Electronic Supplementary Information

for

$Mn(OAc)_3$–Mediated Phosphonation–Lactonization of Alkenoic Acids: Synthesis of Phosphono-γ-butyrolactones

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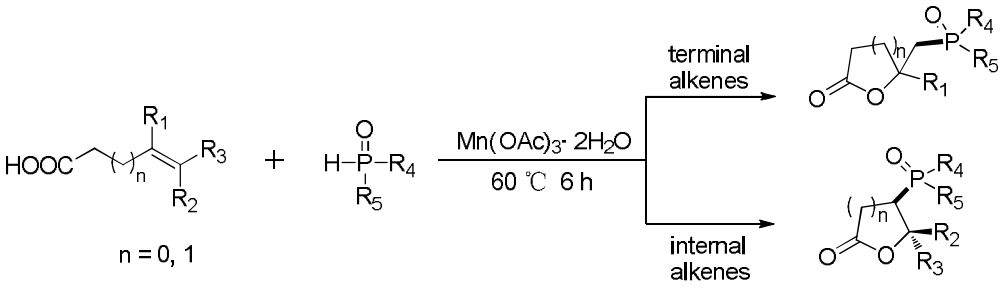
1. General:

All reactions were carried out under N_2. Unless otherwise noted, all reagents were obtained from commercial suppliers and used without further purification. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were measured on Bruker AVⅢ 400M spectrometers with $CDCl_3$ as solvent and tetramethylsilane (TMS) as internal standard or 85% H_3PO_4 as external standard for ^{31}P NMR (162 MHz). Chemical shifts were reported in units (ppm) by assigning TMS resonance in the ^{1}H spectrum as 0.00 ppm and $CDCl_3$ resonance in the ^{13}C spectrum as 77.23 ppm. All coupling constants (*J* values) were reported in Hertz (Hz). Chemical shifts of common trace ^{1}H NMR impurities (ppm): H_2O: 1.56, $CHCl_3$: 7.26, DMSO: 2.50. Column chromatography was performed on silica gel 300-400 mesh. The CAS number of the known compound was listed. The unknown products were further characterized by HRMS-ESI.

2. Experimental procedure for the synthesis of $Mn(OAc)_3\cdot 2H_2O$

The $Mn(OAc)_3\cdot 2H_2O$ was prepared by heating a mixture of 125 mL of acetic acid and 12 g of $Mn(OAc)_2\cdot 4H_2O$ to reflux for 20 min, then slowly adding 2.0 g of $KMnO_4$. After refluxing for an additional 30 min, the mixture was cooled to room temperature and add a mixture of 20 mL of water and 20 mL of acetic anhydride. The manganic acetate was filtered off after 2 h, washed with cold acetic acid and diethyl ether, and then air dried.

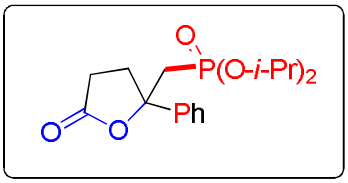
3. Experimental procedure for the phosphonylation of alkenoic acids



An oven-dried Schlenk tube containing $Mn(OAc)_3\cdot 2H_2O$ (0.9 mmol) was evacuated and purged with nitrogen three times. Alkenoic acids (0.30 mmol), *H*-phosphonate (0.6 mmol) and CH_3COOH (2 mL) were sequentially added to the system at room temperature. The reaction mixture was heated with stirring at 60 ^{o}C for 8 hours. The reaction solution was concentrated in vacuo and then added 15 mL saturated sodium bicarbonate solution and extracted with EtOAc (3×10 mL). The combined organic layer was dried over $MgSO_4$, filtered and concentrated in vacuo. The residue was purified by silica gel column chromatography using petroleum ether–AcOEt (3:1-1:1, v/v) as the eluent to give the corresponding products.

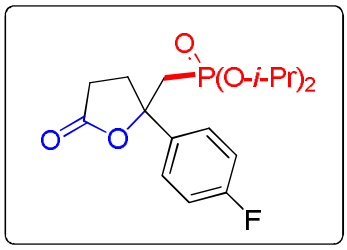
4. Spectral data

Diisopropyl ((5-oxo-2-phenyltetrahydrofuran-2-yl)methyl)phosphonate (3a)



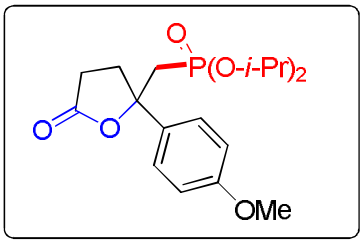
Yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.40 – 7.39 (m, 2H), 7.35 – 7.31 (m, 2H), 7.28 – 7.24 (m, 1H), 4.73 – 4.63 (m, 1H), 4.59 – 4.50 (m, 1H), 3.06 – 2.98 (m, 1H), 2.73 – 2.64 (m, 1H), 2.61 – 2.54 (m, 1H), 2.46 (d, J = 18.7 Hz, 2H), 2.42 – 2.35 (m, 1H), 1.27 (d, J = 6.2 Hz, 6H), 1.18 (d, J = 6.3 Hz, 3H), 1.14 (d, J = 6.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.0 (s), 143.7 (d, J = 9.6 Hz), 128.6 (s), 128.0 (s), 124.7 (s), 85.7 (d, J = 2.4 Hz), 71.1 (d, J = 6.8 Hz), 70.7 (d, J = 6.6 Hz), 40.6 (d, J = 141.1 Hz), 33.7 (d, J = 2.5 Hz), 28.7 (s), 24.14 (d, J = 3.4 Hz), 23.9 (m). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 23.4. HRMS-ESI: *m/z* 341.1504 ($[M+H]^+$, $C_{17}H_{26}O_5P^+$ calcd. 341.1512).

Diisopropyl ((2-(4-fluorophenyl)-5-oxotetrahydrofuran-2-yl)methyl)phosphonate (3b)



White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.44 – 7.38 (m, 2H), 7.08 – 7.02 (m, 2H), 4.73 – 4.65 (m, 1H), 4.61 – 4.52 (m, 1H), 3.06 – 2.99 (m, 1H), 2.75 – 2.67 (m, 1H), 2.64 – 2.57 (m, 1H), 2.48 – 2.40 (m, 3H), 1.30 (d, J = 6.2 Hz, 6H), 1.21 (d, J = 6.2 Hz, 3H), 1.17 (d, J = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 175.8 (s), 162.5 (d, J = 251.3 Hz), 139.38 (d, J = 3.1 Hz), 126.9 (d, J = 8.2 Hz), 115.6 (d, J = 21.7 Hz), 85.6 (s), 71.2 (d, J = 6.6 Hz), 70.8 (d, J = 6.8 Hz), 40.9 (d, J = 140.6 Hz), 33.94 (d, J = 2.5 Hz), 28.8 (s), 24.25 (d, J = 3.7 Hz), 24.0 (m). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 20.9. HRMS-ESI: *m/z* 381.1233 ($[M+Na]^+$, $C_{17}H_{24}FNaO_5P^+$ calcd. 381.1238).

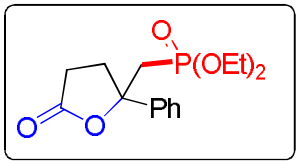
Diisopropyl ((2-(4-methoxyphenyl)-5-oxotetrahydrofuran-2-yl)methyl)phosphonate (3c)



Colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.31 – 7.27 (m, 2H), 6.86 – 6.82 (m, 2H), 4.69 – 4.60 (m, 1H), 4.56 – 4.48 (m, 1H), 3.75 (s, 3H), 2.99 – 2.91 (m, 1H), 2.68 – 2.53 (m, 2H), 2.46 – 2.33 (m, 3H), 1.26 – 1.25 (m, 6H), 1.17 (d, J = 6.1 Hz, 3H), 1.12 (d, J = 6.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.2 (s), 159.4 (s), 135.5 (d, J = 8.9 Hz), 126.1 (s), 113.9 (s),

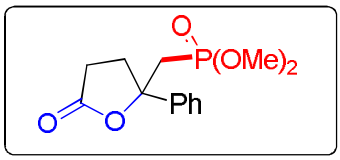
85.8 (s), 71.1 (d, J = 6.7 Hz), 70.7 (d, J = 6.8 Hz), 55.4 (s), 40.8 (d, J = 140.6 Hz), 33.7 (d, J = 2.3 Hz), 28.9 (s), 24.15 (d, J = 3.6 Hz), 23.9 (m). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 20.5. HRMS-ESI: *m/z* 393.1439 ($[M+Na]^+$, $C_{18}H_{27}NaO_6P^+$ calcd. 393.1437).

Diethyl ((5-oxo-2-phenyltetrahydrofuran-2-yl)methyl)phosphonate (3d)



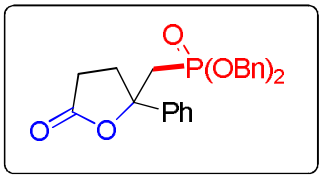
Colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.44 – 7.36 (m, 4H), 7.33 – 7.29 (m, 1H), 4.13 – 4.05 (m, 2H), 3.97 – 3.87 (m, 2H), 3.08 – 3.01 (m, 1H), 2.76 – 2.68 (m, 1H), 2.65 – 2.60 (m, 1H), 2.55 (d, J = 18.6 Hz, 2H), 2.50 – 2.41 (m, 1H), 1.31 (t, J = 7.1 Hz, 3H), 1.18 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.0 (s), 143.45 (d, J = 9.2 Hz), 128.7 (s), 128.2 (s), 124.7 (s), 85.6 (d, J = 2.2 Hz), 62,3 (d, J = 6.5 Hz), 61.85 (d, J = 6.6 Hz), 39.2 (d, J = 139.3 Hz), 33.87 (d, J = 2.9 Hz), 28.7 (s), 16.4 (d, J = 6.2 Hz), 16.26 (d, J = 6.3 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 23.2. HRMS-ESI: *m/z* 335.1021 ($[M+Na]^+$, $C_{15}H_{21}NaO_5P^+$ calcd. 335.1019).

Dimethyl ((5-oxo-2-phenyltetrahydrofuran-2-yl)methyl)phosphonate (3e)



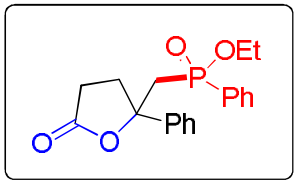
Colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.40 – 7.33 (m, 4H), 7.30 – 7.26 (m, 1H), 3.71 (d, J = 11.2 Hz, 3H), 3.51 (d, J = 11.1 Hz, 3H), 3.03 – 2.96 (m, 1H), 2.72 – 2.64 (m, 1H), 2.61 – 2.56 (m, 1H), 2.55 – 2.50 (m, 1H), 2.46 – 2.38 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 175.9 (s), 143.3 (d, J = 9.5 Hz), 128.8 (s), 128.2 (s), 124.6 (s), 85.38 (d, J = 2.2 Hz), 52.8 (d, J = 6.5 Hz), 52.3 (d, J = 6.6 Hz), 38.4 (d, J = 139.2 Hz), 33.9 (d, J = 3.1 Hz), 28.6 (s). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 25.9. HRMS-ESI: *m/z* 307.0710 ($[M+Na]^+$, $C_{13}H_{17}NaO_5P^+$ calcd. 307.0706).

Dibenzyl ((5-oxo-2-phenyltetrahydrofuran-2-yl)methyl)phosphonate (3f)



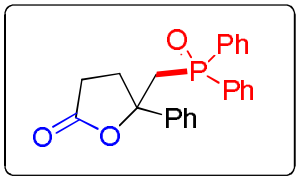
Colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.39 – 7.27 (m, 13H), 7.19 – 7.17 (m, 2H), 4.98 – 4.95 (m, 2H), 4.78 – 4.76 (m, 2H), 3.03 – 2.94 (m, 1H), 2.67 – 2.55 (m, 4H), 2.45 – 2.38 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 175.9 (s), 143.2 (d, J = 9.1 Hz), 136.1 (d, J = 6.0 Hz), 135.96 (d, J = 6.4 Hz), 128.8 (s), 128.7 (s), 128.65 (s), 128.63 (s), 128.57(s), 128.4 (s), 128.25 (s), 128.19 (s), 124.7 (s), 85.5 (d, J = 2.2 Hz), 67.9 (d, J = 6.4 Hz), 67.3 (d, J = 6.7 Hz), 39.7 (d, J = 138.9 Hz), 34.0 (d, J = 3.0 Hz), 28.6 (s). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 25.9. HRMS-ESI: *m/z* 459.1337 ($[M+Na]^+$, $C_{25}H_{25}NaO_5P^+$ calcd. 459.1332)

Ethyl ((5-oxo-2-phenyltetrahydrofuran-2-yl)methyl)(phenyl)phosphinate (3g)



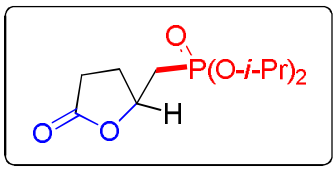
White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.59 – 7.14 (m, 10H), 3.95 – 3.66 (m, 2H), 3.27 – 3.17 (m, 1H), 2.88 – 2.54 (m, 5H), 1.28 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 175.8 (s), 143.9 (d, *J* = 8.0 Hz), 132.6 (d, *J* = 2.9 Hz), 131.65 (d, *J* = 10.2 Hz), 128.9 (s), 128.6 (s), 128.6 (s), 128.0 (s), 124.7(s), 86.2 (s), 61.1 (d, *J* = 6.1 Hz), 60.6 (d, *J* = 6.2 Hz), 43.4 (d, *J* = 35.0 Hz), 33.9 (d, *J* = 2.0 Hz), 28.6 (s), 16.4 (d, *J* = 6.6 Hz), 16.3 (d, *J* = 6.7 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 37.7. HRMS-ESI: *m/z* 345.1252 ($[M+H]^+$, $C_{19}H_{22}O_4P^+$ calcd. 345.1250).

5-((Diphenylphosphoryl)methyl)-5-phenyldihydrofuran-2(3H)-one (3h)



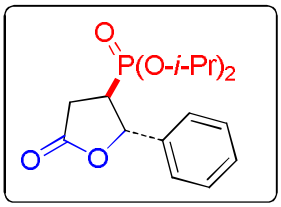
White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.78 – 7.73 (m, 2H), 7.58 – 7.53 (m, 2H), 7.50 – 7.42 (m, 3H), 7.40 – 7.34 (m, 3H), 7.33 – 7.28 (m, 2H), 7.21 – 7.13 (m, 3H), 3.43 – 3.35 (m, 1H), 3.14 – 3.02 (m, 2H), 2.77 – 2.67 (m, 2H), 2.42 – 2.33 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 175.6 (s), 143.0 (d, *J* = 5.9 Hz), 133.5 (d, *J* = 45.0 Hz), 132.5 (d, *J* = 44.7 Hz), 131.75 (d, *J* = 2.6 Hz), 131.4 (d, *J* = 2.6 Hz), 130.45 (d, *J* = 9.6 Hz), 130.2 (d, *J* = 9.5 Hz), 128.6 (d, *J* = 12.1 Hz), 128.43 (s), 128.4 (d, *J* = 11.7 Hz), 128.0 (s), 124.7 (s), 86.73 (d, *J* = 2.3 Hz), 42.2 (d, *J* = 67.3 Hz), 33.5 (s), 28.5 (s). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 25.9. HRMS-ESI: *m/z* 399.1123 ($[M+Na]^+$, $C_{23}H_{21}NaO_3P^+$ calcd. 399.1121).

Diisopropyl ((5-oxotetrahydrofuran-2-yl)methyl)phosphonate (3i)



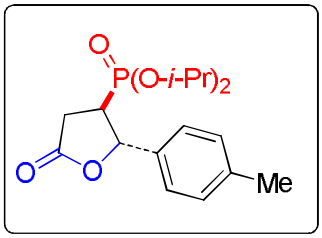
Colourless oil. 4.79 – 4.64 (m, 3H), 2.58 – 2.41 (m, 3H), 2.36 – 2.27 (m, 1H), 2.13 – 1.96 (m, 2H), 1.30 – 1.29 (m, 12H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 176.4 (s), 75.9 (s), 71.08 (d, *J* = 6.8 Hz), 70.9 (d, *J* = 6.6 Hz), 33.7 (d, *J* = 139.2 Hz), 29.0 (d, *J* = 4.7 Hz), 28.7 (s), 24.1 (m). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 22.7. HRMS-ESI: *m/z* 287.1013 ($[M+Na]^+$, $C_{11}H_{21}NaO_5P^+$ calcd. 287.1019).

Diisopropyl ((2S,3R)-5-oxo-2-phenyltetrahydrofuran-3-yl)phosphonate (5a)



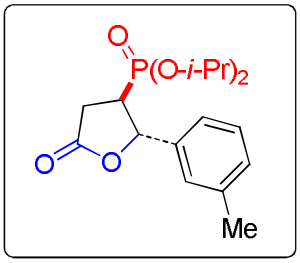
Colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.37 – 7.28 (m, 5H), 5.60 (dd, *J* = 13.5 Hz, *J* = 7.0 Hz, 1H), 4.72 – 4.62 (m, 2H), 2.89 – 2.83 (m, 2H), 2.79 – 2.70 (m, 1H), 1.27 (d, *J* = 1.7 Hz, 3H), 1.26 (d, *J* = 1.7 Hz, 3H), 1.21 (d, *J* = 6.2 Hz, 3H), 1.12 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 174.5 (d, *J* = 12.4 Hz), 138.4 (d, *J* = 5.4 Hz), 129.0 (s), 128.8 (s), 126.1 (s), 81.1 (s), 71.9 (d, *J* = 7.0 Hz), 70.6 (d, *J* = 7.0 Hz), 41.5 (d, *J* = 152.0 Hz), 30.3 (d, *J* = 4.1 Hz), 23.9 (m), 23.67 (d, *J* = 4.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 22.6. HRMS-ESI: *m/z* 349.1178 ($[M+Na]^+$, $C_{16}H_{23}NaO_5P^+$ calcd. 349.1175).

Diisopropyl ((2S,3R)-5-oxo-2-(p-tolyl)tetrahydrofuran-3-yl)phosphonate (5b)



Colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.22 (d, *J* = 8.1 Hz, 2H), 7.16 (d, *J* = 8.1 Hz, 2H), 5.58 (dd, *J* = 13.5 Hz, *J* = 7.0 Hz, 1H), 4.73 – 4.63 (m, 2H), 2.89 – 2.83 (m, 2H), 2.79 – 2.69 (m, 1H), 2.33 (s, 3H), 1.28 (d, *J* = 2.5 Hz, 3H), 1.27 (d, *J* = 2.5 Hz, 3H), 1.21 (d, *J* = 6.2 Hz, 3H), 1.15 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 174.6 (d, *J* = 12.7 Hz), 138.9 (s), 135.5 (d, *J* = 5.3 Hz), 129.4 (s), 126.1 (s), 81.1 (s), 71.8 (d, *J* = 6.8 Hz), 71.5 (d, *J* = 6.8 Hz), 41.5 (d, *J* = 152.5 Hz), 30.4 (d, *J* = 4.1 Hz), 23.9 (m), 23.7 (d, *J* = 4.8 Hz), 21.1 (s). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 22.8. HRMS-ESI: *m/z* 343.1512 ($[M+H]^+$, $C_{17}H_{26}O_5P^+$ calcd. 343.1512).

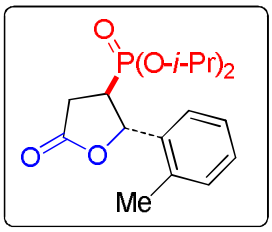
Diisopropyl ((2S,3R)-5-oxo-2-(m-tolyl)tetrahydrofuran-3-yl)phosphonate (5c)



Colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.30 – 7.25 (m, 1H), 7.15 (d, *J* = 7.7 Hz, 3H), 5.61 (dd, *J* = 13.8 Hz, *J* = 6.8 Hz, 1H), 4.77 – 4.67 (m, 2H), 2.92 – 2.86 (m, 2H), 2.82 – 2.73 (m, 1H), 2.36 (s, 3H), 1.31 (d, *J* = 2.0 Hz, 3H), 1.30 (d, *J* = 2.0 Hz, 3H), 1.24 (d, *J* = 6.2 Hz, 3H), 1.18 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 174.6 (d, *J* = 11.9 Hz), 138.6 (s), 138.46 (d, *J* = 5.5 Hz), 129.7 (s), 128.7 (s), 126.7 (s), 123.2 (s), 81.1 (s), 71.93 (d, *J* = 7.0 Hz), 71.65 (d, *J* = 7.1 Hz), 41.5 (d, *J* = 152.0 Hz), 30.25 (d, *J* = 4.0 Hz), 24.0 (m), 23.7 (d, *J* = 4.7 Hz), 21.4 (s). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 23.0. HRMS-ESI: *m/z* 343.1508 ($[M+H]^+$,

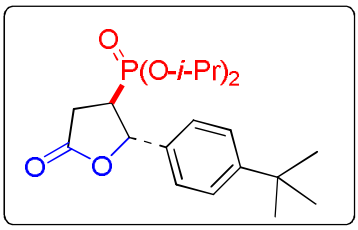
$C_{17}H_{26}O_5P^+$ calcd. 343.1512).

Diisopropyl ((2S,3R)-5-oxo-2-(o-tolyl)tetrahydrofuran-3-yl)phosphonate (5d)



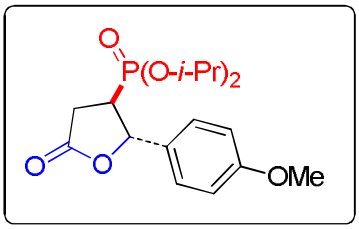
Colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.30 – 7.25 (m, 1H), 7.16 (d, J = 7.7 Hz, 3H), 5.61 (dd, J = 13.8 Hz, J = 6.9 Hz, 1H), 4.77 – 4.67 (m, 2H), 2.92 – 2.86 (m, 2H), 2.82 – 2.72 (m, 1H), 2.36 (s, 3H), 1.31 (d, J = 2.4 Hz, 3H), 1.30 (d, J = 2.4 Hz, 3H), 1.24 (d, J = 6.2 Hz, 3H), 1.18 (d, J = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 174.7 (d, J = 12.1 Hz), 138.6 (s), 138.5 (d, J = 5.5 Hz), 129.7 (s), 128.8 (s), 126.7 (s), 123.2 (s), 81.1 (s), 71.9 (d, J = 7.0 Hz), 71.6 (d, J = 7.0 Hz), 41.5 (d, J = 152.1 Hz), 30.3 (d, J = 4.1 Hz), 24.0 (m), 23.75 (d, J = 4.8 Hz), 21.5 (s). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 22.9. HRMS-ESI: *m/z* 343.1512 ($[M+H]^+$, $C_{17}H_{26}O_5P^+$ calcd. 343.1512).

Diisopropyl ((2S,3R)-2-(4-(tert-butyl)phenyl)-5-oxotetrahydrofuran-3-yl)phosphonate (5e)



Colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.42 – 7.40 (m, 2H), 7.31 – 7.29 (m, 2H), 5.61 (dd, J = 13.4 Hz, J = 7.1 Hz, 1H), 4.75 – 4.65 (m, 2H), 2.92 – 2.86 (m, 2H), 2.84 – 2.76 (m, 1H), 1.32 – 1.29 (m, 15H), 1.22 (d, J = 6.2 Hz, 3H), 1.14 (d, J = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 174.7 (d, J = 12.5 Hz), 152.3 (s), 138.35 (d, J = 5.1 Hz), 126.0 (s), 125.8 (s), 81.1 (s), 71.9 (d, J = 6.7 Hz), 71.7 (d, J = 7.1 Hz), 41.6 (d, J = 152.1 Hz), 34.7 (s), 31.4 (s), 30.5 (d, J = 4.1 Hz), 24.0 (m), 23.7 (d, J = 4.8 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 22.6. HRMS-ESI: *m/z* 405.1804 ($[M+Na]^+$, $C_{20}H_{31}NaO_5P^+$ calcd. 405.1801).

