Supplementary Information

Metal-free direct difunctionalization of alkenes with I_2O_5 and P(O)–H compounds leading to β-iodophosphates

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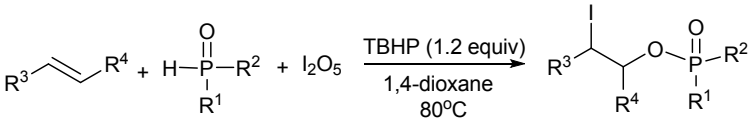
1. General information

All commercially available reagent grade chemicals were purchased from Aldrich, Acros, Alfa Aesar and Beijing Ouhe Chemical Company and used as received without further purification unless otherwise stated. ^{1}H NMR, ^{13}C NMR and ^{31}P NMR were recorded in $CDCl_3$ on a Bruker Avance III 400 spectrometer with TMS as internal standard (400 MHz ^{1}H, 100 MHz ^{13}C, 162 MHz ^{31}P) at room temperature, the chemical shifts (δ) were expressed in ppm and J values were given in Hz. The following abbreviations are used to indicate the multiplicity: singlet (s), doublet (d), triplet (t), quartet (q), doublet of doublets (dd), doublet of triplets (dt), and multiplet (m). All first order splitting patterns were assigned on the basis of the appearance of the multiplet. Splitting patterns that could not be easily interpreted were designated as multiplet (m). Mass analyses and HRMS were obtained on a Finnigan-LCQDECA mass spectrometer and a Bruker Daltonics Bio-TOF-Q mass spectrometer by the ESI method, respectively. Column chromatography was performed on silica gel (200-300 mesh).

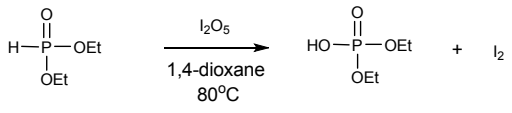
2. General procedure for metal-free difunctionalization of alkenes with I_2O_5 and P(O)–H compounds.



To a mixture of alkene **1** (0.25 mmol), P(O)-H compound **2** (0.5 mmol, **2c** and **2d** (0.75 mmol)), I_2O_5 (0.25 mmol) and TBHP (0.3 mmol) in a 25 mL round-bottomed flack at room temperature, was added the 1,4-dioxane (2 mL). The reaction vessel was allowed to stir at 80 °C for 16-24 h. After the reaction, the solution was concentrated in vacuum. The resulting mixture purified by flash column chromatography using a mixture of petroleum ether and ethyl acetate as eluent to give the desired product **3**.

3. Preliminary mechanistic studies.

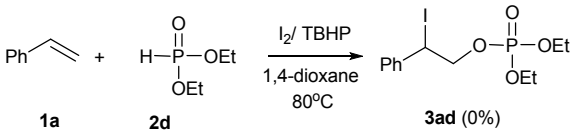
(1) The reaction of diethyl phosphonate **2d** with I_2O_5 under standard conditions.



2d **4d** (48%)

To a mixture of diethyl phosphonate **2d** (0.25 mmol) and I_2O_5 (0.25 mmol) in a 25 mL round-bottomed flack at room temperature, was added the 1,4-dioxane (2 mL). The reaction vessel was allowed to stir at 80 °C for 10 h. After the reaction, the solution was concentrated in vacuum, the desired product **4d** was obtained in 48% yield.

(2) The reaction of styrene **1a**, diethyl phosphonate **2d,** and I_2 in the presence of TBHP.

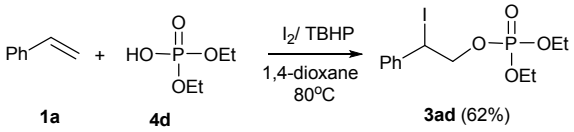


1a **2d** **3ad** (0%)

To a mixture of styrene **1a** (0.25 mmol), diethyl phosphonate **2d** (0.75 mmol)**,** I_2 (0.25 mmol), and TBHP (0.3 mmol) in a 25 mL round-bottomed flack at room

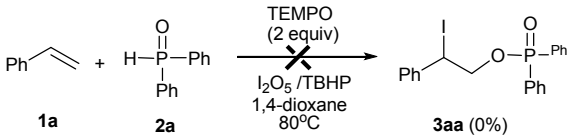
temperature, was added the 1,4-dioxane (2 mL). The reaction vessel was allowed to stir at 80 °C for 16 h. After the reaction, the solution was concentrated in vacuum, none of the desired product **3ad** was detected.

(3) The reaction of styrene **1a**, diethyl hydrogen phosphate **4d,** and I_2 in the presence of TBHP.



To a mixture of styrene **1a** (0.25 mmol), diethyl hydrogen phosphate **4d** (0.75 mmol)**,** I_2 (0.25 mmol), and TBHP (0.3 mmol) in a 25 mL round-bottomed flack at room temperature, was added the 1,4-dioxane (2 mL). The reaction vessel was allowed to stir at 80 °C for 16 h. After the reaction, the solution was concentrated in vacuum, the desired product **3ad** was isolated in 62% yield.

(4) The reaction of styrene **1a** and **2a** with TEMPO under the standard conditions.



To a mixture of styrene **1a** (0.25 mmol), diphenylphosphine oxide **2a** (0.5 mmol)**,** I_2O_5 (0.25 mmol), TBHP (0.3 mmol) and TEMPO (0.5 mmol) in a 25 mL round-bottomed flack at room temperature, was added the 1,4-dioxane (2 mL). The reaction vessel was allowed to stir at 80 °C for 16 h. After the reaction, the solution was concentrated in vacuum, the TEMPO-trapped complex ($Ph_2P(O)$-O-Tempo) was detected by LC-MS analysis and none of the desired product **3aa** was detected.

The copy of LC/MS for TEMPO trapping experiment:

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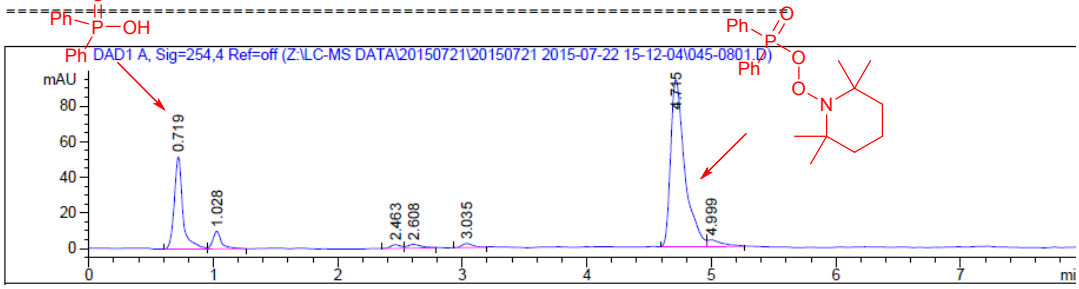

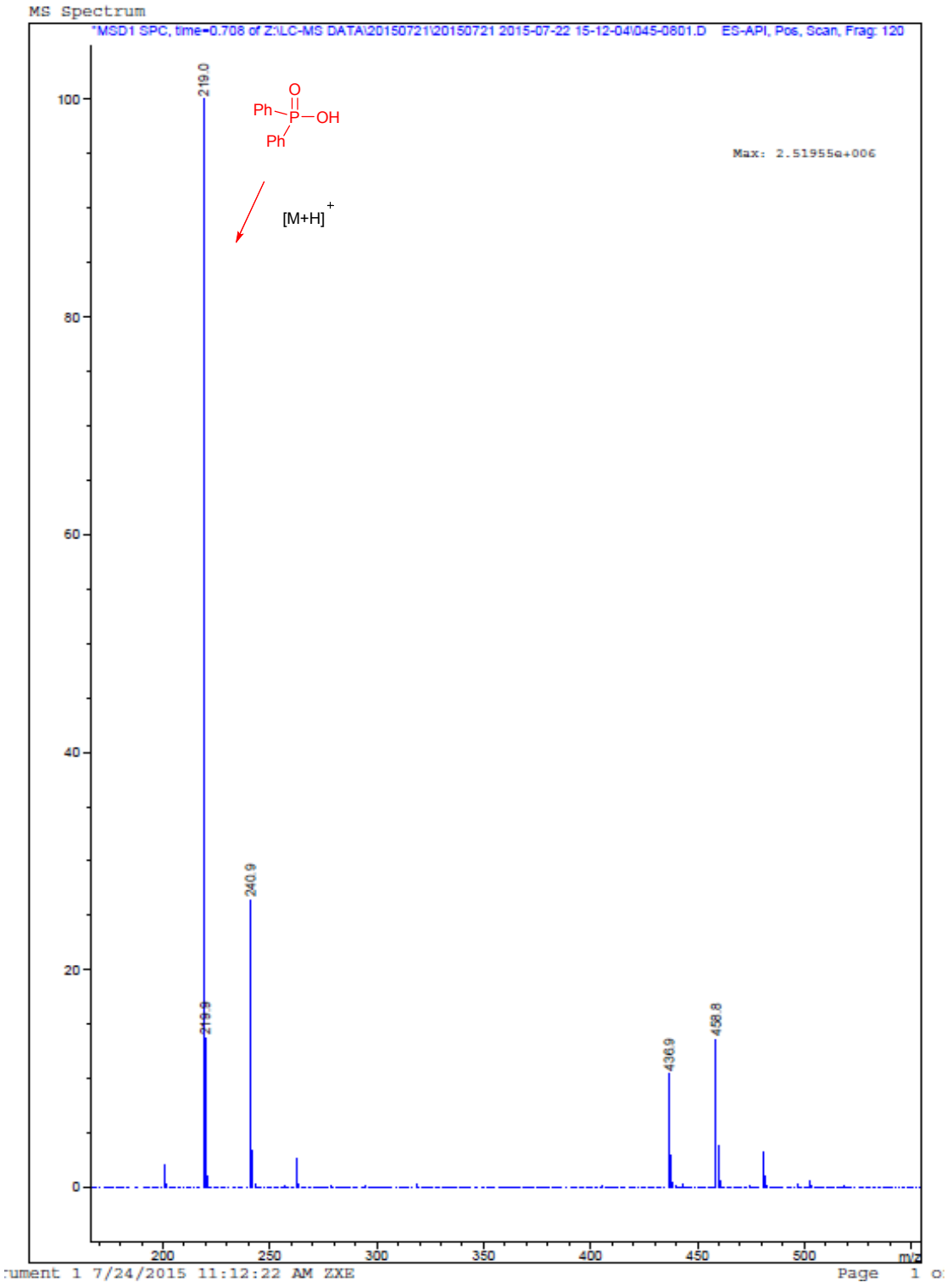
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458.8
[M+H]+
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80
60
40
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400
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m/z
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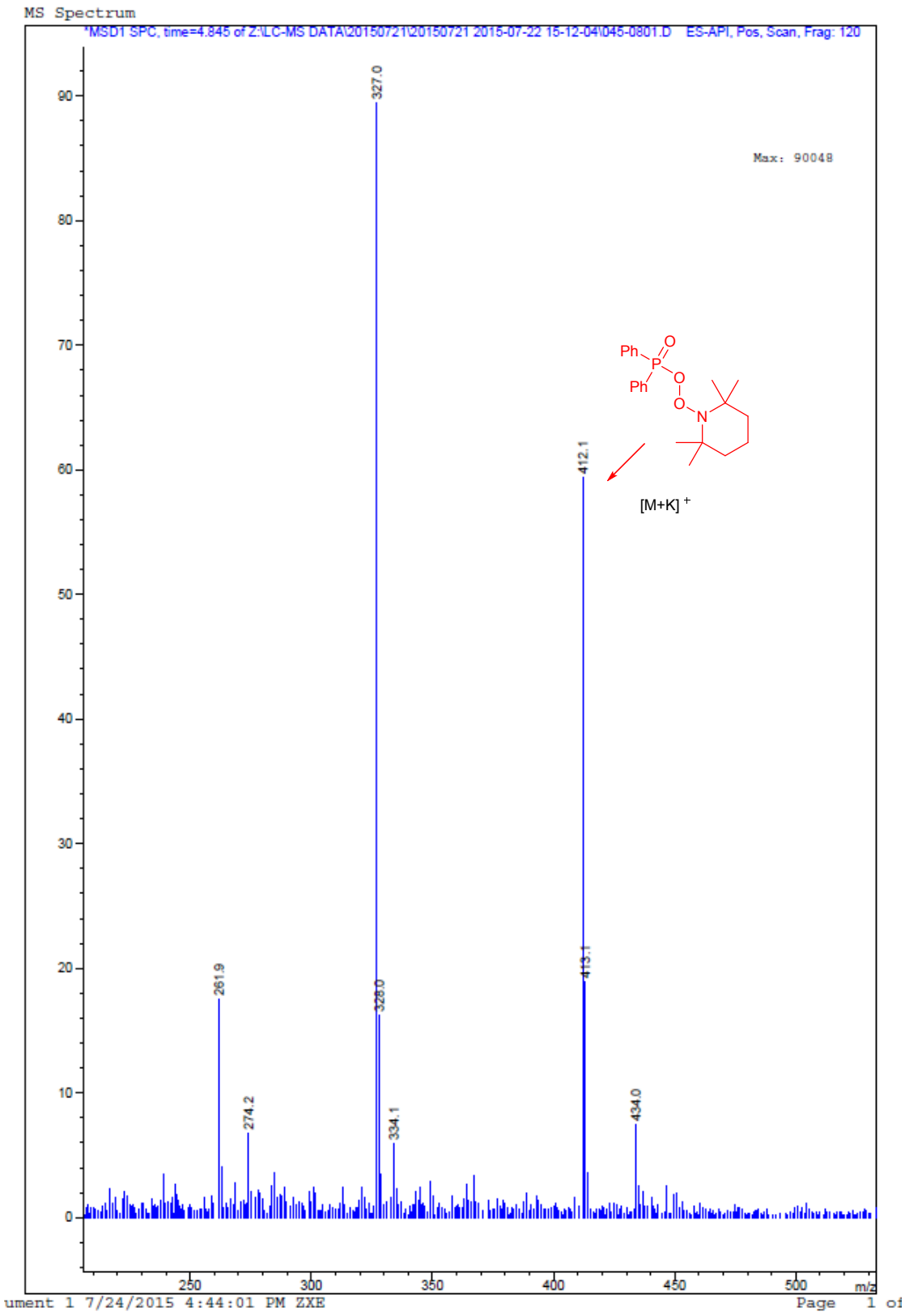
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434.0
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m/z
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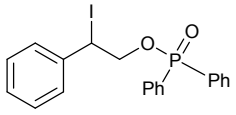
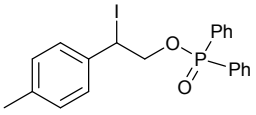
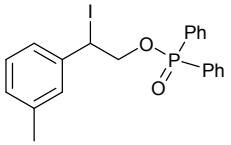
4. Characterization data of products (3aa-3ad)

2-iodo-2-phenylethyl diphenylphosphinate 

Compound **3aa** was obtained in 80% yield according to the general procedure (16h). ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.91-7.86 (m, 2H), 7.70-7.65 (m, 2H), 7.58-7.54 (m, 1H), 7.51-7.44 (m, 3H), 7.36-7.27 (m, 7H), 5.46-5.41 (m, 1H), 3.70 (dd, J_1 = 10.3 Hz, J_2 = 5.4 Hz, 1H), 3.61 (dd, J_1 = 10.3 Hz, J_2= 6.7 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 138.5 (d, J = 3.9 Hz), 132.3 (d, J = 2.8 Hz), 132.2 (d, J = 2.8 Hz), 131.9 (d, J = 10.3 Hz), 131.6 (d, J = 10.4 Hz), 131.5 (d, J = 138.4 Hz), 130.9 (d, J = 134.8 Hz), 128.7, 128.5 (d, J = 13.4 Hz), 128.4, 128.3 (d, J = 13.2 Hz), 126.7, 76.4 (d, J = 5.5 Hz), 10.7 (d, J = 4.5 Hz); ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.4; HRMS calc. for $C_{20}H_{19}O_2PI$ $(M+H)^+$, 449.0167; found, 449.0160.

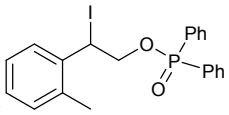
2-iodo-2-p-tolylethyl diphenylphosphinate 

Compound **3ba** was obtained in 67% yield according to the general procedure (20h). ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.90-7.85 (m, 2H), 7.70-7.66 (m, 2H), 7.55 (t, J = 7.2 Hz, 1H), 7.50-7.45 (m, 3H), 7.37-7.32 (m, 2H), 7.17 (d, J = 8.0 Hz, 2H), 7.12 (d, J = 8.0 Hz, 2H), 5.41-5.36 (m, 1H), 3.70 (dd, J_1 = 10.2 Hz, J_2 = 5.4 Hz, 1H), 3.60 (dd, J_1 = 10.2 Hz, J_2 = 6.9 Hz, 1H), 2.35 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 138.6, 135.5 (d, J = 4.0 Hz), 132.3 (d, J = 2.8 Hz), 132.2 (d, J = 2.8 Hz), 132.0 (d, J = 10.4 Hz), 131.6 (d, J = 10.3 Hz), 131.6 (d, J = 136.3 Hz), 131.0 (d, J = 133.8 Hz), 129.1, 128.5 (d, J = 13.3 Hz), 128.3 (d, J = 13.2 Hz), 126.6, 76.5 (d, J = 5.5 Hz), 21.3, 10.9 (d, J = 4.5 Hz); ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.2; HRMS calc. for $C_{21}H_{21}O_2PI$ $(M+H)^+$, 463.0324; found, 463.0327.

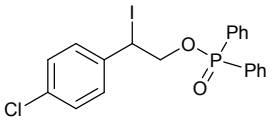
2-iodo-2-m-tolylethyl diphenylphosphinate 

Compound **3ca** was obtained in 63% yield according to the general procedure (20h). ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.91-7.86 (m, 2H), 7.69-7.64 (m, 2H), 7.56 (q, J_1 = 7.4 Hz, J_2 = 1.3 Hz, 1H), 7.51-7.45 (m, 3H), 7.36-7.32 (m, 2H), 7.20 (t, J = 7.6 Hz, 1H), 7.10 (t, J = 4.8 Hz, 2H), 7.02 (s, 1H), 5.43-5.37 (m, 1H), 3.70 (dd, J_1 = 10.3 Hz, J_2 = 5.6 Hz, 1H), 3.60 (dd, J_1 = 10.3 Hz, J_2 = 6.7 Hz, 1H), 2.30 (s, 3H); ^{13}C NMR

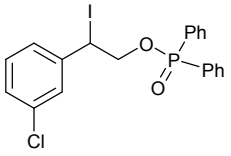
($CDCl_3$, 100 MHz, ppm): δ 138.3 (d, J = 3.7 Hz), 138.0, 132.3 (d, J = 2.7 Hz), 132.2 (d, J = 2.8 Hz), 132.0 (d, J = 10.4 Hz), 131.6 (d, J = 10.4 Hz), 131.6 (d, J = 136.3 Hz), 131.0 (d, J = 133.7 Hz), 129.5, 128.5 (d, J = 13.3 Hz), 128.3, 128.2 (d, J = 13.3 Hz), 127.4, 123.8, 76.6 (d, J = 5.4 Hz), 21.4, 10.6 (d, J = 4.8 Hz); ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.3; HRMS calc. for $C_{21}H_{21}O_2PI$ $(M+H)^+$, 463.0324; found, 463.0328.

