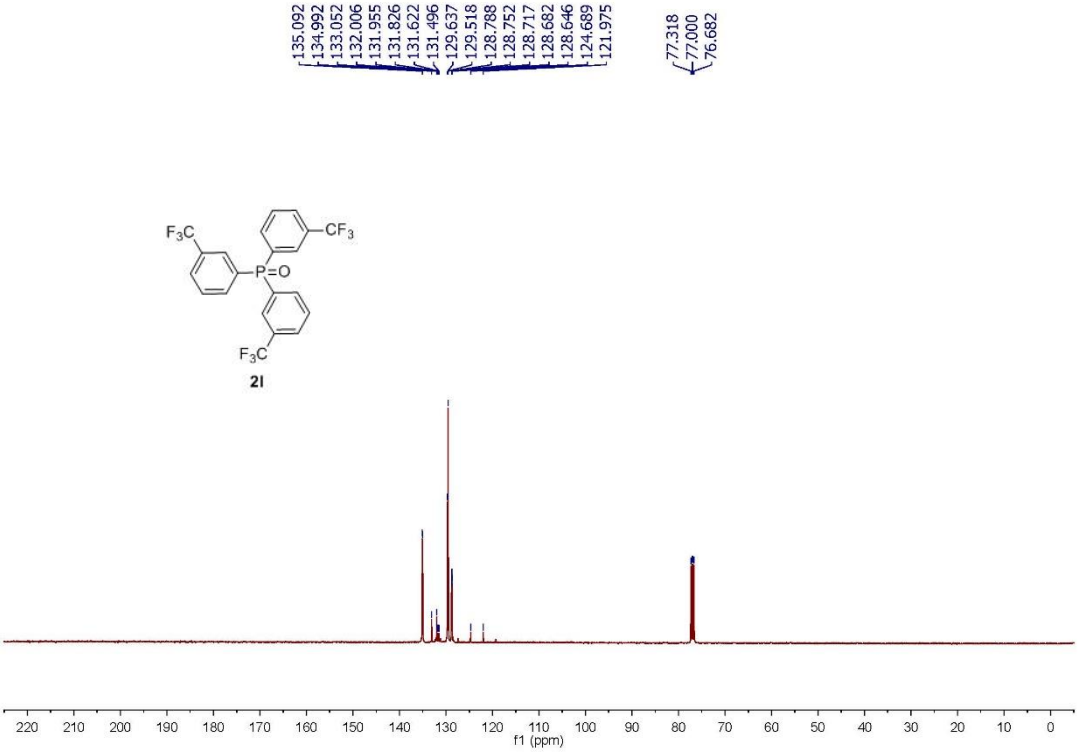

27.274
F
F
P=O
F
2j
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160 -190 -220
f1 (ppm)

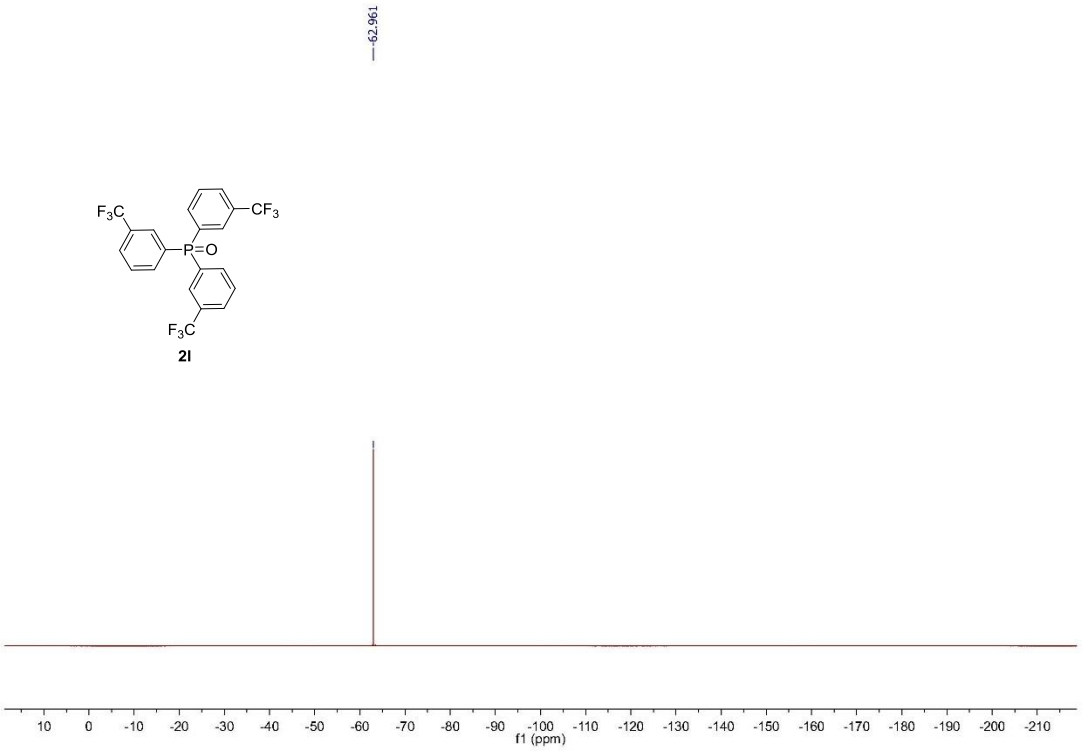

7.606
7.585
7.561
7.481
7.461
7.288
0.000
Cl
Cl
P=O
Cl
2k
5.89
6.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