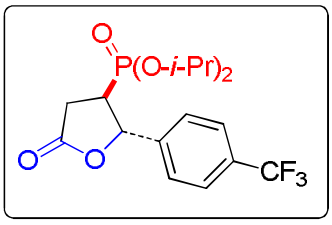
Diisopropyl ((2S,3R)-2-(4-methoxyphenyl)-5-oxotetrahydrofuran-3-yl)phosphonate (5f)



Colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.24 – 7.21 (m, 2H), 6.86 – 6.83 (m, 2H), 6.18 (dd, J = 8.6 Hz, J = 6.4 Hz, 1H), 4.71 – 4.30 (m, 3H), 3.76 (s, 3H), 2.91 – 2.57 (m, 2H), 1.28 (d, J = 6.2 Hz, 6H), 1.19 (d, J = 6.3 Hz, 3H), 1.14 (d, J = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 170.5 (s), 159.4 (s), 130.9 (s), 128.0 (s), 113.7 (s), 81.1 (s), 70.8 (d, J = 6.9 Hz),

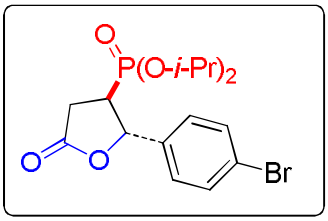
70.6 (d, J = 7.1 Hz), 55.2 (s), 43.7 (d, J = 140.3 Hz), 23.8 (m), 20.6 (s). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 22.6. HRMS-ESI: *m/z* 379.1286 ($[M+Na]^+$, $C_{17}H_{25}NaO_6P^+$ calcd. 379.1281).

Diisopropyl ((2S,3R)-5-oxo-2-(4-(trifluoromethyl)phenyl)tetrahydrofuran-3-yl)phosphonate (5g)



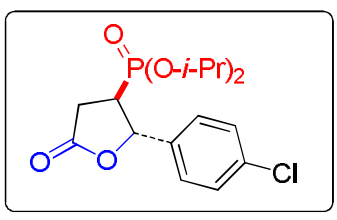
Colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.66 (d, J = 8.2 Hz, 2H), 7.53 (d, J = 8.3 Hz, 2H), 5.70 (dd, J = 13.6 Hz, J = 7.1 Hz, 1H), 4.83 – 4.71 (m, 2H), 2.95 – 2.87 (m, 2H), 2.81 – 2.71 (m, 1H), 1.33 (t, J = 6.0 Hz, 6H), 1.28 (d, J = 6.2 Hz, 3H), 1.23 (d, J = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 174.2 (d, J = 12.1 Hz), 142.7 (d, J = 4.8 Hz), 131.4 (d, J = 33.0 Hz), 130.5 (s), 126.6 (s), 125.9 (q, J = 3.8 Hz), 80.1 (s), 72.4 (d, J = 7.0 Hz), 72.2 (d, J = 6.9 Hz), 41.7 (d, J = 153.2 Hz), 30.2 (d, J = 4.0 Hz), 24.1 (m), 23.9 (d, J = 4.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 21.9. HRMS-ESI: *m/z* 417.1056 ($[M+Na]^+$, $C_{17}H_{22}F_3NaO_5P^+$ calcd. 417.1049).

Diisopropyl ((2S,3R)-2-(4-bromophenyl)-5-oxotetrahydrofuran-3-yl)phosphonate (5h)



Colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.47 (d, J = 8.5 Hz, 2H), 7.23 (d, J = 8.3 Hz, 2H), 5.54 (dd, J = 13.3 Hz, J = 7.4 Hz, 1H), 4.74 – 4.62 (m, 2H), 2.87 – 2.81 (m, 2H), 2.74 – 2.63 (m, 1H), 1.27 (t, J = 5.5 Hz, 6H), 1.21 (d, J = 6.2 Hz, 3H), 1.16 (d, J = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 174.2 (d, J = 13.2 Hz), 137.5 (d, J = 4.9 Hz), 131.9 (s), 127.9 (s), 123.0 (s), 81.3 (s), 72.0 (d, J = 6.9 Hz), 71.7 (d, J = 7.1 Hz), 41.5 (d, J = 152.5 Hz), 30.27 (d, J = 4.0 Hz), 24.0 (m), 23.76 (d, J = 4.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 22.1. HRMS-ESI: *m/z* 427.0279 ($[M+Na]^+$, $C_{16}H_{22}BrNaO_5P^+$ calcd. 427.0280).

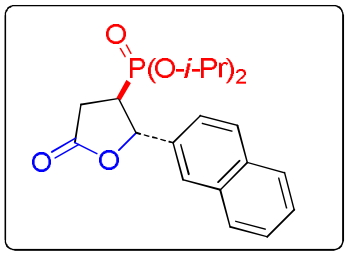
Diisopropyl ((2S,3R)-2-(4-chlorophenyl)-5-oxotetrahydrofuran-3-yl)phosphonate (5i)



Colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.38 – 7.31 (m, 4H), 5.60 (dd, J = 13.2 Hz, J = 7.4 Hz, 1H), 4.78 – 4.67 (m, 2H), 2.92 – 2.86 (m, 2H), 2.77 – 2.67 (m, 1H), 1.31 (t, J = 5.5 Hz, 6H), 1.26 (d, J = 6.2 Hz, 3H), 1.21 (d, J = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm)

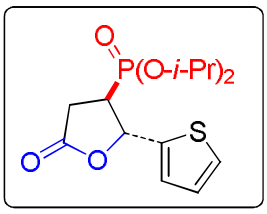
174.2 (d, J = 12.8 Hz), 137.05 (d, J = 4.5 Hz), 134.9 (s), 129.0 (s), 127.6 (s), 81.4 (s), 72.0 (d, J = 6.9 Hz), 71.7 (d, J = 7.1 Hz), 41.6 (d, J = 152.3 Hz), 30.36 (d, J = 4.1 Hz), 24.0 (m), 23.8 (d, J = 4.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 22.0. HRMS-ESI: *m/z* 383.0789 ($[M+Na]^+$, $C_{16}H_{22}ClNaO_5P^+$ calcd. 383.0786).

Diisopropyl ((2S,3R)-2-(naphthalen-2-yl)-5-oxotetrahydrofuran-3-yl)phosphonate (5j)



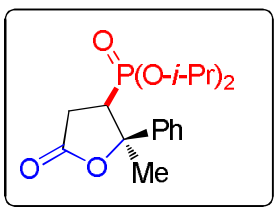
Colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.91 – 7.89 (m, 1H), 7.87 – 7.84 (m, 2H), 7.54 – 7.51 (m, 2H), 7.49 – 7.46 (m, 2H), 5.84 (dd, J = 14.1 Hz, J = 6.6 Hz, 1H), 4.81 – 4.70 (m, 2H), 2.98 – 2.82 (m, 3H), 1.32 (dd, J = 6.1 Hz, J = 4.3 Hz, 6H), 1.25 (d, J = 6.2 Hz, 3H), 1.19 (d, J = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 174.7 (d, J = 12.0 Hz), 136.0 (d, J = 5.5 Hz), 133.6 (s), 133.2 (s), 132.3 (d, J = 10.1 Hz), 129.1 (s), 128.3 (s), 127.9 (s), 126.9 (s), 125.6 (s), 123.2 (s), 81.2 (s), 72.1 (d, J = 7.1 Hz), 71.8 (d, J = 7.3 Hz), 41.6 (d, J = 151.8 Hz), 30.3 (d, J = 3.9 Hz), 24.0 (m). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 22.4. HRMS-ESI: *m/z* 399.1335 ($[M+Na]^+$, $C_{20}H_{25}NaO_5P^+$ calcd. 399.1332).

Diisopropyl ((2R,3R)-5-oxo-2-(thiophen-2-yl)tetrahydrofuran-3-yl)phosphonate (5k)



Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.31 – 7.30 (m, 1H), 7.11 – 7.10 (m, 1H), 6.96 – 6.94 (m, 1H), 5.79 (dd, J = 12.7 Hz, J = 6.6 Hz, 1H), 4.70 – 4.61 (m, 2H), 2.92 – 2.82 (m, 3H), 1.26 – 1.24 (m, 6H), 1.17 – 1.15 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 173.6 (d, J = 12.8 Hz), 140.9 (d, J = 6.0 Hz), 127.0 (s), 126.7 (s), 125.8 (s), 77.1 (s), 71.8 (d, J = 6.9 Hz), 71.7 (d, J = 7.1 Hz), 67.7 (s), 41.6 (d, J = 153.0 Hz), 23.8 (m). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 21.8. HRMS-ESI: *m/z* 355.0735 ($[M+Na]^+$, $C_{14}H_{21}NaO_5PS^+$ calcd. 355.0740).

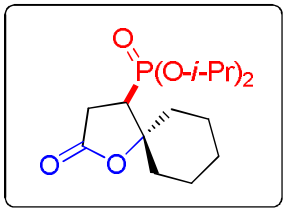
Diisopropyl ((2R,3R)-2-methyl-5-oxo-2-phenyltetrahydrofuran-3-yl)phosphonate (5l)



Colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.53 – 7.50 (m, 2H), 7.39 – 7.35 (m, 2H),

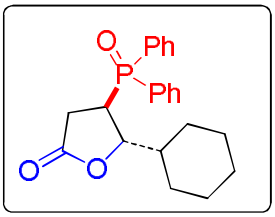
7.33 – 7.28 (m, 1H), 4.83 – 4.70 (m, 2H), 3.03 – 2.88 (m, 2H), 2.82 – 2.72 (m, 1H), 1.92 (s, 3H), 1.36 – 1.29 (m, 12H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 174.1 (d, *J* = 12.5 Hz), 144.4 (d, *J* = 5.9 Hz), 128.6 (s), 128.1 (s), 124.7 (s), 87.2 (s), 71.7 (d, *J* = 7.2 Hz), 71.6 (d, *J* = 6.9 Hz), 45.1 (d, *J* = 149.6 Hz), 31.54 (d, *J* = 3.9 Hz), 25.4 (d, *J* = 3.1 Hz), 24.0 (m). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 21.6. HRMS-ESI: *m/z* 343.1516 ($[M+H]^+$, $C_{17}H_{26}O_5P^+$ calcd. 343.1512).

(R)-Diisopropyl (2-oxo-1-oxaspiro[4.5]decan-4-yl)phosphonate (5m)



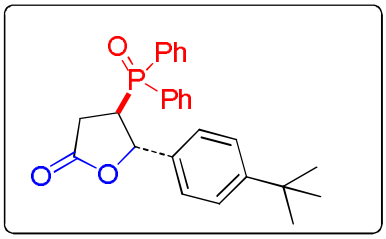
White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 4.77 – 4.69 (m, 2H), 2.95 – 2.84 (m, 1H), 2.71 – 2.64 (m, 1H), 2.46 – 2.37 (m, 1H), 1.99 – 1.88 (m, 2H), 1.81 – 1.54 (m, 8H), 1.34 – 1.30 (m, 12H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 174.2 (d, *J* = 18.9 Hz), 87.03 (d, *J* = 18.9 Hz), 71.36 (d, *J* = 5.8 Hz), 71.28 (d, *J* = 5.2 Hz), 44.5 (d, *J* = 153.5 Hz), 37.6 (s), 33.15 (d, *J* = 2.7 Hz), 30.96 (d, *J* = 3.3 Hz), 25.0 (s), 24.2 (m), 22.8 (s), 21.7 (s). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 21.7. HRMS-ESI: *m/z* 319.1700 ($[M+H]^+$, $C_{15}H_{28}O_5P^+$ calcd. 319.1699).

(4R,5S)-5-Cyclohexyl-4-(diphenylphosphoryl)dihydrofuran-2(3H)-one (5n)



White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.84 – 7.74 (m, 4H), 7.57 – 7.46 (m, 6H), 4.64 (dd, *J* = 13.9 Hz, *J* = 6.0 Hz, 1H), 3.26 – 3.19 (m, 1H), 2.87 – 2.64 (m, 2H), 1.66 – 1.52 (m, 4H), 1.47 – 1.42 (m, 1H), 1.05 – 0.94 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 174.36 (d, *J* = 7.7 Hz), 132.7 (s), 131.1 (d, *J* = 2.7 Hz), 131.0 (d, *J* = 2.3 Hz), 130.0 (d, *J* = 6.3 Hz), 129.2 (s), 129.1 (s), 129.0 (s), 82.8 (s), 41.94 (d, *J* = 4.5 Hz), 36.2 (d, *J* = 73.2 Hz), 29.77 (d, *J* = 2.1 Hz), 29.6 (s), 26.0 (d, *J* = 4.5 Hz), 25.65 (d, *J* = 3.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 31.0. HRMS-ESI: *m/z* 391.1435 ($[M+Na]^+$, $C_{22}H_{25}NaO_3P^+$ calcd. 391.1434).

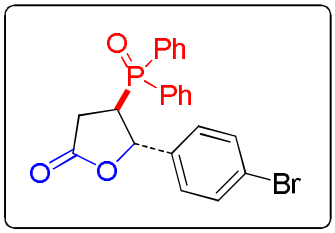
(4R,5S)-5-(4-(Tert-butyl) phenyl)-4-(diphenylphosphoryl)dihydrofuran-2(3H)-one (5o)



White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.78 – 7.73 (m, 2H), 7.53 – 7.41 (m, 5H), 7.35 – 7.31 (m, 1H), 7.20 – 7.16 (m, 2H), 7.08 (d, *J* = 8.4 Hz, 2H), 6.90 (d, *J* = 8.3 Hz, 2H), 5.69 (t, *J* =

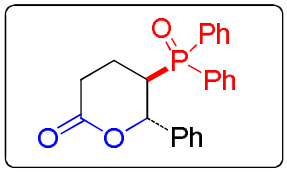
9.2 Hz, 1H), 3.54 (q, J = 9.4 Hz, 1H), 3.21 – 3.09 (m, 1H), 2.79 – 2.71 (m, 1H), 1.23 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 174.1 (d, J = 11.8 Hz), 151.8 (s), 134.6 (s), 132.38 (d, J = 2.4 Hz), 131.9 (d, J = 2.5 Hz), 130.9 (d, J = 9.4 Hz), 130.4 (d, J = 9.2 Hz), 129.0 (d, J = 11.7 Hz), 128.4 (d, J = 11.9 Hz), 126.2 (s), 125.4 (s), 80.5 (s), 43.3 (d, J = 73.6 Hz), 34.4 (s), 31.2 (s), 30.1 (s). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 27.6. HRMS-ESI: *m/z* 441.1592 ([M+Na]$^+$, $C_{26}H_{27}NaO_3P^+$ calcd. 441.1590).

(4R,5S)-5-(4-Bromophenyl)-4-(diphenylphosphoryl)dihydrofuran-2(3H)-one (5p)



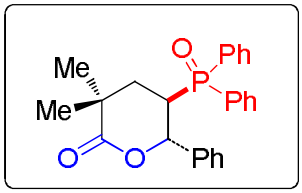
White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.76 – 7.71 (m, 2H), 7.56 – 7.43 (m, 6H), 7.30 – 7.26 (m, 2H), 7.21 (d, J = 8.4 Hz, 2H), 6.82 (d, J = 8.4 Hz, 2H), 5.67 (t, J = 9.4 Hz, 1H), 3.37 (q, J = 9.5 Hz, 1H), 3.21 – 3.10 (m, 1H), 2.76 – 2.68 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 173.8 (s), 137.0 (s), 132.7 (s), 132.4 (s), 131.7 (s), 131.0 (d, J = 8.9 Hz), 130.6 (d, J = 9.0 Hz), 129.2 (d, J = 11.7 Hz), 128.8 (d, J = 12.0 Hz), 128.1 (s), 123.0 (s), 79.9 (s), 43.7 (d, J = 73.0 Hz), 30.2 (s). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 27.4. HRMS-ESI: *m/z* 463.0068 ([M+Na]$^+$, $C_{22}H_{18}BrNaO_3P^+$ calcd. 463.0069).

(5R,6S)-5-(Diphenylphosphoryl)-6-phenyltetrahydro-2H-pyran-2-one (5q)



White solid. ^{1}H NMR (400 MHz, C_2D_6OS) δ (ppm) 7.92 – 7.87 (m, 2H), 7.54 – 7.48 (m, 5H), 7.29 – 7.24 (m, 1H), 7.20 – 7.14 (m, 4H), 7.04 – 7.01 (m, 3H), 5.63 (dd, J = 9.5 Hz, J = 6.2 Hz, 1H), 3.74 – 3.67 (m, 1H), 2.84 – 2.76 (m, 1H), 2.60 – 2.50 (m, 1H), 2.10 – 1.98 (m, 1H), 1.92 – 1.79 (m, 1H). ^{13}C NMR (100 MHz, C_2D_6OS) δ (ppm) 170.5 (s), 137.4 (s), 133.1 (d, J = 67.6 Hz), 132.1 (d, J = 67.6 Hz), 131.5 (d, J = 2.2 Hz), 130.7 (d, J = 2.8 Hz), 130.2 (d, J = 8.9 Hz), 130.0 (d, J = 9.1 Hz), 128.7 (d, J = 11.4 Hz), 128.3 (s), 127.8 (d, J = 11.7 Hz), 127.7 (s), 127.6 (s), 80.17 (d, J = 3.5 Hz), 36.5 (d, J = 69.7 Hz), 28.4 (d, J = 8.3 Hz), 19.7 (s). ^{31}P NMR (162 MHz, C_2D_6OS) δ (ppm) 31.0. HRMS-ESI: *m/z* 399.1126 ([M+Na]$^+$, $C_{23}H_{21}NaO_3P^+$ calcd. 399.1121).

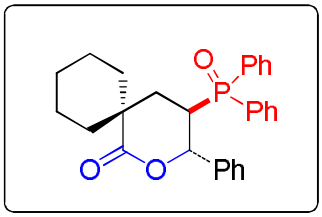
(5R,6S)-5-(Diphenylphosphoryl)-3,3-dimethyl-6-phenyltetrahydro-2H-pyran-2-one (5r)



White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.74 – 7.69 (m, 2H), 7.49 – 7.40 (m, 3H), 7.36

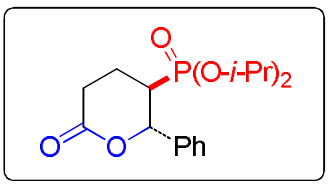
– 7.31 (m, 2H), 7.17 – 7.12 (m, 3H), 7.05 – 7.01 (m, 2H), 6.96 – 6.89 (m, 3H), 5.78 (dd, *J* = 11.0 Hz, *J* = 5.5 Hz, 1H), 3.22 – 3.14 (m, 1H), 2.39 – 2.31 (m, 1H), 1.62 – 1.56 (m, 1H), 1.39 (s), 1.32 (s). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 175.7 (s), 137.5 (s), 132.6 (s), 131.96 (d, *J* = 2.5 Hz), 131.6 (s), 131.0 (d, *J* = 3.0 Hz), 130.3 (d, *J* = 8.8 Hz), 130.2 (d, *J* = 9.0 Hz), 129.05 (d, *J* = 11.6 Hz), 128.9 (s), 128.2 (s), 128.1 (d, *J* = 12.1 Hz), 127.7 (s), 83.12 (d, *J* = 4.0 Hz), 38.2 (d, *J* = 11.6 Hz), 37.6 (d, *J* = 150.1 Hz), 35.7 (s), 27.5 (s), 27.4 (s). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 29.1. HRMS-ESI: *m/z* 427.1432 ($[M+Na]^+$, $C_{25}H_{25}NaO_3P^+$ calcd. 427.1434).

(3S,4R)-4-(Diphenylphosphoryl)-3-phenyl-2-oxaspiro[5.5]undecan-1-one (5s)



White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.76 (t, *J* = 7.5 Hz, 2H), 7.52 – 7.45 (m, 3H), 7.35 (t, *J* = 8.2 Hz, 2H), 7.19 – 7.12 (m, 3H), 7.07 – 7.04 (m, 2H), 6.97 – 6.92 (m, 3H), 5.73 (dd, *J* = 10.2 Hz, *J* = 4.6 Hz, 1H), 3.14 – 3.02 (m, 1H), 2.17 – 2.07 (m, 2H), 1.98 – 1.90 (m, 2H), 1.82 – 1.75 (m, 1H), 1.67 – 1.52 (m, 3H), 1.48 – 1.43 (m, 1H), 1.38 – 1.29 (m, 1H), 1.20 – 1.02 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 175.7 (s), 137.7 (s), 131.5 (d, *J* = 101.8 Hz), 130.3 (t, *J* = 9.0 Hz), 129.0 (d, *J* = 10.8 Hz), 128.9 (s), 128.2 (s), 128.1 (s), 127.7 (s), 82.3 (s), 41.7 (d, *J* = 10.5 Hz), 37.2 (d, *J* = 71.0 Hz), 35.6 (s), 33.5 (s), 30.4 (s), 25.3 (s), 20.9 (s), 20.8 (s). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 29.6. HRMS-ESI: *m/z* 467.1750 ($[M+Na]^+$, $C_{28}H_{29}NaO_3P^+$ calcd. 467.1747).

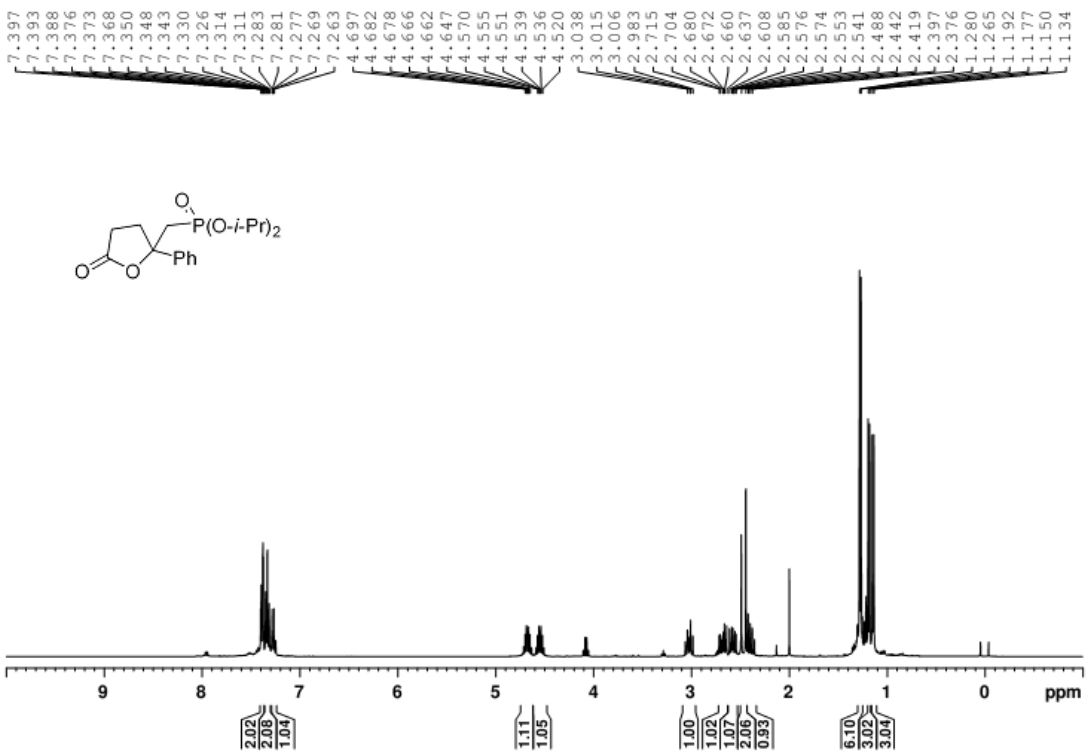
Diisopropyl ((2S,3R)-6-oxo-2-phenyltetrahydro-2H-pyran-3-yl)phosphonate (5t)



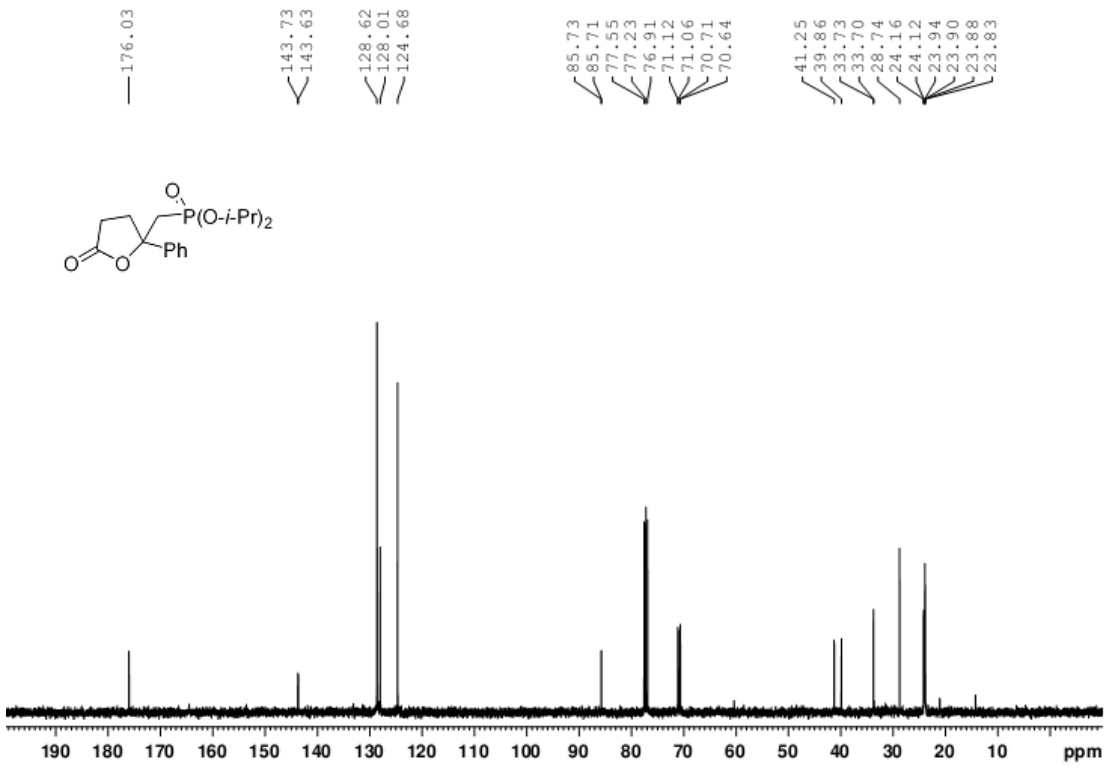
White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.38 – 7.30 (m, 5H), 5.53 (t, *J* = 7.9 Hz, 1H), 4.64 – 4.52 (m, 2H), 2.83 – 2.74 (m, 1H), 2.61 – 2.53 (m, 1H), 2.46 – 2.36 (m, 1H), 2.26 – 2.17 (m, 2H), 1.24 (d, *J* = 6.2 Hz, 3H), 1.15 (d, *J* = 6.2 Hz, 6H), 1.08 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 170.7 (s), 138.7 (d, *J* = 4.8 Hz), 128.9 (s), 128.6 (s), 127.3 (s), 81.2 (s), 71.46 (d, *J* = 7.1 Hz), 71.0 (d, *J* = 7.2 Hz), 38.7 (d, *J* = 144.1 Hz), 28.8 (d, *J* = 9.3 Hz), 24.0 (m), 23.65 (d, *J* = 3.7 Hz), 19.76 (d, *J* = 3.8 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ (ppm) 23.8. HRMS-ESI: *m/z* 341.1511 ($[M+H]^+$, $C_{17}H_{26}O_5P^+$ calcd. 341.1512).

