

Supporting Information

Lewis Acid Catalyzed Phosphorylation of Alcohol

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Contents

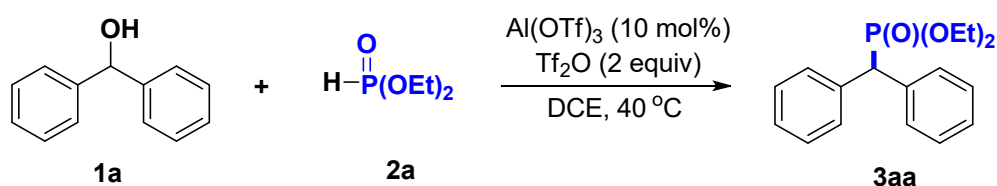
General	S2
Experimental Section	S2
1. General procedure for synthesis of 3	S2
2. Analytical Data of Products	S2
¹ H, ¹³ C and ³¹ P NMR and spectra of Products	S8

General

All chemicals were purchased from Aldrich and J&K Chemical and used without further purification. Thin-layer chromatography (TLC) was performed using 60 mesh silica gel plates visualized with short-wavelength UV light (254 nm). Silica gel 60 (230-400 mesh) was used for column chromatography. ^1H , ^{13}C and ^{31}P NMR spectra were recorded using CDCl_3 solvent on a Bruker advance III 400 spectrometer (400 MHz for ^1H , 162 MHz for ^{31}P and 101 MHz for ^{13}C), The chemical shift is given in dimensionless δ values and is frequency referenced relative to TMS in ^1H and ^{13}C NMR spectroscopy. HRMS were measured with Bruker micro TOF-Q II mass spectrometer equipped with electrospray ionization (ESI).

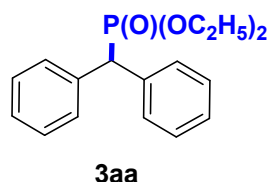
Experimental Section

1. General procedure for synthesis of 3



An oven-dried 10 mL screw-capped vial containing **1a** (0.3 mmol, 1.0 equiv), $\text{Al}(\text{OTf})_3$ (0.03 mmol, 0.1 equiv), and DCE (2 mL) was added *via* syringe, diethyl phosphonate **2a** (0.75 mmol, 2.5 equiv), Tf_2O (0.6 mmol, 2.0 equiv), and then heated to 40°C in an oil bath until the starting material has disappeared for 14 hours (monitored by TLC). And then the solvent was removed in vacuo and residue was purified by column chromatography on a short silica gel column using EA/PE as eluent to afford the desired product **3aa**.

2. Analytical Data of Products



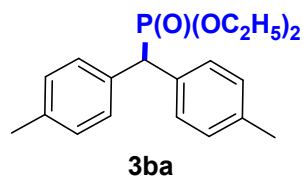
Colorless liquid. ^1H NMR (400 MHz, CDCl_3) δ 7.53 (d, $J = 7.8$ Hz, 4H), 7.30 (t, $J = 7.4$ Hz, 4H), 7.27 – 7.18 (m, 2H), 4.43 (d, $J = 25.1$ Hz, 1H), 4.05 – 3.91 (m, 2H), 3.91 – 3.75 (m, 2H), 1.10 (t, $J = 7.1$ Hz, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 136.72 (d, $J = 5.2$ Hz), 129.36 (d, $J = 8.0$ Hz), 128.47

(d, $J = 1.2$ Hz), 127.03 (d, $J = 2.1$ Hz), 62.59 (d, $J = 7.0$ Hz), 51.19 (d, $J = 138.2$ Hz), 16.13 (d, $J = 5.8$ Hz).

^{31}P NMR (162 MHz, CDCl_3) δ 25.07.

HRMS (ESI) Calcd. for $\text{C}_{17}\text{H}_{21}\text{O}_3\text{P}$ $[\text{M}+\text{Na}]^+$: 327.1121, found 327.1120.

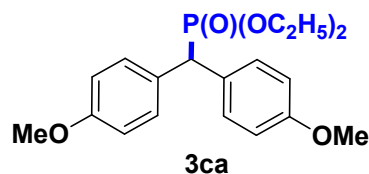


Yellowish liquid. ^1H NMR (400 MHz, CDCl_3) δ 7.39 (d, $J = 8.1$ Hz, 4H), 7.10 (d, $J = 8.0$ Hz, 4H), 4.36 (d, $J = 25.1$ Hz, 1H), 4.06 – 3.91 (m, 2H), 3.91 – 3.74 (m, 2H), 2.29 (s, 6H), 1.12 (t, $J = 7.1$ Hz, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 136.52 (d, $J = 2.3$ Hz), 133.85 (d, $J = 5.2$ Hz), 129.14 (d, $J = 1.3$ Hz), 129.07, 62.50 (d, $J = 7.1$ Hz), 50.27 (d, $J = 138.3$ Hz), 16.16 (d, $J = 5.8$ Hz), 20.92.

^{31}P NMR (162 MHz, CDCl_3) δ 25.57.

HRMS (ESI) Calcd. for $\text{C}_{19}\text{H}_{25}\text{O}_3\text{P}$ $[\text{M}+\text{Na}]^+$: 355.1434, found 355.1429.

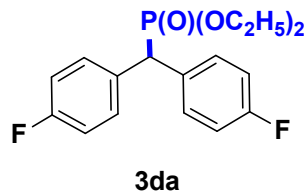


Yellowish liquid. ^1H NMR (400 MHz, CDCl_3) δ 7.42 (d, $J = 7.0$ Hz, 4H), 6.84 (d, $J = 8.6$ Hz, 4H), 4.33 (d, $J = 25.3$ Hz, 1H), 4.04 – 3.90 (m, 2H), 3.90 – 3.79 (m, 2H), 3.76 (s, 6H), 1.13 (t, $J = 7.1$ Hz, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 158.51 (d, $J = 2.0$ Hz), 130.30 (d, $J = 8.0$ Hz), 129.17 (d, $J = 5.0$ Hz), 113.86 (d, $J = 1.0$ Hz), 62.47 (d, $J = 7.1$ Hz), 55.11, 49.33 (d, $J = 138.8$ Hz), 16.21 (d, $J = 5.8$ Hz).

^{31}P NMR (162 MHz, CDCl_3) δ 25.63.

HRMS (ESI) Calcd. for $\text{C}_{19}\text{H}_{25}\text{O}_3\text{P}$ $[\text{M}+\text{Na}]^+$: 387.1332, found 387.1330.



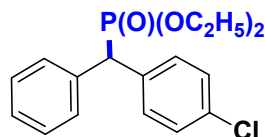
Yellowish liquid. ^1H NMR (400 MHz, CDCl_3) δ 7.54 – 7.41 (m, 4H), 7.01 (t, $J = 8.6$ Hz, 4H), 4.40 (d, $J = 25.3$ Hz, 1H), 4.07 – 3.92 (m, 2H), 3.92 – 3.78 (m, 2H), 1.13 (t, $J = 7.1$ Hz, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 161.94 (dd, $J_{\text{C-P}} = 2.3$ Hz, $J_{\text{C-F}} = 246.3$ Hz), 132.48

(dd, $J_{C-P} = 3.9$ Hz, $J_{C-F} = 4.6$ Hz), 130.88 (t, $J = 8.0$ Hz), 115.46 (dd, $J_{C-P} = 1.0$ Hz, $J_{C-F} = 21.4$ Hz), 62.68 (d, $J = 7.1$ Hz), 49.45 (d, $J = 139.6$ Hz), 16.17 (d, $J = 5.8$ Hz).

^{31}P NMR (162 MHz, CDCl_3) δ 24.50.

HRMS (ESI) Calcd. for $\text{C}_{17}\text{H}_{19}\text{F}_2\text{O}_3\text{P}$ [$\text{M} + \text{Na}$] $^+$: 363.0932, found 363.0932.



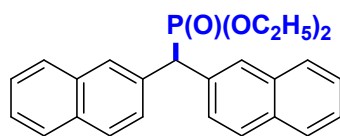
3ea

Yellowish liquid. ^1H NMR (400 MHz, CDCl_3) δ 7.55 – 7.40 (m, 4H), 7.36 – 7.19 (m, 5H), 4.40 (d, $J = 25.1$ Hz, 1H), 4.07 – 3.92 (m, 2H), 3.92 – 3.75 (m, 2H), 1.19 – 1.03 (m, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 136.31 (d, $J = 5.3$ Hz), 135.44 (d, $J = 5.2$ Hz), 133.00 (d, $J = 2.6$ Hz), 130.69 (d, $J = 8.0$ Hz), 129.29 (d, $J = 7.9$ Hz), 128.61, 127.25 (d, $J = 2.1$ Hz), 62.75 (d, $J = 7.1$ Hz), 62.55 (d, $J = 7.1$ Hz), 50.49 (d, $J = 138.8$ Hz), 16.16.

^{31}P NMR (162 MHz, CDCl_3) δ 24.47.

HRMS (ESI) Calcd. for $\text{C}_{17}\text{H}_{20}\text{ClO}_3\text{P}$ [$\text{M} + \text{Na}$] $^+$: 361.0731, found 361.0733.



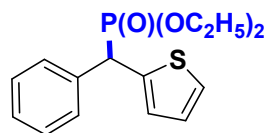
3fa

Brown solid. mp 107-109 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.08 (s, 2H), 7.88 – 7.62 (m, 8H), 7.48 – 7.33 (m, 4H), 4.78 (d, $J = 25.0$ Hz, 1H), 4.11 – 3.94 (m, 2H), 3.94 – 3.77 (m, 2H), 1.10 (t, $J = 7.1$ Hz, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 134.21 (d, $J = 5.2$ Hz), 133.29 (d, $J = 1.3$ Hz), 132.33 (d, $J = 1.3$ Hz), 128.24 (d, $J = 8.8$ Hz), 128.16, 127.91, 127.55 (d, $J = 7.7$ Hz), 127.45, 126.03, 125.86, 62.68 (d, $J = 7.0$ Hz), 51.24 (d, $J = 138.7$ Hz), 16.20 (d, $J = 5.8$ Hz).

^{31}P NMR (162 MHz, CDCl_3) δ 24.86.

HRMS (ESI) Calcd. for $\text{C}_{25}\text{H}_{25}\text{O}_3\text{P}$ [$\text{M} + \text{Na}$] $^+$: 427.1434, found 427.1430.



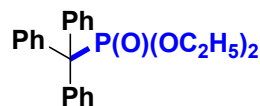
3ga

Brown liquid. ^1H NMR (400 MHz, CDCl_3) δ 7.52 (d, $J = 7.5$ Hz, 2H), 7.33 (t, $J = 7.6$ Hz, 2H), 7.30 – 7.22 (m, 2H), 7.19 (d, $J = 5.2$ Hz, 1H), 7.01 – 6.92 (m, 1H), 4.66 (d, $J = 25.6$ Hz, 1H), 4.11 – 3.86 (m, 3H), 3.84 – 3.70 (m, 1H), 1.19 (t, $J = 7.1$ Hz, 3H), 1.10 (t, $J = 7.1$ Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 138.71 (d, *J* = 5.3 Hz), 136.33 (d, *J* = 5.6 Hz), 129.28 (d, *J* = 7.0 Hz), 128.57 (d, *J* = 1.8 Hz), 127.48 (d, *J* = 2.5 Hz), 127.04 (d, *J* = 7.4 Hz), 126.89 (d, *J* = 2.4 Hz), 124.88 (d, *J* = 2.6 Hz), 63.05 (d, *J* = 7.0 Hz), 62.89 (d, *J* = 7.2 Hz), 46.32 (d, *J* = 140.2 Hz), 16.26 (d, *J* = 5.9 Hz), 16.18 (d, *J* = 5.8 Hz).

³¹P NMR (162 MHz, CDCl₃) δ 23.14.

HRMS (ESI) Calcd. for C₁₅H₁₉O₃PS [M+Na]⁺: 333.0685, found 333.0681.



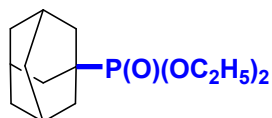
3ha

Yellowish solid. mp 104-106 °C. **¹H NMR (400 MHz, CDCl₃)** δ 7.39 – 7.19 (m, 15H), 4.10 – 3.92 (m, 2H), 3.92 – 3.75 (m, 2H), 1.09 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 141.55 (d, *J* = 5.5 Hz), 130.66 (d, *J* = 6.3 Hz), 127.77 (d, *J* = 1.0 Hz), 126.84 (d, *J* = 1.7 Hz), 63.26 (d, *J* = 8.2 Hz), 62.91 (d, *J* = 136.2 Hz), 16.11 (d, *J* = 5.7 Hz).

³¹P NMR (121 MHz, CDCl₃) δ 27.06.

HRMS (ESI) Calcd. for C₂₃H₂₅O₃P [M+Na]⁺: 403.1434, found 403.1430.



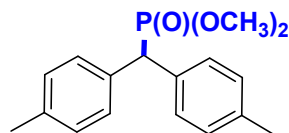
3ia

Yellowish liquid. **¹H NMR (400 MHz, CDCl₃)** δ 4.16 – 4.04 (m, 4H), 1.98 (s, 3H), 1.94 – 1.84 (m, 6H), 1.79 – 1.68 (m, 6H), 1.31 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 61.57 (d, *J* = 7.3 Hz), 36.50 (d, *J* = 1.7 Hz), 35.36 (d, *J* = 4.2 Hz), 34.18 (d, *J* = 147.3 Hz), 27.08 (d, *J* = 12.2 Hz), 16.52 (d, *J* = 5.7 Hz).

³¹P NMR (121 MHz, CDCl₃) δ 33.77.

HRMS (ESI) Calcd. for C₁₄H₂₅O₃P [M+Na]⁺: 295.1434, found 295.1432.



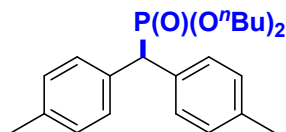
3bb

Yellowish liquid. **¹H NMR (400 MHz, CDCl₃)** δ 7.39 (d, *J* = 6.6 Hz, 4H), 7.12 (d, *J* = 7.9 Hz, 4H), 4.39 (d, *J* = 25.1 Hz, 1H), 3.56 (d, *J* = 10.7 Hz, 6H), 2.30 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 136.77 (d, *J* = 2.4 Hz), 133.62 (d, *J* = 5.2 Hz), 129.32 (d, *J* = 1.4 Hz), 129.09 (d, *J* = 8.0 Hz), 53.31 (d, *J* = 7.2 Hz), 49.89 (d, *J* = 138.3 Hz), 20.98.

^{31}P NMR (162 MHz, CDCl_3) δ 27.88.

HRMS (ESI) Calcd. for $\text{C}_{17}\text{H}_{21}\text{O}_3\text{P}$ $[\text{M}+\text{Na}]^+$: 327.1121, found 327.1123.



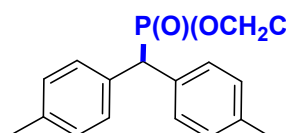
3bc

Yellowish liquid. ^1H NMR (400 MHz, CDCl_3) δ 7.39 (d, $J = 6.7$ Hz, 4H), 7.10 (d, $J = 7.9$ Hz, 4H), 4.36 (d, $J = 25.1$ Hz, 1H), 3.96 – 3.84 (m, 2H), 3.82 – 3.70 (m, 2H), 2.29 (s, 6H), 1.54 – 1.36 (m, 4H), 1.32 – 1.16 (m, 5H), 0.81 (t, $J = 7.4$ Hz, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 136.50 (d, $J = 2.3$ Hz), 134.03 (d, $J = 5.2$ Hz), 129.22, 129.14, 66.15 (d, $J = 7.3$ Hz), 50.34 (d, $J = 138.4$ Hz), 32.37 (d, $J = 5.9$ Hz), 20.93, 18.51, 13.46.

^{31}P NMR (162 MHz, CDCl_3) δ 25.41.

HRMS (ESI) Calcd. for $\text{C}_{23}\text{H}_{33}\text{O}_3\text{P}$ $[\text{M}+\text{Na}]^+$: 411.2060, found 411.2058.



