Support information for

Article

Lipase-Catalyzed Phospha-Michael Addition Reactions under Mild Conditions

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The Time Course

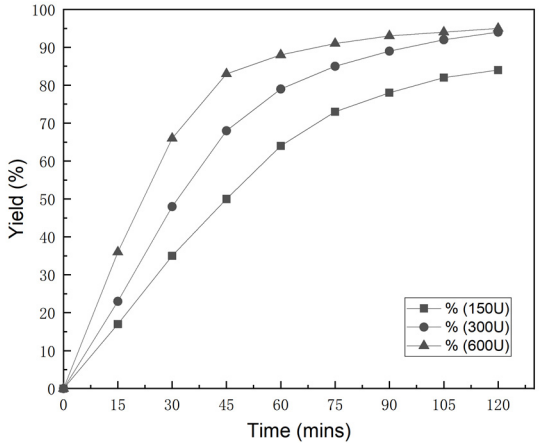


Figure S1. The time course of different dosage lipase-catalyzed phospha-Michael addition reaction. Reaction conditions: **1a** (0.5 mmol), **2** (0.5 mmol), Novozym 435, rt, and EtOH (1 mL).

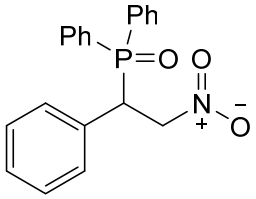
Reusability of Novozym 435

Table S1. Reusability of Novozym 435 on the phospha-Michael addition reaction.

Cycle	Enzyme weight (mg)	Yield (%)
1	20.0	94
2	19.8	93
3	19.4	92
4	19.2	89
5	18.9	86
6	18.5	81
7	18.2	78
8	17.9	72

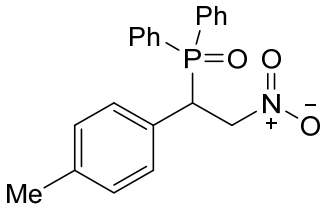
Reaction conditions: **1a** (0.5 mmol), **2** (0.5 mmol), Novozym 435 (300 U = 20mg), rt, and EtOH (1 mL).

Data of Products



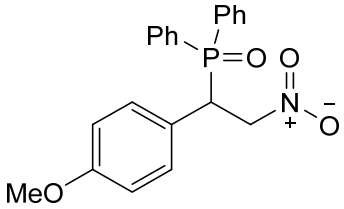
3a *((2-nitro-1-phenylethyl) diphenylphosphine oxide)*

White solid, yield: 165mg, 94%; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.18 – 8.10 (m, 2H), 7.78 – 7.70 (m, 2H), 7.68 – 7.62 (m, 3H), 7.41 (td, J = 7.2, 1.6 Hz, 3H), 7.37 – 7.31 (m, 2H), 7.23 – 7.12 (m, 3H), 5.15 – 4.97 (m, 2H), 4.91 – 4.81 (m, 1H). ^{13}C NMR (151 MHz, DMSO) δ 133.20, 132.62, 131.52, 131.21, 131.15, 131.06, 131.00, 130.62, 130.41, 129.81, 129.73, 128.98, 128.90, 128.83, 128.37, 76.47, 76.06, 44.44, 44.02. ^{31}P NMR (243 MHz, DMSO) δ 30.84.



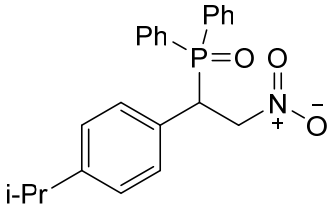
3b *((2-nitro-1-(p-tolyl) ethyl) diphenylphosphine oxide)*

White solid, yield: 173mg, 95%; ^{1}H NMR (400 MHz, Chloroform-d) δ 8.01 – 7.94 (m, 2H), 7.67 – 7.56 (m, 3H), 7.51 – 7.45 (m, 2H), 7.40 (dd, J = 7.2, 1.6 Hz, 1H), 7.30 (td, J = 6.4, 5.6, 3.6 Hz, 2H), 7.17 (dd, J = 8.0, 2.0 Hz, 2H), 7.02 (d, J = 7.6 Hz, 2H), 5.11 – 5.01 (m, 1H), 4.77 – 4.71 (m, 1H), 4.41 (s, 1H), 2.26 (d, J = 1.6 Hz, 3H). ^{13}C NMR (151 MHz, DMSO) δ 135.71, 130.35, 129.74, 128.85, 128.79, 128.69, 128.23, 128.09, 127.59, 127.40, 127.15, 126.91, 126.05, 125.97, 114.09, 74.98, 74.77, 74.56, 73.57, 73.53, 43.28, 42.86, 18.73. ^{31}P NMR (243 MHz, DMSO) δ 24.87.



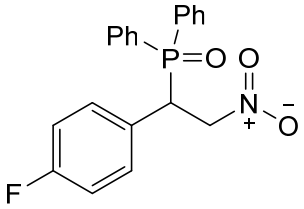
3c *((1-(4-methoxyphenyl)-2-nitroethyl) diphenylphosphine oxide)*

White solid, yield: 169mg, 89%; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.15 – 8.07 (m, 2H), 7.81 – 7.72 (m, 2H), 7.65 (dt, J = 6.0, 3.6 Hz, 3H), 7.45 – 7.32 (m, 5H), 6.80 – 6.74 (m, 2H), 5.06 – 4.90 (m, 2H), 4.84 – 4.77 (m, 1H), 3.67 (s, 3H). ^{13}C NMR (151 MHz, DMSO) δ 159.06, 133.08, 132.53, 131.18, 131.07, 130.28, 129.75, 129.67, 128.99, 128.91, 124.34, 114.20, 76.34, 55.38, 55.06, 43.58, 43.15. ^{31}P NMR (243 MHz, DMSO) δ 30.56.



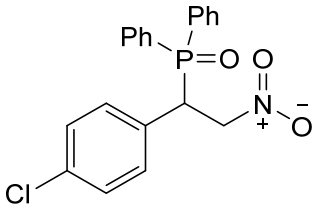
3d *((1-(4-isopropylphenyl)-2-nitroethyl) diphenylphosphine oxide)*

White solid, yield: 157mg, 80%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.01 (t, *J* = 9.2 Hz, 2H), 7.66 (dq, *J* = 14.0, 7.2 Hz, 3H), 7.43 (q, *J* = 7.6, 6.4 Hz, 3H), 7.31 (d, *J* = 2.8 Hz, 1H), 7.28 (s, 1H), 7.21 (d, *J* = 8.0 Hz, 2H), 7.09 (d, *J* = 7.2 Hz, 2H), 5.13 (s, 1H), 4.78 (d, *J* = 13.6 Hz, 1H), 4.45 (d, *J* = 10.8 Hz, 1H), 2.89 – 2.79 (m,1H), 1.20 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 149.03, 132.73, 132.53, 132.04, 131.31, 131.14, 130.76, 130.47, 129.81, 129.35, 129.27, 128.61, 128.28, 128.21, 126.84, 75.89, 75.71, 45.77, 45.33, 33.68, 23.82. ^{31}P NMR (243 MHz, $CDCl_3$) δ 30.26.



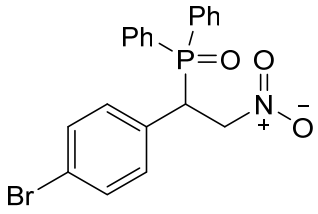
3e *((1-(4-fluorophenyl)-2-nitroethyl) diphenylphosphine oxide)*

White solid, yield: 160mg 87%; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.20 – 8.08 (m, 2H), 7.74 (ddd, *J* = 11.6, 6.8, 1.6 Hz, 2H), 7.69 – 7.63 (m, 3H), 7.51 – 7.33 (m, 5H), 7.06 (t, *J* = 8.8 Hz, 2H), 5.12 – 5.07 (m, 2H), 4.85 (s, 1H). ^{13}C NMR (151 MHz, DMSO) δ 139.74, 139.53, 133.09, 132.49, 131.88, 131.31, 131.25, 131.09, 131.04, 130.74, 130.39, 129.73, 129.65, 129.31, 128.93, 128.86, 115.70, 115.55, 76.24, 76.23, 43.56, 43.14. ^{31}P NMR (243 MHz, DMSO) δ 29.97.



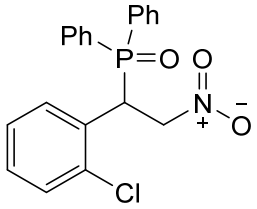
3f *((1-(4-chlorophenyl)-2-nitroethyl) diphenylphosphine oxide)*

White solid, yield: 169mg, 88%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.99 (dd, *J* = 11.2, 7.6 Hz, 2H), 7.70 – 7.59 (m, 3H), 7.53 – 7.41 (m, 3H), 7.34 (dt, *J* = 10.8, 5.2 Hz, 2H), 7.27 (dd, *J* = 8.4, 1.6 Hz, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 5.06 (d, *J* = 3.2 Hz, 1H), 4.78 – 4.70 (m, 1H), 4.47 – 4.38 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 134.43, 132.92, 132.34, 131.15, 131.09, 130.95, 130.89, 130.74, 130.32, 130.13, 129.48, 129.40, 129.05, 128.59, 128.51, 75.69, 75.65, 45.46, 45.04. ^{31}P NMR (243 MHz, $CDCl_3$) δ 29.53.



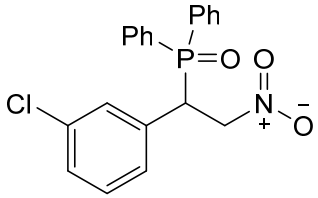
3g *((1-(4-bromophenyl)-2-nitroethyl) diphenylphosphine oxide)*

White solid, yield: 193mg, 90%; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.13 (td, J = 8.0, 3.6 Hz, 2H), 7.76 (dd, J = 11.6, 7.6 Hz, 2H), 7.69 – 7.62 (m, 3H), 7.48 – 7.35 (m, 7H), 5.13 – 5.05 (m, 2H), 4.90 – 4.80 (m, 1H). ^{13}C NMR (151 MHz, DMSO) δ 133.13, 132.71, 132.60, 132.19, 131.99, 131.67, 131.29, 131.10, 131.03, 130.65, 130.27, 129.75, 129.67, 128.94, 121.60, 76.00, 43.75, 43.33. ^{31}P NMR (243 MHz, DMSO) δ 29.69.



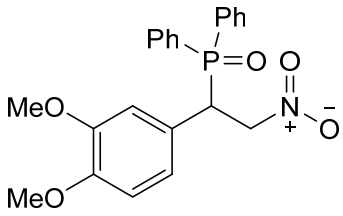
3h *((1-(2-chlorophenyl)-2-nitroethyl) diphenylphosphine oxide)*

White solid, yield: 161mg 84%; ^{1}H NMR (400 MHz, Chloroform-d) δ 8.10 (ddd, J = 11.2, 7.6, 1.6 Hz, 2H), 7.89 (d, J = 7.6 Hz, 1H), 7.74 – 7.64 (m, 3H), 7.49 – 7.39 (m, 3H), 7.34 (dt, J = 8.4, 4.0 Hz, 1H), 7.26 (dd, J = 7.6, 3.2 Hz, 2H), 7.19 (d, J = 4.0 Hz, 2H), 5.25 (dd, J = 9.2, 3.2 Hz, 1H), 5.20 – 5.11 (m, 1H), 4.79 (ddd, J = 13.6, 6.0, 3.2 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 134.95, 132.97, 132.26, 131.94, 131.30, 131.24, 130.95, 130.89, 130.19, 129.91, 129.71, 129.56, 129.47, 129.41, 128.42, 128.20, 128.12, 127.52, 75.39, 75.38, 41.14, 40.72. ^{31}P NMR (243 MHz, $CDCl_3$) δ 30.73.



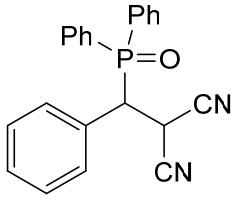
3i *((1-(3-chlorophenyl)-2-nitroethyl) diphenylphosphine oxide)*

White solid, yield: 154mg, 80%; ^{1}H NMR (400 MHz, Chloroform-d) δ 8.01 (dd, J = 11.2, 7.6 Hz, 2H), 7.67 (t, J = 9.2 Hz, 3H), 7.53 – 7.43 (m, 3H), 7.36 (dt, J = 8.8, 4.4 Hz, 2H), 7.28 – 7.16 (m, 4H), 5.15 – 5.03 (m, 1H), 4.80 – 4.73 (m, 1H), 4.46 – 4.36 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 134.61, 133.83, 132.98, 132.41, 131.19, 131.13, 130.99, 130.93, 130.01, 129.65, 129.49, 129.42, 128.57, 128.49, 127.47, 75.45, 45.76, 45.34. ^{31}P NMR (243 MHz, $CDCl_3$) δ 29.82.



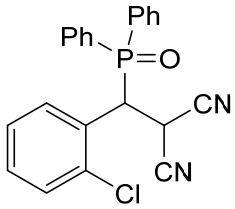
3j *(1-(3,4-dimethoxyphenyl)-2-nitroethyl) diphenylphosphine oxide*

White solid, yield: 154mg, 75%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.99 (dd, *J* = 11.2, 7.6 Hz, 2H), 7.70 – 7.59 (m, 3H), 7.53 – 7.41 (m, 3H), 7.34 (dt, *J* = 10.8, 5.2 Hz, 2H), 7.27 (dd, *J* = 8.4, 1.6 Hz, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 5.06 (d, *J* = 3.2 Hz, 1H), 4.78 – 4.70 (m, 1H), 4.47 – 4.38 (m, 1H), 3.88 (s, 3H), 3.77 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 134.43, 132.92, 132.34, 131.15, 131.09, 130.95, 130.89, 130.74, 130.32, 130.13, 129.47, 129.40, 129.04, 128.59, 128.51, 122.45, 122.05, 122.00, 75.67, 55.45, 55.45, 45.46, 45.04. ^{31}P NMR (243 MHz, $CDCl_3$) δ 29.53.



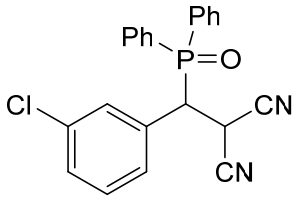
5a *(2-((diphenylphosphoryl)(phenyl)methyl) malononitrile)*

White solid, yield: 163mg, 92%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.05 – 7.97 (m, 2H), 7.72 – 7.60 (m, 3H), 7.56 – 7.49 (m, 2H), 7.45 – 7.39 (m, 3H), 7.31 (td, *J* = 5.2, 4.8, 3.2 Hz, 5H), 4.78 (t, *J* = 7.6 Hz, 1H), 4.09 (s, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 134.43, 132.92, 132.34, 131.15, 131.09, 130.95, 130.89, 130.74, 130.32, 130.13, 129.47, 129.40, 129.04, 128.59, 128.51, 111.14, 111.09, 111.05, 75.67, 45.46, 45.04, 24.31. ^{31}P NMR (243 MHz, $CDCl_3$) δ 29.53.



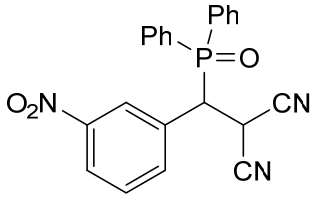
5b *(2-((2-chlorophenyl) (diphenylphosphoryl)methyl) malononitrile)*

White solid, yield: 173mg, 89%; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.20 – 8.12 (m, 2H), 7.99 (d, *J* = 8.0 Hz, 1H), 7.76 – 7.64 (m, 3H), 7.50 (dd, *J* = 11.6, 7.6 Hz, 2H), 7.42 (t, *J* = 7.2 Hz, 2H), 7.36 – 7.26 (m, 4H), 5.66 (dd, *J* = 8.4, 6.8 Hz, 1H), 5.22 – 5.15 (m, 1H). ^{13}C NMR (151 MHz, DMSO) δ 134.63, 134.19, 133.99, 133.45, 132.74, 132.28, 132.20, 131.84, 131.78, 130.97, 130.76, 130.28, 130.14, 129.68, 129.60, 129.24, 129.16, 128.81, 128.73, 113.20, 113.13, 112.01, 111.91, 42.11, 41.83, 25.05. ^{31}P NMR (243 MHz, DMSO) δ 29.01.



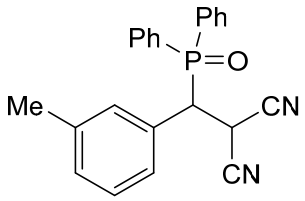
5c *(2-((3-chlorophenyl) (diphenylphosphoryl)methyl) malononitrile)*

White solid, yield: 165mg, 85%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.02 (dd, *J* = 11.6, 7.6 Hz, 2H), 7.75 – 7.63 (m, 3H), 7.59 – 7.44 (m, 3H), 7.36 (d, *J* = 7.2 Hz, 5H), 7.27 (d, *J* = 8.0 Hz, 1H), 4.74 (s, 1H), 4.03 (t, *J* = 7.6 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 135.15, 133.43, 131.32, 131.30, 131.20, 131.18, 130.53, 129.87, 129.65, 129.48, 129.41, 128.65, 128.57, 127.72, 110.94, 110.93, 47.07, 46.67, 24.62. ^{31}P NMR (243 MHz, $CDCl_3$) δ 28.36.