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

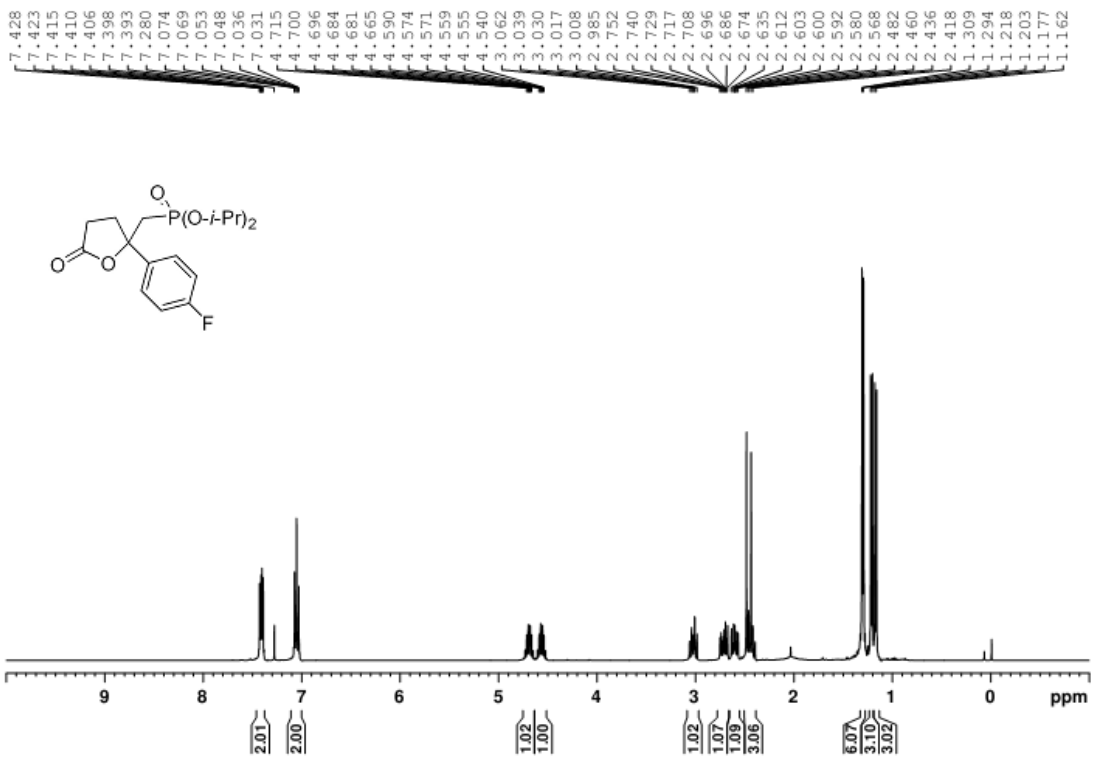
3a



3a



3b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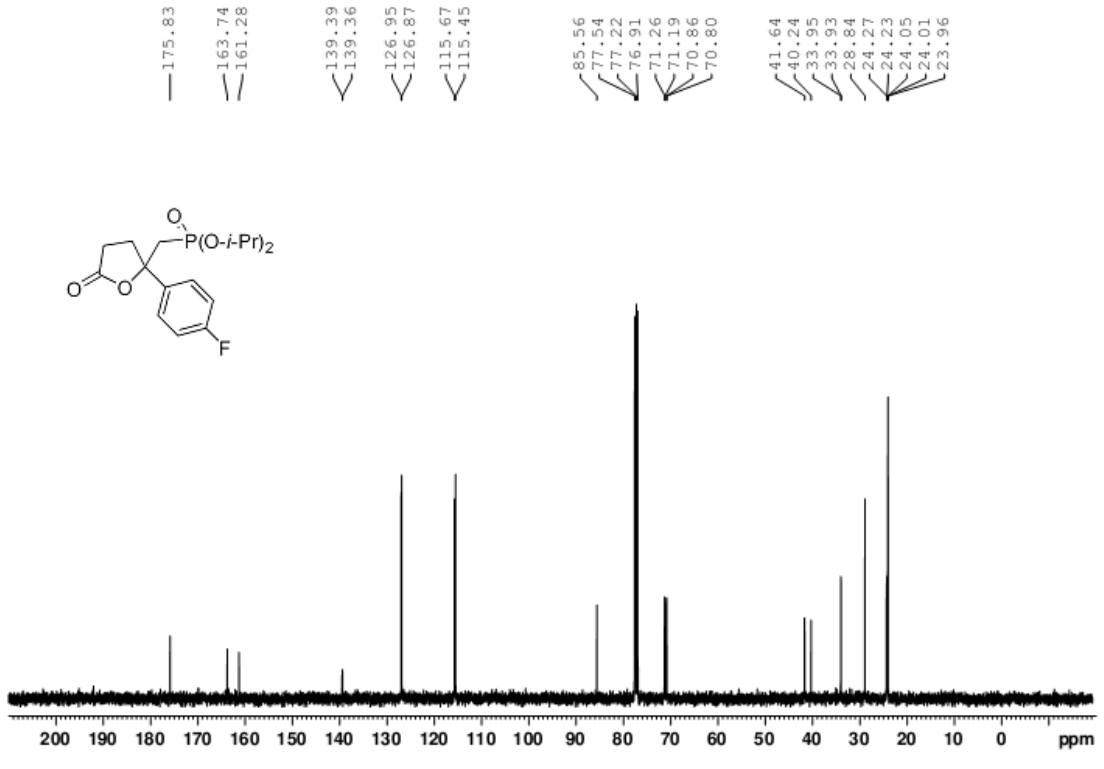
7.410
7.406
7.398
7.393
7.280
7.074
7.069
7.053
7.048
7.036
7.031
4.715
4.700
4.696
4.684
4.681
4.665
4.590
4.574
4.571
4.559
4.555
4.540
3.062
3.039
3.030
3.017
3.008
2.985
2.752
2.740
2.729
2.717
2.708
2.696
2.686
2.674
2.635
2.612
2.603
2.600
2.592
2.580
2.568
2.482
2.460
2.436
2.418
1.309
1.294
1.218
1.203
1.177
1.162
O
P(O-i-Pr)2
O
O
F
9
8
7
6
5
4
3
2
1
0
ppm
2.01
2.00
1.02
1.00
1.02
1.07
1.09
3.06
6.07
3.10
3.02

3b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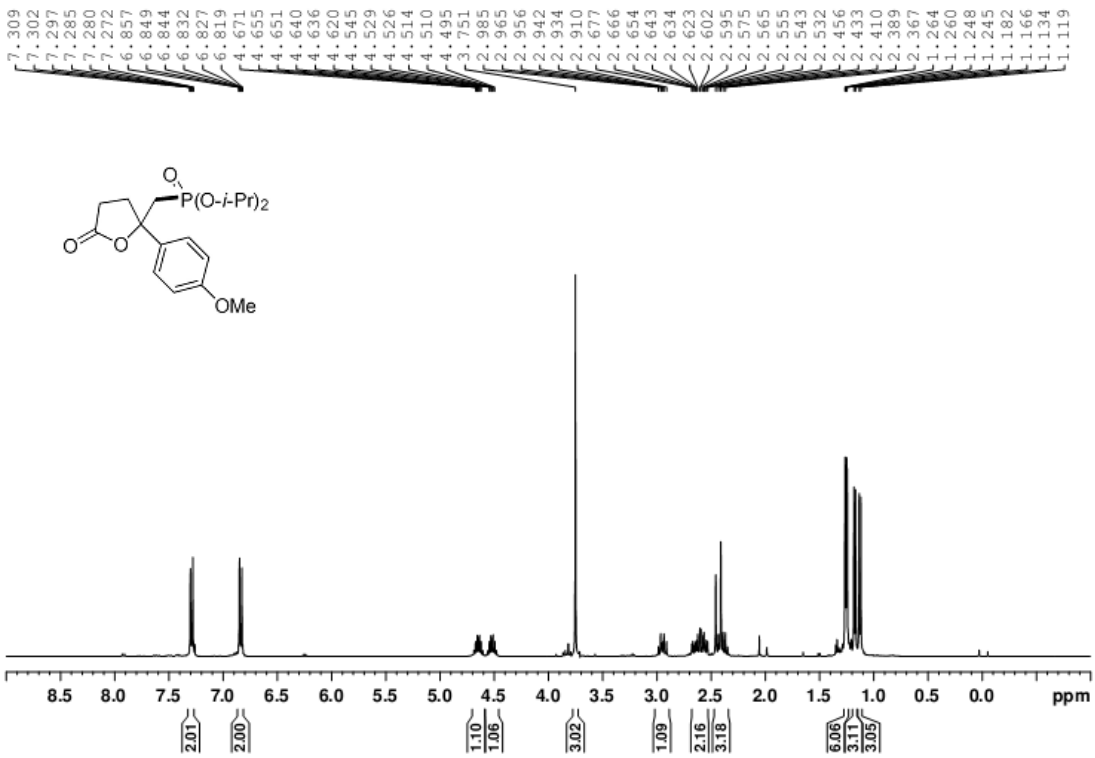
163.74
161.28
139.39
139.36
126.95
126.87
115.67
115.45
85.56
77.54
77.22
76.91
71.26
71.19
70.86
70.80
41.64
40.24
33.95
33.93
28.84
24.27
24.23
24.05
24.01
23.96
O
P(O-i-Pr)2
O
O
F
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3c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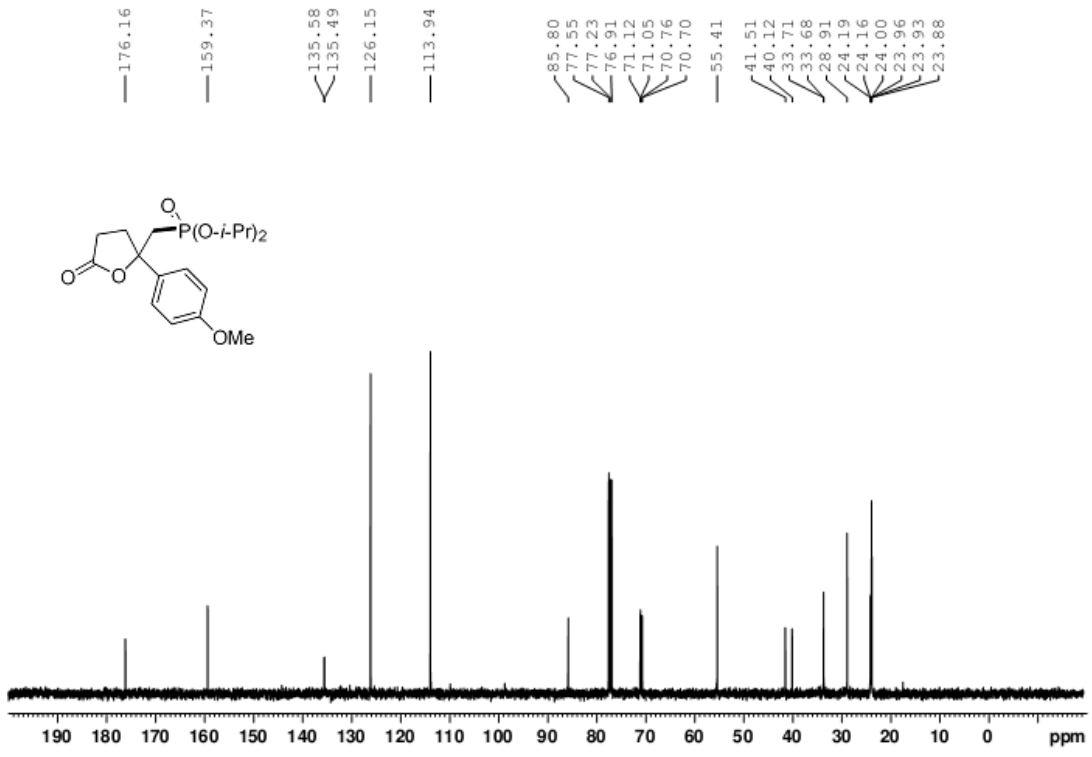
7.297
7.285
7.280
7.272
6.857
6.849
6.844
6.832
6.827
6.819
4.671
4.655
4.651
4.640
4.636
4.620
4.545
4.529
4.526
4.514
4.510
4.495
3.751
2.985
2.965
2.956
2.942
2.934
2.910
2.677
2.666
2.654
2.643
2.634
2.623
2.602
2.595
2.575
2.565
2.555
2.543
2.532
2.456
2.433
2.410
2.389
2.367
1.264
1.260
1.248
1.245
1.182
1.166
1.134
1.119
O
P(O-i-Pr)2
O
O
OMe
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 ppm
2.01
2.00
1.10
1.06
3.02
1.09
2.16
3.18
6.06
3.11
3.05

3c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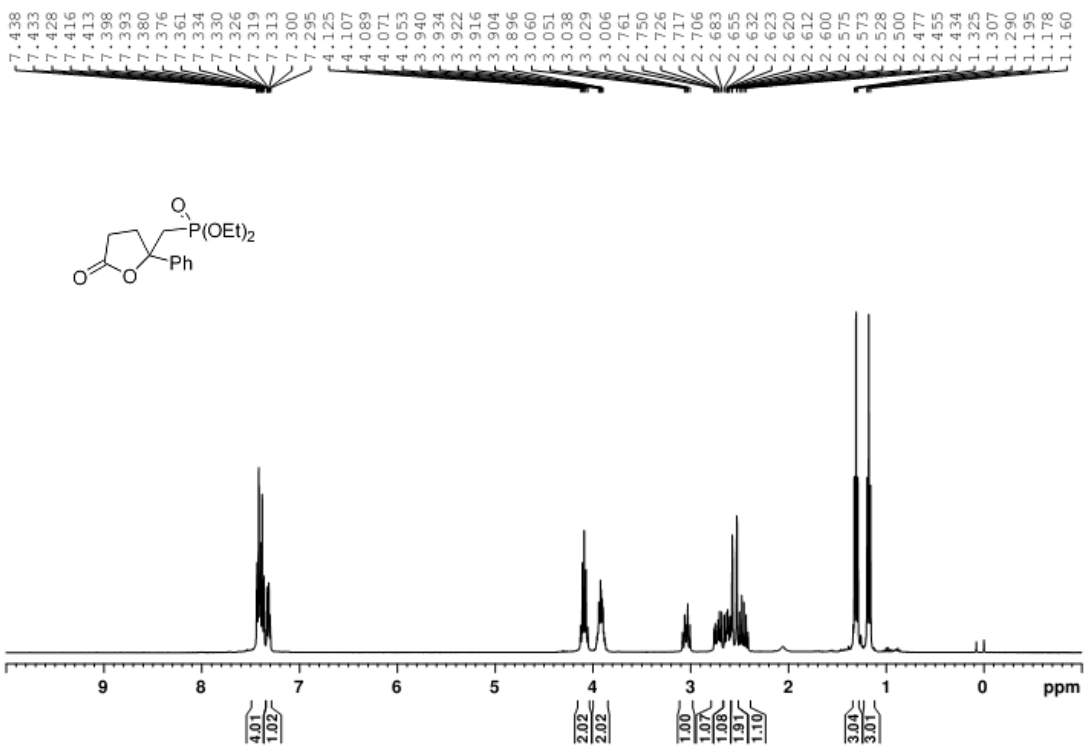
159.37
135.58
135.49
126.15
113.94
85.80
77.55
77.23
76.91
71.12
71.05
70.76
70.70
55.41
41.51
40.12
33.71
33.68
28.91
24.19
24.16
24.00
23.96
23.93
23.88
O
P(O-i-Pr)2
O
O
OMe
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

3d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7.393
7.380
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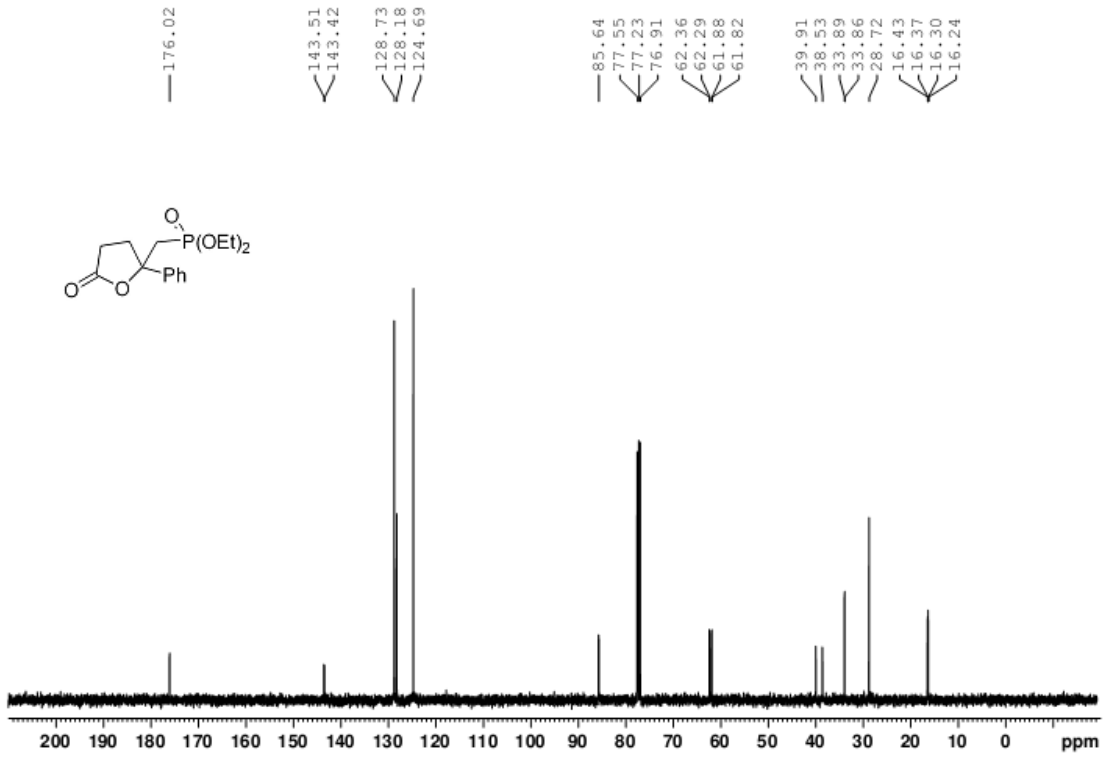
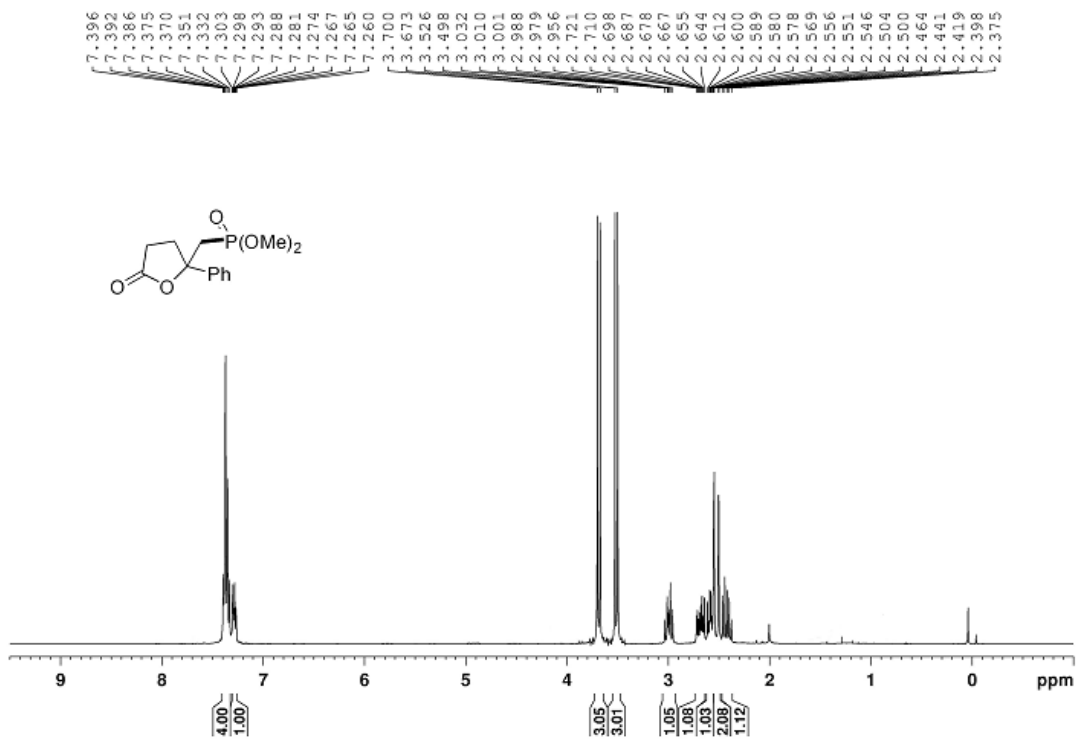
7.361
7.334
7.330
7.326
7.319
7.313
7.300
7.295
4.125
4.107
4.089
4.071
4.053
3.940
3.934
3.922
3.916
3.904
3.896
3.060
3.051
3.038
3.029
3.006
2.761
2.750
2.726
2.717
2.706
2.683
2.655
2.632
2.623
2.620
2.612
2.600
2.575
2.573
2.528
2.500
2.477
2.455
2.434
1.325
1.307
1.290
1.195
1.178
1.160
O
P(OEt)2
Ph
O
O
9
8
7
6
5
4
3
2
1
0
ppm
4.01
1.02
2.02
2.02
1.00
1.07
1.08
1.91
1.10
3.04
3.01