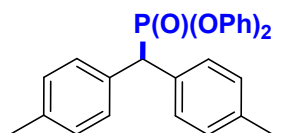
3bd

Yellowish liquid. ^1H NMR (400 MHz, CDCl_3) δ 7.38 (d, $J = 8.0$, 4H), 7.14 (d, $J = 7.9$ Hz, 4H), 4.54 (d, $J = 26.4$ Hz, 1H), 4.40 – 4.20 (m, 2H), 4.01 – 3.79 (m, 2H), 2.31 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 137.59 (d, $J = 2.5$ Hz), 131.78 (d, $J = 5.3$ Hz), 129.61 (d, $J = 1.4$ Hz), 129.06 (d, $J = 8.6$ Hz), 122.42 (qd, $J_{\text{C-F}} = 277.6$, $J_{\text{C-P}} = 7.9$ Hz), 62.51 (qd, $J_{\text{C-P}} = 38.0$, $J_{\text{C-P}} = 6.5$ Hz), 50.09 (d, $J = 139.1$ Hz), 20.95.

^{31}P NMR (162 MHz, CDCl_3) δ 28.48.

HRMS (ESI) Calcd. for $\text{C}_{19}\text{H}_{19}\text{F}_6\text{O}_3\text{P}$ $[\text{M}+\text{Na}]^+$: 463.0868, found 463.0870.



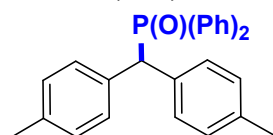
3be

White solid. mp 169-171 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.47 (d, $J = 6.5$ Hz, 4H), 7.27 – 6.99 (m, 10H), 6.85 (d, $J = 8.4$ Hz, 4H), 4.73 (d, $J = 25.5$ Hz, 1H), 2.31 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 150.49 (d, $J = 9.9$ Hz), 137.09 (d, $J = 2.4$ Hz), 132.85 (d, $J = 5.3$ Hz), 129.44, 129.42 (d, $J = 1.8$ Hz), 129.36, 124.85, 120.58 (d, $J = 4.2$ Hz), 50.45 (d, $J = 139.5$ Hz), 21.00.

^{31}P NMR (162 MHz, CDCl_3) δ 18.40.

HRMS (ESI) Calcd. for $C_{27}H_{25}O_3P$ $[M+Na]^+$: 451.1434, found 451.1487.



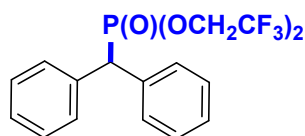
3bf

White solid. mp 258-260 °C. 1H NMR (400 MHz, $CDCl_3$) δ 7.70 – 7.53 (m, 4H), 7.45 – 7.27 (m, 10H), 6.97 (d, J = 8.0 Hz, 4H), 4.69 (d, J = 9.0 Hz, 1H), 2.21 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 136.35 (d, J = 1.7 Hz), 134.28 (d, J = 4.6 Hz), 132.73 (d, J = 97.7 Hz), 131.31, 131.23, 129.52 (d, J = 6.8 Hz), 129.14, 128.15 (d, J = 11.5 Hz), 52.51 (d, J = 66.6 Hz), 20.96.

^{31}P NMR (162 MHz, $CDCl_3$) δ 31.00.

HRMS (ESI) Calcd. for $C_{27}H_{25}OP$ $[M+Na]^+$: 419.1535, found 419.1533.



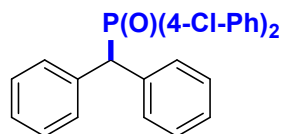
3bg

Yellowish solid. mp 88-90 °C. 1H NMR (400 MHz, $CDCl_3$) δ 7.51 (d, J = 7.3 Hz, 4H), 7.39 – 7.26 (m, 6H), 4.62 (d, J = 26.4 Hz, 1H), 4.37 – 4.19 (m, 2H), 3.99 – 3.82 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 134.60 (d, J = 5.4 Hz), 128.96 (d, J = 1.4 Hz), 127.91 (d, J = 2.3 Hz), 122.35 (qd, J_{C-F} = 277.5, J_{C-P} = 7.9 Hz), 62.53 (qd, J_{C-F} = 38.0, J_{C-P} = 6.5 Hz), 50.89 (d, J = 139.3 Hz).

^{31}P NMR (162 MHz, $CDCl_3$) δ 28.06.

HRMS (ESI) Calcd. for $C_{17}H_{15}F_6O_3P$ $[M+Na]^+$: 435.0555, found 435.0559.



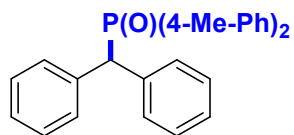
3bh

White solid. mp 248-250 °C. 1H NMR (400 MHz, $CDCl_3$) δ 7.57 – 7.45 (m, 8H), 7.30 (dd, J = 8.6, 2.4 Hz, 4H), 7.24 – 7.12 (m, 6H), 4.70 (d, J = 8.9 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 138.19 (d, J = 3.4 Hz), 136.45 (d, J = 4.7 Hz), 132.55 (d, J = 9.5 Hz), 130.52 (d, J = 99.4 Hz), 129.63 (d, J = 6.8 Hz), 128.73, 128.62, 127.21 (d, J = 1.8 Hz), 53.34 (d, J = 67.1 Hz).

^{31}P NMR (162 MHz, $CDCl_3$) δ 30.25.

HRMS (ESI) Calcd. for $C_{25}H_{19}ClOP$ $[M+Na]^+$: 459.0443, found 459.0440.



3bi

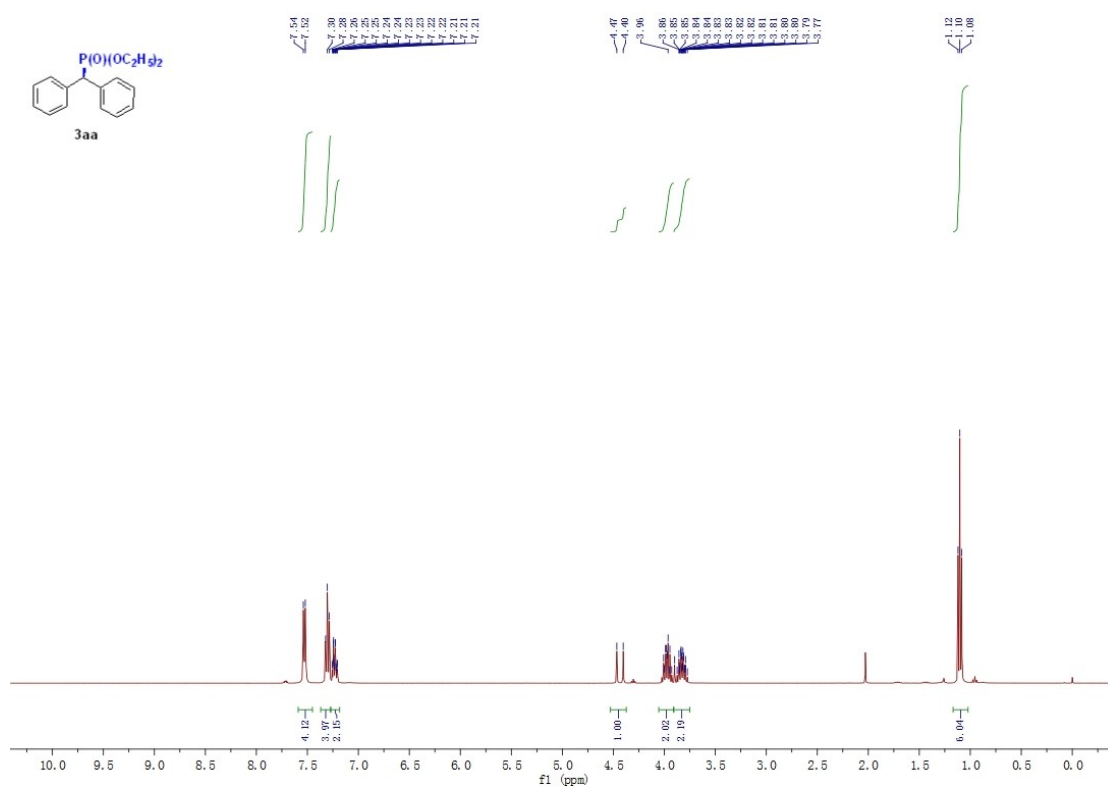
White solid. mp 264-266 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.57 – 7.50 (m, 8H), 7.26 – 7.14 (m, 10H), 4.76 (d, $J = 9.0$ Hz, 1H), 2.35 (s, 6H).

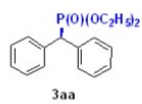
^{13}C NMR (101 MHz, CDCl_3) δ 141.60 (d, $J = 2.7$ Hz), 137.43 (d, $J = 4.7$ Hz), 131.24 (d, $J = 9.0$ Hz), 129.79 (d, $J = 6.7$ Hz), 128.96, 128.85, 128.39, 126.77 (d, $J = 1.7$ Hz), 53.56 (d, $J = 66.1$ Hz), 21.47.

^{31}P NMR (162 MHz, CDCl_3) δ 31.49.

HRMS (ESI) Calcd. for $\text{C}_{27}\text{H}_{25}\text{OP}$ $[\text{M}+\text{Na}]^+$: 419.1535, found 419.1537.

^1H , ^{13}C and ^{31}P NMR and spectra of Products

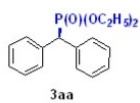
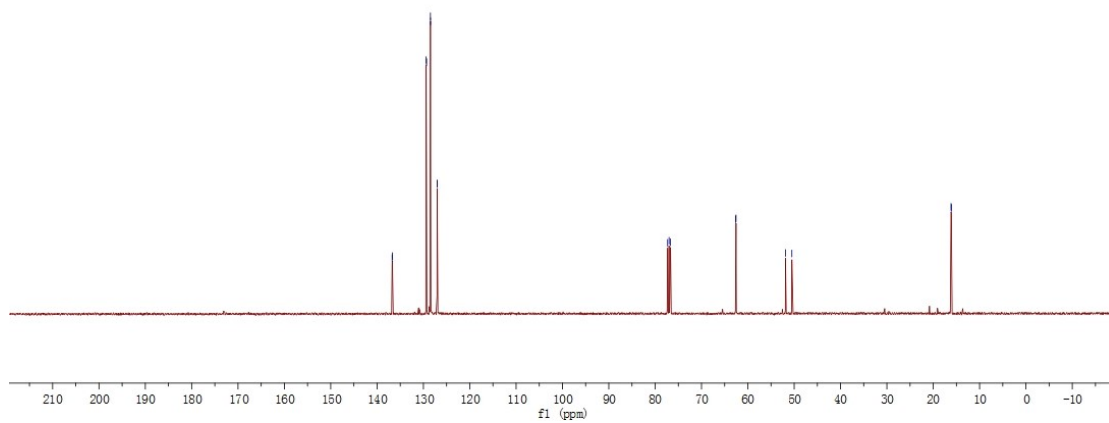




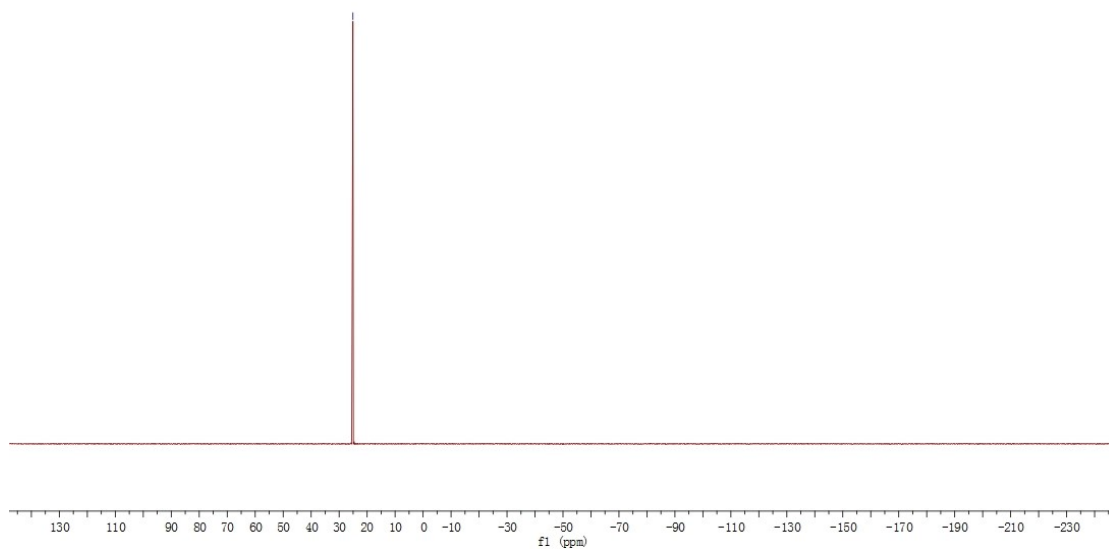
136.75
 136.09
 129.40
 129.32
 128.47
 127.94
 127.02

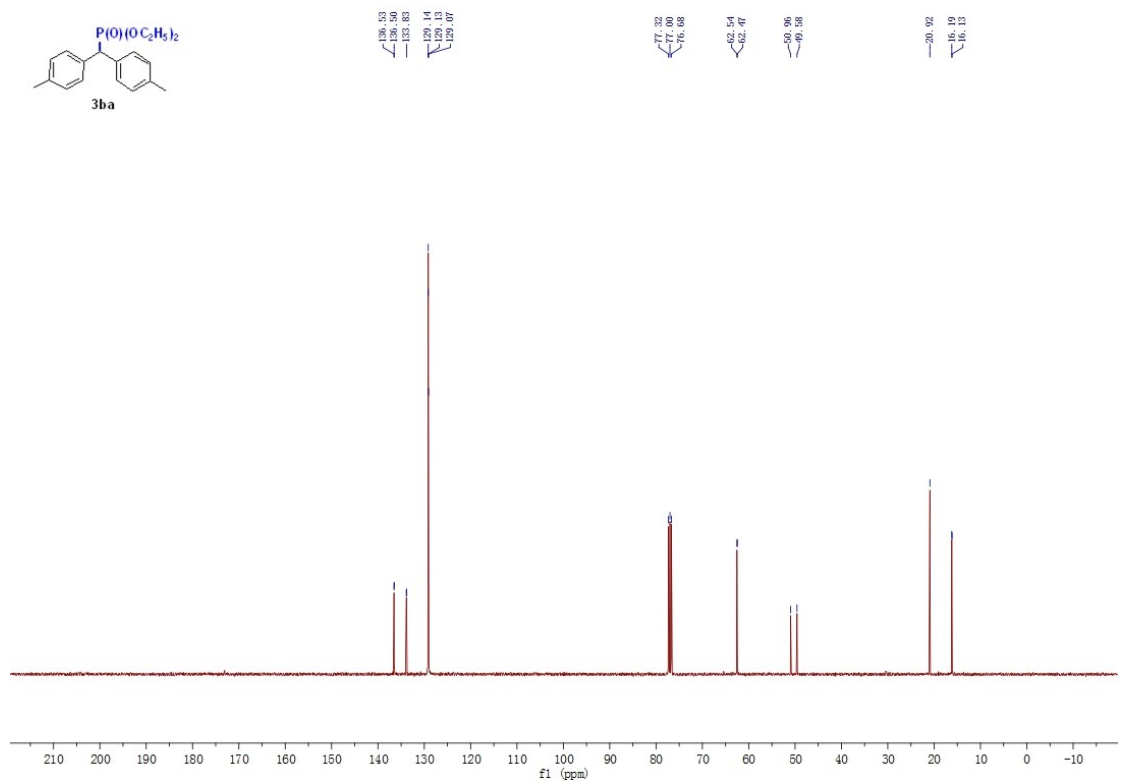
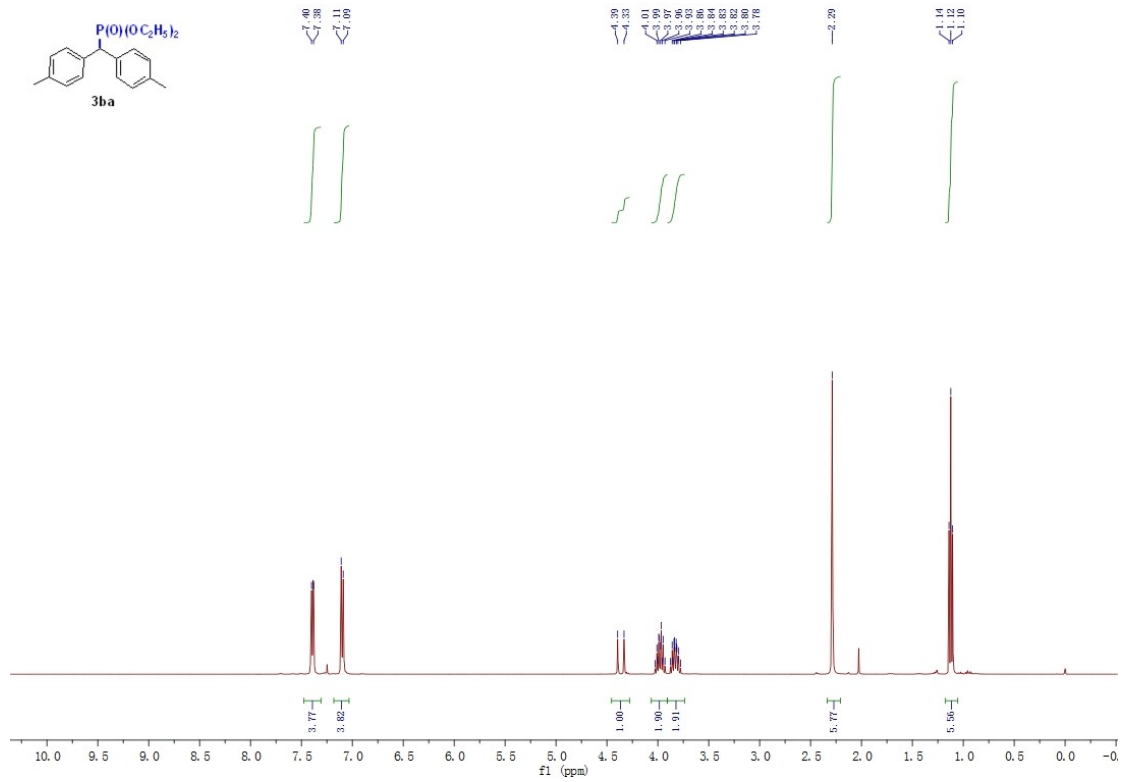
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 51.88
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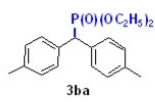
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16.10