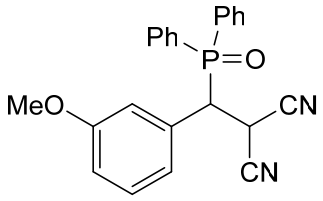
5d *(2-((diphenylphosphoryl)(3-nitrophenyl) methyl) malononitrile)*

White solid, yield: 164mg, 82%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.19 (d, *J* = 8.8 Hz, 2H), 8.09 – 8.02 (m, 2H), 7.96 (d, *J* = 7.6 Hz, 1H), 7.71 (ddd, *J* = 18.8, 7.6, 2.4 Hz, 3H), 7.57 (ddd, *J* = 11.2, 8.0, 2.0 Hz, 3H), 7.44 (d, *J* = 7.2 Hz, 1H), 7.35 (td, *J* = 7.6, 3.2 Hz, 2H), 4.75 (s, 1H), 4.20 (t, *J* = 7.2 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 135.18, 133.71, 133.09, 132.91, 131.33, 131.27, 130.95, 130.89, 130.47, 129.67, 129.59, 128.91, 128.83, 125.11, 124.25, 110.68, 46.98, 46.57, 29.71, 24.47. ^{31}P NMR (243 MHz, $CDCl_3$) δ 28.24.



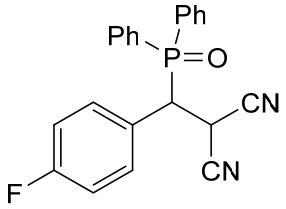
5e *(2-((diphenylphosphoryl)(m-tolyl) methyl) malononitrile)*

White solid, yield: 157mg, 85%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.03 – 7.97 (m, 2H), 7.66 (dtd, *J* = 14.8, 7.2, 2.4 Hz, 3H), 7.55 – 7.48 (m, 2H), 7.43 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.33 (dd, *J* = 7.6, 3.6 Hz, 2H), 7.22 – 7.11 (m, 4H), 4.83 (s, 1H), 4.03 (t, *J* = 8.0 Hz, 1H), 2.28 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 133.17, 132.69, 132.37, 131.36, 130.58, 130.30, 130.13, 129.30, 129.22, 129.12, 128.88, 128.35, 128.27, 126.77, 111.31, 111.30, 47.42, 47.00, 24.70, 21.30. ^{31}P NMR (243 MHz, $CDCl_3$) δ 28.64.



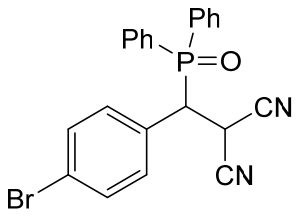
5f *(2-((diphenylphosphoryl)(3-methoxyphenyl) methyl) malononitrile)*

White solid, yield: 156mg, 81%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.04 – 7.95 (m, 2H), 7.71 – 7.53 (m, 5H), 7.45 (d, *J* = 1.6 Hz, 1H), 7.33 (d, *J* = 3.2 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 1H), 7.01 – 6.92 (m, 2H), 6.86 (dd, *J* = 8.4, 2.4 Hz, 1H), 4.77 (s, 1H), 4.05 (t, *J* = 8.0 Hz, 1H), 3.73 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 133.20, 132.46, 132.03, 131.36, 131.36, 130.31, 129.65, 129.35, 129.27, 128.85, 128.45, 128.46, 128.38, 122.08, 115.48, 114.83, 111.28, 55.29, 47.43, 47.01, 24.78. ^{31}P NMR (243 MHz, $CDCl_3$) δ 30.84.



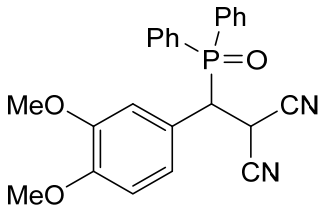
5g *2-((diphenylphosphoryl)(4-fluorophenyl) methyl) malononitrile*

White solid, yield: 145mg, 78%; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.17 – 8.09 (m, 2H), 7.69 – 7.62 (m, 5H), 7.53 (s, 4H), 7.44 – 7.32 (m, 3H), 5.46 (t, *J* = 7.3 Hz, 1H), 5.17 (t, *J* = 7.1 Hz, 1H). ^{13}C NMR (151 MHz, DMSO) δ 133.11, 132.60, 132.46, 132.39, 132.32, 131.88, 131.62, 131.56, 131.37, 131.30, 130.82, 130.76, 130.16, 130.10, 129.49, 129.42, 128.87, 128.79, 116.00, 115.85, 115.09, 114.95, 113.17, 112.93, 79.57, 79.35, 79.14, 42.23, 41.80, 25.47. ^{31}P NMR (243 MHz, DMSO) δ 28.58. ^{19}F NMR (565 MHz, DMSO) δ -112.90.



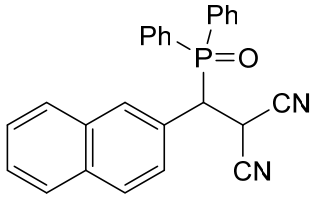
5h *2-((4-bromophenyl) (diphenylphosphoryl)methyl) malononitrile*

White solid, yield: 171mg, 79%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.03 – 7.97 (m, 2H), 7.83 – 7.50 (m, 7H), 7.44 (dtd, *J* = 8.4, 3.9, 2.0 Hz, 3H), 7.35 (td, *J* = 7.6, 3.2 Hz, 1H), 7.02 (t, *J* = 8.6 Hz, 1H), 4.69 (t, *J* = 7.2 Hz, 1H), 4.05 (t, *J* = 7.6 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 137.03, 136.79, 133.15, 132.51, 131.97, 131.28, 130.69, 130.43, 130.25, 129.81, 129.27, 128.80, 128.45, 111.89, 111.37, 78.00, 77.95, 42.23, 41.80, 25.47. ^{31}P NMR (243 MHz, $CDCl_3$) δ 29.69.



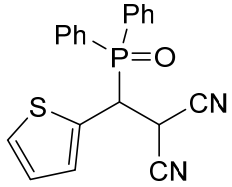
5i *(2-((3,4-dimethoxyphenyl) (diphenylphosphoryl)methyl) malononitrile)*

White solid, yield: 174mg, 84%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.02 – 7.92 (m, 2H), 7.72 – 7.53 (m, 5H), 7.47 (t, *J* = 7.6 Hz, 1H), 7.36 (td, *J* = 7.6, 2.8 Hz, 2H), 6.96 (d, *J* = 5.6 Hz, 2H), 6.82 (d, *J* = 8.4 Hz, 1H), 4.73 (s, 1H), 4.04 – 3.96 (m, 1H), 3.88 (s, 3H), 3.77 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 139.74, 139.53, 133.21, 132.49, 131.88, 131.31, 131.25, 131.09, 131.04, 130.74, 130.39, 129.73, 129.65, 129.31, 128.94, 128.86, 78.00, 77.95, 47.42, 47.01, 24.70. ^{31}P NMR (243 MHz, $CDCl_3$) δ 28.47.



5j *(2-((diphenylphosphoryl)(naphthalen-2-yl) methyl) malononitrile)*

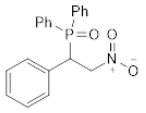
White solid, yield: 142mg, 70%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.03 (dd, *J* = 11.6, 7.6 Hz, 2H), 7.91 (s, 1H), 7.81 (t, *J* = 9.2 Hz, 4H), 7.73 – 7.61 (m, 3H), 7.58 – 7.46 (m, 5H), 7.25 (td, *J* = 7.6, 3.2 Hz, 2H), 4.85 (t, *J* = 7.6 Hz, 1H), 4.23 (t, *J* = 8.0 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 133.23, 133.11, 132.45, 132.02, 131.35, 131.29, 130.38, 129.72, 129.49, 129.38, 129.31, 129.26, 128.79, 128.46, 128.38, 128.18, 127.72, 127.07, 126.80, 126.48, 111.36, 111.30, 111.21, 47.54, 47.12, 24.97. ^{31}P NMR (243 MHz, $CDCl_3$) δ 28.51.



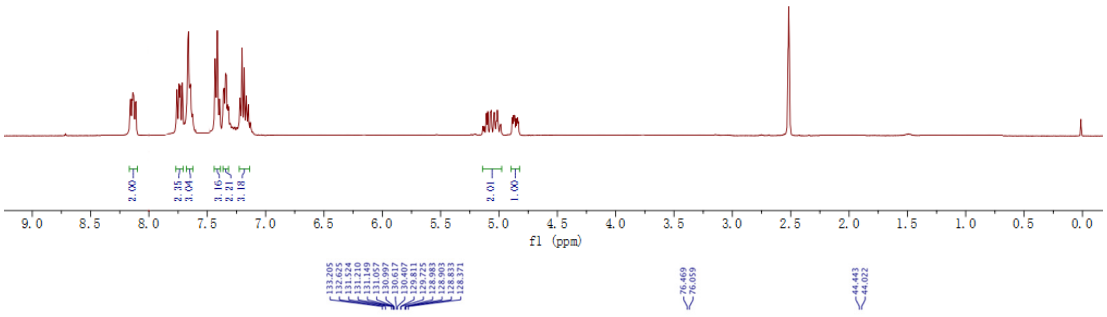
5k *(2-((diphenylphosphoryl)(thiophen-2-yl) methyl) malononitrile)*

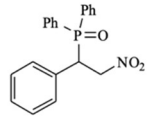
White solid, yield: 130mg, 72%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.96 (q, *J* = 7.6, 6.4 Hz, 2H), 7.86 – 7.74 (m, 2H), 7.70 (d, *J* = 7.2 Hz, 1H), 7.64 (d, *J* = 7.6 Hz, 4H), 7.52 (s, 1H), 7.41 (d, *J* = 7.2 Hz, 2H), 7.25 (d, *J* = 3.2 Hz, 1H), 4.75 (s, 1H), 4.40 (s, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 133.37, 132.78, 132.35, 131.89, 131.64, 131.58, 131.41, 131.34, 130.80, 130.68, 129.81, 129.42, 129.34, 129.16, 128.87, 128.55, 128.47, 128.36, 128.16, 127.85, 127.45, 126.82, 111.25, 111.19, 110.96, 110.92, 42.97, 42.54, 25.62. ^{31}P NMR (243 MHz, $CDCl_3$) δ 28.36.

Spectra of Products



^{1}H-3a 400MHz





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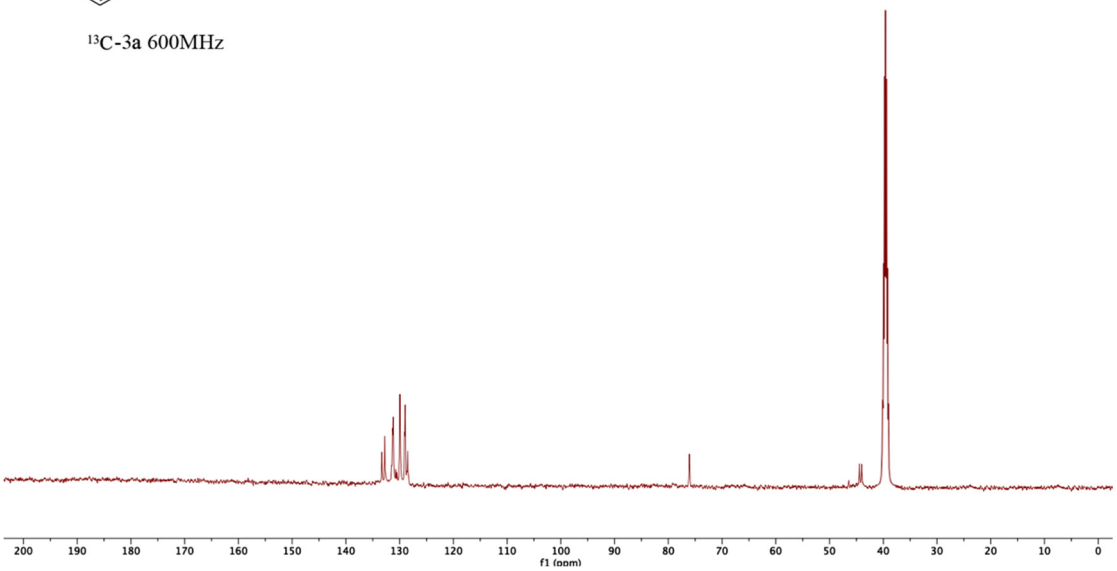
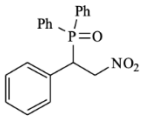
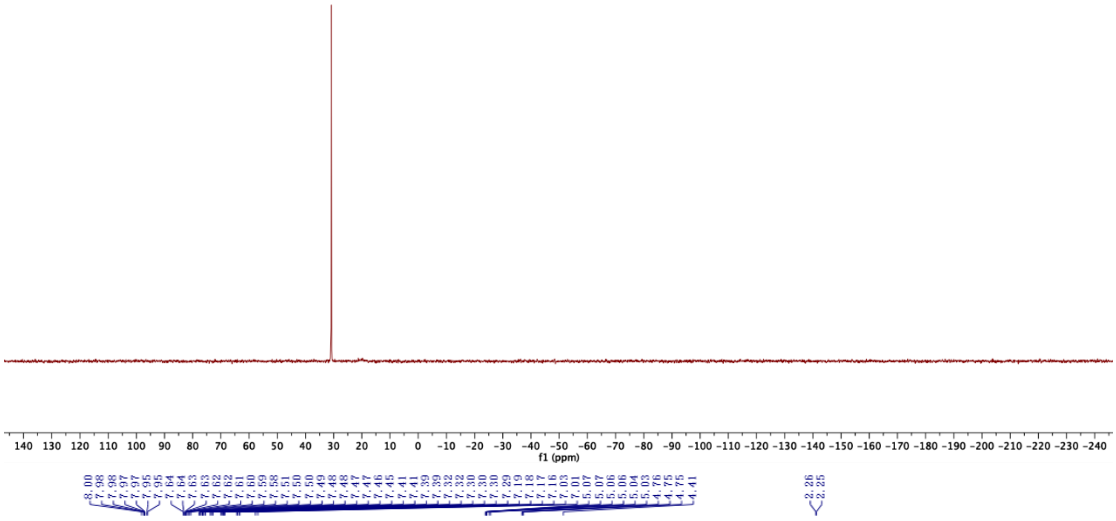
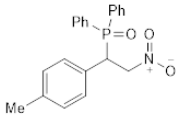




^{31}P 3-a 600MHz





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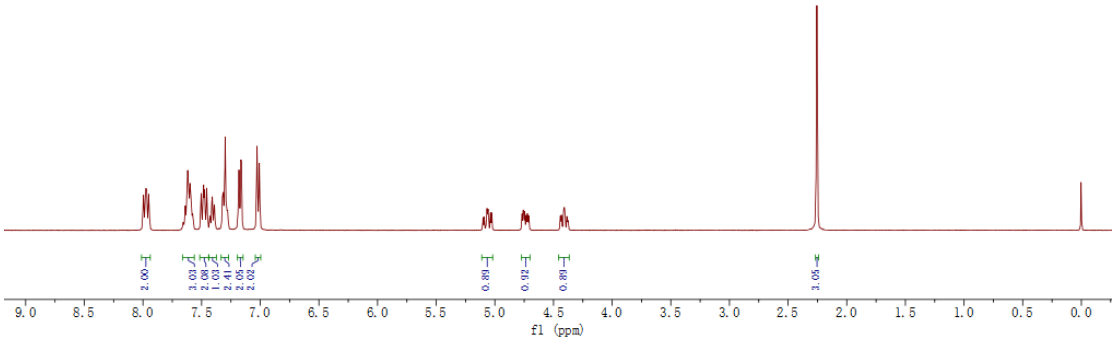

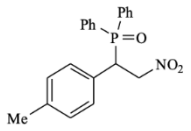

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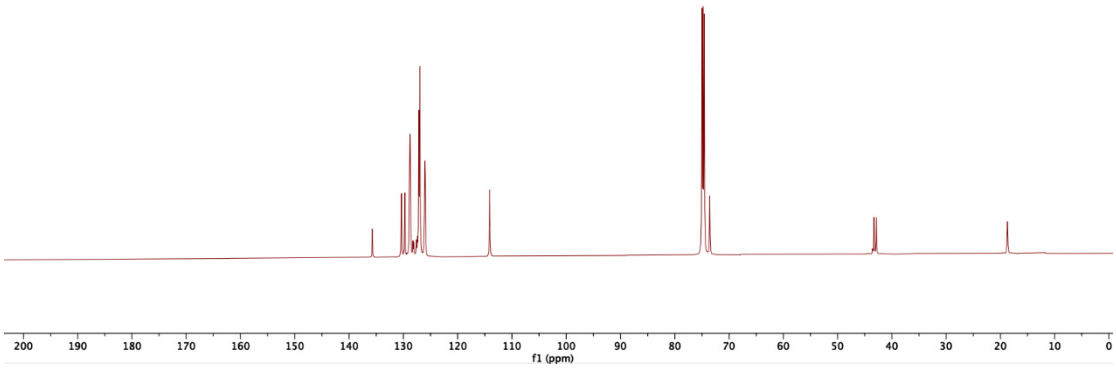