3d


176.02
143.51
143.42
128.73
128.18
124.69
85.64
77.55
77.23
76.91
62.36
62.29
61.88
61.82
39.91
38.53
33.89
33.86
28.72
16.43
16.37
16.30
16.24
O
P(OEt)2
Ph
O
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3e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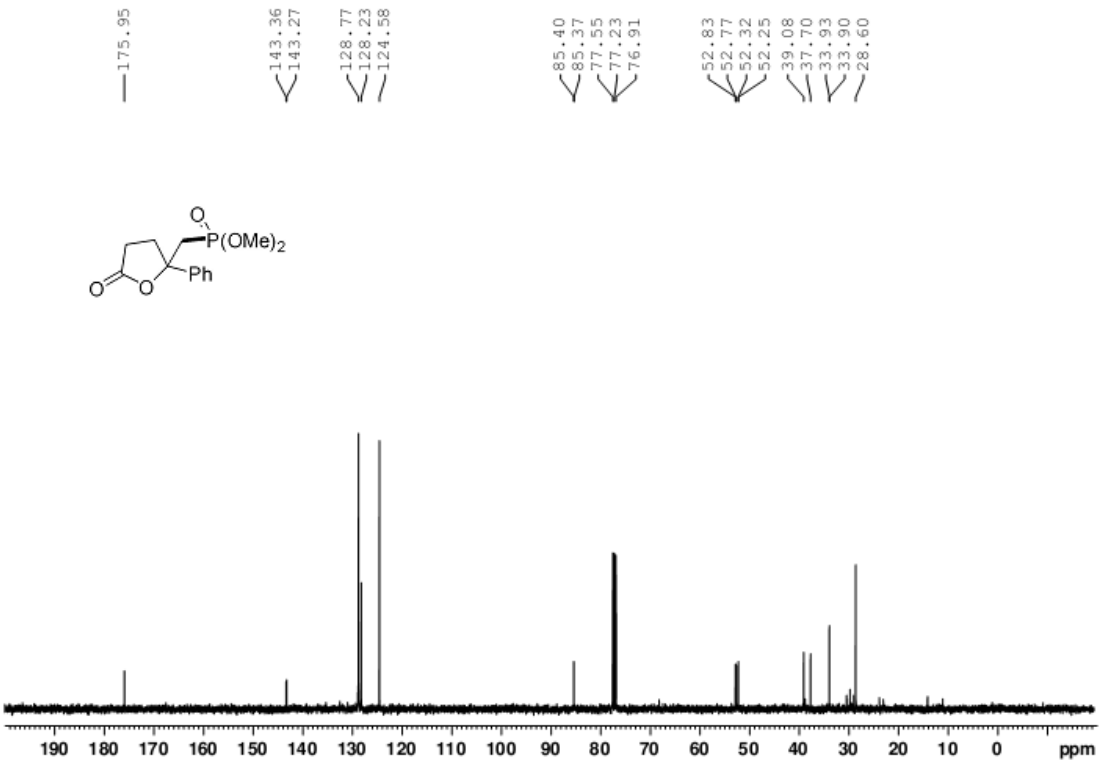
7.392
7.386
7.375
7.370
7.351
7.332
7.303
7.298
7.293
7.288
7.281
7.274
7.267
7.265
7.260
3.700
3.673
3.526
3.498
3.032
3.010
3.001
2.988
2.979
2.956
2.721
2.710
2.698
2.687
2.678
2.667
2.655
2.644
2.612
2.600
2.589
2.580
2.578
2.569
2.556
2.551
2.546
2.504
2.500
2.464
2.441
2.419
2.398
2.375
O
P(OMe)2
Ph
O
O
9
8
7
6
5
4
3
2
1
0
ppm
4.00
1.00
3.05
3.01
1.05
1.08
1.03
2.08
1.12

3e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143.36
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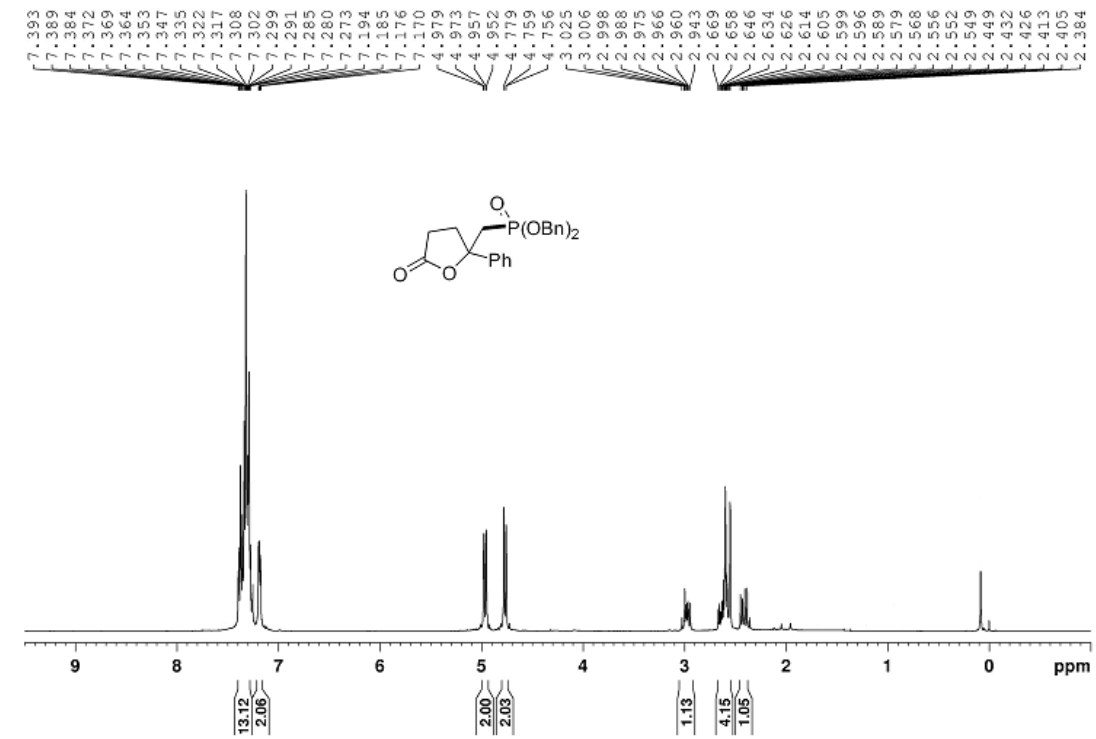
128.23
124.58
85.40
85.37
77.55
77.23
76.91
52.83
52.77
52.32
52.25
39.08
37.70
33.93
33.90
28.60
O
P(OMe)2
Ph
O
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3f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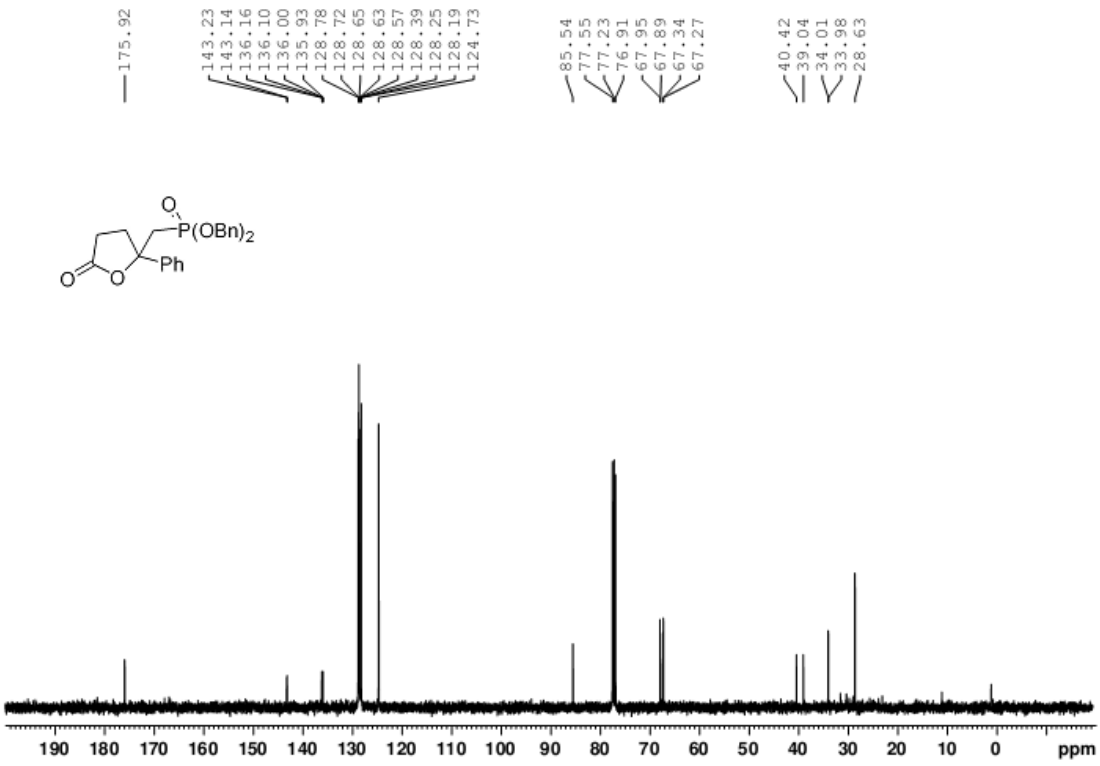
7.389
7.384
7.372
7.369
7.364
7.353
7.347
7.335
7.322
7.317
7.308
7.302
7.299
7.291
7.285
7.280
7.273
7.194
7.185
7.176
7.170
4.979
4.973
4.957
4.952
4.779
4.759
4.756
3.025
3.006
2.998
2.988
2.975
2.966
2.960
2.943
2.669
2.658
2.646
2.634
2.626
2.614
2.605
2.599
2.596
2.589
2.579
2.568
2.556
2.552
2.549
2.449
2.432
2.426
2.413
2.405
2.384
O
P(OBn)2
Ph
O
O
9
8
7
6
5
4
3
2
1
0
ppm
13.12
2.06
2.00
2.03
1.13
4.15
1.05

3f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136.16
136.10
136.00
135.93
128.78
128.72
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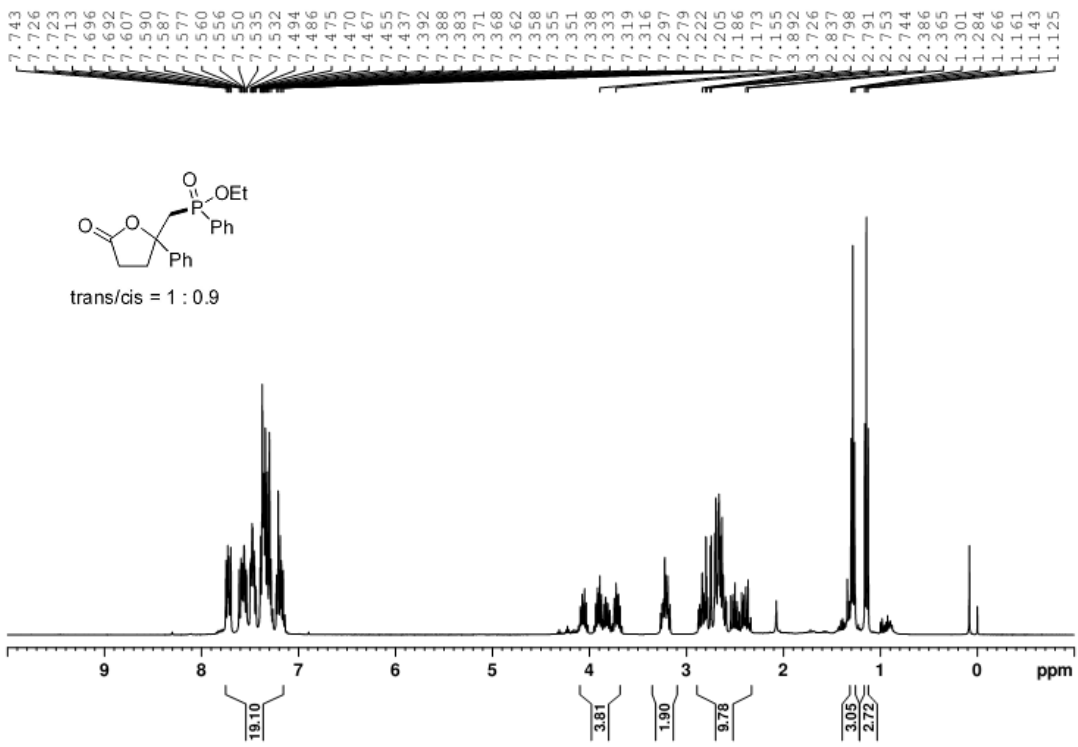
128.63
128.57
128.39
128.25
128.19
124.73
85.54
77.55
77.23
76.91
67.95
67.89
67.34
67.27
40.42
39.04
34.01
33.98
28.63
O
P(OBn)2
Ph
O
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3g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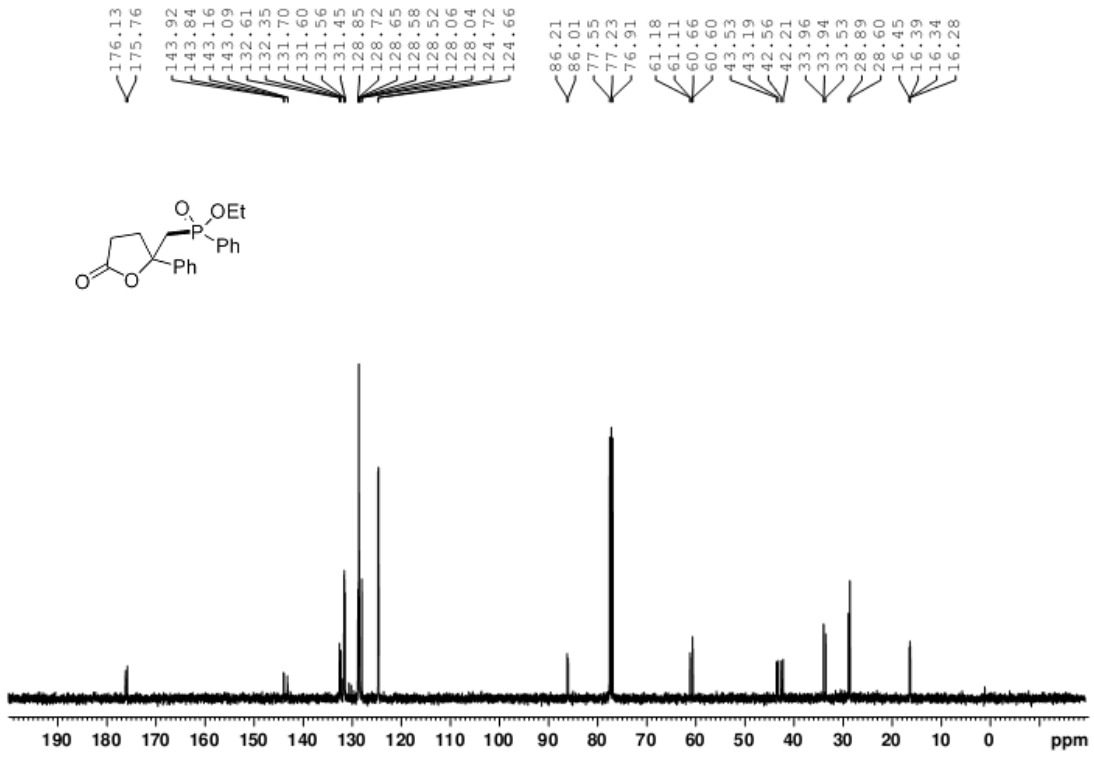
7.723
7.713
7.696
7.692
7.607
7.590
7.587
7.577
7.560
7.556
7.550
7.535
7.532
7.494
7.486
7.475
7.470
7.467
7.455
7.437
7.392
7.388
7.383
7.371
7.368
7.362
7.358
7.355
7.351
7.338
7.333
7.319
7.316
7.297
7.279
7.222
7.205
7.186
7.173
7.155
3.892
3.726
2.837
2.798
2.791
2.753
2.744
2.386
2.365
1.301
1.284
1.266
1.161
1.143
1.125
OEt
P
Ph
O
O
Ph
trans/cis = 1 : 0.9
9
8
7
6
5
4
3
2
1
0
ppm
19.10
3.81
1.90
9.78
3.05
2.72

3g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143.09
132.61
132.35
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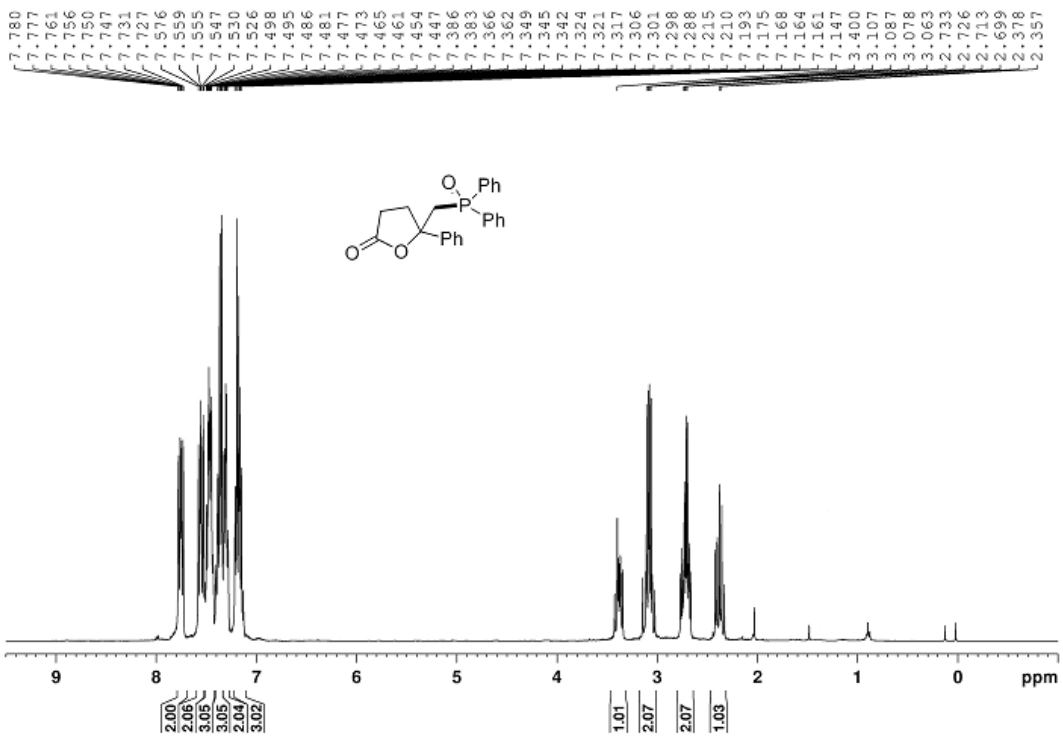
131.60
131.56
131.45
128.85
128.72
128.65
128.58
128.52
128.06
128.04
124.72
124.66
86.21
86.01
77.55
77.23
76.91
61.18
61.11
60.66
60.60
43.53
43.19
42.56
42.21
33.96
33.94
33.53
28.89
28.60
16.45
16.39
16.34
16.28
O
OEt
P
Ph
O
O
Ph
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3h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7.727
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7.547
7.530
7.526
7.498
7.495
7.486
7.481
7.477
7.473
7.465
7.461
7.454
7.447
7.386
7.383
7.366
7.362
7.349
7.345
7.342
7.324
7.321
7.317
7.306
7.301
7.298
7.288
7.215
7.210
7.193
7.175
7.168
7.164
7.161
7.147
3.400
3.107
3.087
3.078
3.063
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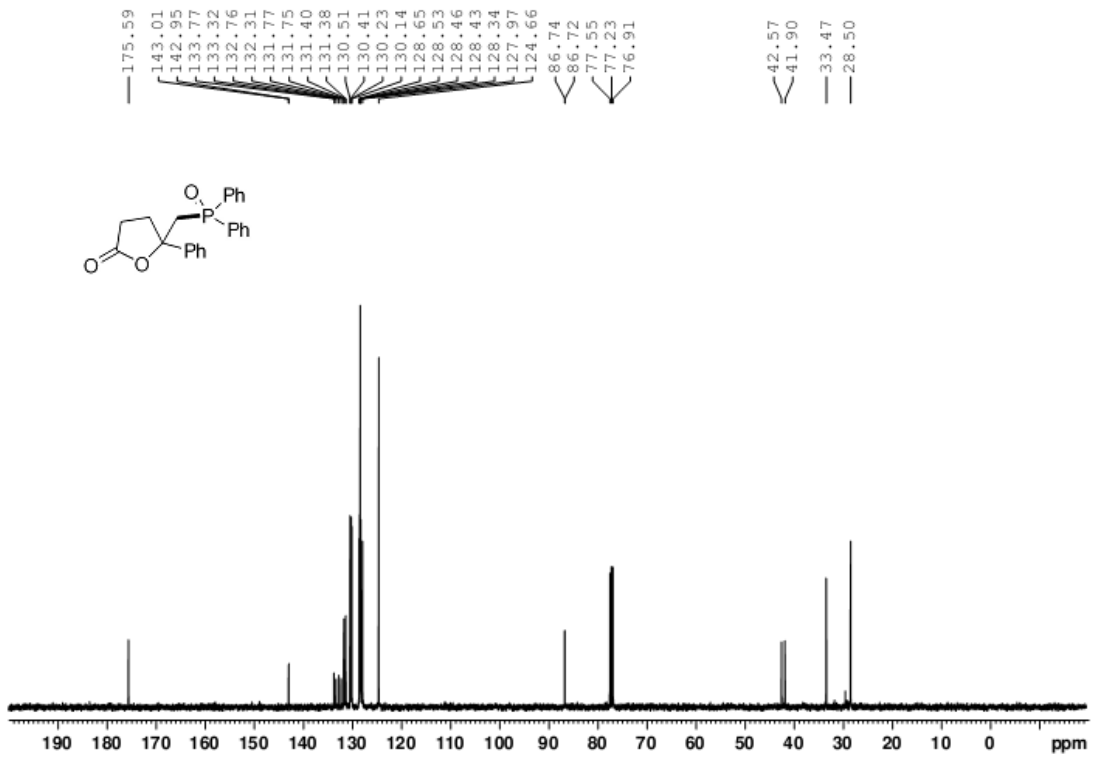
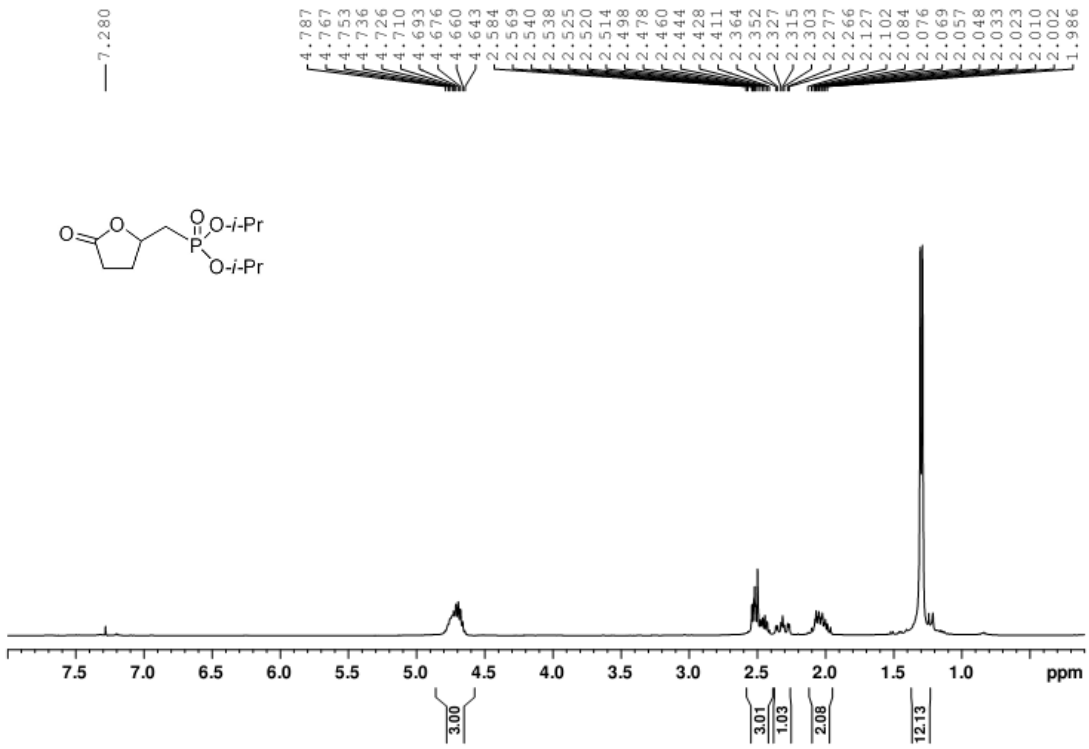
2.713
2.699
2.378
2.357
O
P
Ph
Ph
Ph
O
O
9
8
7
6
5
4
3
2
1
0
ppm
2.00
2.06
3.05
3.05
2.04
3.02
1.01
2.07
2.07
1.03