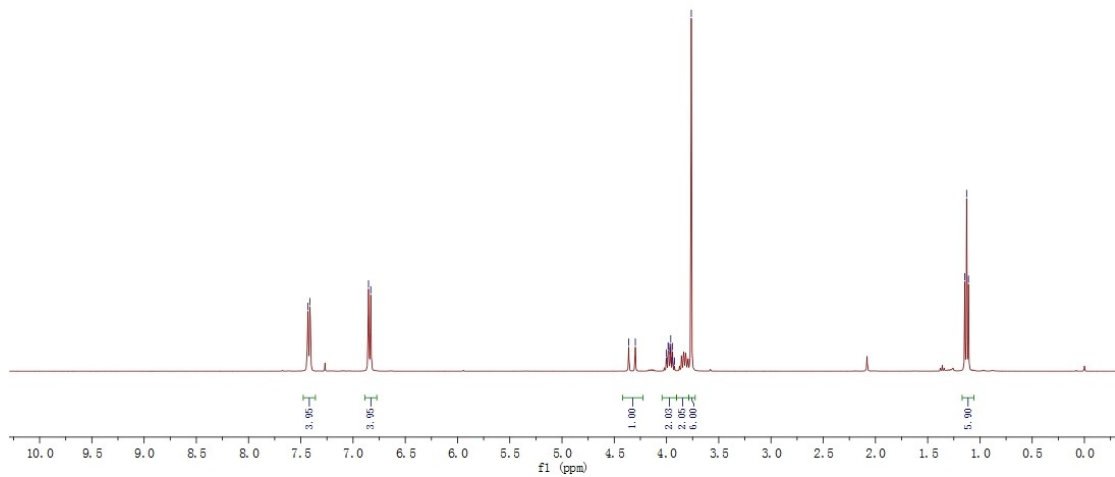
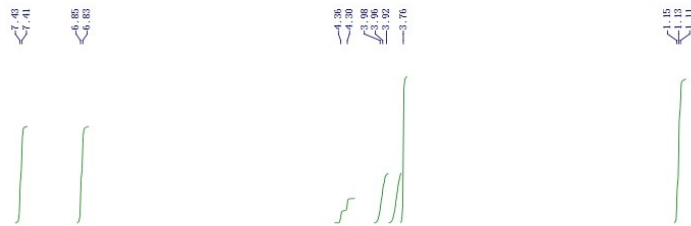
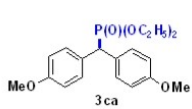
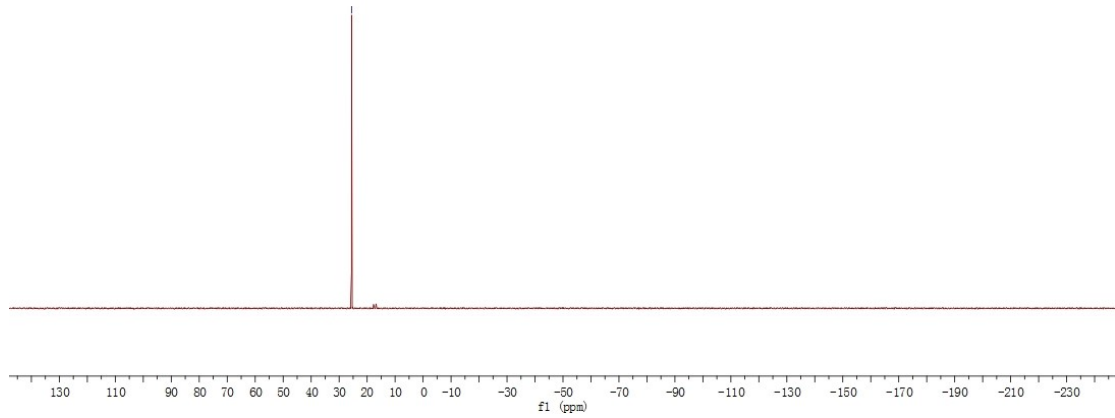
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—25.57