2-iodo-2-o-tolylethyl diphenylphosphinate 

Compound **3da** was obtained in 64% yield according to the general procedure (20h). ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.90-7.85 (m, 2H), 7.65-7.60 (m, 2H), 7.60-7.54 (m, 1H), 7.51-7.43 (m, 4H), 7.33-7.28 (m, 2H), 7.24-7.18 (m, 2H), 7.04 (t, J = 6.4 Hz, 1H), 5.70-5.65 (m, 1H), 3.72 (dd, J_1 = 10.2 Hz, J_2 = 6.0 Hz, 1H), 3.59 (dd, J_1 = 10.1 Hz, J_2 = 7.2 Hz, 1H), 2.09 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 137.0 (d, J = 3.4 Hz), 135.2, 132.3 (d, J = 2.6 Hz), 132.1 (d, J = 2.5 Hz), 132.0 (d, J = 10.3 Hz), 131.6 (d, J = 10.3 Hz), 131.6 (d, J = 137.1 Hz), 130.9 (d, J = 133.7 Hz), 130.3, 128.6, 128.5 (d, J = 13.7 Hz), 128.2 (d, J = 13.1 Hz), 126.4, 126.3, 72.9 (d, J = 5.3 Hz), 19.2, 9.4 (d, J = 4.6 Hz); ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.1; HRMS calc. for $C_{21}H_{21}O_2PI$ $(M+H)^+$, 463.0324; found, 463.0332.

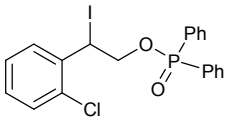
2-(4-chlorophenyl)-2-iodoethyl diphenylphosphinate 

Compound **3ea** was obtained in 76% yield according to the general procedure (20h). ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.90-7.85 (m, 2H), 7.70-7.64 (m, 2H), 7.60-7.56 (m, 1H), 7.52-7.47 (m, 3H), 7.39-7.35 (m, 2H), 7.28 (t, J = 3.3 Hz, 2H), 7.22 (d, J = 8.5 Hz, 2H), 5.43-5.38 (m, 1H), 3.66 (dd, J_1 = 10.4 Hz，J_2 = 5.2 Hz, 1H), 3.57 (dd, J_1 = 10.3 Hz, J_2 = 6.9 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 137.1 (d, J = 3.9 Hz), 134.6, 132.5 (d, J = 2.8 Hz), 132.4 (d, J = 2.8 Hz), 131.8 (d, J = 10.4 Hz), 131.6 (d, J = 10.4 Hz), 131.3 (d, J = 138.0 Hz), 130.7 (d, J = 133.9 Hz), 128.6, 128.6 (d, J = 13.3 Hz), 128.5 (d, J = 13.2 Hz), 128.1, 75.6 (d, J = 5.4 Hz), 10.3 (d, J = 4.5 Hz); ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.8; HRMS calc. for $C_{20}H_{18}O_2PClI$ $(M+H)^+$, 482.9778; found, 482.9774.

2-(3-chlorophenyl)-2-iodoethyl diphenylphosphinate 

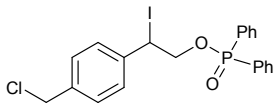
Compound **3fa** was obtained in 68% yield according to the general procedure (20h). ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.91-7.86 (m, 2H), 7.67 (m, 2H), 7.61-7.57 (m, 1H), 7.53-7.48 (m, 3H), 7.39-7.35 (m, 2H), 7.27-7.22 (m, 3H), 7.17 (d, J = 7.4 Hz, 1H), 5.42-5.37 (m, 1H), 3.65 (dd, J_1 = 10.4 Hz, J_2 = 5.3 Hz, 1H), 3.57 (dd, J_1 = 10.4 Hz, J_2 = 6.7 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 140.5 (d, J = 3.8 Hz), 134.3, 132.5 (d, J = 2.8 Hz), 132.4 (d, J = 2.8 Hz), 131.9 (d, J = 10.4 Hz), 131.6 (d, J = 10.5 Hz), 131.2 (d, J = 139.7 Hz), 130.5 (d, J = 135.4 Hz), 129.7, 128.9, 128.6 (d, J = 13.4 Hz), 128.4 (d, J = 13.2 Hz), 126.8, 125.0, 75.4 (d, J = 5.3 Hz), 10.2 (d, J = 4.4 Hz); ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.9; HRMS calc. for $C_{20}H_{18}O_2PClI$ $(M+H)^+$, 482.9778; found, 482.9776.

2-(2-chlorophenyl)-2-iodoethyl diphenylphosphinate



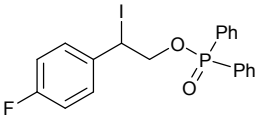
Compound **3ga** was obtained in 60% yield according to the general procedure (20h). ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.94-7.89 (m, 2H), 7.77-7.72 (m, 2H), 7.61-7.57 (m, 2H), 7.53-7.47 (m, 3H), 7.40-7.35 (m, 2H), 7.32-7.25 (m, 3H), 5.77-5.72 (m, 1H), 3.73-3.67 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 136.4 (d, J = 3.8 Hz), 132.5 (d, J = 2.6 Hz), 132.4 (d, J = 2.7 Hz), 131.9 (d, J = 10.5 Hz), 131.8, 131.7 (d, J = 10.5 Hz), 131.1 (d, J = 137.6 Hz), 130.7 (d, J = 134.4 Hz), 129.7, 129.5, 128.6 (d, J = 1.5 Hz), 128.5 (d, J = 1.7 Hz), 128.4, 126.8, 72.3 (d, J = 5.0 Hz), 10.3 (d, J = 4.5 Hz); ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.6; HRMS calc. for $C_{20}H_{18}O_2PClI$ $(M+H)^+$, 482.9778; found, 482.9774.

2-(4-(chloromethyl)phenyl-2-iodoethyl diphenylphosphinate

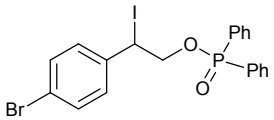


Compound **3ha** was obtained in 63% yield according to the general procedure (24h). ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.91-7.86 (m, 2H), 7.69-7.64 (m, 2H), 7.59-7.55 (m, 1H), 7.51-7.46 (m, 3H), 7.37-7.32 (m, 4H), 7.28 (d, J = 7.2 Hz, 2H), 5.45-5.40 (m, 1H), 4.58 (s, 2H), 3.69 (dd, J_1 = 10.4 Hz, J_2 = 5.4 Hz, 1H), 3.59 (dd, J_1 = 10.3 Hz, J_2 = 6.8 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 138.7 (d, J = 3.8 Hz), 137.9, 132.4 (d, J = 2.8 Hz), 132.3 (d, J = 2.8 Hz), 131.9 (d, J = 10.4 Hz), 131.6 (d, J = 10.4 Hz), 131.4 (d, J = 137.8 Hz), 130.8 (d, J = 133.9 Hz), 128.6, 128.5 (d, J = 13.6 Hz), 128.4 (d, J = 13.2 Hz), 127.1, 75.9 (d, J = 5.4 Hz), 45.7, 10.4 (d, J = 4.5 Hz); ^{31}P NMR (162

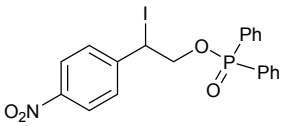
MHz, $CDCl_3$): δ 32.5; HRMS calc. for $C_{21}H_{20}O_2PClI$ $(M+H)^+$, 496.9934; found, 496.9944.

2-(4-fluorophenyl)-2-iodoethyl diphenylphosphinate 

Compound **3ia** was obtained in 74% yield according to the general procedure (24h). ^1H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.90-7.85 (m, 2H), 7.69-7.63 (m, 2H), 7.60-7.56 (m, 1H), 7.52-7.46 (m, 3H), 7.38-7.33 (m, 2H), 7.28-7.24 (m, 2H), 7.02-6.96 (m, 2H), 5.45-5.40 (m, 1H), 3.68 (dd, J_1 = 10.4 Hz, J_2 = 5.4 Hz, 1H), 3.57 (dd, J_1 = 10.3 Hz, J_2 = 6.9 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 162.5 (d, J = 198.2 Hz), 134.4 (t, J = 3.5 Hz), 132.4 (d, J = 2.8 Hz), 132.3 (d, J = 2.8 Hz), 131.9 (d, J = 10.3 Hz), 131.6 (d, J = 10.3 Hz), 131.4 (d, J = 138.1 Hz), 130. (d, J = 133.9 Hz), 128.6 (d, J = 5.2 Hz), 128.5 (d, J = 5.7 Hz), 128.3, 115.3 (d, J = 21.6 Hz), 75.6 (d, J = 5.5 Hz), 10.5 (d, J = 4.7 Hz); ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.5; HRMS calc. for $C_{20}H_{18}O_2PIF$ $(M+H)^+$, 467.0073; found, 467.0065.

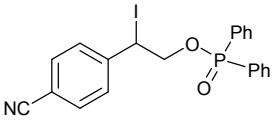
2-(4-bromophenyl)-2-iodoethyl diphenylphosphinate 

Compound **3ja** was obtained in 72% yield according to the general procedure (24h). ^1H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.90-7.84 (m, 2H), 7.70-7.64 (m, 2H), 7.60-7.56 (m, 1H), 7.52-7.48 (m, 3H), 7.44 (d, J = 8.4 Hz, 2H), 7.36 (t, J = 3.9 Hz, 2H), 7.16 (d, J = 8.4 Hz, 2H), 5.41-5.35 (m, 1H), 3.66 (dd, J_1 = 10.4 Hz, J_2 = 5.2 Hz, 1H), 3.56 (dd, J_1 = 10.3 Hz, J_2 = 6.9 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 137.6 (d, J = 3.9 Hz), 132.5 (d, J = 2.8 Hz), 132.4 (d, J = 2.8 Hz), 131.8 (d, J = 10.4 Hz), 131.6 (d, J = 10.4 Hz), 131.6, 131.3 (d, J = 137.9 Hz), 130.7 (d, J = 133.7 Hz), 128.6 (d, J = 13.4 Hz), 128.5 (d, J = 14.2 Hz), 128.4, 122.8, 75.6 (d, J = 5.3 Hz), 10.2 (d, J = 4.5 Hz); ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.7; HRMS calc. for $C_{20}H_{18}O_2PBrI$ $(M+H)^+$, 526.9272; found, 526.9276.

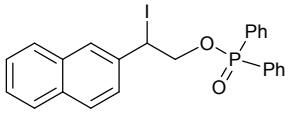
2-iodo-2-(4-nitrophenyl)ethyl diphenylphosphinate 

Compound **3ka** was obtained in 70% yield according to the general procedure (24h). ^1H NMR ($CDCl_3$, 400 MHz, ppm): δ 8.18 (d, J = 8.7 Hz, 2H), 7.92-7.87 (m, 2H), 7.72-7.67 (m, 2H), 7.61 (t, J = 7.4 Hz, 1H), 7.54-7.48 (m, 5H), 7.41-7.36 (m, 2H),

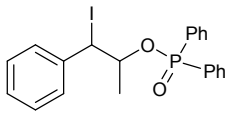
5.54-5.49 (m, 1H), 3.67-3.57 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 147.9, 145.6 (d, J = 3.8 Hz), 132.8 (d, J = 2.8 Hz), 132.7 (d, J = 2.8 Hz), 131.7 (d, J = 9.4 Hz), 131.6 (d, J = 9.5 Hz), 130.8 (d, J = 137.2 Hz), 130.4 (d, J = 134.3 Hz), 128.8 (d, J = 13.6 Hz), 128.6 (d, J = 13.4 Hz), 127.6, 123.7, 74.6 (d, J = 5.1 Hz), 9.8 (d, J = 4.7 Hz); ^{31}P NMR (162 MHz, $CDCl_3$): δ 33.5; HRMS calc. for $C_{20}H_{18}NO_4PI$ $(M+H)^+$, 494.0018; found, 494.0018.

2-(4-cyanophenyl)-2-iodoethyl diphenylphosphinate 

Compound **3la** was obtained in 83% yield according to the general procedure (24h). ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.90-7.85 (m, 2H), 7.70-7.65 (m, 2H), 7.60 (t, J = 8.4 Hz, 3H), 7.53-7.49 (m, 3H), 7.42-7.35 (m, 4H), 5.48-5.43 (m, 1H), 3.63-3.55 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 143.7 (d, J = 3.7 Hz), 132.7 (d, J = 2.8 Hz), 132.6 (d, J = 2.7 Hz), 132.3, 131.7 (d, J = 10.6 Hz), 131.6 (d, J = 10.6 Hz), 130.9 (d, J = 137.3 Hz), 130.5 (d, J = 134.3 Hz), 128.7 (d, J = 13.4 Hz), 128.5 (d, J = 13.3 Hz), 127.4, 118.4, 112.6, 74.8 (d, J = 5.2 Hz), 9.8 (d, J = 4.5 Hz); ^{31}P NMR (162 MHz, $CDCl_3$): δ 33.2; HRMS calc. for $C_{21}H_{18}NO_2PI$ $(M+H)^+$, 474.0120; found, 474.0124.

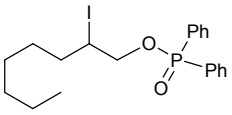
2-iodo-2-(naphthalen-2-yl)ethyl diphenylphosphinate 

Compound **3ma** was obtained in 71% yield according to the general procedure (24h). ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.94-7.89 (m, 2H), 7.85-7.78 (m, 3H), 7.71-7.65 (m, 3H), 7.59-7.55 (m, 1H), 7.52-7.48 (m, 4H), 7.45 (dd, J_1 = 8.5 Hz, J_2 = 1.6 Hz, 1H), 7.42-7.38 (m, 1H), 7.30-7.25 (m, 2H), 5.64-5.59 (m, 1H), 3.81 (dd, J_1 = 10.3 Hz, J_2 = 5.4 Hz, 1H), 3.71 (dd, J_1 = 10.3 Hz, J_2 = 7.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 135.7 (d, J = 4.0 Hz), 133.4, 132.8, 132.4 (d, J = 2.8 Hz), 132.2 (d, J = 2.8 Hz), 131.9 (d, J = 10.4 Hz), 131.6 (d, J = 10.3 Hz), 131.5 (d, J = 138.5 Hz), 130.9 (d, J = 133.4 Hz), 128.5 (d, J = 13.1 Hz), 128.3 (d, J = 13.5 Hz), 128.2, 127.7, 126.8, 126.5, 126.4, 123.6, 76.7 (d, J = 5.6 Hz), 10.4 (d, J = 4.3 Hz); ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.5; HRMS calc. for $C_{24}H_{21}O_2PI$ $(M+H)^+$, 499.0324; found, 499.0323.

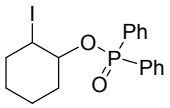
1-iodo-1-phenylpropan-2-yl diphenylphosphinate 

Compound **3na** was obtained in 67% yield according to the general procedure (24h). ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.90-7.85 (m, 2H), 7.60-7.55 (m, 3H), 7.51-7.47

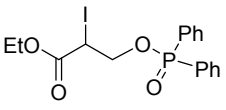
(m, 2H), 7.41 (t, J = 7.5 Hz, 1H), 7.28-7.24 (m, 7H), 5.23-5.19 (m, 1H), 4.61-4.54 (m, 1H), 1.97 (d, J = 1.7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 137.7 (d, J = 2.7 Hz), 132.3 (d, J = 2.8 Hz), 132.0 (d, J = 2.8 Hz), 131.9 (d, J = 10.4 Hz), 131.6 (d, J = 10.3 Hz), 131.5 (d, J = 139.4 Hz), 130.9 (d, J = 133.1 Hz), 128.6, 128.5 (d, J = 13.4 Hz), 128.1 (d, J = 13.2 Hz), 127.9, 127.6, 80.6 (d, J = 5.7 Hz), 31.2 (d, J = 5.3 Hz), 24.4; ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.2; HRMS calc. for $C_{21}H_{21}O_2PI$ $(M+H)^+$, 463.0324; found, 463.0329.

2-iodooctyl diphenylphosphinate 

Compound **3oa** was obtained in 56% yield according to the general procedure (24h). ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.89-7.82 (m, 4H), 7.57-7.53 (m, 2H), 7.50-7.45 (m, 4H), 4.21-4.16 (m, 1H), 3.42 (d, J = 4.4 Hz, 2H), 1.81-1.74 (m, 2H), 1.27-1.22 (m, 8H), 0.86 (t, J = 6.7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 132.3 (d, J = 2.3 Hz), 131.8 (t, J = 136.7 Hz),131.7 (t, J = 11.2 Hz), 128.6 (d, J = 2.9 Hz), 128.5 (d, J = 2.8 Hz), 73.7 (d, J = 6.0 Hz), 35.8 (d, J = 4.2 Hz), 31.6, 28.8, 24.5, 22.5, 14.1, 11.4; ^{31}P NMR (162 MHz, $CDCl_3$): δ 31.2; HRMS calc. for $C_{20}H_{27}O_2PI$ $(M+H)^+$, 457.0793; found, 457.0804.

2-iodocyclohexyl diphenylphosphinate 

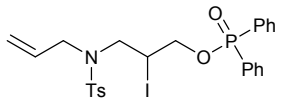
Compound **3pa** was obtained in 68% yield according to the general procedure (24h). ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.93-7.88 (m, 4H), 7.57-7.53 (m, 2H), 7.50-7.46 (m, 4H), 4.52-4.45 (m, 1H), 3.35-4.30 (m, 1H), 2.43-2.33 (m, 2H), 2.02-1.94 (m, 1H), 1.83-1.77 (m, 1H), 1.66-1.57 (m, 2H), 1.44-1.36 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 132.2 (d, J = 138.0 Hz), 132.1 (t, J = 2.1 Hz), 132.0 (t, J = 10.4 Hz), 131.7 (d, J = 10.1 Hz), 131.4 (d, J = 135.7 Hz), 128.5 (d, J = 5.2 Hz), 128.4 (d, J = 5.3 Hz), 78.7 (d, J = 6.0 Hz), 36.3, 33.1 (d, J = 6.7 Hz), 32.5, 25.8, 22.8; ^{31}P NMR (162 MHz, $CDCl_3$): δ 30.5; HRMS calc. for $C_{18}H_{21}O_2PI$ $(M+H)^+$, 427.0324; found, 427.0327.

ethyl 3-(diphenylphosphoryloxy)-2-iodopropanoate 

Compound **3qa** was obtained in 55% yield according to the general procedure (24h). ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.86-7.77 (m, 4H), 7.60-7.54 (m, 2H), 7.52-7.45

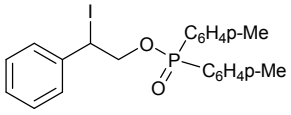
(m, 4H), 4.62-4.58 (m, 1H), 4.46-4.39 (m, 1H), 4.35-4.29 (m, 1H), 4.24 (q, J = 7.1 Hz 2H), 1.28 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 169.4, 132.5 (t, J = 2.8 Hz), 132.4 (t, J = 2.7 Hz), 131.8 (d, J = 10.3 Hz), 131.5 (d, J = 10.3 Hz), 131.0 (d, J = 136.2 Hz), 130.6 (d, J = 134.8 Hz), 128.7 (d, J = 8.0 Hz), 128.5 (d, J = 8.0 Hz), 65.8 (d, J = 5.3 Hz), 62.2, 16.8 (d, J = 7.0 Hz), 13.7; ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.6; HRMS calc. for $C_{17}H_{19}O_4PI$ $(M+H)^+$, 445.0066; found, 445.0067.

