Supporting Information

Efficient Nickel-catalyzed Phosphinylation of C-S Bonds Forming C-P Bonds

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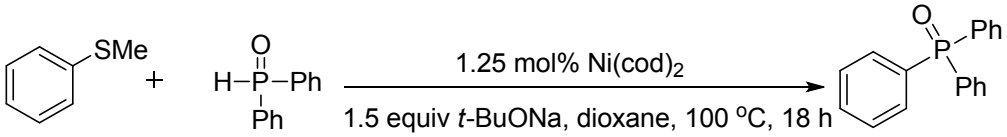
1. General information

All reactions were carried out in oven-dried Schlenk tubes under N_2 atmosphere. Dry solvents were obtained by purification according to standard methods. Reagents were used as received unless otherwise noted. Column chromatography was performed using Silica Gel 60 (particle size 37–54 μm). The pure products were obtained by means of column chromatography. ^{1}H NMR, ^{13}C NMR and ^{31}P NMR data were acquired on a 400MHz spectrometer (400 MHz for ^{1}H, 100 MHz for ^{13}C, and 162 MHz for ^{31}P NMR spectroscopy). Chemical shifts for ^{1}H NMR are referred to internal Me_4Si (0 ppm) and reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz) and integration. Data for ^{31}P NMR were relative to H_3PO_4 (85% solution in D_2O, 0 ppm). HRMS were conducted in the Analytical Center at Hunan University, China.

2. Procedure for the Efficient Nickel-catalyzed Phosphinylation of C-S Bonds Forming C-P Bonds



1a **2a** **3a**

Typical procedure: In a glove box (O_2 < 0.5 ppm, H_2O < 0.5 ppm), 0.4 mmol diphenylphosphine oxide **2a**, 1.25 mol% $Ni(cod)_2$, 0.6 mmol *t*-BuONa and 1.0 mL dry dioxane were charged into a 10 mL schlenk tube. After the schlenk tube was taken out, 0.4 mmol methyl(phenyl)sulfane **1a** was added under N_2 atmosphere. Then the mixture was stirred at 100 °C for 18 h. After removal of the volatile, the residues were passed through a short silica chromatography (particle size 37–54 μm, pether/ethyl acetate as eluent) to afford analytically pure organophosphorus compounds **3a**.

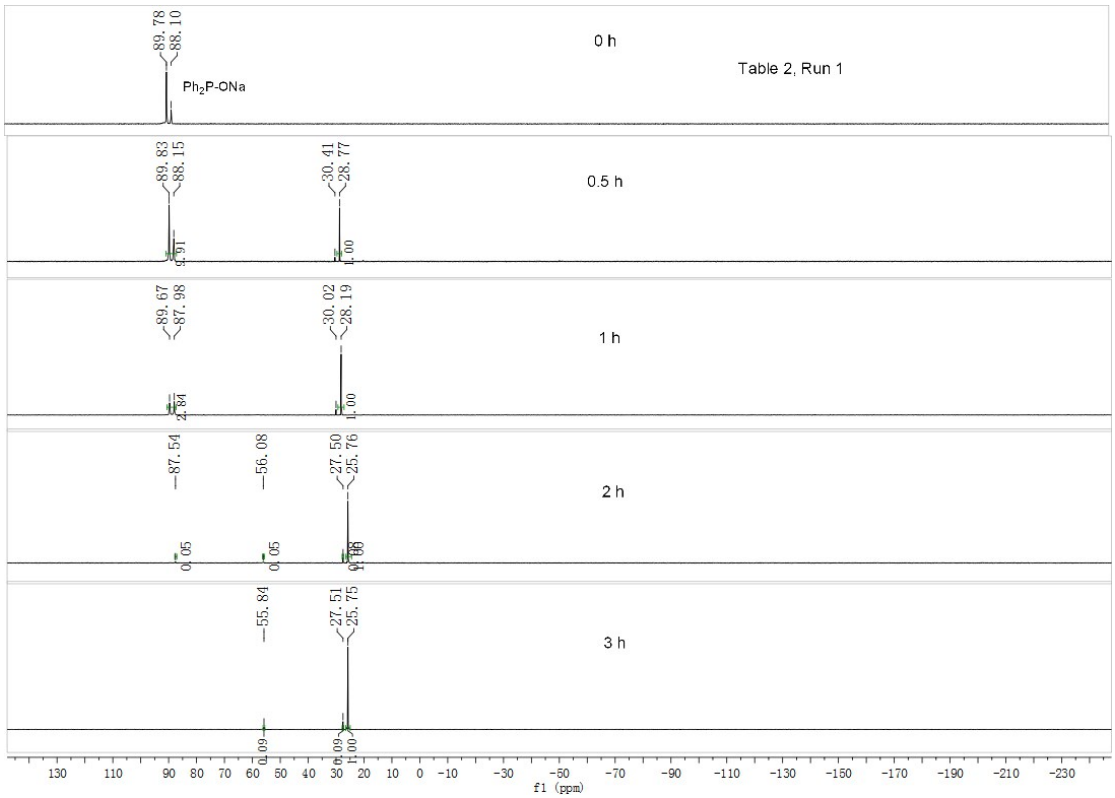
Procedure at 10 mmol scale: In a glove box (O_2 < 0.5 ppm, H_2O < 0.5 ppm), 10 mmol (2.021 g) diphenylphosphine oxide **2a**, 0.1 mol% (0.003 g) $Ni(cod)_2$, 15 mmol (1.442 g) *t*-BuONa and 20 mL dry dioxane were charged into a 100 mL schlenk tube. After the tube was taken out, 10 mmol (1.242 g) methyl(phenyl)sulfane **1a** was added under N_2 atmosphere. Then the mixture was heated at 120 °C for 30 h. The mixture was extracted with CH_2Cl_2. The crude product after removing the

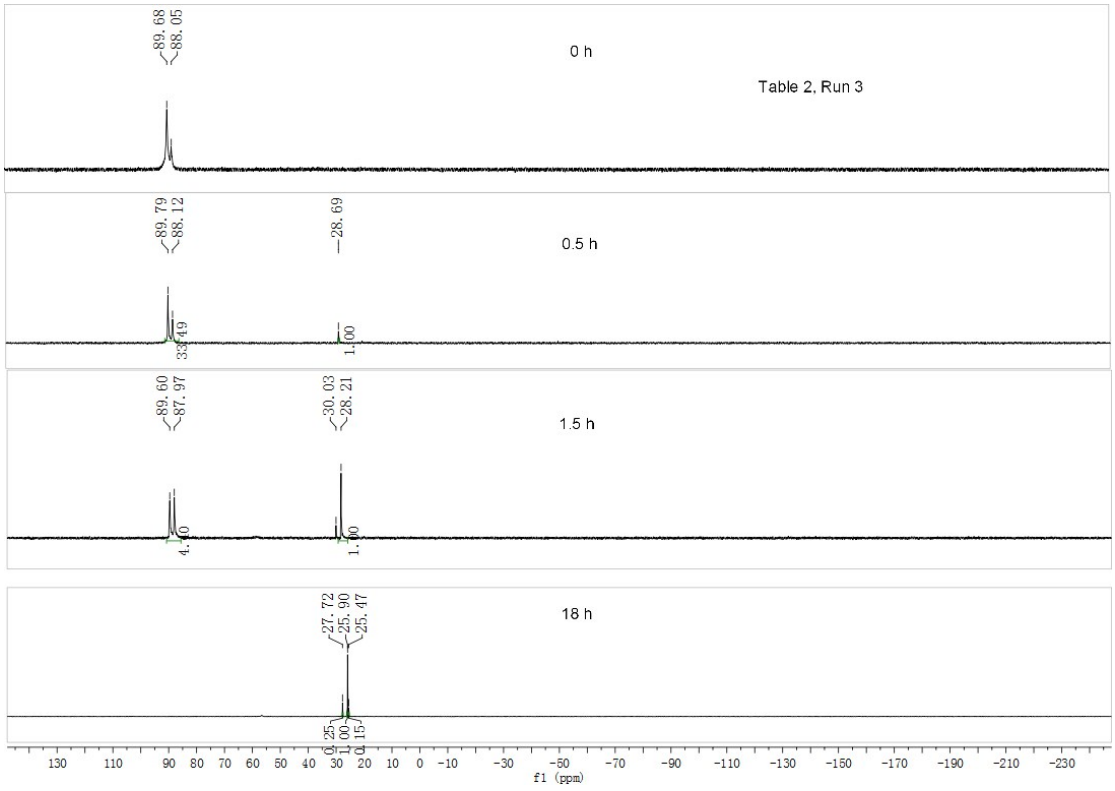
volatiles under a reduced pressure was passed through a short SiO_2 column using EtOAc as an eluent to give the spectroscopically pure **3a** in 92% yield (2.560 g).

3. ^{31}P NMR data of the reaction mixture

Procedure: In a glove box (O_2 < 0.5 ppm, H_2O < 0.5 ppm), 1 mmol **2**, 1.25 mol% $Ni(cod)_2$, 1.5 mmol *t*-BuONa (For *n*-Bu_2POH, *t*-BuOK was used as a base) and 3.0 mL dry dioxane (due to the multiple sampling for one reaction, the solvent was enlarged) were charged into a 10 mL schlenk tube. After the schlenk tube was taken out, 1 mmol **1** (if solid, **1** was added in the glove box; for sulfones: 1.1 mmol was loaded) was added under N_2 atmosphere. Samples for ^{31}P NMR detection were acquired in a glove box.

Copies of ^{31}P NMR spectra




89.68
88.05
0 h
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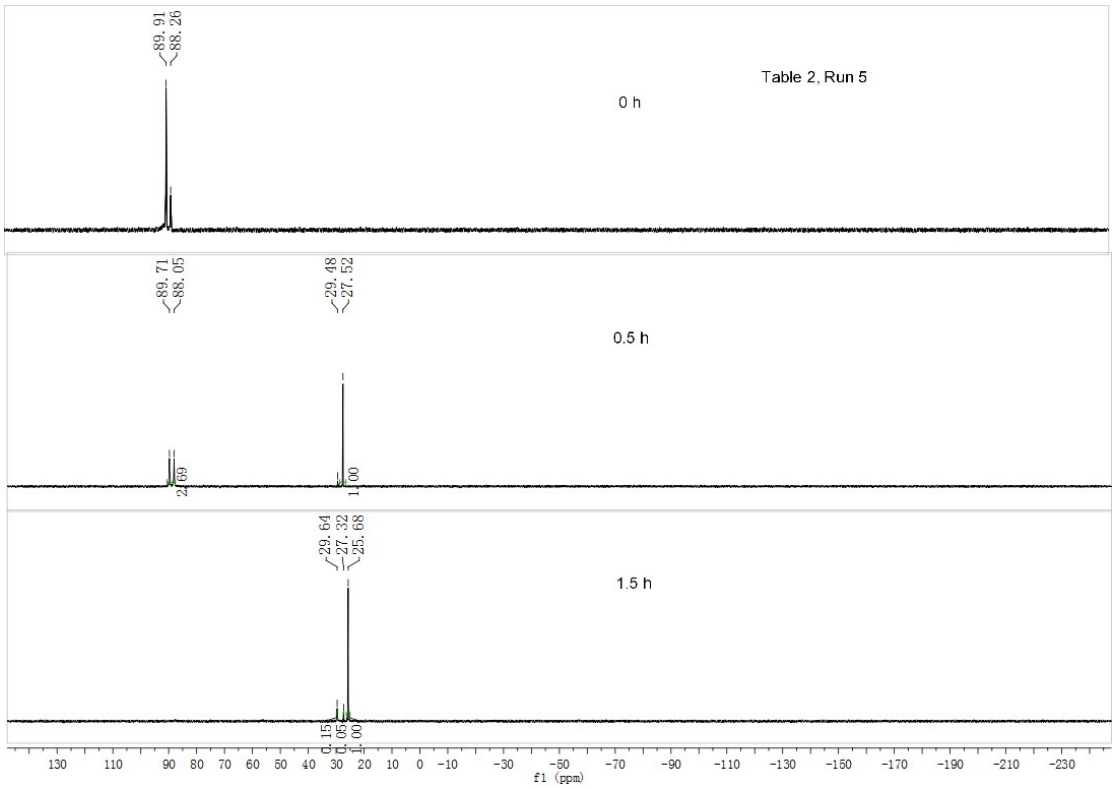
88.12
28.69
0.5 h
33.49
1.00
89.60
87.97
30.03
28.21
1.5 h
4.10
1.00
27.72
25.90
25.47
18 h
0.25
1.00
0.15
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)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88.26
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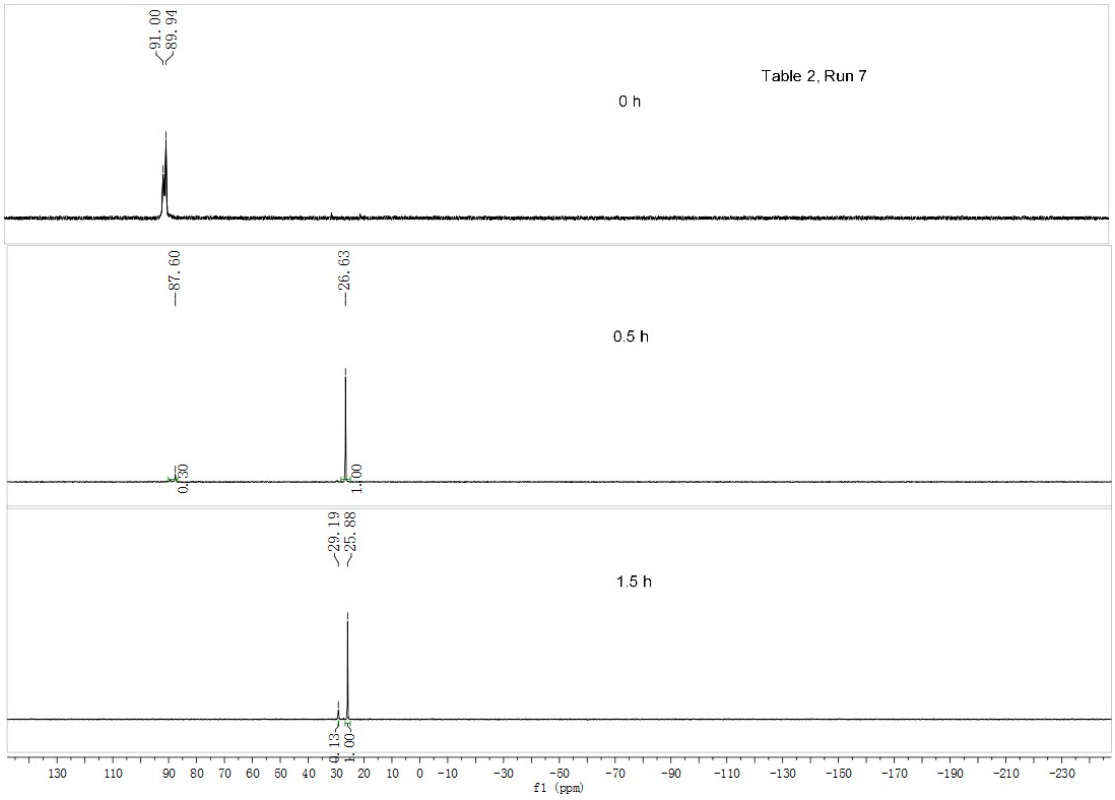
88.05
29.48
27.52
0.5 h
2.69
1.00
29.64
27.32
25.68
1.5 h
0.15
0.05
1.00
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)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1.5 h
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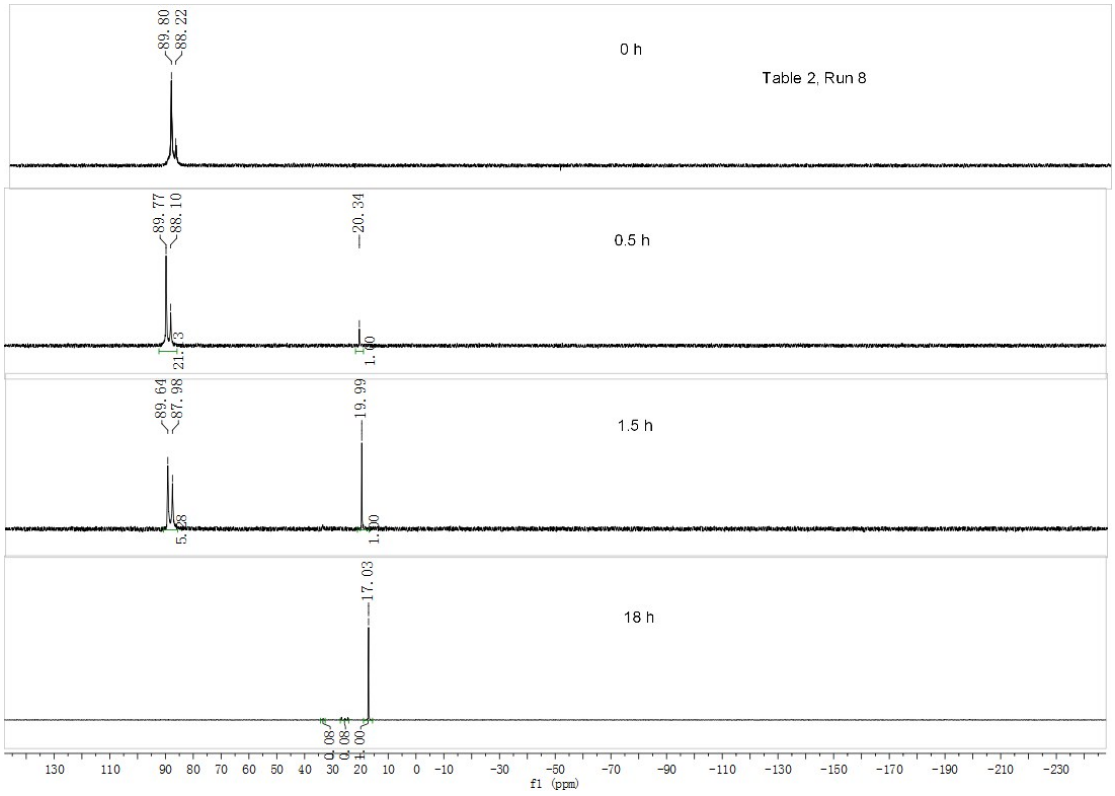
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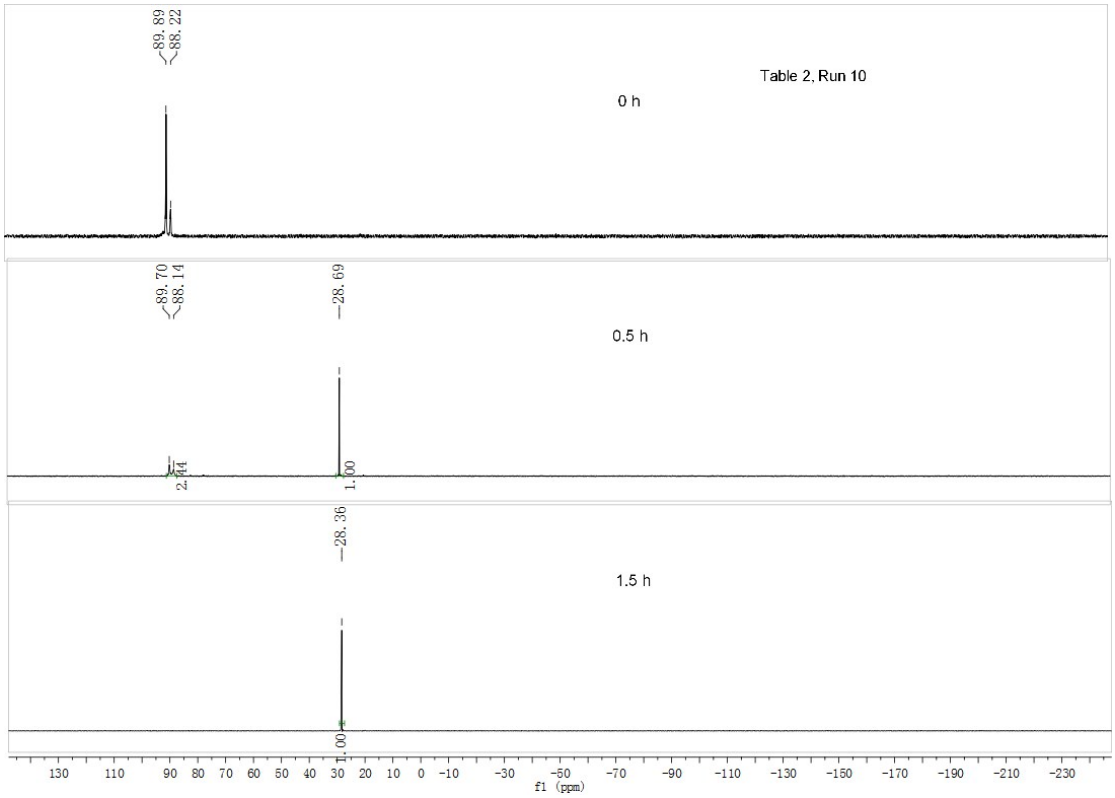
1.5 h
18 h
f1 (ppm)


89.89
88.22
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88.14
28.69
0.5 h
2.44
1.00
28.36
1.5 h
1.00
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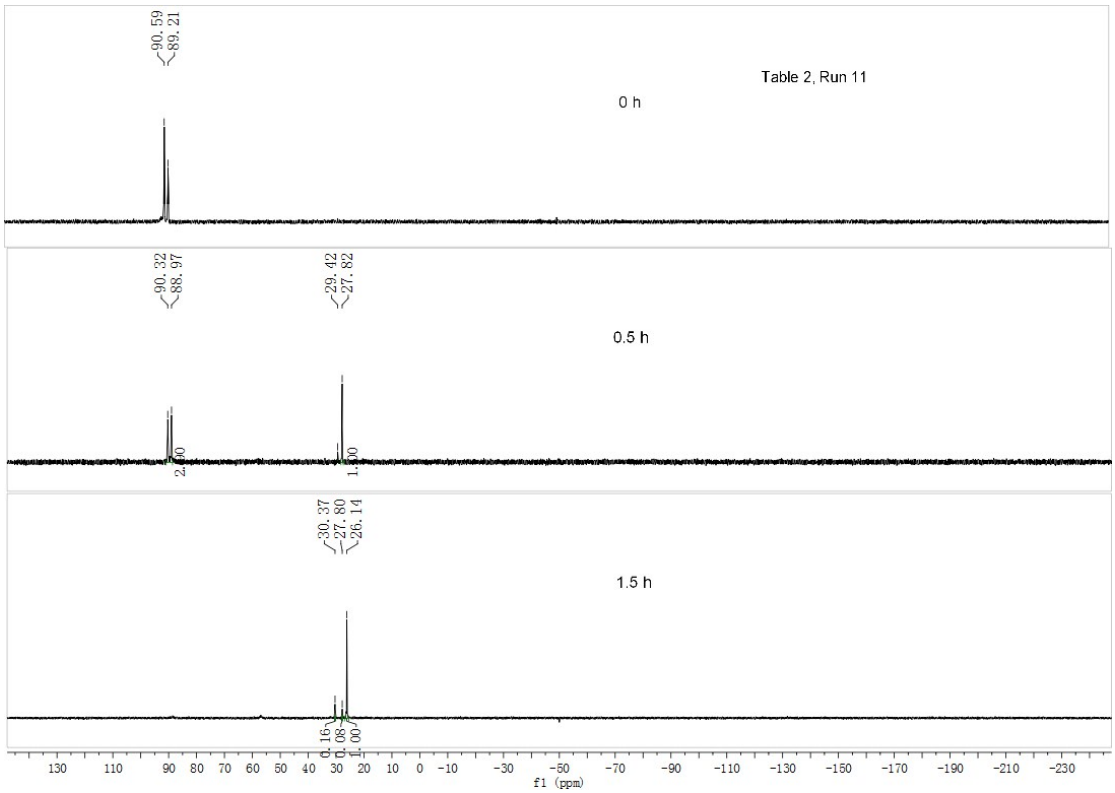
f1 (ppm)