158.52
158.50

130.24
130.26

113.86
113.85

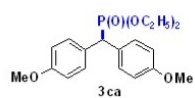
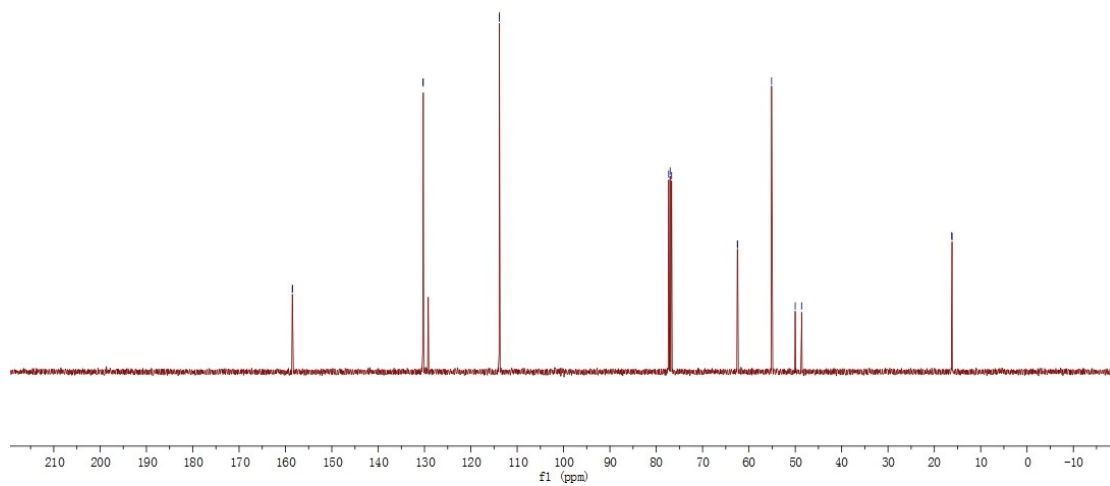
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62.50
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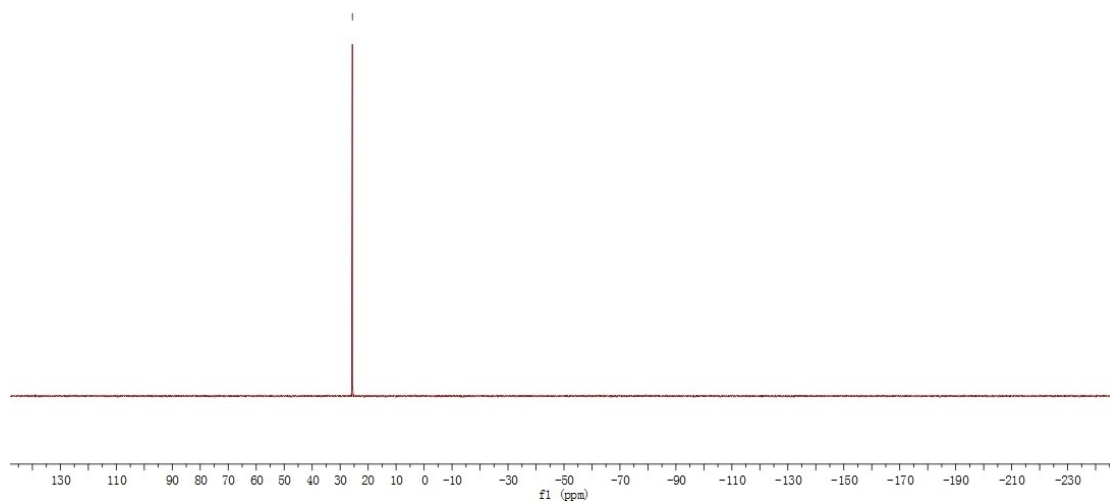
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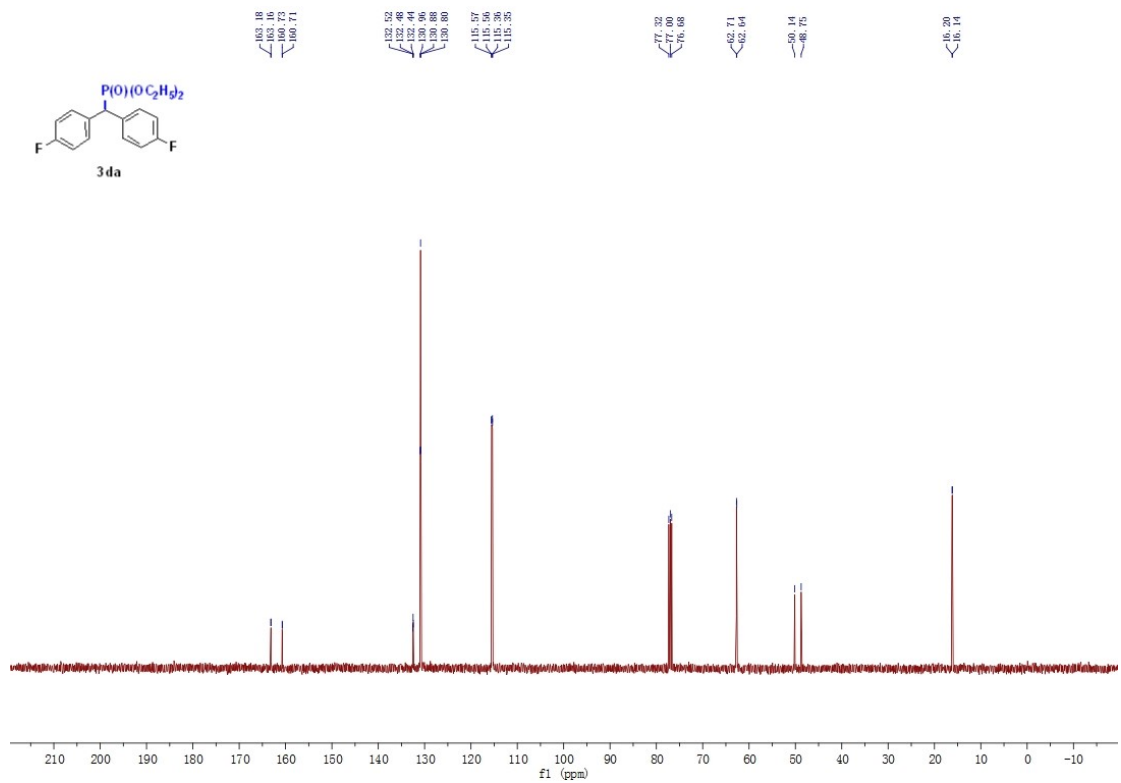
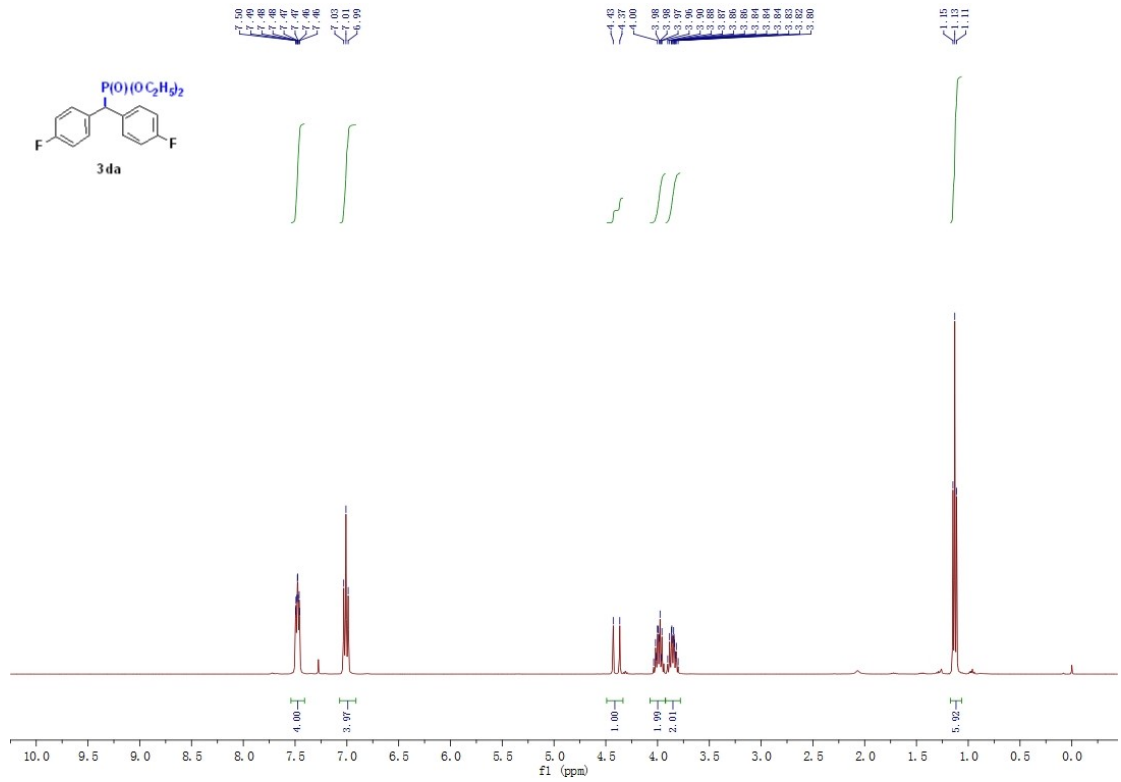
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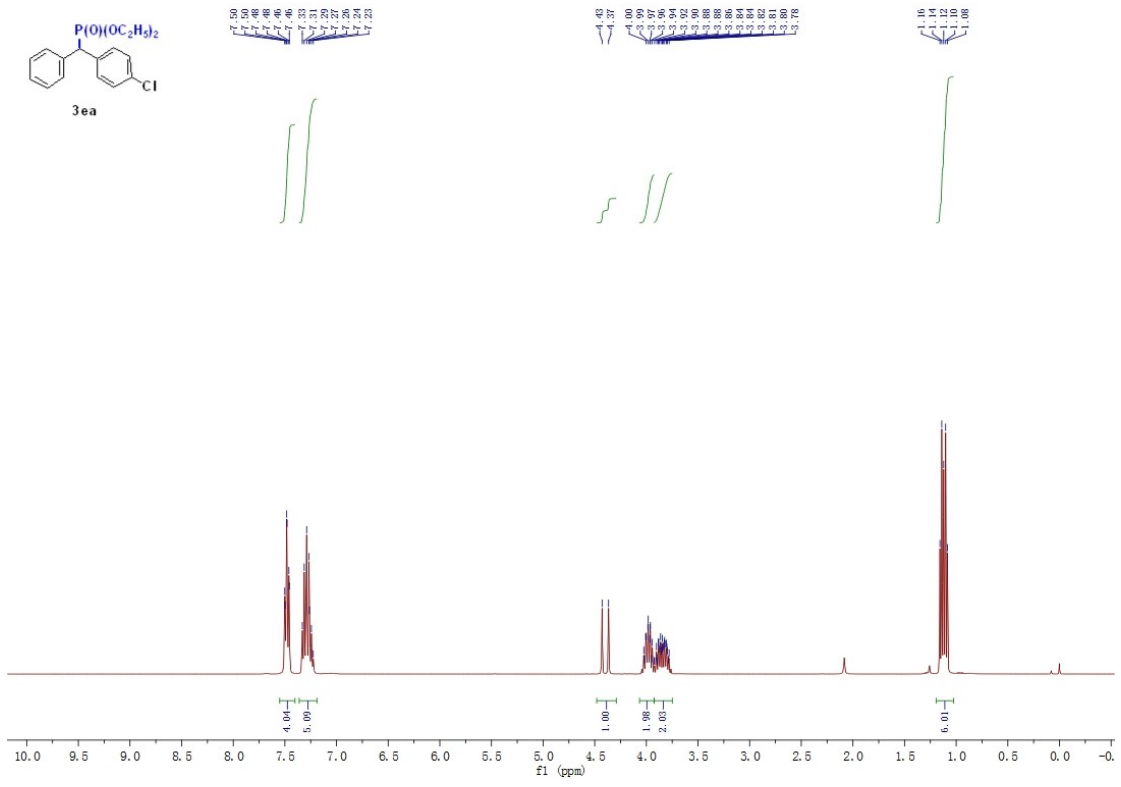
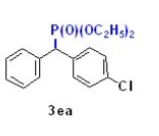
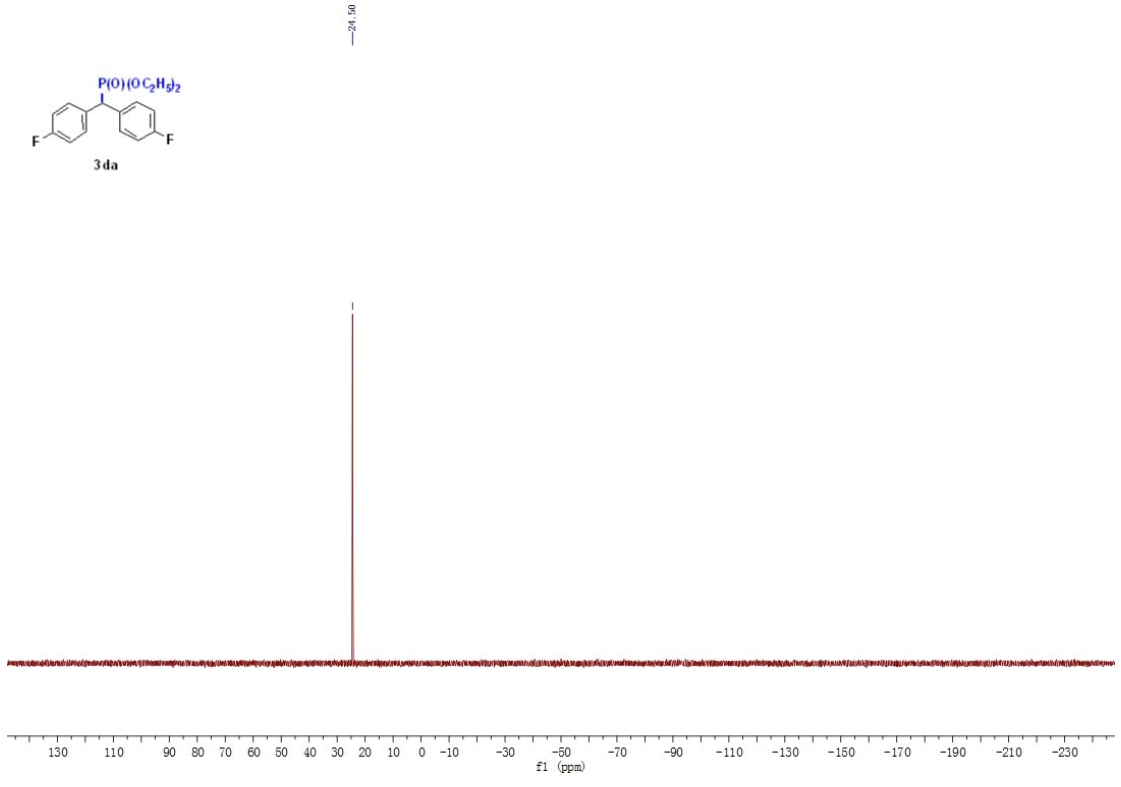
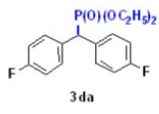
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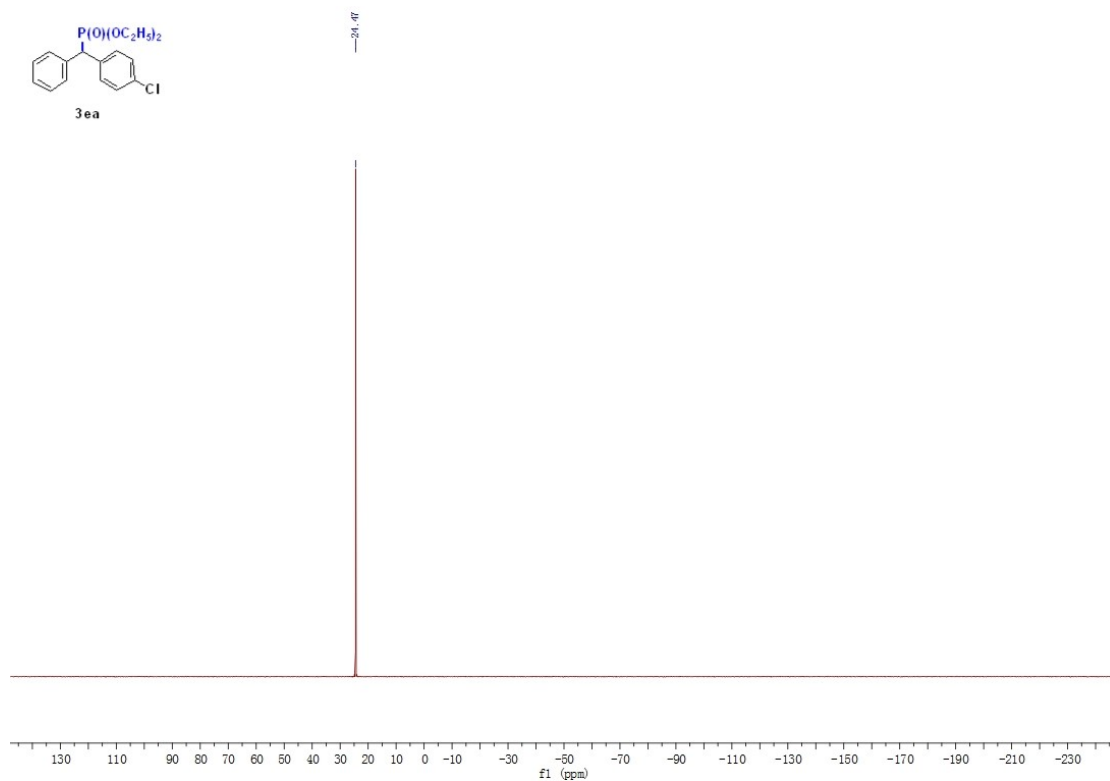
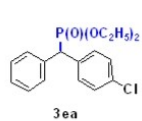
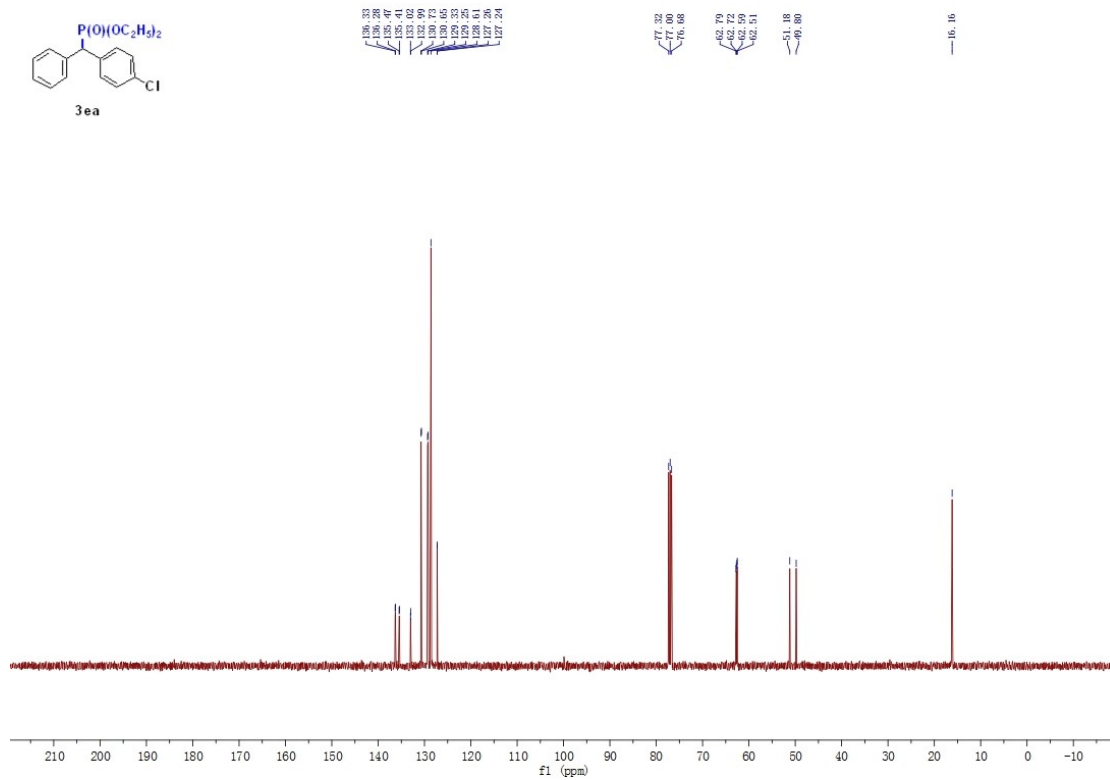
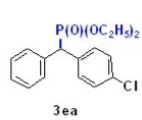