3-(N-allyl-4-methylphenylsulfonamido)-2-iodopropyl diphenylphosphinate

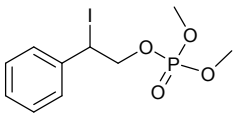


Compound **3ra** was obtained in 45% yield according to the general procedure (24h). ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.93-7.88 (m, 4H), 7.69 (d, J = 1.7 Hz, 2H), 7.57 (t, J = 7.4 Hz, 2H), 7.52-7.48 (m, 4H), 7.29 (d, J = 8.1 Hz, 2H), 5.54-5.46 (m, 1H), 5.08 (d, J = 5.5 Hz, 1H), 5.04 (s, 1H), 4.61-4.55 (m, 1H), 4.35-4.29 (m, 1H), 4.27-4.21 (m, 1H), 3.93 (dd, J_1 = 6.5 Hz, J_2 = 15.6 Hz, 1H), 3.78 (dd, J_1 = 6.9 Hz, J_2 = 15.6 Hz, 1H), 3.64 (dd, J_1 = 8.4 Hz, J_2 = 15.0 Hz, 1H), 3.45 (dd, J_1 = 6.8 Hz, J_2 = 15.0 Hz, 1H), 2.44 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 143.8, 135.9, 132.5 (t, J = 2.3 Hz), 132.2, 131.8 (d, J = 10.3 Hz), 131.7 (d, J = 10.2 Hz), 130.7 (d, J = 135.1 Hz), 129.9, 129.6 (d, J = 146.5 Hz), 128.6 (d, J = 13.2 Hz), 120.3, 66.3 (d, J = 5.2 Hz), 52.4, 52.0, 28.0 (d, J = 8.1 Hz), 21.5; ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.1; HRMS calc. for $C_{25}H_{28}O_4PSIN$ $(M+H)^+$, 596.0521; found, 596.0525.

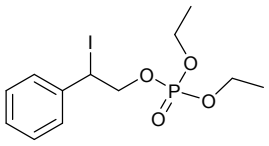
2-iodo-2-phenylethyl dip-tolylphosphinate



Compound **3ab** was obtained in 76% yield according to the general procedure (18h). ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.78-7.73 (m, 2H), 7.59-7.54 (m, 2H), 7.33-7.27 (m, 7H), 7.16-7.13 (m, 2H), 5.41-5.36 (m, 1H), 3.69 (dd, J_1 = 2.6 Hz, J_2 = 1.3 Hz, 1H), 3.60 (dd, J_1 = 2.6 Hz, J_2 = 1.7 Hz, 1H), 2.41 (s, 3H), 2.35 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 142.8 (d, J = 3.0 Hz), 142.7 (d, J = 2.9 Hz), 138.8 (d, J = 4.1 Hz), 131.9 (d, J = 10.7 Hz), 131.6 (d, J = 10.8 Hz), 129.2 (d, J = 13.9 Hz), 129.1 (d, J = 13.9 Hz), 128.6, 128.5 (d, J = 140.3 Hz), 128.4, 127.8 (d, J = 136.5 Hz), 126.7, 76.0 (d, J = 5.4 Hz), 21.7 (d, J = 0.9 Hz), 21.6 (d, J = 1.0 Hz), 11.2 (d, J = 4.2 Hz); ^{31}P NMR (162 MHz, $CDCl_3$): δ 33.5; HRMS calc. for $C_{22}H_{22}O_2IPNa$ $(M+Na)^+$, 499.0300; found, 499.0303.

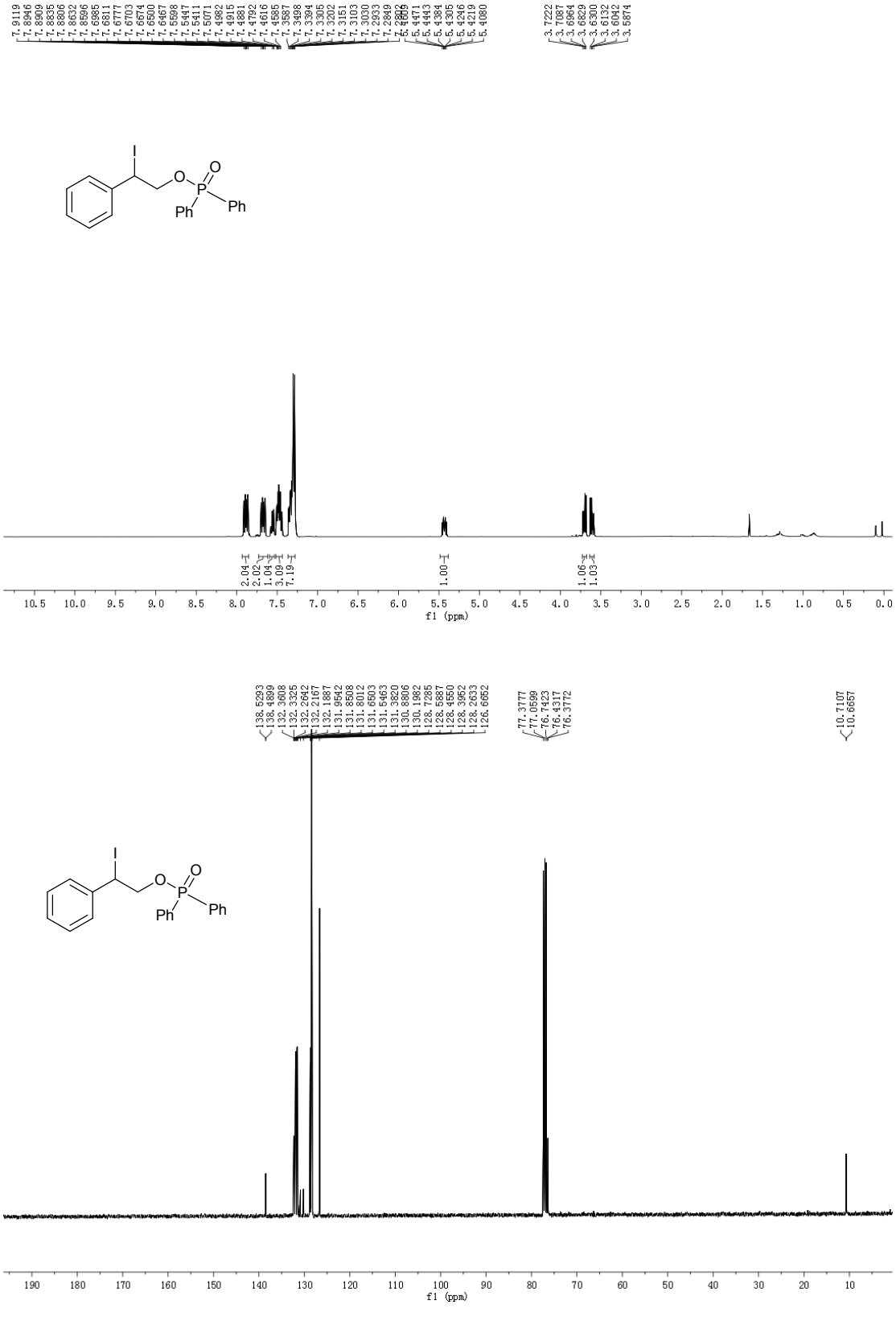
2-iodo-2-phenylethyl dimethyl phosphate 

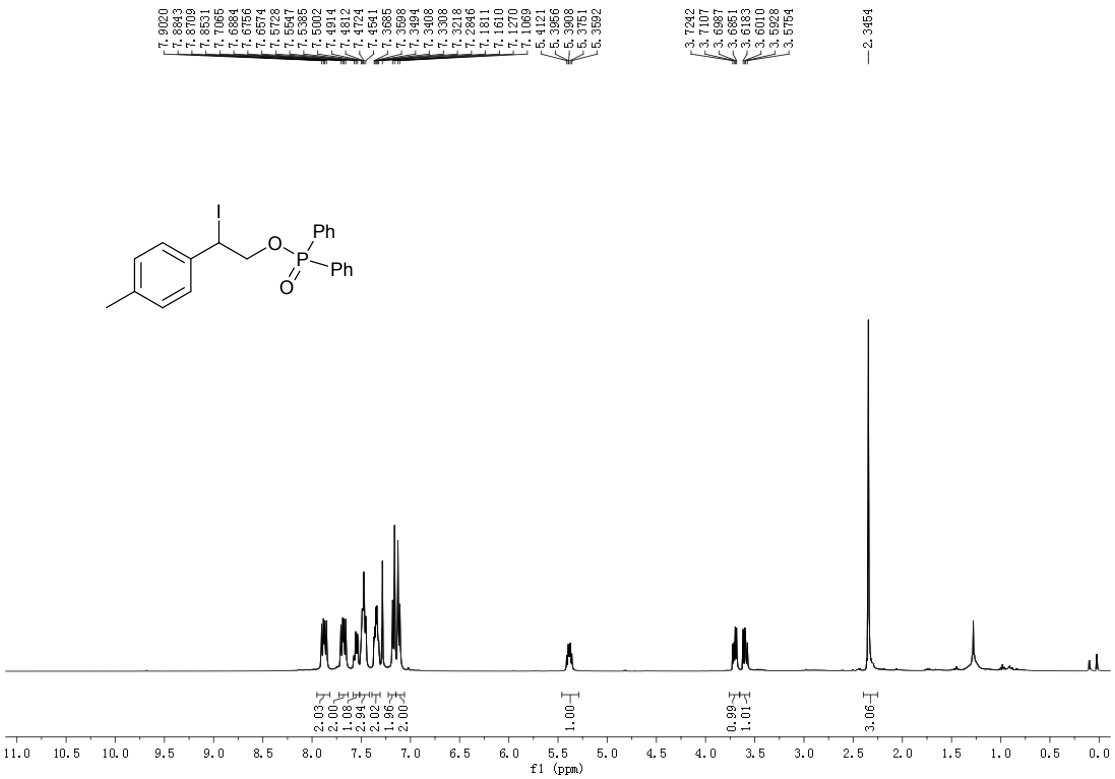
Compound **3ac** was obtained in 52% yield according to the general procedure (14h). ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.42-7.38 (m, 5H), 5.46-5.41 (m, 1H), 3.79 (d, J = 11.2 Hz, 3H), 3.61-3.50 (m, 5H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 138.3 (d, J = 2.9 Hz), 129.1, 128.7, 126.4, 79.5 (d, J = 5.0 Hz), 54.7 (d, J = 5.9 Hz), 54.3 (d, J = 6.0 Hz), 9.0 (d, J = 8.1 Hz); ^{31}P NMR (162 MHz, $CDCl_3$): δ -0.01; HRMS calc. for $C_{10}H_{14}O_4PINa$ $(M+Na)^+$, 378.9572; found, 378.9574.

diethyl 2-iodo-2-phenylethyl phosphate 

Compound **3ad** was obtained in 51% yield according to the general procedure (14h). ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.41-7.38 (m, 5H), 5.45-5.40 (m, 1H), 4.22-4.08 (m, 2H), 3.95-3.89 (m, 2H), 3.61-3.50 (m, 2H), 1.32 (dt, J_1 = 7.8 Hz, J_2 = 0.7 Hz, 3H), 1.16 (dt, J_1 = 7.8 Hz, J_2 = 0.7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 138.4 (d, J = 3.0 Hz), 129.0, 128.6, 126.5, 79.2 (d, J = 5.0 Hz), 64.2 (d, J = 5.8 Hz), 63.9 (d, J = 5.8 Hz), 16.1 (d, J = 7.1 Hz), 15.9 (d, J = 7.0 Hz), 9.3 (d, J = 8.1 Hz); ^{31}P NMR (162 MHz, $CDCl_3$): δ -2.42; HRMS calc. for $C_{12}H_{19}O_4PI$ $(M+H)^+$, 385.0066; found, 385.0066.