139.108
139.079
133.331
133.223
130.744
129.684
129.169
129.041
77.318
77.000
76.683
Cl
Cl
P=O
Cl
2k
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

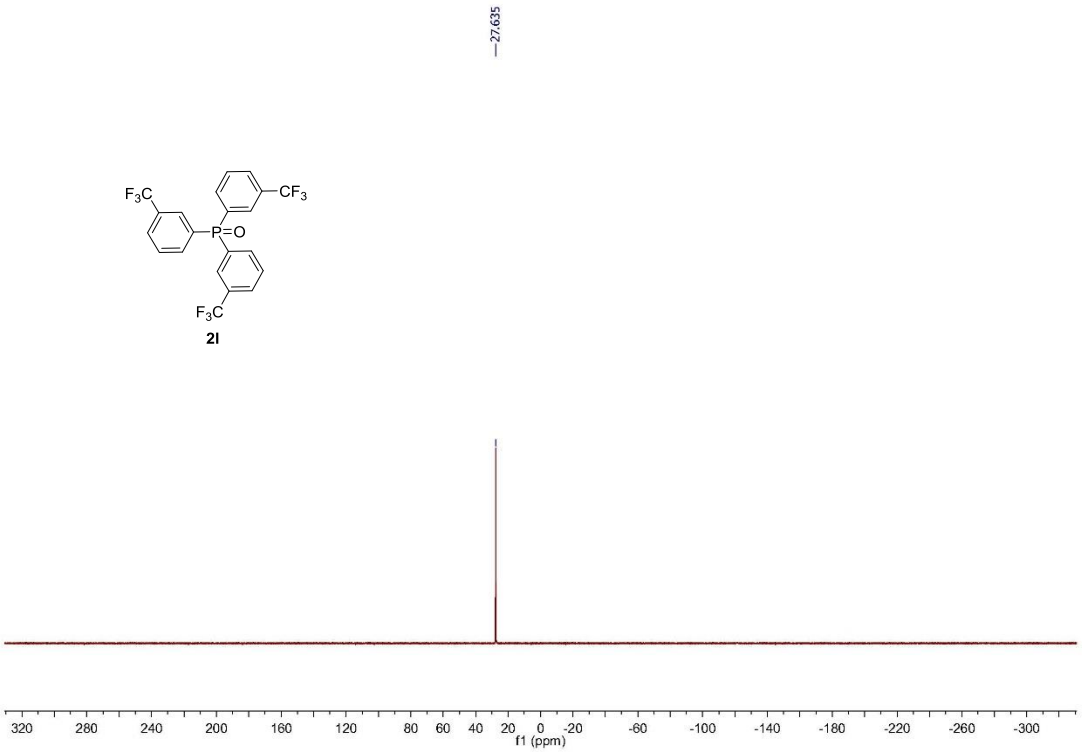

30.519
Cl
Cl
P=O
Cl
2k
320 280 240 200 160 120 80 60 40 20 0 -20 -60 -100 -140 -180 -220 -260 -300
f1 (ppm)


8.038
8.007
7.900
7.881
7.828
7.798
7.711
7.704
7.692
7.684
7.672
7.665
7.282
0.000
F3C
CF3
P=O
F3C
2l
3.00
6.11
3.08
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