90.59
89.21
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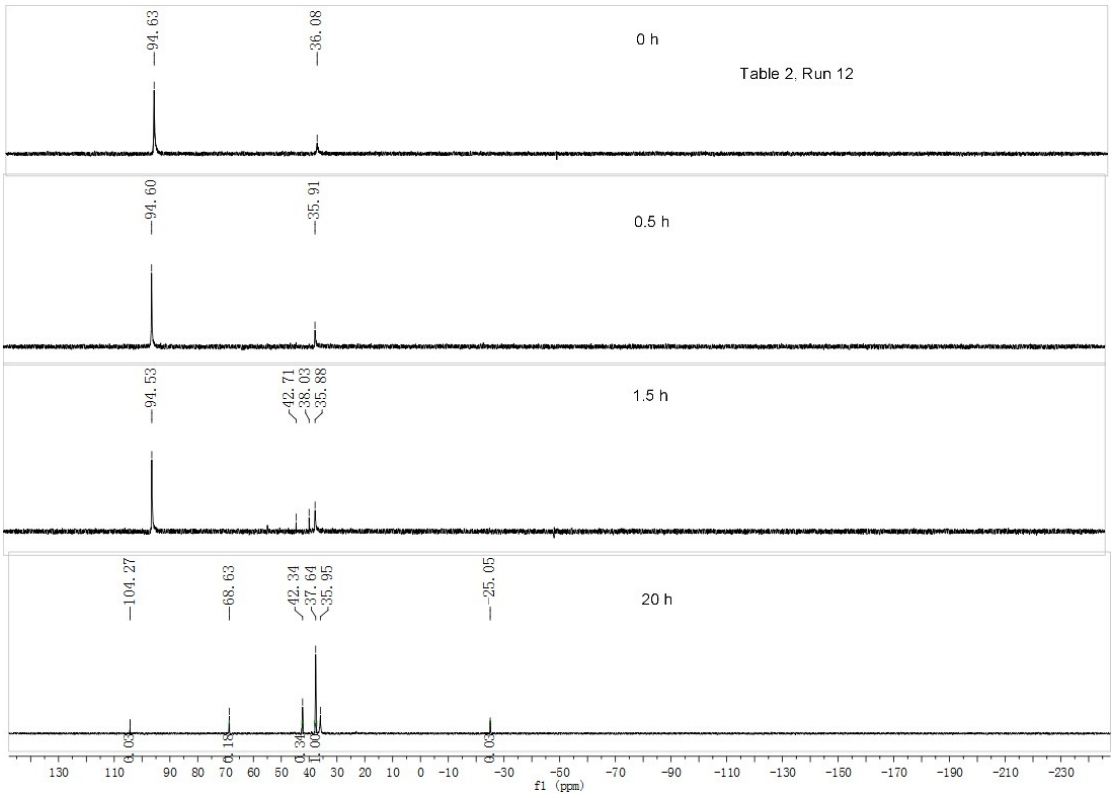
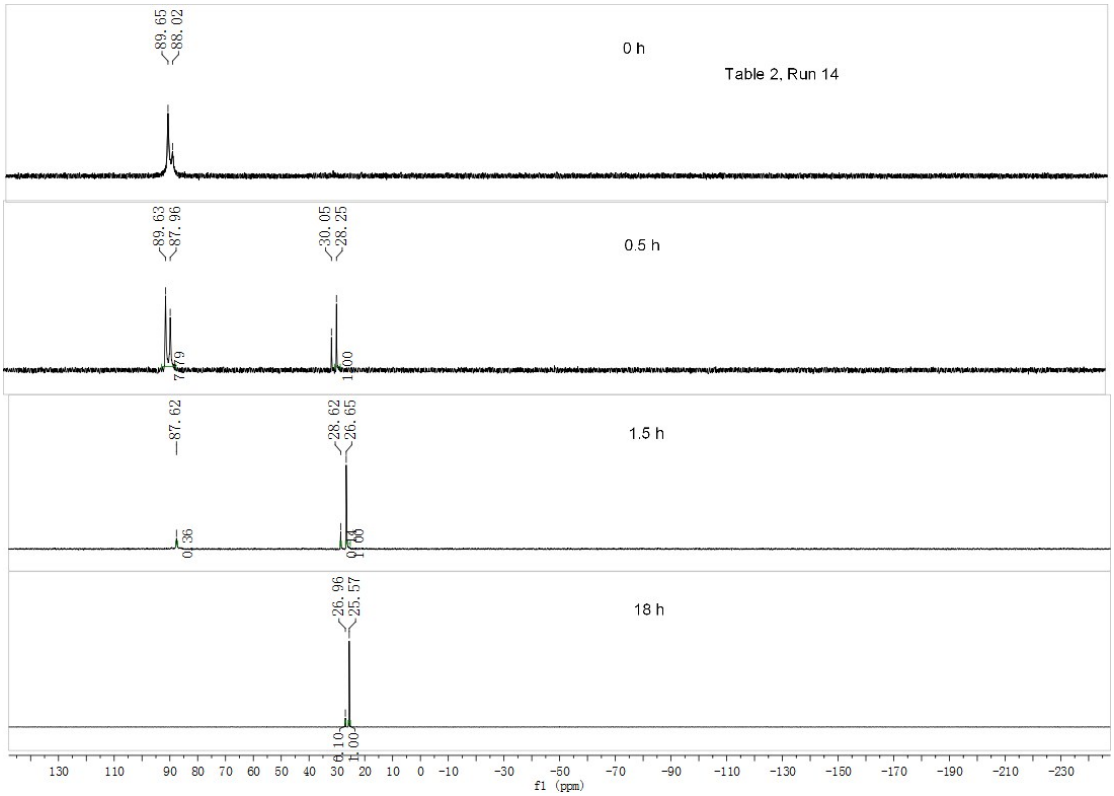
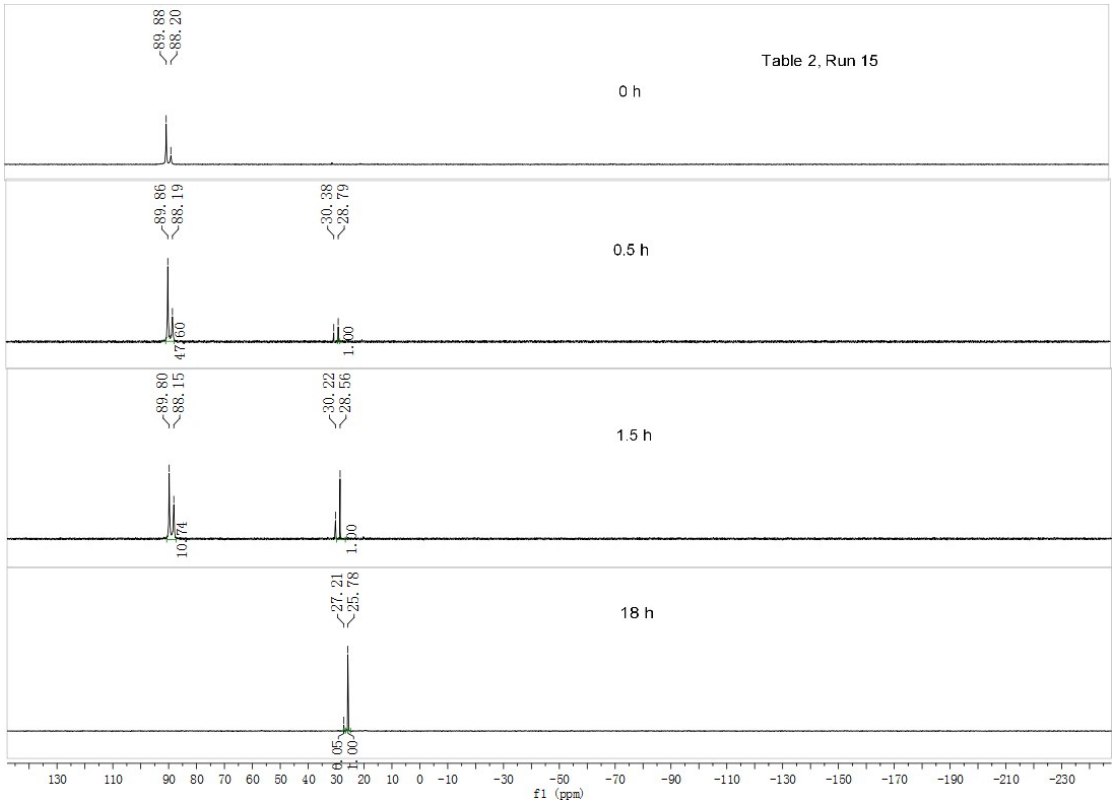
27.82
0.5 h
2.40
1.00
30.37
27.80
26.14
1.5 h
0.16
0.08
1.00
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)



Note: the ^{31}P signal of *n*-$Bu_2P(O)K$ is at 94.6 ppm, the ^{31}P signal of *n*-$Bu_2P(O)Ph$ is at 37.6 ppm.

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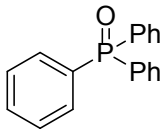
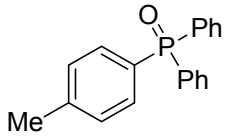
88.20
0.5 h
89.86
88.19
30.38
28.79
4.60
1.00
1.5 h
89.80
88.15
30.22
28.56
10.74
1.00
18 h
27.21
25.78
6.05
1.00
f1 (ppm)

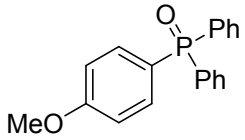
4. Characterization and analytical data of products 3



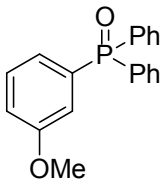
Triphenylphosphine oxide (3a).[1] White solid; ^{1}H NMR (400 MHz $CDCl_3$): δ 7.70–7.65 (m, 6H), 7.57–7.53 (m, 3H), 7.48–7.44 (m, 6H). ^{13}C NMR (100 MHz $CDCl_3$): δ 132.6 (d, $J_{C\text{-}P}$ = 103.3 Hz), 132.1 (d, $J_{C\text{-}P}$ = 9.8 Hz), 132.0 (d, $J_{C\text{-}P}$ = 2.7 Hz), 128.5 (d, $J_{C\text{-}P}$ = 12.0 Hz). ^{31}P NMR (162 MHz $CDCl_3$): δ 29.16. MS (EI): 277.



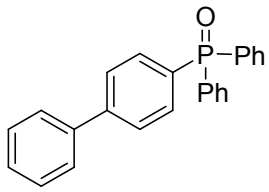
Diphenyl(p-tolyl)phosphine oxide (3b).[1] White solid; ^{1}H NMR (400 MHz $CDCl_3$): δ 7.66 (dd, J = 8.0 Hz, J = 12.0 Hz, 4H), 7.58–7.50 (m, 4H), 7.46–7.43 (m, 4H), 7.26 (dd, J = 1.6 Hz, J = 8.0 Hz, 2H), 2.39 (s, 3H). ^{13}C NMR (100 MHz $CDCl_3$): δ 142.5 (d, $J_{C\text{-}P}$ = 2.6 Hz), 132.8 (d, $J_{C\text{-}P}$ = 103.4 Hz), 132.1 (d, $J_{C\text{-}P}$ = 10.1 Hz), 132.1 (d, $J_{C\text{-}P}$ = 9.8 Hz), 131.8 (d, $J_{C\text{-}P}$ = 2.7 Hz), 129.3 (d, $J_{C\text{-}P}$ = 12.5 Hz), 129.1 (d, $J_{C\text{-}P}$ = 106.0 Hz), 128.5 (d, $J_{C\text{-}P}$ = 12.0 Hz), 21.6 (d, $J_{C\text{-}P}$ = 0.9 Hz). ^{31}P NMR (162 MHz $CDCl_3$): δ 29.23. MS (EI): 291.



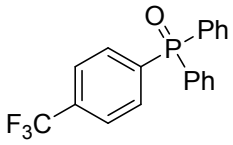
(4-Methoxyphenyl)diphenylphosphine oxide (3c).[1] White solid; ^{1}H NMR (400 MHz $CDCl_3$): δ 7.66 (dd, J = 8.0 Hz, J = 12.0 Hz, 4H), 7.61–7.51 (m, 4H), 7.46–7.43 (m, 4H), 6.97 (dd, J = 2.0 Hz, J = 8.8 Hz, 2H), 3.84 (s, 3H). ^{13}C NMR (100 MHz $CDCl_3$): δ 162.5 (d, $J_{C\text{-}P}$ = 2.8 Hz), 134.0 (d, $J_{C\text{-}P}$ = 11.2 Hz), 133.0 (d, $J_{C\text{-}P}$ = 103.8 Hz), 132.0 (d, $J_{C\text{-}P}$ = 9.8 Hz), 131.8 (d, $J_{C\text{-}P}$ = 2.7 Hz), 128.5 (d, $J_{C\text{-}P}$ = 12.0 Hz), 123.6 (d, $J_{C\text{-}P}$ = 109.8 Hz), 114.1 (d, $J_{C\text{-}P}$ = 13.1 Hz), 55.4. ^{31}P NMR (162 MHz $CDCl_3$): δ 29.08. MS (EI): 307.



(3-Methoxyphenyl)diphenylphosphine oxide (3d).[2] White solid; ^{1}H NMR (400 MHz $CDCl_3$): δ 7.66 (dd, J = 8.0 Hz, J = 12.0 Hz, 4H), 7.54 (t, J = 7.6 Hz, 2H), 7.48–7.44 (m, 4H), 7.38–7.33 (m, 1H), 7.39 (d, J = 13.2 Hz, 1H), 7.14 (dd, J = 7.6 Hz, J = 12.0 Hz, 1H), 7.07 (dd, J = 1.6 Hz, J = 8.0 Hz, 1H), 3.79 (s, 3H). ^{13}C NMR (100 MHz $CDCl_3$): δ 159.6 (d, $J_{C\text{-}P}$ = 14.7 Hz), 133.8 (d, $J_{C\text{-}P}$ = 102.6 Hz), 132.4 (d, $J_{C\text{-}P}$ = 103.7 Hz), 132.1 (d, $J_{C\text{-}P}$ = 9.8 Hz), 132.0 (d, $J_{C\text{-}P}$ = 2.7 Hz), 129.7 (d, $J_{C\text{-}P}$ = 14.2 Hz), 128.5 (d, $J_{C\text{-}P}$ = 12.1 Hz), 124.4 (d, $J_{C\text{-}P}$ = 10.0 Hz), 118.2 (d, $J_{C\text{-}P}$ = 2.7 Hz), 116.8 (d, $J_{C\text{-}P}$ = 10.7 Hz), 55.4. ^{31}P NMR (162 MHz $CDCl_3$): δ 29.31. MS (EI): 307.

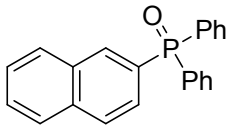


[1,1'-Biphenyl]-4-yldiphenylphosphine oxide (3e).[1] White solid; ^{1}H NMR (400 MHz $CDCl_3$): δ 7.79–7.68 (m, 8H), 7.61–7.54 (m, 4H), 7.50–7.37 (m, 7H). ^{13}C NMR (100 MHz $CDCl_3$): δ 144.8 (d, $J_{C\text{-}P}$ = 2.6 Hz), 139.9, 132.6 (d, $J_{C\text{-}P}$ = 10.2 Hz), 132.5 (d, $J_{C\text{-}P}$ = 103.8 Hz), 132.1 (d, $J_{C\text{-}P}$ = 9.9 Hz), 132.0 (d, $J_{C\text{-}P}$ = 4.5 Hz), 131.0 (d, $J_{C\text{-}P}$ = 104.7 Hz), 130.0, 128.6 (d, $J_{C\text{-}P}$ = 12.1 Hz), 128.2, 127.3, 127.2. ^{31}P NMR (162 MHz $CDCl_3$): δ 29.32. MS (EI): 353.

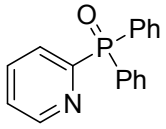


diphenyl(4-(trifluoromethyl)phenyl)phosphine oxide (3f).[1] White solid; ^{1}H NMR (400 MHz $CDCl_3$): δ 7.75 (dd, J = 8.0 Hz, J = 11.2 Hz, 2H), 7.64 (d, J = 7.6 Hz, 2H), 7.58 (dd, J = 7.6 Hz, J = 12.0 Hz, 4H), 7.50 (t, J = 7.2 Hz, 2H), 7.42–7.39 (m, 4H). ^{13}C NMR (100 MHz $CDCl_3$): δ 137.1 (d, $J_{C\text{-}P}$ = 99.3 Hz), 133.7 (dq, $J_{C\text{-}P}$ = 2.9 Hz, $J_{C\text{-}F}$ = 32.6 Hz), 132.6 (d, $J_{C\text{-}P}$ = 10.1 Hz), 132.4 (d, $J_{C\text{-}P}$ = 2.7 Hz), 132.0 (d, $J_{C\text{-}P}$ = 10.0 Hz), 131.6 (d, $J_{C\text{-}P}$ = 102.2 Hz), 128.8 (d, $J_{C\text{-}P}$ = 12.2 Hz), 125.4 (dq, $J_{C\text{-}P}$ = 12.1 Hz, $J_{C\text{-}F}$ = 3.5 Hz), 122.2 (q, $J_{C\text{-}F}$ = 271.2 Hz). ^{31}P NMR (162 MHz $CDCl_3$):

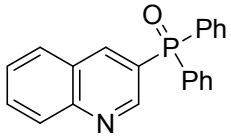
δ 28.15. MS (EI): 345.



Naphthalen-2-yldiphenylphosphine oxide (3g).[1] White solid; ^{1}H NMR (400 MHz $CDCl_3$): δ 8.29 (d, J = 13.6 Hz, 1H), 7.91–7.86 (m, 3H), 7.72 (dd, J = 7.6 Hz, J = 12.0 Hz, 4H), 7.67–7.54 (m, 5H), 7.47 (dd, J = 7.6 Hz, J = 7.2 Hz, 4H). ^{13}C NMR (100 MHz $CDCl_3$): δ 134.7 (d, $J_{C\text{-}P}$ = 2.3 Hz), 134.1 (d, $J_{C\text{-}P}$ = 9.3 Hz), 132.5 (d, $J_{C\text{-}P}$ = 103.8 Hz), 132.4 (d, $J_{C\text{-}P}$ = 13.1 Hz), 132.2 (d, $J_{C\text{-}P}$ = 9.9 Hz), 132.1 (d, $J_{C\text{-}P}$ = 2.6 Hz), 129.5 (d, $J_{C\text{-}P}$ = 102.0 Hz), 129.0, 128.6 (d, $J_{C\text{-}P}$ = 12.1 Hz), 128.3 (d, $J_{C\text{-}P}$ = 11.6 Hz), 128.3, 127.9, 127.0, 126.9 (d, $J_{C\text{-}P}$ = 10.6 Hz). ^{31}P NMR (162 MHz $CDCl_3$): δ 29.42. MS (EI): 327.

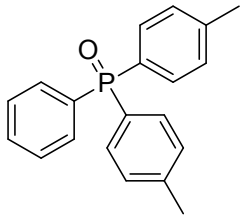


Diphenyl(pyridin-2-yl)phosphine oxide (3h).[3] White solid; ^{1}H NMR (400 MHz $CDCl_3$): δ 8.77 (d, J = 4.0 Hz, 1H), 8.30 (t, J = 6.8 Hz, 1H), 7.91–7.81 (m, 5H), 7.52–7.37 (m, 7H). ^{13}C NMR (100 MHz $CDCl_3$): δ 156.4 (d, $J_{C\text{-}P}$ = 130.9 Hz), 150.2 (d, $J_{C\text{-}P}$ = 19.0 Hz), 136.2 (d, $J_{C\text{-}P}$ = 9.3 Hz), 132.2 (d, $J_{C\text{-}P}$ = 103.4 Hz), 132.1 (d, $J_{C\text{-}P}$ = 9.4 Hz), 131.9 (d, $J_{C\text{-}P}$ = 2.8 Hz), 128.349 (d, $J_{C\text{-}P}$ = 12.1 Hz), 128.346 (d, $J_{C\text{-}P}$ = 19.8 Hz), 125.3 (d, $J_{C\text{-}P}$ = 3.2 Hz). ^{31}P NMR (162 MHz $CDCl_3$): δ 20.80. MS (EI): 278.

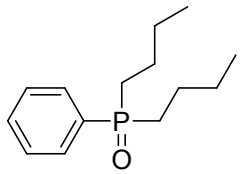


Diphenyl(quinolin-3-yl)phosphine oxide (3i).[4] White solid; ^{1}H NMR (400 MHz $CDCl_3$): δ 9.02–9.00 (m, 1H), 8.64 (d, J = 13.2 Hz, 1H), 8.16 (d, J = 8.4 Hz, 1H), 7.89–7.82 (m, 2H), 7.73 (dd, J = 8.0 Hz, J = 12.0 Hz, 4H), 7.63–7.58 (m, 3H), 7.52–7.49 (dd, J = 7.2 Hz, J = 7.2 Hz, 4H). ^{13}C NMR (100 MHz $CDCl_3$): δ 150.8 (d, $J_{C\text{-}P}$ = 12.4 Hz), 149.1 (d, $J_{C\text{-}P}$ = 1.3 Hz), 142.1 (d, $J_{C\text{-}P}$ = 7.4 Hz), 132.5 (d, $J_{C\text{-}P}$ = 2.8 Hz), 132.1 (d, $J_{C\text{-}P}$ = 10.1 Hz), 131.9, 131.8 (d, $J_{C\text{-}P}$ = 104.7 Hz), 129.5,

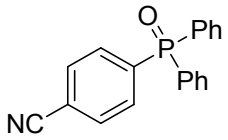
128.8 (d, $J_{C\text{-}P}$ = 12.3 Hz), 128.8, 127.7, 126.8 (d, $J_{C\text{-}P}$ = 10.4 Hz), 126.0 (d, $J_{C\text{-}P}$ = 100.5 Hz). ^{31}P NMR (162 MHz $CDCl_3$): δ 26.37. MS (EI): 328.



Phenyldi-p-tolylphosphine oxide (3j).[1] White solid; ^{1}H NMR (400 MHz $CDCl_3$): δ 7.65 (dd, J = 7.6 Hz, J = 12.0 Hz, 2H), 7.57–7.52 (m, 5H), 7.46–7.42 (m, 2H), 7.26 (d, J = 6.8 Hz, 4H), 2.39 (s, 6H). ^{13}C NMR (100 MHz $CDCl_3$): δ 142.4 (d, $J_{C\text{-}P}$ = 2.8 Hz), 132.9 (d, $J_{C\text{-}P}$ = 103.3 Hz), 132.1 (d, $J_{C\text{-}P}$ = 10.2 Hz), 132.1 (d, $J_{C\text{-}P}$ = 9.8 Hz), 131.8 (d, $J_{C\text{-}P}$ = 2.7 Hz), 129.3 (d, $J_{C\text{-}P}$ = 12.5 Hz), 129.2 (d, $J_{C\text{-}P}$ = 106.5 Hz), 128.4 (d, $J_{C\text{-}P}$ = 12.1 Hz), 21.6 (d, $J_{C\text{-}P}$ = 1.0 Hz). ^{31}P NMR (162 MHz $CDCl_3$): δ 29.64. MS (EI): 305.

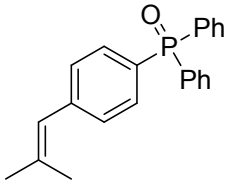


Dibutyl(phenyl)phosphine oxide (3k).[1] White solid; ^{1}H NMR (400 MHz $CDCl_3$): δ 7.70 (dd, J = 8.4 Hz, J = 8.4 Hz, 2H), 7.51 (b, 3H), 2.02–1.81 (m, 4H), 1.60 (br, 2H), 1.38 (br, 6H), 0.87 (t, J = 6.8 Hz, 6H). ^{13}C NMR (100 MHz $CDCl_3$): δ 132.7 (d, $J_{C\text{-}P}$ = 91.9 Hz), 131.4 (d, $J_{C\text{-}P}$ = 2.6 Hz), 130.4 (d, $J_{C\text{-}P}$ = 8.7 Hz), 128.6 (d, $J_{C\text{-}P}$ = 11.0 Hz), 29.7 (d, $J_{C\text{-}P}$ = 68.1 Hz), 24.1 (d, $J_{C\text{-}P}$ = 14.5 Hz), 23.5 (d, $J_{C\text{-}P}$ = 4.0 Hz), 13.56. ^{31}P NMR (162 MHz $CDCl_3$): δ 40.71. MS (EI): 238.

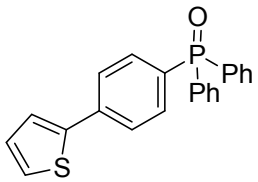


4-(Diphenylphosphoryl)benzonitrile (3m).[3] White solid; ^{1}H NMR (400 MHz $CDCl_3$): δ 7.84–7.75 (m, 4H), 7.68–7.58 (m, 6H), 7.52–7.49 (m, 4H). ^{13}C NMR (100 MHz $CDCl_3$): δ 138.3 (d, $J_{C\text{-}P}$ = 99.0 Hz), 132.7 (d, $J_{C\text{-}P}$ = 5.3 Hz), 132.6 (d, $J_{C\text{-}P}$ = 1.6 Hz), 132.0 (d, $J_{C\text{-}P}$ = 11.9 Hz), 132.0 (d, $J_{C\text{-}P}$ = 10.0 Hz), 131.0 (d, $J_{C\text{-}P}$ = 105.0 Hz), 128.9 (d, $J_{C\text{-}P}$ = 12.3 Hz), 117.9 (d, $J_{C\text{-}P}$ = 1.3 Hz),

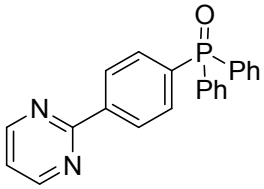
115.7 (d, $J_{C\text{-}P}$ = 3.1 Hz). ^{31}P NMR (162 MHz $CDCl_3$): δ 28.20. MS (EI): 302.



(4-(2-Methylprop-1-en-1-yl)phenyl)diphenylphosphine oxide (3n). White solid, m.p. 125–126 ^{o}C; ^{1}H NMR (400 MHz $CDCl_3$): δ 7.71–7.66 (dd, J = 8.0 Hz, J = 12.0 Hz, 4H), 7.62–7.52 (m, 4H), 7.46 (dd, J = 7.2 Hz, J = 7.2 Hz, 4H), 7.31 (d, J = 6.8 Hz, 2H), 6.27 (s, 1H), 1.92 (s, 3H), 1.87 (s, 3H). ^{13}C NMR (100 MHz $CDCl_3$): δ 142.5 (d, $J_{C\text{-}P}$ = 2.8 Hz), 138.2, 132.6 (d, $J_{C\text{-}P}$ = 103.6 Hz), 132.1 (d, $J_{C\text{-}P}$ = 9.9 Hz), 131.9 (d, $J_{C\text{-}P}$ = 5.5 Hz), 131.9 (d, $J_{C\text{-}P}$ = 1.8 Hz), 129.1 (d, $J_{C\text{-}P}$ = 106.0 Hz), 128.7 (d, $J_{C\text{-}P}$ = 12.4 Hz), 128.5 (d, $J_{C\text{-}P}$ = 12.1 Hz), 124.3 (d, $J_{C\text{-}P}$ = 1.1 Hz), 27.1, 19.6. ^{31}P NMR (162 MHz $CDCl_3$): δ 29.50. HRMS: Cal. for $C_{22}H_{21}O_1P_1$ 332.1330. Found [M] 332.1316. IR: 3056, 2929, 2912, 1653, 1183, 1119, 871, 721 cm^{-1}.

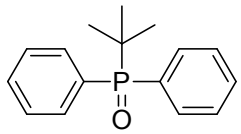


Diphenyl(4-(thiophen-2-yl)phenyl)phosphine oxide (3o). Pale yellow oil; ^{1}H NMR (400 MHz $CDCl_3$): δ 7.72–7.64 (m, 8H), 7.55 (t, J = 7.6 Hz, 2H), 7.49–7.45 (m, 4H), 7.39 (d, J = 3.6 Hz, 1H), 7.33 (d, J = 4.8 Hz, 1H), 7.09 (dd, J = 4.4 Hz, J = 4.0 Hz, 1H). ^{13}C NMR (100 MHz $CDCl_3$): δ 142.9 (d, $J_{C\text{-}P}$ = 1.2 Hz), 137.8 (d, $J_{C\text{-}P}$ = 2.9 Hz), 132.8 (d, J = 10.2 Hz), 132.3 (d, $J_{C\text{-}P}$ = 104.4 Hz), 132.1 (d, $J_{C\text{-}P}$ = 9.9 Hz), 132.1 (d, $J_{C\text{-}P}$ = 2.9 Hz), 130.9 (d, $J_{C\text{-}P}$ = 105.1 Hz), 128.6 (d, $J_{C\text{-}P}$ = 12.1 Hz), 128.4, 126.3, 125.7 (d, $J_{C\text{-}P}$ = 12.4 Hz), 124.5. ^{31}P NMR (162 MHz $CDCl_3$): δ 29.30. HRMS: Cal. for $C_{22}H_{17}O_1P_1S_1$ 360.0738. Found 360.0723. IR: 3055, 1433, 1181, 1117, 733, 697 cm^{-1}.

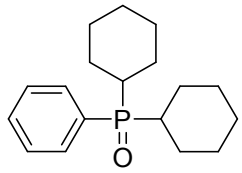


Diphenyl(4-(pyrimidin-2-yl)phenyl)phosphine oxide (3p). White solid; m.p.: 181–182 ^{o}C, ^{1}H

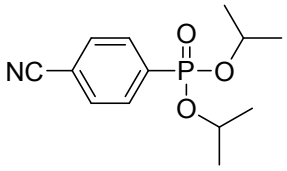
NMR (400 MHz $CDCl_3$): δ 8.83 (d, J = 4.8 Hz, 2H), 8.54 (d, J = 7.2 Hz, 2H), 7.82 (dd, J = 8.4 Hz, J = 11.4 Hz, 2H), 7.71 (dd, J = 7.6 Hz, J = 12.0 Hz, 4H), 7.56 (t, J = 7.6 Hz, 2H), 7.48 (dd, J = 6.4 Hz, J = 6.4 Hz, 4H), 7.24 (d, $J_{C\text{-}P}$ = 4.8 Hz, 1H). ^{13}C NMR (100 MHz $CDCl_3$): δ 163.8, 157.4, 140.9 (d, $J_{C\text{-}P}$ = 2.8 Hz), 134.8 (d, $J_{C\text{-}P}$ = 102.4 Hz), 132.4 (d, $J_{C\text{-}P}$ = 10.0 Hz), 132.4 (d, $J_{C\text{-}P}$ = 103.8 Hz), 132.1 (d, $J_{C\text{-}P}$ = 9.8 Hz), 132.1, 128.6 (d, $J_{C\text{-}P}$ = 12.1 Hz), 128.1 (d, $J_{C\text{-}P}$ = 12.2 Hz), 119.8. ^{31}P NMR (162 MHz $CDCl_3$): δ 28.96. HRMS: Cal. for $C_{22}H_{17}O_1N_2P_1$ 356.1078. Found [M-H] 355.0987. IR: 3030, 1559, 1419, 1183, 1116, 708 cm^{-1}.