5. Copies of NMR spectra for 3aa−3ad

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7.4914
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7.3494
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5.4121
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3.6987
3.6851
3.6183
3.6010
3.5928
3.5764
2.3454
I
O
P
Ph
Ph
O
2.03
2.00
1.08
2.94
2.02
1.96
2.00
1.00
0.99
1.01
3.06
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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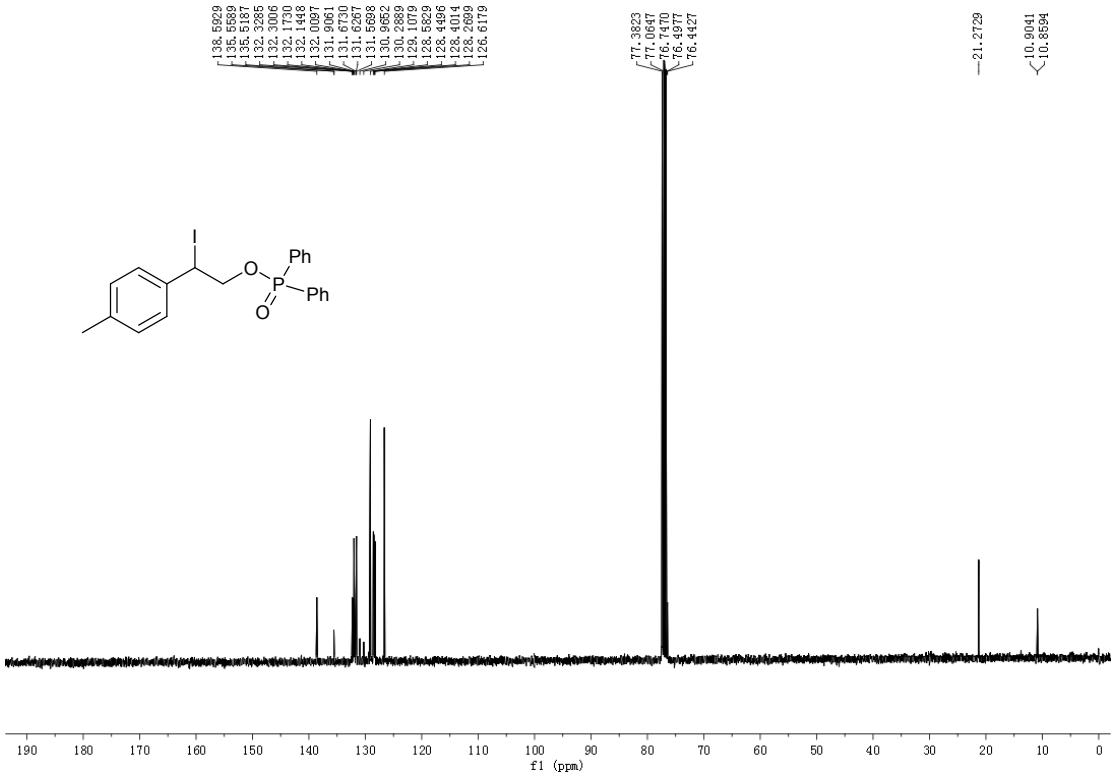
0.0
f1 (ppm)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I
O
P
Ph
Ph
O
190
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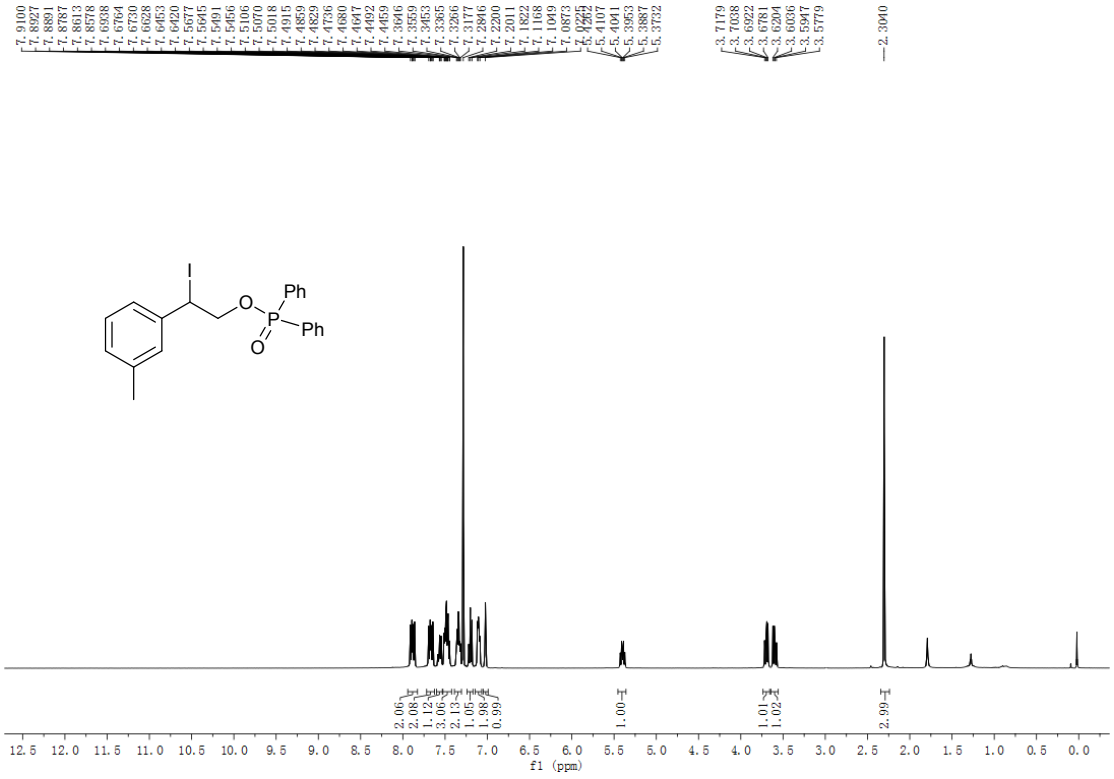
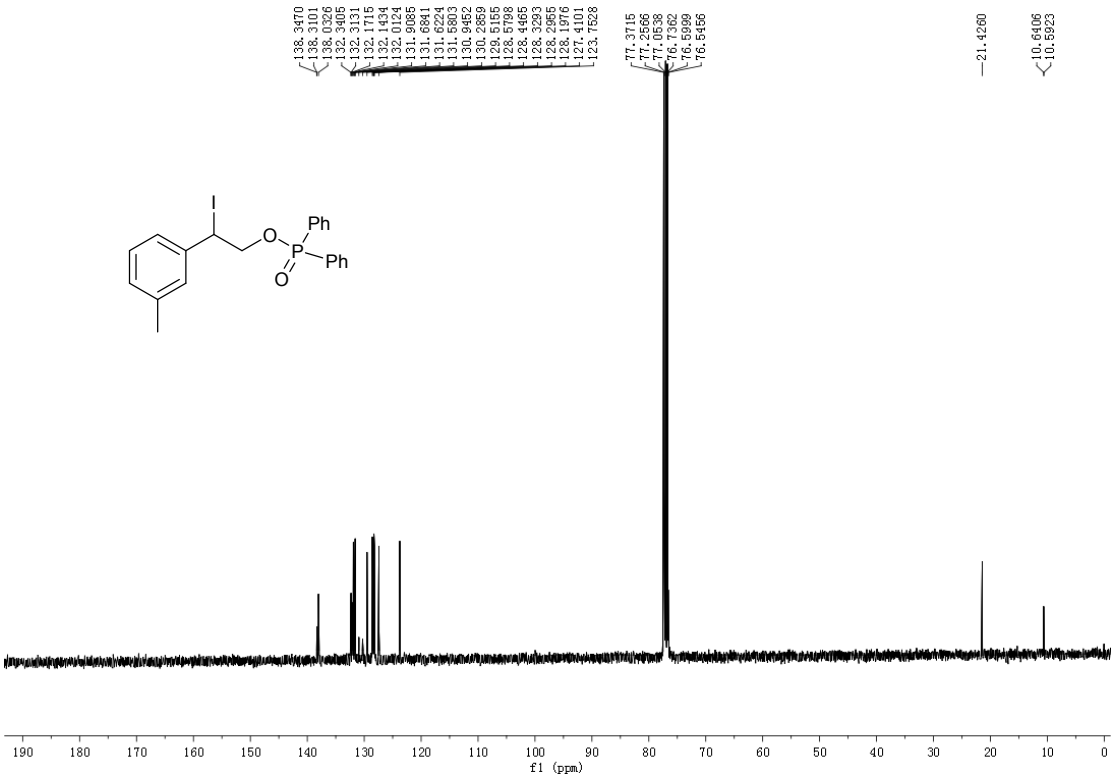
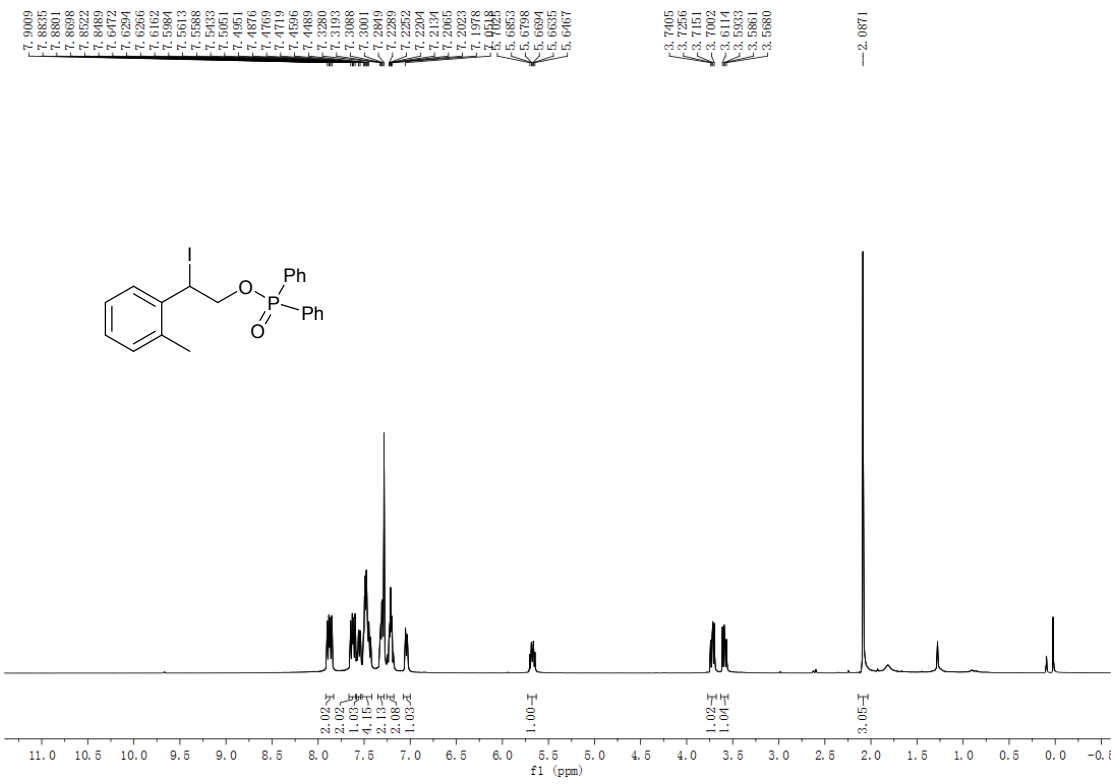
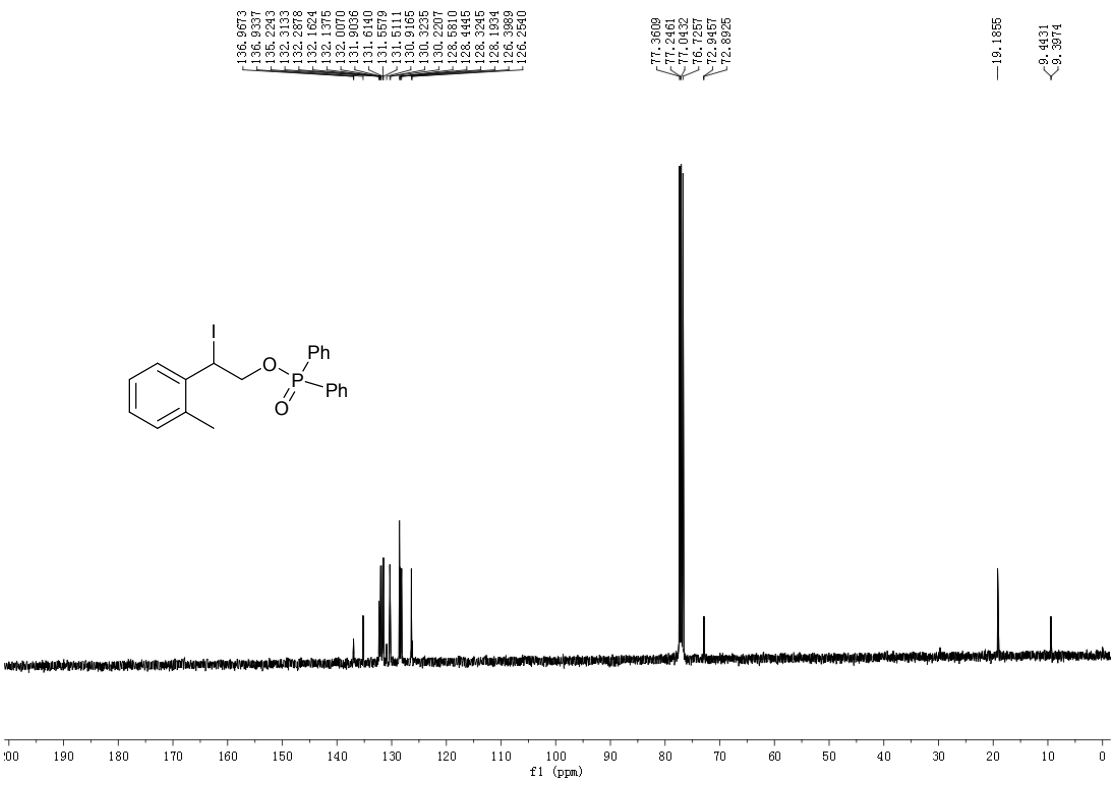
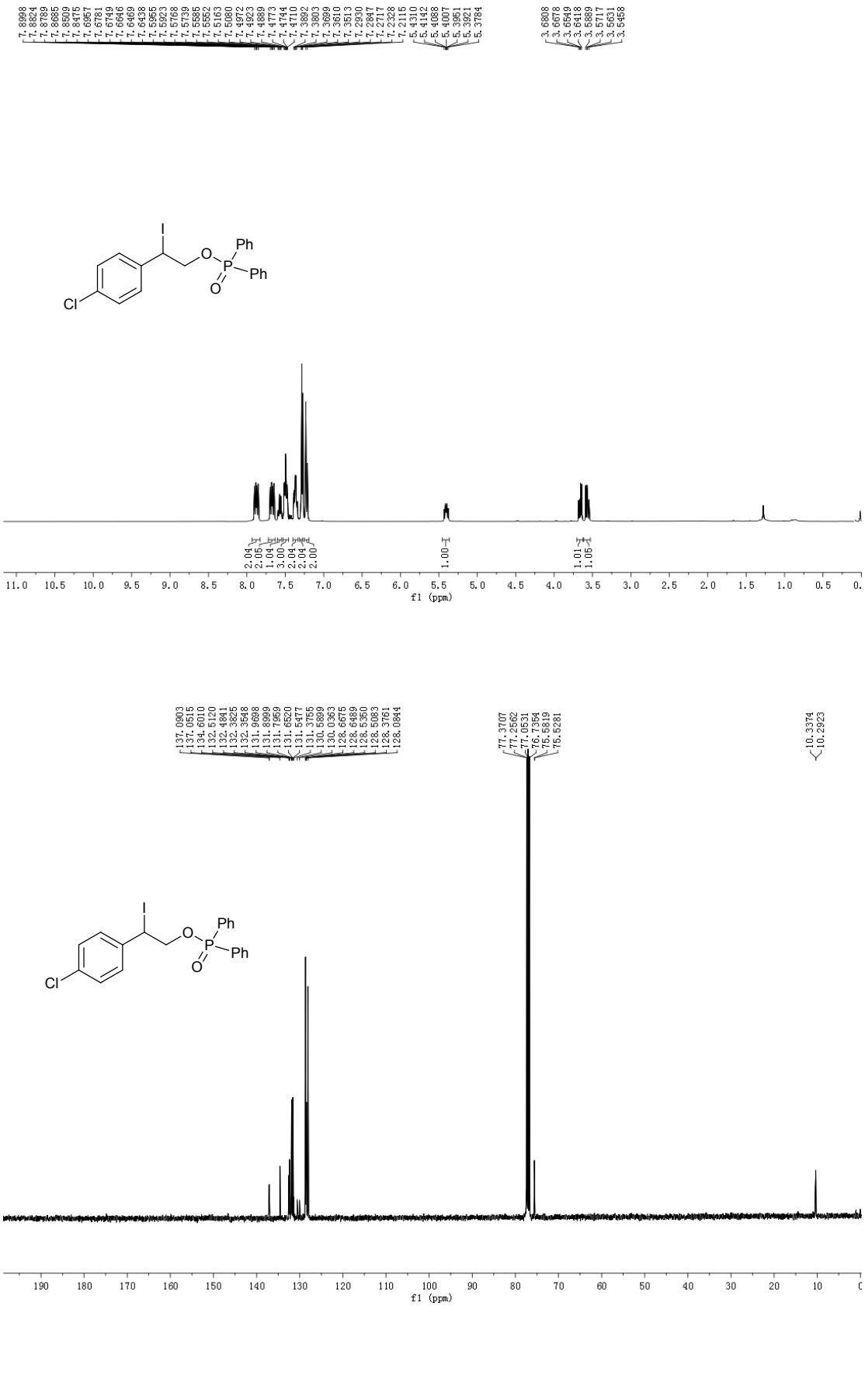
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

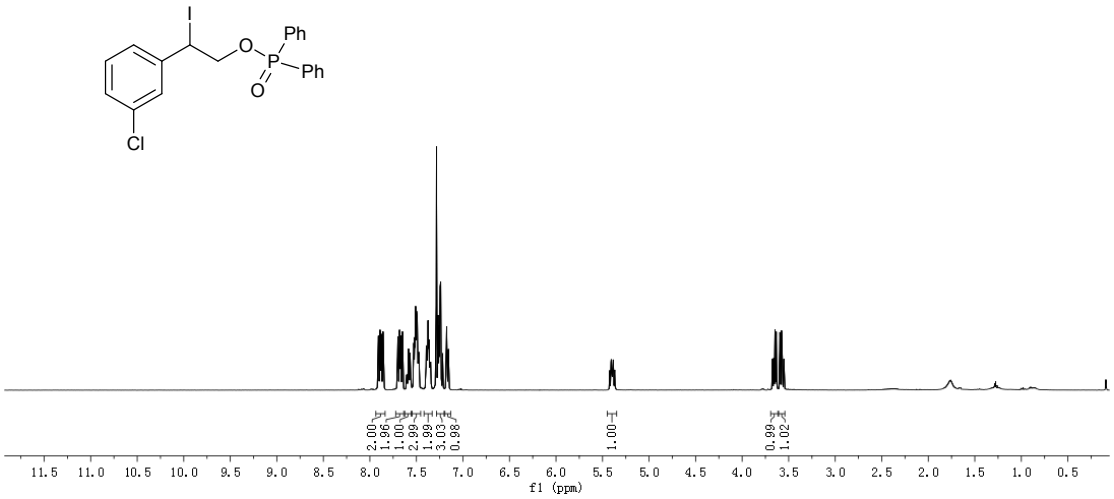

I
O
P
Ph
Ph
O
2.06
2.08
1.12
3.06
2.13
1.05
1.98
0.99
1.00
1.01
1.02
2.99
f1 (ppm)

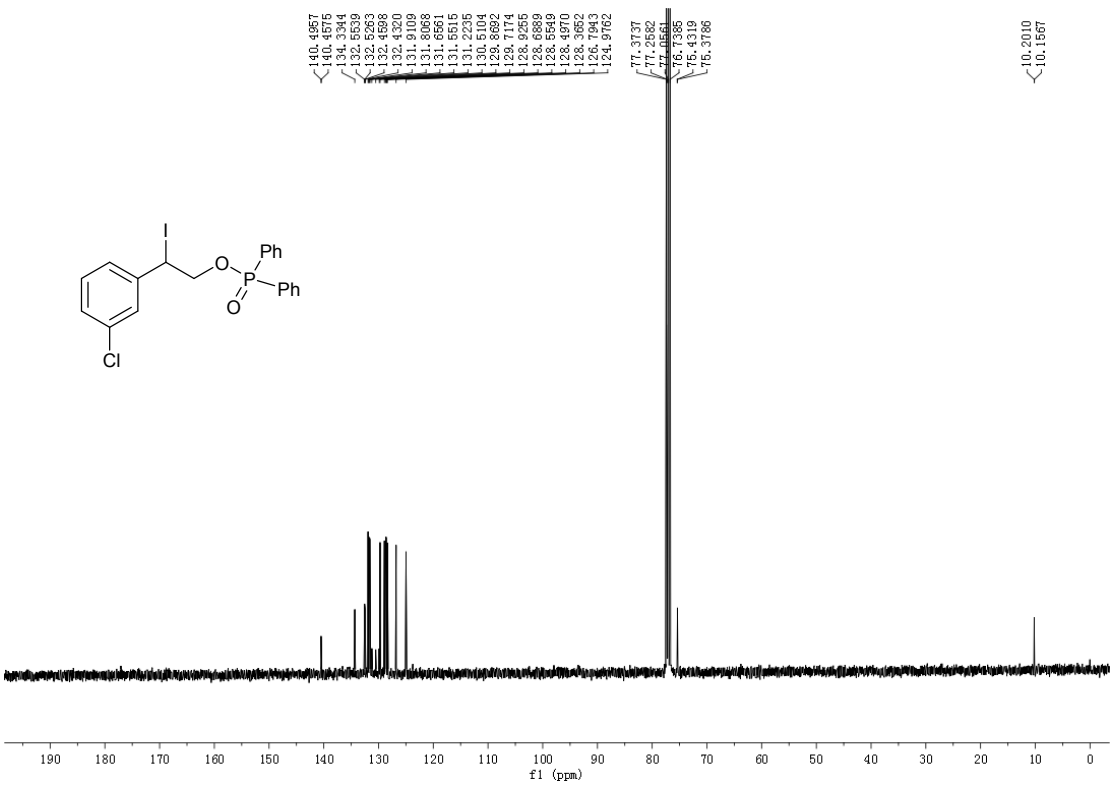

I
O
P
Ph
Ph
O
f1 (ppm)

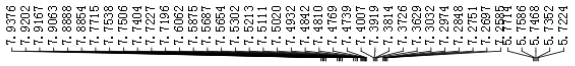

I
O
P
Ph
Ph
O
f1 (ppm)

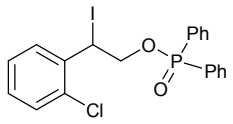

I
O
P
Ph
Ph
O
f1 (ppm)

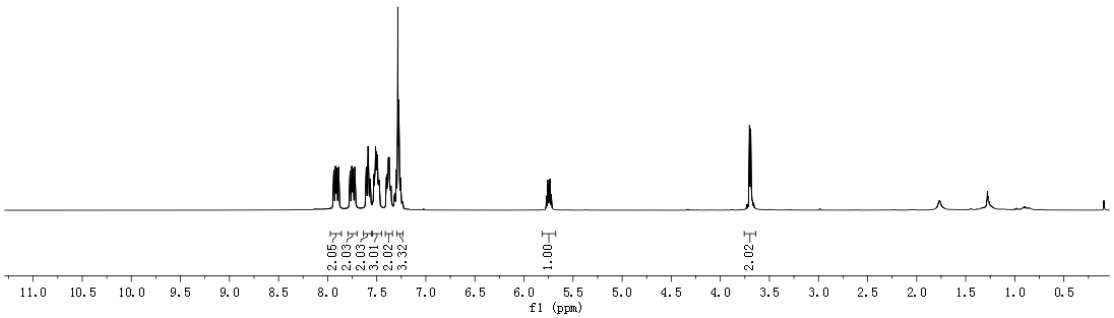

7.8898
7.8824
7.8789
7.8685
7.8659
7.8475
7.6957
7.6781
7.6749
7.6646
7.6469
7.6438
7.5865
7.5823
7.5768
7.5739
7.5585
7.5552
7.5163
7.5080
7.4972
7.4923
7.4889
7.4773
7.4744
7.4710
7.3892
7.3803
7.3699
7.3610
7.3513
7.2930
7.2847
7.2717
7.2328
7.2116
5.4310
5.4142
5.4088
5.4007
5.3951
5.3921
5.3784
3.6808
3.6678
3.6549
3.6418
3.6289
3.5717
3.5621
3.5456
I
O
P
Ph
Ph
O
Cl
2.04
2.05
1.04
3.00
2.04
2.04
2.00
1.00
1.01
1.05
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.
f1 (ppm)
137.0903
137.0515
134.6010
132.5120
132.4841
132.3825
132.3548
131.9698
131.8999
131.7969
131.6520
131.5477
131.3755
130.5889
130.0363
128.6675
128.6489
128.5360
128.5083
128.3761
128.0844
77.3707
77.2562
77.0531
76.7354
75.5819
75.5281
10.3374
10.2923
I
O
P
Ph
Ph
O
Cl
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