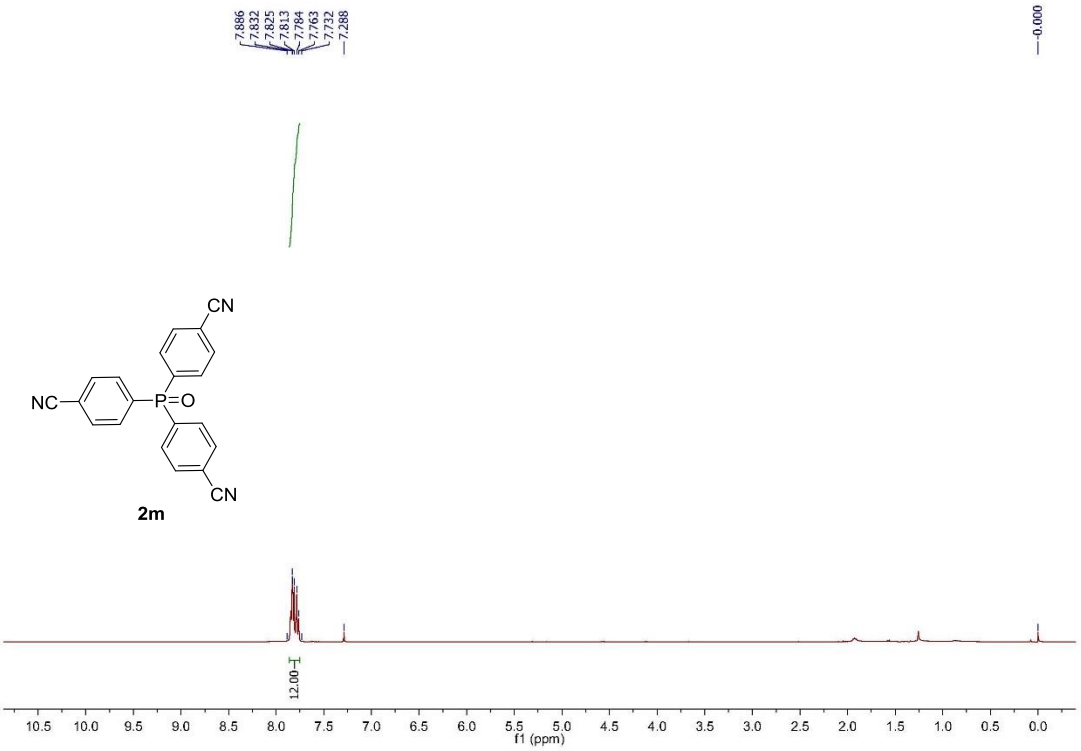

135.092
134.992
133.052
132.006
131.955
131.826
131.622
131.496
129.637
129.518
128.788
128.752
128.717
128.682
128.646
124.689
121.975
77.318
77.000
76.682
F3C
CF3
P=O
F3C
2l
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