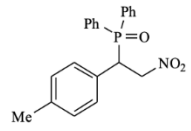

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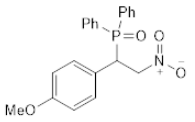
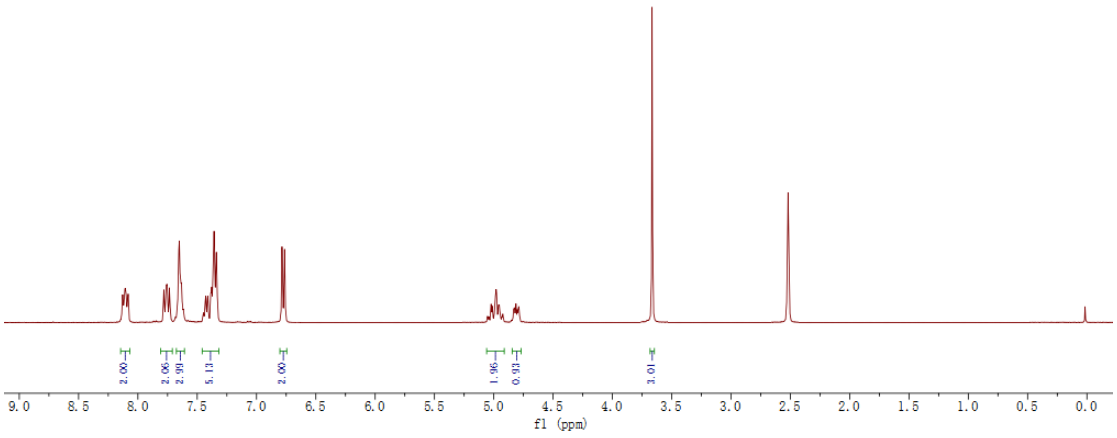
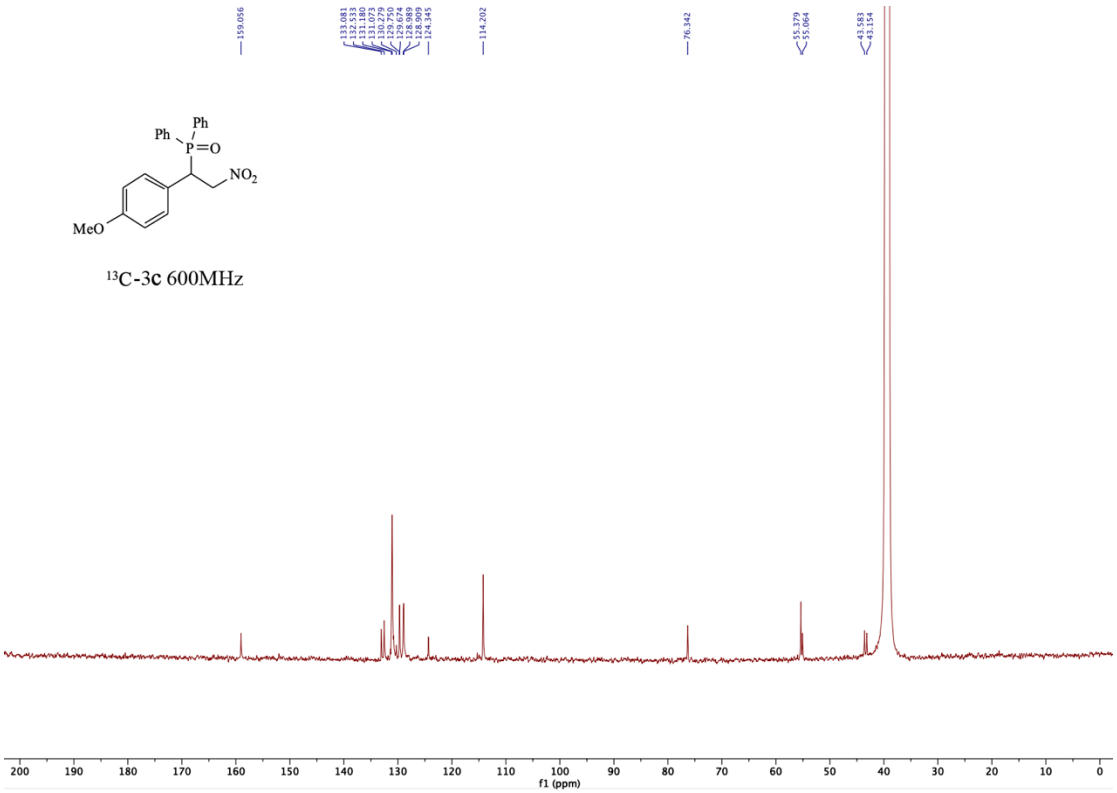


^{1}H-3c 400MHz





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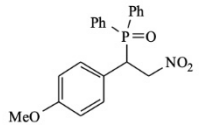

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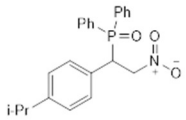
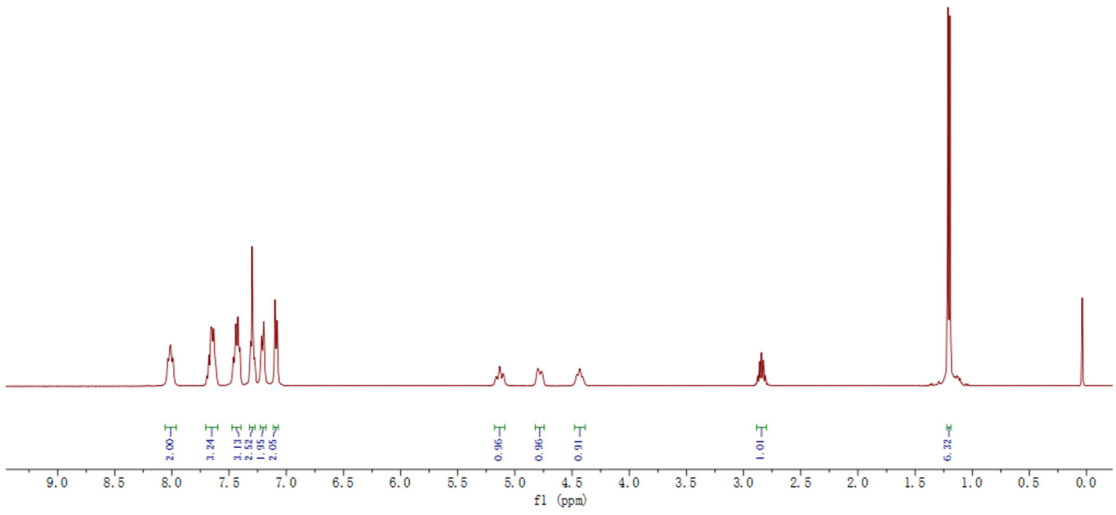
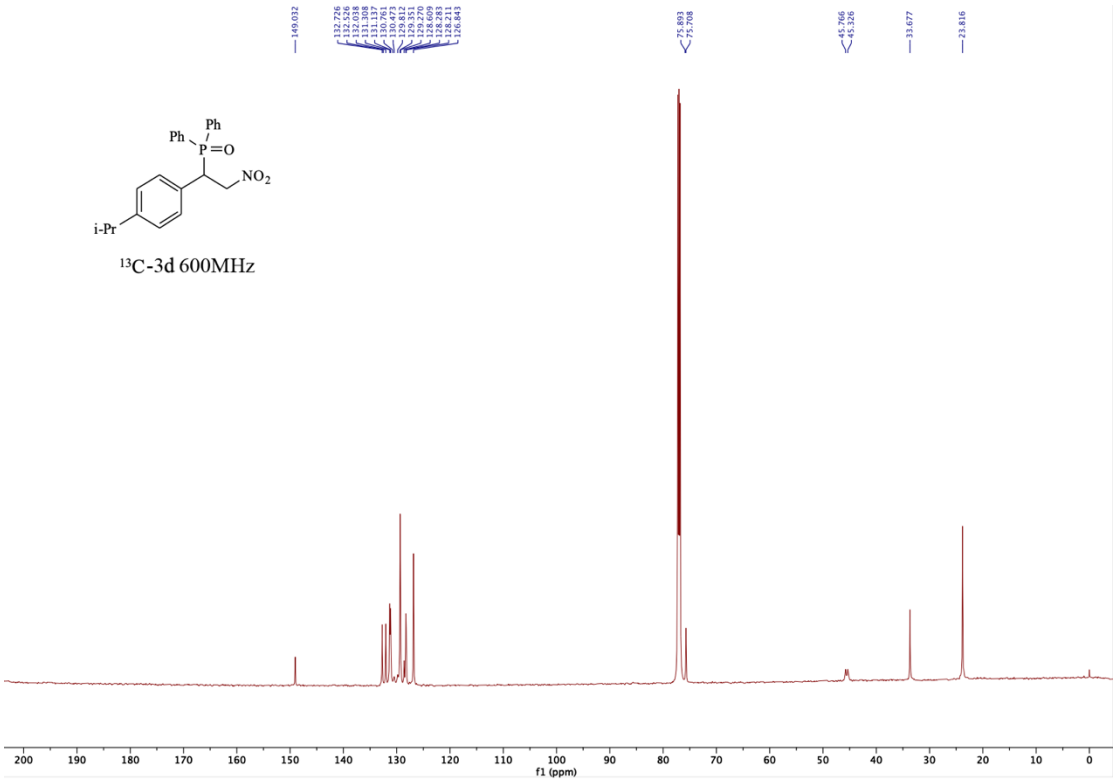


^{1}H-3d 400MHz





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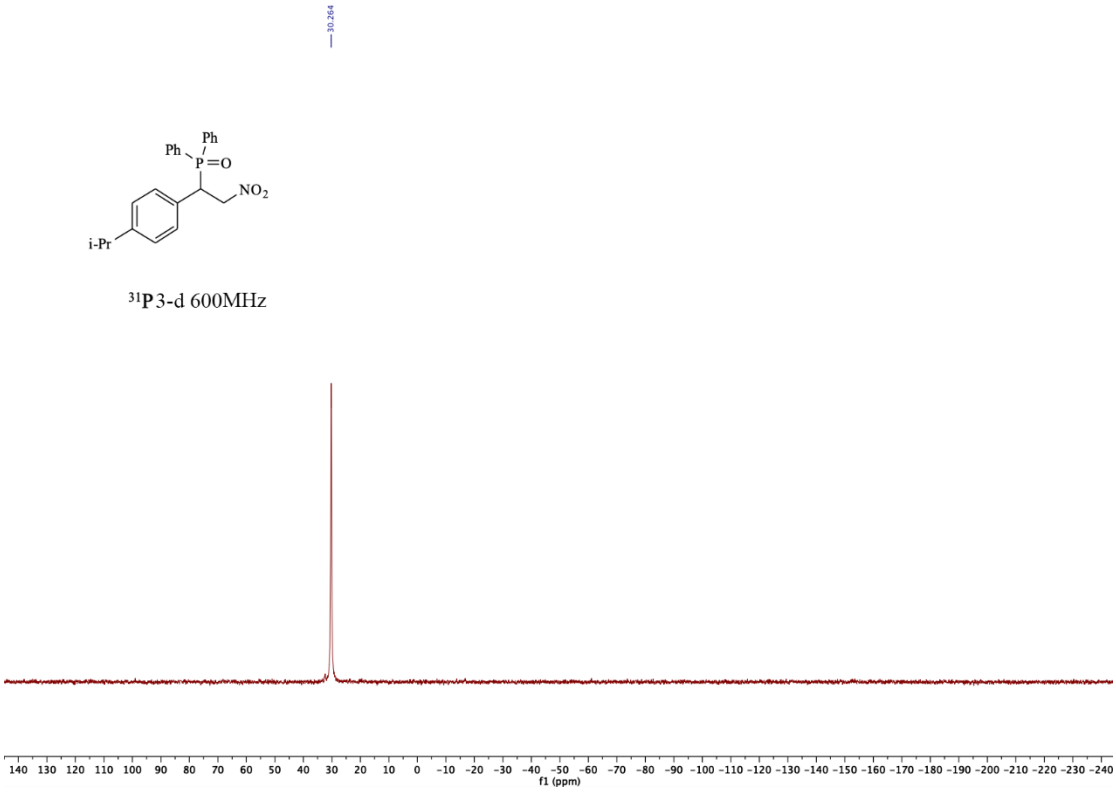

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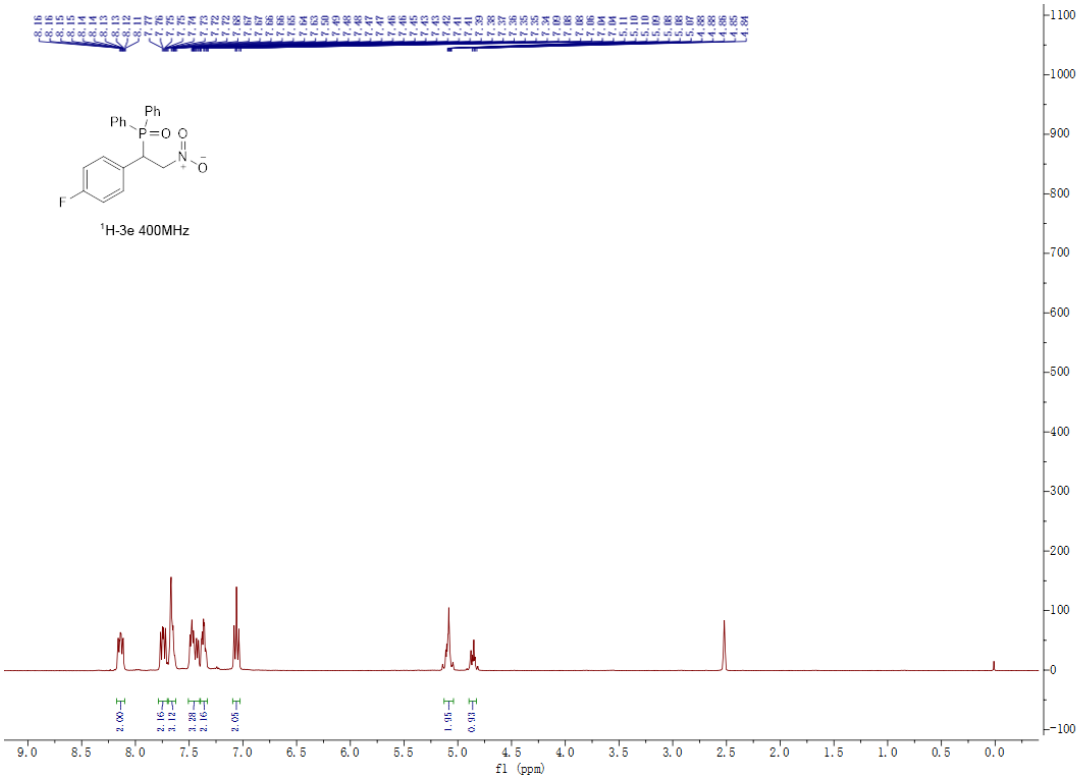

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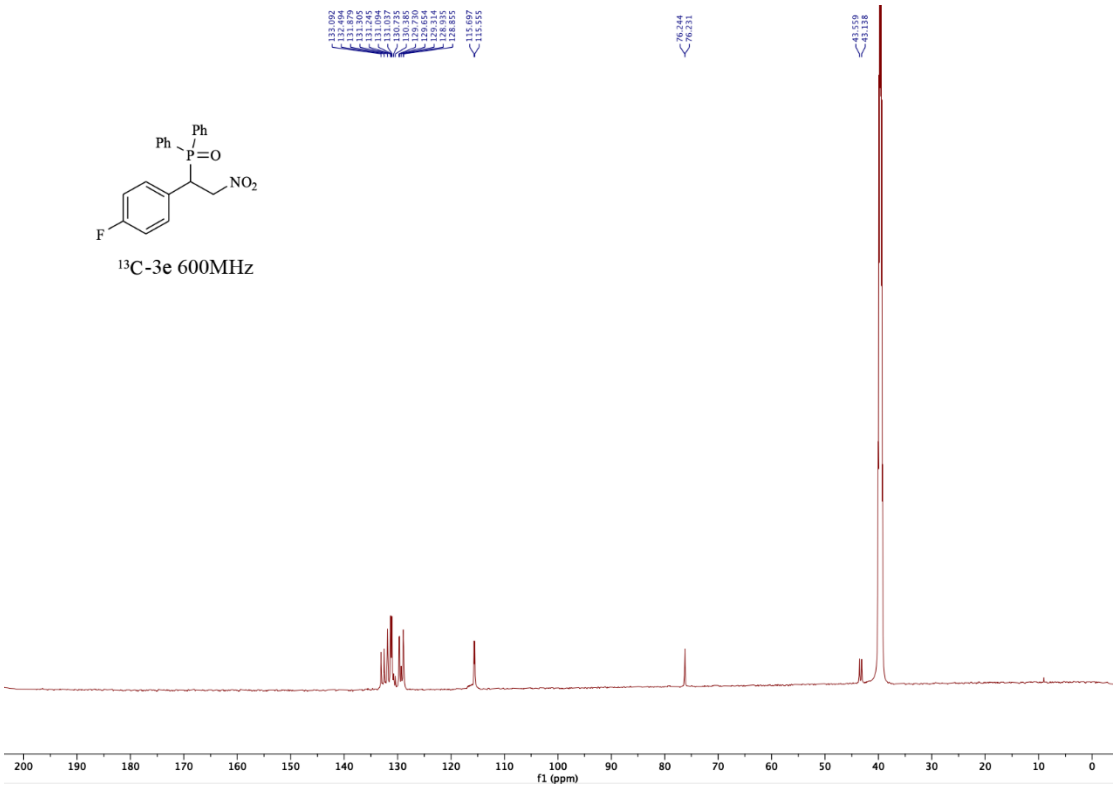

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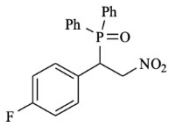

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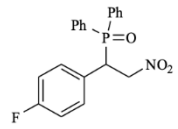
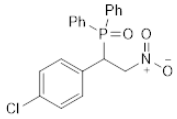