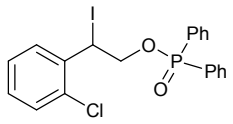

I
O
P
Ph
Ph
O
Cl
2.00
1.96
1.00
2.99
1.99
3.03
0.98
1.00
0.99
1.02
f1 (ppm)

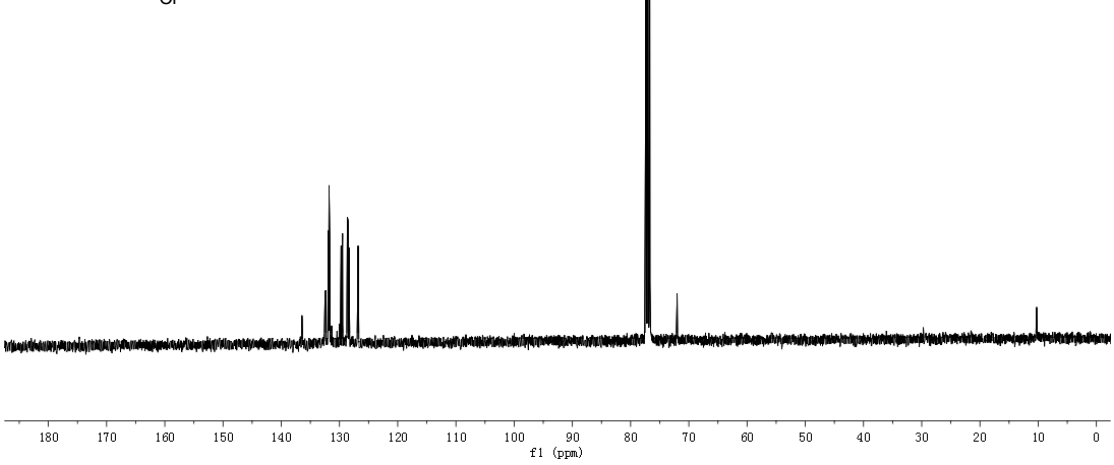

I
O
P
Ph
Ph
O
Cl
f1 (ppm)

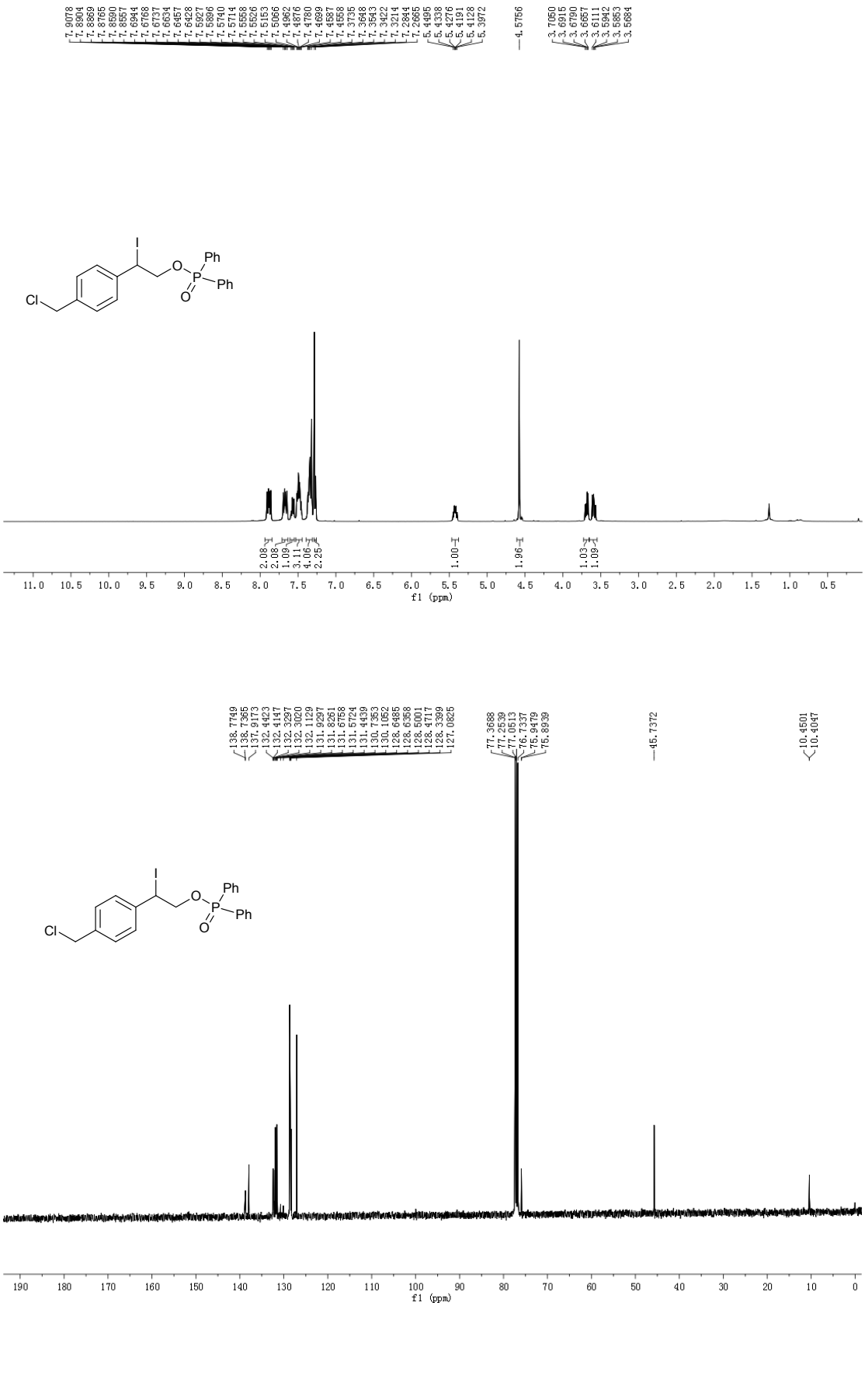


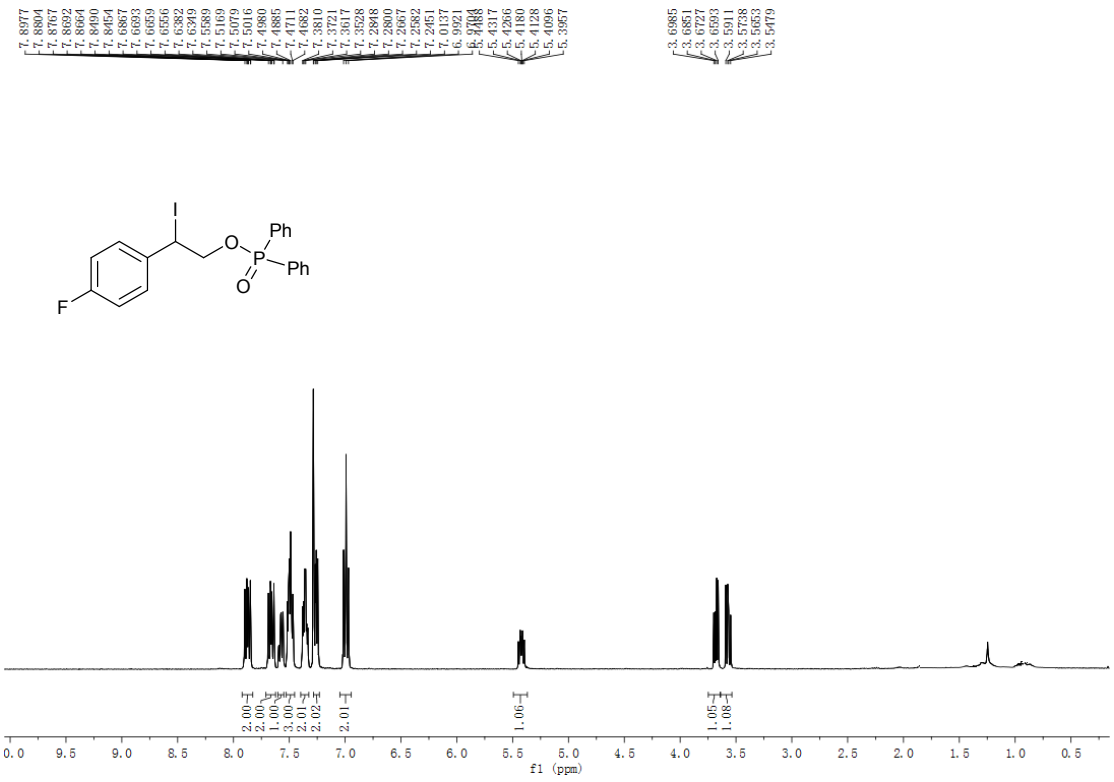

I
O
P
Ph
Ph
O
Cl

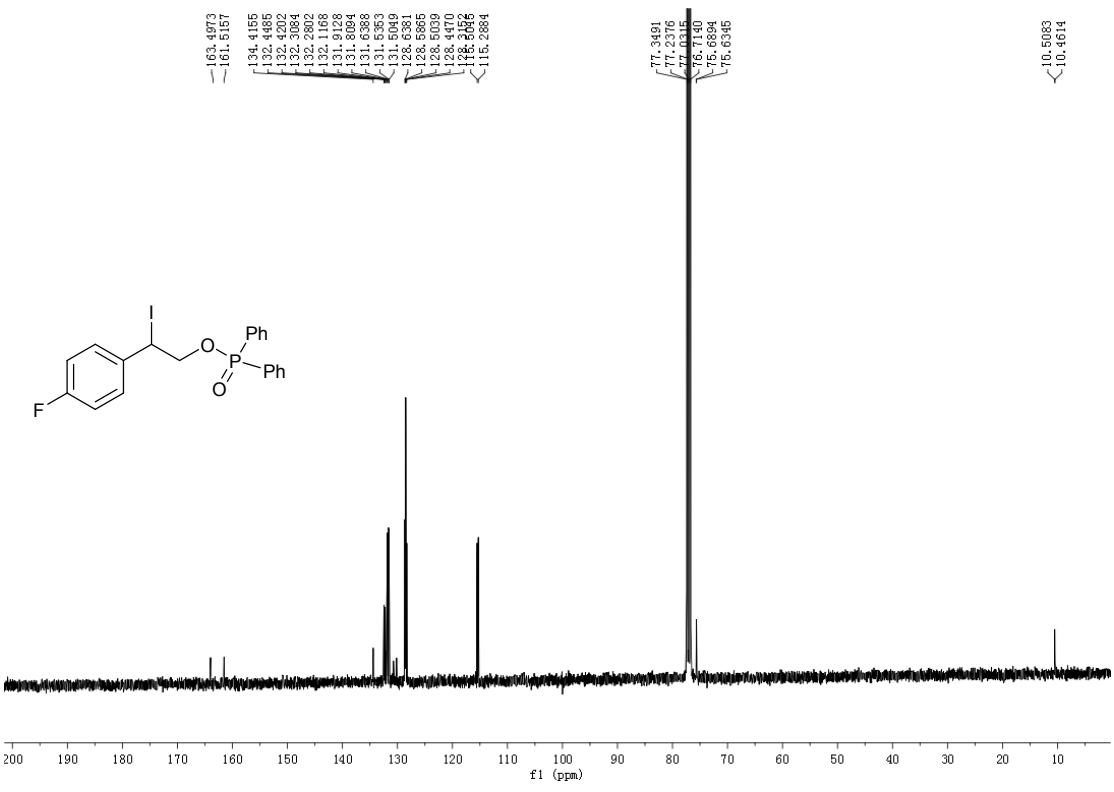

2.05
2.03
2.03
3.01
2.02
3.32
1.00
2.02
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

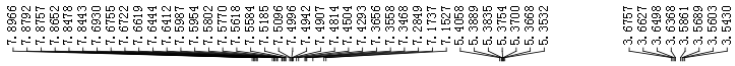

I
O
P
Ph
Ph
O
Cl


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


7.9078
7.8904
7.8869
7.8765
7.8690
7.8557
7.6944
7.6768
7.6737
7.6634
7.6457
7.6428
7.5927
7.5896
7.5740
7.5714
7.5558
7.5526
7.5153
7.5066
7.4962
7.4876
7.4780
7.4689
7.4587
7.4558
7.3755
7.3648
7.3543
7.3422
7.3214
7.2844
7.2665
5.4496
5.4338
5.4276
5.4191
5.4128
5.3972
4.5756
3.7050
3.6915
3.6790
3.6657
3.6111
3.5942
3.5853
3.5684
I
O
P
Ph
Ph
O
Cl
2.08
2.08
1.09
3.11
4.06
2.25
1.00
1.96
1.03
1.09
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
f1 (ppm)
138.7749
138.7365
137.9173
132.4423
132.4147
132.3297
132.3020
132.1129
131.9297
131.8261
131.6758
131.5724
131.4439
130.7363
130.1052
128.6485
128.6358
128.5001
128.4717
128.3399
127.0825
77.3688
77.2539
77.1513
76.7337
76.9479
75.8939
45.7372
10.4501
10.4047
I
O
P
Ph
Ph
O
Cl
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


I
O
Ph
P
Ph
O
F
f1 (ppm)


I
O
Ph
P
Ph
O
F
f1 (ppm)


7.8966
7.8792
7.8757
7.8652
7.8478
7.8443
7.6930
7.6755
7.6722
7.6619
7.6444
7.6412
7.5987
7.5954
7.5802
7.5770
7.5618
7.5584
7.5185
7.5096
7.4996
7.4942
7.4907
7.4814
7.4504
7.4293
7.3666
7.3568
7.3468
7.2849
7.1737
7.1527
5.4068
5.3889
5.3835
5.3754
5.3700
5.3668
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3.6757
3.6627
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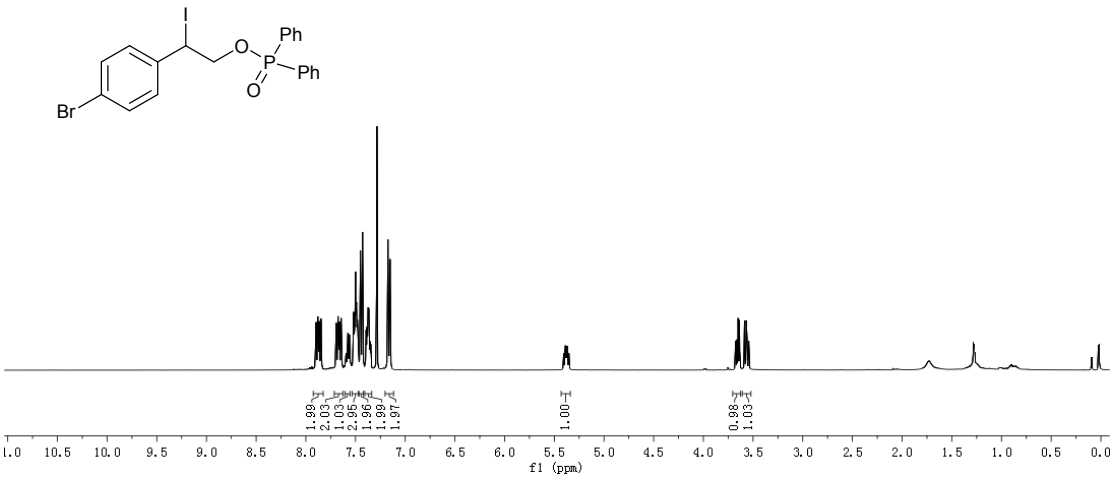
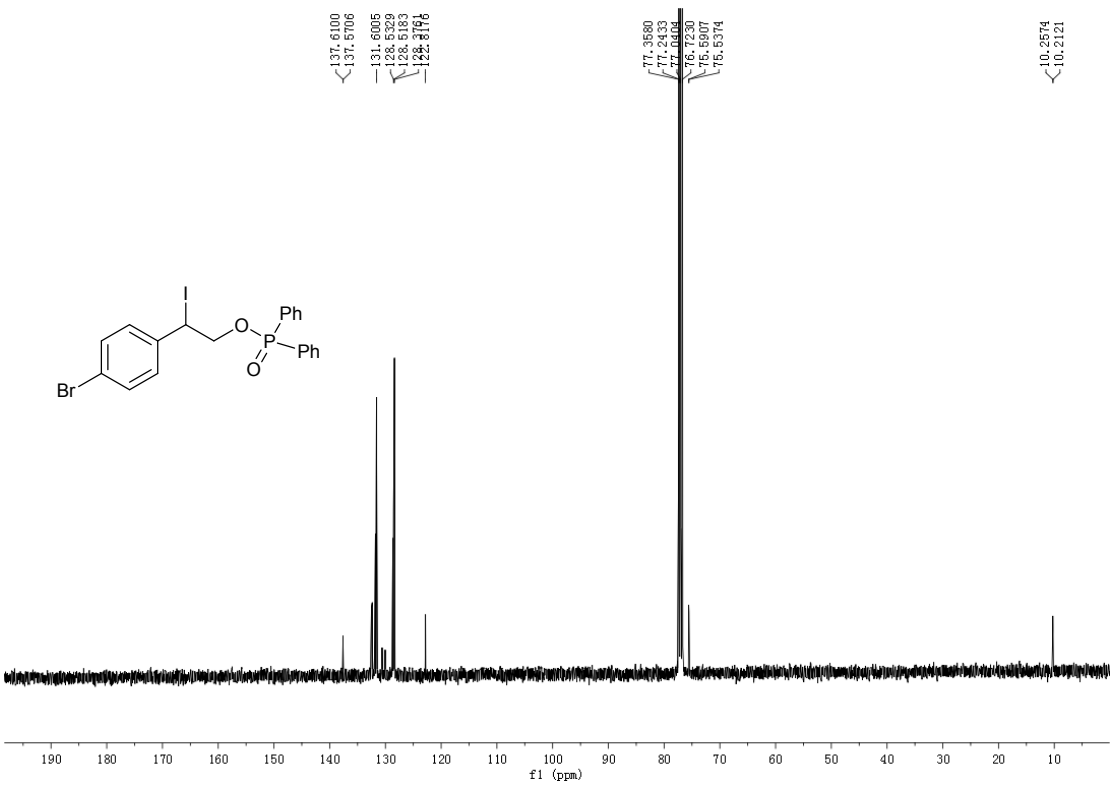
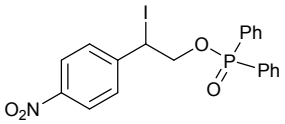
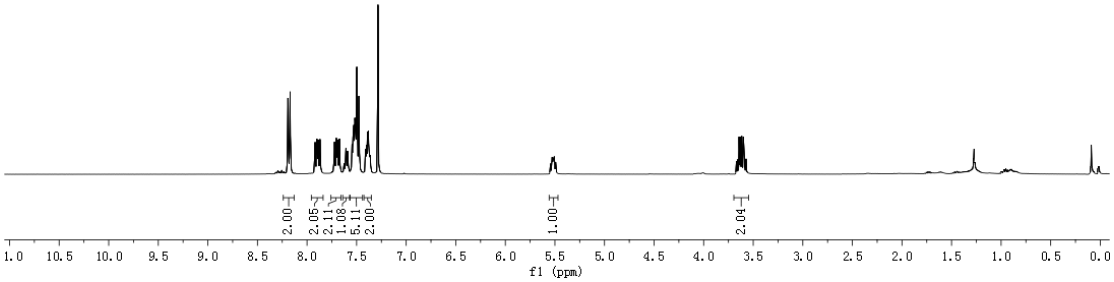
3.6368
3.5861
3.5689
3.5603
3.5430