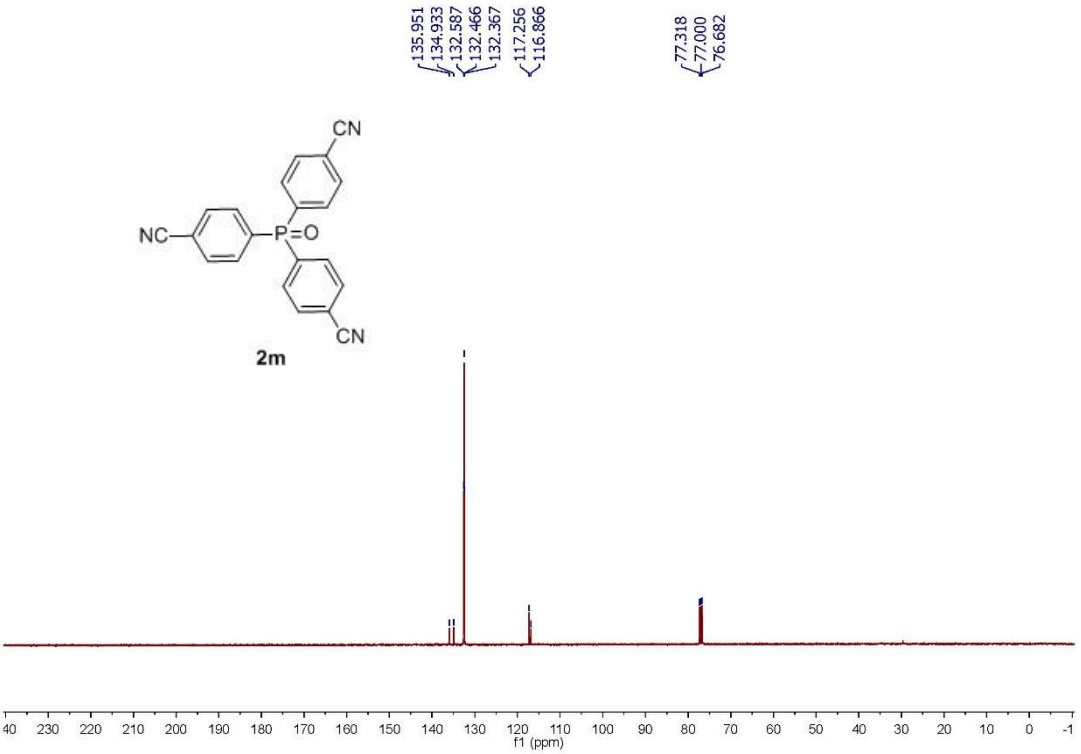

-62.961
F3C
CF3
P=O
F3C
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

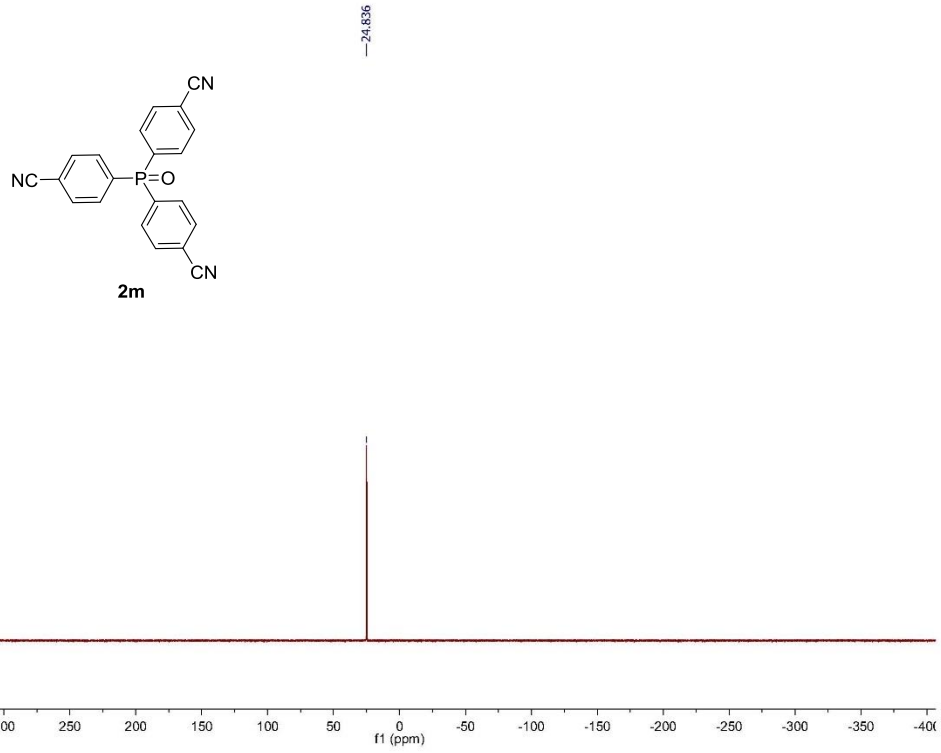
2l

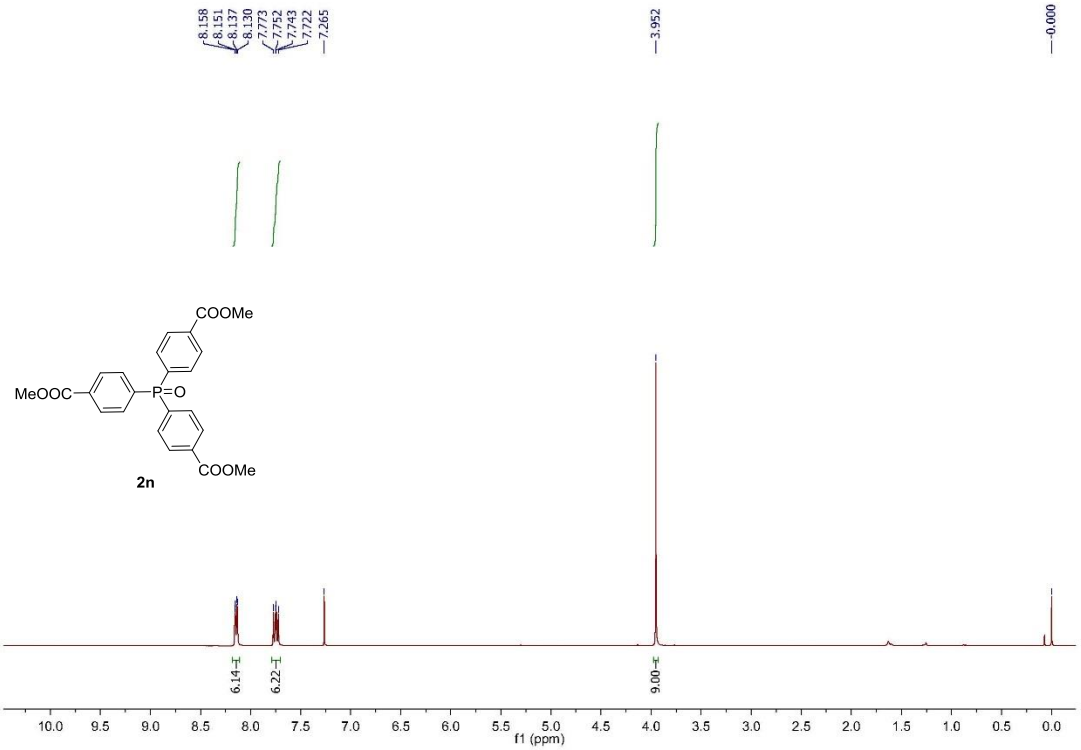

-27.635
F3C
CF3
P=O
F3C
320 280 240 200 160 120 80 60 40 20 0 -20 -60 -100 -140 -180 -220 -260 -300
f1 (ppm)