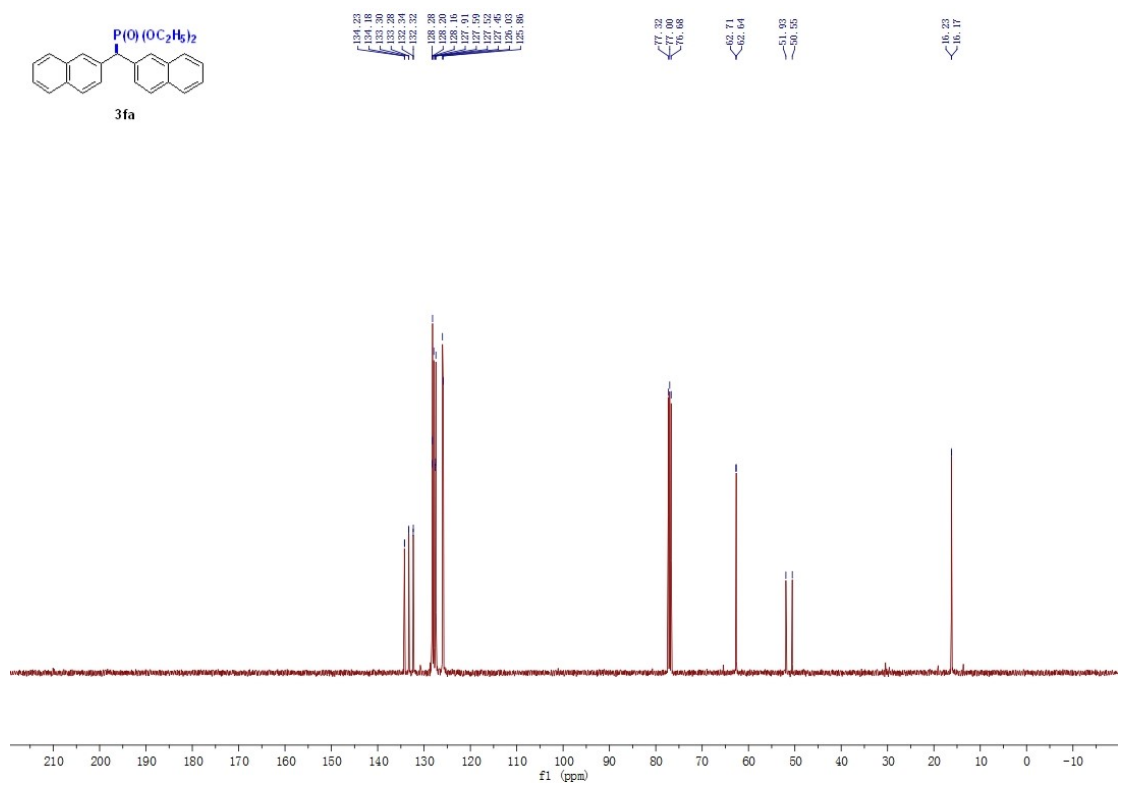
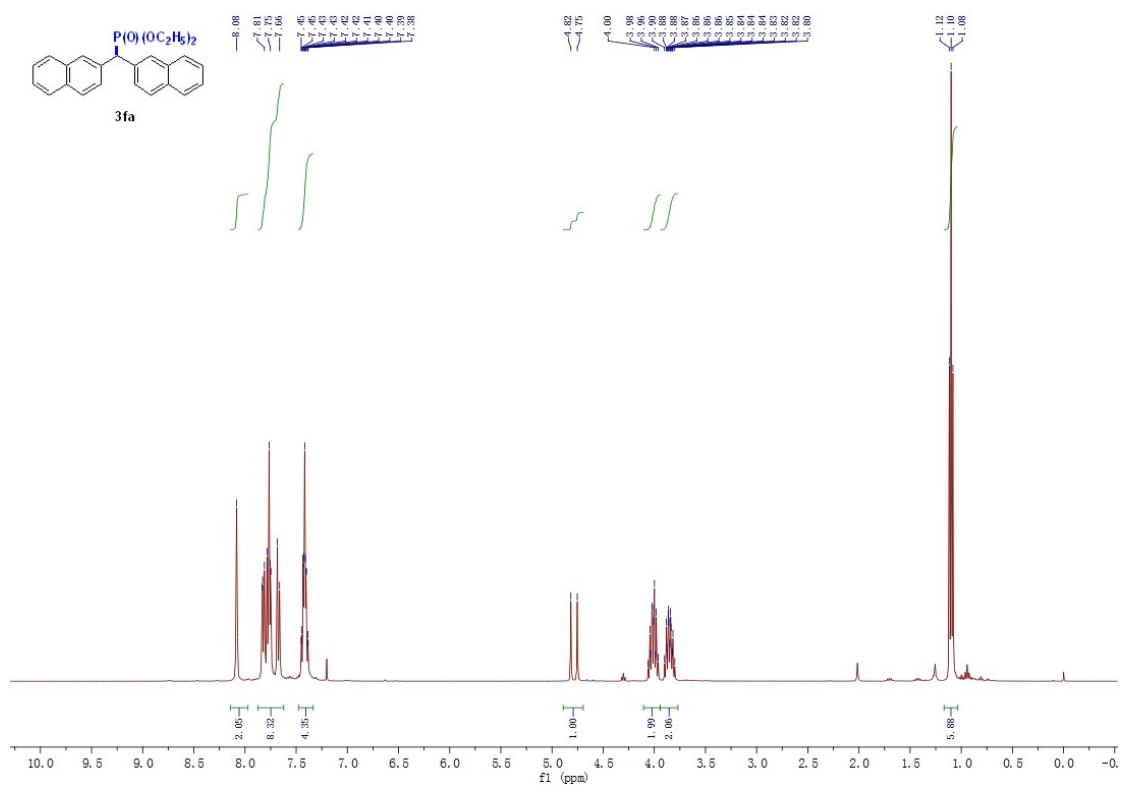
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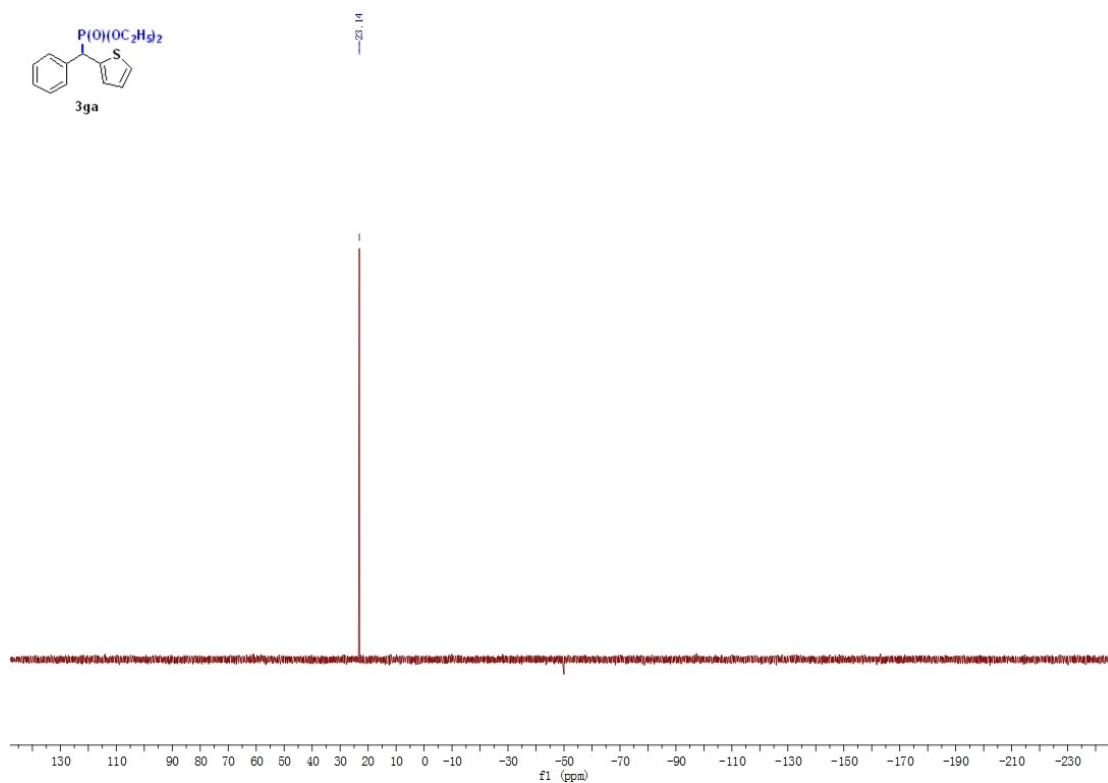
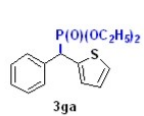
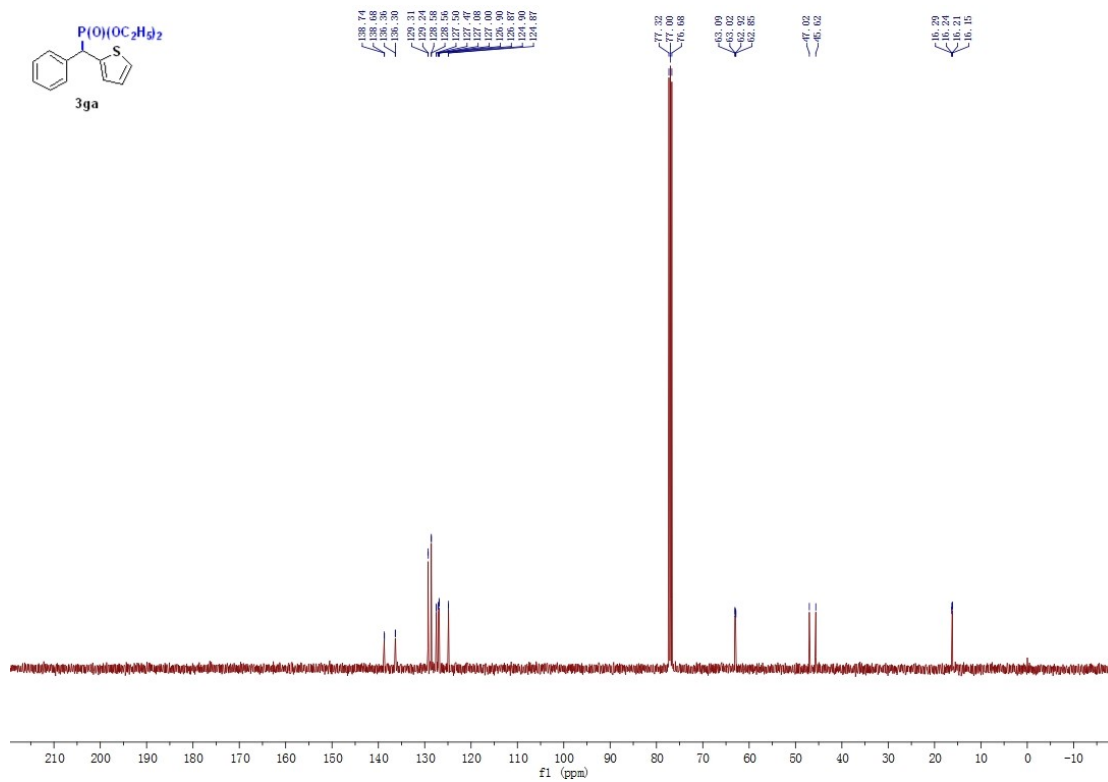
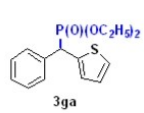


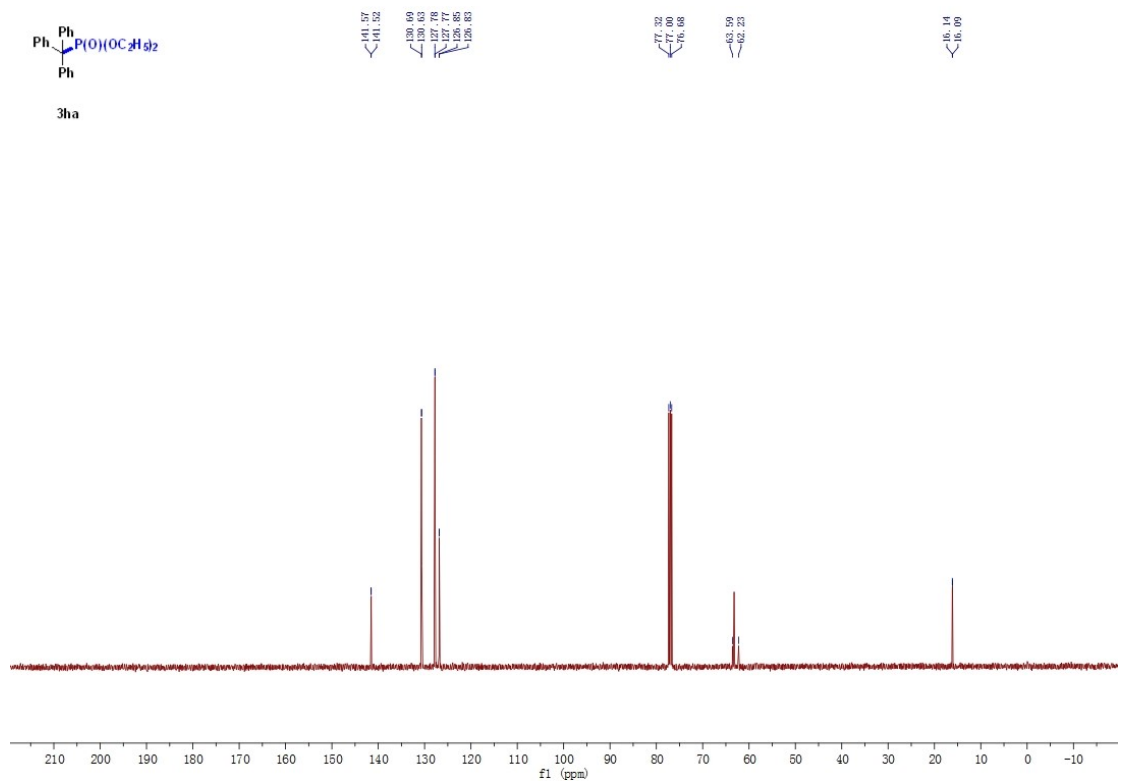
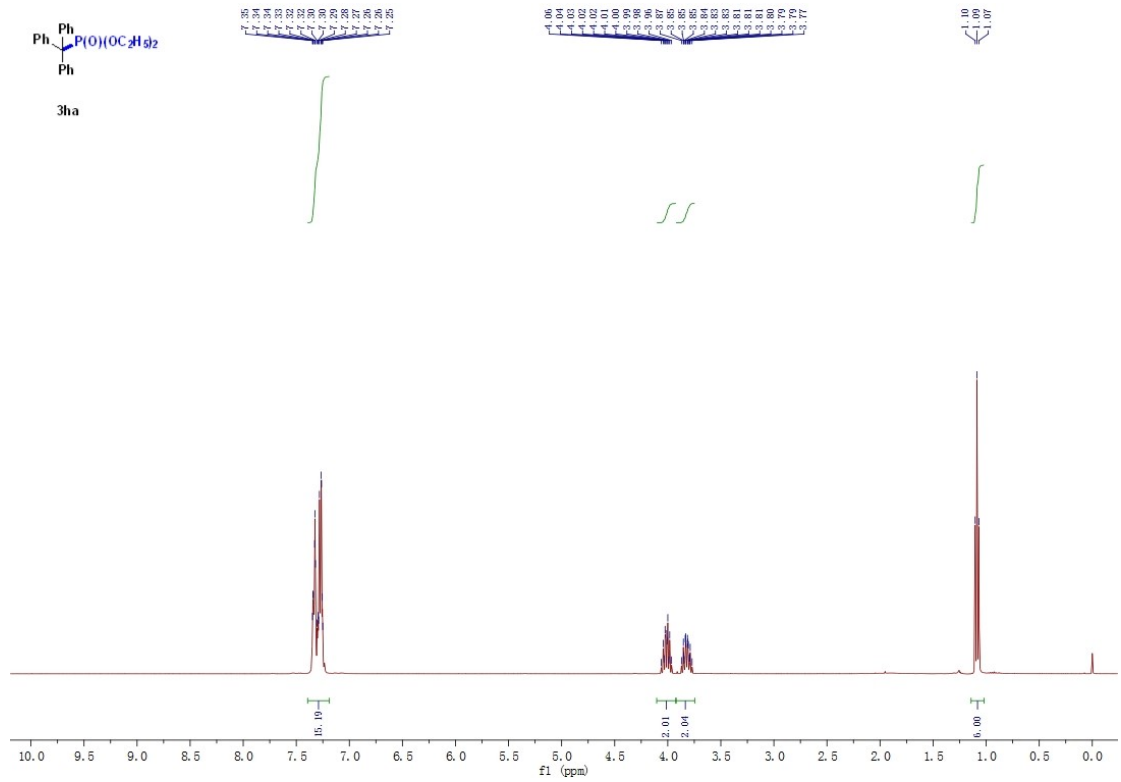


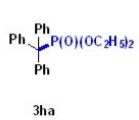




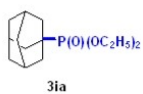
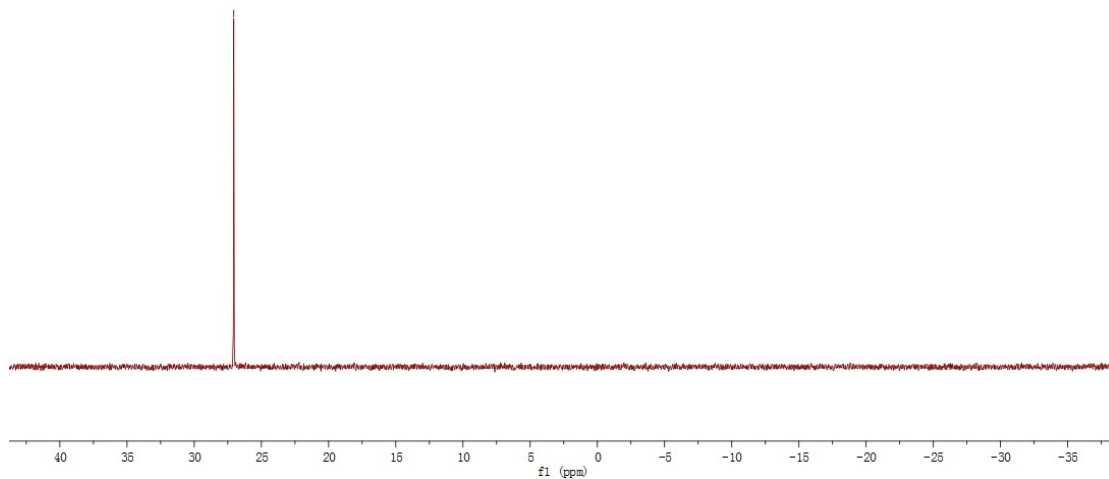