^{19}F 3-e 600 MHz



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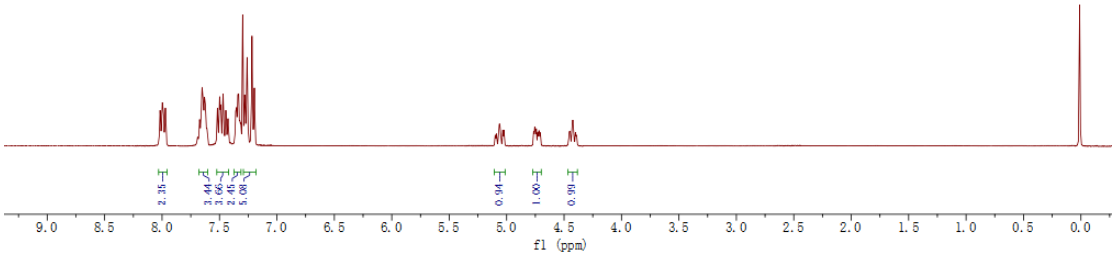

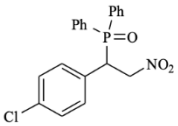

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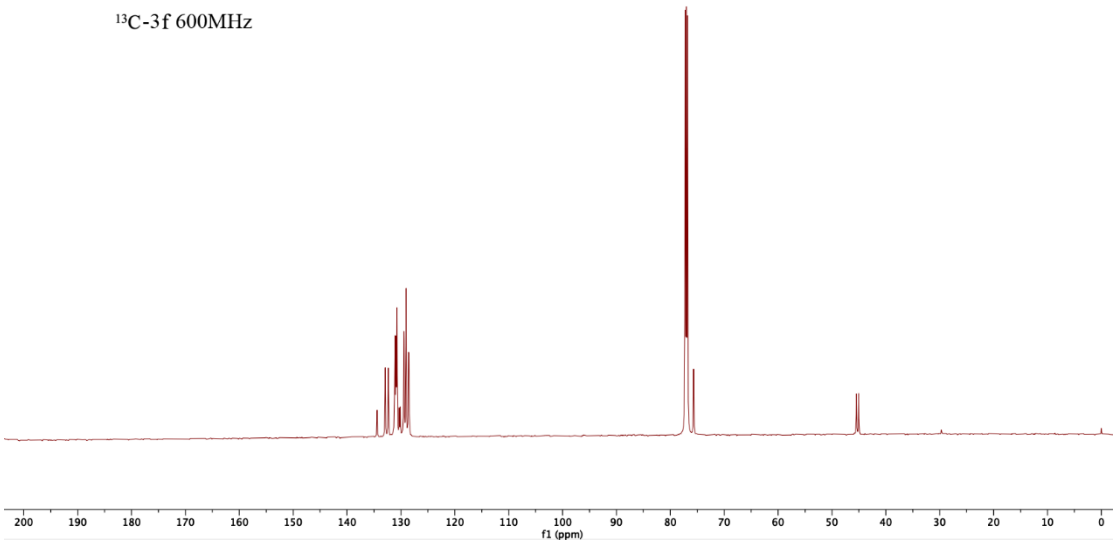

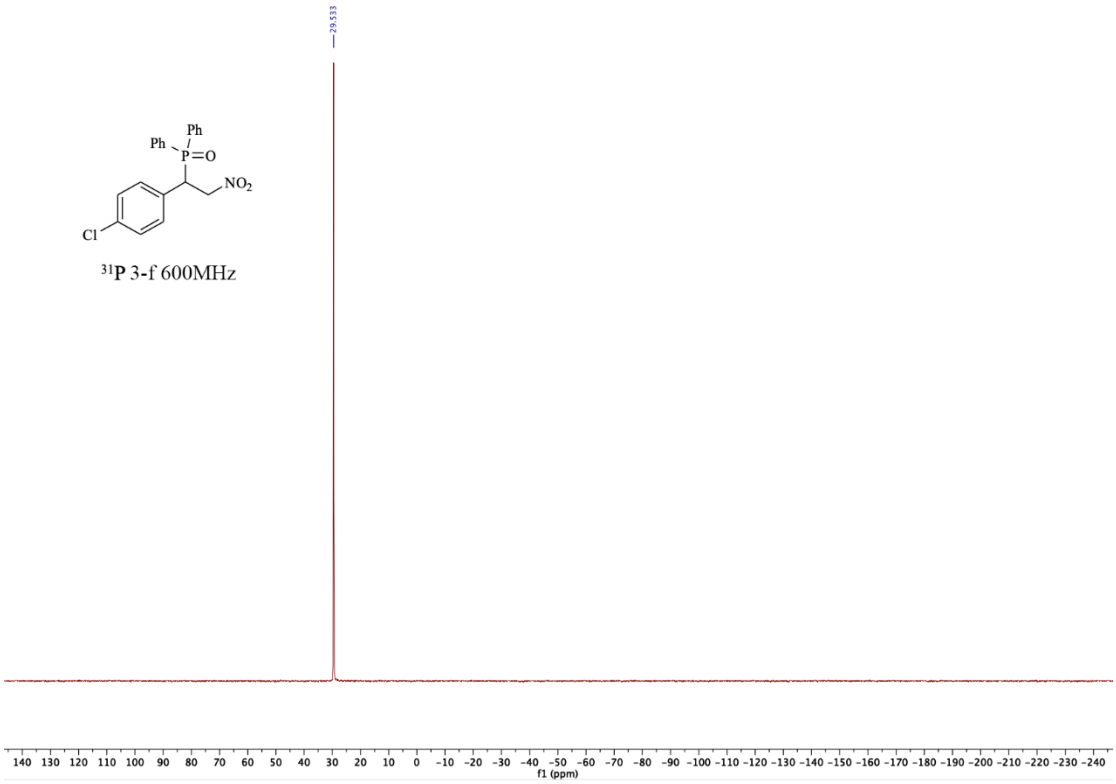

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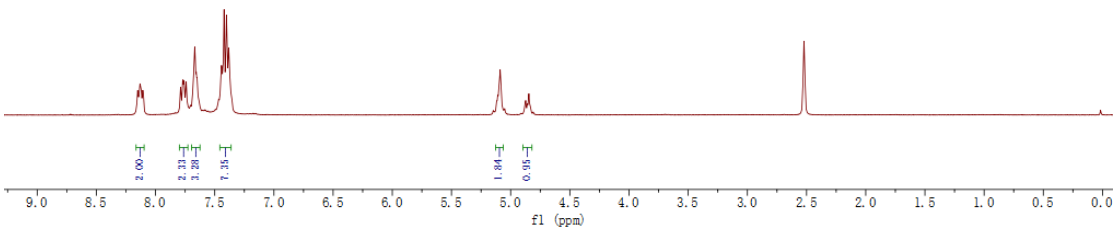

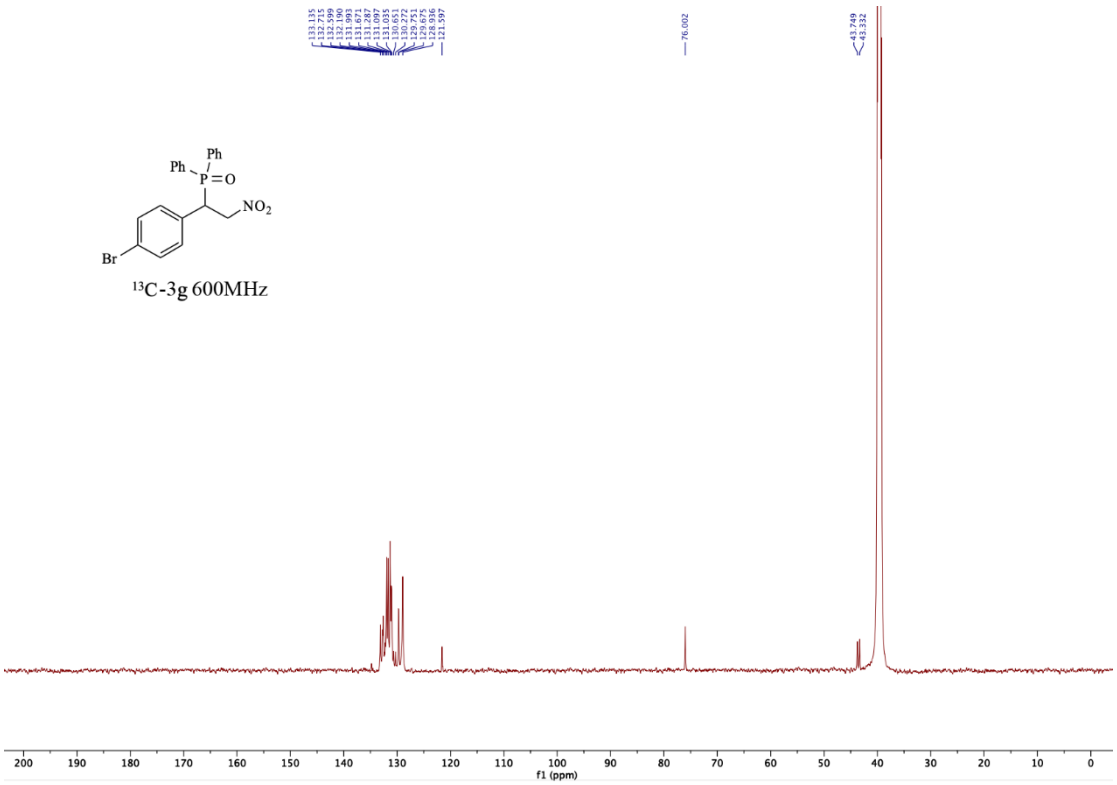

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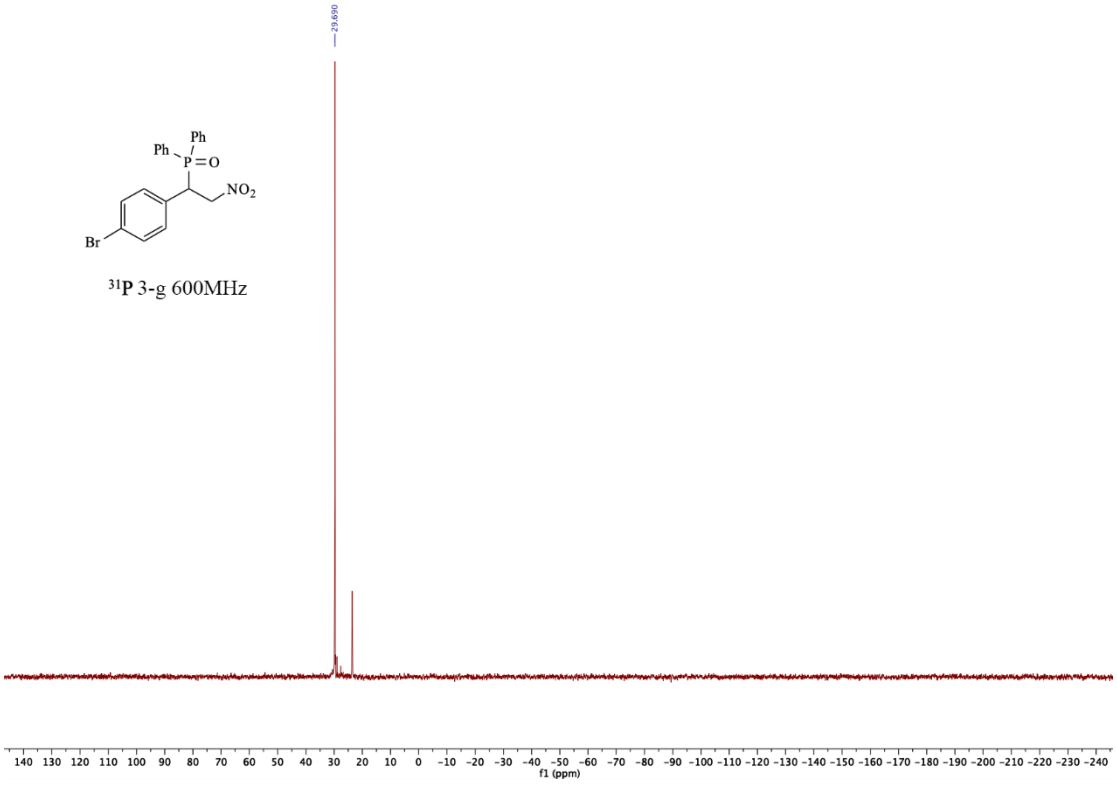

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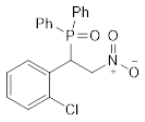

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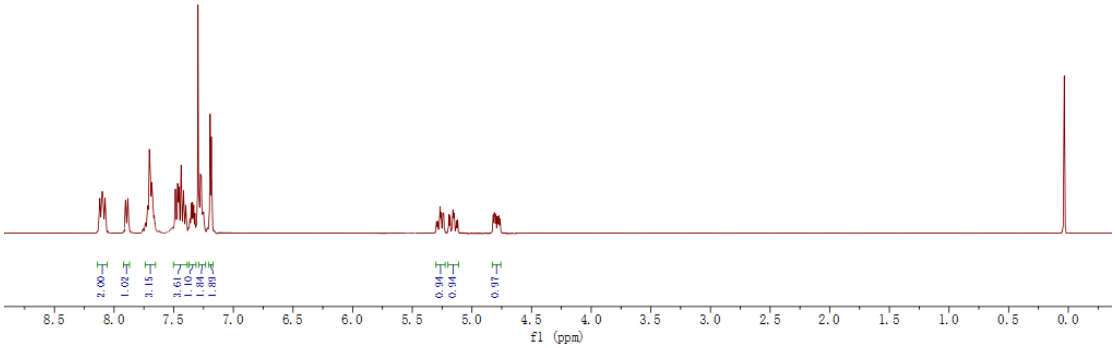

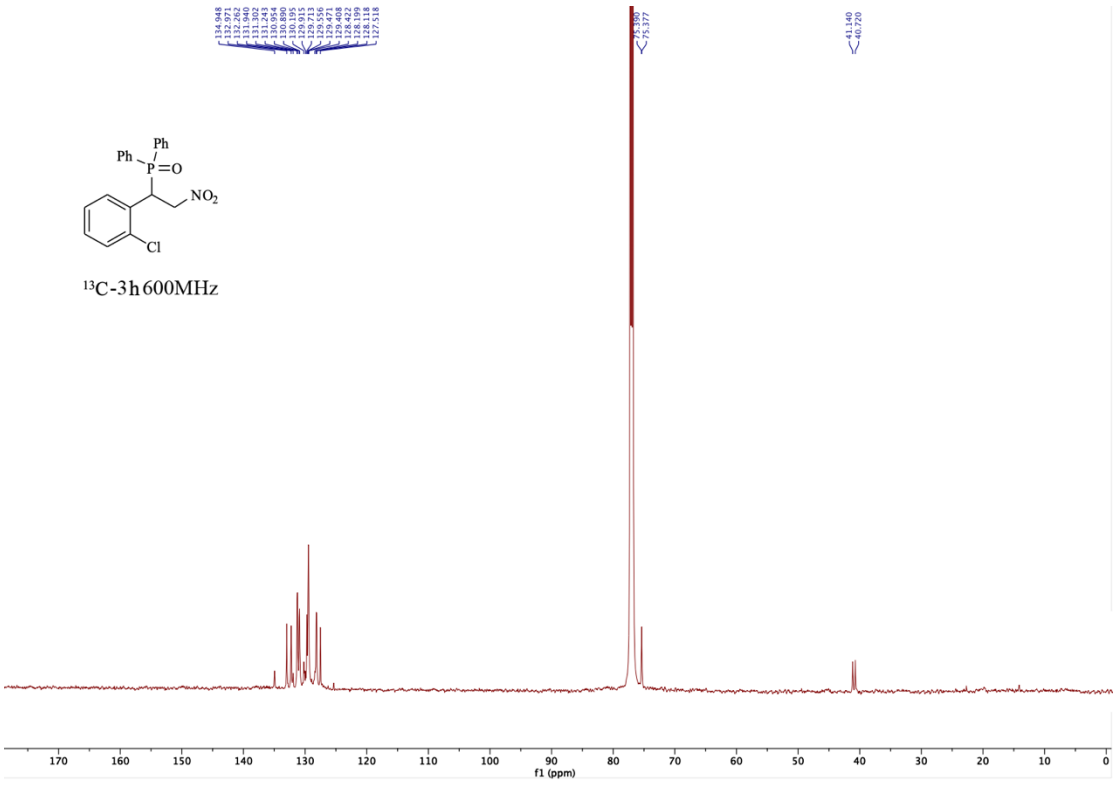

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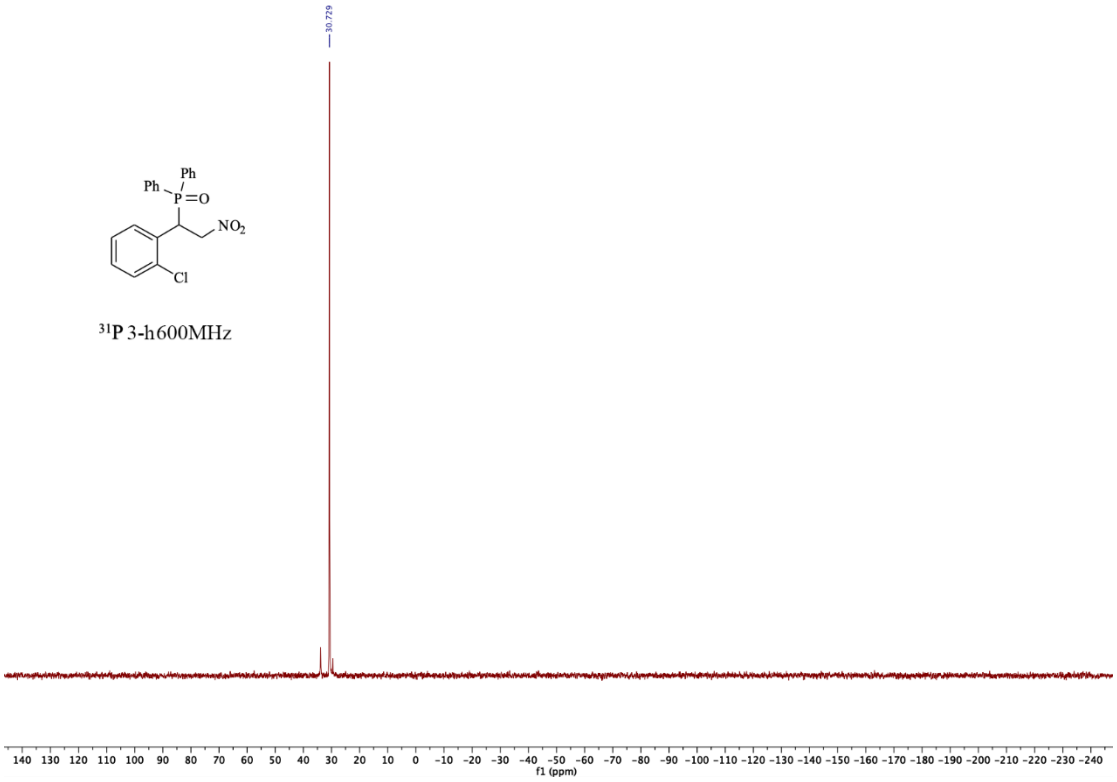