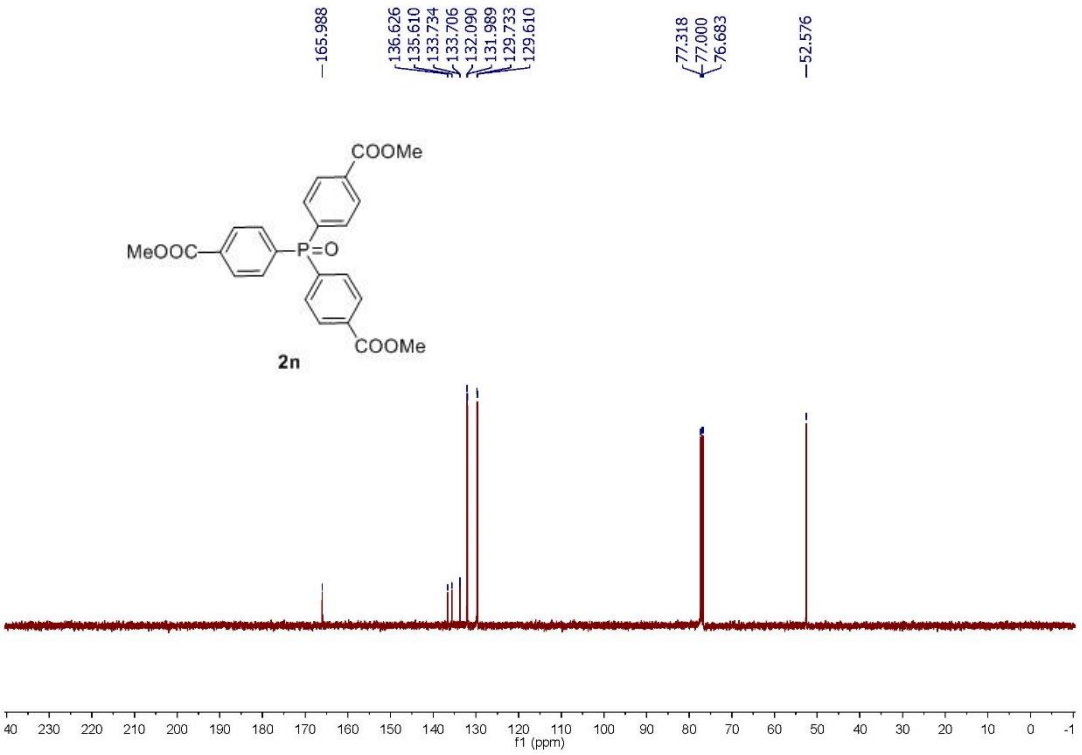
2l

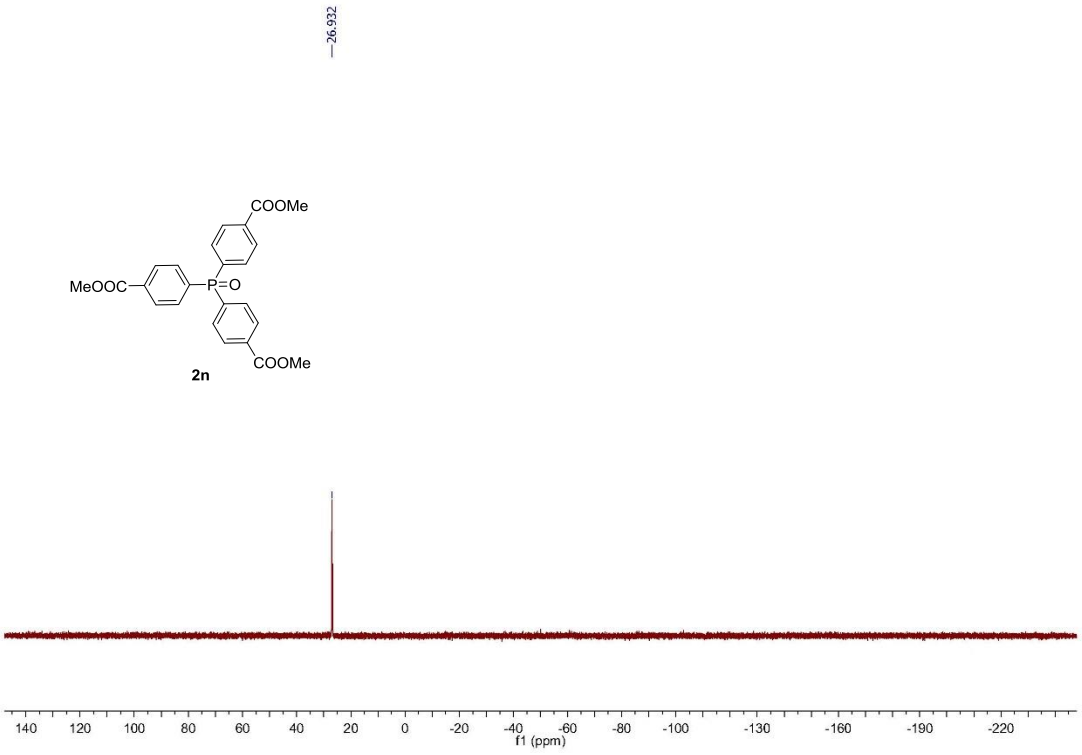

7.886
7.832
7.825
7.813
7.784
7.763
7.732
7.288
-0.000
CN
NC
P=O
CN
2m
12.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