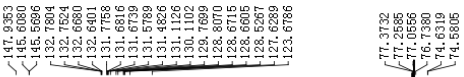

I
O
P
Ph
Ph
O
Br
1.99
2.03
1.03
2.96
1.96
1.99
1.97
1.00
0.98
1.03
f1 (ppm)

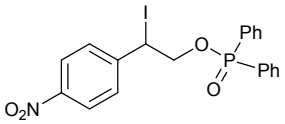

137.6100
137.5706
131.6005
128.5329
128.5183
77.3580
77.2433
77.0286
76.7230
75.5907
75.5374
10.2574
10.2121
I
O
P
Ph
Ph
O
Br
f1 (ppm)

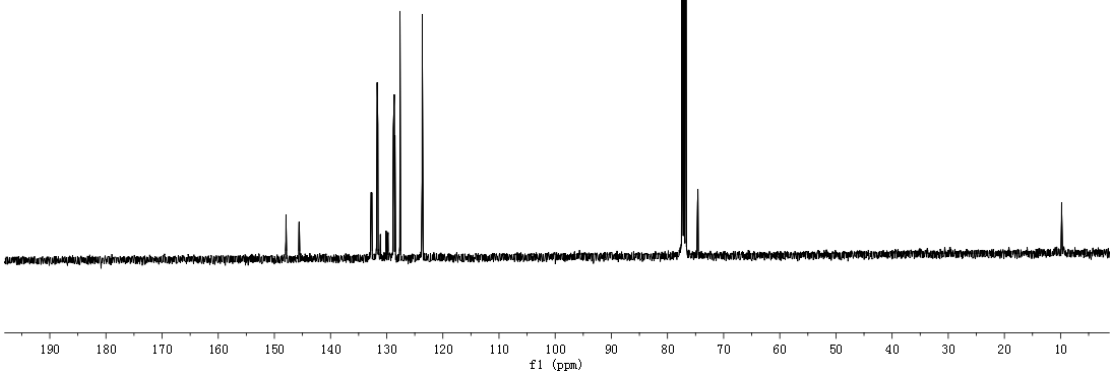

8.1924
8.1707
7.9184
7.9007
7.8976
7.8869
7.8691
7.7226
7.7047
7.6914
7.6735
7.6260
7.6076
7.5918
7.5371
7.5274
7.5175
7.4980
7.4764
7.4092
7.4003
7.3900
7.3811
7.3713
7.3622
7.2848
5.5434
5.5279
5.5207
5.5150
5.5060
5.4923
3.6691
3.6568
3.6426
3.6304
3.6158
3.5996
3.5895
3.5731


I
O
Ph
P
Ph
O
O2N


2.00
2.05
2.11
1.08
5.11
2.00
1.00
2.04
1.0 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
f1 (ppm)


147.9353
145.6080
145.5696
132.7804
132.7524
132.6680
132.6401
131.7758
131.6816
131.6739
131.5789
131.4826
131.1126
130.1102
129.7699
128.8070
128.6715
128.6605
128.5267
127.6289
123.6786
77.3732
77.2585
77.0556
76.7380
74.6319
74.5805
8.8173
8.7705


I
O
Ph
P
Ph
O
O2N


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


7.9037
7.8860
7.8830
7.8724
7.8545
7.7035
7.6855
7.6728
7.6544
7.6189
7.5980
7.5768
7.5314
7.5240
7.5119
7.5060
7.4942
7.4910
7.4875
7.4225
7.4018
7.3927
7.3820
7.3732
7.3633
7.3542
7.2843
5.4793
5.4643
5.4568
5.4509
5.4428
5.4283
3.6308
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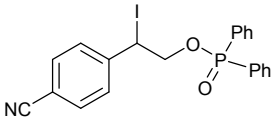
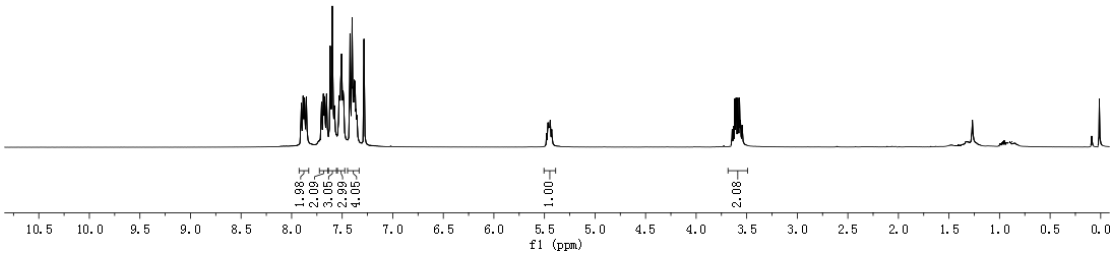
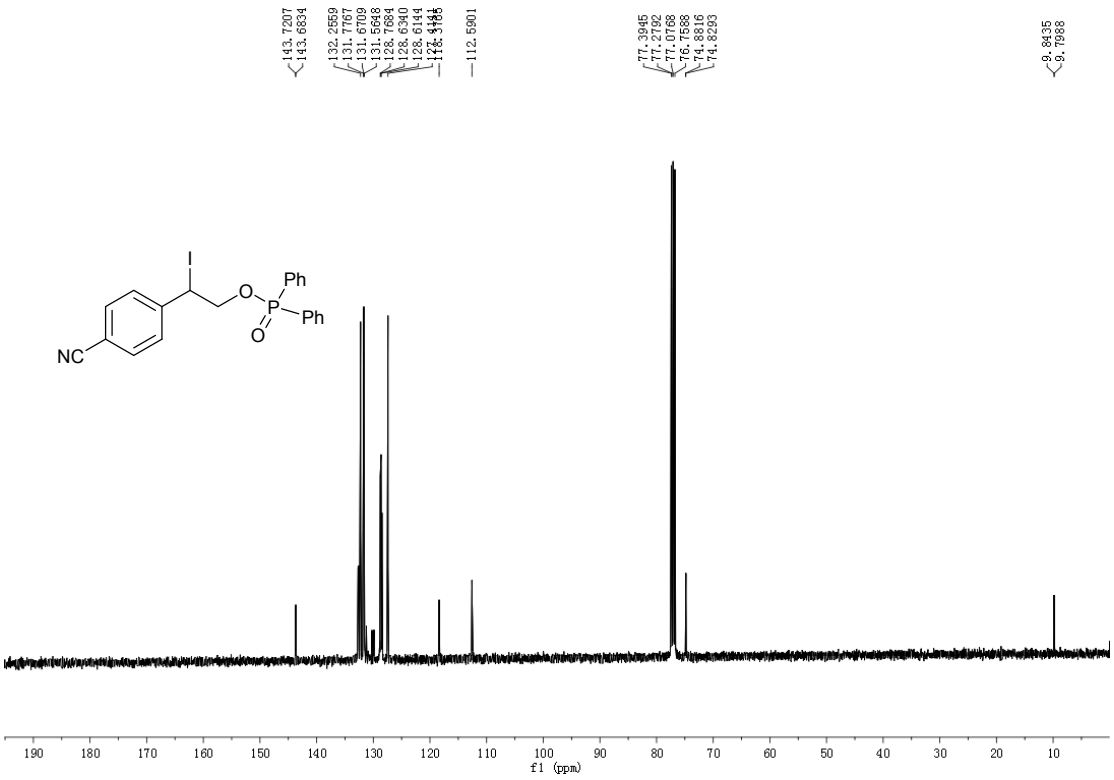
3.6045
3.5876
3.5713
3.5614
3.5450


I
O
P
Ph
Ph
O
NC


1.98
2.09
3.05
2.99
4.05
1.00
2.08
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


143.7207
143.6834
132.2559
131.7767
131.6709
131.5648
128.7684
128.6340
128.6144
112.5901
77.3945
77.2792
77.0768
76.7588
74.8816
74.8293
9.8435
9.7988
I
O
P
Ph
Ph
O
NC
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


7.9413
7.9240
7.9204
7.9100
7.8926
7.8891
7.8499
7.8390
7.8322
7.8113
7.8006
7.7873
7.7771
7.7062
7.6884
7.6576
7.6545
7.5745
7.5714
7.5561
7.5527
7.5205
7.5179
7.5150
7.5077
7.5009
7.4964
7.4841
7.4691
7.4650
7.4378
7.4337
7.3988
7.3961
7.2965
7.2841
7.2762
7.2674
5.6258
5.6210
5.6115
5.6066
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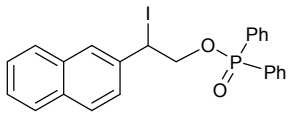
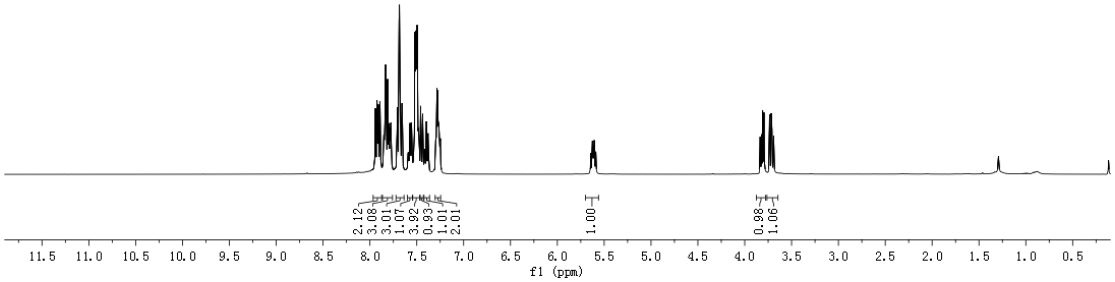
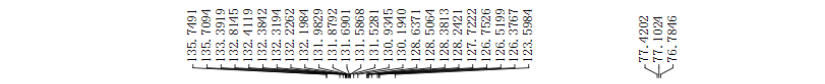
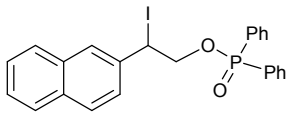
3.8345
3.8210
3.8087
3.7953
3.7349
3.7173
3.7092
3.6916

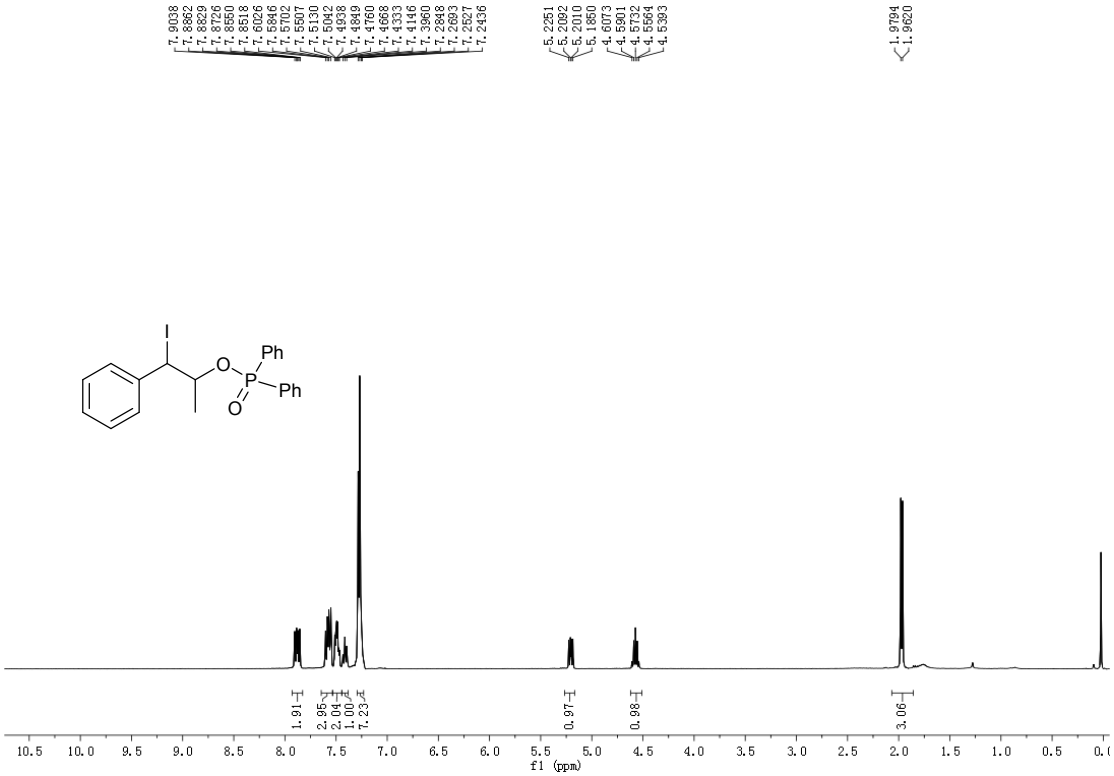

I
O
P
Ph
Ph
O


2.12
3.08
3.01
1.07
3.92
0.93
1.01
2.01
1.00
0.98
1.06
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
f1 (ppm)


135.7491
135.7094
133.3919
132.8145
132.4119
132.3842
132.3194
132.2262
132.1984
131.9829
131.8792
131.6901
131.5868
131.5281
130.9345
130.1940
128.6371
128.5064
128.3813
128.2421
127.7222
126.7526
126.5199
126.3767
123.5984
77.4202
77.1024
76.7846
10.4637
10.4205


I
O
P
Ph
Ph
O

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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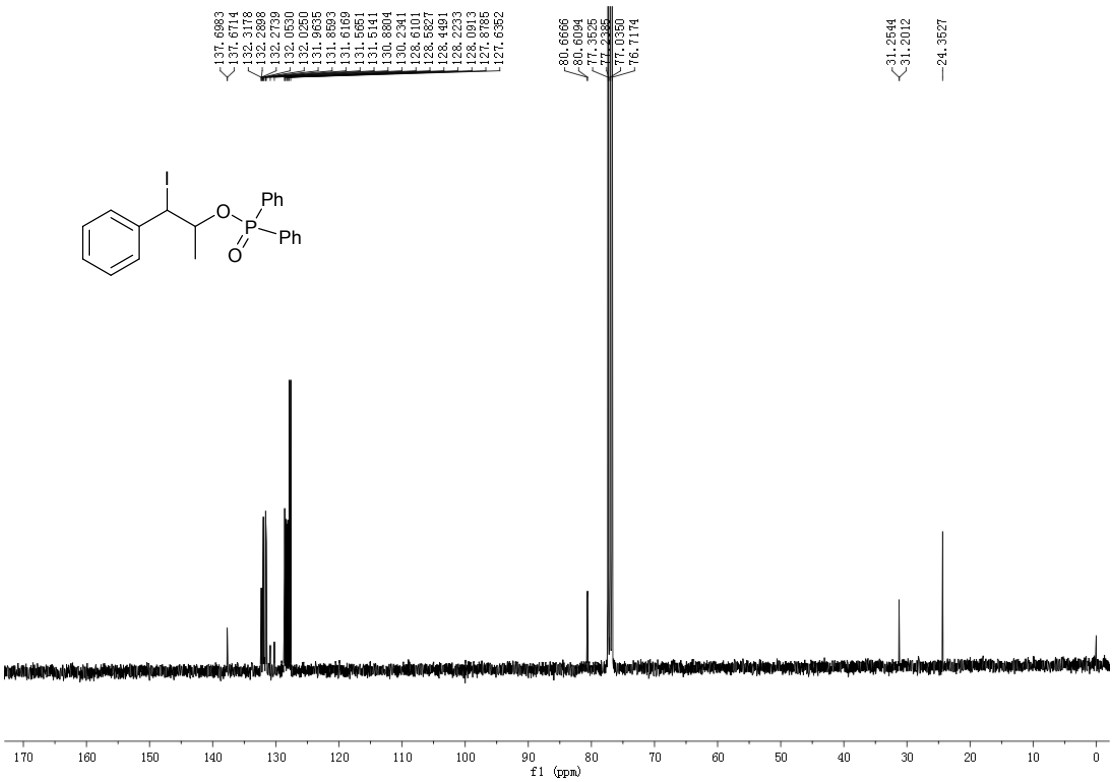
7.5130
7.5042
7.4938
7.4849
7.4760
7.4668
7.4333
7.4146
7.3960
7.2848
7.2693
7.2527
7.2436
5.2251
5.2092
5.2010
5.1850
4.6073
4.5901
4.5732
4.5564
4.5393
1.9794
1.9620
I
O
P
O
Ph
Ph
1.91
2.95
2.04
1.00
7.23
0.97
0.98
3.06
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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131.5141
130.8804
130.2341
128.6101
128.5827
128.4491
128.2233
128.0913
127.8785
127.6352
80.6666
80.6094
77.3525
77.2365
77.0350
76.7174
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31.2012
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I
O
P
O
Ph
Ph
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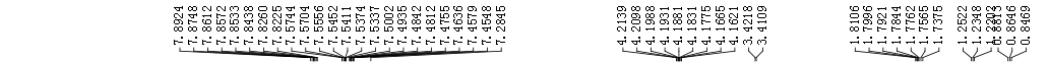
f1 (ppm)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7.5452
7.5411
7.5374
7.5337
7.5002
7.4935
7.4842
7.4812
7.4755
7.4636
7.4579
7.4548
7.2845
4.2139
4.2098
4.1988
4.1931
4.1881
4.1831
4.1775
4.1665
4.1621
3.4218
3.4109
1.8106
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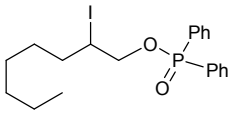
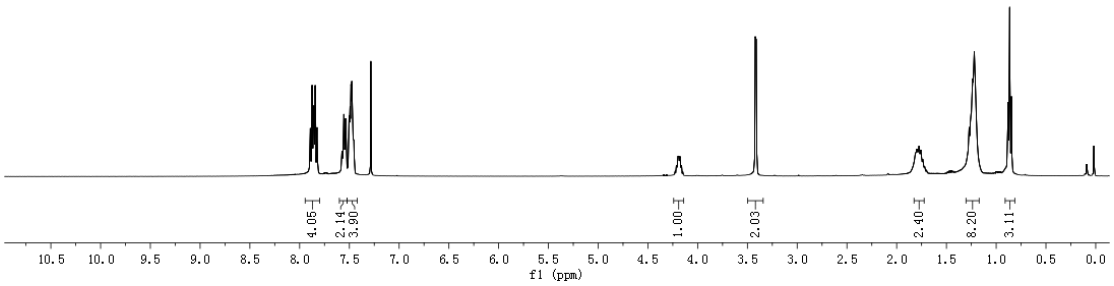
0.8469