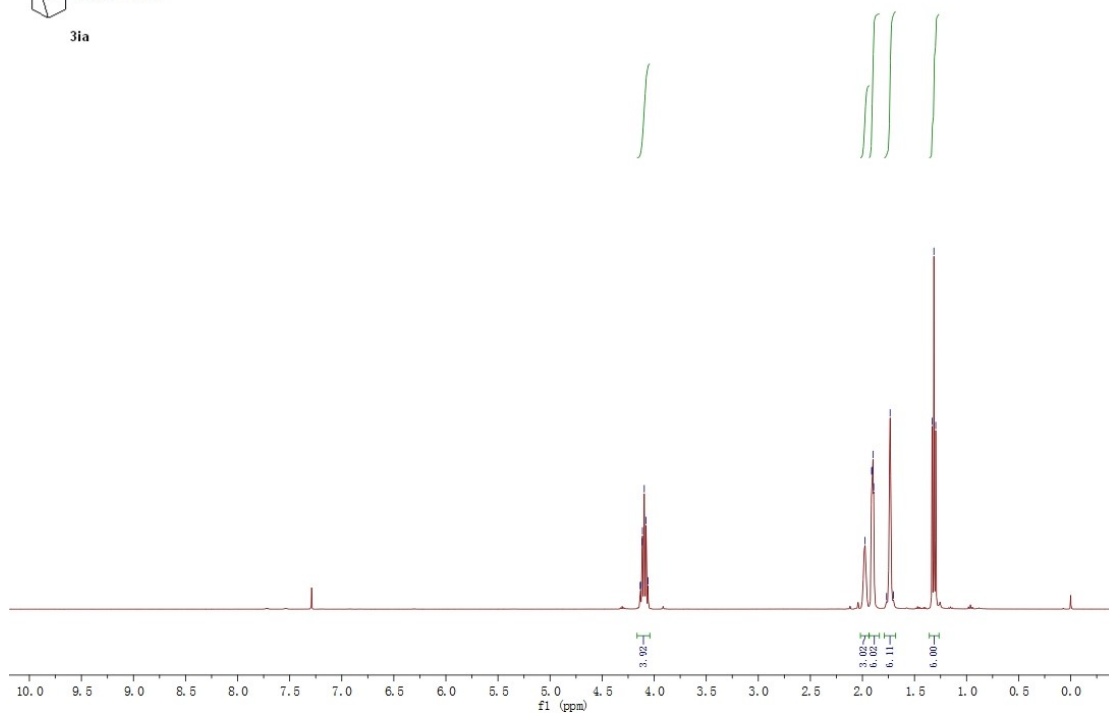


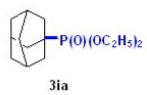
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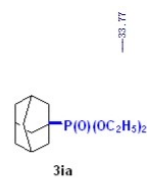
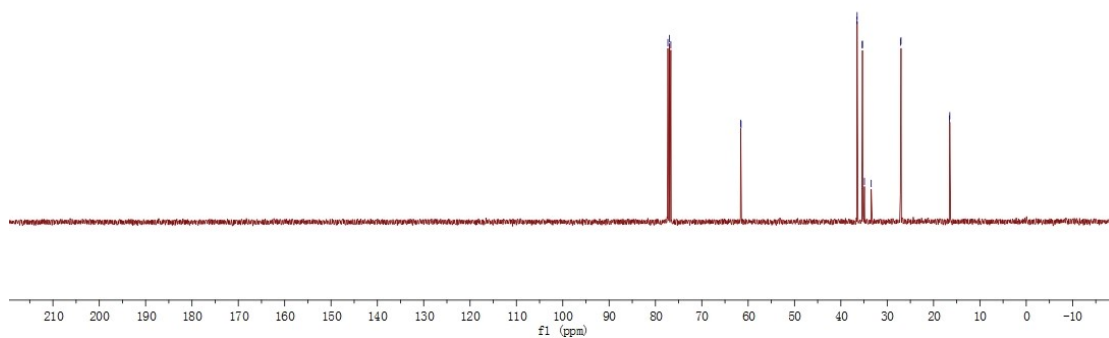
13.32, 12.12, 11.11, 10.08, 10.06

1.88, 1.91, 1.90, 1.89, 1.88, 1.72, 1.68, 1.33, 1.30

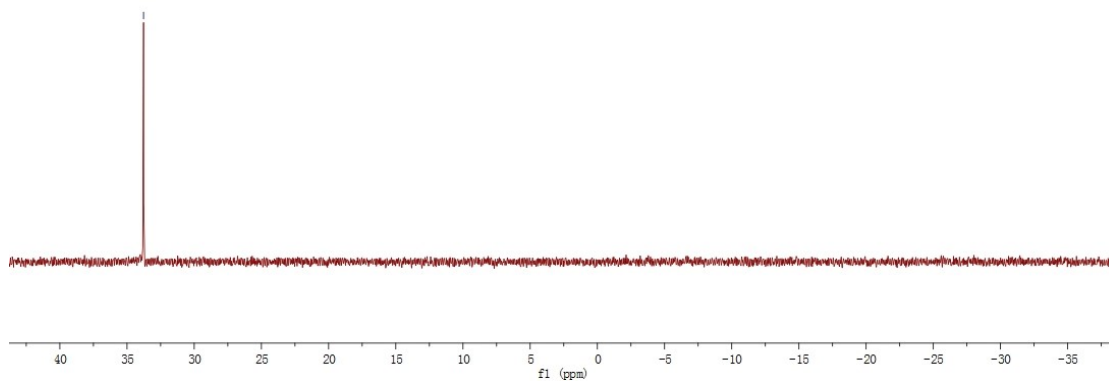


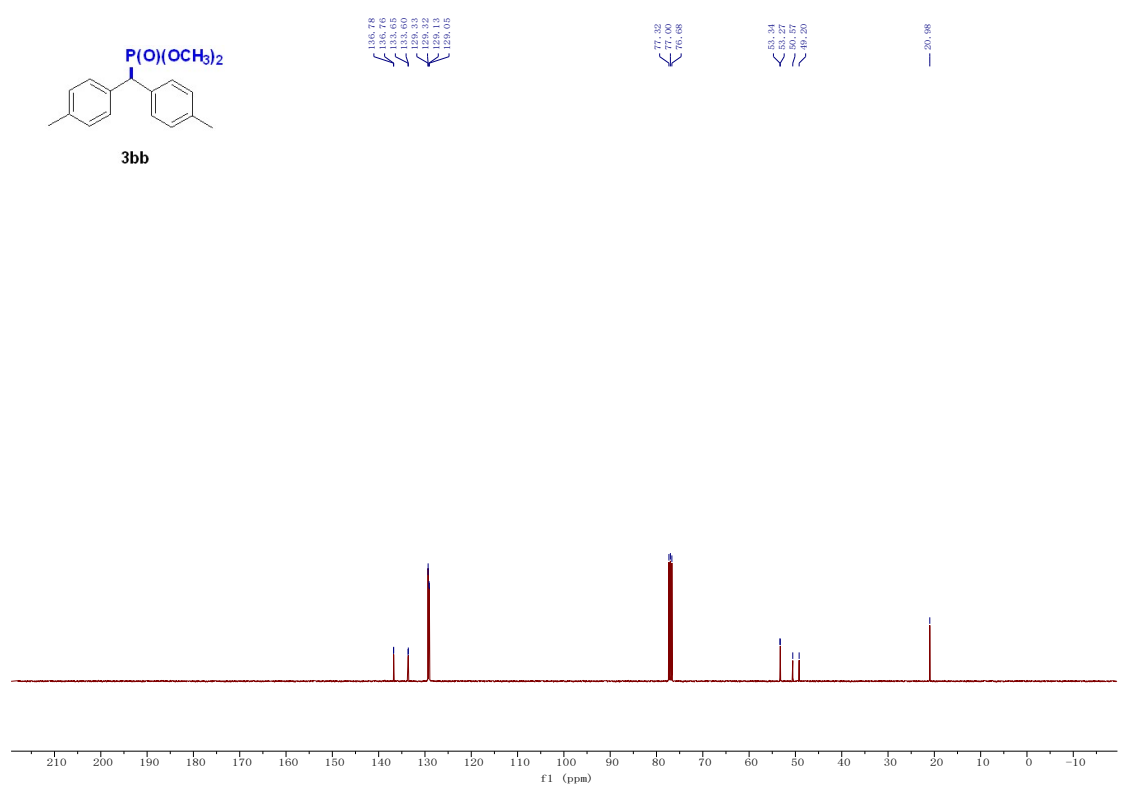
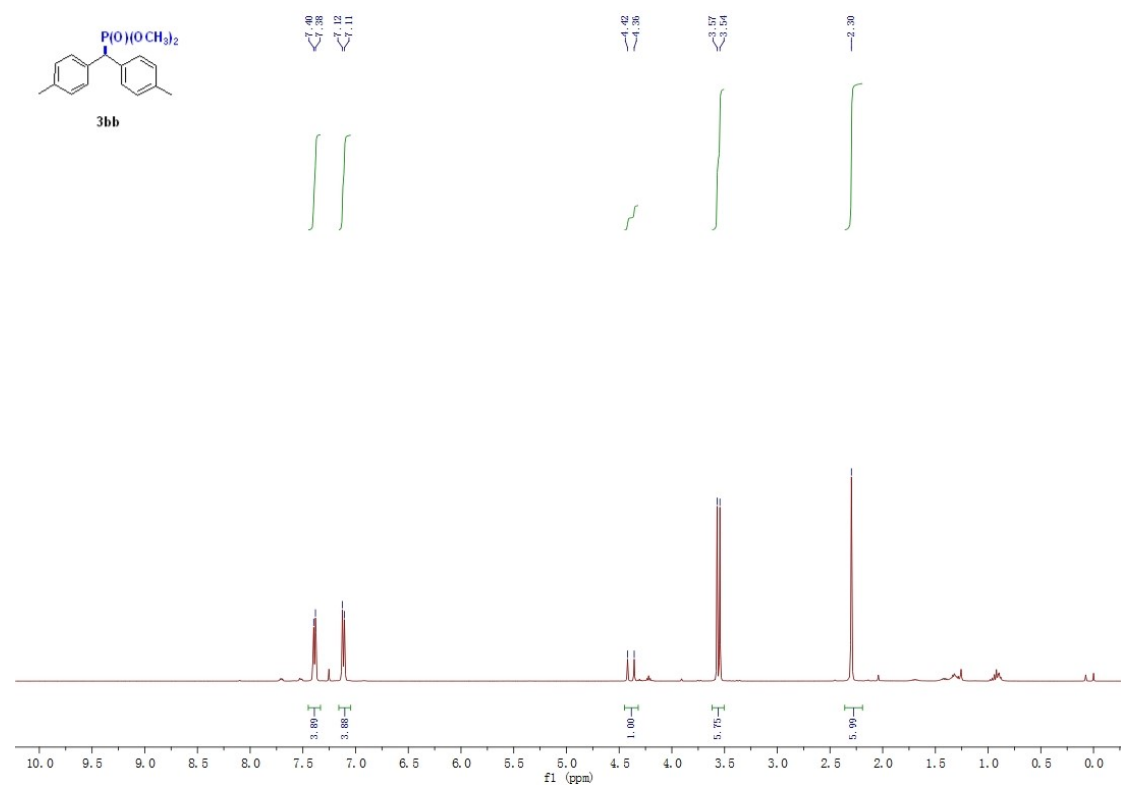


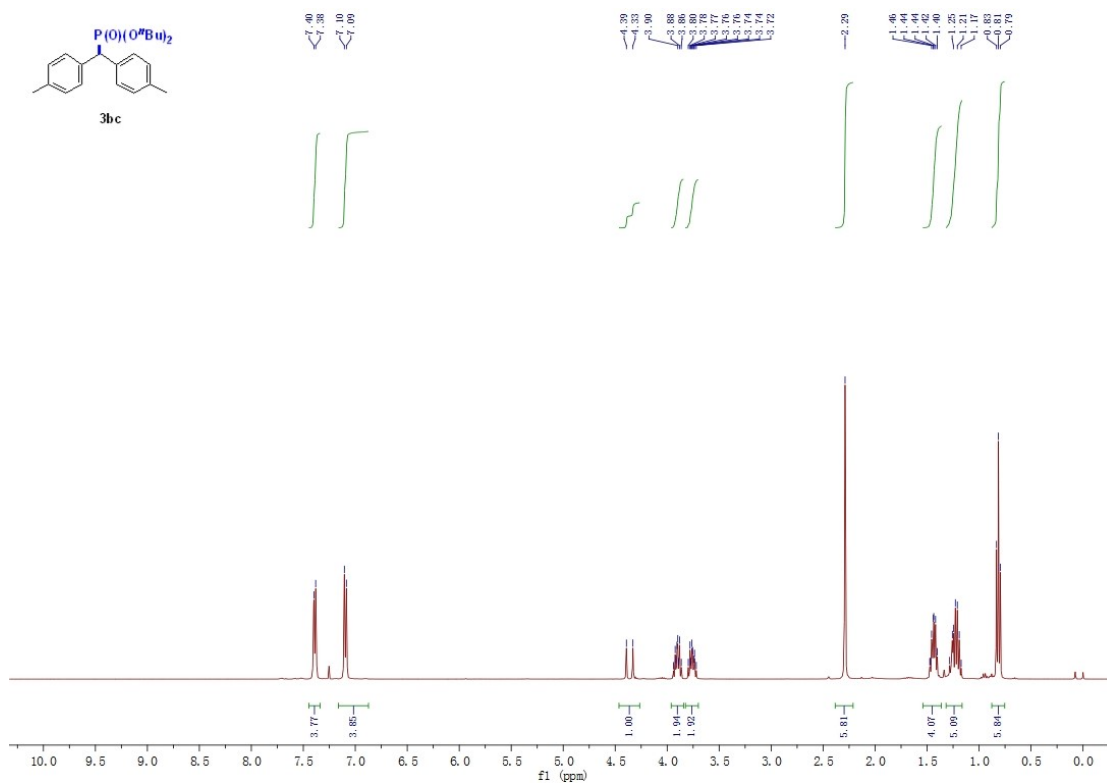
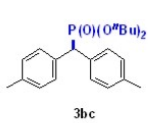
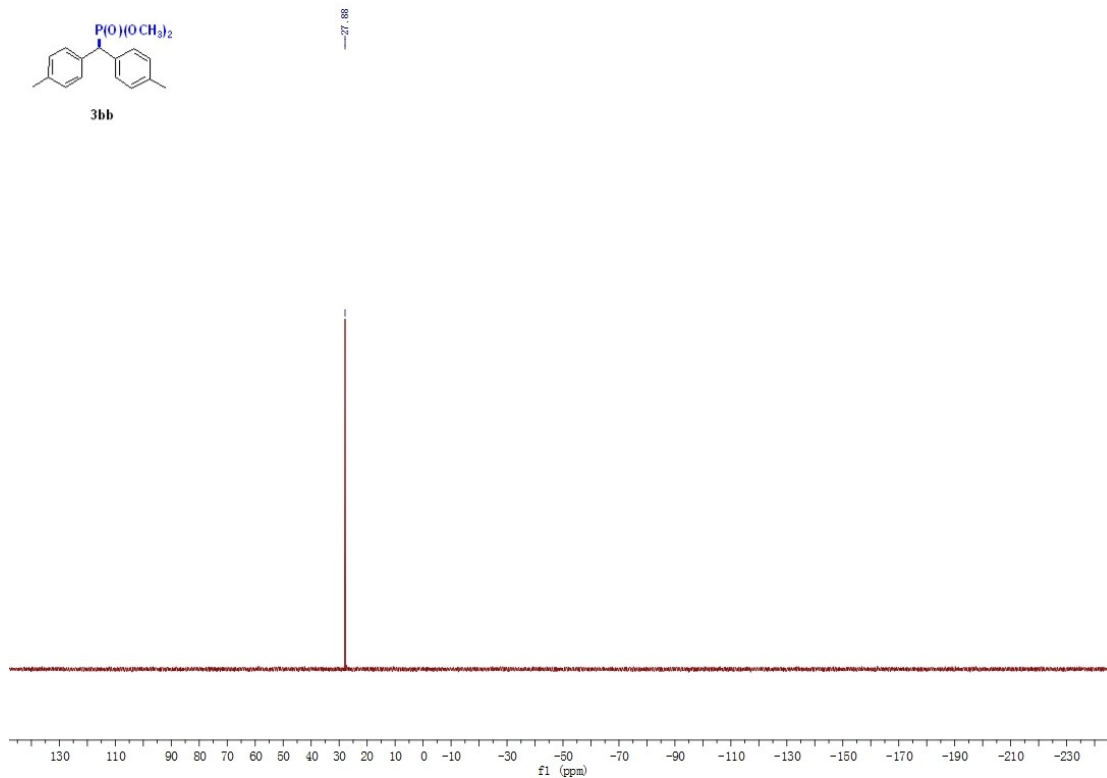
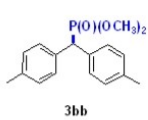
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36.49
35.38
34.92
33.46
32.82
16.55
16.49