Tert-butyldiphenylphosphine oxide (3q).[1] White solid; ^{1}H NMR (400 MHz $CDCl_3$): δ 7.96 (dd, J = 8.8 Hz, J = 8.8 Hz, 4H), 7.54–7.46 (m, 6H), 1.25 (d, J = 15.2 Hz, 9H). ^{13}C NMR (100 MHz $CDCl_3$): δ 132.2 (d, $J_{C\text{-}P}$ = 8.1 Hz), 131.5 (d, $J_{C\text{-}P}$ = 2.6 Hz), 131.2 (d, $J_{C\text{-}P}$ = 89.8 Hz), 128.3 (d, $J_{C\text{-}P}$ = 10.8 Hz), 34.0 (d, $J_{C\text{-}P}$ = 70.5 Hz), 25.2. ^{31}P NMR (162 MHz $CDCl_3$): δ 38.73. MS (EI): 258.

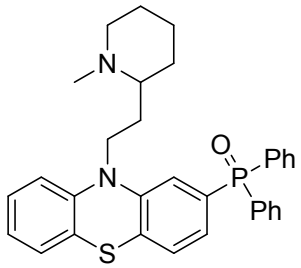


Dicyclohexyl(phenyl)phosphine oxide (3r).[5] White solid; ^{1}H NMR (400 MHz $CDCl_3$): δ 7.59 (t, J = 7.60 Hz, 2H), 7.45–7.40 (m, 3H), 1.98 (br, 4H), 1.75–1.53 (m, 8H), 1.24–1.05 (m, 10H). ^{13}C NMR (100 MHz $CDCl_3$): δ 131.5 (d, $J_{C\text{-}P}$ = 7.6 Hz), 131.2 (d, $J_{C\text{-}P}$ = 1.8 Hz), 129.9 (d, $J_{C\text{-}P}$ = 84.3 Hz), 128.3 (d, $J_{C\text{-}P}$ = 10.3 Hz), 35.1 (d, $J_{C\text{-}P}$ = 66.9 Hz), 26.5 (d, $J_{C\text{-}P}$ = 11.6 Hz), 26.4 (d, $J_{C\text{-}P}$ = 11.8 Hz), 25.8, 25.5, 24.6 (d, $J_{C\text{-}P}$ = 2.9 Hz). ^{31}P NMR (162 MHz $CDCl_3$): δ 45.40. MS (EI): 289.



Diisopropyl (4-cyanophenyl)phosphonate (3s).[6] Colorless oil; ^{1}H NMR (400 MHz $CDCl_3$): δ

7.95–7.90 (m, 2H), 7.76–7.73 (m, 2H), 4.78–4.70 (m, 2H), 1.39 (d, J = 6.0 Hz, 6H), 1.24 (d, J = 6.0 Hz, 6H). ^{13}C NMR (100 MHz $CDCl_3$): δ 135.5 (d, $J_{C\text{-}P}$ = 187.3 Hz), 132.2 (d, $J_{C\text{-}P}$ = 9.7 Hz), 131.9 (d, $J_{C\text{-}P}$ = 14.9 Hz), 118.0 (d, $J_{C\text{-}P}$ = 1.3 Hz), 115.7 (d, $J_{C\text{-}P}$ = 3.4 Hz), 71.7 (d, $J_{C\text{-}P}$ = 5.8 Hz), 24.0 (d, $J_{C\text{-}P}$ = 4.1 Hz), 23.9 (d, $J_{C\text{-}P}$ = 4.7 Hz). ^{31}P NMR (162 MHz $CDCl_3$): δ 13.11. MS (EI): 267.



(10-(2-(1-Methylpiperidin-2-yl)ethyl)-10H-phenothiazin-2-yl)diphenylphosphine oxide (3t). Colorless oil; ^{1}H NMR (400 MHz $CDCl_3$): δ 7.66 (dd, J = 7.6 Hz, J = 10.8 Hz, 4H), 7.63–7.46 (m, 6H), 7.28–7.11 (m, 4H), 7.05–7.00 (m, 1H), 7.96–7.86 (m, 2H), 3.93–3.75 (m, 2H), 2.84 (d, J = 11.6 Hz, 1H), 2.14–2.05 (m, 6H), 1.80–1.59 (m, 5H), 1.38–1.28 (m, 2H). ^{13}C NMR (100 MHz $CDCl_3$): δ 145.4 (d, $J_{C\text{-}P}$ = 14.0 Hz), 144.6, 132.9 (d, $J_{C\text{-}P}$ = 6.8 Hz), 132.1, 132.0 (d, $J_{C\text{-}P}$ = 10.0 Hz), 132.0, 131.9 (d, $J_{C\text{-}P}$ = 3.4 Hz), 131.3 (d, $J_{C\text{-}P}$ = 100.9 Hz), 130.9 (d, $J_{C\text{-}P}$ = 2.9 Hz), 128.6 (d, $J_{C\text{-}P}$ = 12.1 Hz), 128.1 (d, $J_{C\text{-}P}$ = 105.7 Hz), 127.7 (d, $J_{C\text{-}P}$ = 15.3 Hz), 127.3 (d, $J_{C\text{-}P}$ = 14.2 Hz), 127.3 (d, $J_{C\text{-}P}$ = 8.8 Hz), 126.1 (d, $J_{C\text{-}P}$ = 10.8 Hz), 124.2, 123.1, 118.3 (d, $J_{C\text{-}P}$ = 11.1 Hz), 116.0, 62.0, 57.0, 43.9, 42.5, 30.2, 29.2, 25.1, 23.7. ^{31}P NMR (162 MHz $CDCl_3$): δ 29.50. HRMS: Cal. for $C_{32}H_{33}O_1N_2P_1S_1$ 524.2051. Found 524.2044. IR: 3055, 2931, 2852, 1458, 1402, 1180, 1118, 751, 697 cm^{-1}.

5. Rerferences

(1) J.-S. Zhang, T. Chen, J. Yang, L.-B. Han, *Chem. Comm.,* 2015, **51**, 7540.

(2) X. Zhang, H. Liu, X. Hu, G. Tang, J. Zhu, Y. Zhao, *Org. Lett.,* 2011, **13**, 3478.

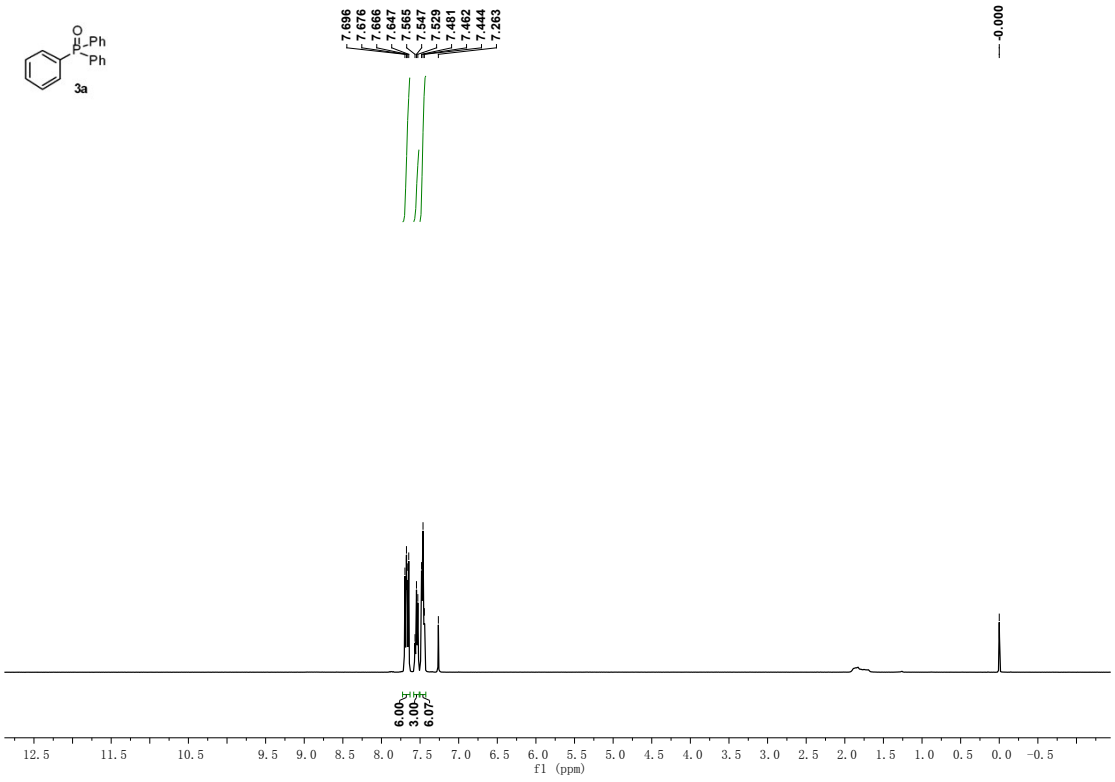
(3) Y.-L. Zhao, G.-J. Wu, F.-S. Han, *Chem. Commun.,* 2012, **48**, 5868.

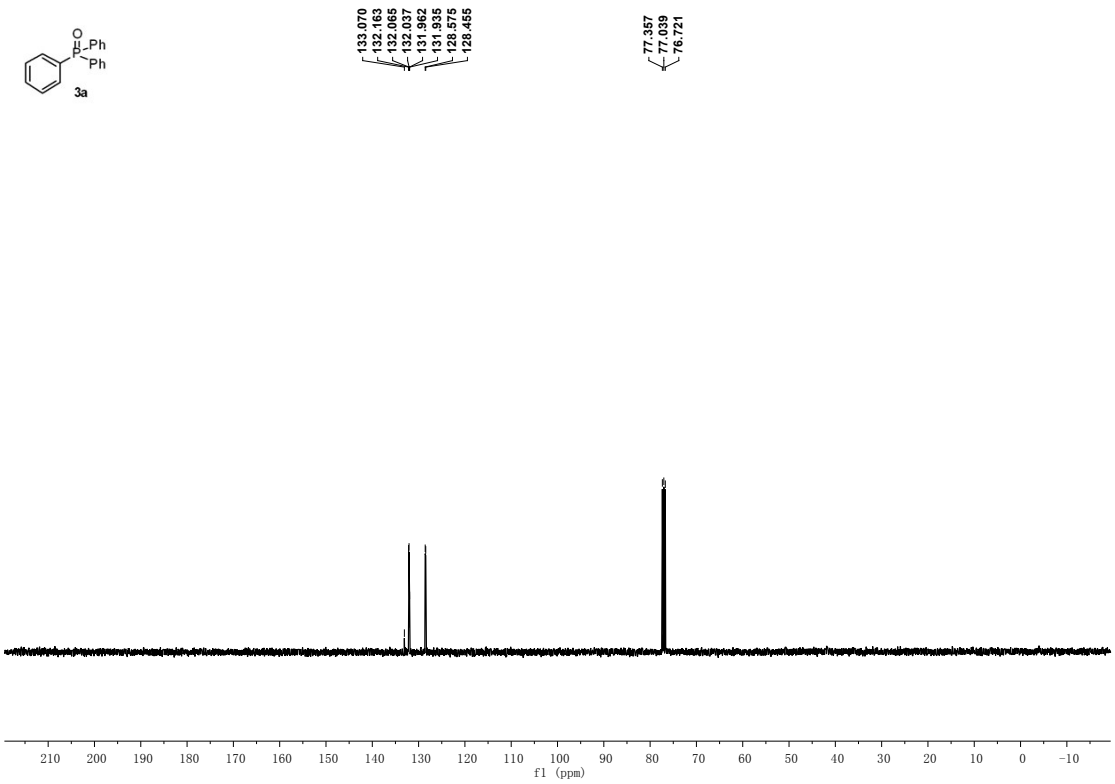
(4) Y.-L. Zhao, G.-J. Wu, Y. Li, L.-X. Gao, F.-S. Han, *Chem. Eur. J.,* 2012, **18**, 9622.

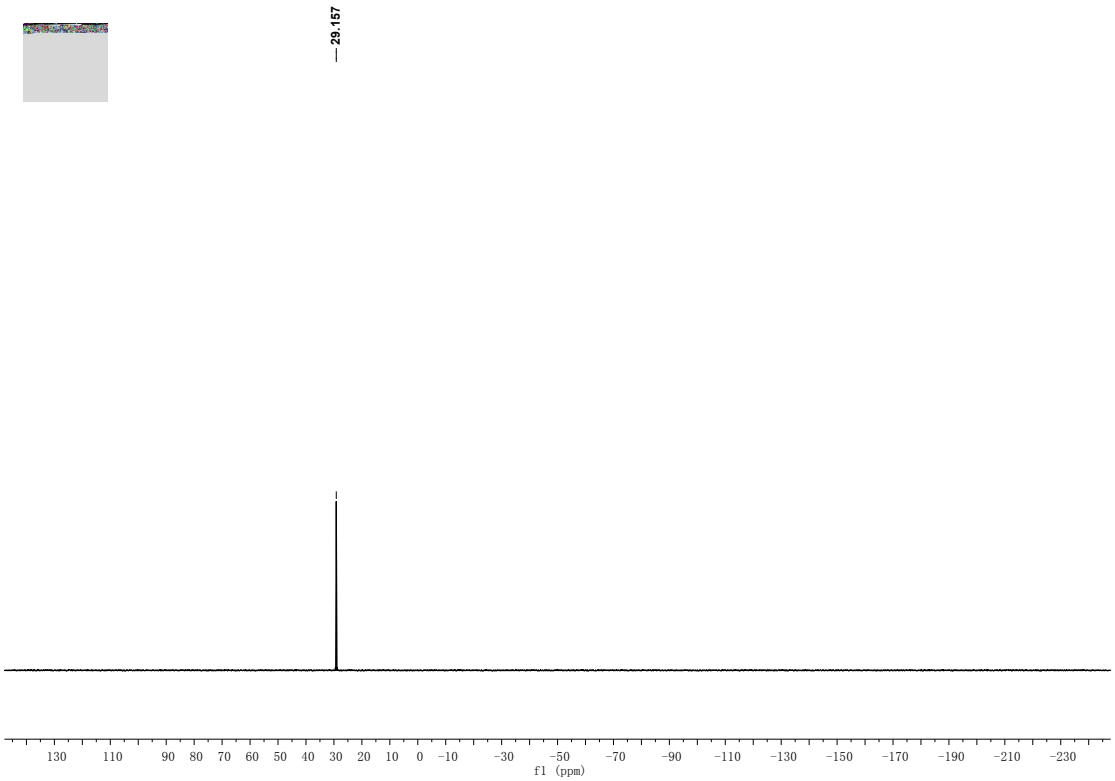
(5) R. Berrino, S. Cacchi, G. Fabrizi, A. Goggiamani, P. Stabile, *Org. Biomol. Chem.,* 2010, **8**, 4518.

(6) Belabassi, Y.; Alzghari, S.; Montchamp, J.-L. *J. Organometallic Chem.,* 2008, **693**, 3178.