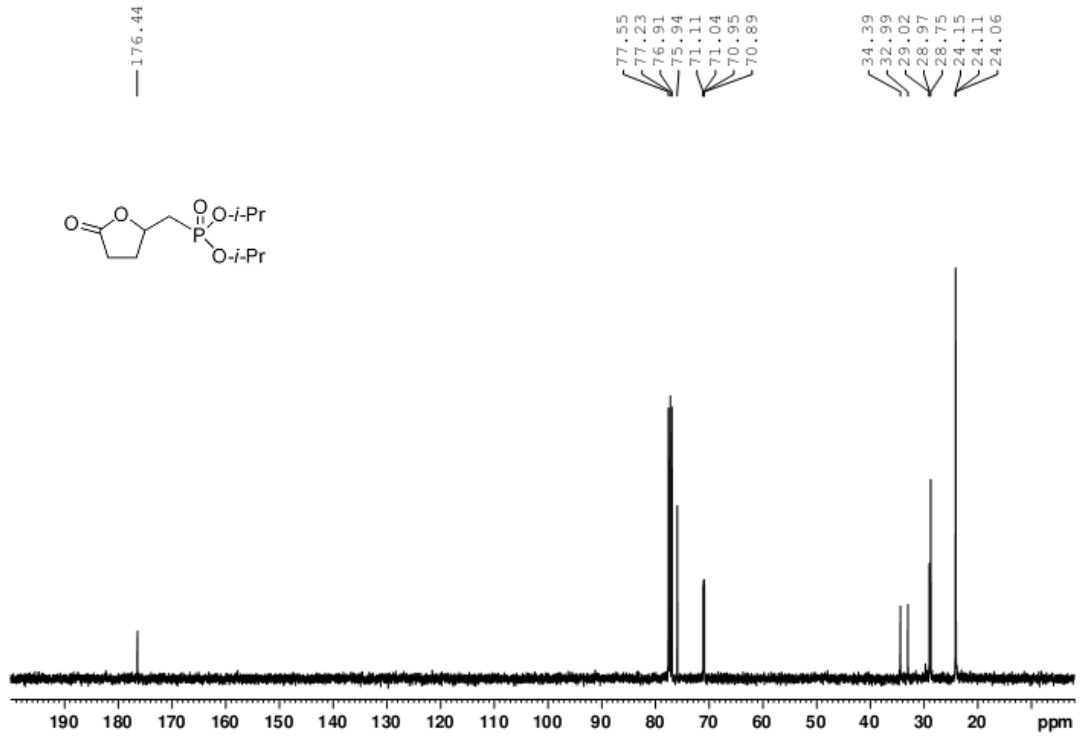
3h


175.59
143.01
142.95
133.77
133.32
132.76
132.31
131.77
131.75
131.40
131.38
130.51
130.41
130.23
130.14
128.65
128.53
128.46
128.43
128.34
127.97
124.66
86.74
86.72
77.55
77.23
76.91
42.57
41.90
33.47
28.50
O
P
Ph
Ph
Ph
O
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3i


7.280
4.787
4.767
4.753
4.736
4.726
4.710
4.693
4.676
4.660
4.643
2.584
2.569
2.540
2.538
2.525
2.520
2.514
2.498
2.478
2.460
2.444
2.428
2.411
2.364
2.352
2.327
2.315
2.303
2.277
2.266
2.127
2.102
2.084
2.076
2.069
2.057
2.048
2.033
2.023
2.010
2.002
1.986
O-i-Pr
O-i-Pr
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
ppm
3.00
3.01
1.03
2.08
12.13

3i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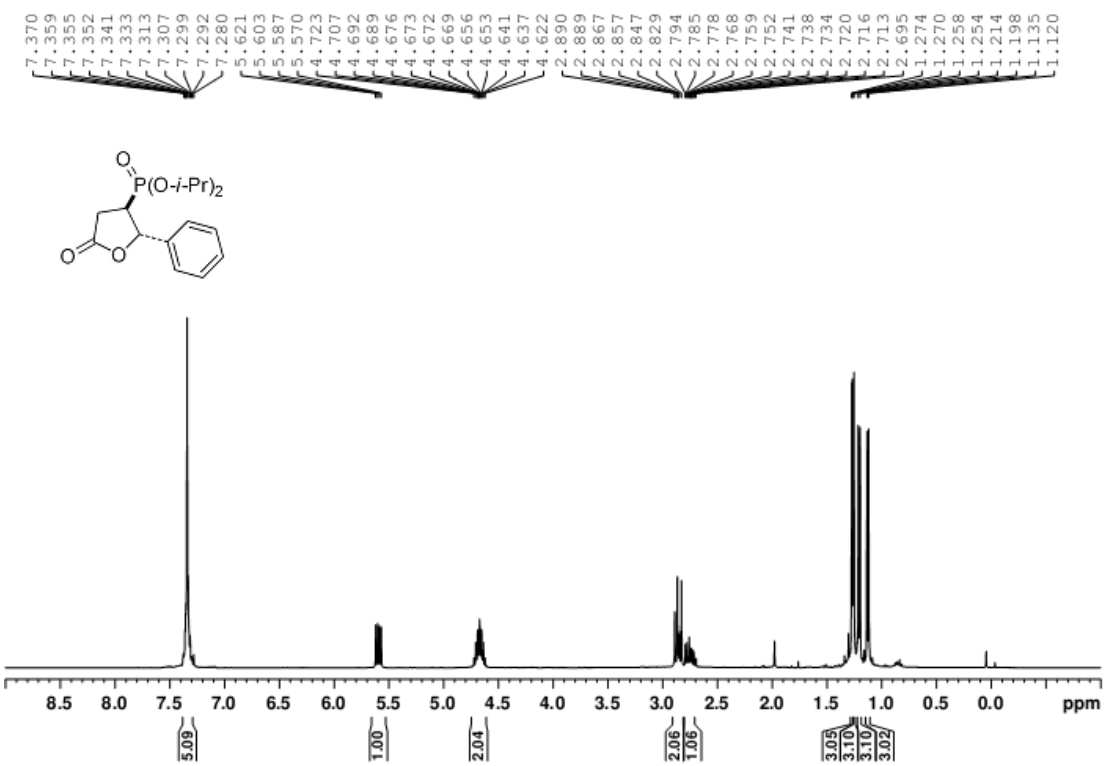
77.55
77.23
76.91
75.94
71.11
71.04
70.95
70.89
34.39
32.99
29.02
28.97
28.75
24.15
24.11
24.06
O-i-Pr
O-i-Pr
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

5a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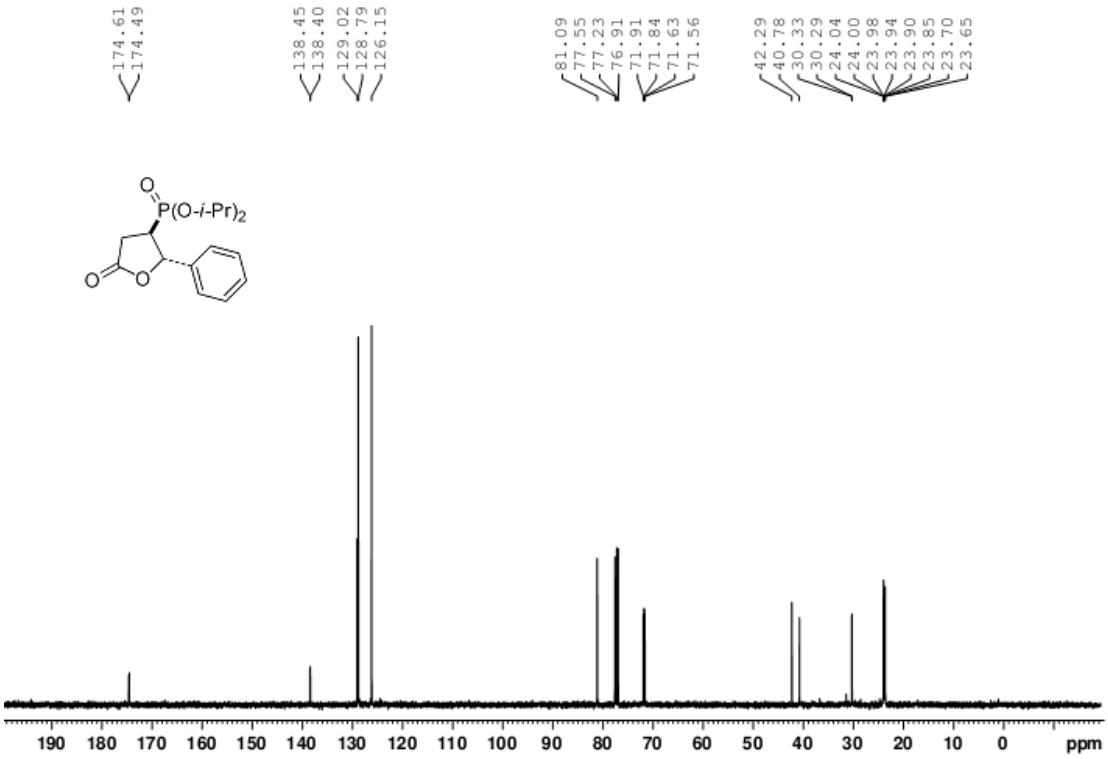
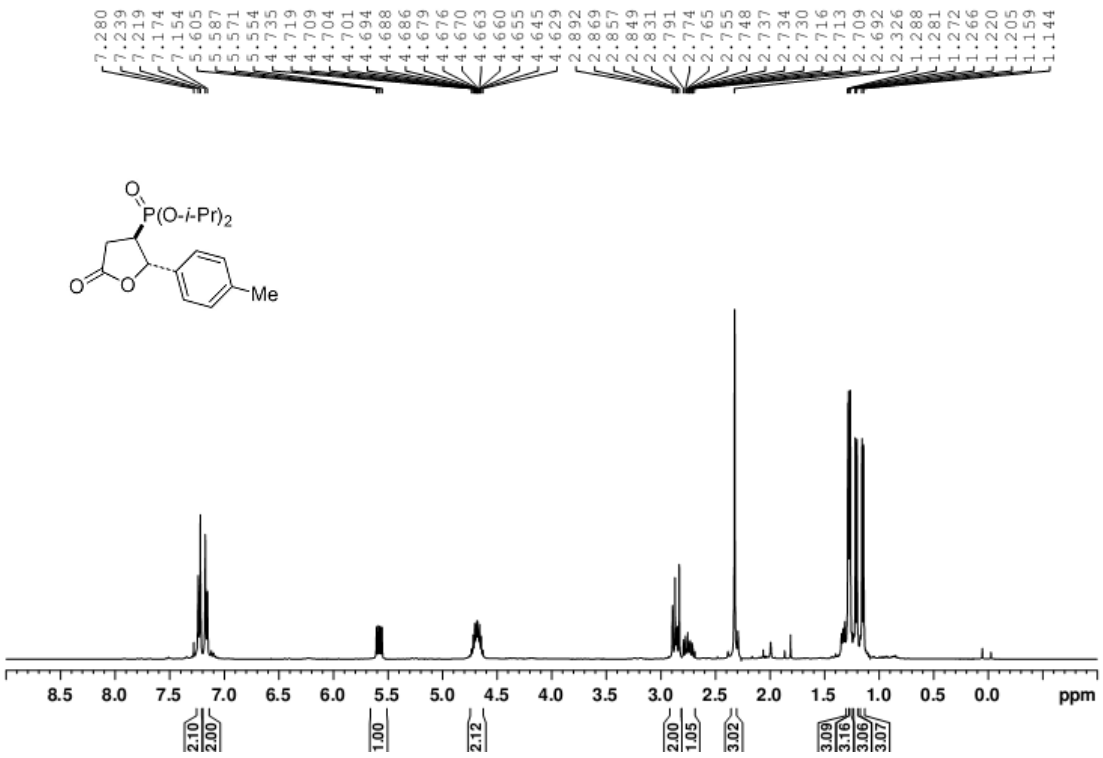
7.359
7.355
7.352
7.341
7.333
7.313
7.307
7.299
7.292
7.280
5.621
5.603
5.587
5.570
4.723
4.707
4.692
4.689
4.676
4.673
4.672
4.669
4.656
4.653
4.641
4.637
4.622
2.890
2.889
2.867
2.857
2.847
2.829
2.794
2.785
2.778
2.768
2.759
2.752
2.741
2.738
2.734
2.720
2.716
2.713
2.695
1.274
1.270
1.258
1.254
1.214
1.198
1.135
1.120
O
P(O-i-Pr)2
O
O
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 ppm
5.09
1.00
2.04
2.06
1.06
3.05
3.10
3.10
3.02

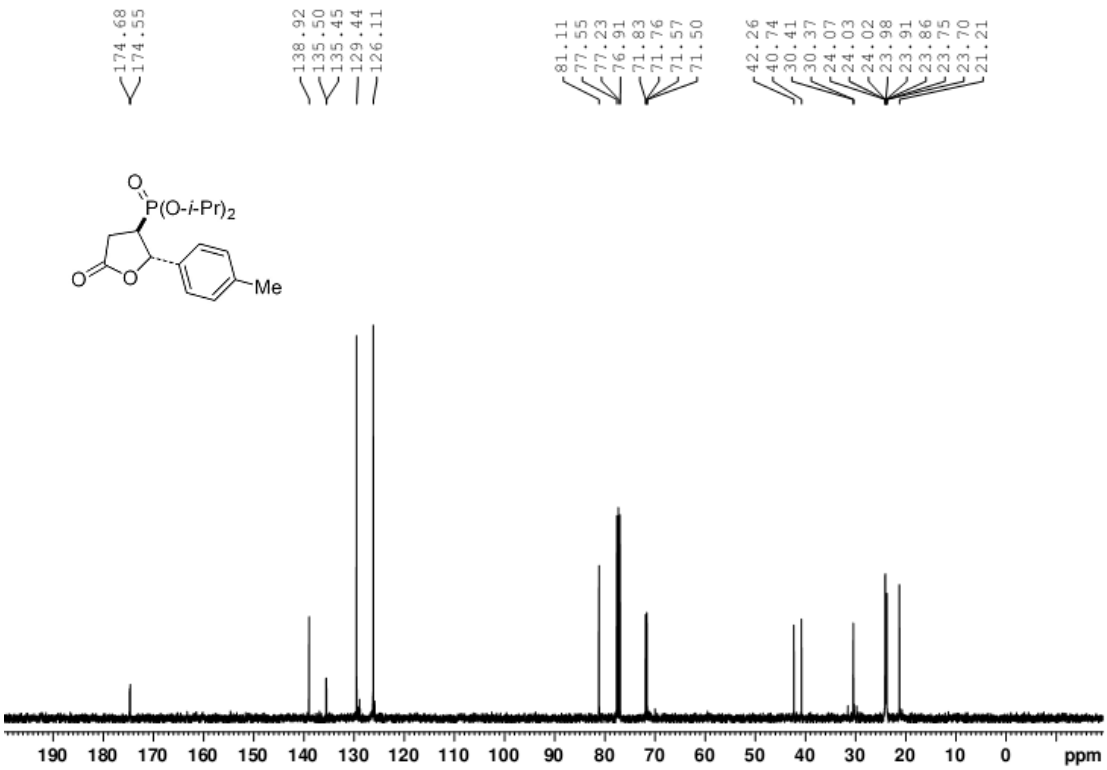
5a


174.61
174.49
138.45
138.40
129.02
128.79
126.15
81.09
77.55
77.23
76.91
71.91
71.84
71.63
71.56
42.29
40.78
30.33
30.29
24.04
24.00
23.98
23.94
23.90
23.85
23.70
23.65
O
P(O-i-Pr)2
O
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

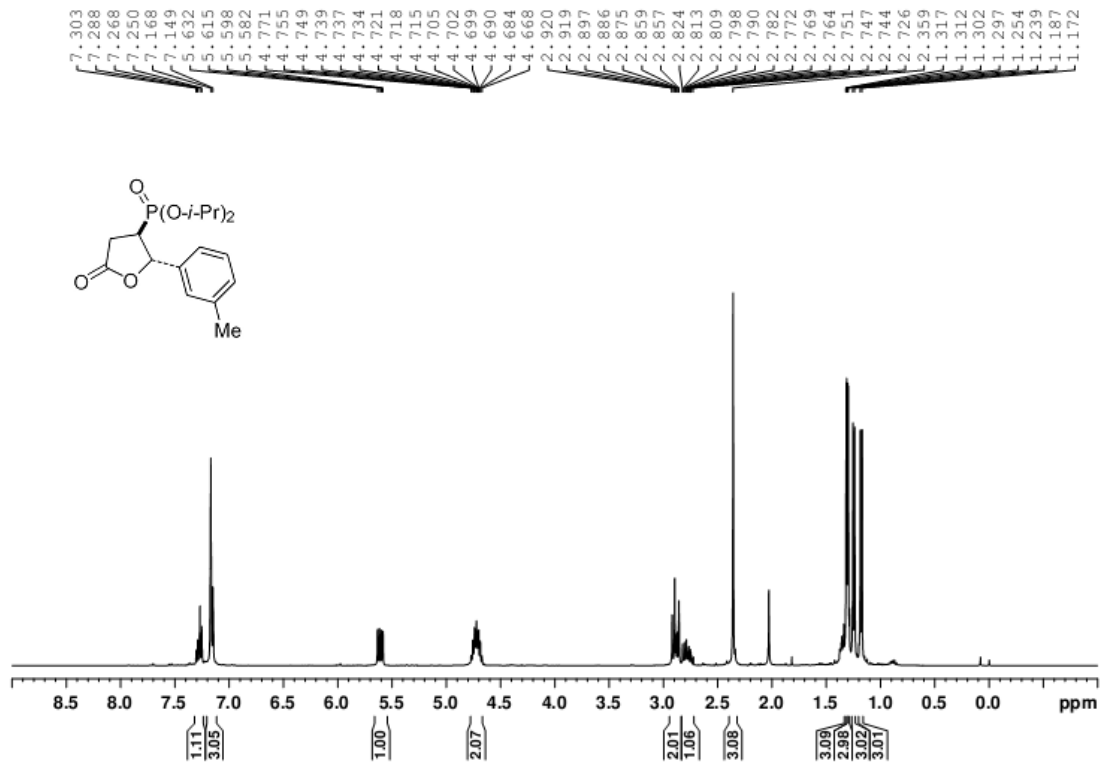
5b


7.280
7.239
7.219
7.174
7.154
5.605
5.587
5.571
5.554
4.735
4.719
4.709
4.704
4.701
4.694
4.688
4.686
4.679
4.676
4.670
4.663
4.660
4.655
4.645
4.629
2.892
2.869
2.857
2.849
2.831
2.791
2.774
2.765
2.755
2.748
2.737
2.734
2.730
2.716
2.713
2.709
2.692
2.326
1.288
1.281
1.272
1.266
1.220
1.205
1.159
1.144
P(O-i-Pr)2
Me
ppm

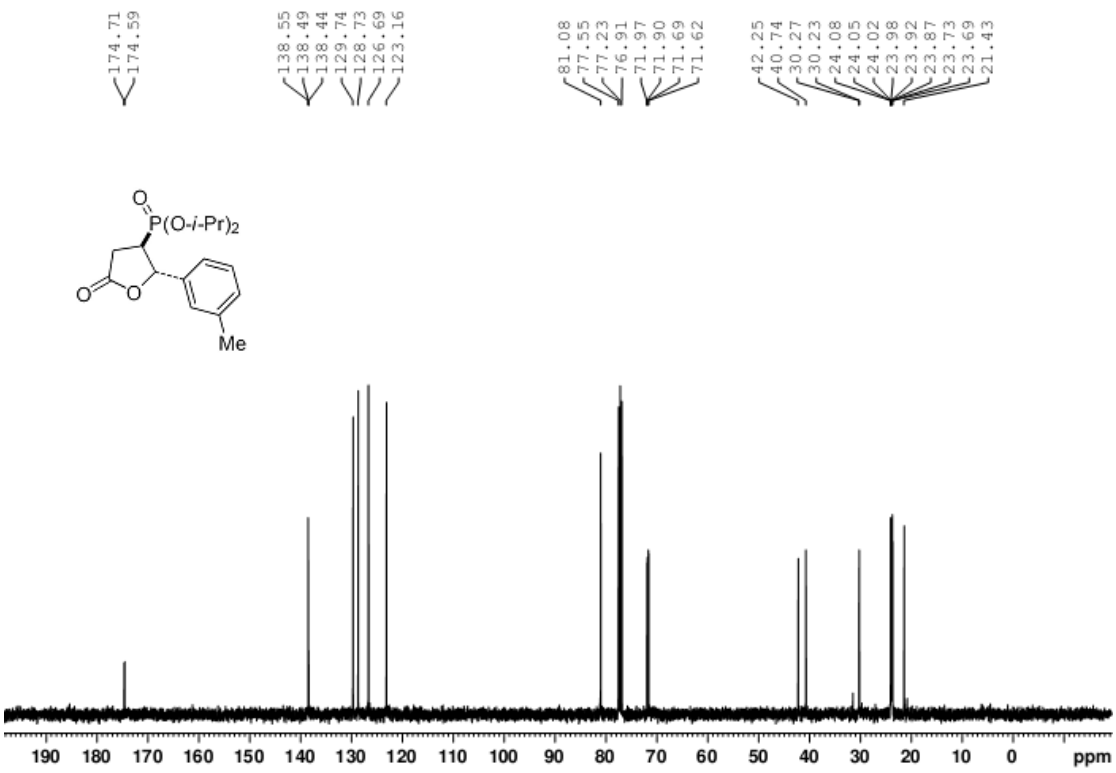
5b


174.68
174.55
138.92
135.50
135.45
129.44
126.11
81.11
77.55
77.23
76.91
71.83
71.76
71.57
71.50
42.26
40.74
30.41
30.37
24.07
24.03
24.02
23.98
23.91
23.86
23.75
23.70
21.21
P(O-i-Pr)2
Me
ppm

5c


7.303
7.288
7.268
7.250
7.168
7.149
5.632
5.615
5.598
5.582
4.771
4.755
4.749
4.739
4.737
4.734
4.721
4.718
4.715
4.705
4.702
4.699
4.690
4.684
4.668
2.920
2.919
2.897
2.886
2.875
2.859
2.857
2.824
2.813
2.809
2.798
2.790
2.782
2.772
2.769
2.764
2.751
2.747
2.744
2.726
2.359
1.317
1.312
1.302
1.297
1.254
1.239
1.187
1.172
P(O-i-Pr)2
O
O
Me
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
ppm
1.11
3.05
1.00
2.07
2.01
1.06
3.08
3.09
2.98
3.02
3.01