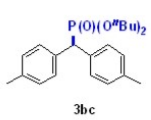


33.77









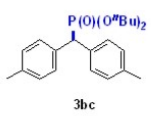
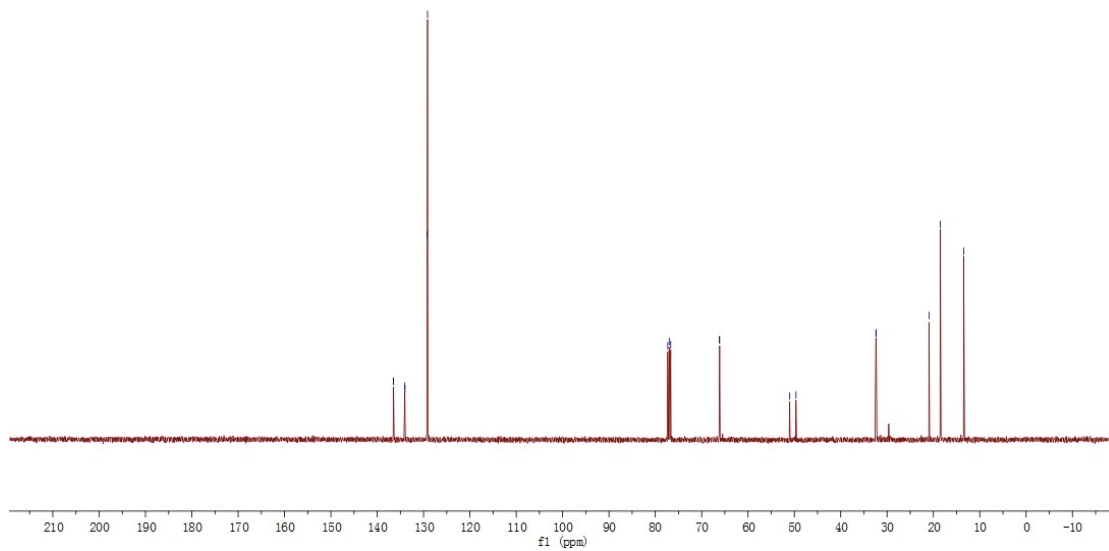
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 133.22
 133.14

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 77.00
 76.68
 66.19
 66.11

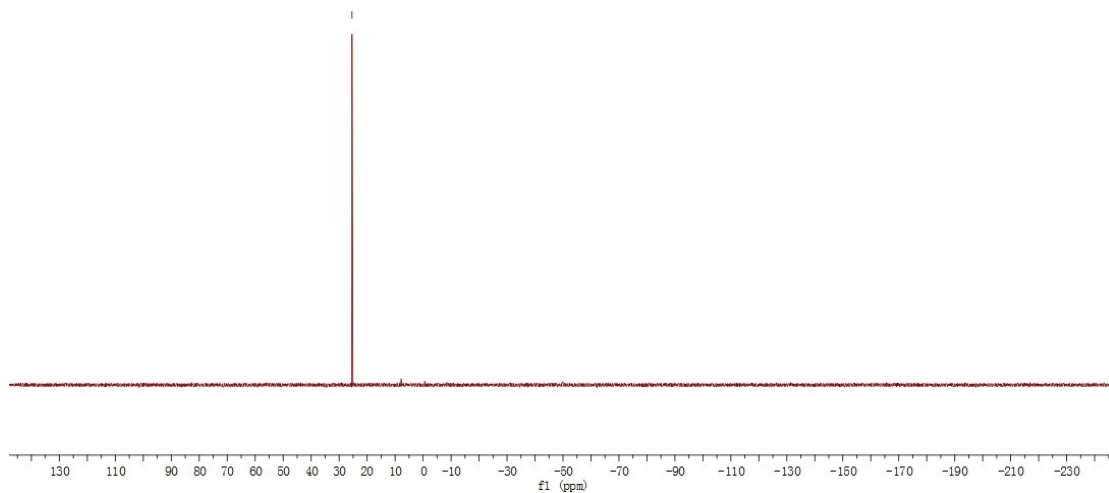
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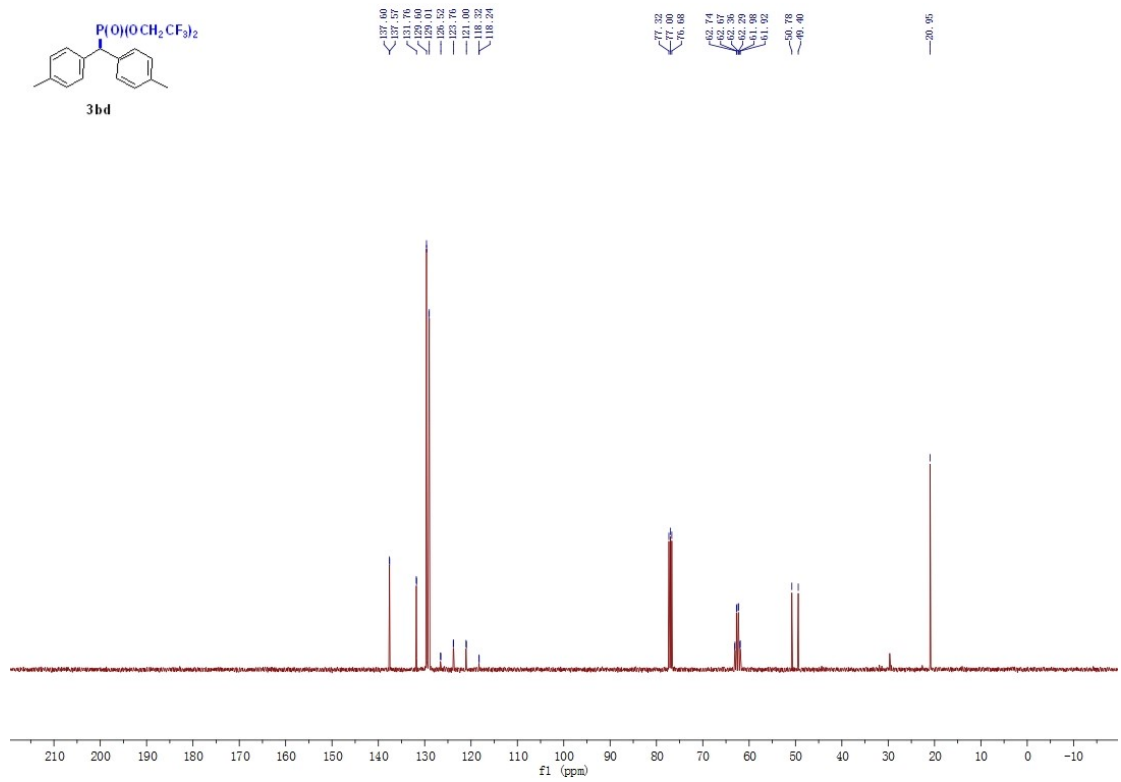
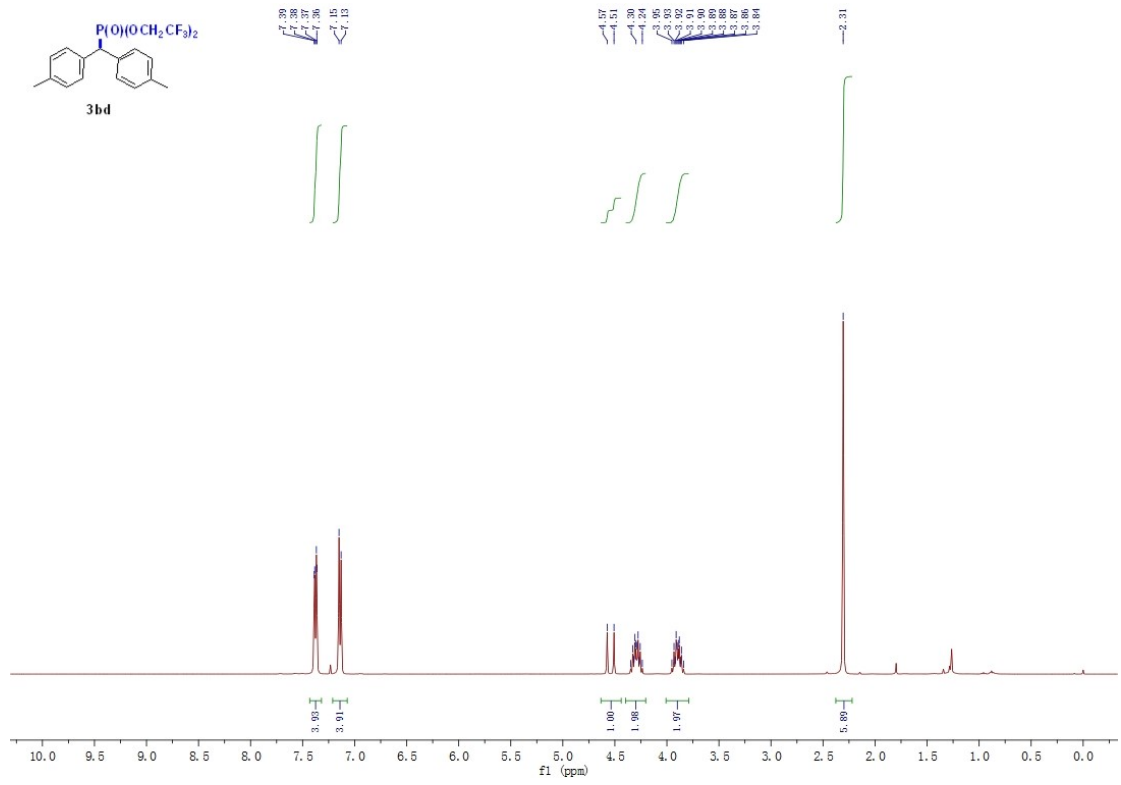
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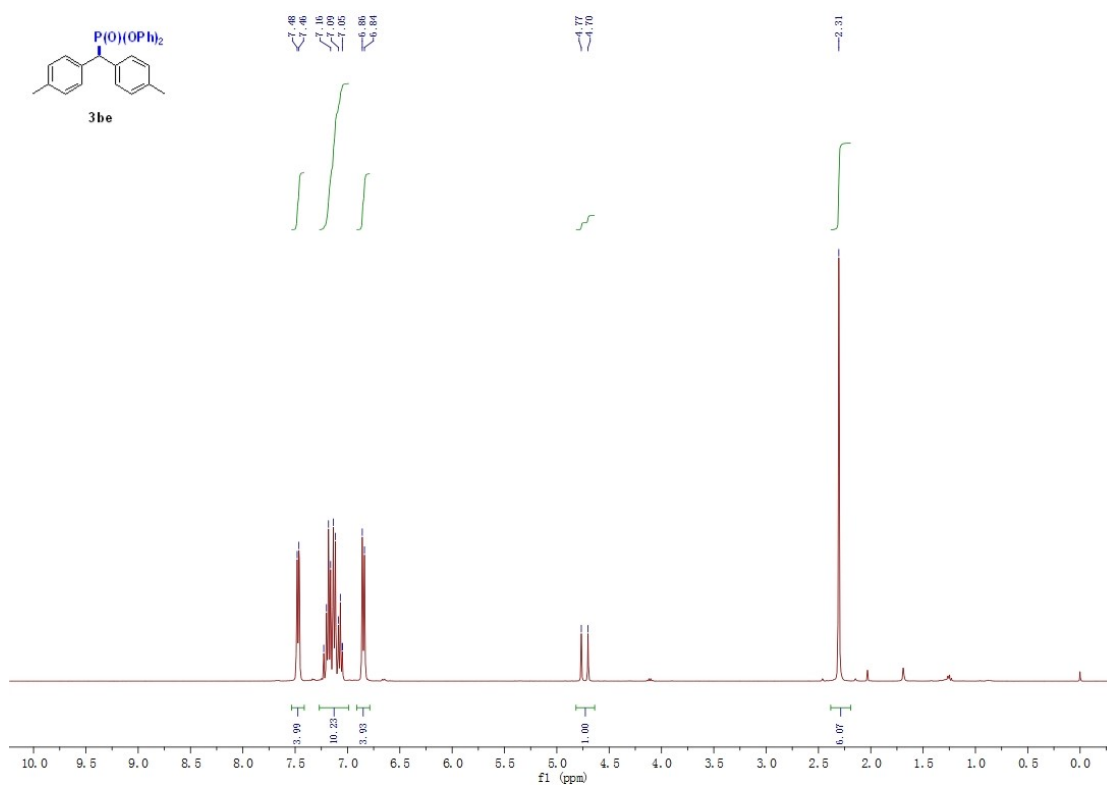
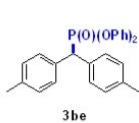
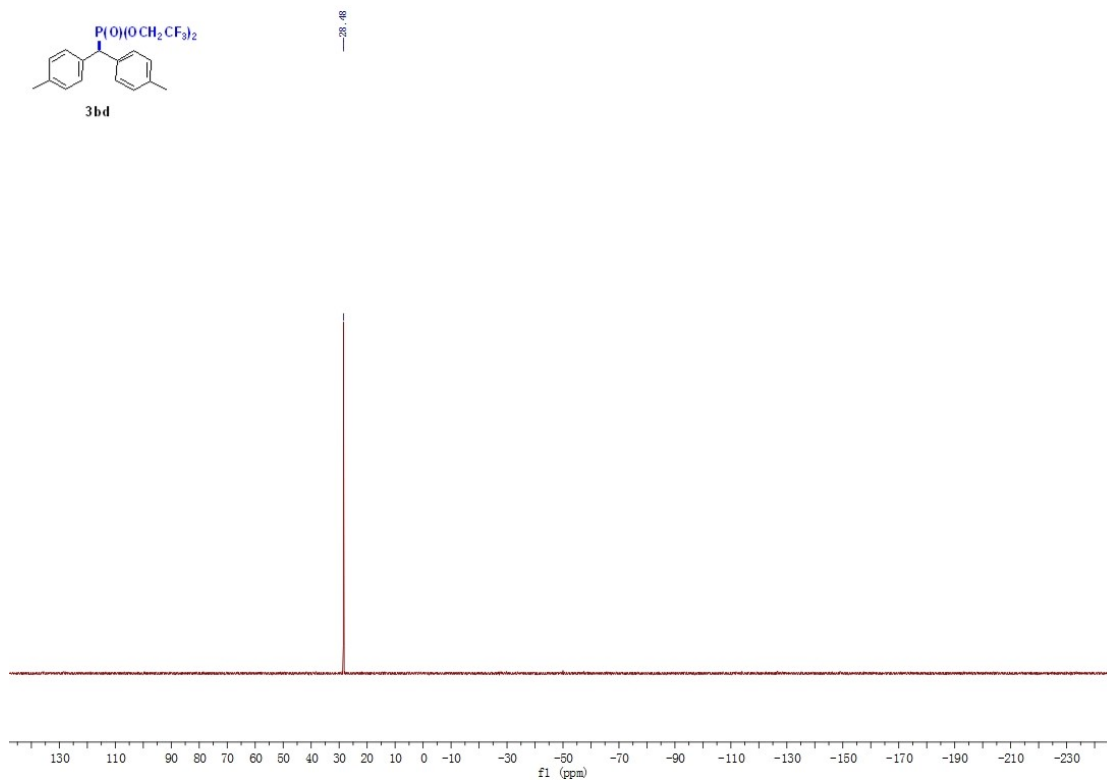
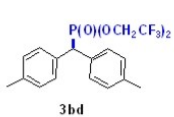
20.93
 19.51
 13.46