6. Copies of ^{1}H NMR, ^{13}C NMR and ^{31}P NMR spectra



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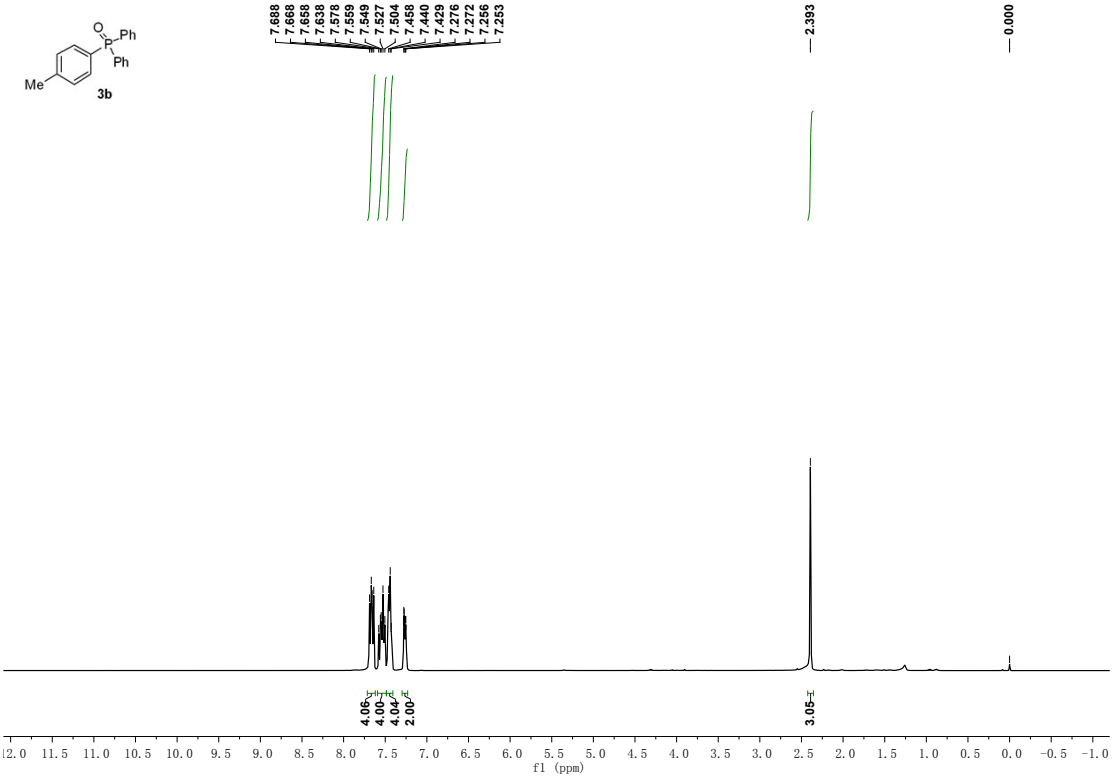
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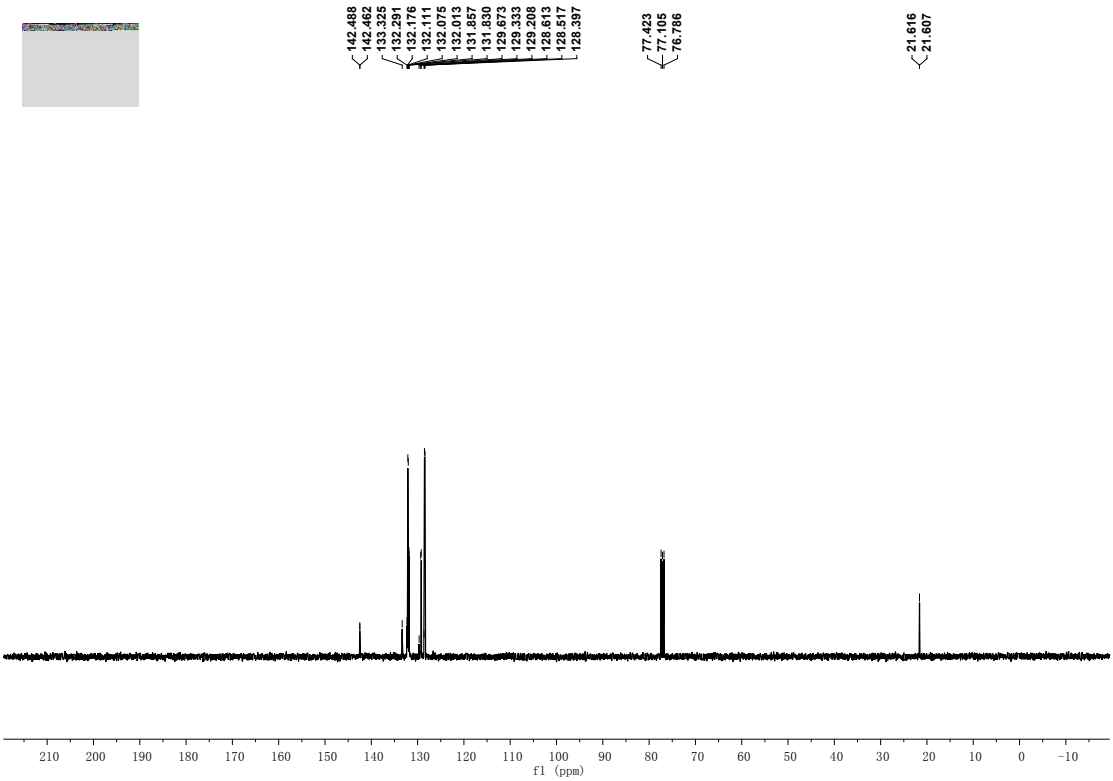
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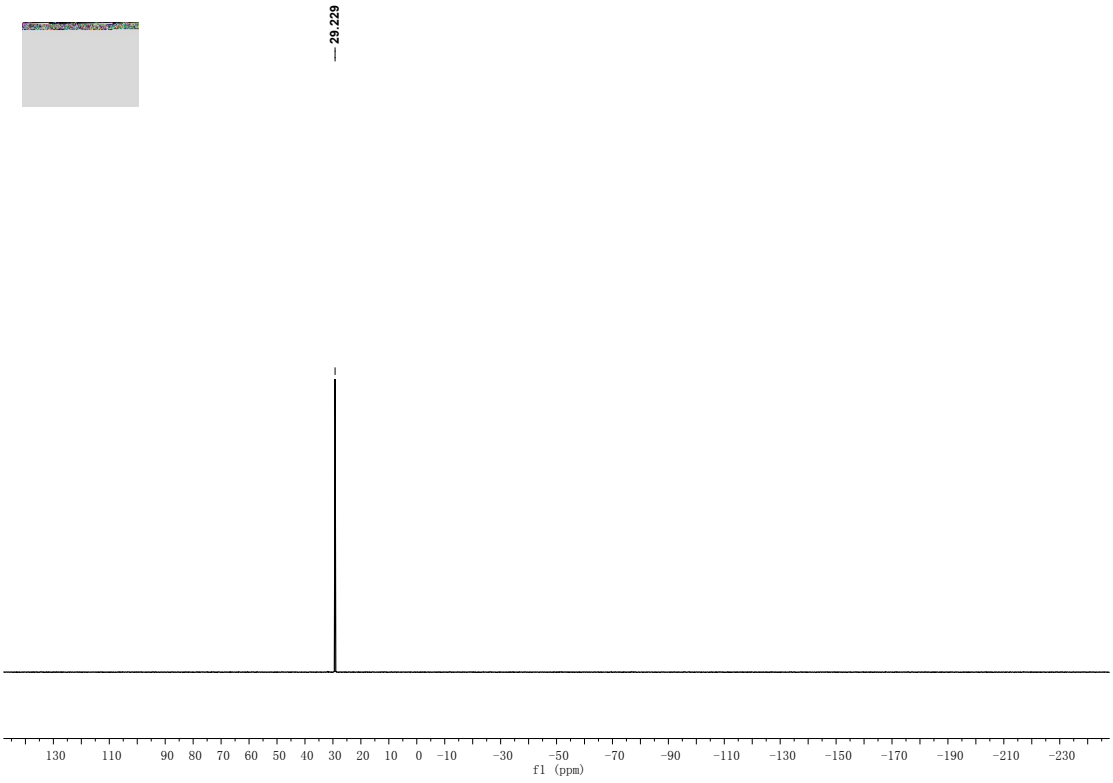
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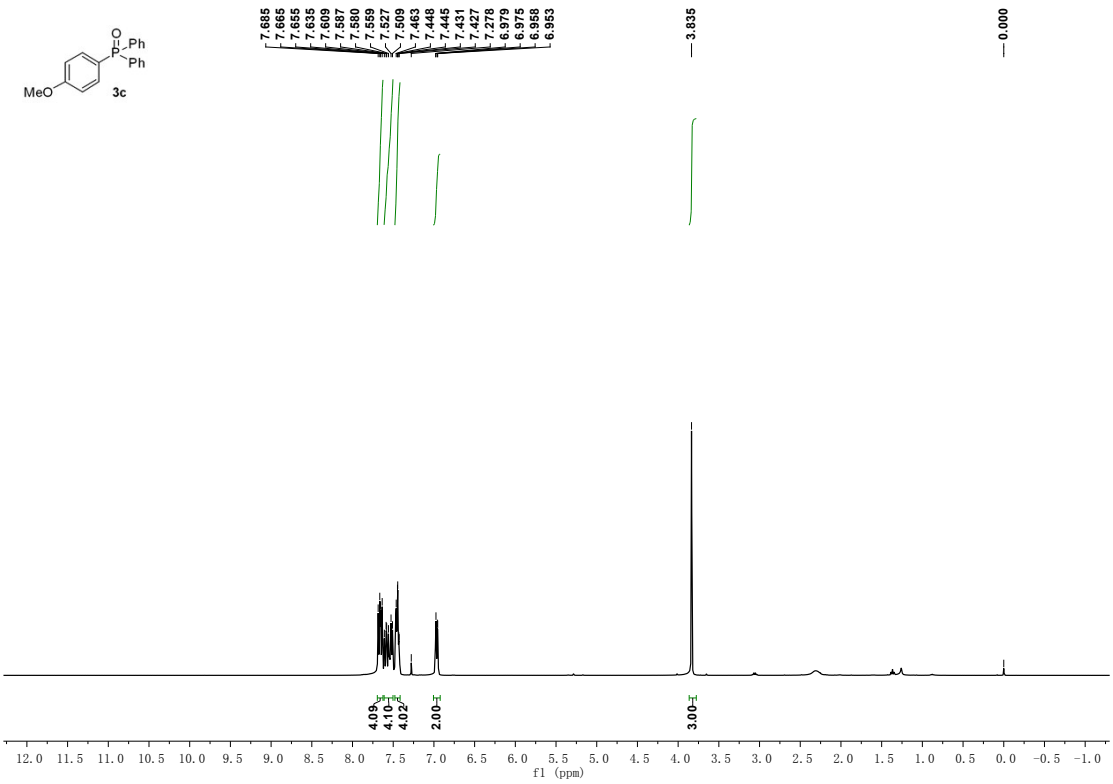
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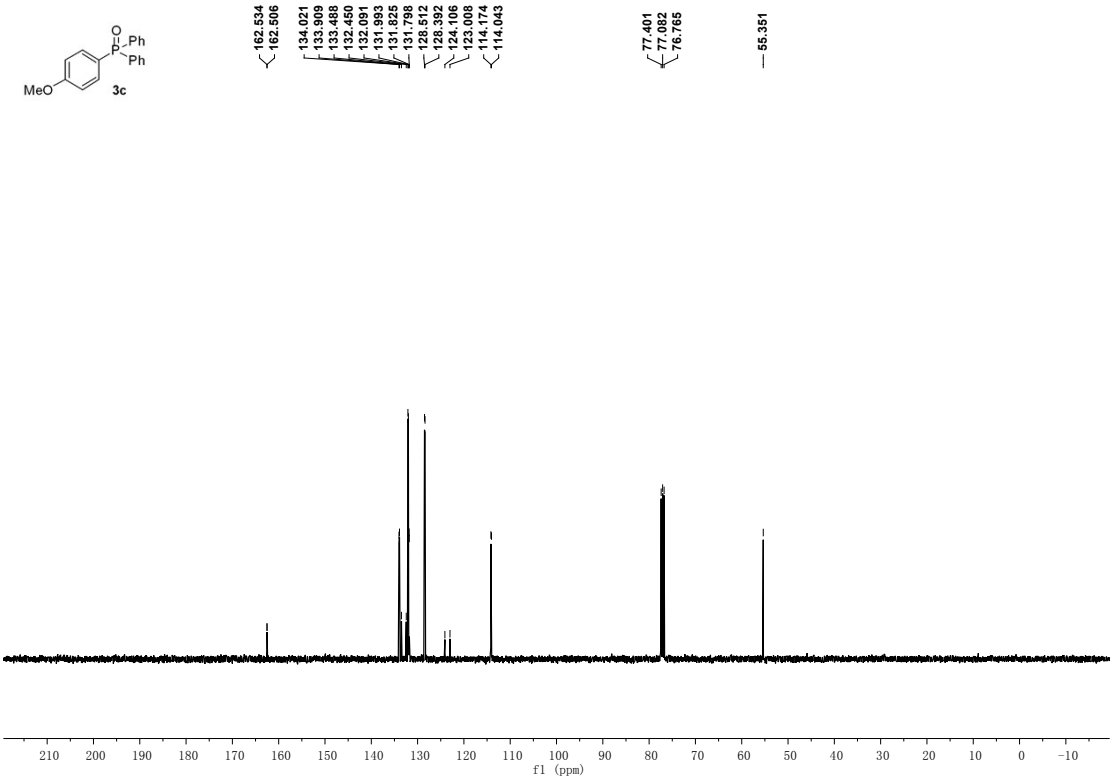
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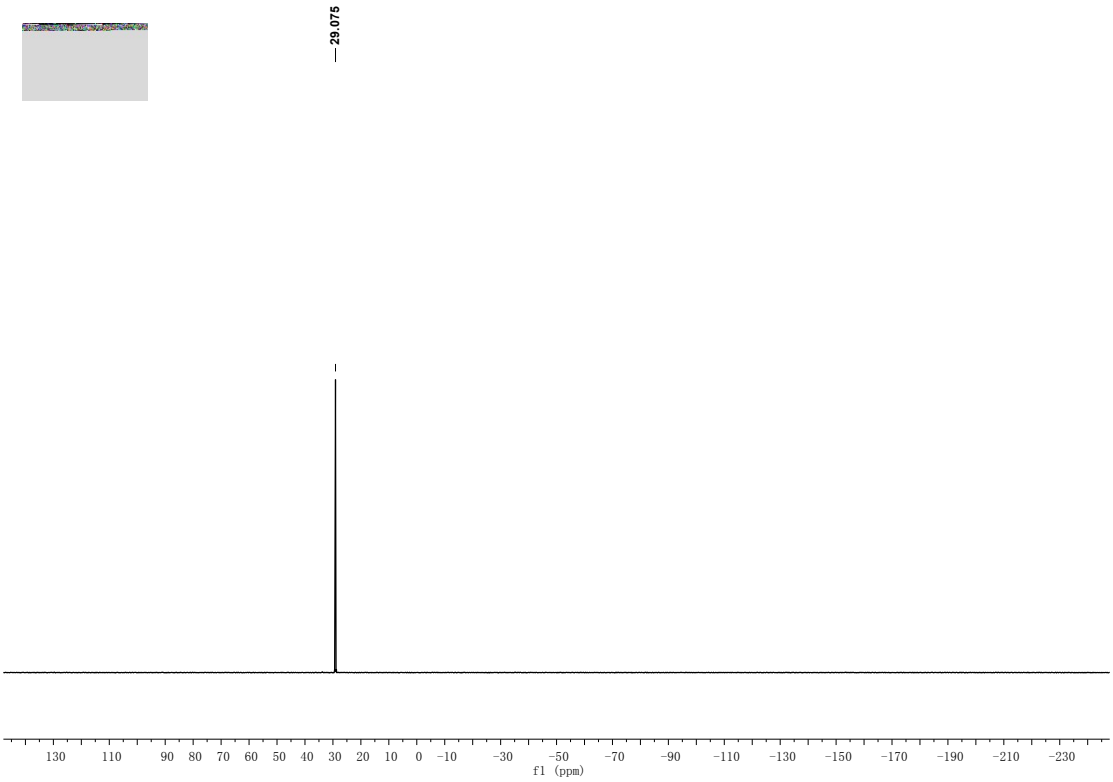
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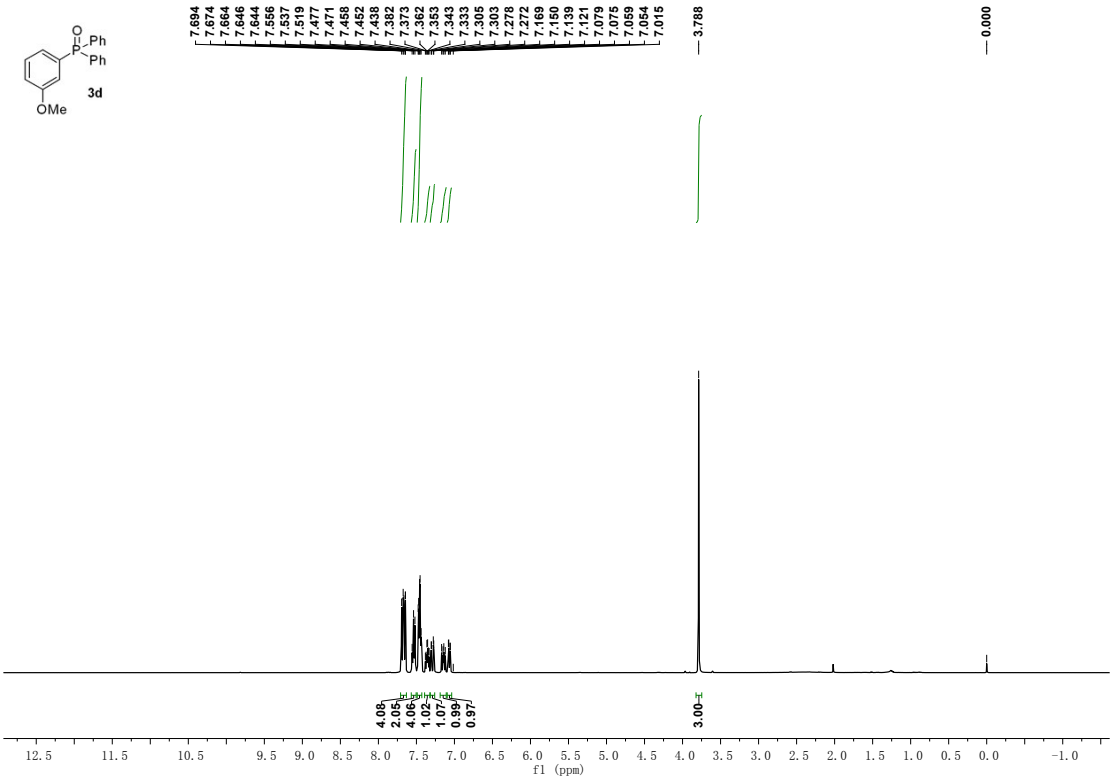
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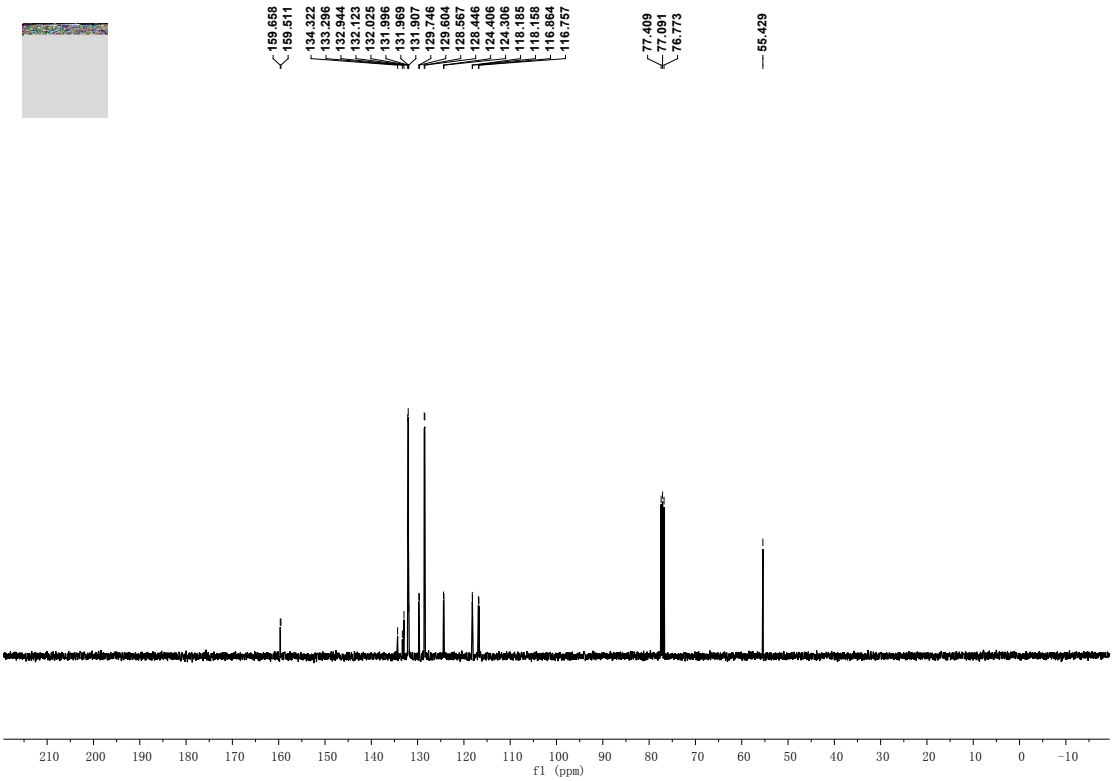
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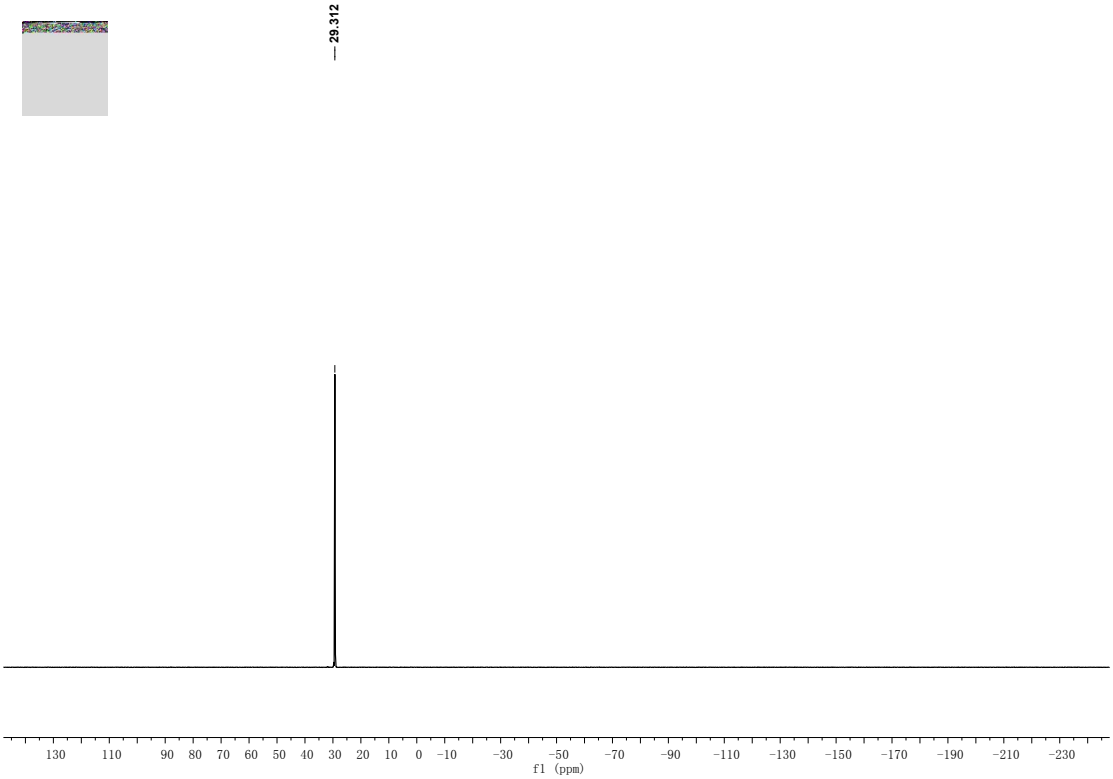
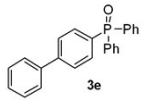
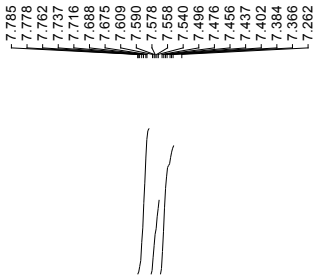
131.907
129.746
129.604
128.567
128.446
124.406
124.306
118.185
118.158
116.864
116.757
77.409
77.091
76.773
55.429
f1 (ppm)


29.312
f1 (ppm)


O
P–Ph
Ph
3e


7.785
7.778
7.762
7.737
7.716
7.688
7.675
7.609
7.590
7.578
7.558
7.540
7.496
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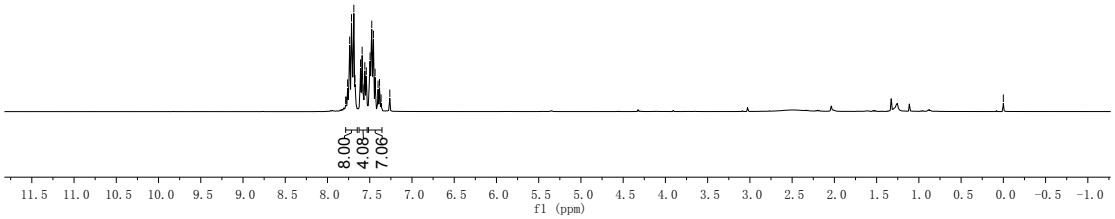
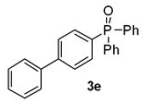
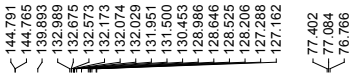
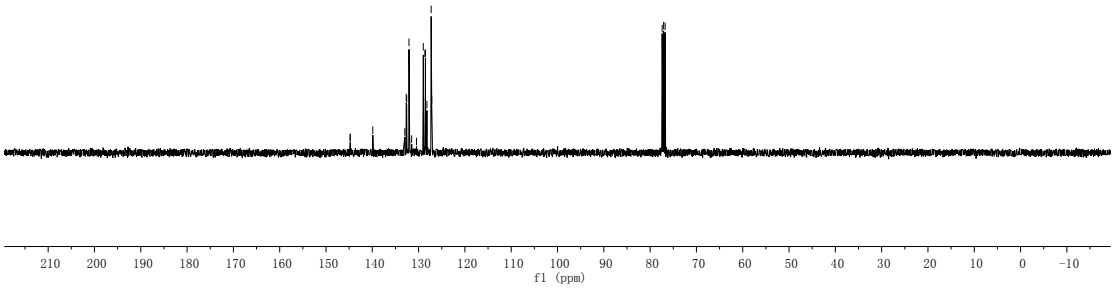
7.456
7.437
7.402
7.384
7.366
7.262

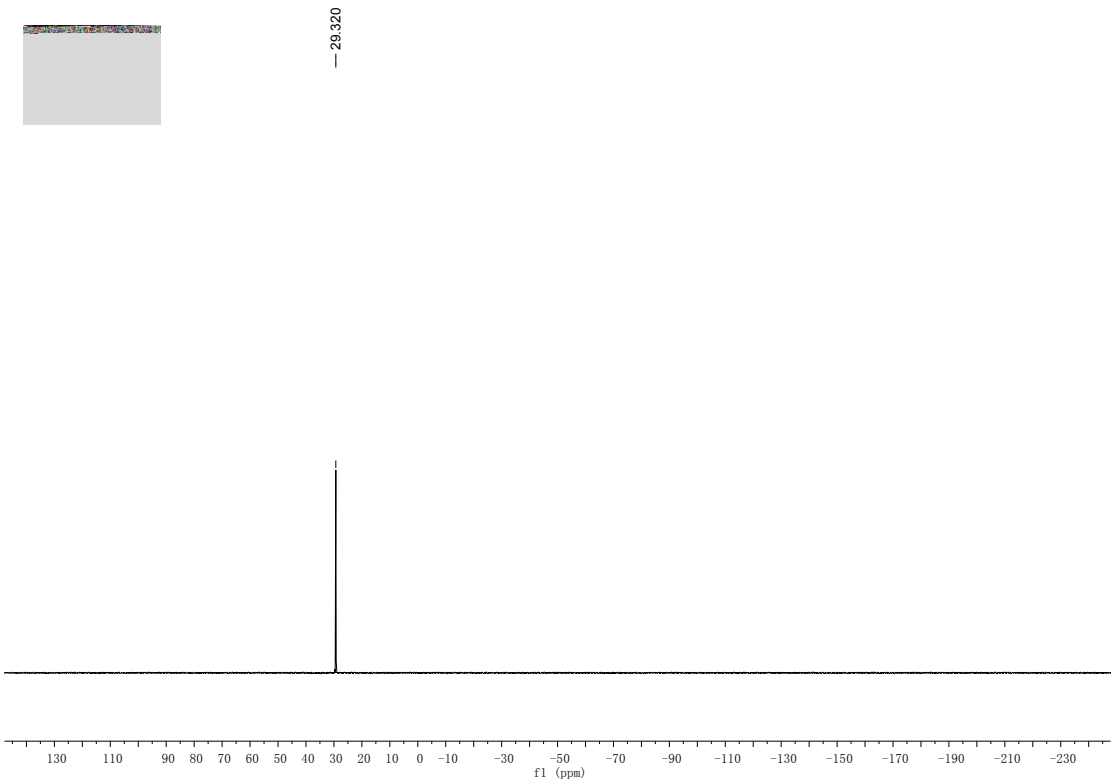

0.000

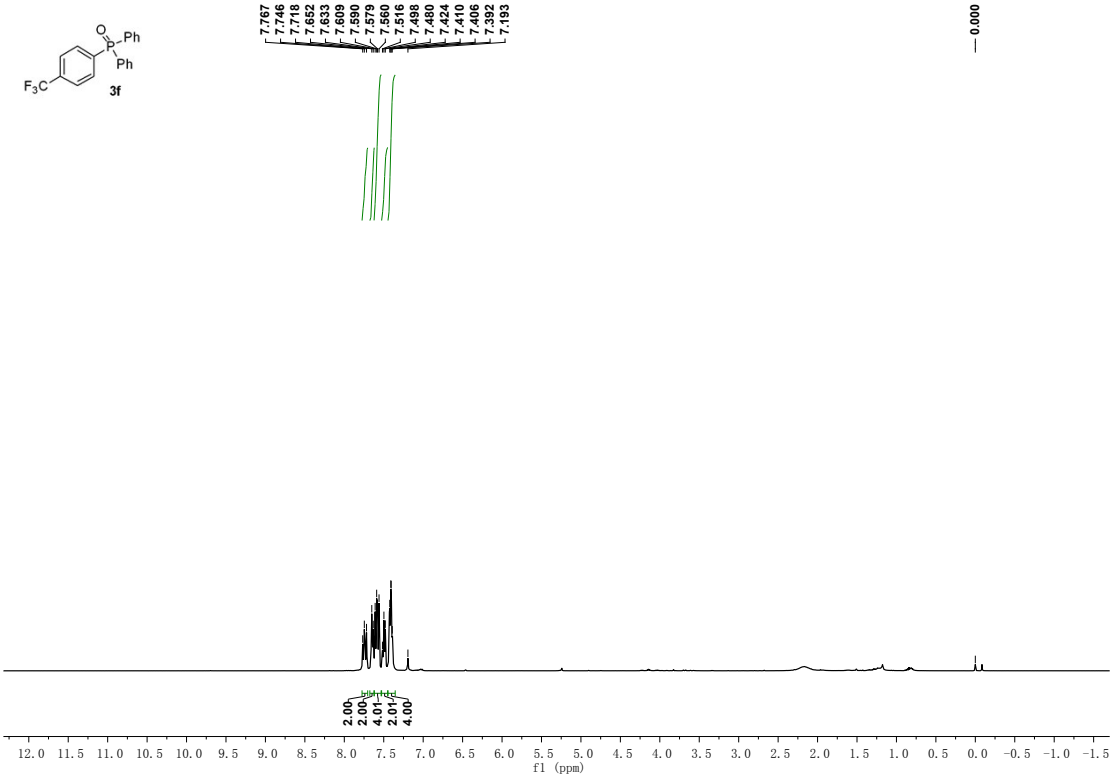

8.00
4.08
7.06
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

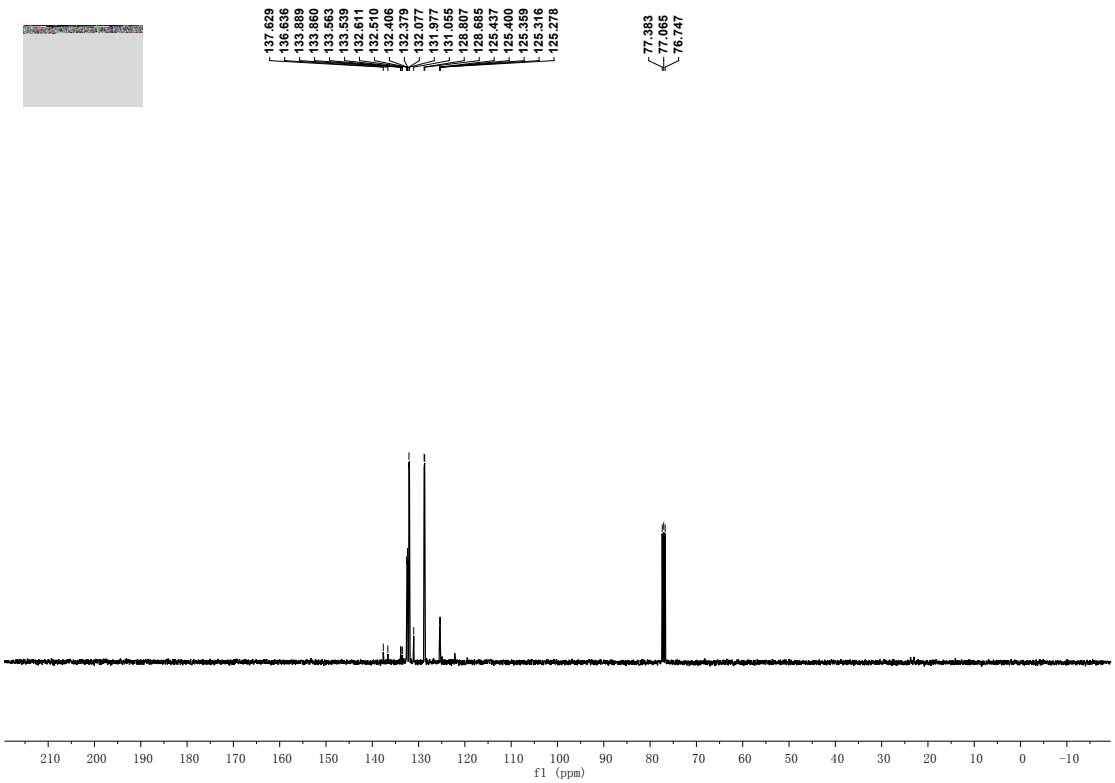

O
P–Ph
Ph
3e

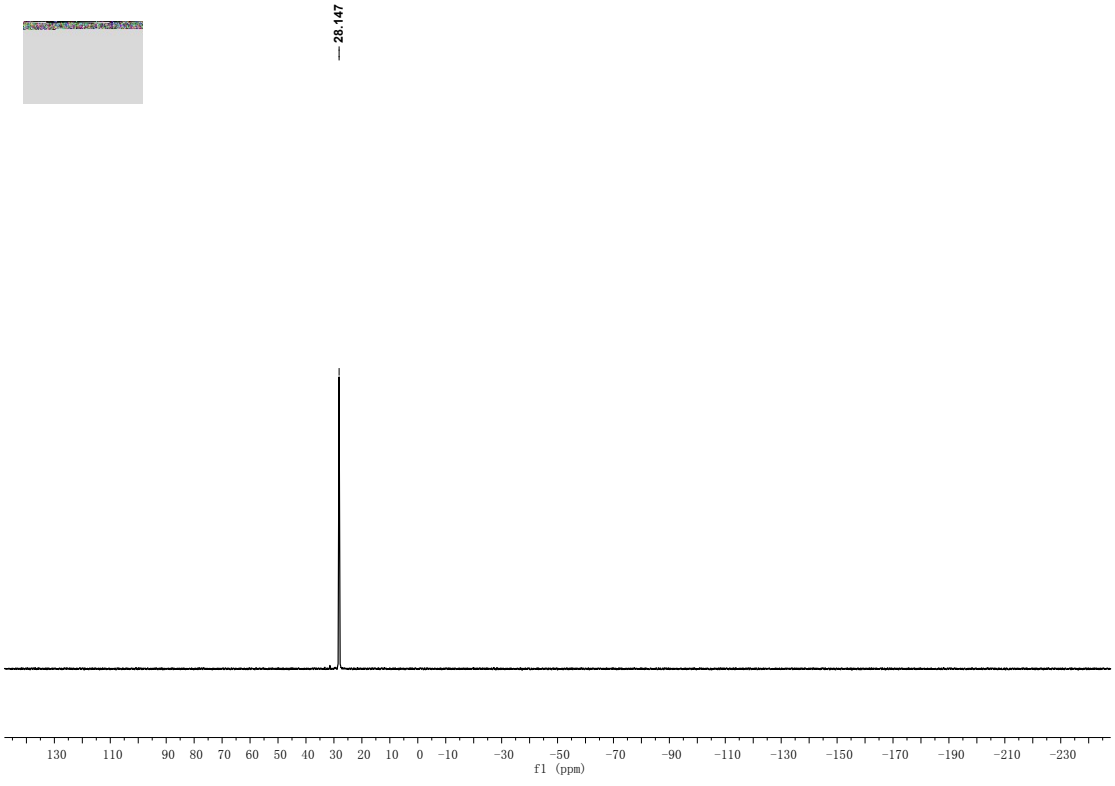

144.791
144.765
139.893
132.989
132.675
132.573
132.173
132.074
132.029
131.951
131.500
130.453
128.986
128.646
128.525
128.206
127.288
127.162
77.402
77.084
76.766