5c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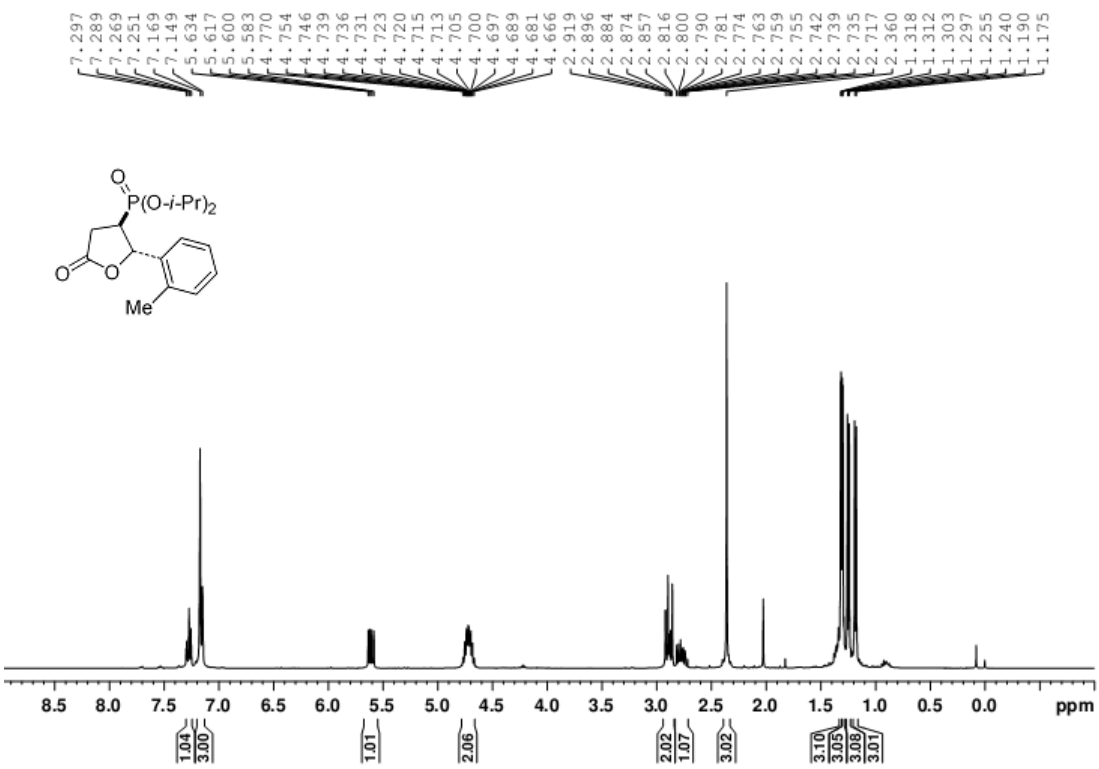
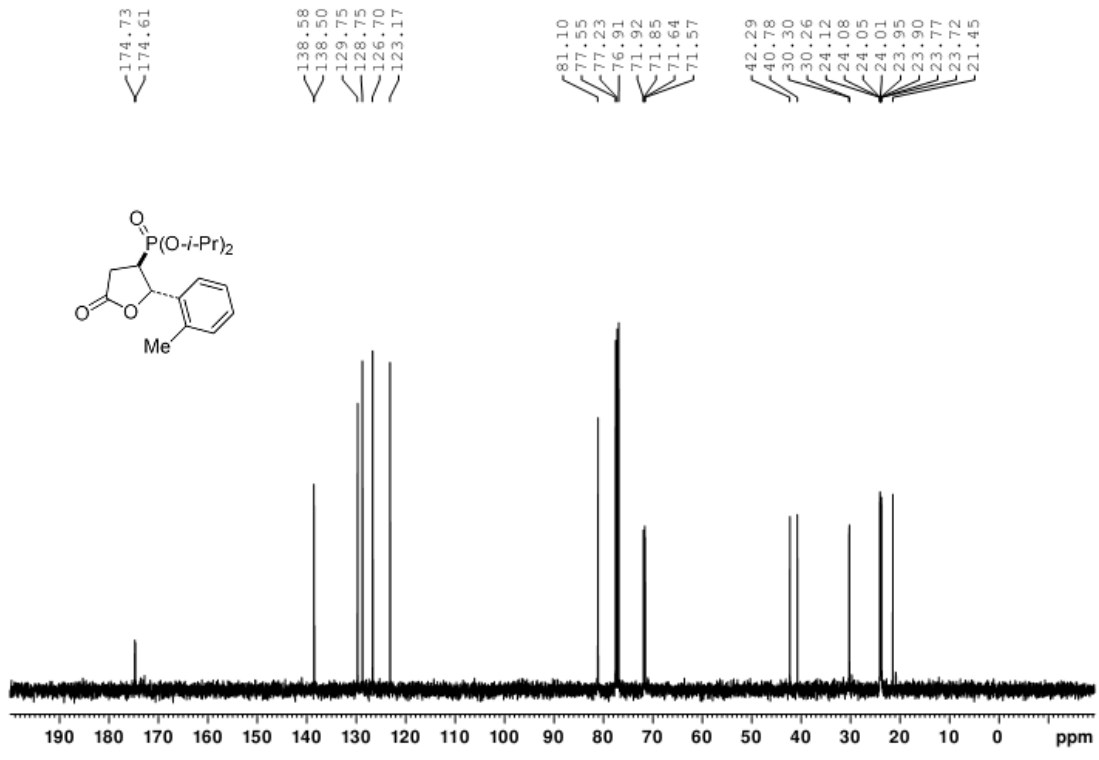
174.59
138.55
138.49
138.44
129.74
128.73
126.69
123.16
81.08
77.55
77.23
76.91
71.97
71.90
71.69
71.62
42.25
40.74
30.27
30.23
24.08
24.05
24.02
23.98
23.92
23.87
23.73
23.69
21.43
P(O-i-Pr)2
O
O
Me
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

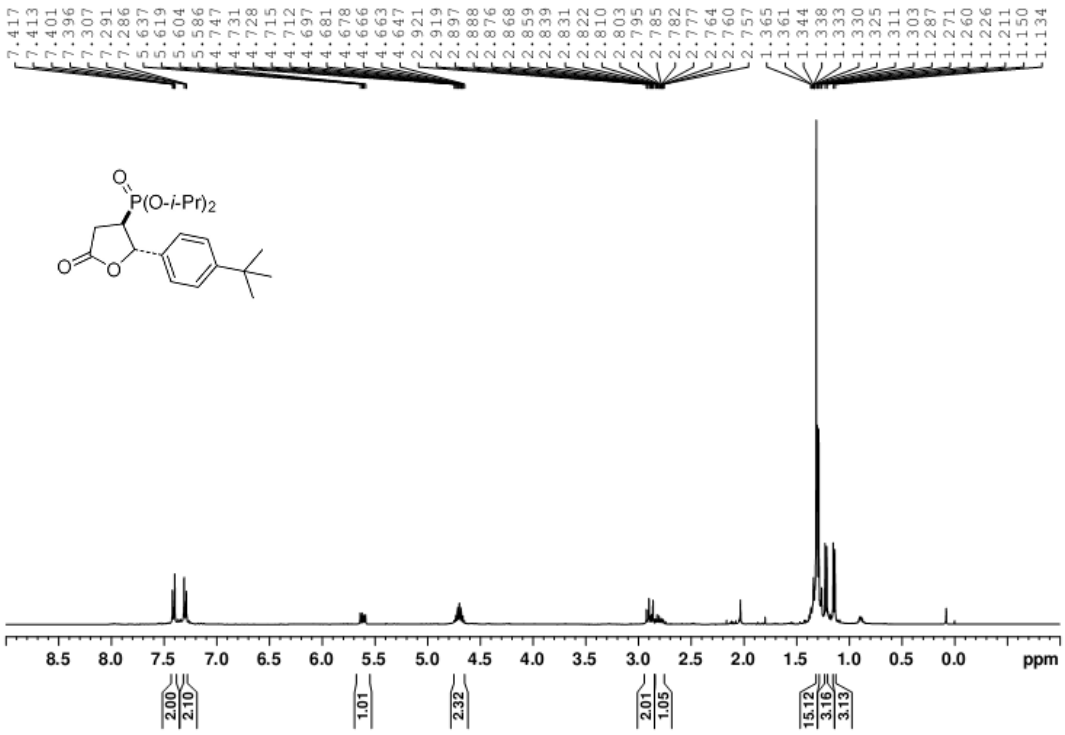
5d


7.297
7.289
7.269
7.251
7.169
7.149
5.634
5.617
5.600
5.583
4.770
4.754
4.746
4.739
4.736
4.731
4.723
4.720
4.715
4.713
4.705
4.700
4.697
4.689
4.681
4.666
2.919
2.896
2.884
2.874
2.857
2.816
2.800
2.790
2.781
2.774
2.763
2.759
2.755
2.742
2.739
2.735
2.717
2.360
1.318
1.312
1.303
1.297
1.255
1.240
1.190
1.175
P(O-i-Pr)2
Me
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 ppm
1.04
3.00
1.01
2.06
2.02
1.07
3.02
3.10
3.05
3.08
3.01

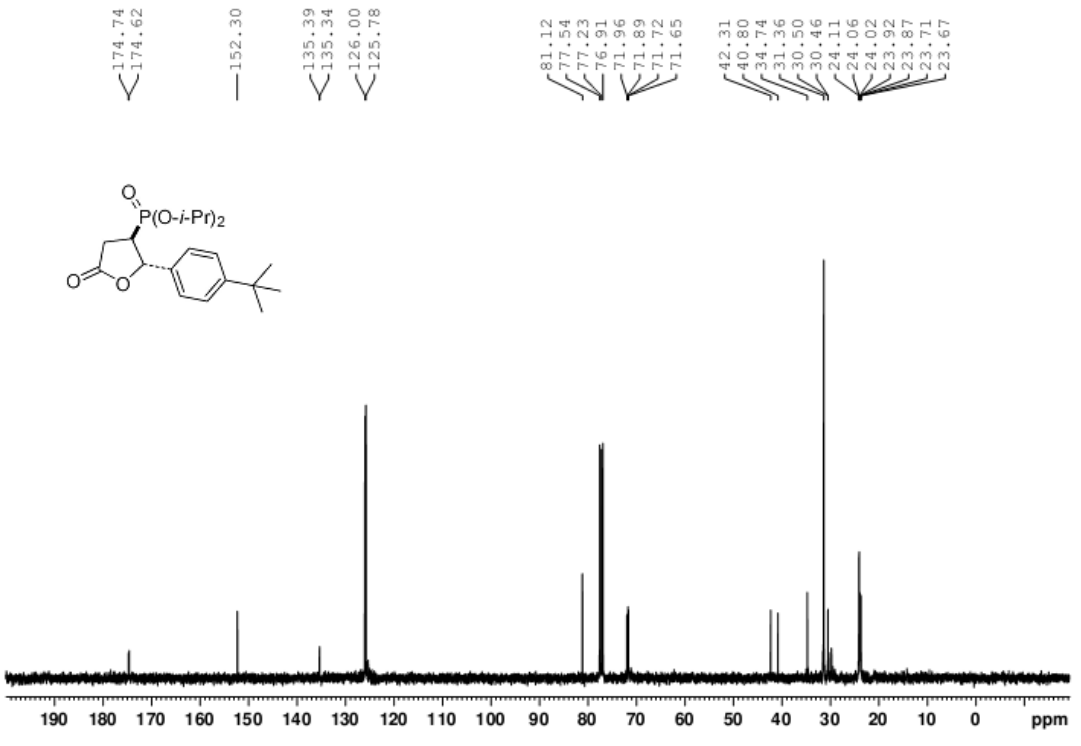
5d


174.73
174.61
138.58
138.50
129.75
128.75
126.70
123.17
81.10
77.55
77.23
76.91
71.92
71.85
71.64
71.57
42.29
40.78
30.30
30.26
24.12
24.08
24.05
24.01
23.95
23.90
23.77
23.72
21.45
P(O-i-Pr)2
Me
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

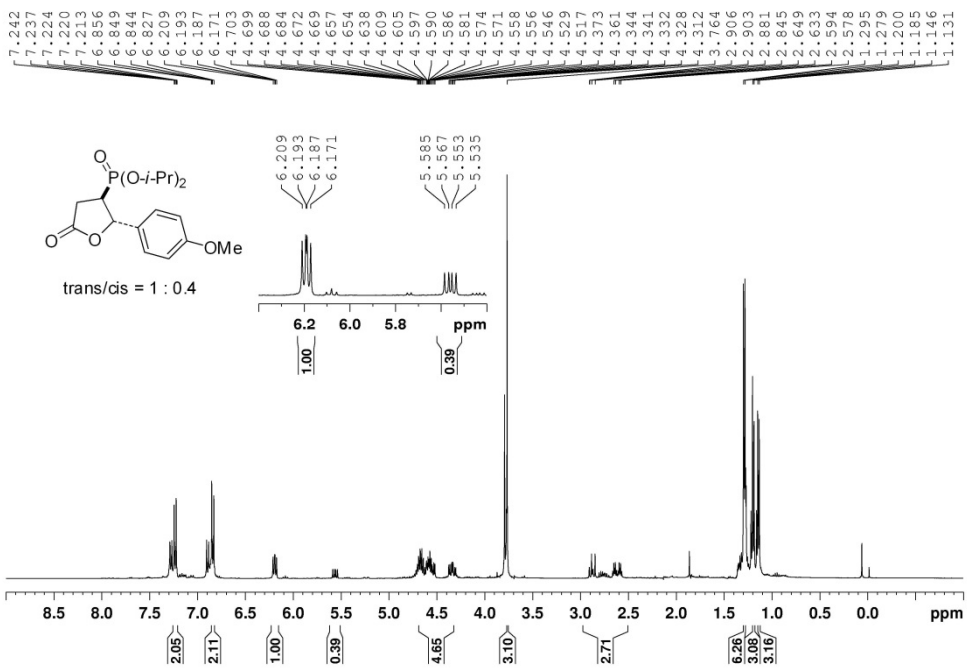
5e


7.417
7.413
7.401
7.396
7.307
7.291
7.286
5.637
5.619
5.604
5.586
4.747
4.731
4.728
4.715
4.712
4.697
4.681
4.678
4.666
4.663
4.647
2.921
2.919
2.897
2.888
2.876
2.868
2.859
2.839
2.831
2.822
2.810
2.803
2.795
2.785
2.782
2.777
2.764
2.760
2.757
1.365
1.361
1.344
1.338
1.333
1.330
1.325
1.311
1.303
1.287
1.271
1.260
1.226
1.211
1.150
1.134
P(O-i-Pr)2
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 ppm
2.00
2.10
1.01
2.32
2.01
1.05
15.12
3.16
3.13

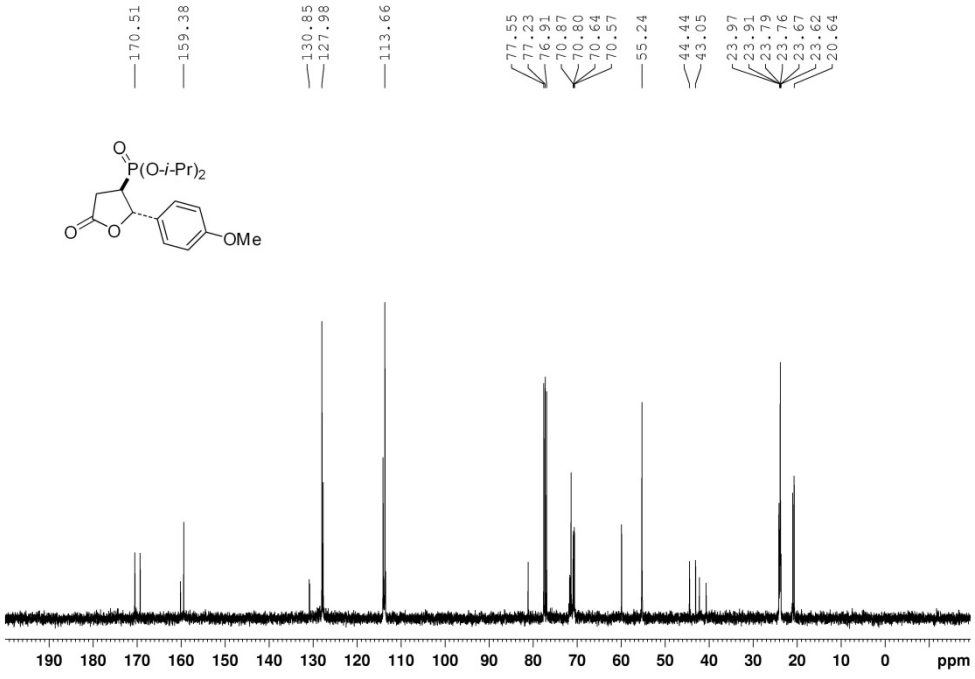
5e


174.74
174.62
152.30
135.39
135.34
126.00
125.78
81.12
77.54
77.23
76.91
71.96
71.89
71.72
71.65
42.31
40.80
34.74
31.36
30.50
30.46
24.11
24.06
24.02
23.92
23.87
23.71
23.67
P(O-i-Pr)2
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

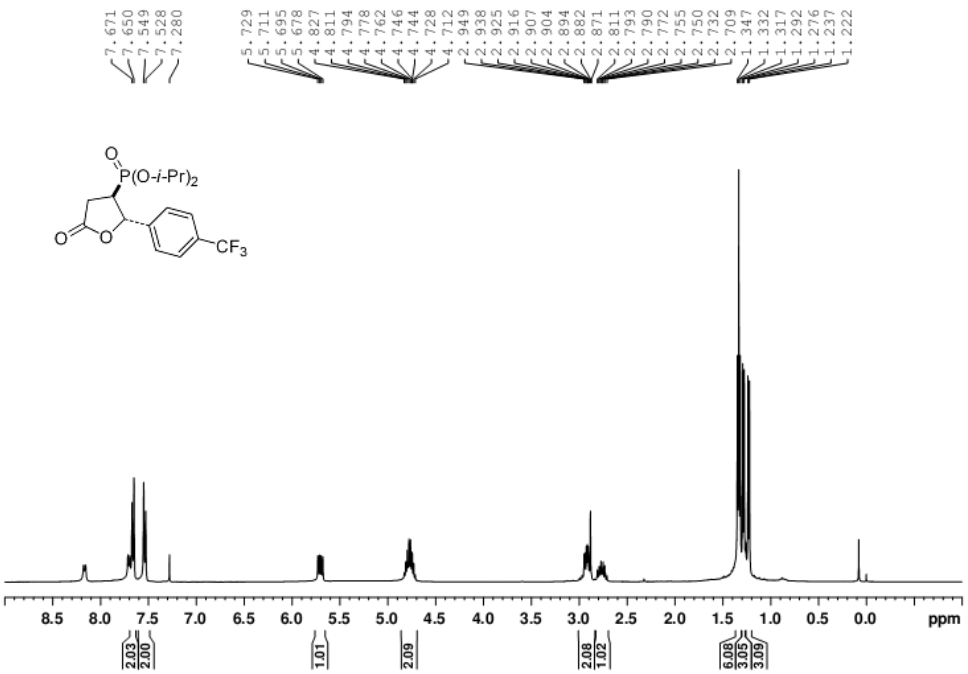
5f


7.242
7.237
7.224
7.220
7.213
6.856
6.849
6.844
6.827
6.209
6.193
6.187
6.171
4.703
4.699
4.688
4.684
4.672
4.669
4.657
4.654
4.638
4.609
4.605
4.597
4.590
4.586
4.581
4.574
4.571
4.558
4.556
4.546
4.529
4.517
4.373
4.361
4.344
4.341
4.332
4.328
4.312
3.764
2.906
2.903
2.881
2.845
2.649
2.633
2.594
2.578
1.295
1.279
1.200
1.185
1.146
1.131
P(O-i-Pr)2
OMe
trans/cis = 1 : 0.4
6.209
6.193
6.187
6.171
5.585
5.567
5.553
5.535
6.2
6.0
5.8
ppm
1.00
0.39
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
ppm
2.05
2.11
1.00
0.39
4.65
3.10
2.71
6.26
3.08
3.16

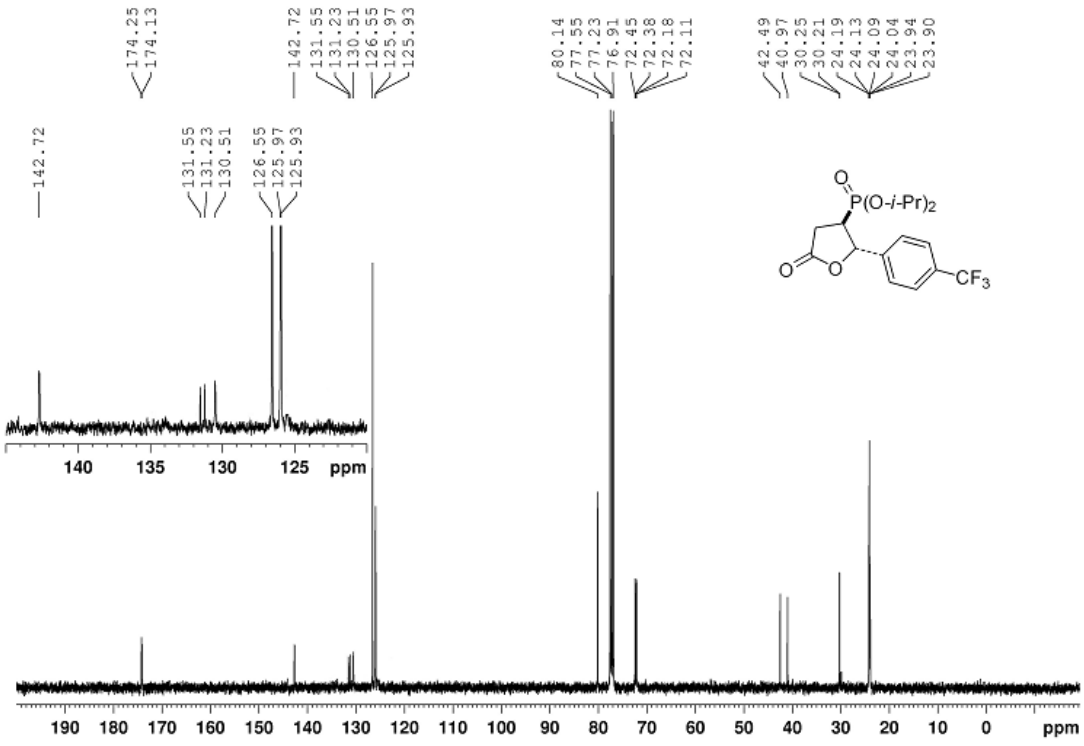
5f


170.51
159.38
130.85
127.98
113.66
77.55
77.23
76.91
70.87
70.80
70.64
70.57
55.24
44.44
43.05
23.97
23.91
23.79
23.76
23.67
23.62
20.64
P(O-i-Pr)2
OMe
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

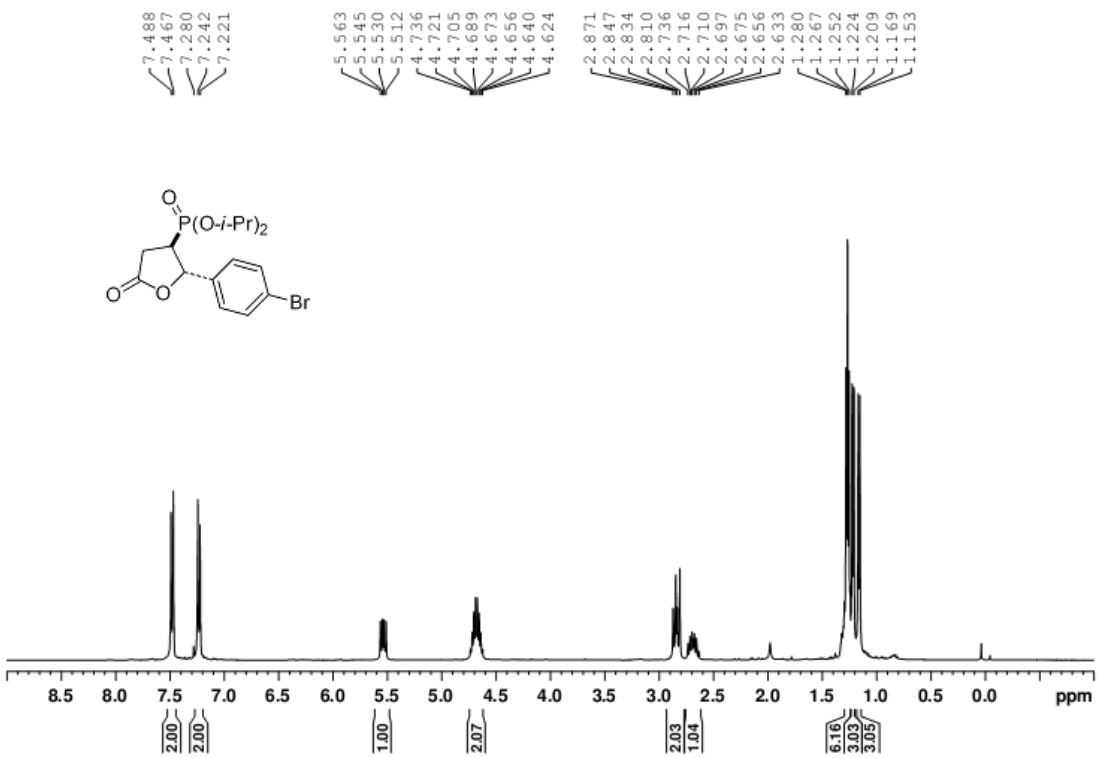
5g


7.671
7.650
7.549
7.528
7.280
5.729
5.711
5.695
5.678
4.827
4.811
4.794
4.778
4.762
4.746
4.744
4.728
4.712
2.949
2.938
2.925
2.916
2.907
2.904
2.894
2.882
2.871
2.811
2.793
2.790
2.772
2.755
2.750
2.732
2.709
1.347
1.332
1.317
1.292
1.276
1.237
1.222
P(O-i-Pr)2
CF3
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 ppm
2.03
2.00
1.01
2.09
2.08
1.02
6.08
3.05
3.09