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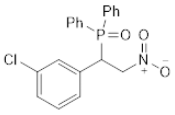

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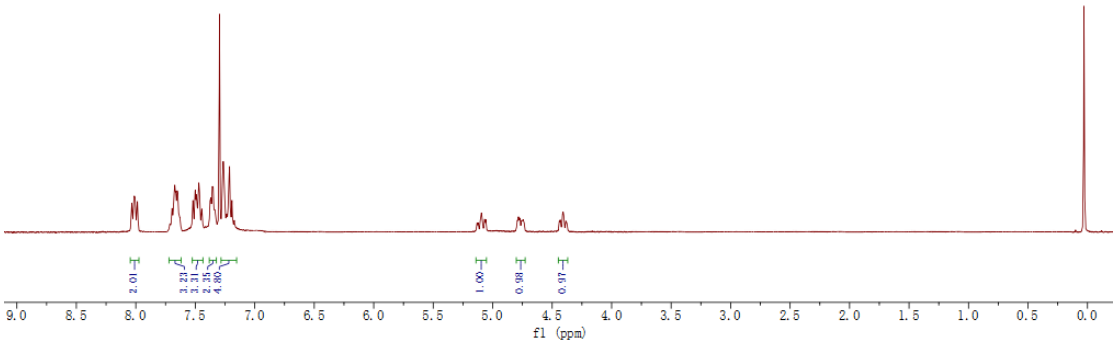

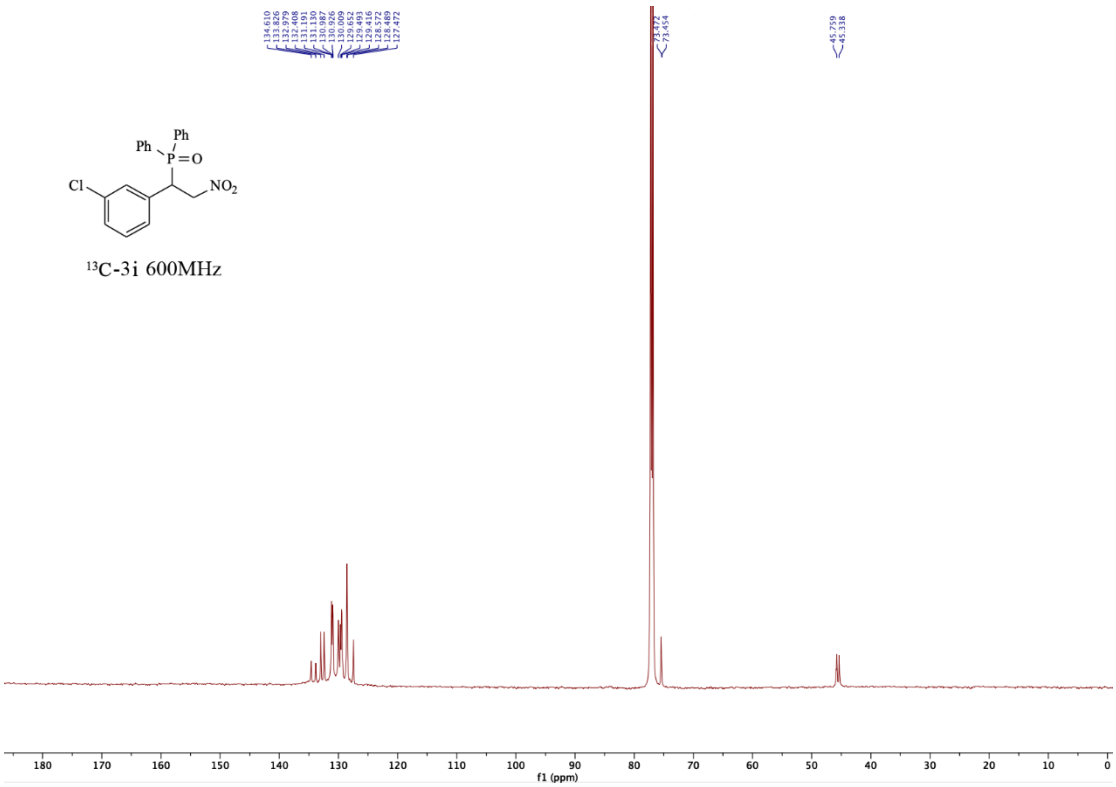

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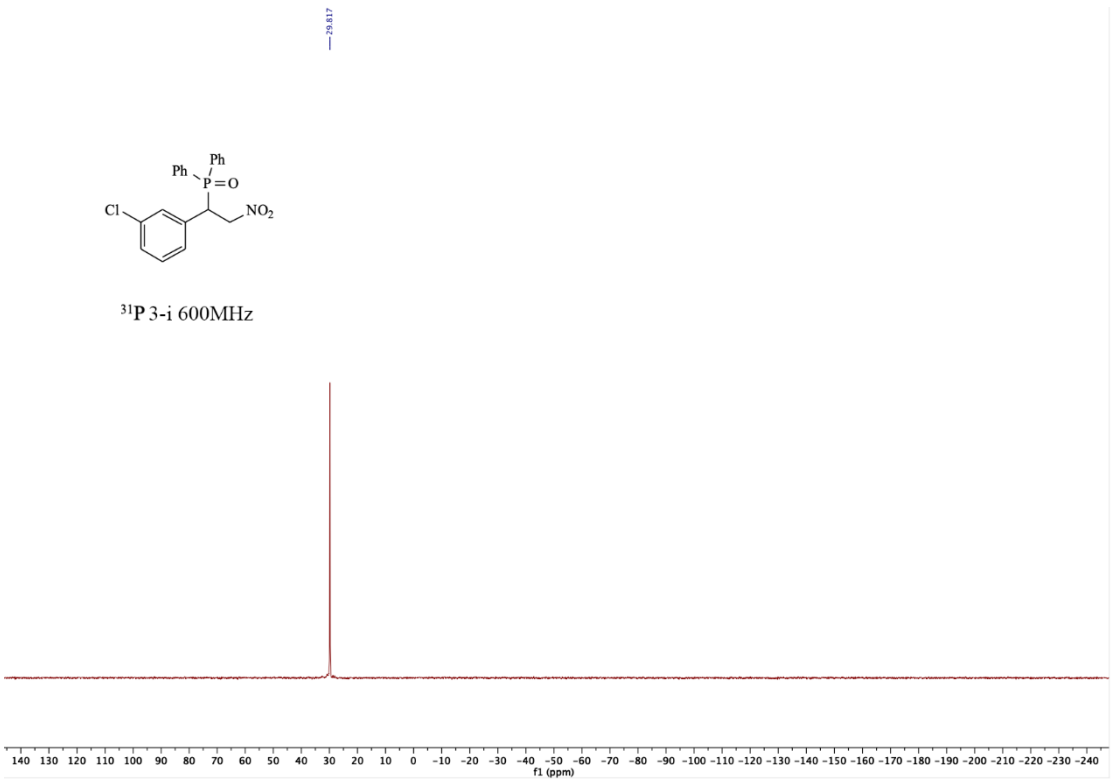

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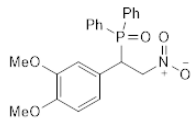

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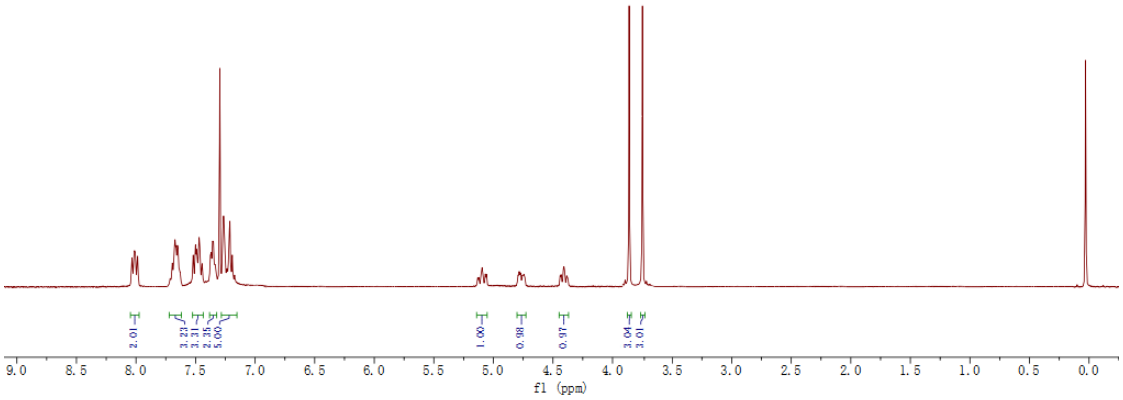

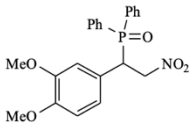

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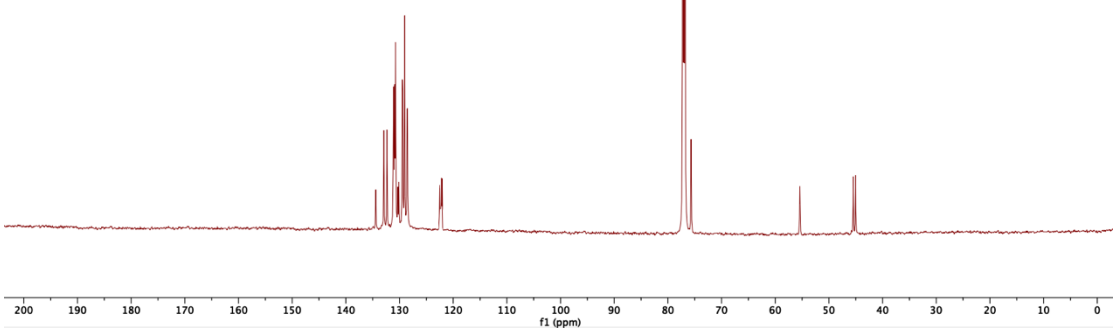

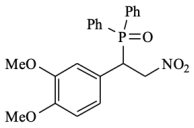

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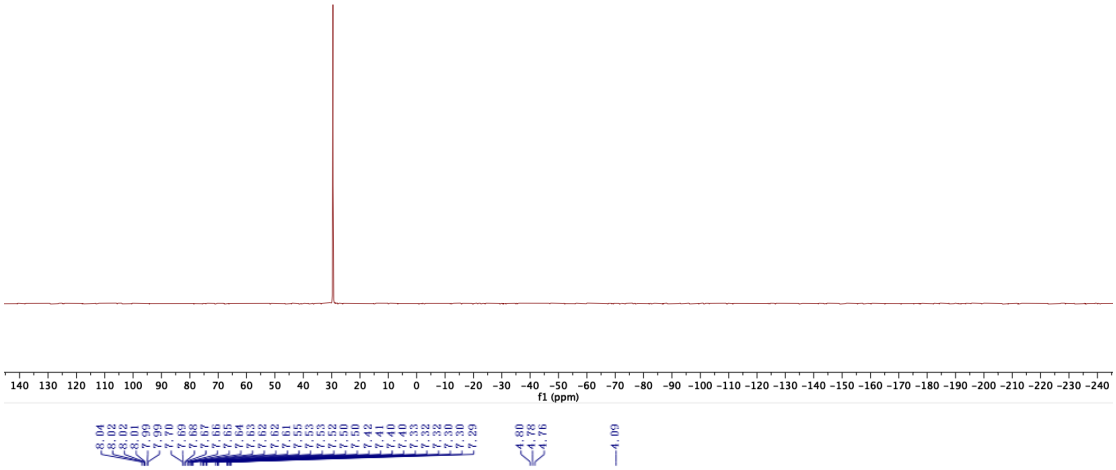
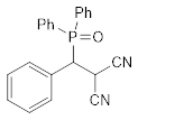
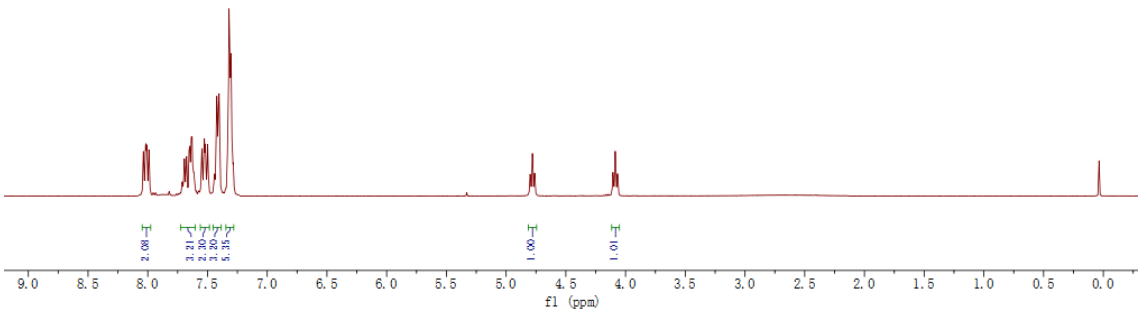
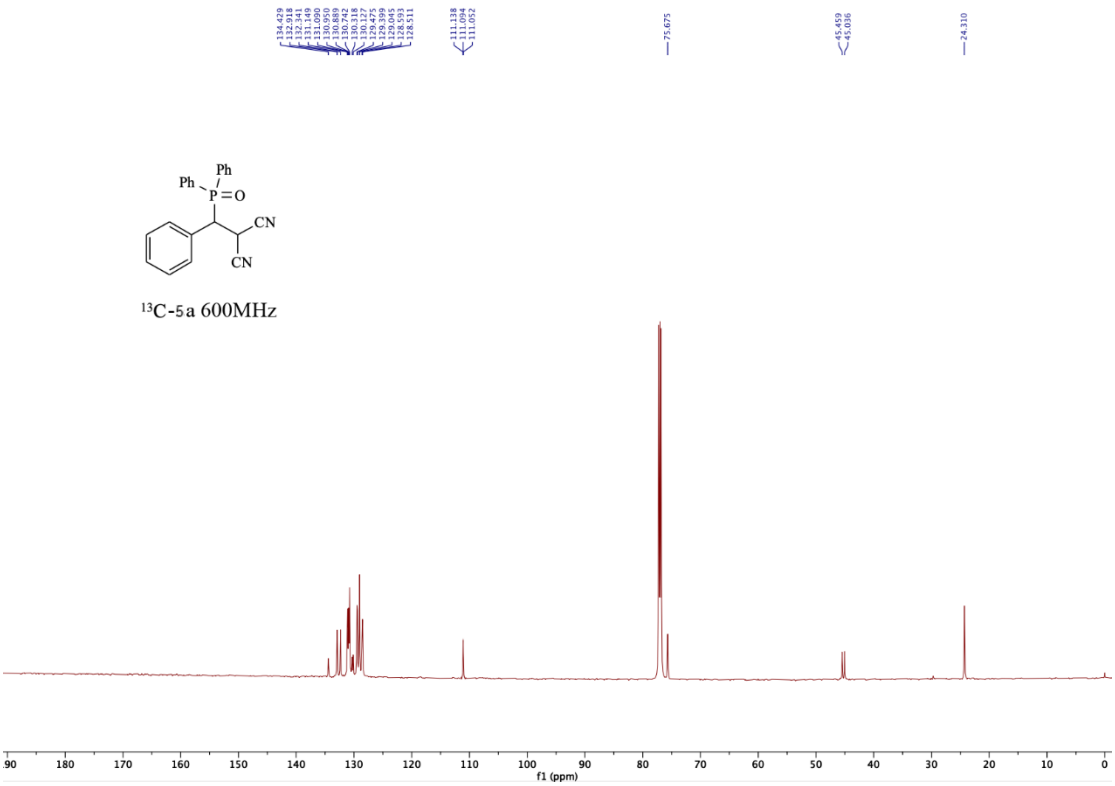