I
O
P
Ph
Ph
O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2.14
3.90
1.00
2.03
2.40
8.20
3.11
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


132.2978
132.2746
131.8246
131.7130
131.6079
128.6010
128.5721
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77.3726
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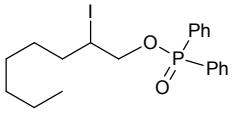
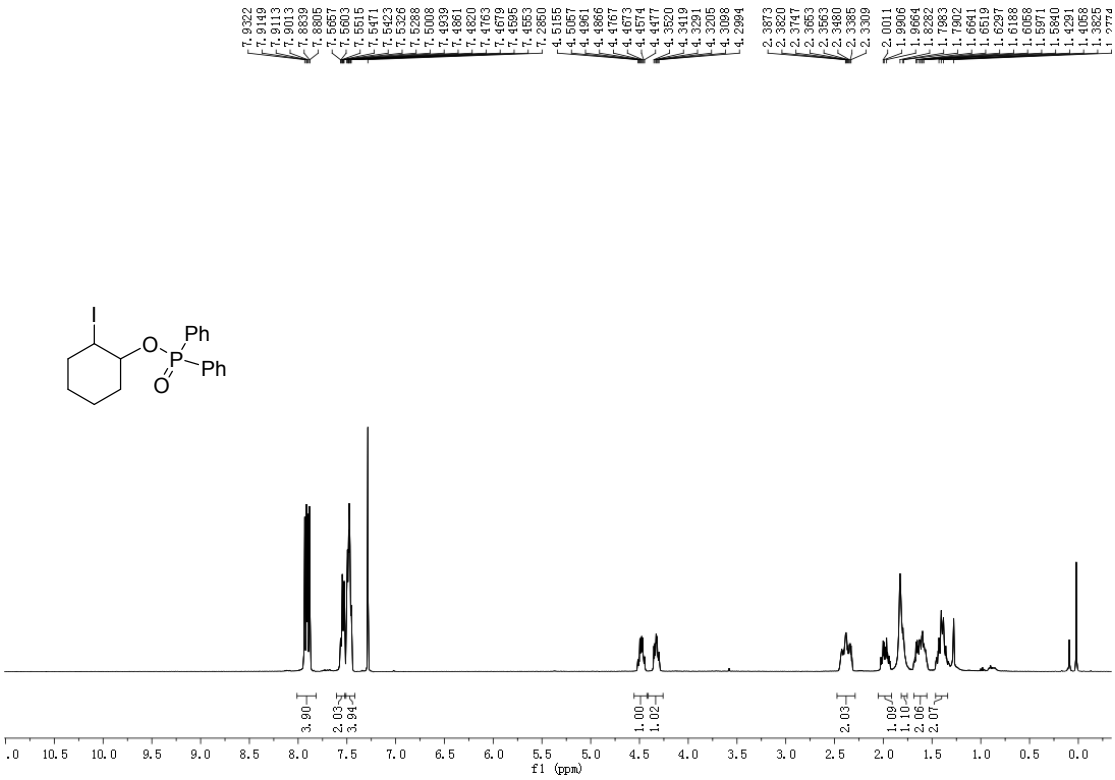

I
O
P
Ph
Ph
O

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


I
O
P
Ph
Ph
O
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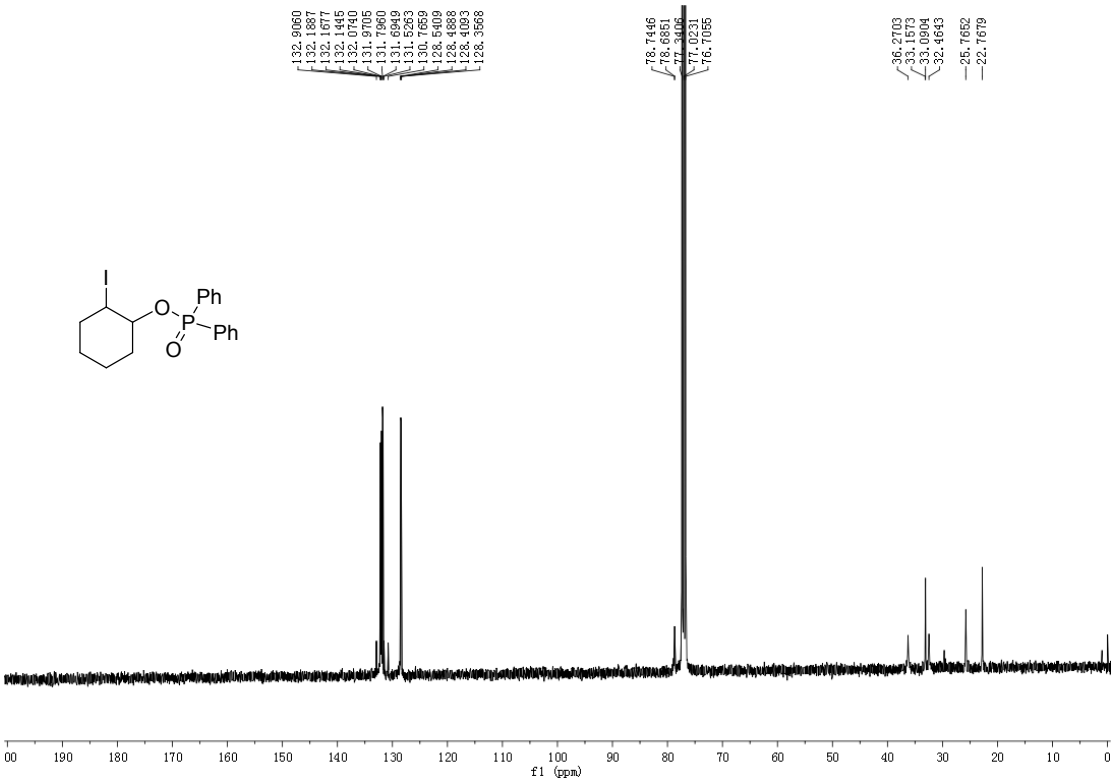
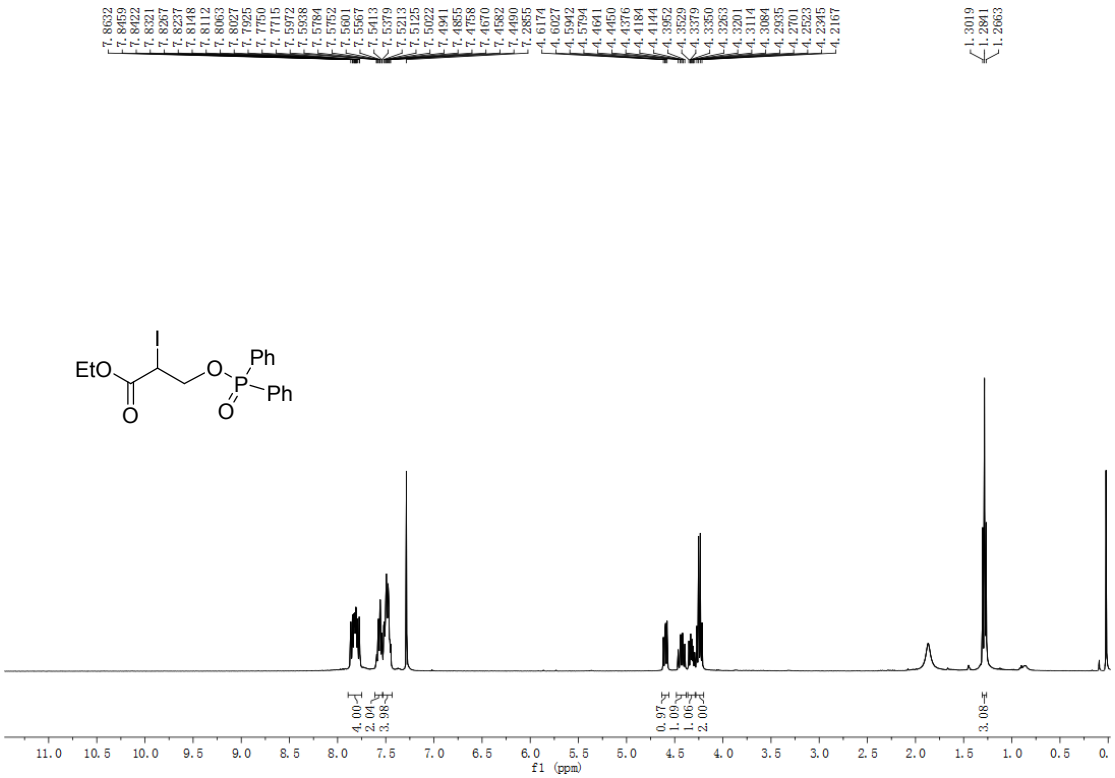
2.03
3.94
1.00
1.02
2.03
1.09
1.10
2.06
2.07
f1 (ppm)