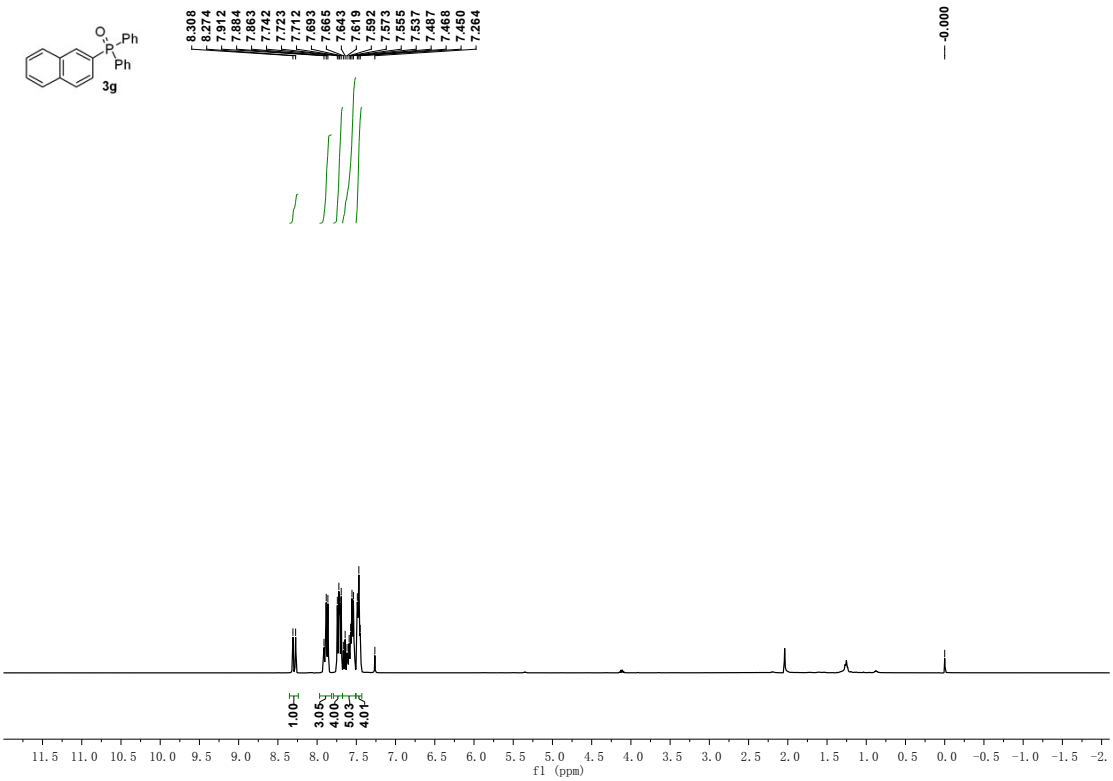

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

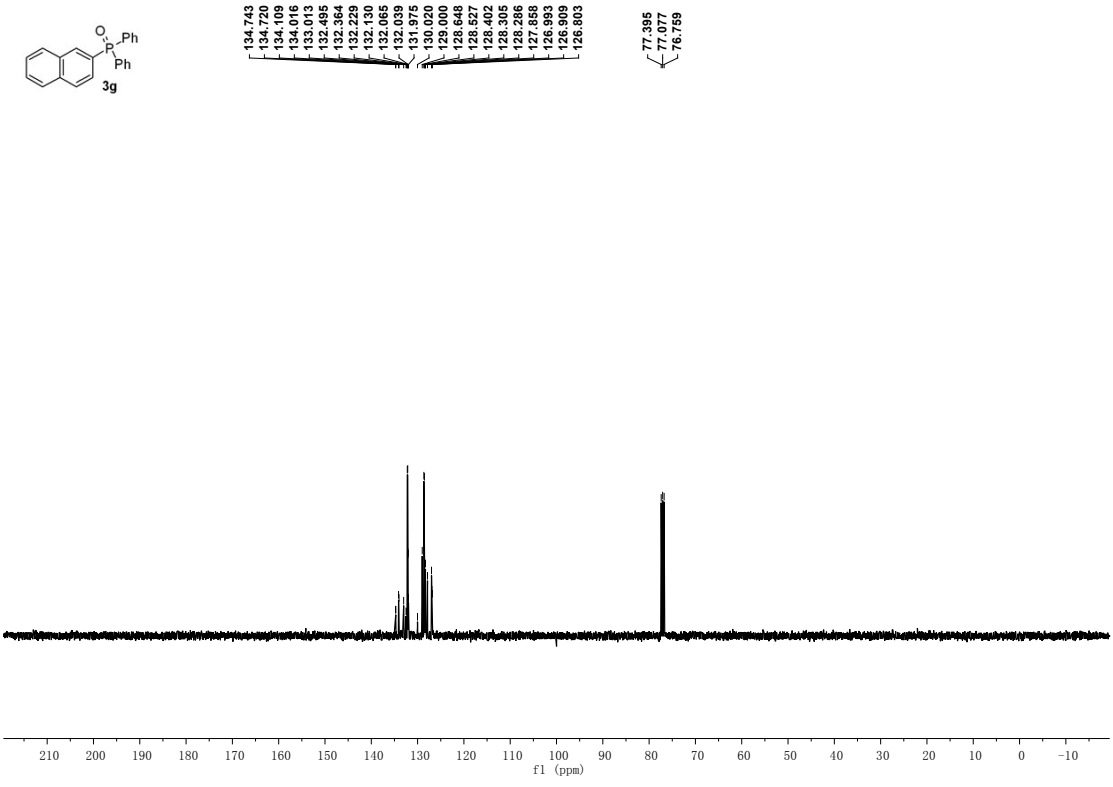

29.320
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

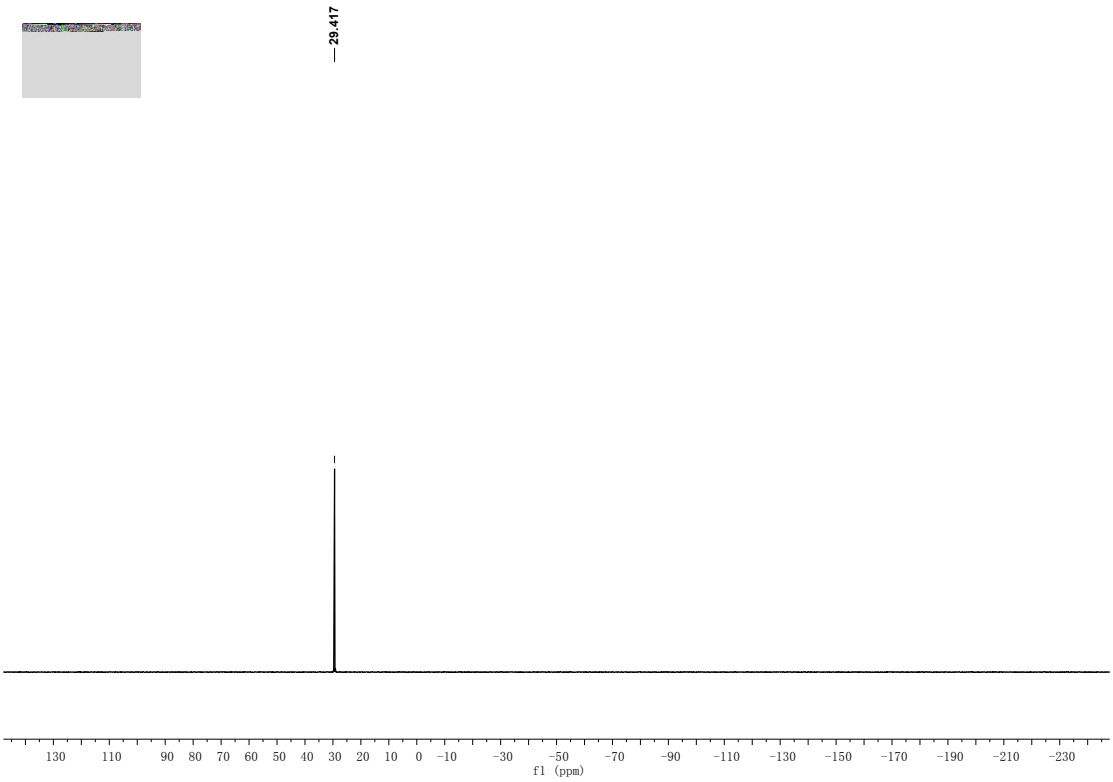

O
Ph
P
Ph
F3C
3f
7.767
7.746
7.718
7.652
7.633
7.609
7.590
7.579
7.560
7.516
7.498
7.480
7.424
7.410
7.406
7.392
7.193
0.000
2.00
2.00
4.01
2.01
4.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

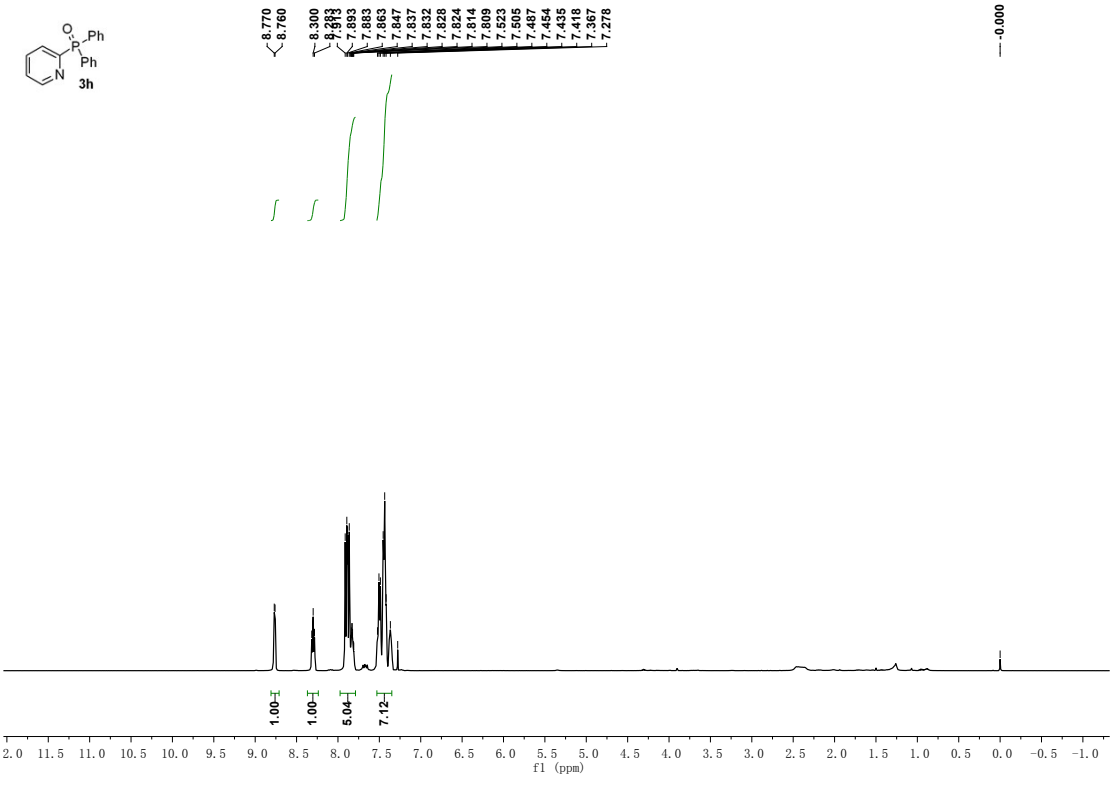

137.629
136.636
133.889
133.860
133.563
133.539
132.611
132.510
132.406
132.379
132.077
131.977
131.055
128.807
128.685
125.437
125.400
125.359
125.316
125.278
77.383
77.065
76.747
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

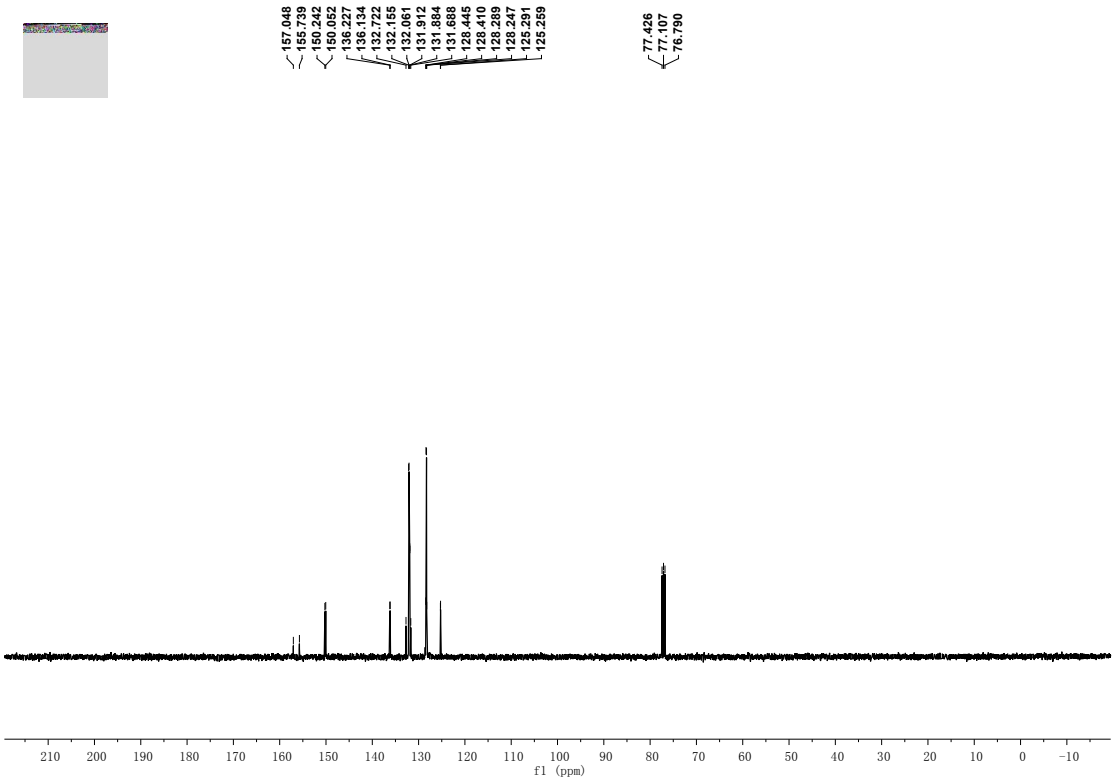

28.147
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

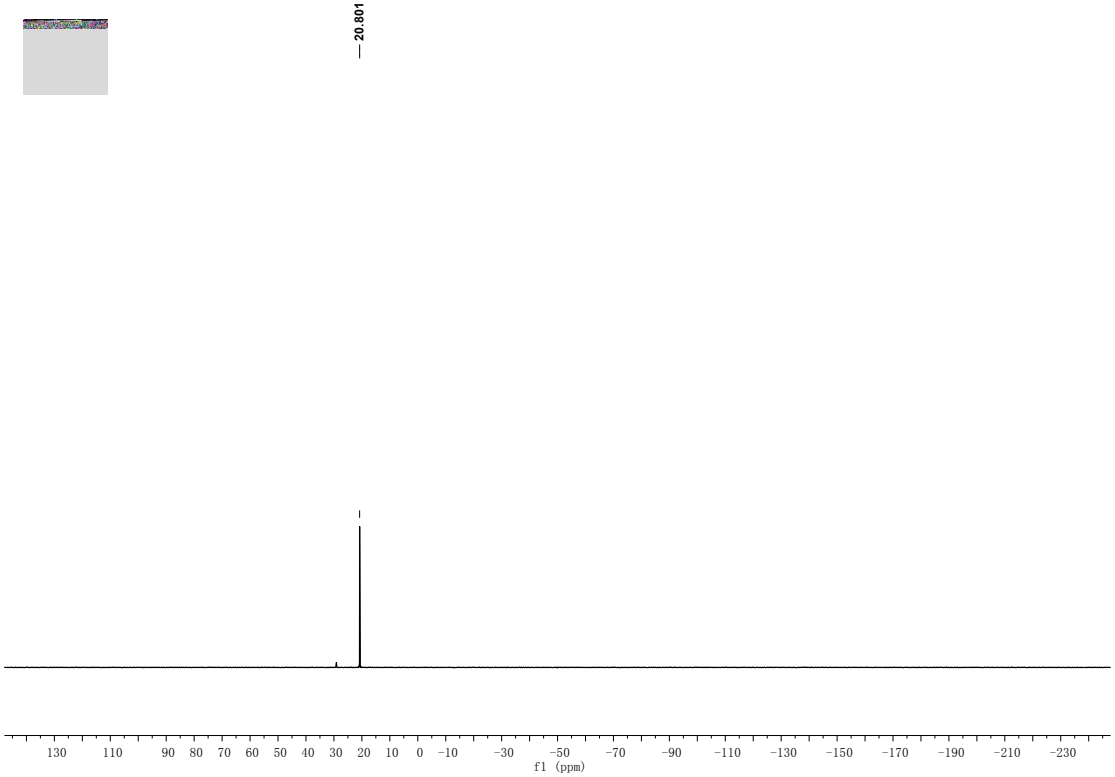

3g
8.308
8.274
7.912
7.884
7.863
7.742
7.723
7.712
7.693
7.665
7.643
7.619
7.592
7.573
7.555
7.537
7.487
7.468
7.450
7.264
-0.000
1.00
3.05
4.00
5.03
4.01
f1 (ppm)

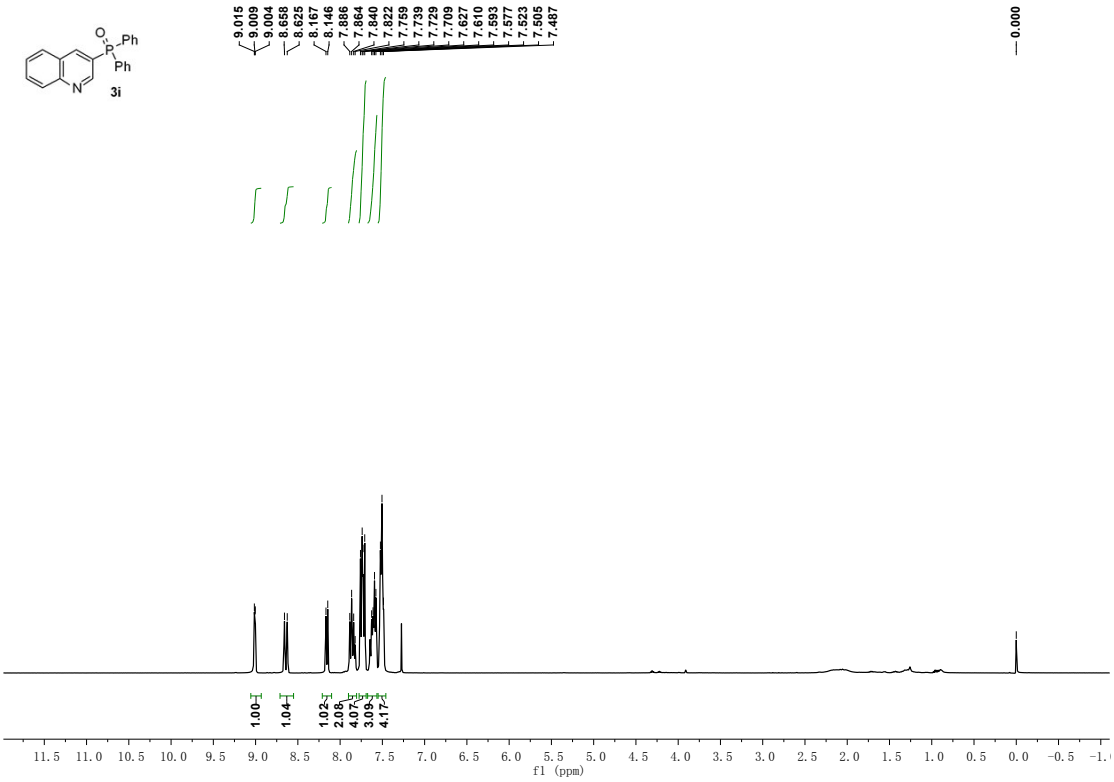

3g
134.743
134.720
134.109
134.016
133.013
132.495
132.364
132.229
132.130
132.065
132.039
131.975
130.020
129.000
128.648
128.527
128.402
128.305
128.286
127.858
126.993
126.909
126.803
77.395
77.077
76.759
f1 (ppm)

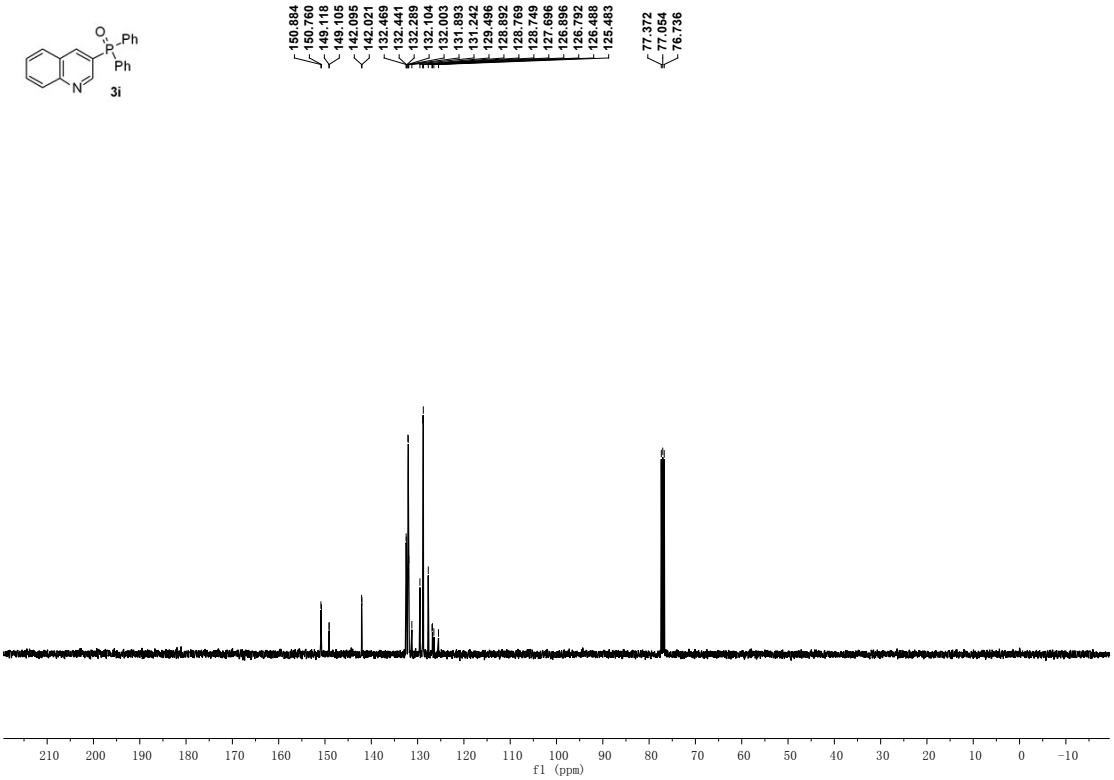

29.417
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

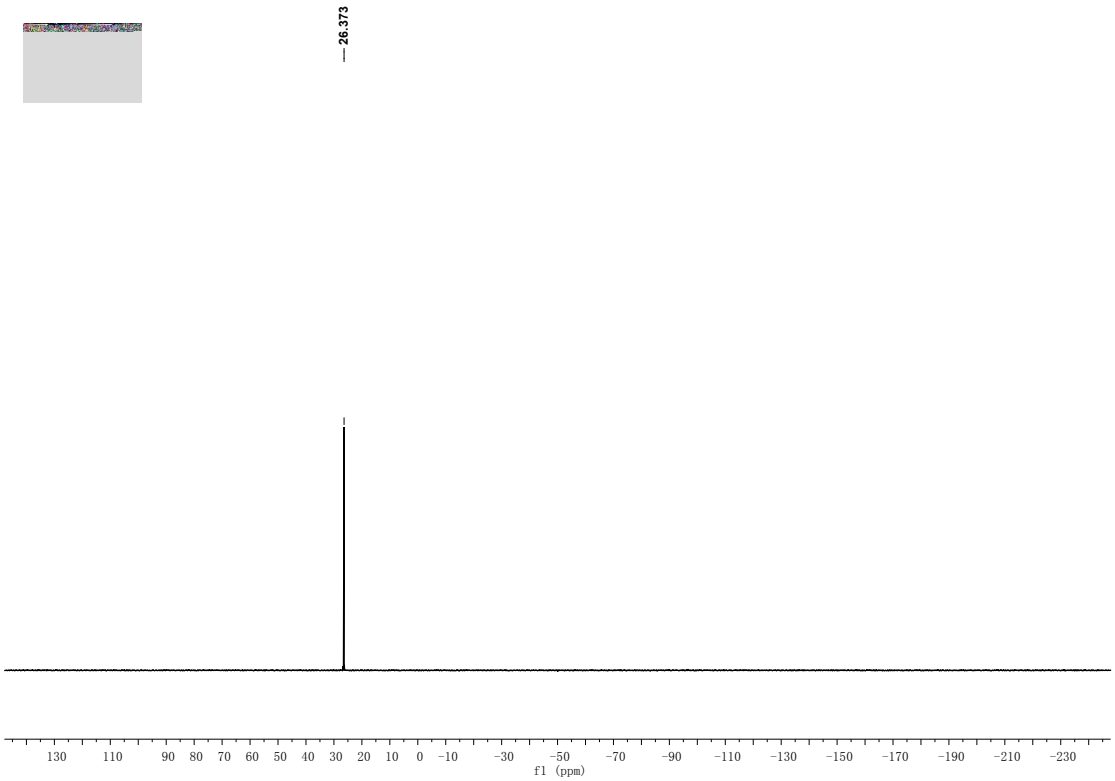

3h
8.770
8.760
8.300
7.893
7.883
7.863
7.847
7.837
7.832
7.828
7.824
7.814
7.809
7.523
7.505
7.497
7.454
7.435
7.418
7.367
7.278
0.000
1.00
1.00
5.04
7.12
2.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

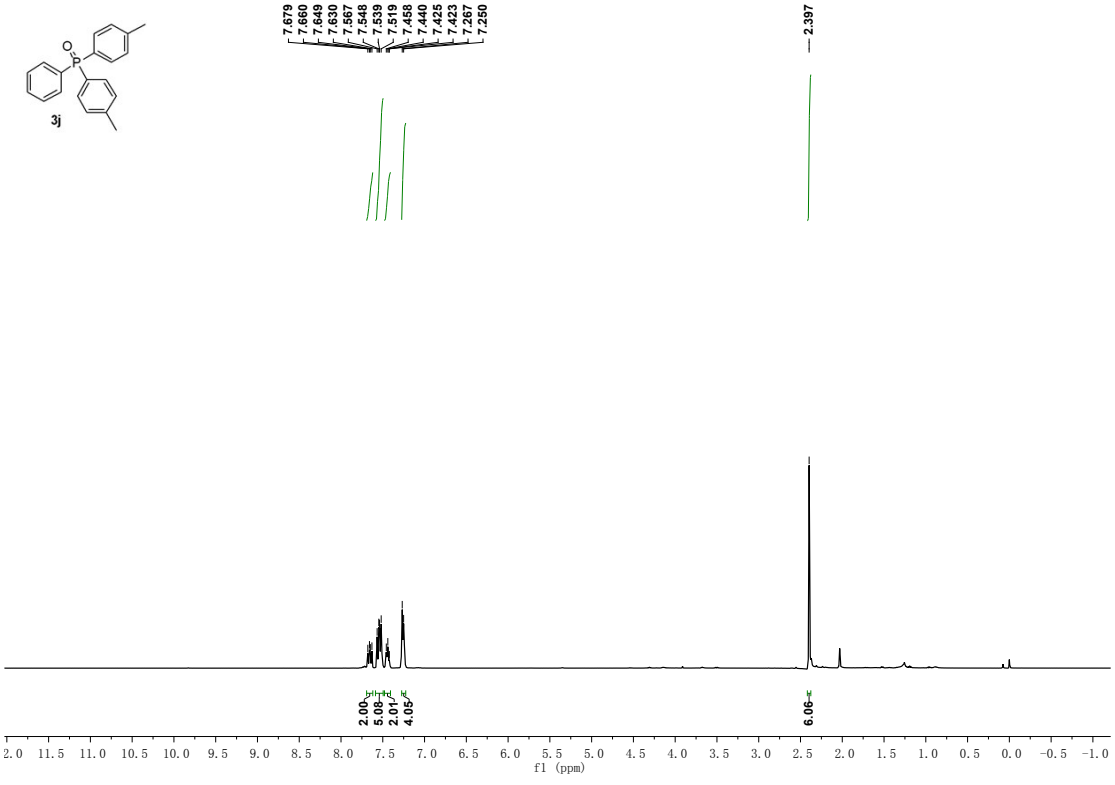

157.048
155.739
150.242
150.052
136.227
136.134
132.722
132.155
132.061
131.912
131.884
131.688
128.445
128.410
128.289
128.247
125.291
125.259
77.426
77.107
76.790
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