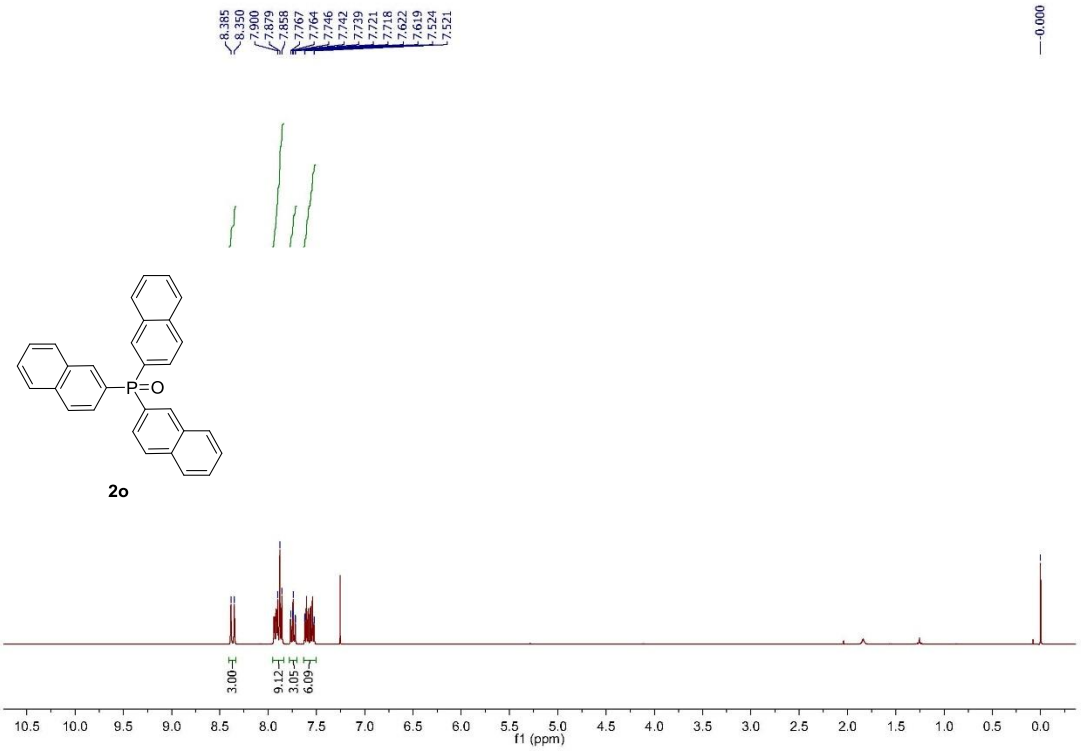

135.951
134.933
132.587
132.466
132.367
117.256
116.866
77.318
77.000
76.682
CN
NC
P=O
CN
2m
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