I
O
P
Ph
Ph
O
f1 (ppm)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7.8027
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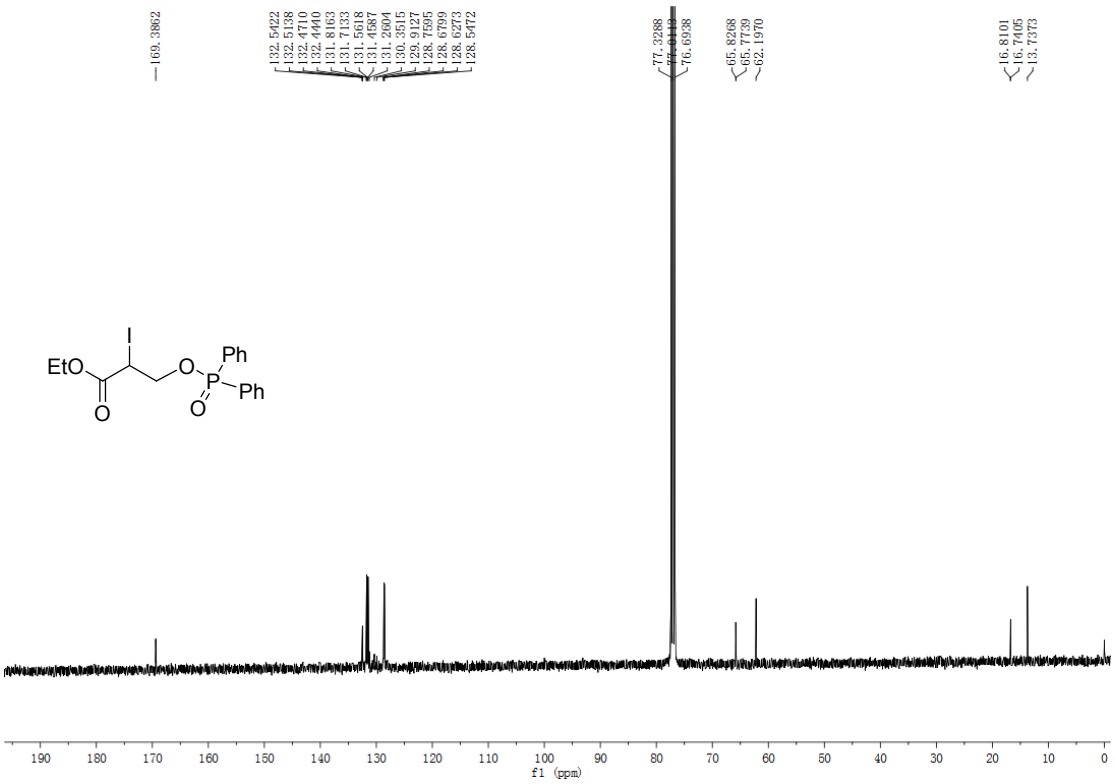
7.7750
7.7715
7.5972
7.5938
7.5784
7.5752
7.5601
7.5567
7.5413
7.5379
7.5213
7.5125
7.5022
7.4941
7.4855
7.4758
7.4670
7.4582
7.4490
7.2855
4.6174
4.6027
4.5942
4.5794
4.4641
4.4450
4.4376
4.4184
4.4144
4.3952
4.3529
4.3379
4.3350
4.3263
4.3201
4.3114
4.3084
4.2935
4.2701
4.2523
4.2345
4.2167
1.3019
1.2841
1.2663
EtO
O
I
O
P
Ph
Ph
O
4.00
2.04
3.98
0.97
1.09
1.06
2.00
3.08
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.
f1 (ppm)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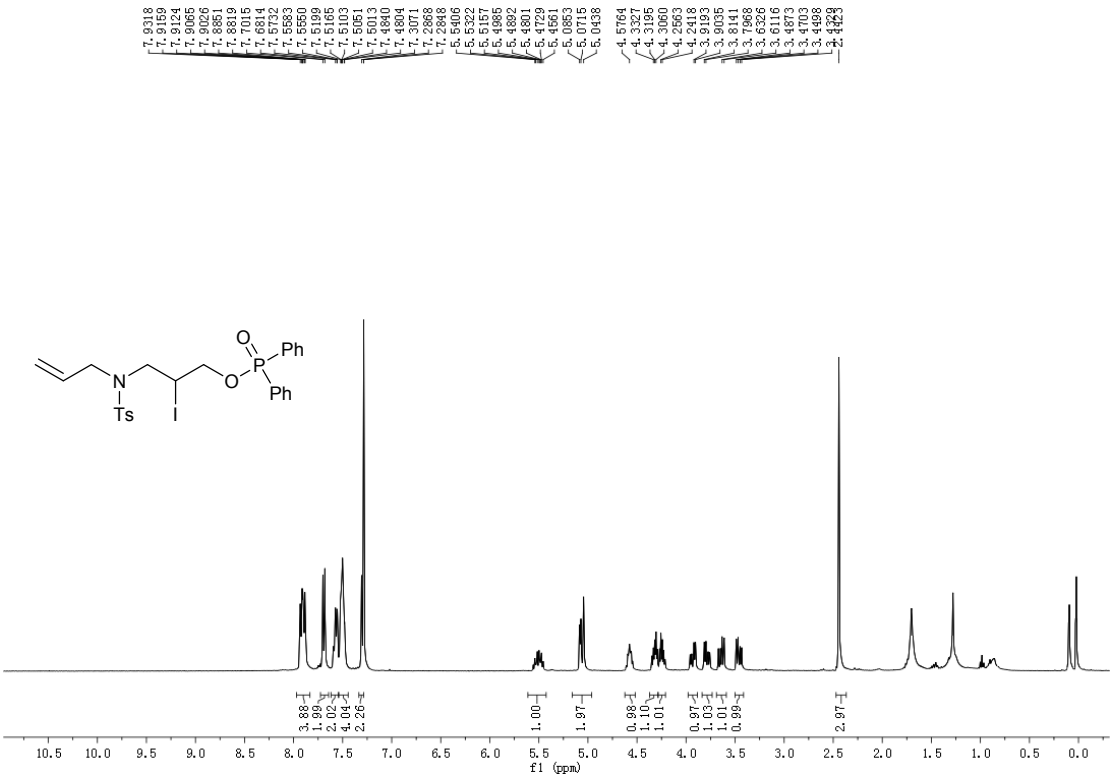
131.4587
131.2604
130.3515
129.9127
128.7595
128.6799
128.6273
128.5472
77.3288
77.0110
76.6938
65.8268
65.7739
62.1970
16.8101
16.7405
13.7373
EtO
O
I
O
P
Ph
Ph
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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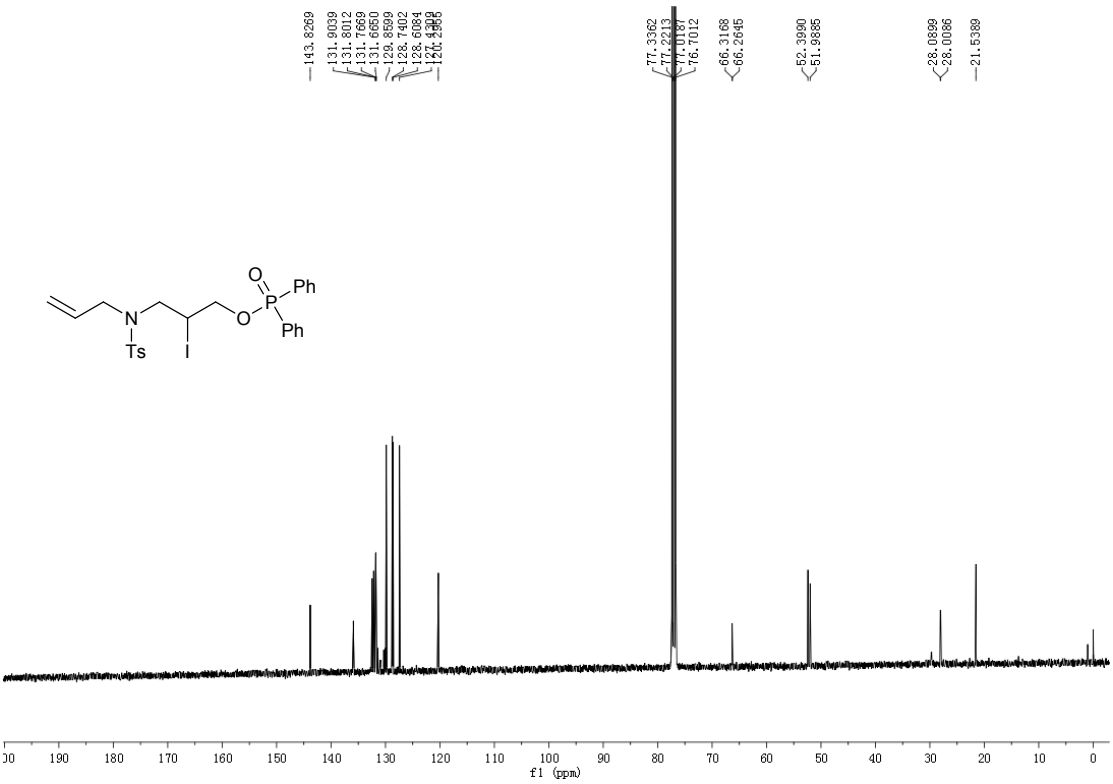
7.5683
7.5550
7.5199
7.5165
7.5103
7.5051
7.5013
7.4840
7.4804
7.3071
7.2868
7.2848
5.5406
5.5322
5.5157
5.4985
5.4892
5.4801
5.4729
5.4561
5.0853
5.0715
5.0438
4.5764
4.3327
4.3195
4.3060
4.2563
4.2418
3.9193
3.9036
3.8141
3.7968
3.6326
3.6116
3.4873
3.4703
3.4498
2.4423
O
P
Ph
Ph
N
Ts
O
I
3.88
1.99
2.02
4.04
2.26
1.00
1.97
0.98
1.10
1.01
0.97
1.03
1.01
0.99
2.97
f1 (ppm)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76.7012
66.3168
66.2645
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28.0086
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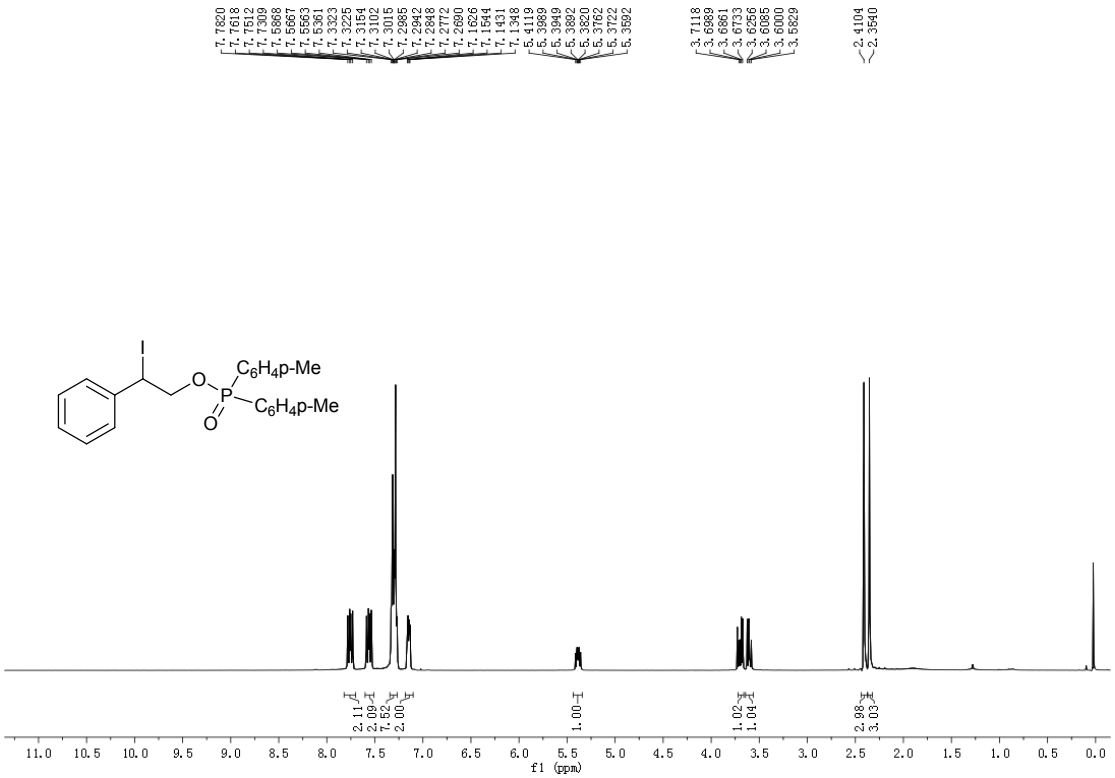
O
P
Ph
Ph
N
Ts
O
I
f1 (ppm)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7.5563
7.5361
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7.3225
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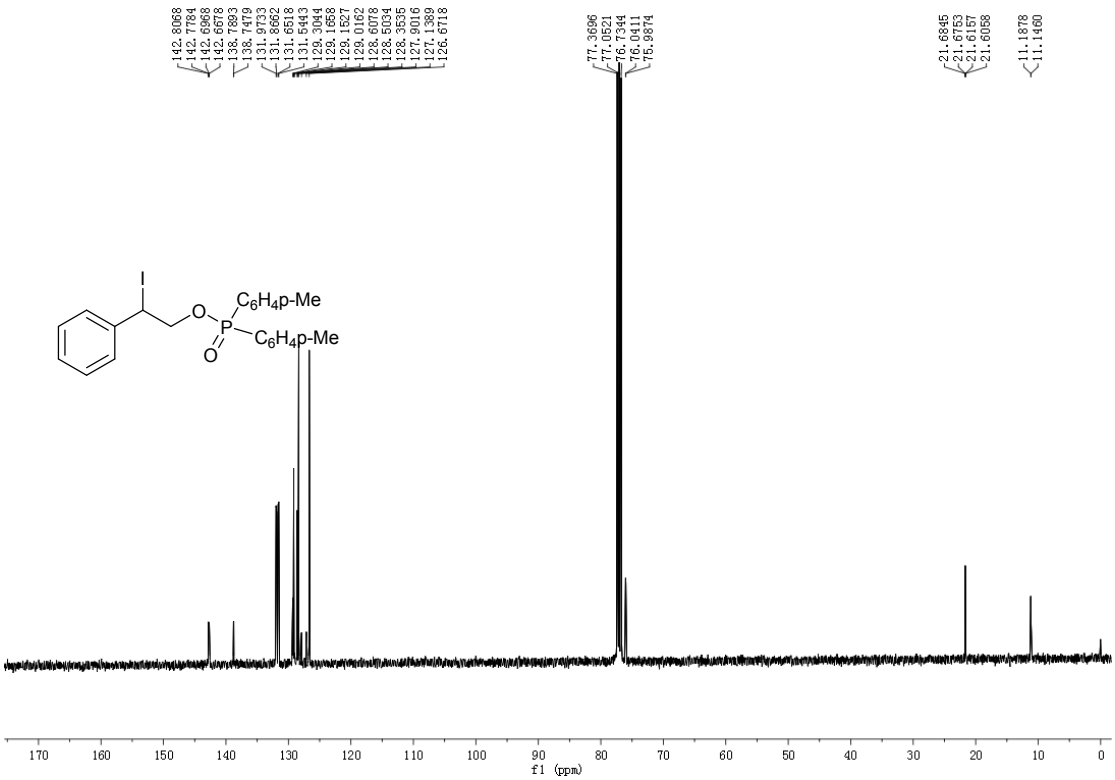
7.3102
7.3015
7.2985
7.2942
7.2848
7.2772
7.2690
7.1626
7.1544
7.1431
7.1348
5.4119
5.3989
5.3949
5.3892
5.3820
5.3762
5.3722
5.3692
3.7118
3.6989
3.6861
3.6733
3.6256
3.6086
3.6000
3.5829
2.4104
2.3540
I
O
P
O
C6H4p-Me
C6H4p-Me
2.11
2.09
7.52
2.00
1.00
1.02
1.04
2.98
3.03
11.0 10.5 10.0 9.5 9.0 8.5 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
f1 (ppm)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128.6078
128.5034
128.3535
127.9016
127.1389
126.6718
77.3696
77.0521
76.7344
76.0411
75.9874
21.6845
21.6753
21.6157
21.6058
11.1878
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O
P
O
C6H4p-Me
C6H4p-Me
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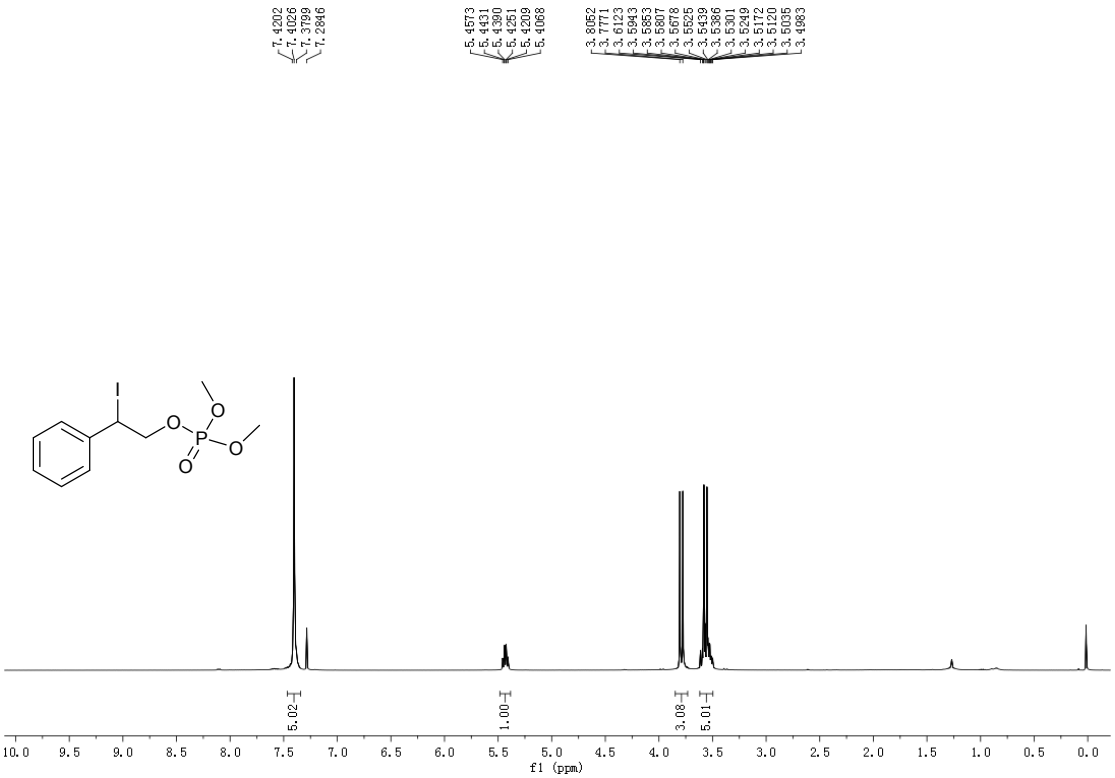
f1 (ppm)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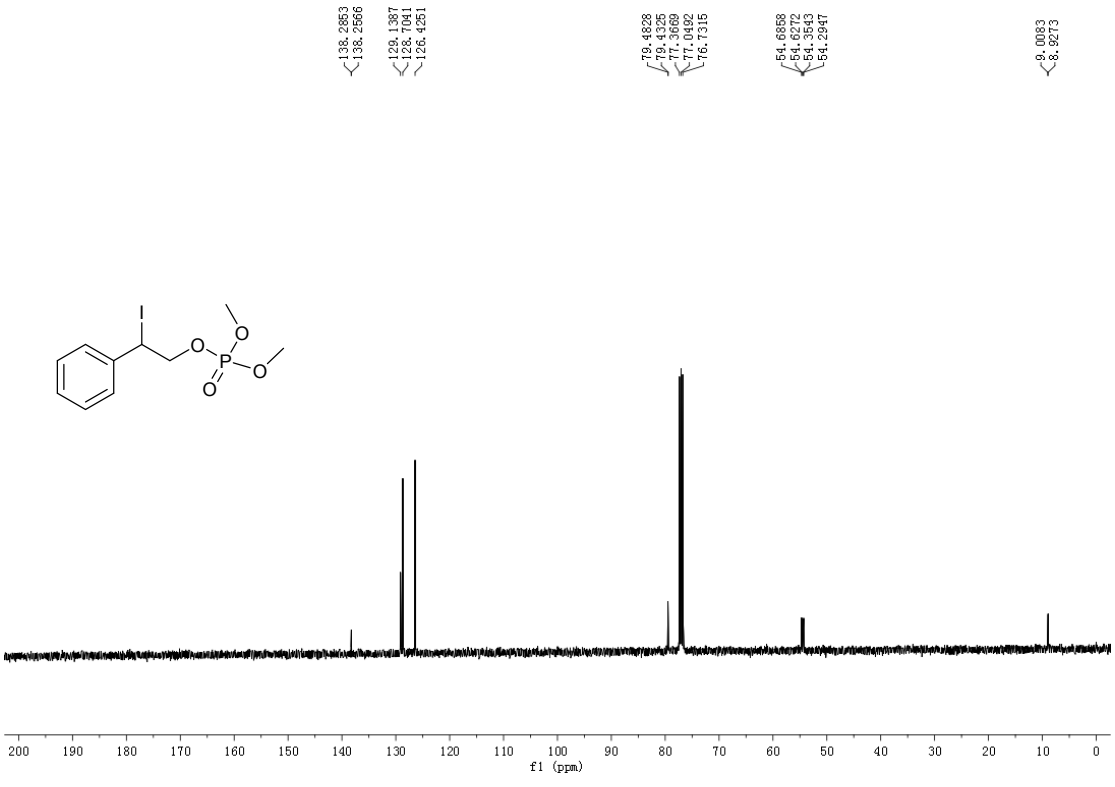
3.7771
3.6423
3.5943
3.5883
3.5807
3.5678
3.5525
3.5438
3.5386
3.5301
3.5249
3.5172
3.5120
3.5055
3.4983
I
O
O
P
O
O
5.02
1.00
3.08
5.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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77.0492
76.7315
54.6858
54.6272
54.3543
54.2947
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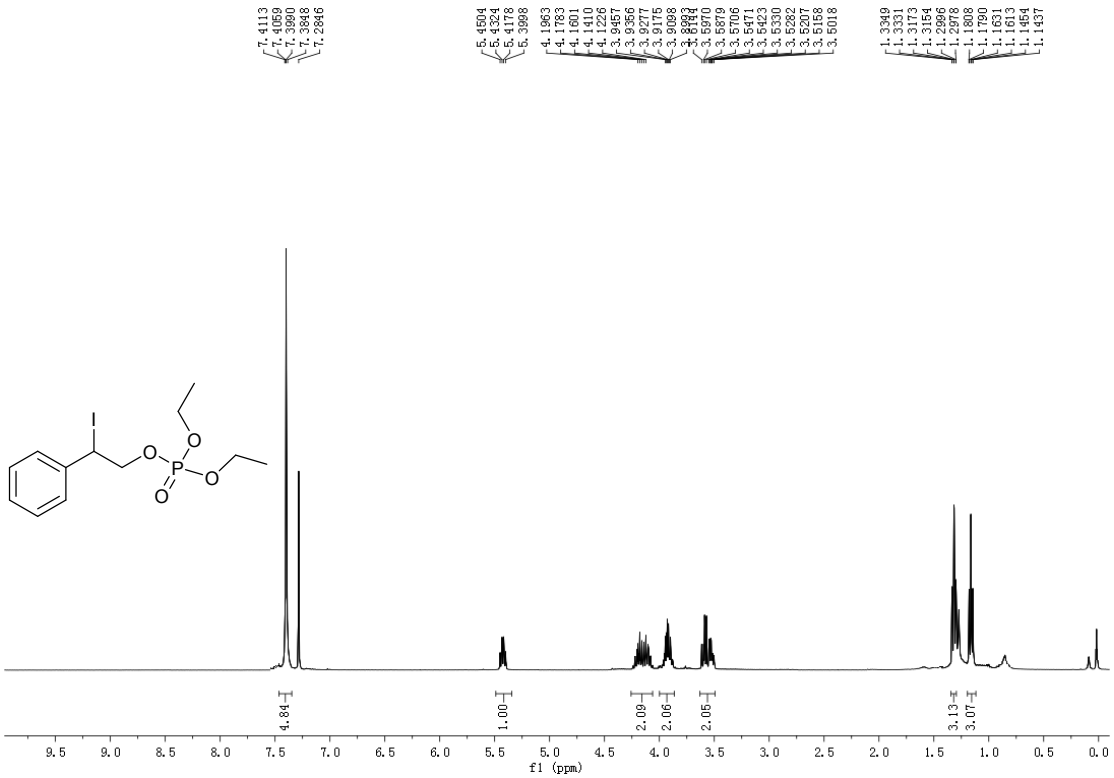
I
O
O
P
O
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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4.1410
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3.9457
3.9356
3.9277
3.9175
3.9008
3.8883
3.8744
3.5970
3.5879
3.5706
3.5471
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3.5207
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3.5018
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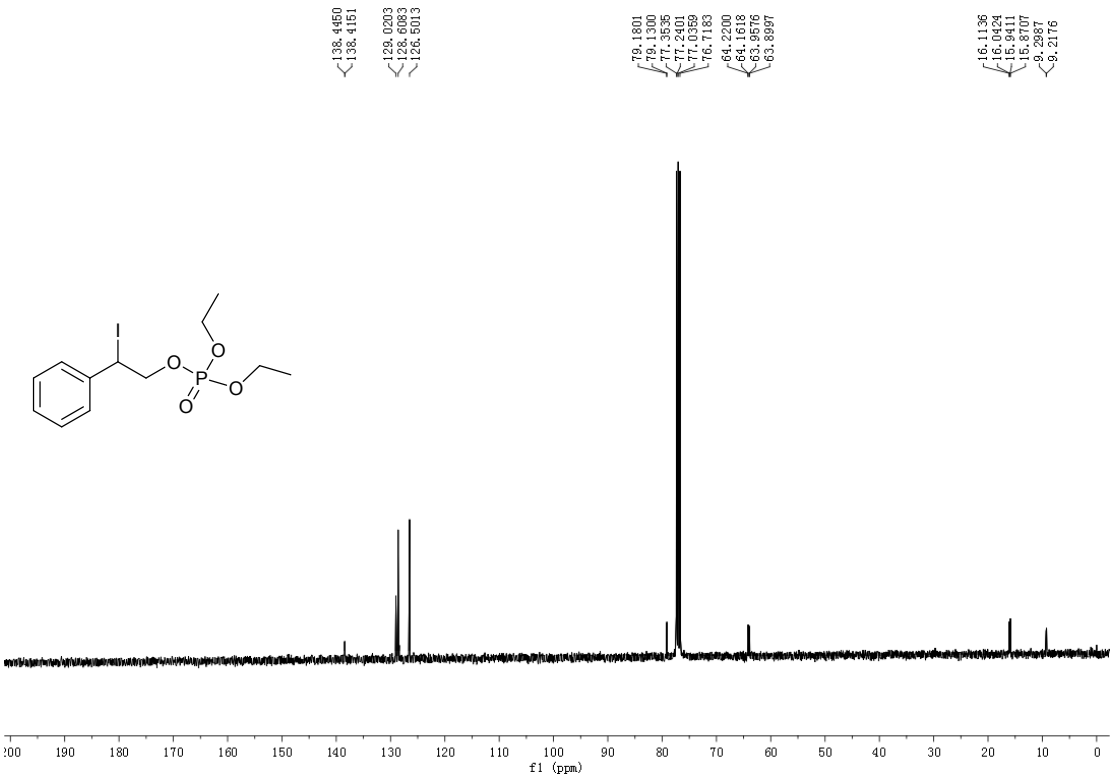
1.3331
1.3173
1.3154
1.2996
1.2978
1.1808
1.1790
1.1631
1.1613
1.1454
1.1437
I
O
O
P
O
O
O
4.84
1.00
2.09
2.06
2.05
3.13
3.07
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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64.2200
64.1618
63.9576
63.8997
16.1136
16.0424
15.9411
15.8707
9.2987
9.2176
I
O
O
P
O
O
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)