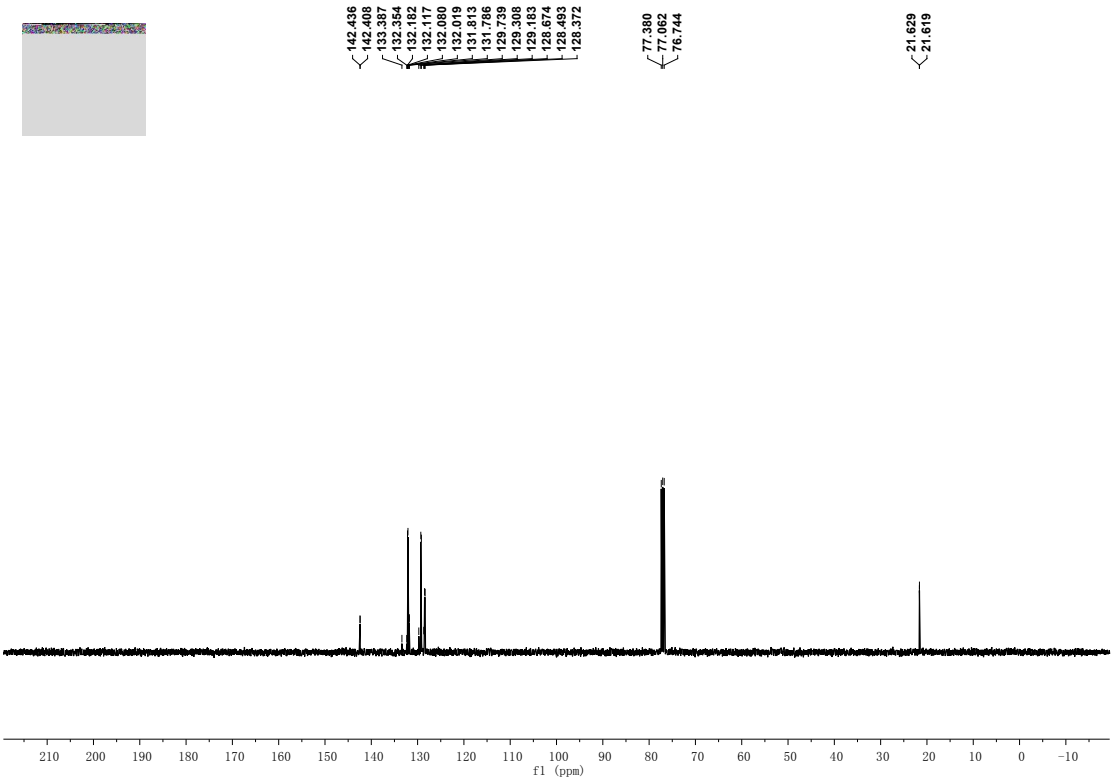

20.801
130 110 90 80 70 60 50 40 30 20 10 0 −10 −30 −50 −70 −90 −110 −130 −150 −170 −190 −210 −230
f1 (ppm)

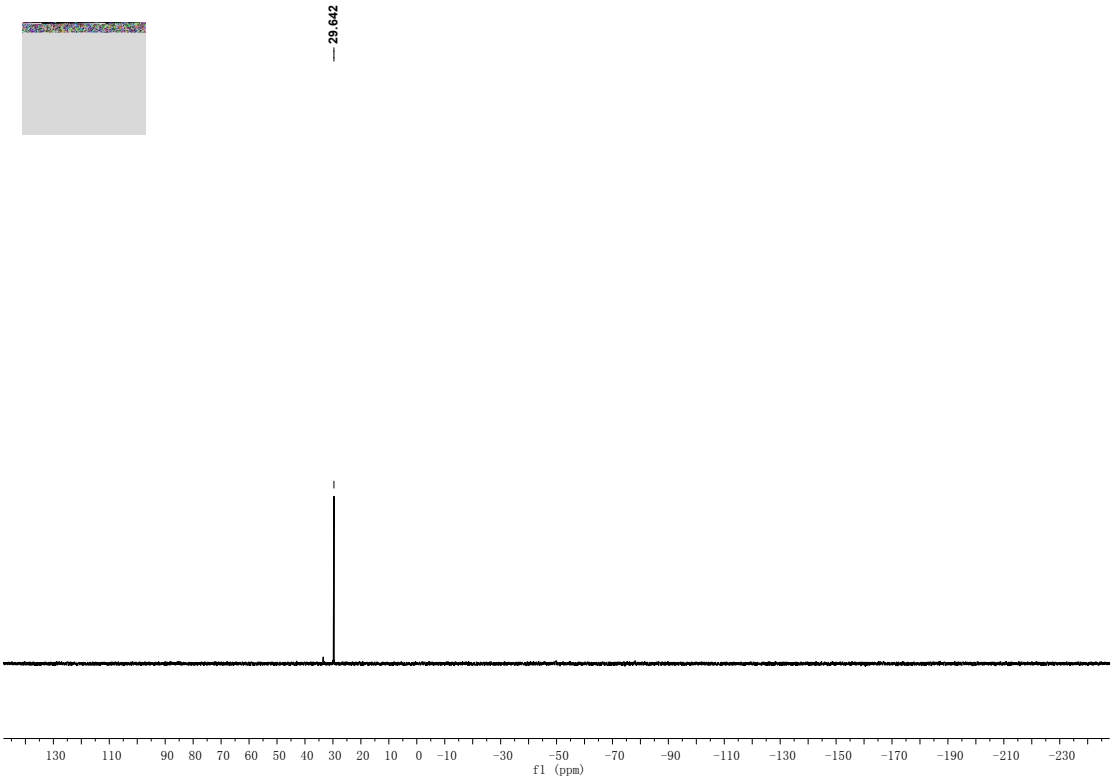

3i
9.015
9.009
9.004
8.658
8.625
8.167
8.146
7.886
7.864
7.840
7.822
7.759
7.739
7.729
7.709
7.627
7.610
7.593
7.577
7.523
7.505
7.487
0.000
1.00
1.04
1.02
2.08
4.07
3.09
4.17
f1 (ppm)

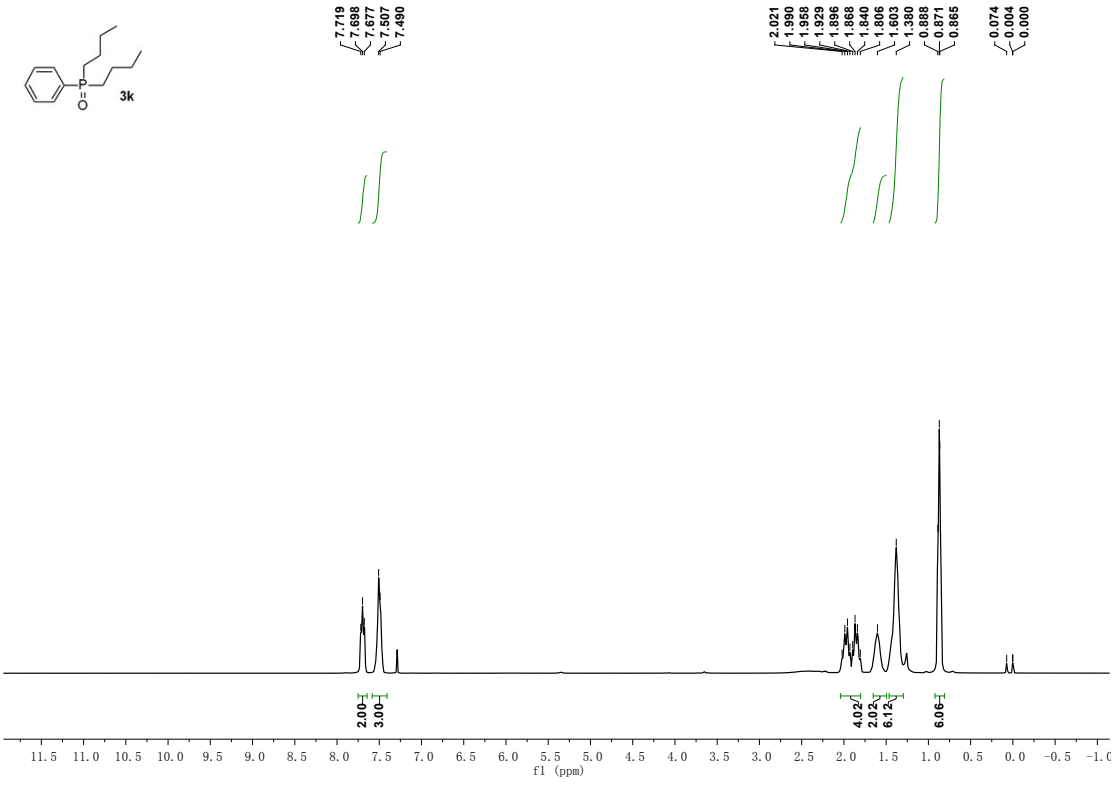

3i
150.884
150.760
149.118
149.105
142.095
142.021
132.469
132.441
132.289
132.104
132.003
131.893
131.242
129.496
128.892
128.769
128.749
127.696
126.896
126.792
126.488
125.483
77.372
77.054
76.736
f1 (ppm)

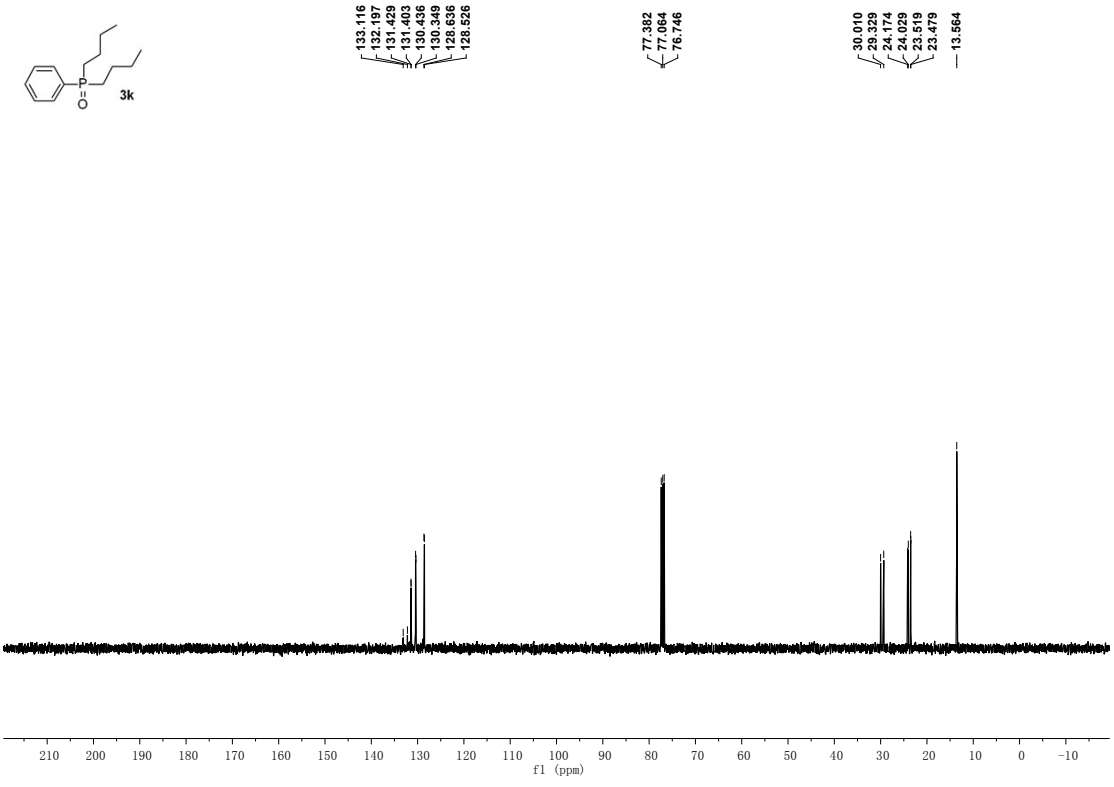

26.373
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

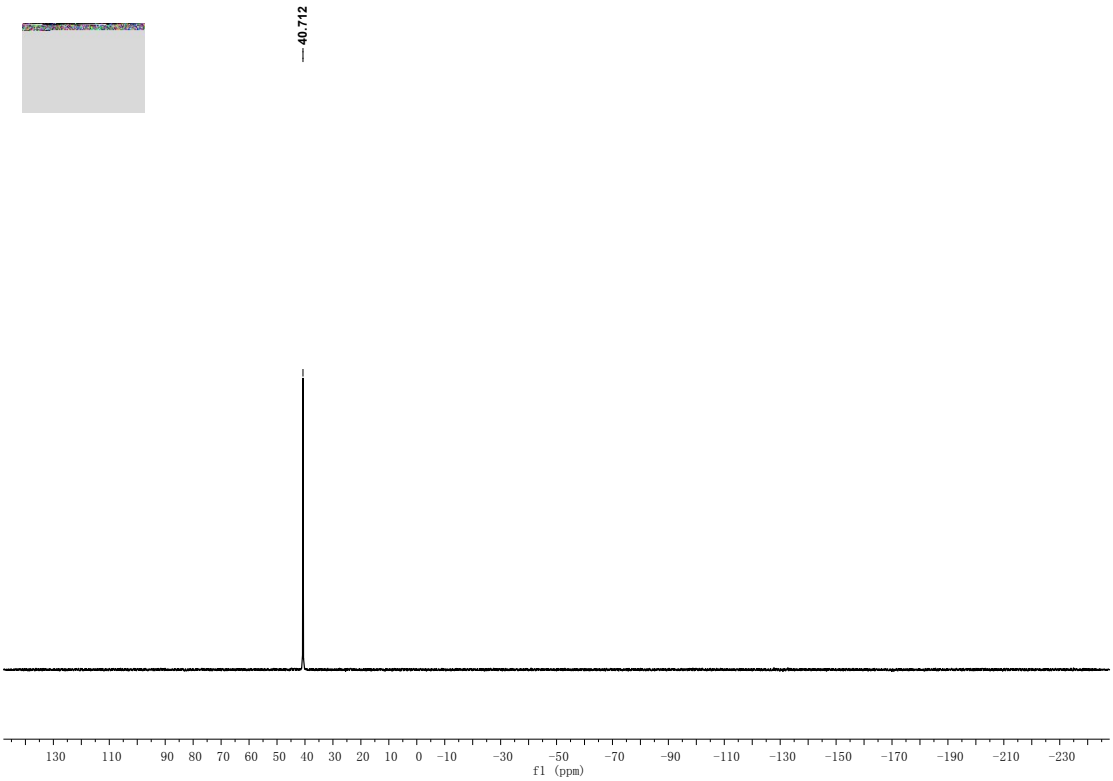

3j
7.679
7.660
7.649
7.630
7.567
7.548
7.539
7.519
7.458
7.440
7.425
7.423
7.267
7.250
2.397
2.00
5.08
2.01
4.05
6.06
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

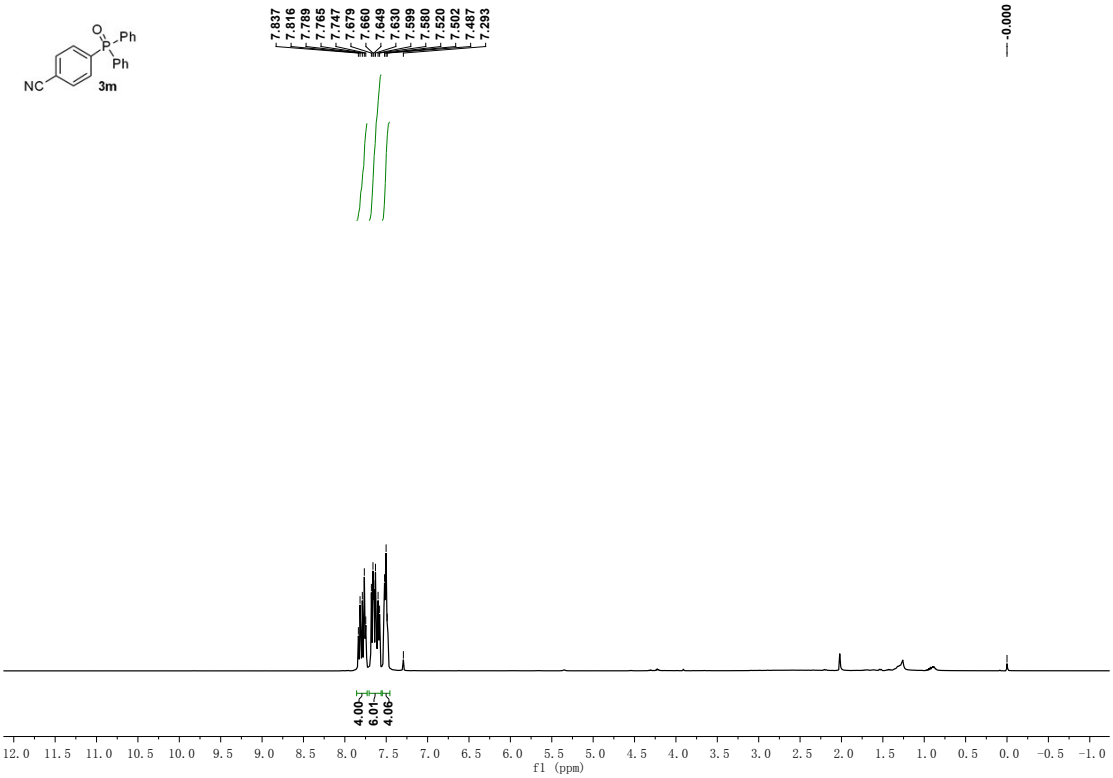

142.436
142.408
133.387
132.354
132.182
132.117
132.080
132.019
131.813
131.786
129.739
129.308
129.183
128.674
128.493
128.372
77.380
77.062
76.744
21.629
21.619
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

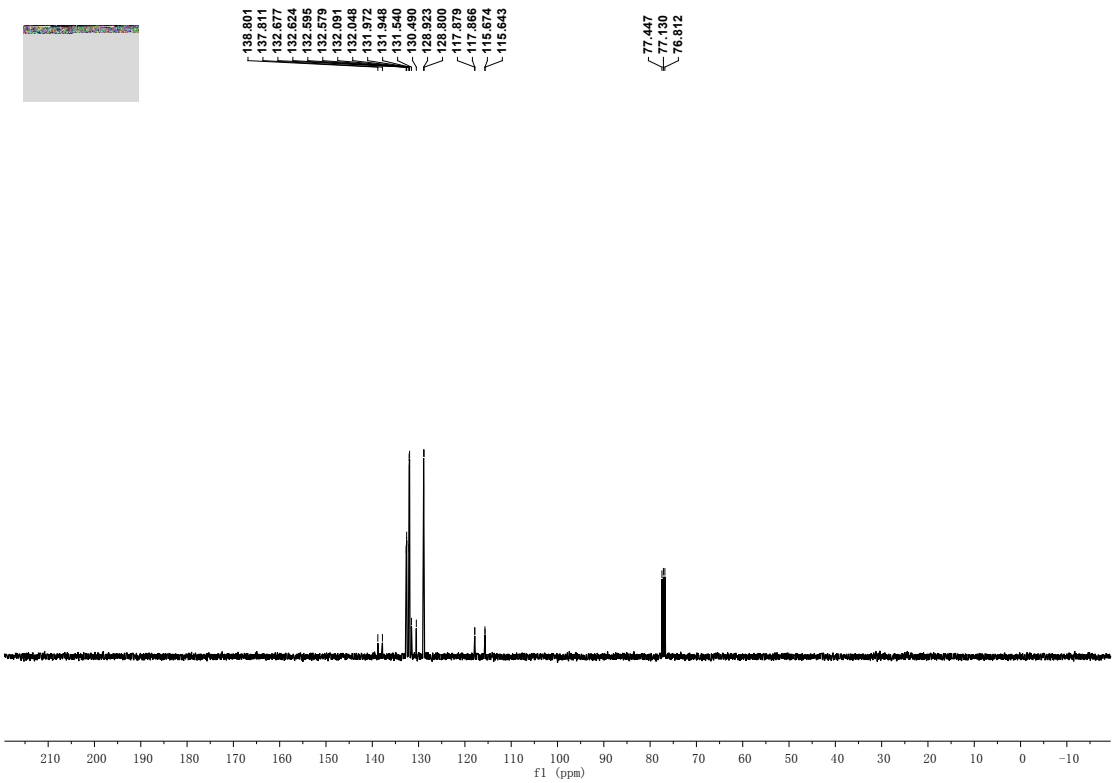

29.642
130 110 90 80 70 60 50 40 30 20 10 0 −10 −30 −50 −70 −90 −110 −130 −150 −170 −190 −210 −230
f1 (ppm)

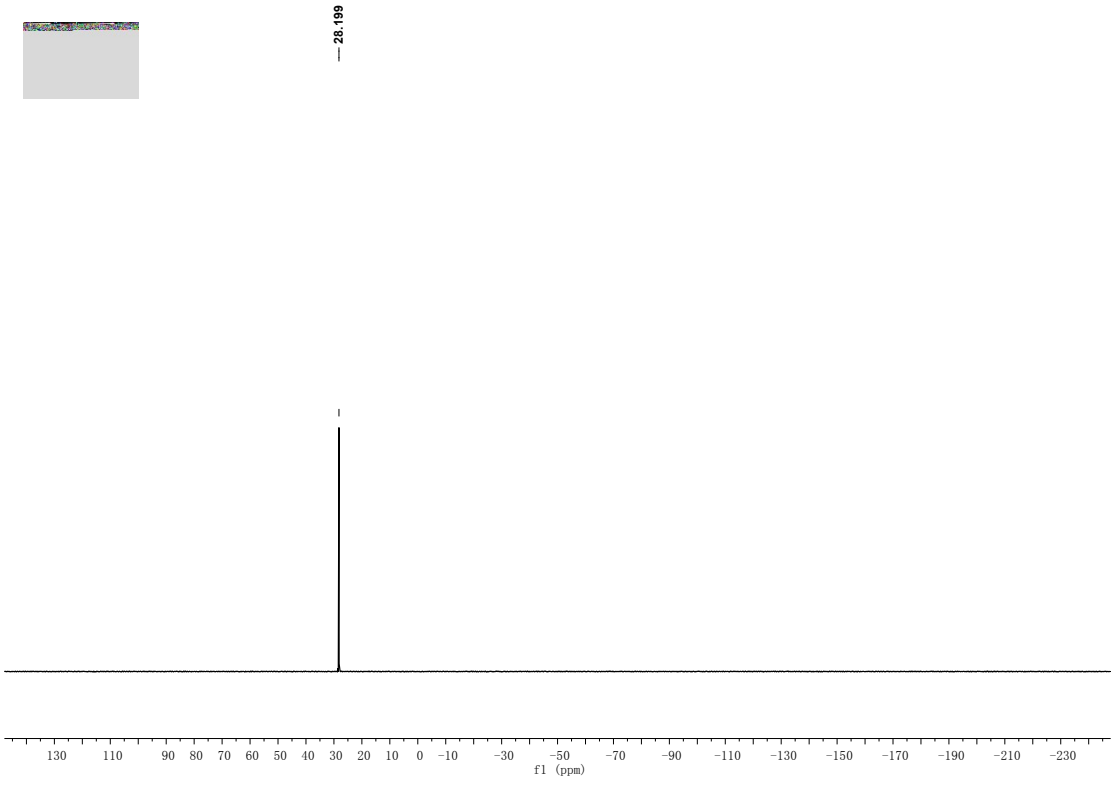

3k
7.719
7.698
7.677
7.507
7.490
2.021
1.990
1.958
1.929
1.896
1.868
1.840
1.806
1.603
1.380
0.888
0.871
0.865
0.074
0.004
0.000
2.00
3.00
4.02
2.02
6.12
6.06
f1 (ppm)

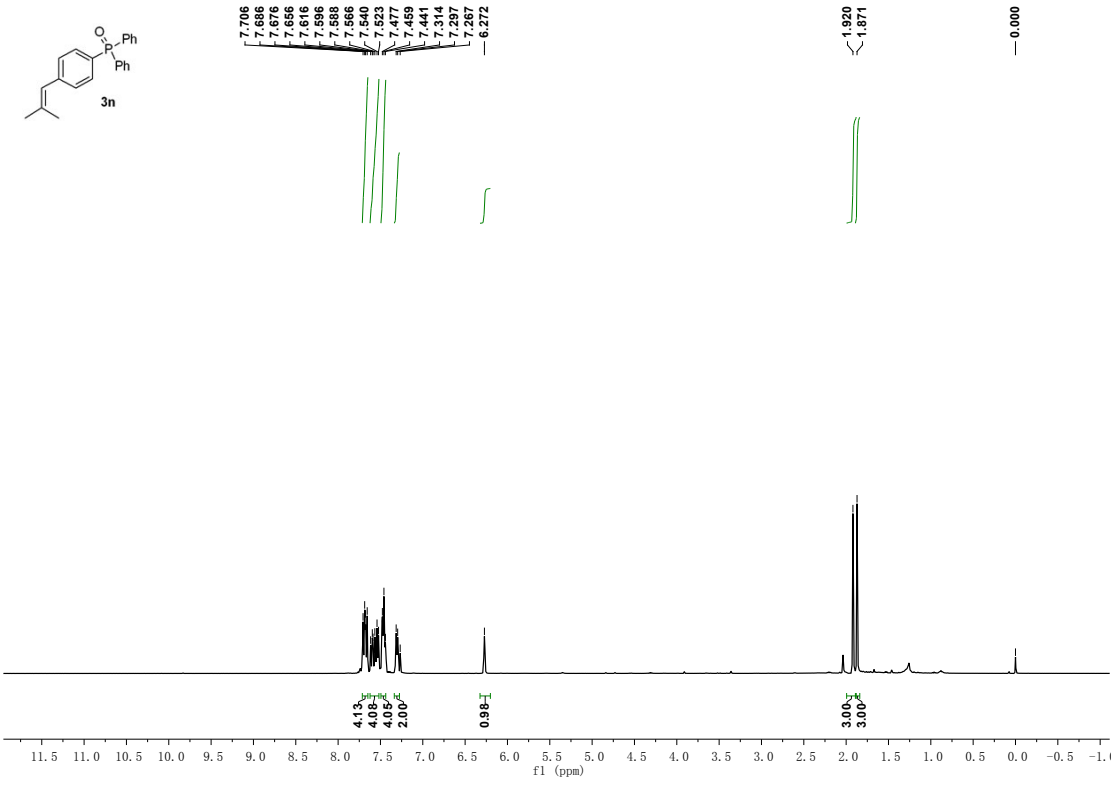

3k
133.116
132.197
131.429
131.403
130.436
130.349
128.636
128.526
77.382
77.064
76.746
30.010
29.329
24.174
24.029
23.519
23.479
13.564
f1 (ppm)