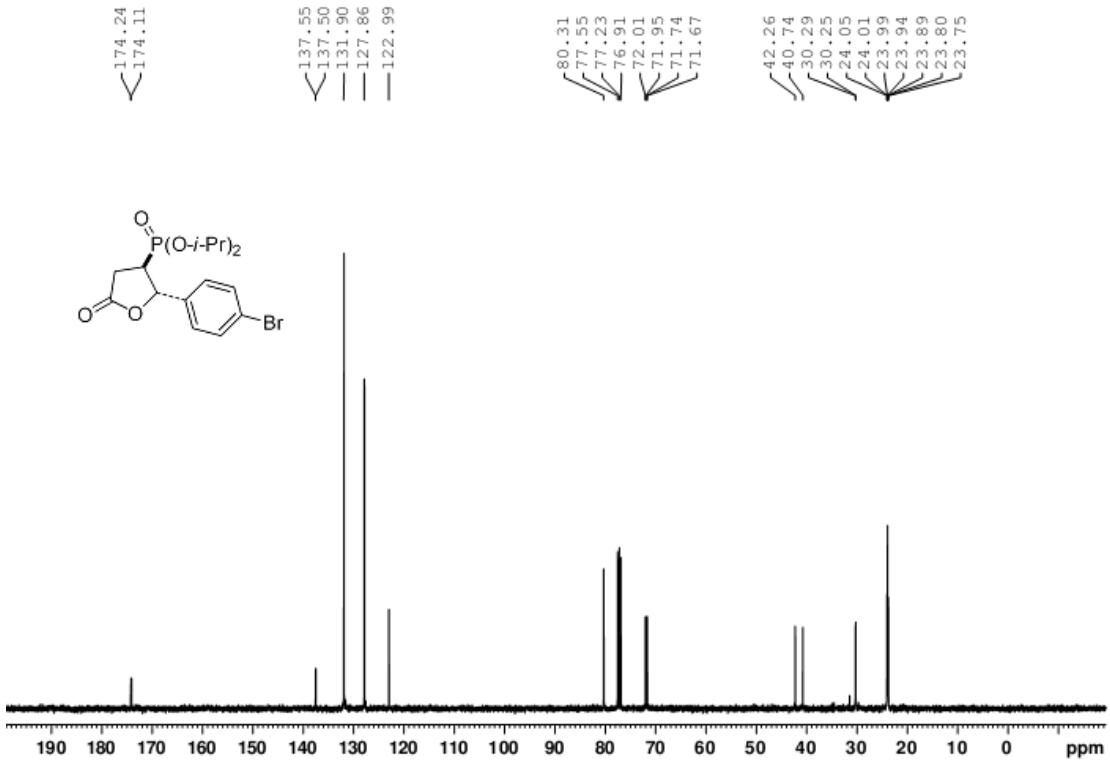
5g


174.25
174.13
142.72
131.55
131.23
130.51
126.55
125.97
125.93
80.14
77.55
77.23
76.91
72.45
72.38
72.18
72.11
42.49
40.97
30.25
30.21
24.19
24.13
24.09
24.04
23.94
23.90
P(O-i-Pr)2
CF3
140 135 130 125 ppm
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

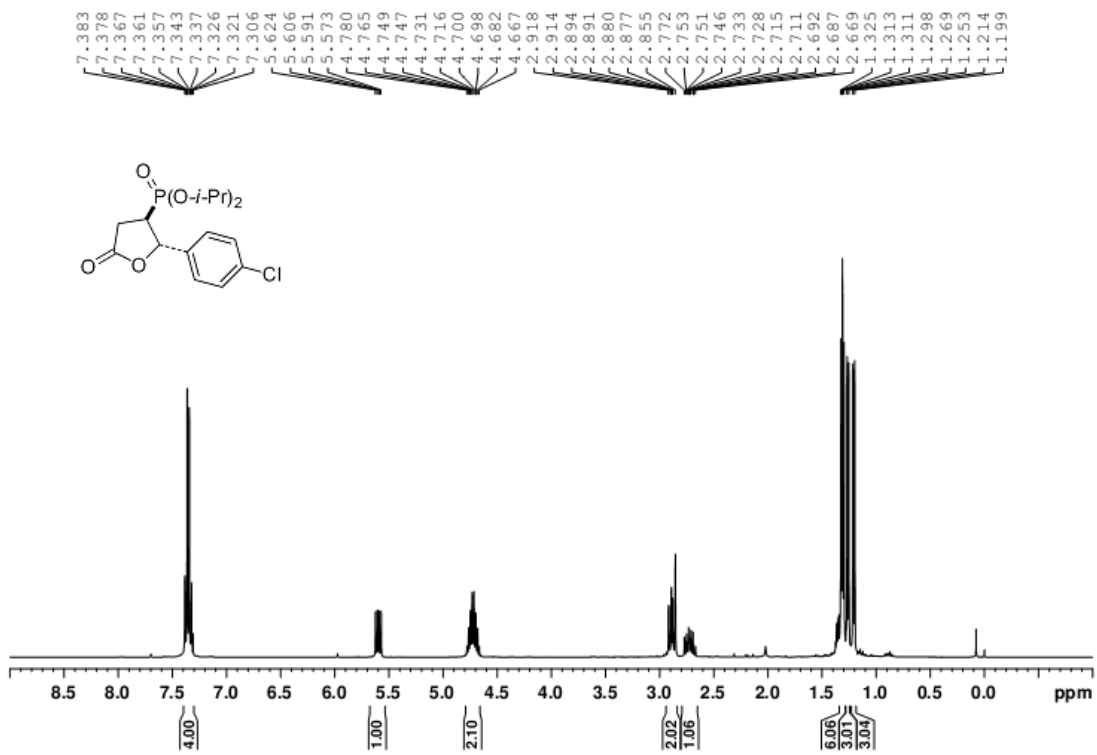
5h


7.488
7.467
7.280
7.242
7.221
5.563
5.545
5.530
5.512
4.736
4.721
4.705
4.689
4.673
4.656
4.640
4.624
2.871
2.847
2.834
2.810
2.736
2.716
2.710
2.697
2.675
2.656
2.633
1.280
1.267
1.252
1.224
1.209
1.169
1.153
O
P(O-i-Pr)2
O
O
Br
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 ppm
2.00
2.00
1.00
2.07
2.03
1.04
6.16
3.03
3.05

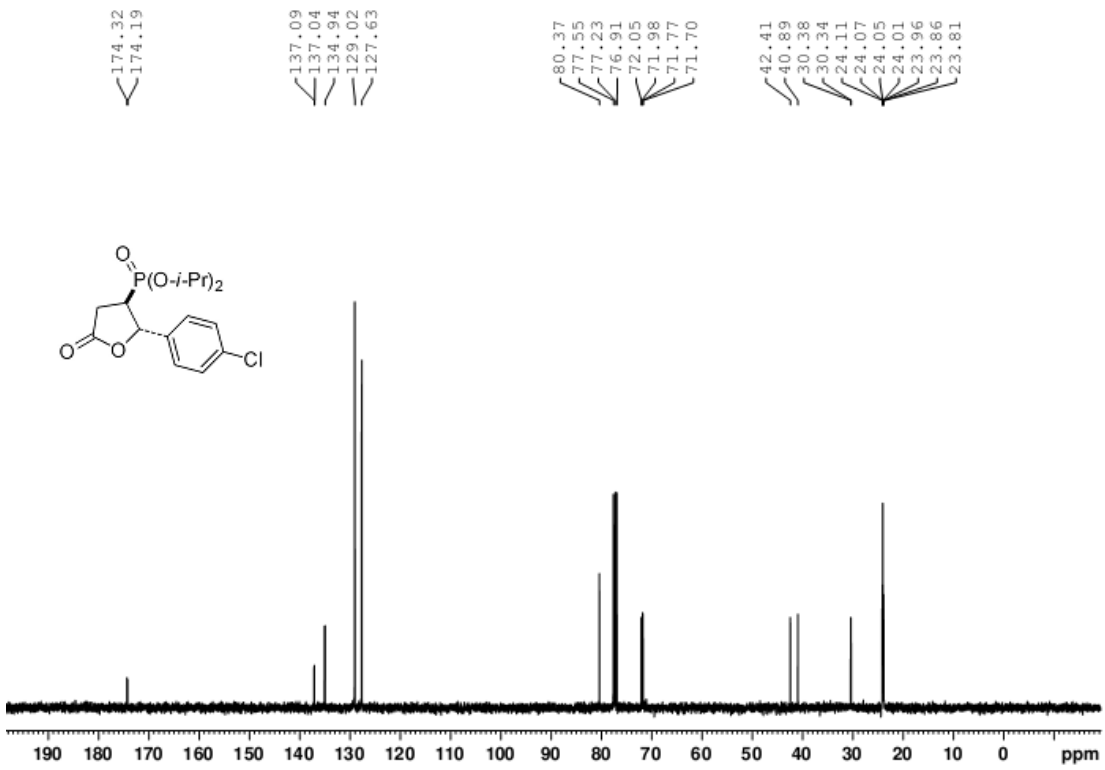
5h


174.24
174.11
137.55
137.50
131.90
127.86
122.99
80.31
77.55
77.23
76.91
72.01
71.95
71.74
71.67
42.26
40.74
30.29
30.25
24.05
24.01
23.99
23.94
23.89
23.80
23.75
O
P(O-i-Pr)2
O
O
Br
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

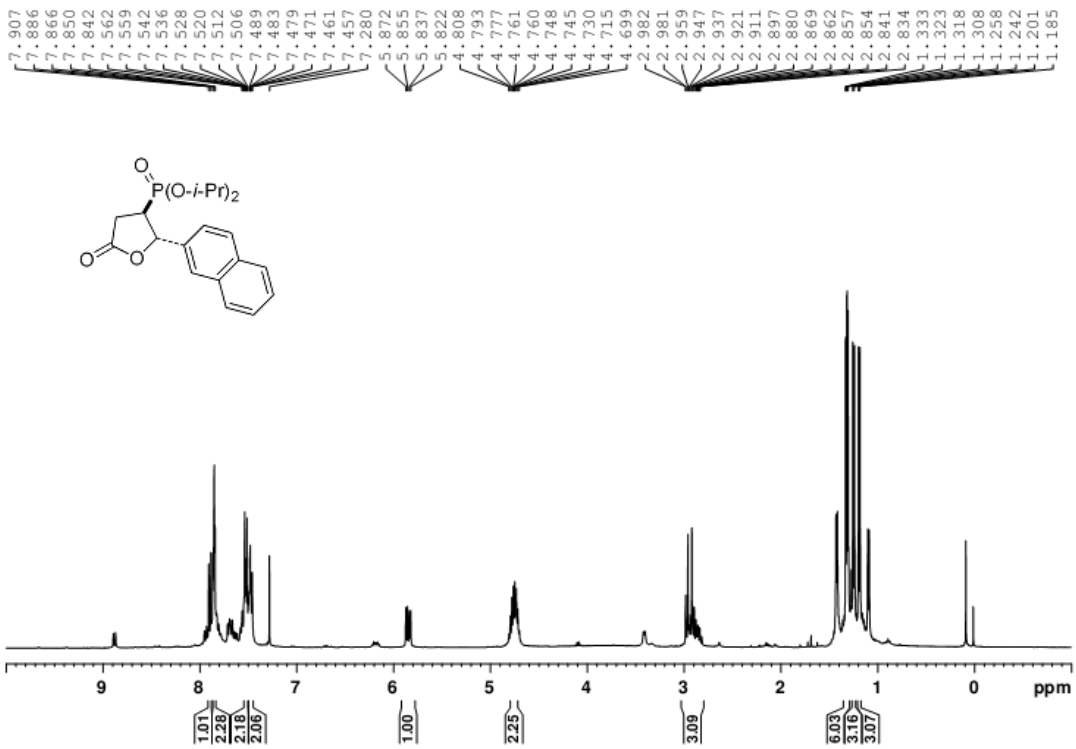
5i


7.383
7.378
7.367
7.361
7.357
7.343
7.337
7.326
7.321
7.306
5.624
5.606
5.591
5.573
4.780
4.765
4.749
4.747
4.731
4.716
4.700
4.698
4.682
4.667
2.918
2.914
2.894
2.891
2.880
2.877
2.855
2.772
2.753
2.751
2.746
2.733
2.728
2.715
2.711
2.692
2.687
2.669
1.325
1.313
1.311
1.298
1.269
1.253
1.214
1.199
P(O-i-Pr)2
O
O
Cl
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 ppm
4.00
1.00
2.10
2.02
1.06
6.06
3.01
3.04

5i


174.32
174.19
137.09
137.04
134.94
129.02
127.63
80.37
77.55
77.23
76.91
72.05
71.98
71.77
71.70
42.41
40.89
30.38
30.34
24.11
24.07
24.05
24.01
23.96
23.86
23.81
P(O-i-Pr)2
O
O
Cl
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

5j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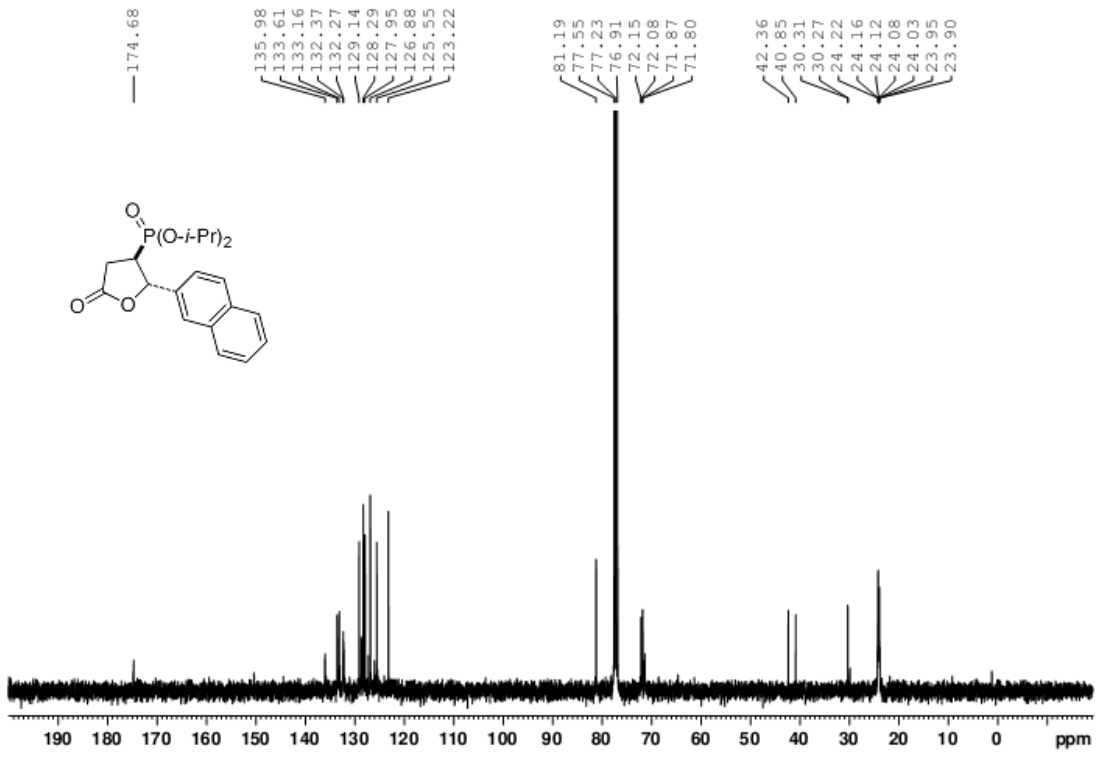
7.886
7.866
7.850
7.842
7.562
7.559
7.542
7.536
7.528
7.520
7.512
7.506
7.489
7.483
7.479
7.471
7.461
7.457
7.280
5.872
5.855
5.837
5.822
4.808
4.793
4.777
4.761
4.760
4.748
4.745
4.730
4.715
4.699
2.982
2.981
2.959
2.947
2.937
2.921
2.911
2.897
2.880
2.869
2.862
2.857
2.854
2.841
2.834
1.333
1.323
1.318
1.308
1.258
1.242
1.201
1.185
P(O-i-Pr)2
ppm
1.01
2.28
2.18
2.06
1.00
2.25
3.09
6.03
3.16
3.07

5j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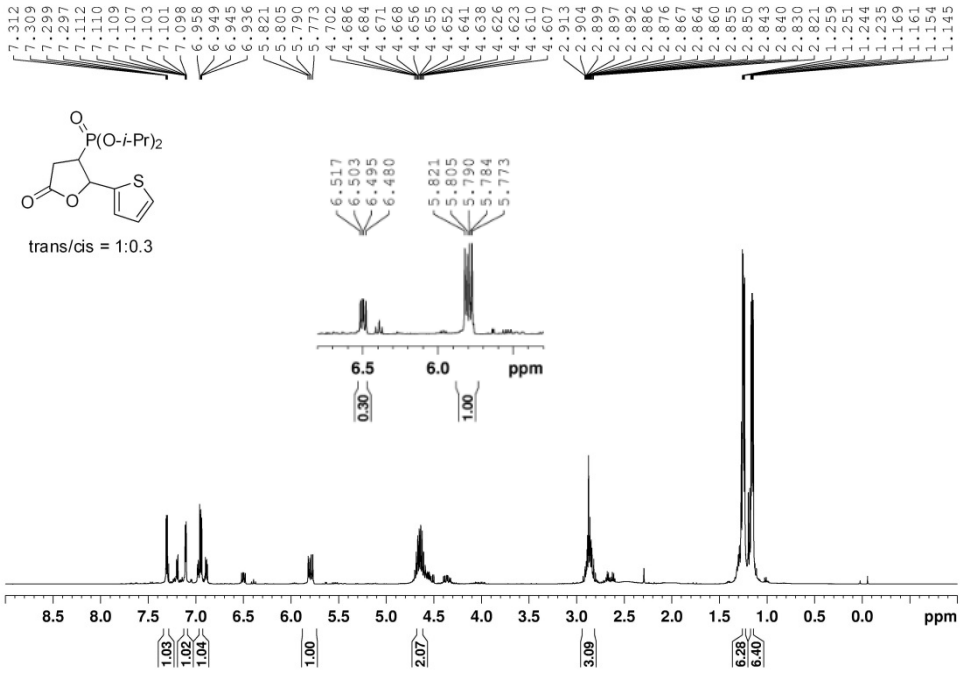
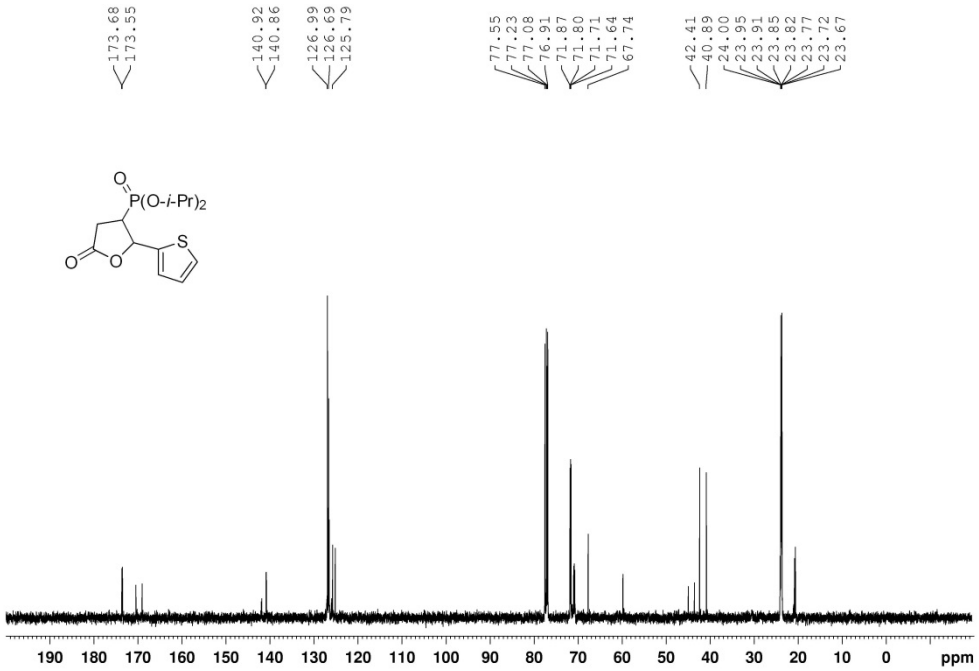
135.98
133.61
133.16
132.37
132.27
129.14
128.29
127.95
126.88
125.55
123.22
81.19
77.55
77.23
76.91
72.15
72.08
71.87
71.80
42.36
40.85
30.31
30.27
24.22
24.16
24.12
24.08
24.03
23.95
23.90
P(O-i-Pr)2
ppm