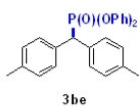


25.41







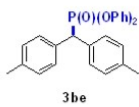
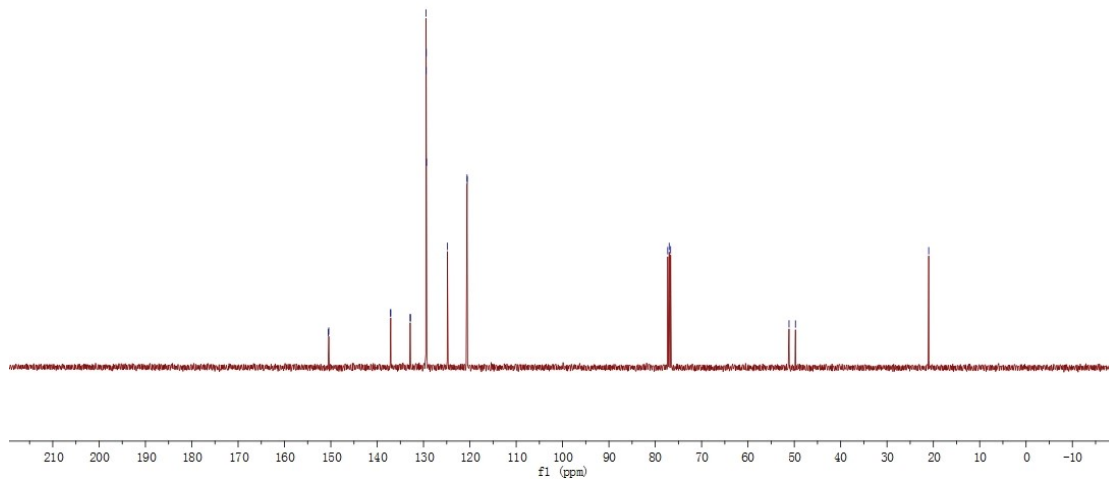


159.54
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 124.85
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 120.84

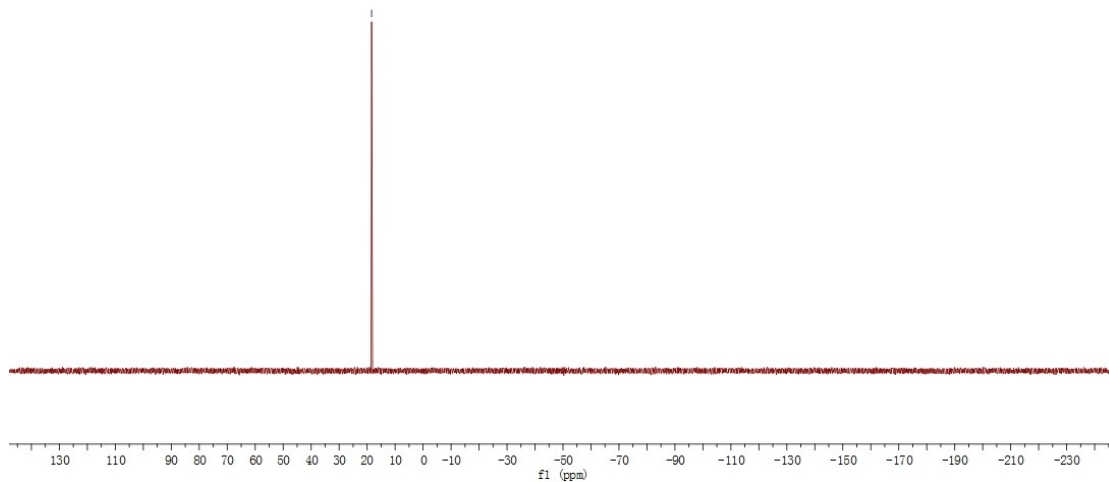
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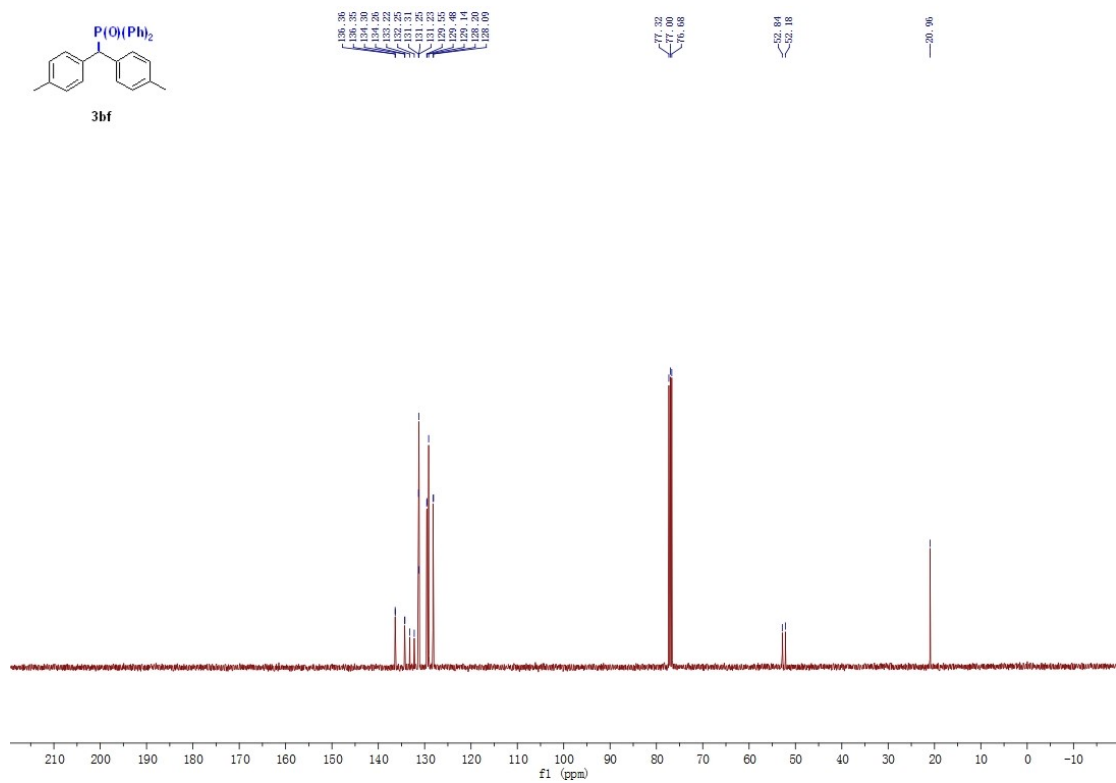
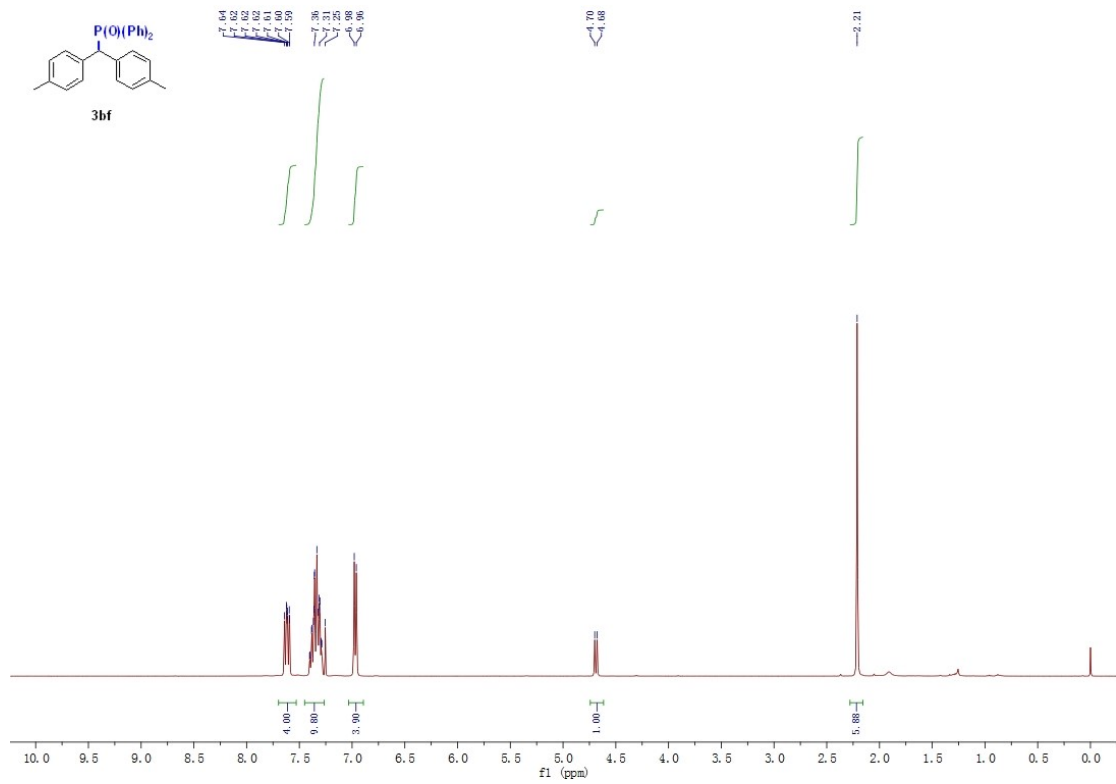
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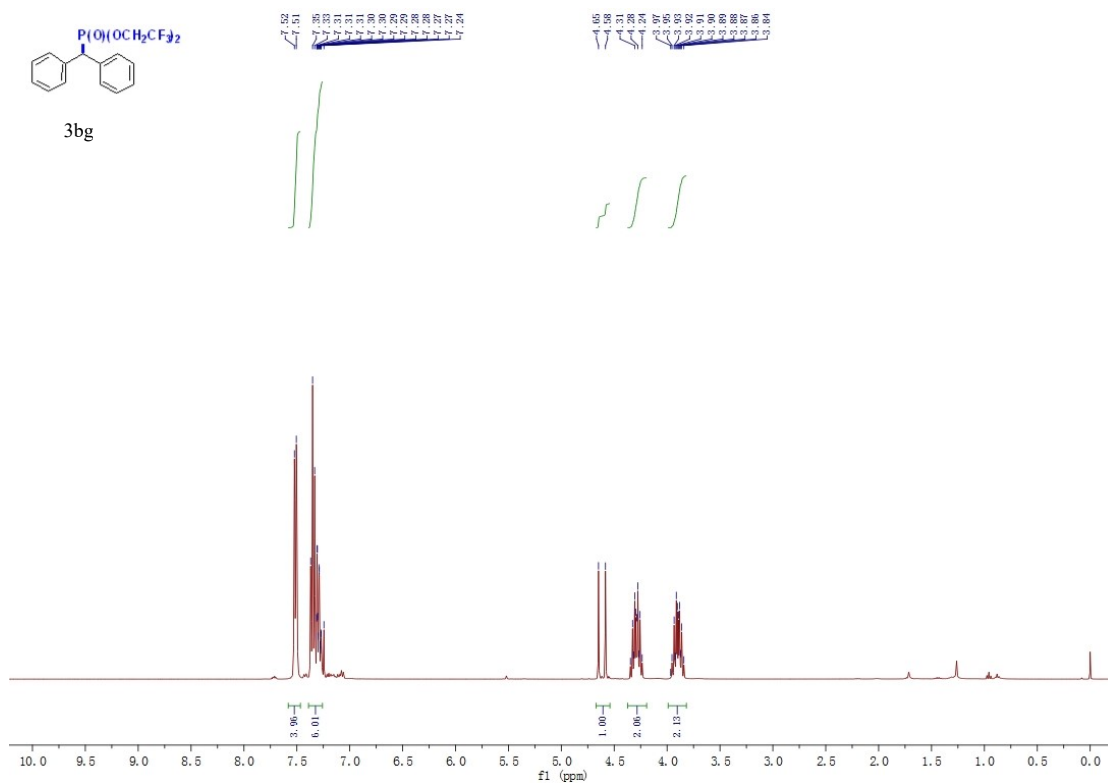
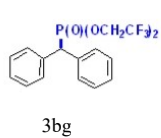
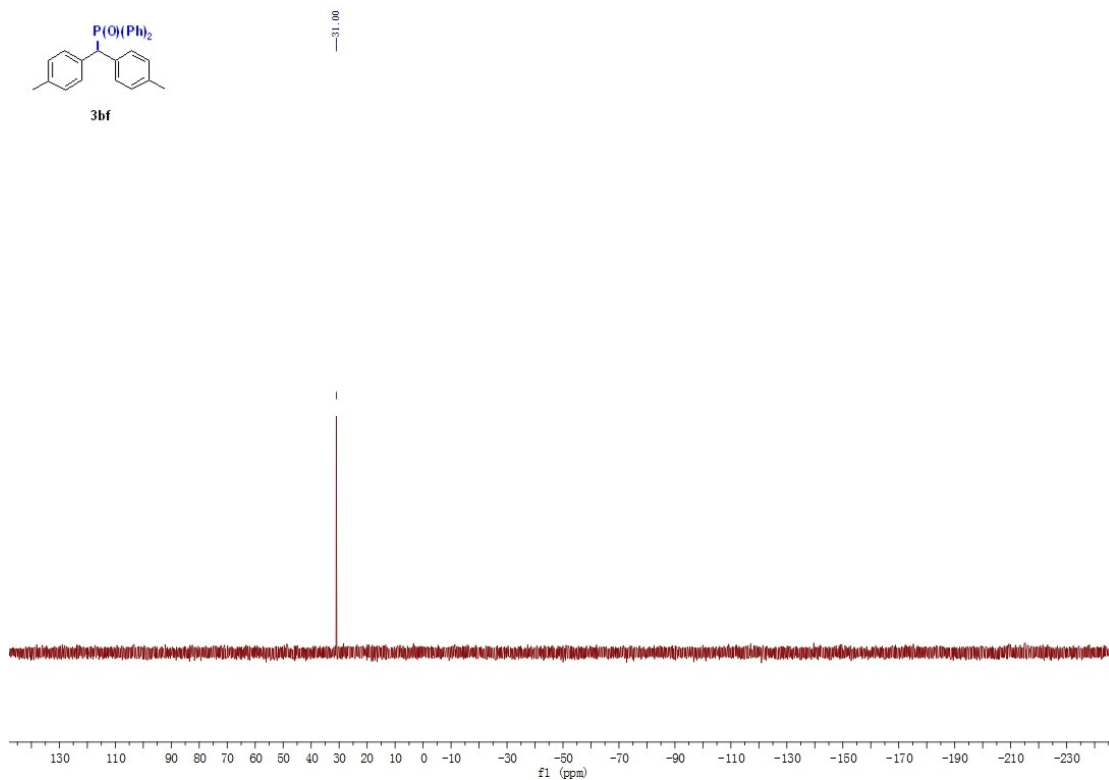
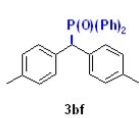
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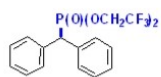


18.40





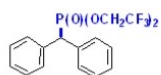
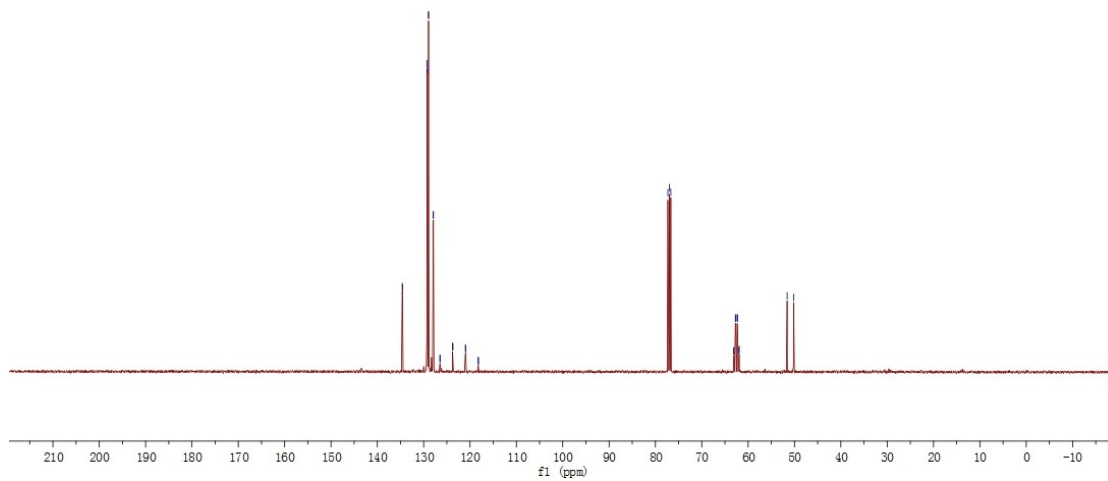




3bg

129.63
128.96
127.90
126.44
120.93
118.17

77.32
76.68
62.75
62.37
61.91
61.93
-51.58
-50.20



3bg

-28.06