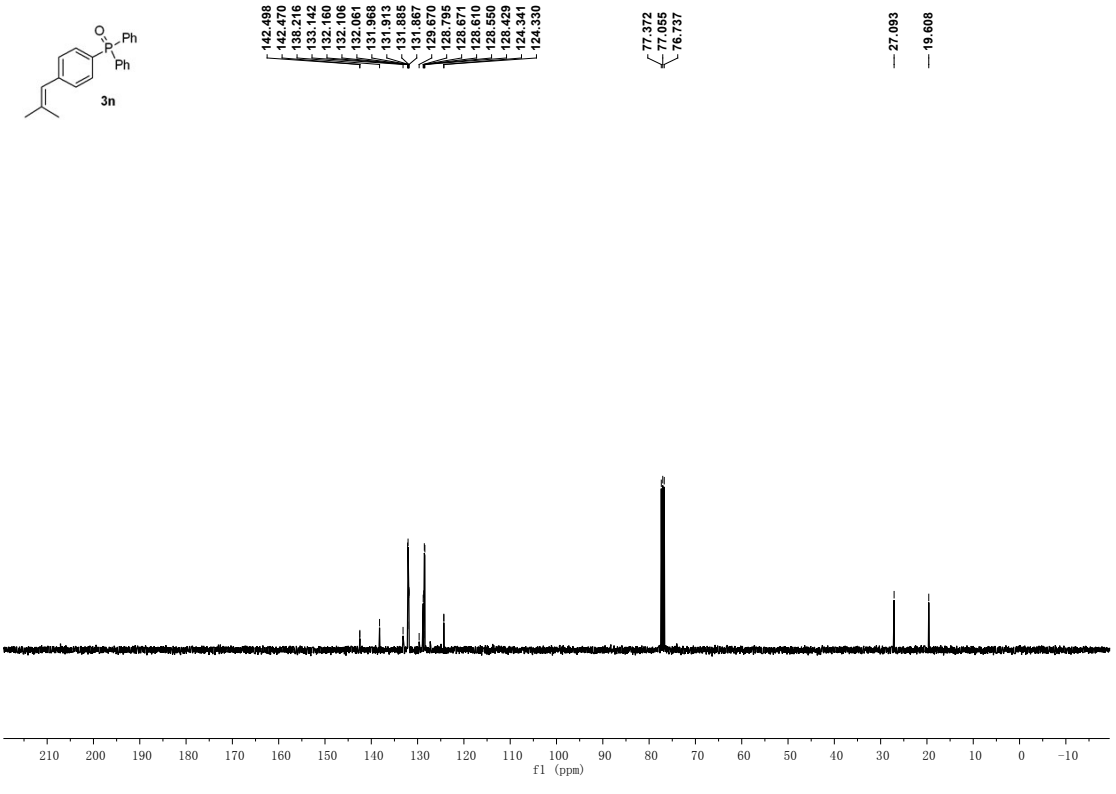

40.712
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

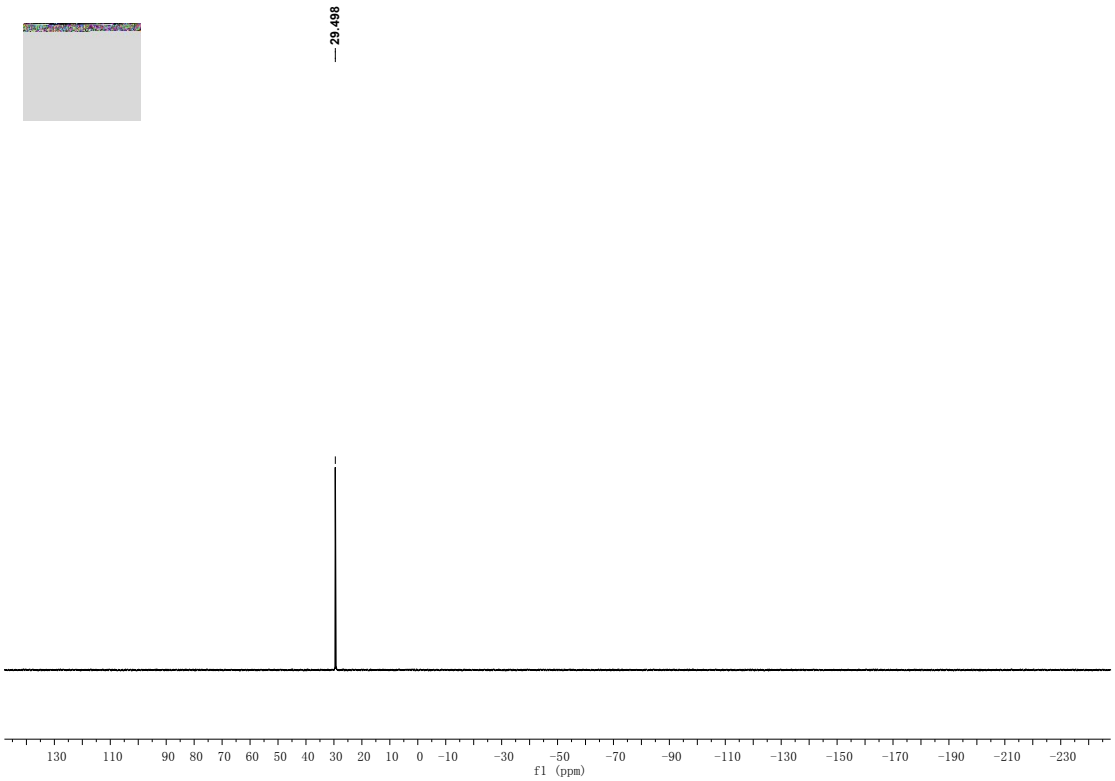

7.837
7.816
7.789
7.765
7.747
7.679
7.660
7.649
7.630
7.599
7.580
7.520
7.502
7.487
7.293
0.000
Ph
Ph
O
P
NC
3m
4.00
6.01
4.06
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

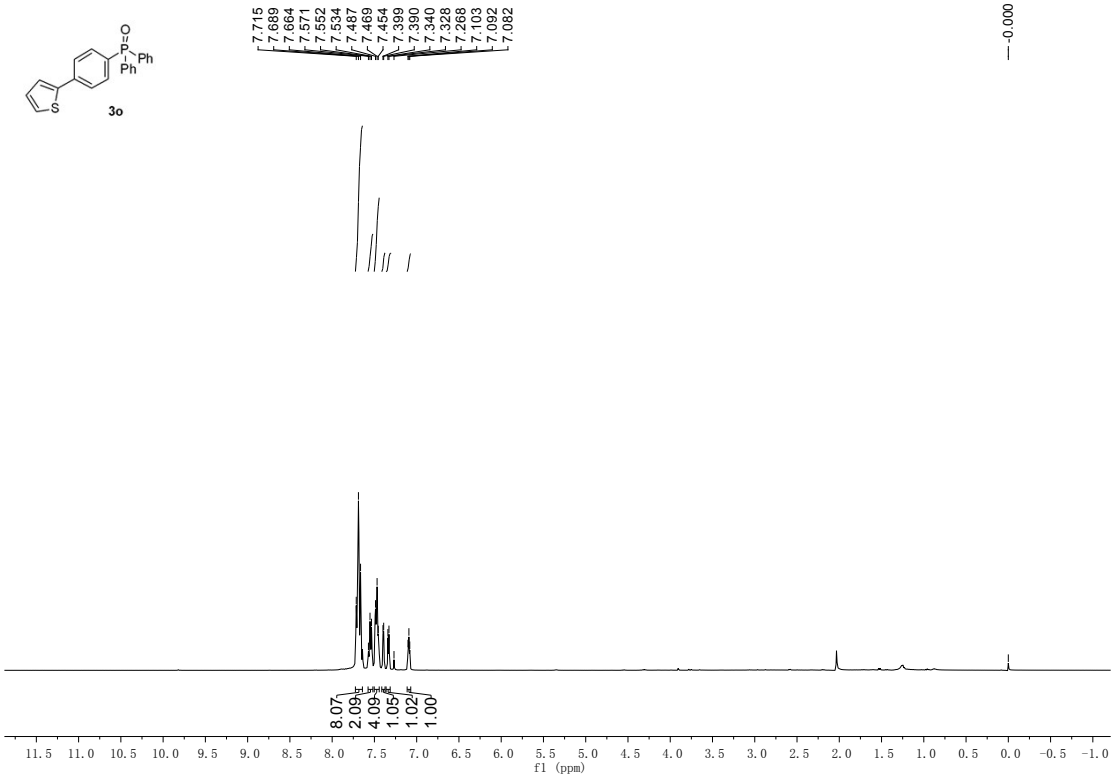

138.801
137.811
132.677
132.624
132.595
132.579
132.091
132.048
131.972
131.948
131.540
130.490
128.923
128.800
117.879
117.866
115.674
115.643
77.447
77.130
76.812
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

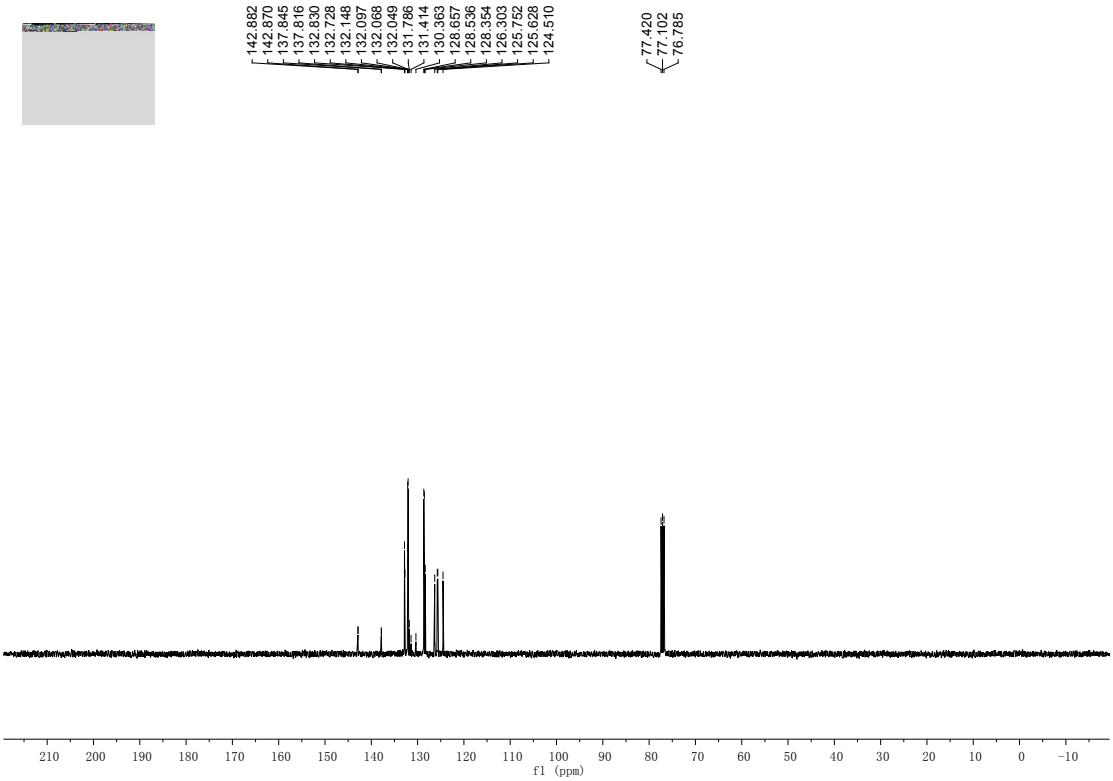

28.199
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

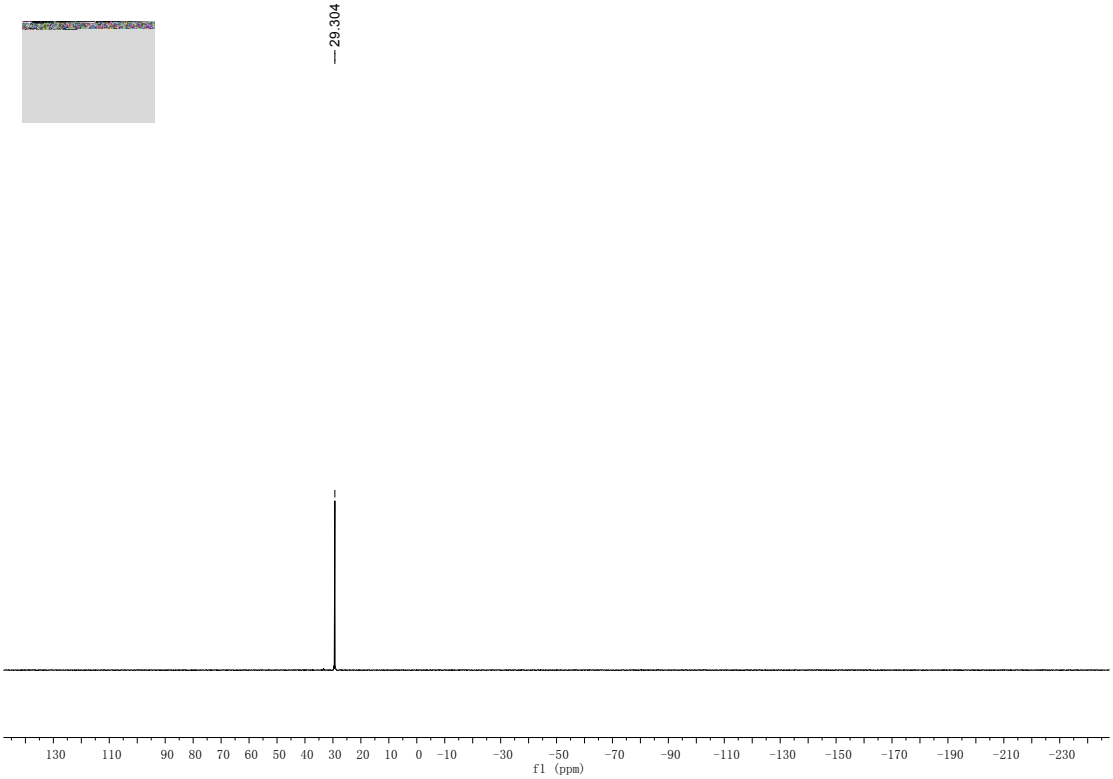

3n
7.706
7.686
7.676
7.656
7.616
7.596
7.588
7.566
7.540
7.523
7.477
7.459
7.441
7.314
7.297
7.267
6.272
1.920
1.871
0.000
4.13
4.08
4.05
2.00
0.98
3.00
3.00
f1 (ppm)

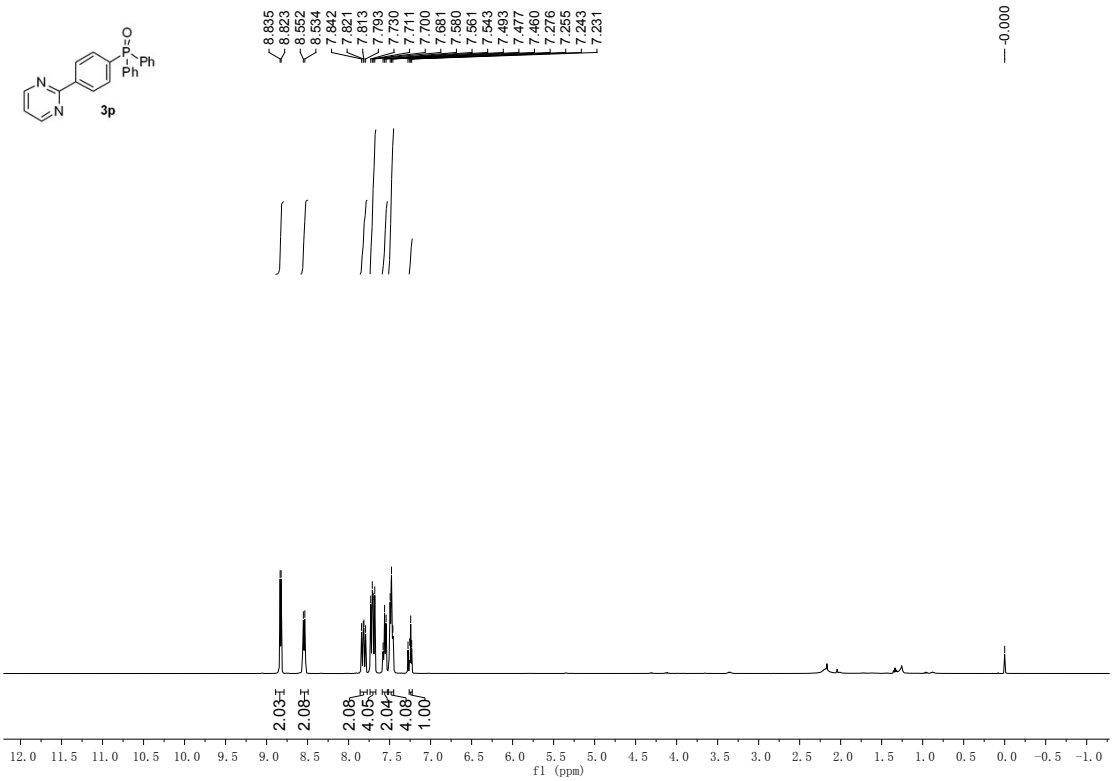

3n
142.498
142.470
138.216
133.142
132.160
132.106
132.061
131.968
131.913
131.885
131.867
129.670
128.795
128.671
128.610
128.550
128.429
124.341
124.330
77.372
77.055
76.737
27.093
19.608
f1 (ppm)

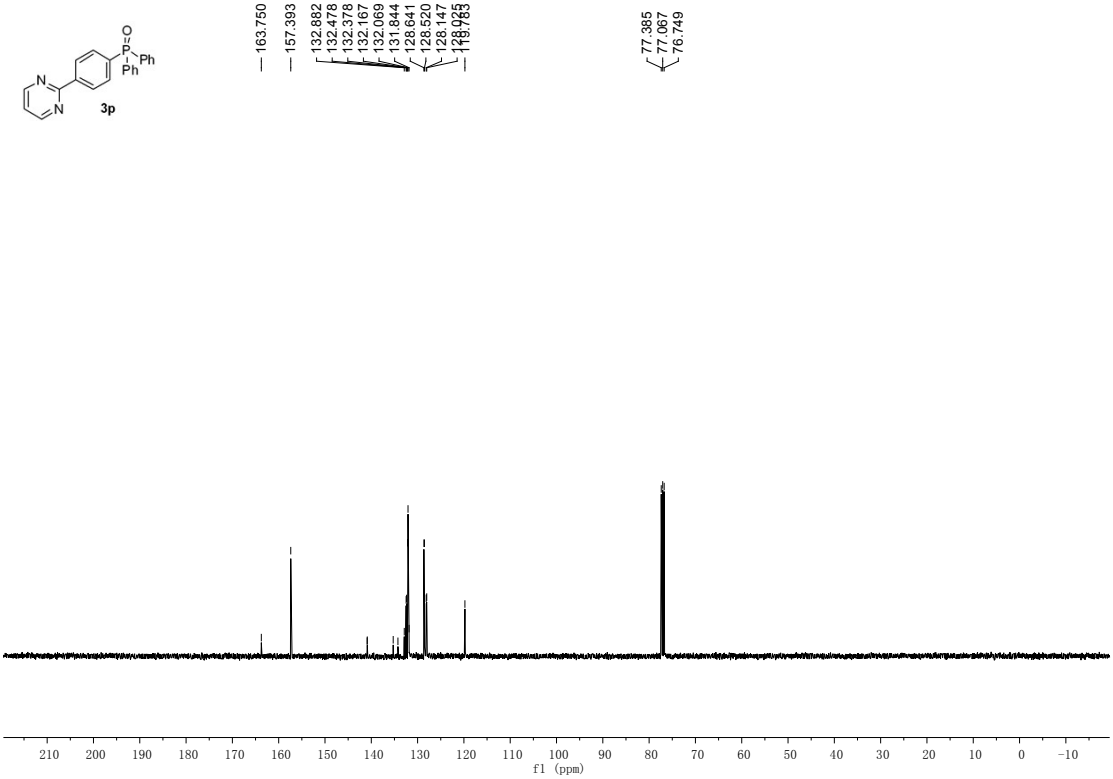

29.498
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

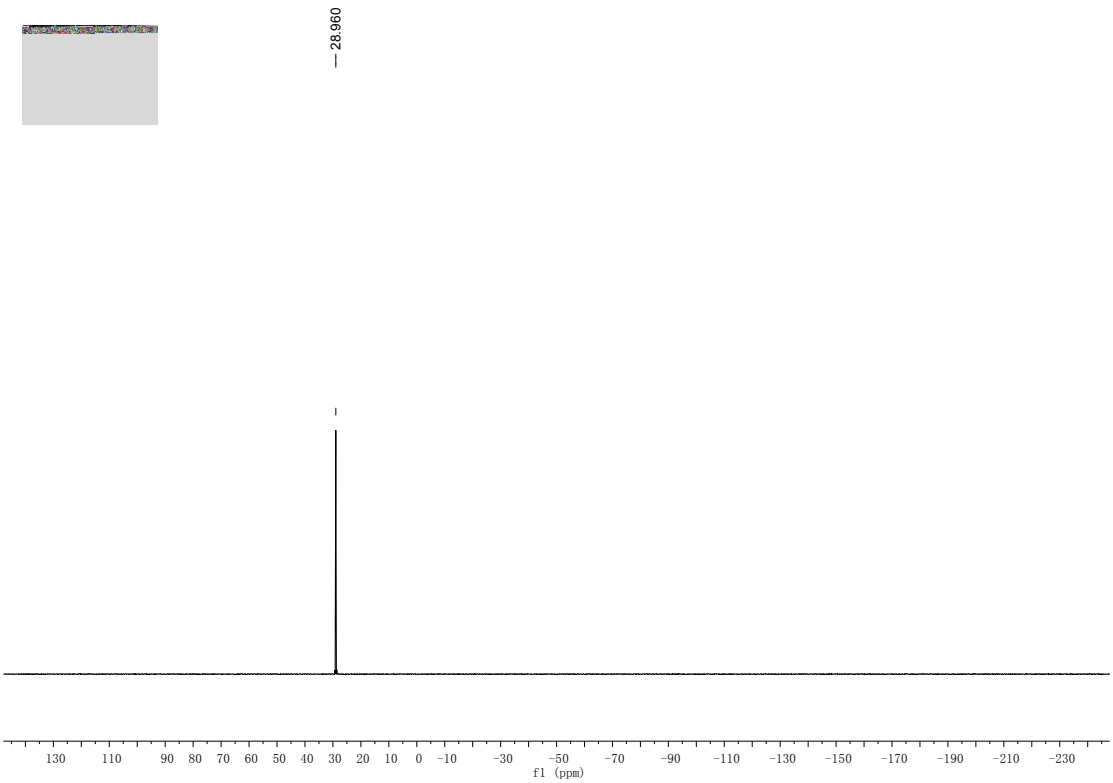

7.715
7.689
7.664
7.571
7.552
7.534
7.487
7.469
7.454
7.399
7.390
7.340
7.328
7.268
7.103
7.092
7.082
-0.000
3o
8.07
2.09
4.09
1.05
1.02
1.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

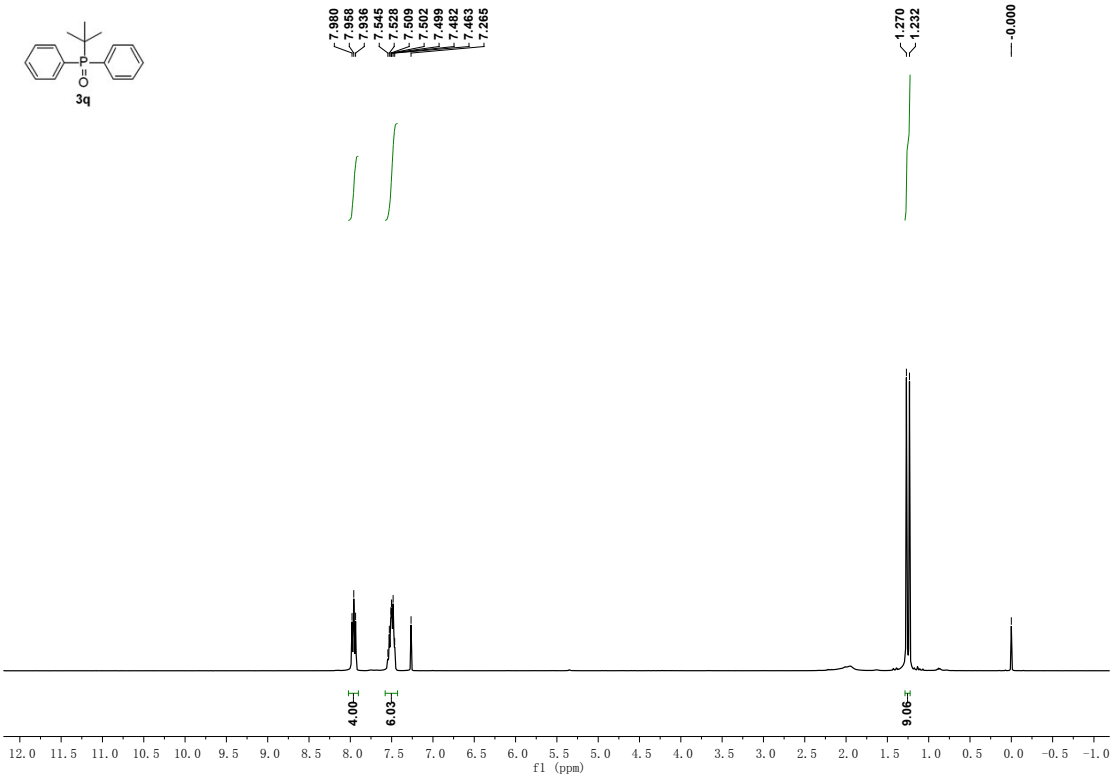

142.882
142.870
137.845
137.816
132.830
132.728
132.148
132.097
132.068
132.049
131.786
131.414
130.363
128.657
128.536
128.354
126.303
125.752
125.628
124.510
77.420
77.102
76.785
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