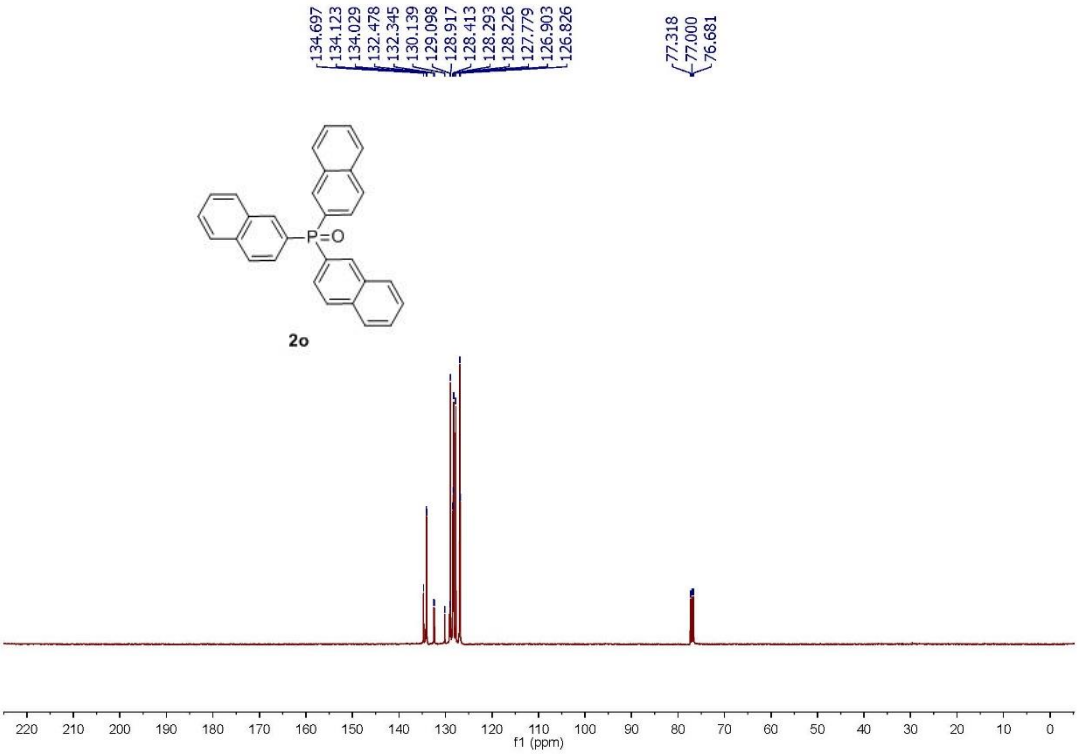

24.836
CN
NC
P=O
CN
2m
f1 (ppm)

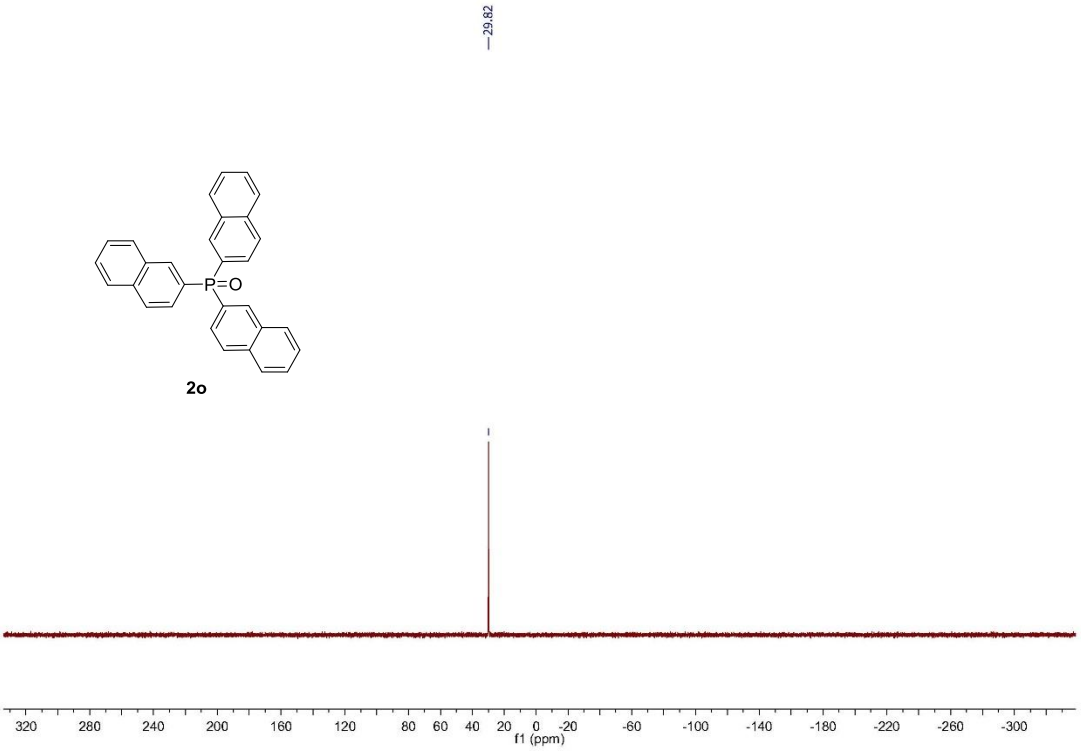

8.158
8.151
8.137
8.130
7.773
7.752
7.743
7.722
7.265
3.952
0.000
COOMe
MeOOC
P=O
COOMe
2n
6.14
6.22
9.00
f1 (ppm)

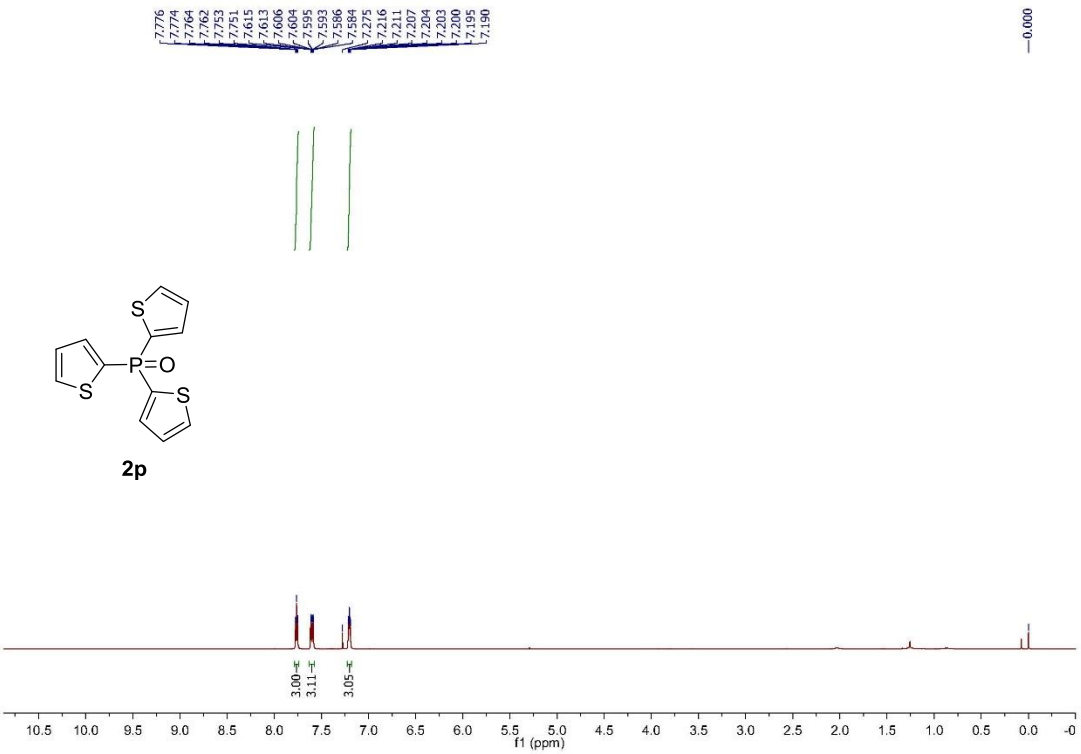

165.988
136.626
135.610
133.734
133.706
132.090
131.989
129.733
129.610
77.318
77.000
76.683
52.576
COOMe
MeOOC
P=O
COOMe
2n
40 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