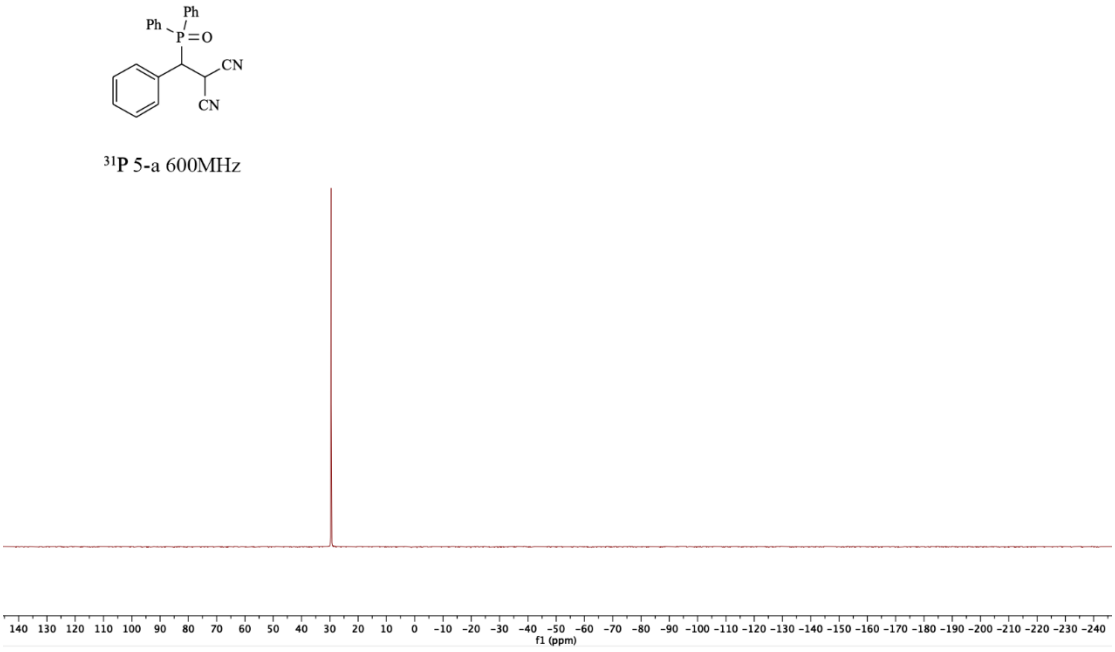


^{1}H-5a 400MHz

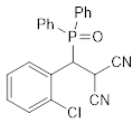




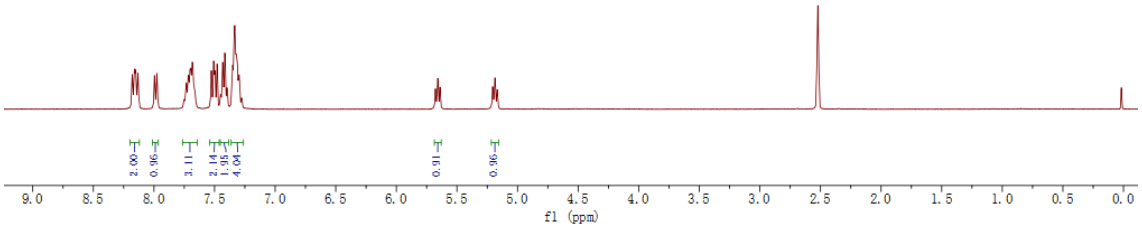
^{13}C-5a 600MHz

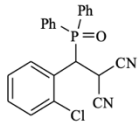


^{31}P 5-a 600MHz



^{1}H-5b 400MHz





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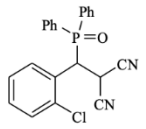

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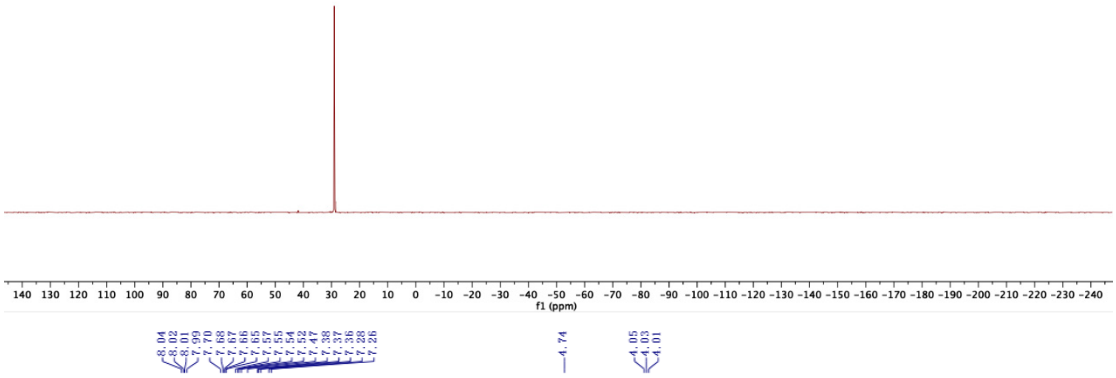
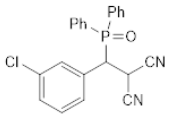
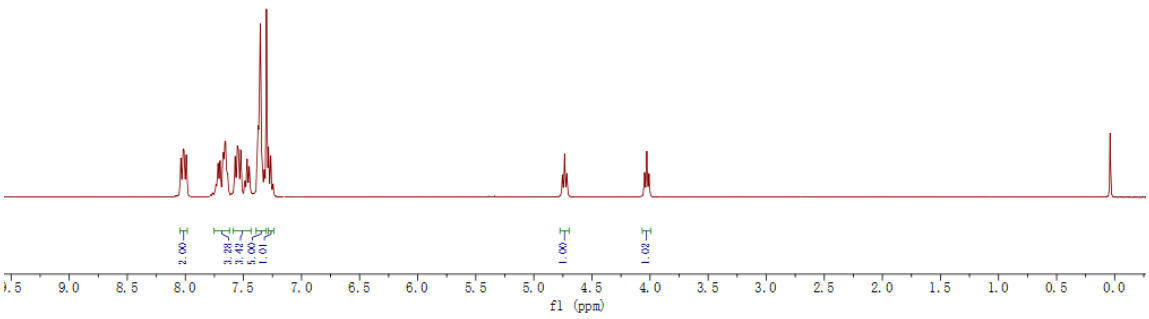
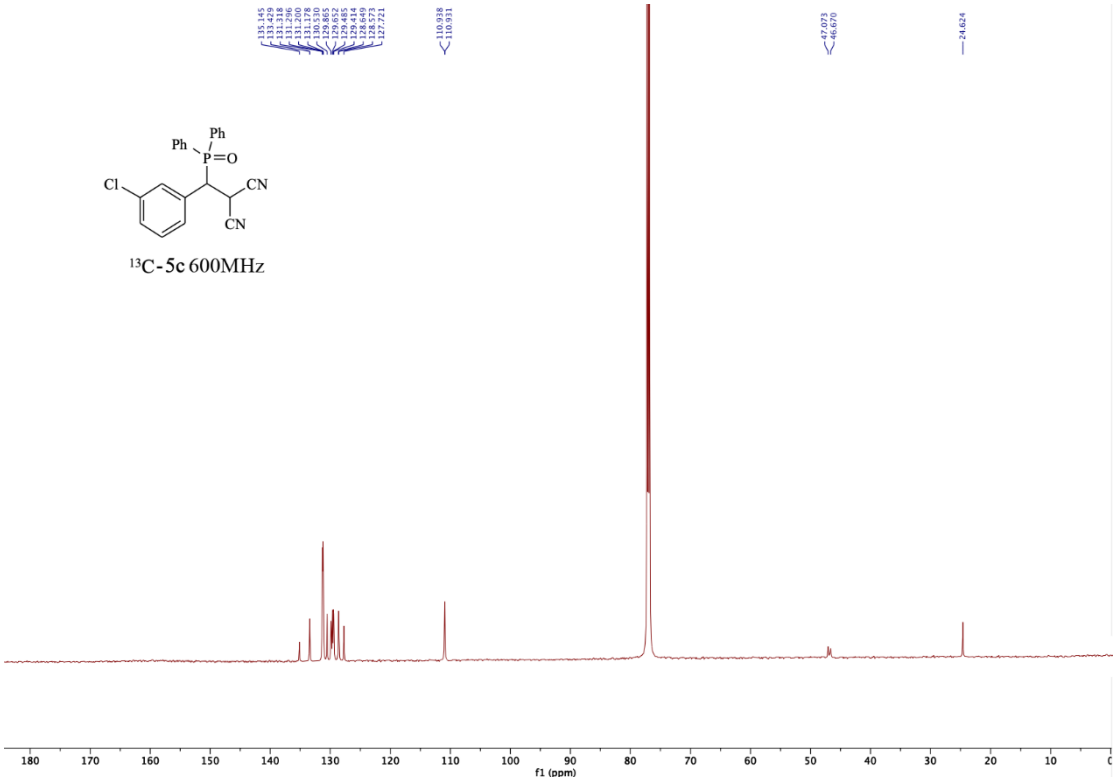




^{1}H-5c 400MHz





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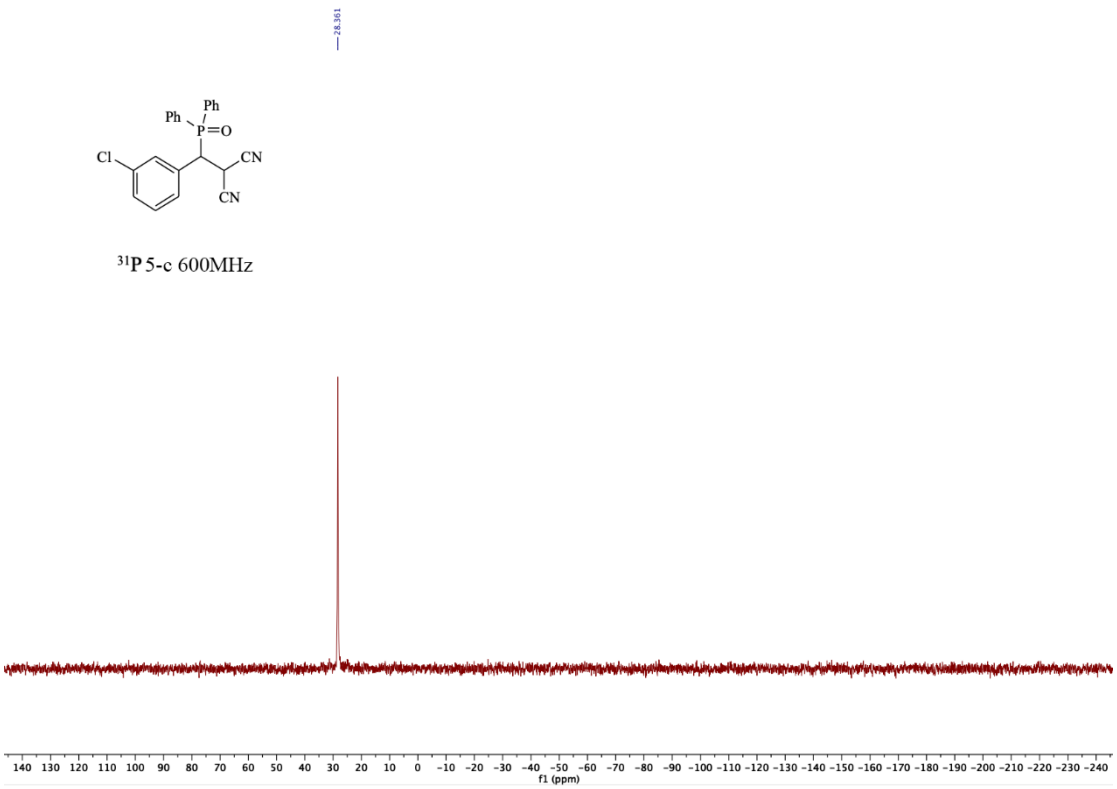

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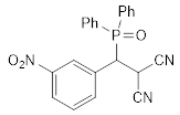
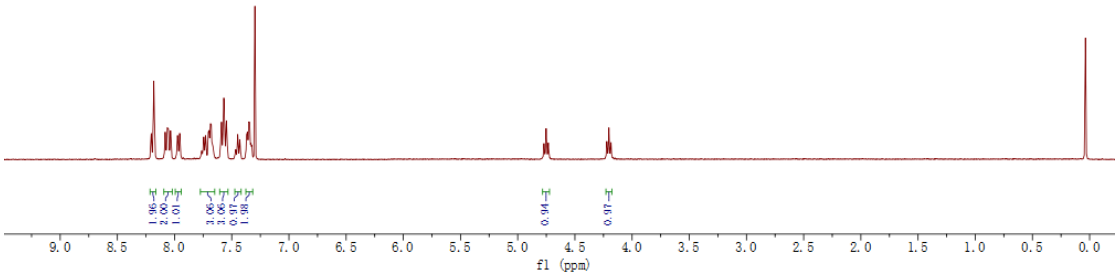
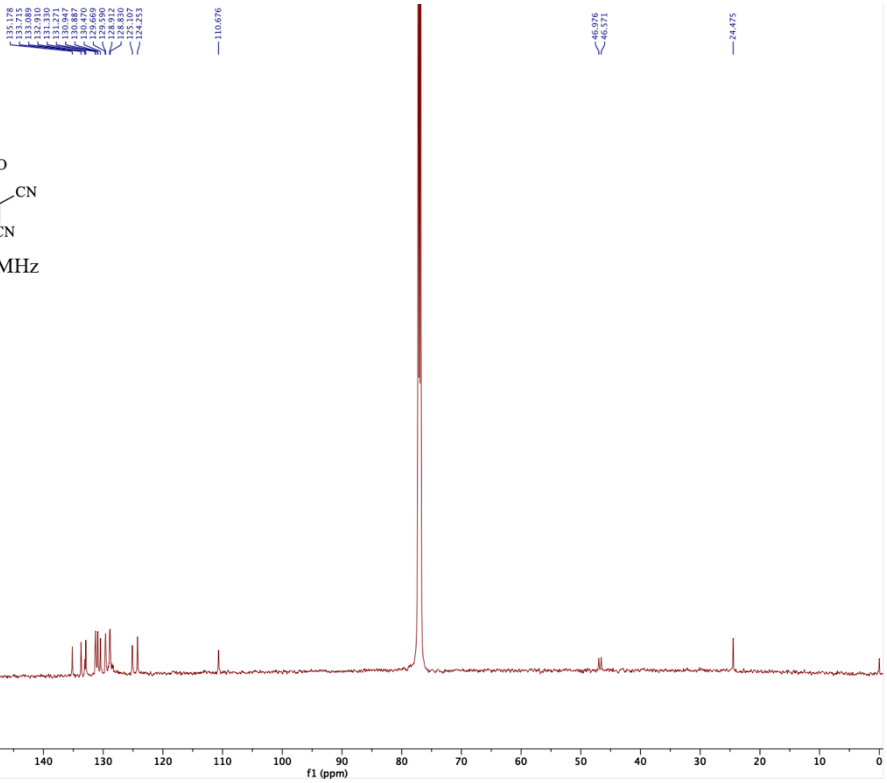
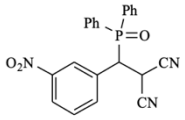