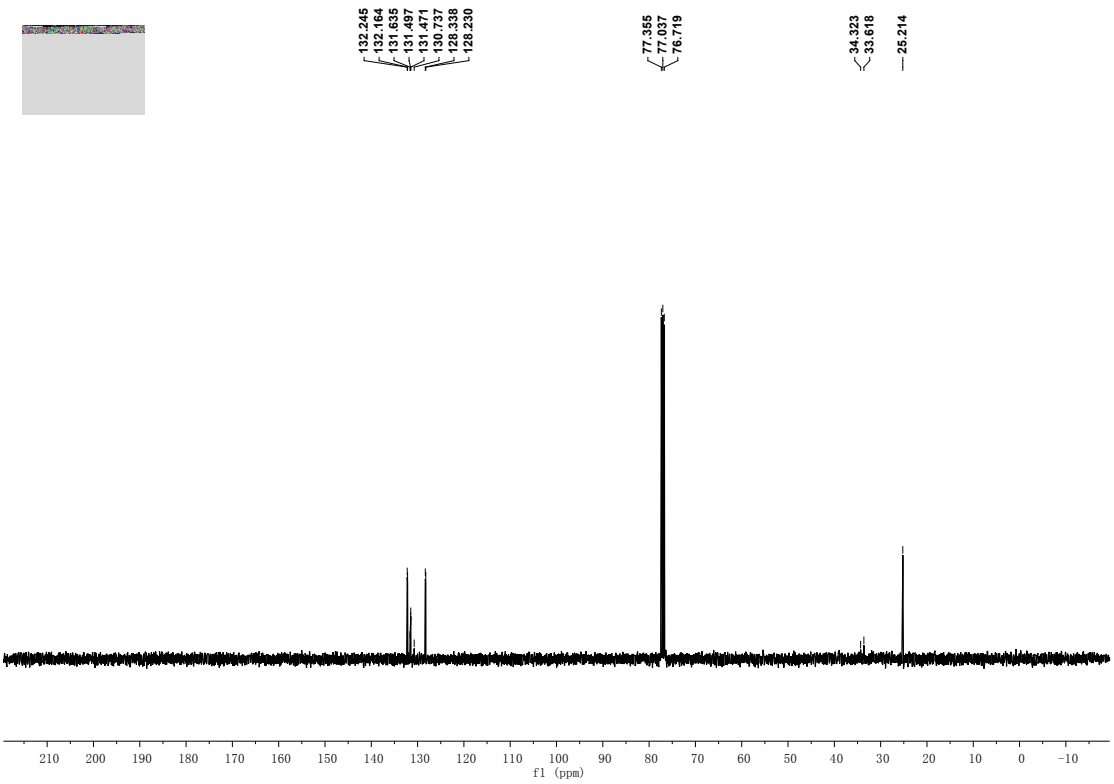

29.304
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

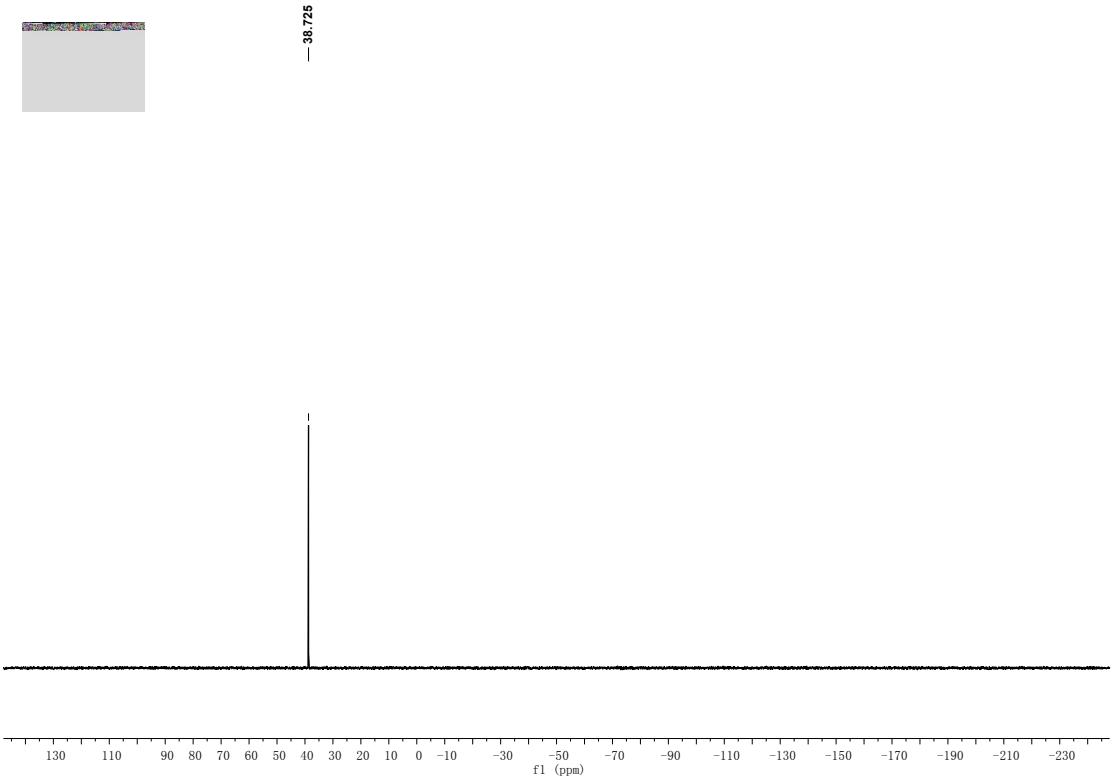

3p
8.835
8.823
8.552
8.534
7.842
7.821
7.813
7.793
7.730
7.711
7.700
7.681
7.580
7.561
7.543
7.493
7.477
7.460
7.276
7.255
7.243
7.231
-0.000
2.03
2.08
2.08
4.05
2.04
4.08
1.00
f1 (ppm)

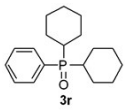

3p
163.750
157.393
132.882
132.478
132.378
132.167
132.069
131.844
128.641
128.520
128.147
128.025
119.783
77.385
77.067
76.749
f1 (ppm)


28.960
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

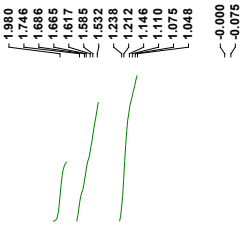

3q
7.980
7.958
7.936
7.545
7.528
7.509
7.502
7.499
7.482
7.463
7.265
1.270
1.232
-0.000
4.00
6.03
9.06
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

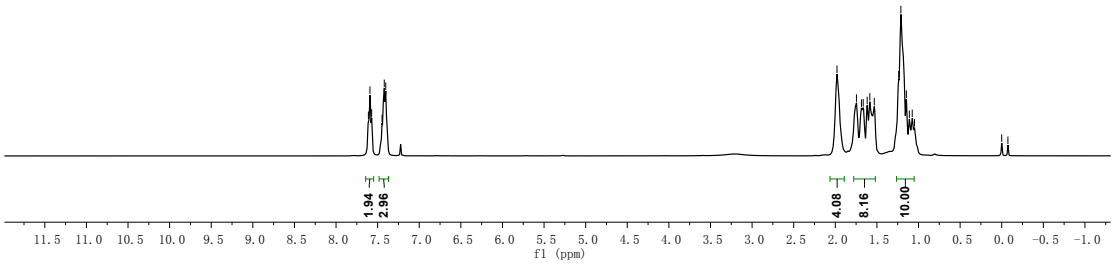

132.245
132.164
131.635
131.497
131.471
130.737
128.338
128.230
77.355
77.037
76.719
34.323
33.618
25.214
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

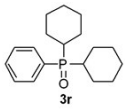

38.725
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

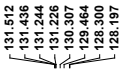

3r


7.611
7.592
7.572
7.447
7.421
7.403

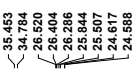

1.980
1.746
1.686
1.665
1.617
1.585
1.532
1.238
1.212
1.146
1.110
1.075
1.048
-0.000
-0.075

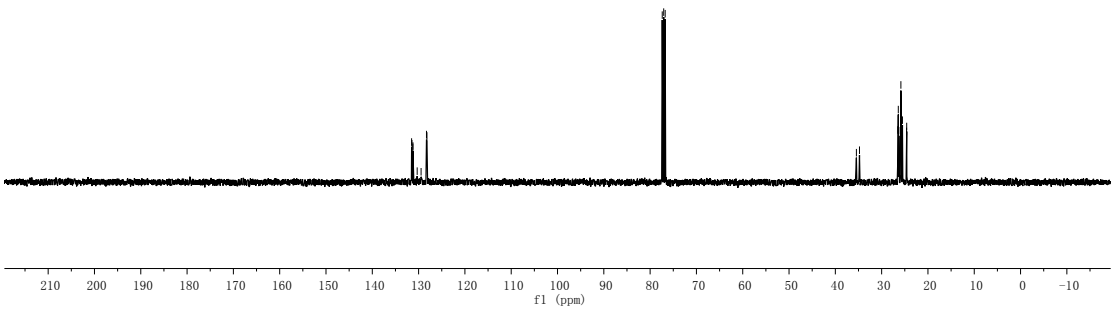

1.94
2.96
4.08
8.16
10.00
f1 (ppm)


3r

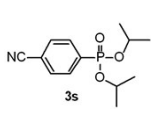

131.512
131.436
131.244
131.226
130.307
129.464
128.300
128.197


77.384
77.067
76.749

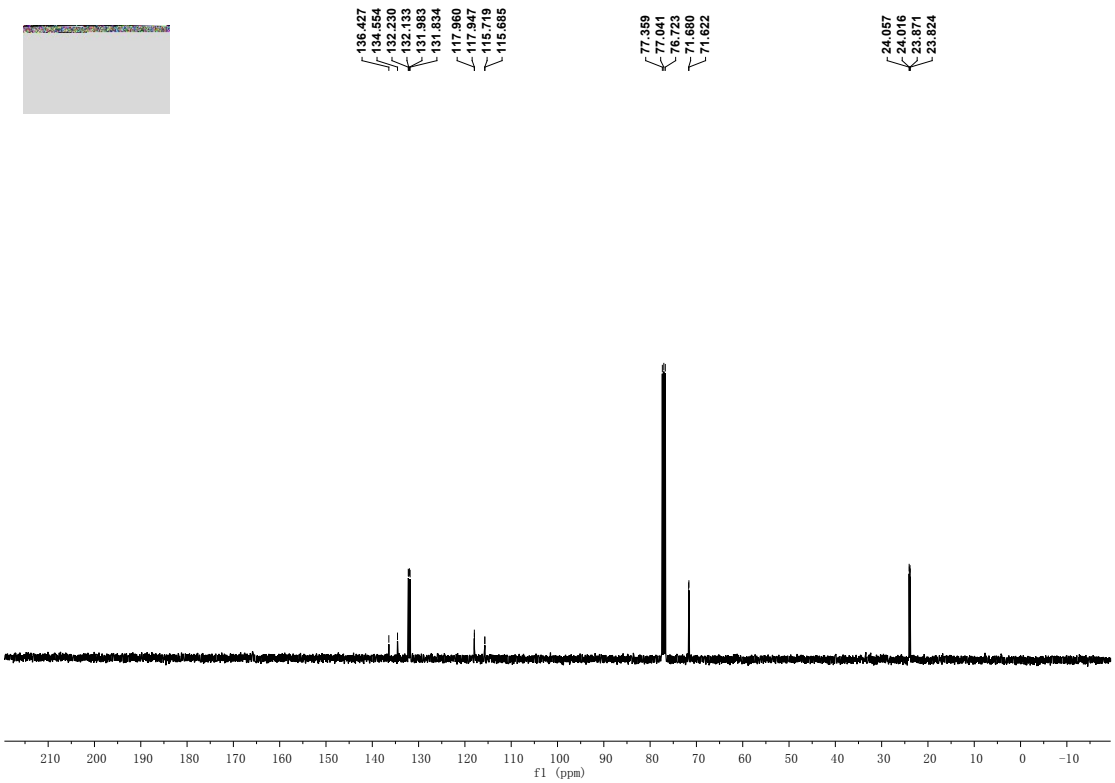

35.453
34.784
26.520
26.404
26.286
25.844
25.507
24.617
24.588

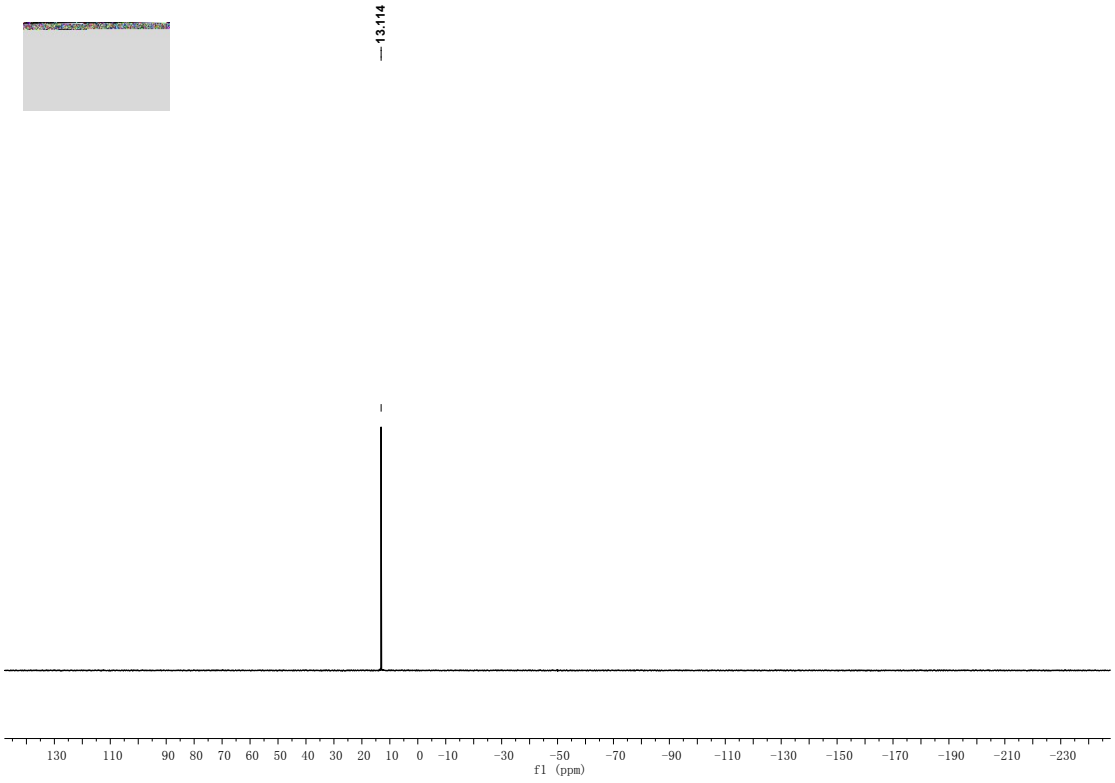

f1 (ppm)

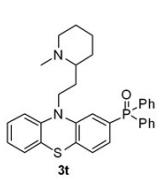
45.399
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

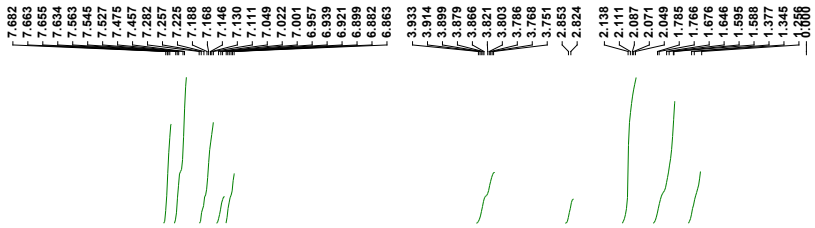

NC
O
P
O
O
3s

7.949
7.929
7.917
7.897
7.758
7.751
7.739
7.732
7.275
4.776
4.760
4.745
4.729
4.712
4.696
1.396
1.381
1.249
1.234
-0.000
2.00
2.03
2.02
6.05
6.06
11.5 11.0 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 -1.0 -1.5
f1 (ppm)


136.427
134.554
132.230
132.133
131.983
131.834
117.960
117.947
115.719
115.685
77.359
77.041
76.723
71.680
71.622
24.057
24.016
23.871
23.824
f1 (ppm)


13.114
f1 (ppm)


Ph
Ph
3t


7.682
7.663
7.655
7.634
7.563
7.545
7.527
7.475
7.457
7.282
7.257
7.225
7.188
7.168
7.146
7.130
7.111
7.049
7.022
7.001
6.957
6.939
6.921
6.899
6.882
6.863
3.933
3.914
3.899
3.879
3.866
3.821
3.803
3.786
3.768
3.751
2.853
2.824
2.138
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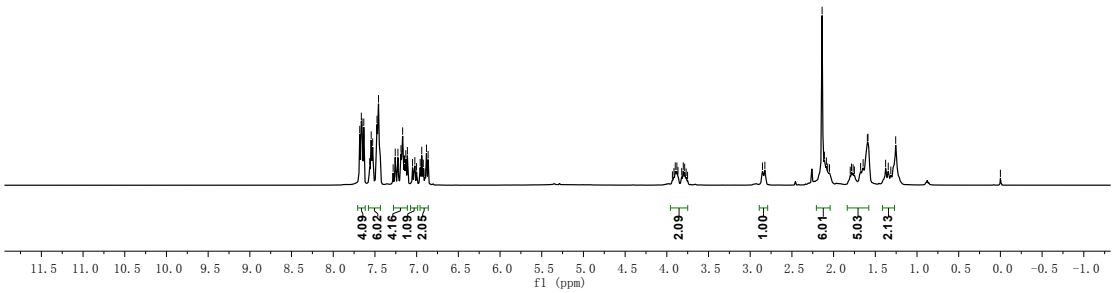
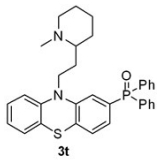
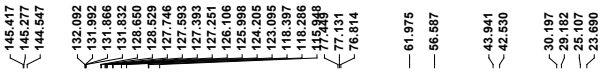
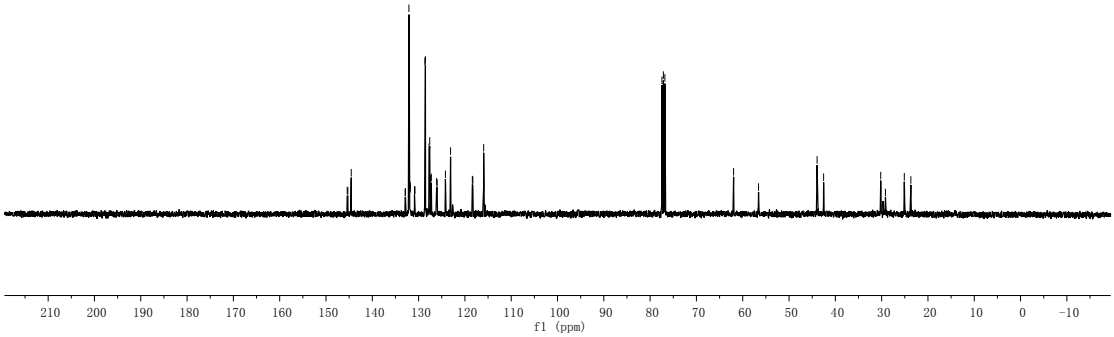
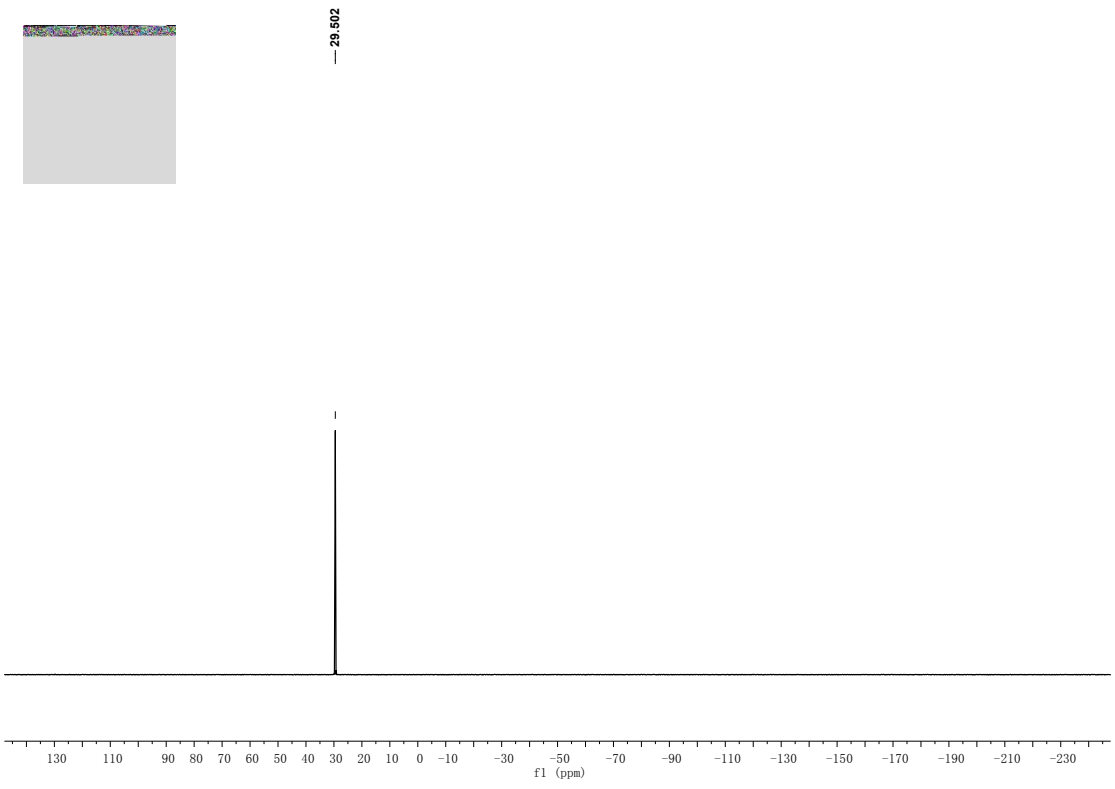
1.676
1.646
1.595
1.588
1.377
1.345


4.09
6.02
4.16
1.09
2.05
2.09
1.00
6.01
5.03
2.13
f1 (ppm)

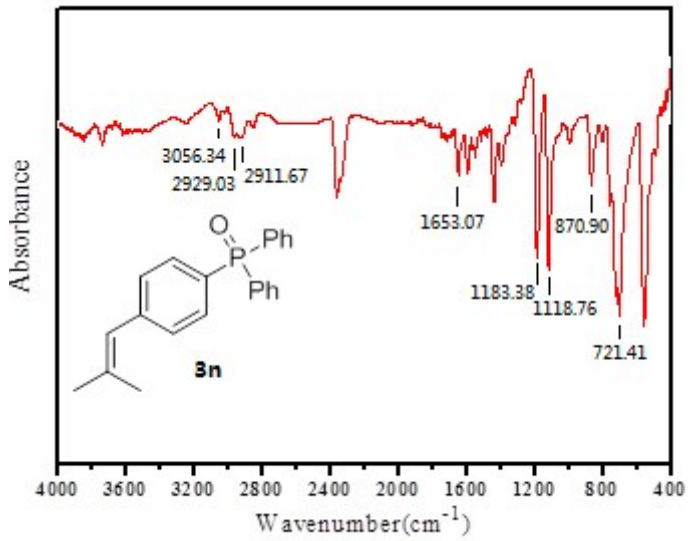

Ph
Ph
3t

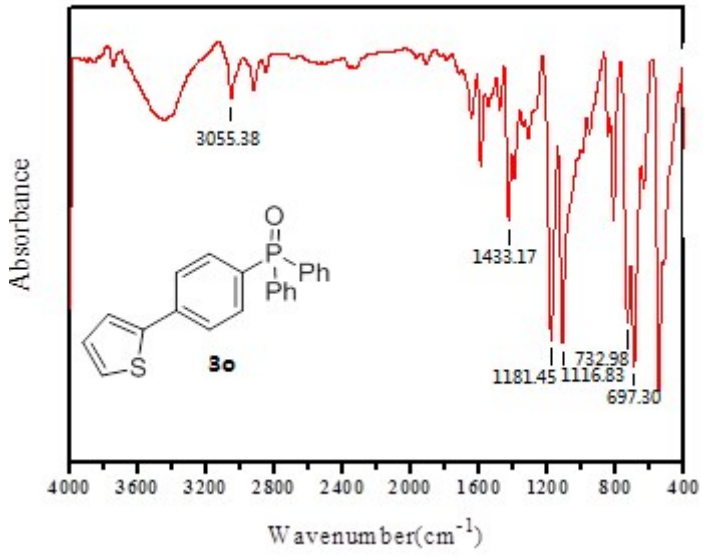

145.417
145.277
144.547
132.092
131.992
131.866
131.832
128.650
128.529
127.746
127.593
127.393
127.251
126.106
125.998
124.205
123.095
118.397
118.286
77.131
76.814
61.975
56.587
43.941
42.530
30.197
29.182
25.107
23.690

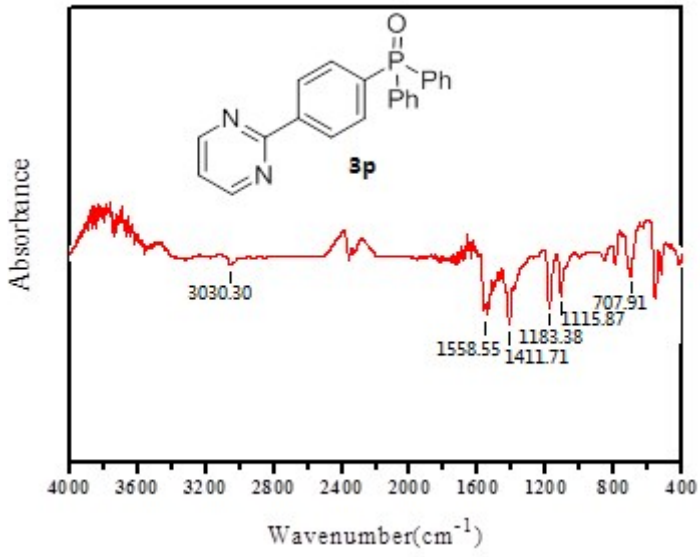

f1 (ppm)

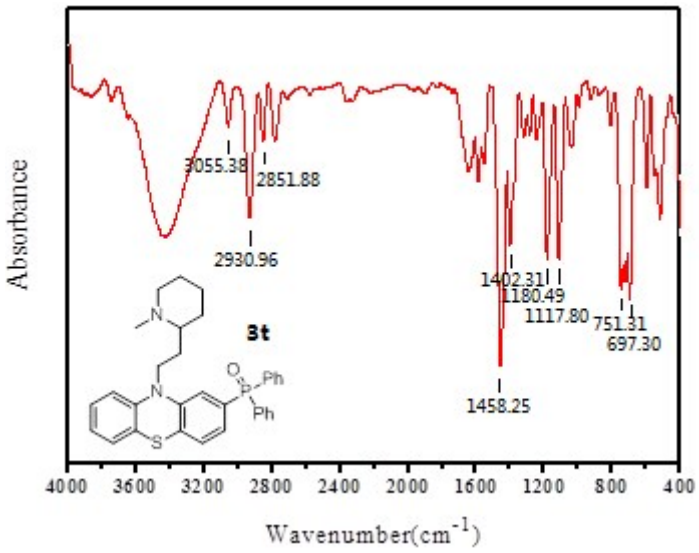

29.502
130
110
90
80
70
60
50
40
30
20
10
0
−10
−30
−50
−70
−90
−110
−130
−150
−170
−190
−210
−230
f1 (ppm)

7. Copies of IR spectra








Absorbance
3055.38
2851.88
2930.96
1402.31
1180.49
1117.80
751.31
697.30
1458.25
3t
Ph
Ph
O
P
N
S
4000
3600
3200
2800
2400
2000
1600
1200
800
400
Wavenumber(cm-1)