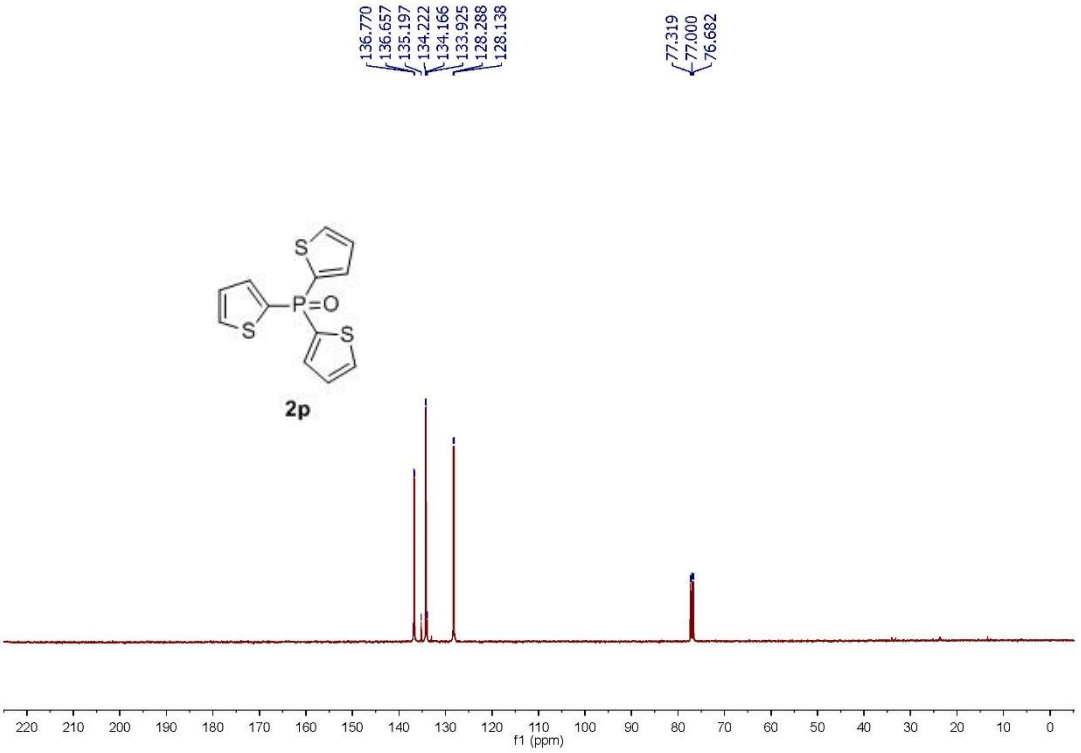

26.932
COOMe
MeOOC
P=O
COOMe
2n
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160 -190 -220
f1 (ppm)

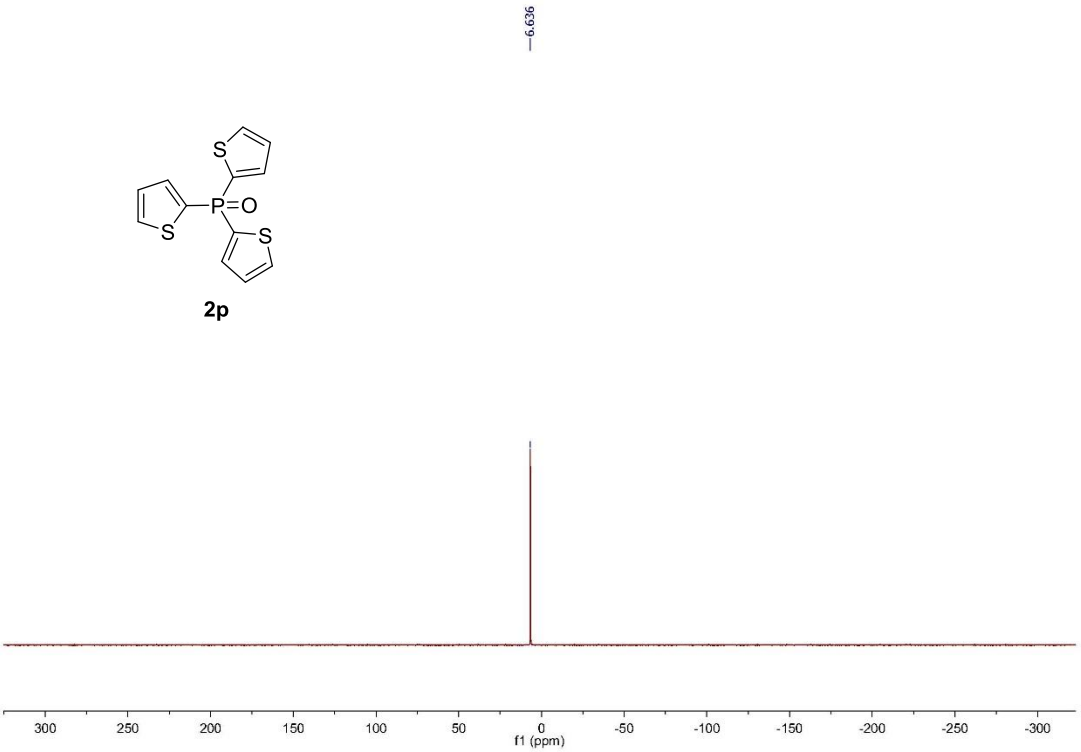

8.385
8.350
7.900
7.879
7.858
7.767
7.764
7.746
7.742
7.739
7.721
7.718
7.622
7.619
7.524
7.521
-0.000
P=O
2o
3.00
9.12
3.05
6.09
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


134.697
134.123
134.029
132.478
132.345
130.139
129.098
128.917
128.413
128.293
128.226
127.779
126.903
126.826
77.318
77.000
76.681
P=O
2o
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


29.82
P=O
2o
320 280 240 200 160 120 80 60 40 20 0 -20 -60 -100 -140 -180 -220 -260 -300
f1 (ppm)


7.776
7.774
7.764
7.762
7.753
7.751
7.615
7.613
7.606
7.604
7.595
7.593
7.586
7.584
7.275
7.216
7.211
7.207
7.204
7.203
7.200
7.195
7.190
0.000
S
P=O
S
S
2p
3.00
3.11
3.05
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)


136.770
136.657
135.197
134.222
134.166
133.925
128.288
128.138
77.319
77.000
76.682
S
P=O
S
S
2p
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


6.636
S
P=O
S
S
2p
300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300
f1 (ppm)

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