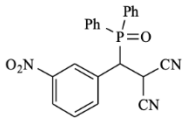
^{1}H-5d 400MHz



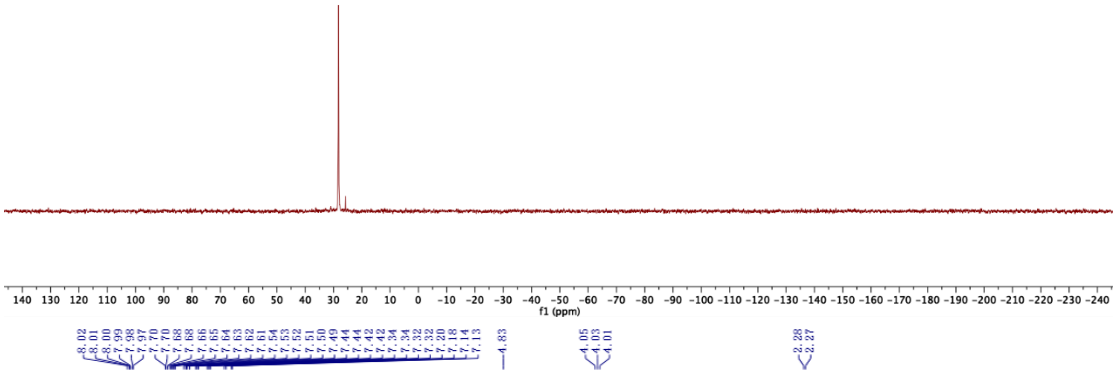


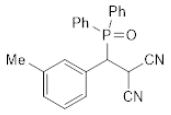


^{13}C-5d 600MHz

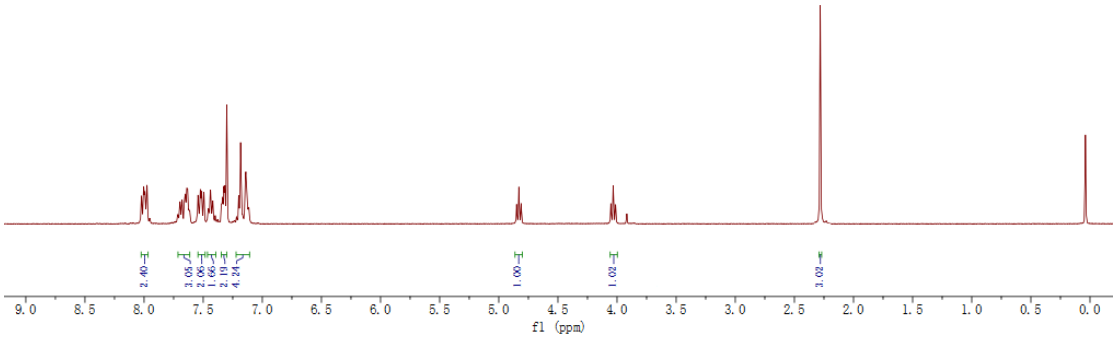


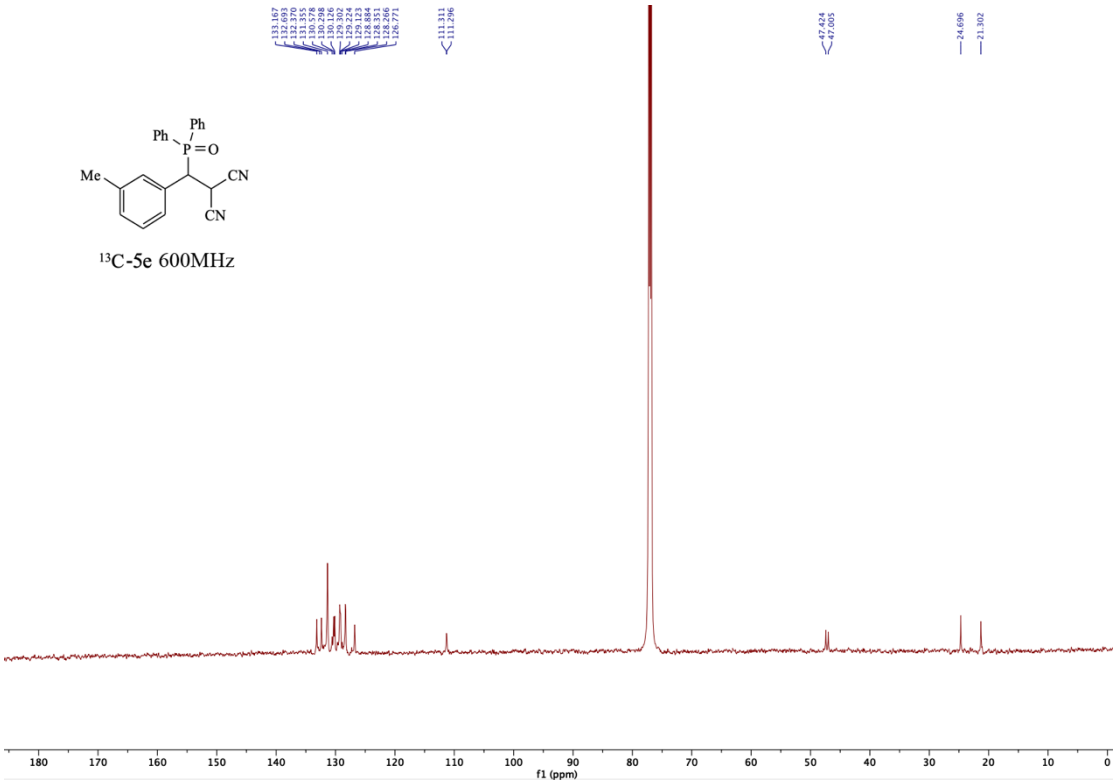
^{31}P 5-d 600MHz





^{1}H-5e 400MHz





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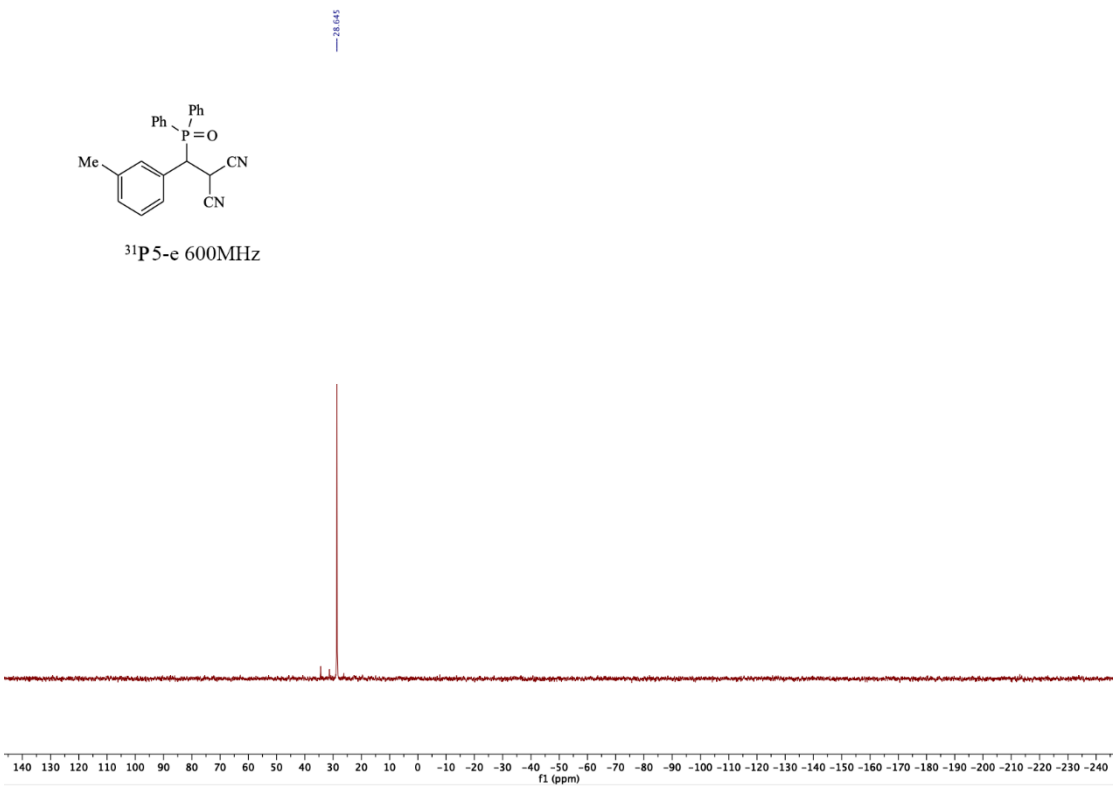

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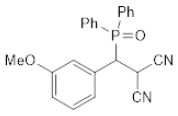
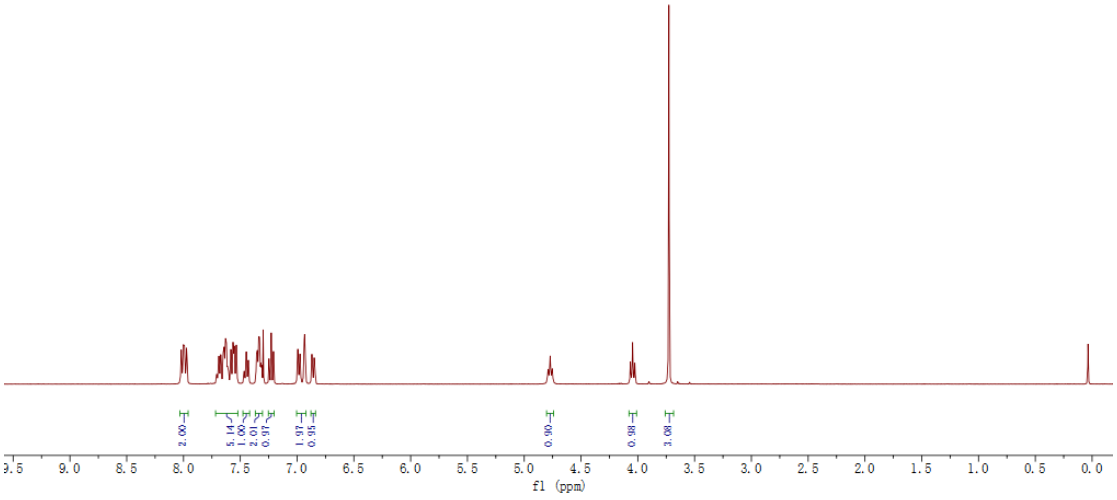
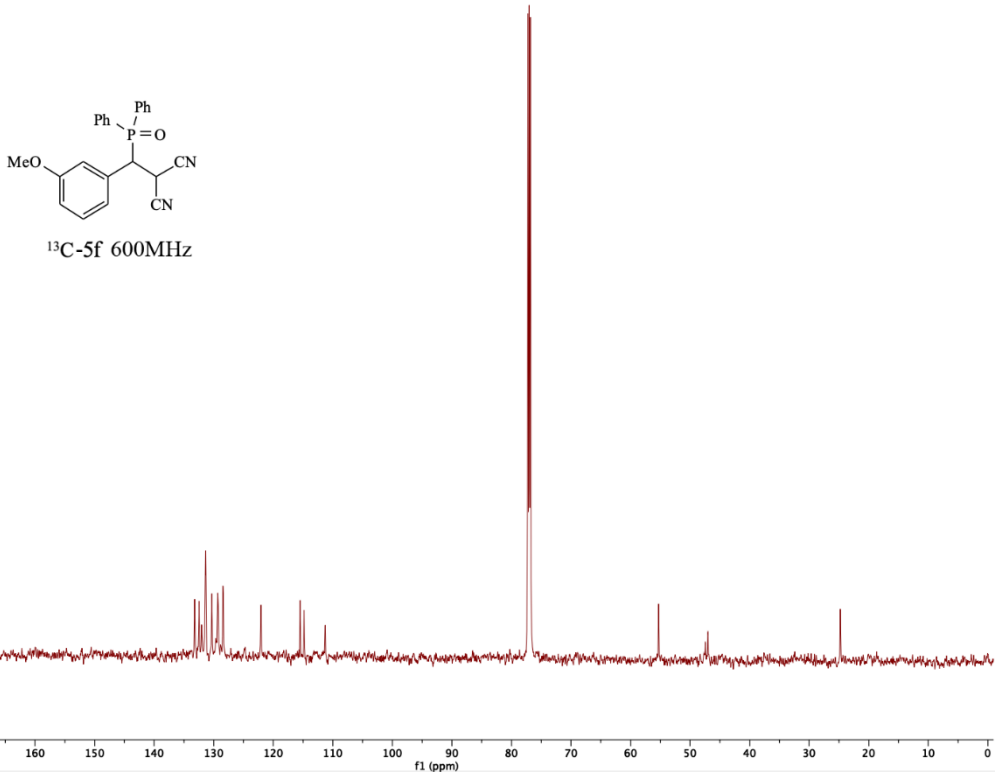


^{1}H-5f 400MHz





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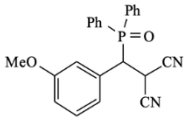
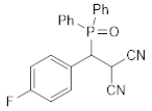
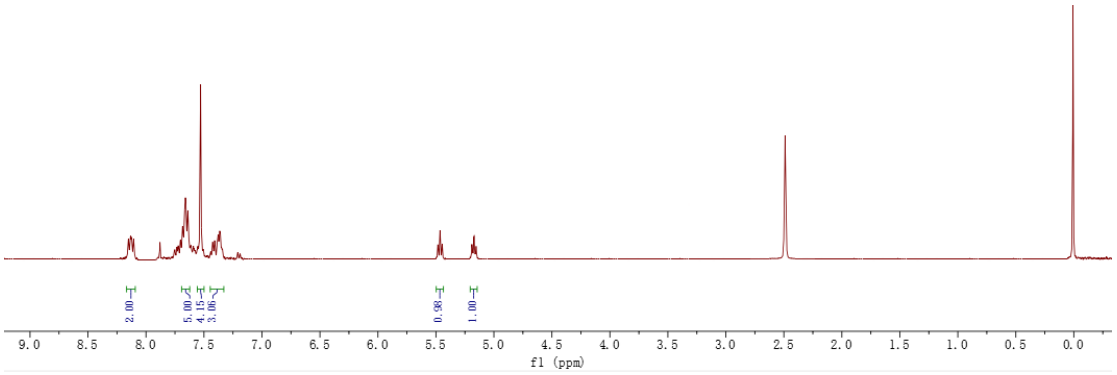
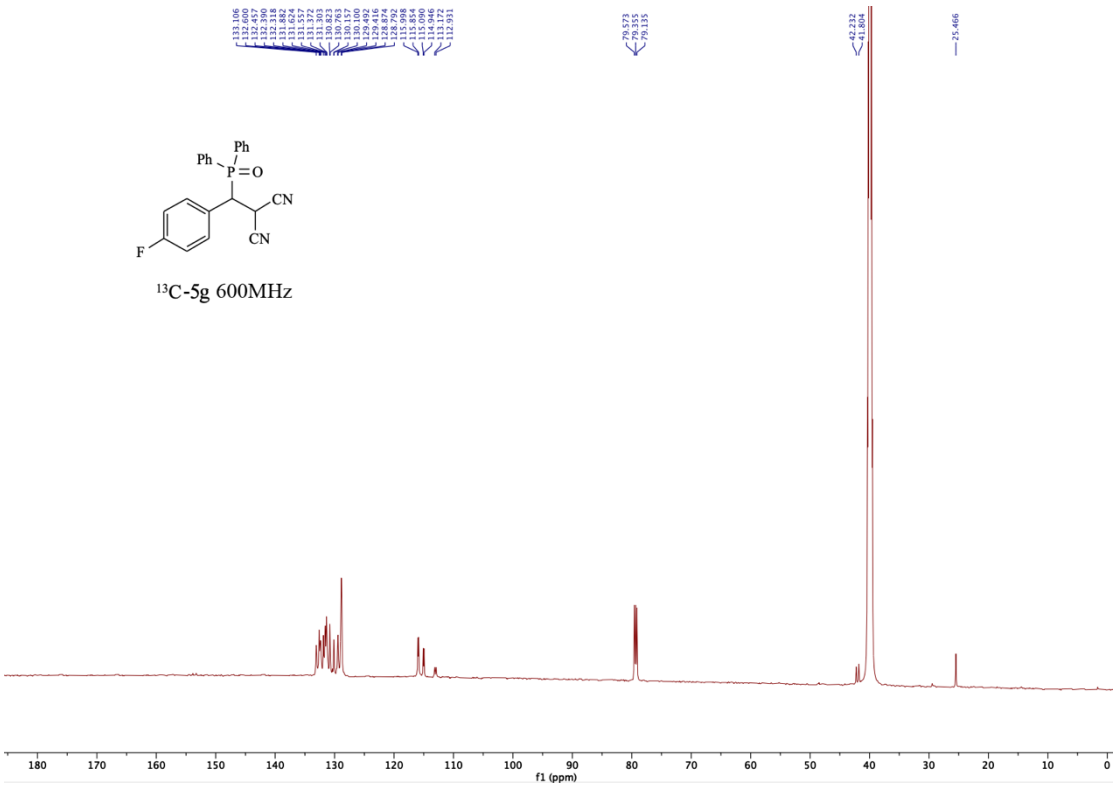


^{31}P 5-f 600MHz



^{1}H-5g 400MHz





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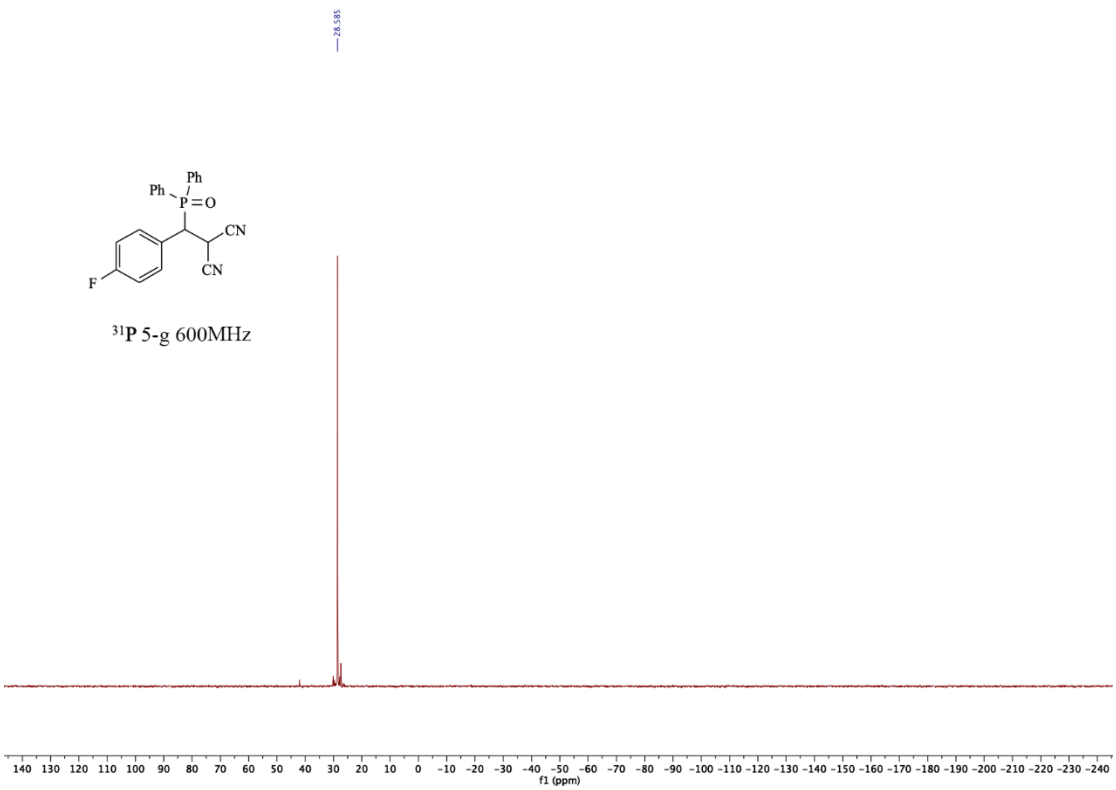
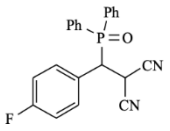
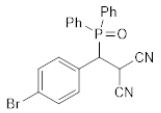


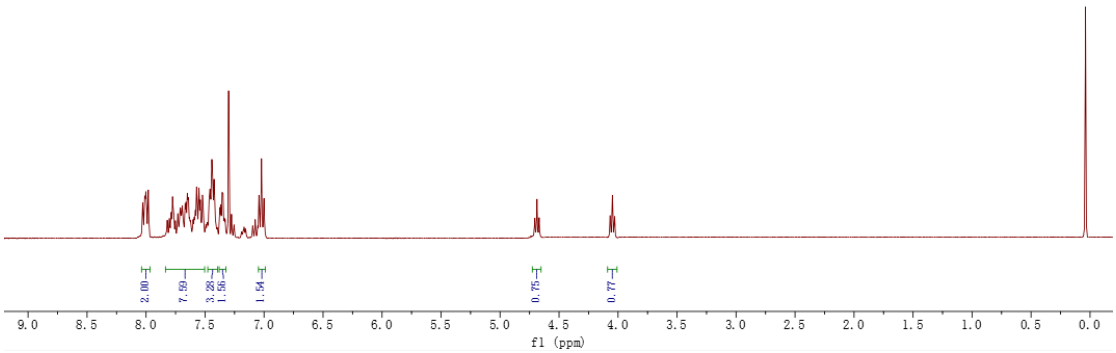
^{31}**P** 5-g 600MHz

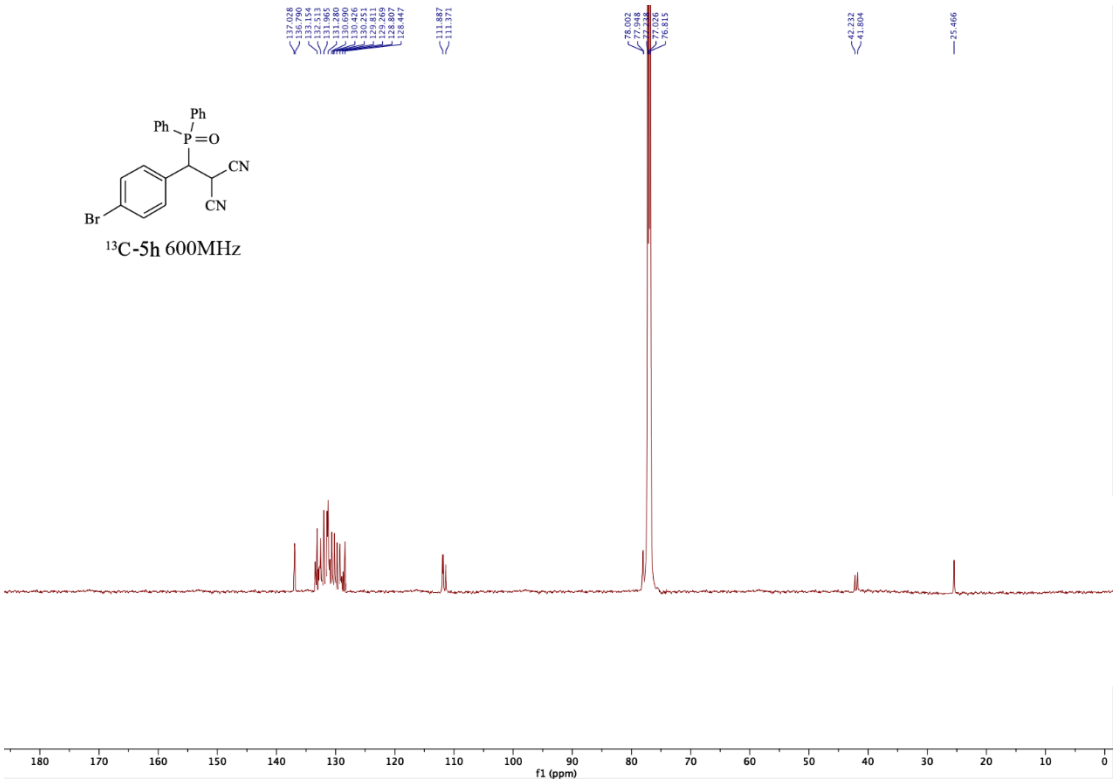


^{19}F 5-g 600MHz



^{1}H-5h 400MHz





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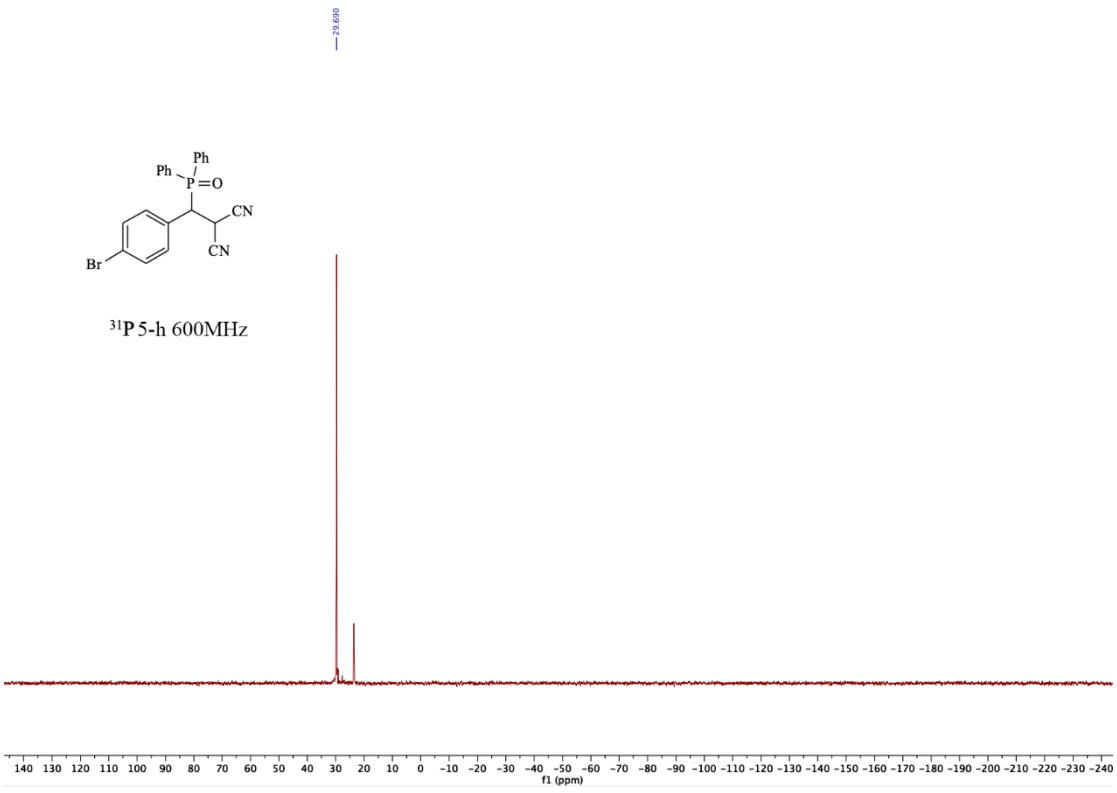
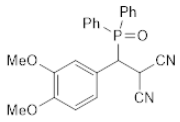
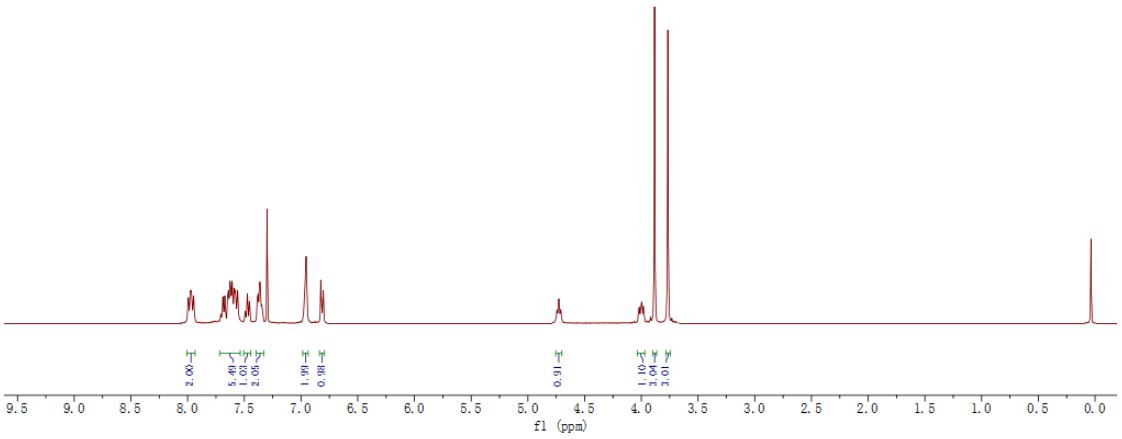
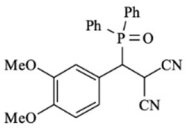


^{31}P 5-h 600MHz



^{1}H-5i 400MHz





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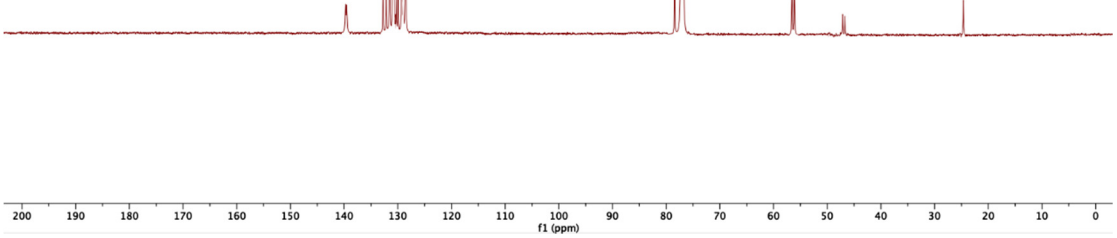
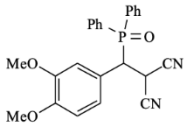
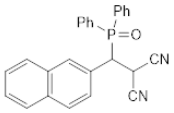
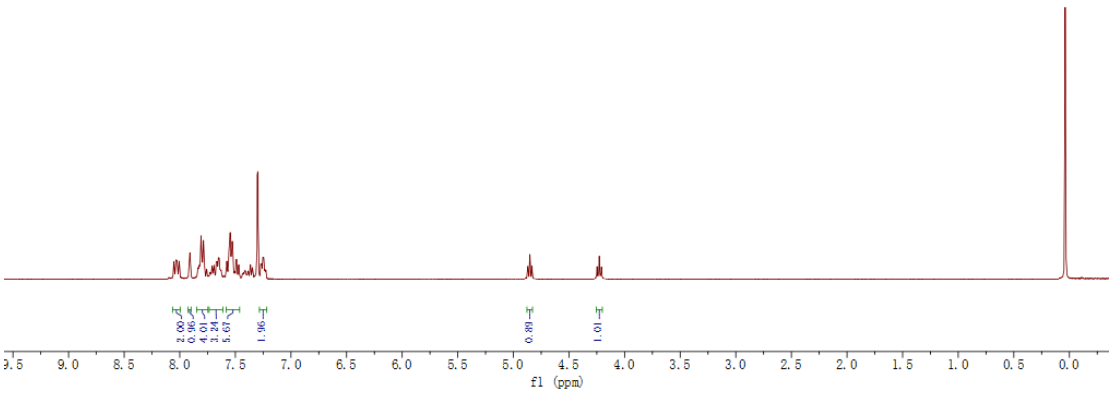


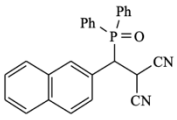


^{31}P 5-i 600MHz



^{1}H-5j 400MHz





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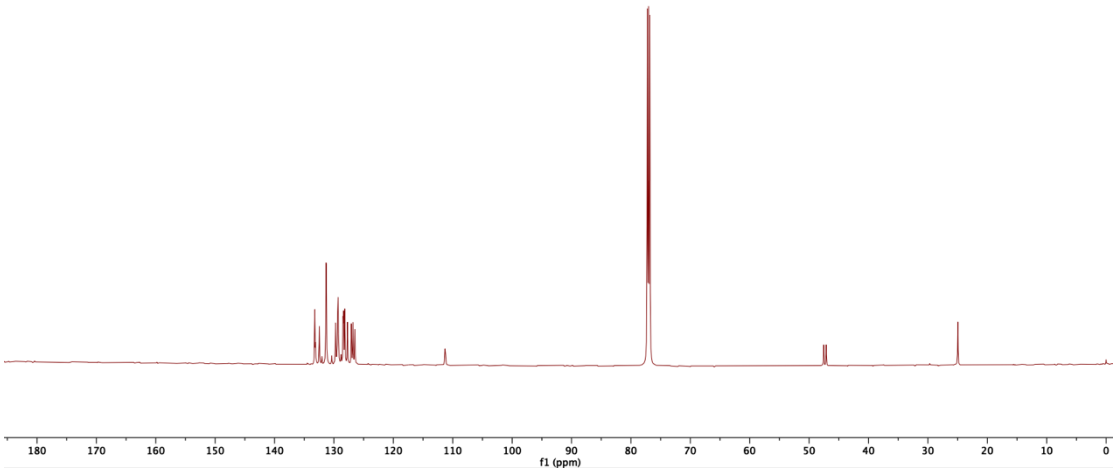
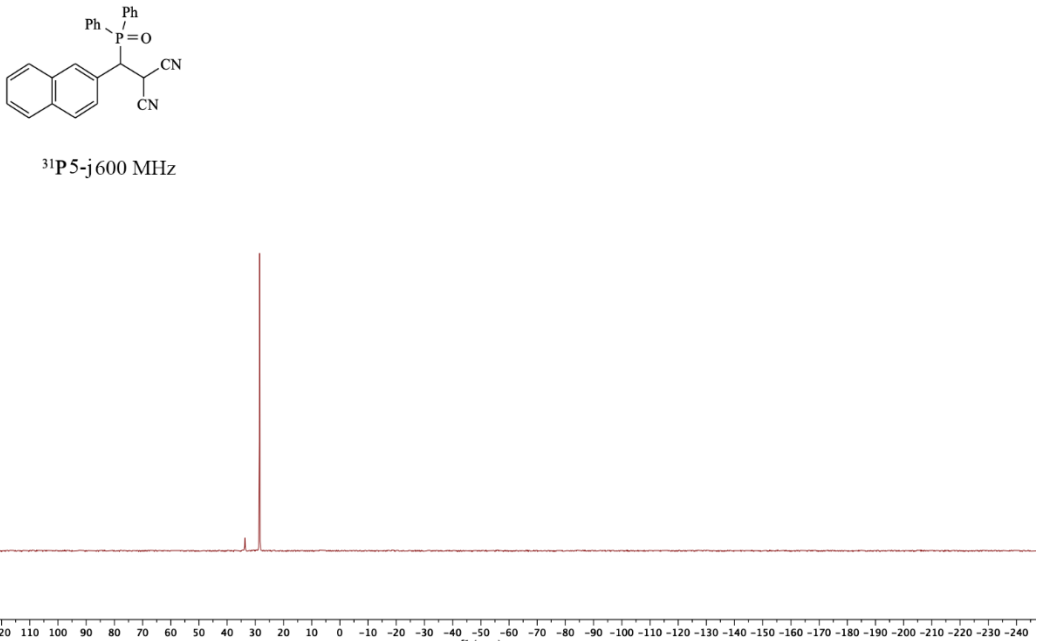
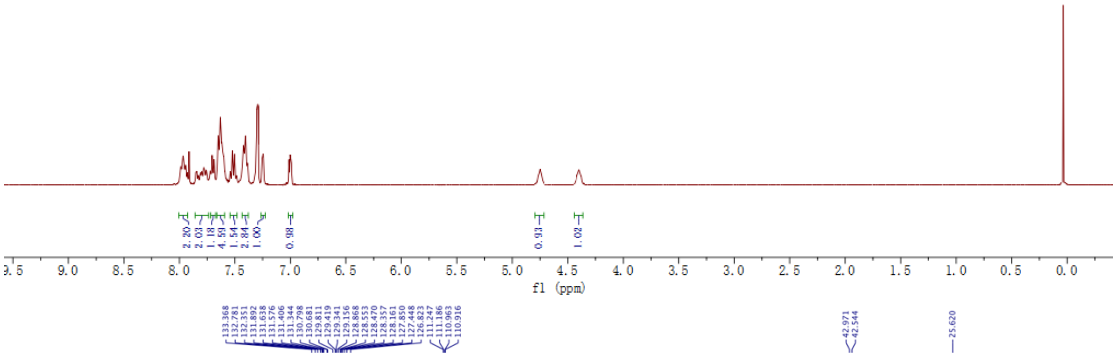
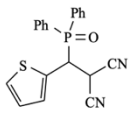




^{31}P 5-j 600 MHz

^{1}H-5k 400MHz





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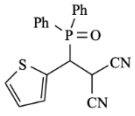

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