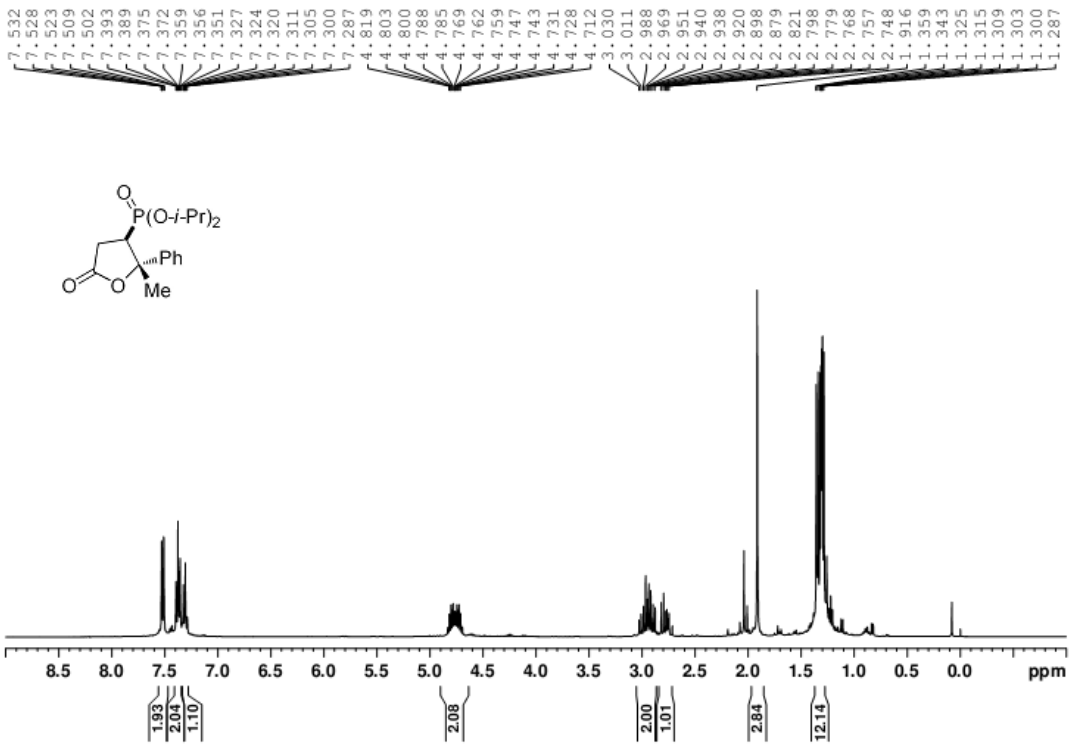
5k



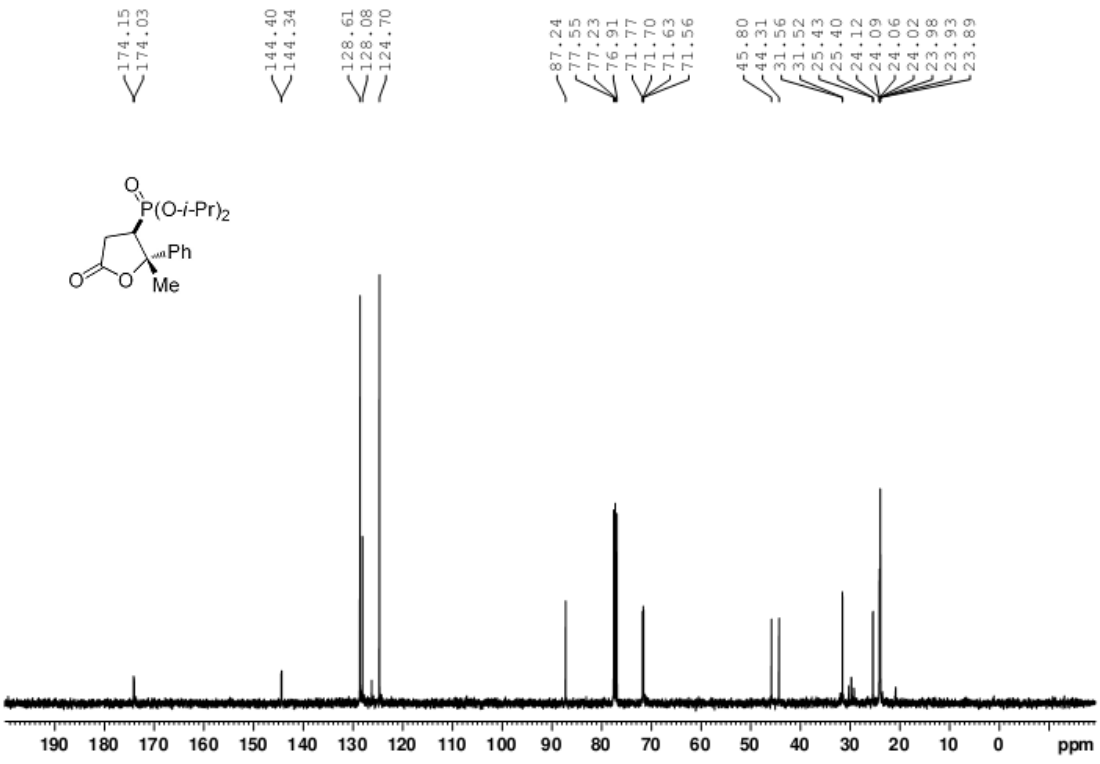
5k



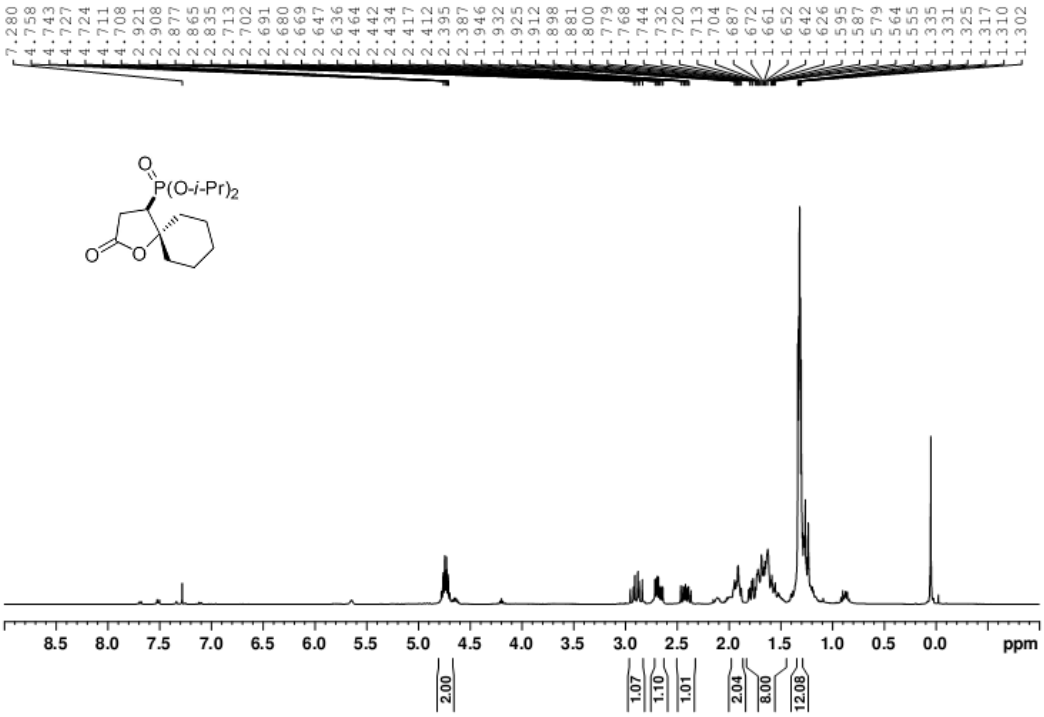
5l


7.532
7.528
7.523
7.509
7.502
7.393
7.389
7.375
7.372
7.359
7.356
7.351
7.327
7.324
7.320
7.311
7.305
7.300
7.287
4.819
4.803
4.800
4.788
4.785
4.769
4.762
4.759
4.747
4.743
4.731
4.728
4.712
3.030
3.011
2.988
2.969
2.951
2.940
2.938
2.920
2.898
2.879
2.821
2.798
2.779
2.768
2.757
2.748
1.916
1.359
1.343
1.325
1.315
1.309
1.303
1.300
1.287
P(O-i-Pr)2
Ph
Me
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 ppm
1.93
2.04
1.10
2.08
2.00
1.01
2.84
12.14

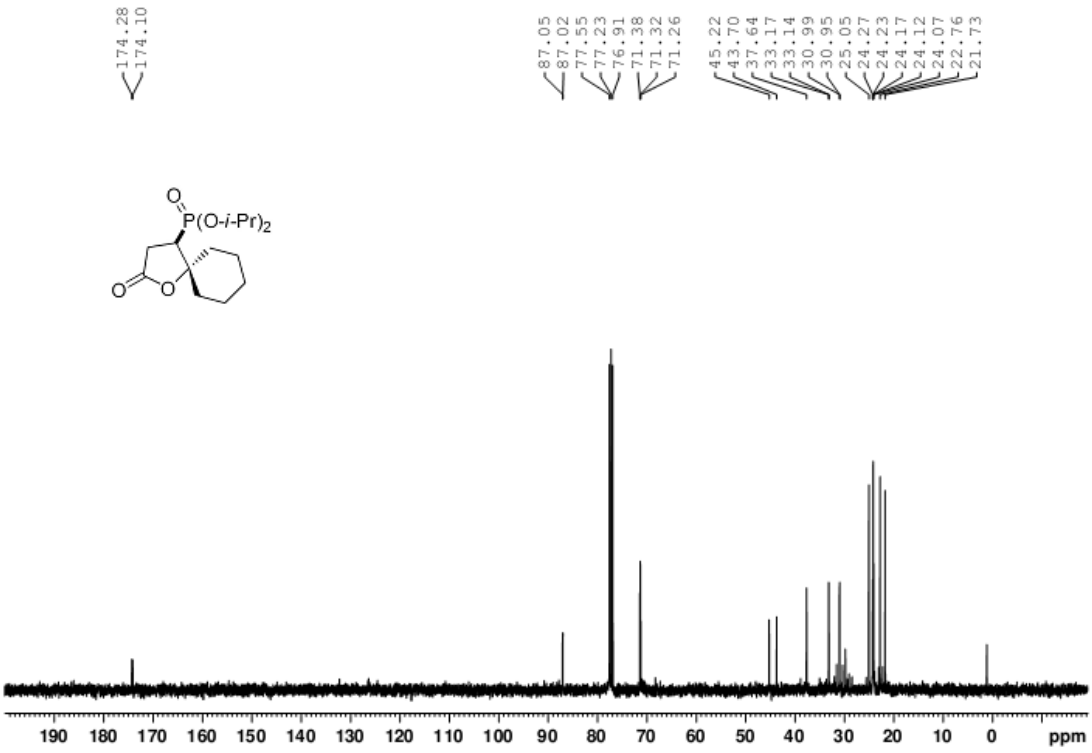
5l


174.15
174.03
144.40
144.34
128.61
128.08
124.70
87.24
77.55
77.23
76.91
71.77
71.70
71.63
71.56
45.80
44.31
31.56
31.52
25.43
25.40
24.12
24.09
24.06
24.02
23.98
23.93
23.89
P(O-i-Pr)2
Ph
Me
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

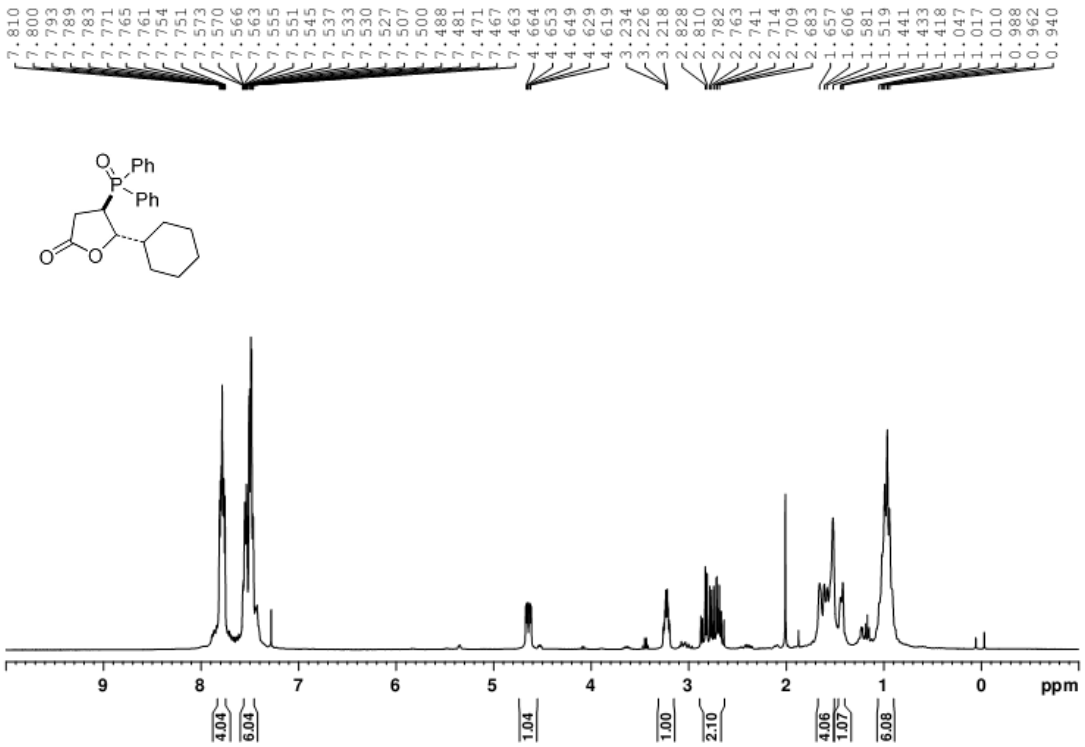
5m


7.280
4.758
4.743
4.727
4.724
4.711
4.708
2.921
2.908
2.877
2.865
2.835
2.713
2.702
2.691
2.680
2.669
2.647
2.636
2.464
2.442
2.434
2.417
2.412
2.395
2.387
1.946
1.932
1.925
1.912
1.898
1.881
1.800
1.779
1.768
1.744
1.732
1.720
1.713
1.704
1.687
1.672
1.661
1.652
1.642
1.626
1.595
1.587
1.579
1.564
1.555
1.335
1.331
1.325
1.317
1.310
1.302
P(O-i-Pr)2
O
O
O
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 ppm
2.00
1.07
1.10
1.01
2.04
8.00
12.08

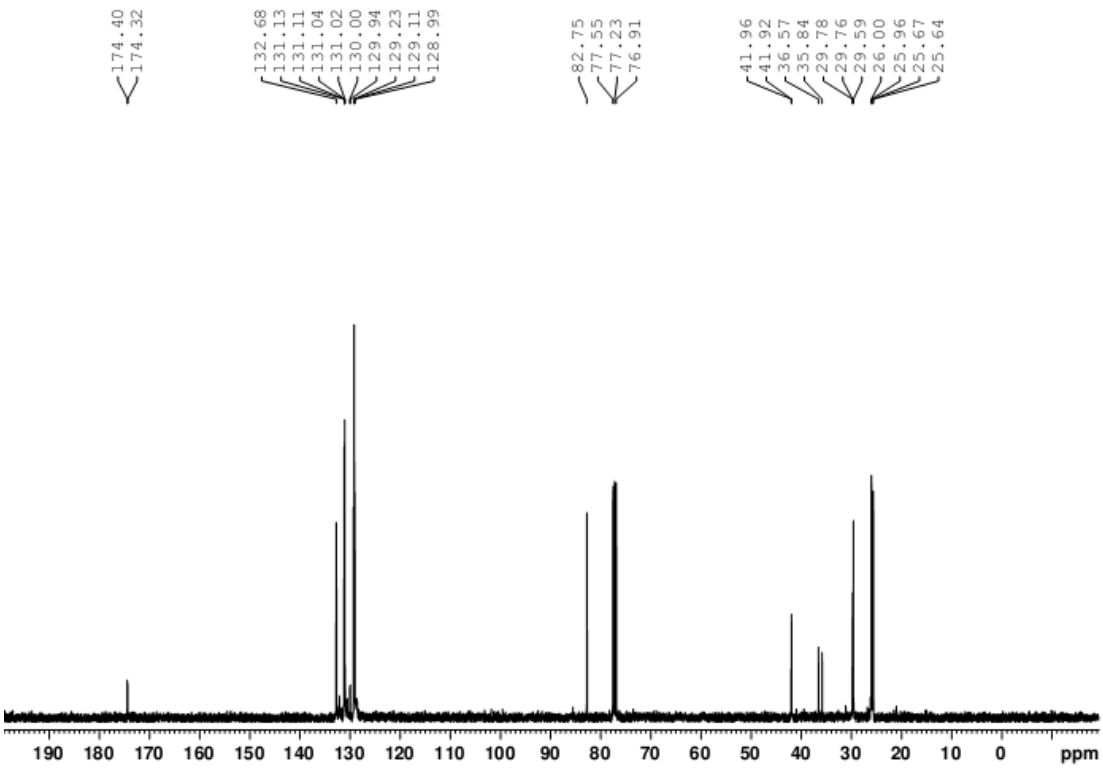
5m


174.28
174.10
87.05
87.02
77.55
77.23
76.91
71.38
71.32
71.26
45.22
43.70
37.64
33.17
33.14
30.99
30.95
25.05
24.27
24.23
24.17
24.12
24.07
22.76
21.73
P(O-i-Pr)2
O
O
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

5n


7.810
7.800
7.793
7.789
7.783
7.771
7.765
7.761
7.754
7.751
7.573
7.570
7.566
7.563
7.555
7.551
7.545
7.537
7.533
7.530
7.527
7.507
7.500
7.488
7.481
7.471
7.467
7.463
4.664
4.653
4.649
4.629
4.619
3.234
3.226
3.218
2.828
2.810
2.782
2.763
2.741
2.714
2.709
2.683
1.657
1.606
1.581
1.519
1.441
1.433
1.418
1.047
1.017
1.010
0.988
0.962
0.940
O
P
Ph
Ph
O
O
9
8
7
6
5
4
3
2
1
0
ppm
4.04
6.04
1.04
1.00
2.10
4.06
1.07
6.08

5n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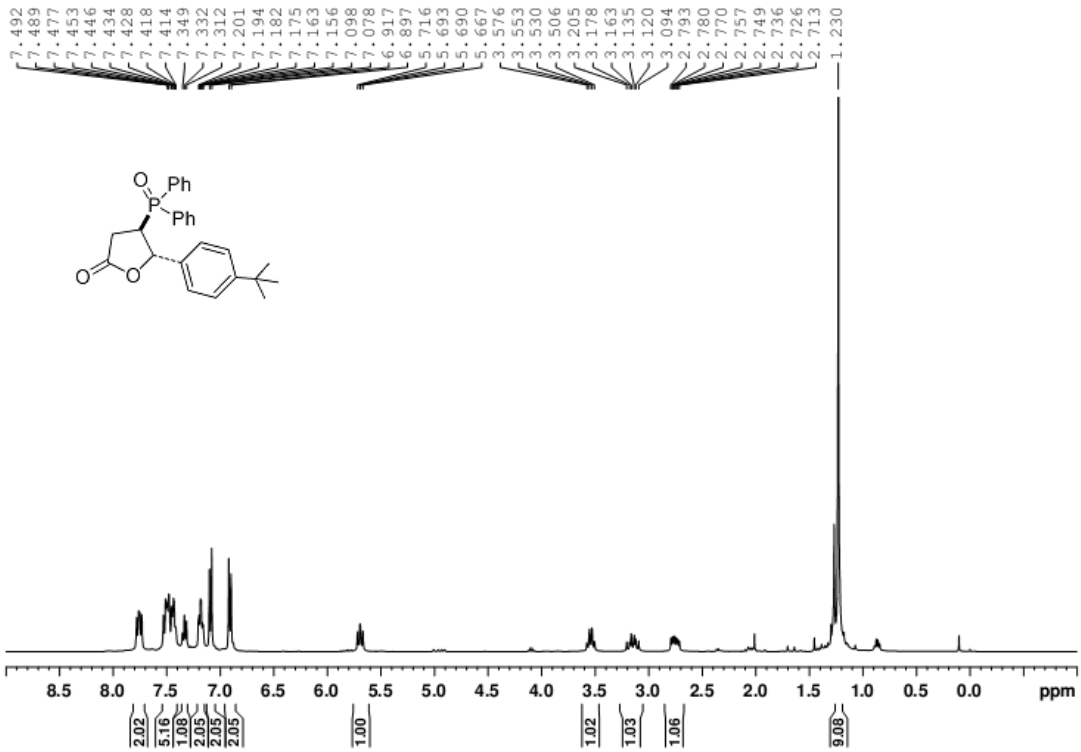
174.32
132.68
131.13
131.11
131.04
131.02
130.00
129.94
129.23
129.11
128.99
82.75
77.55
77.23
76.91
41.96
41.92
36.57
35.84
29.78
29.76
29.59
26.00
25.96
25.67
25.64
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

5o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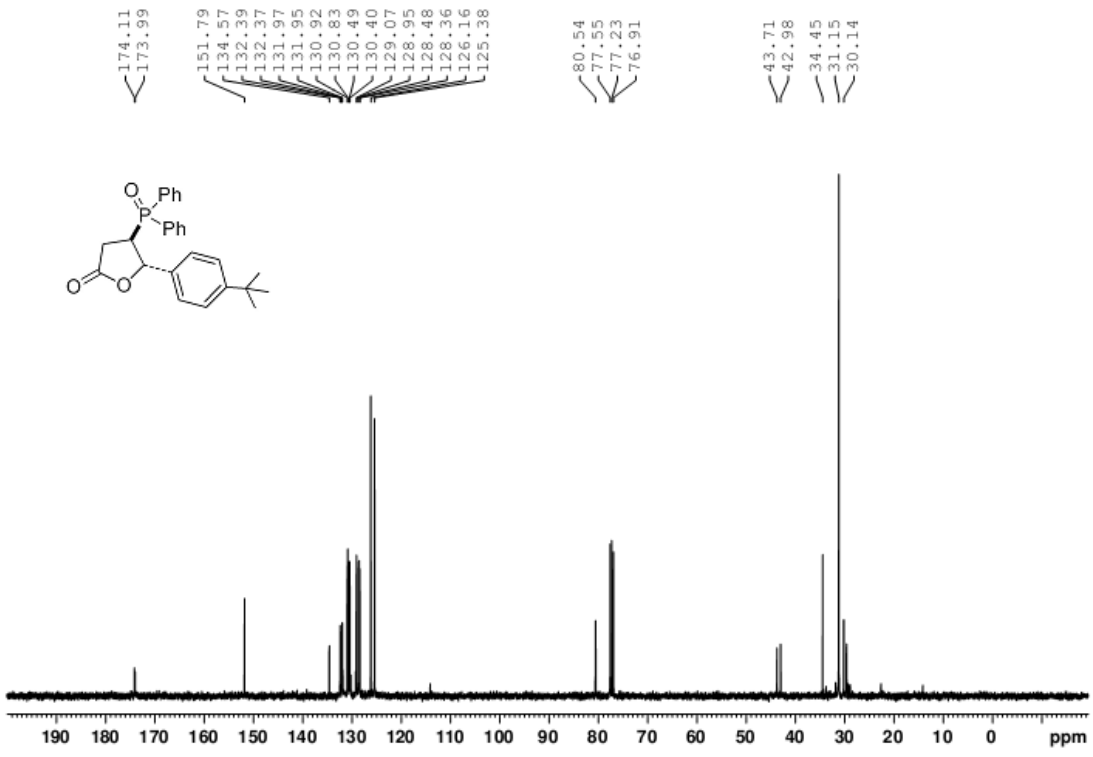
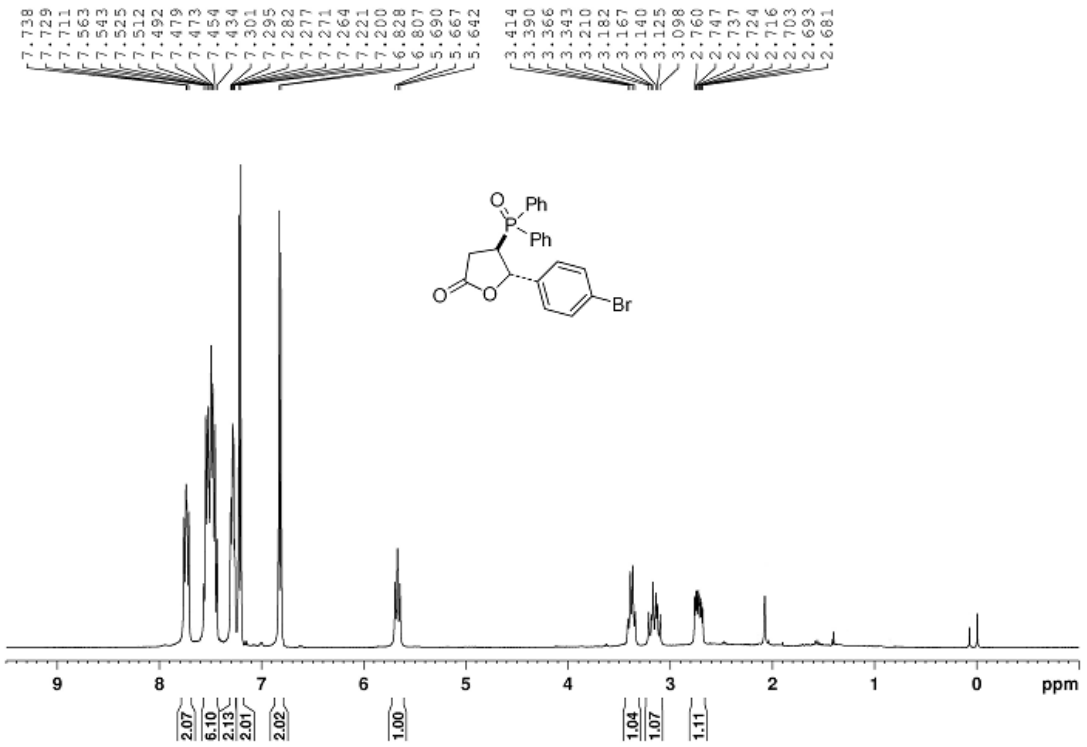
7.477
7.453
7.446
7.434
7.428
7.418
7.414
7.349
7.332
7.312
7.201
7.194
7.182
7.175
7.163
7.156
7.098
7.078
6.917
6.897
5.716
5.693
5.690
5.667
3.576
3.553
3.530
3.506
3.205
3.178
3.163
3.135
3.120
3.094
2.793
2.780
2.770
2.757
2.749
2.736
2.726
2.713
1.230
Ph
Ph
O
O
O
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.02
5.16
1.08
2.05
2.05
2.05
1.00
1.02
1.03
1.06
9.08

5o


174.11
173.99
151.79
134.57
132.39
132.37
131.97
131.95
130.92
130.83
130.49
130.40
129.07
128.95
128.48
128.36
126.16
125.38
80.54
77.55
77.23
76.91
43.71
42.98
34.45
31.15
30.14
Ph
Ph
O
O
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

5p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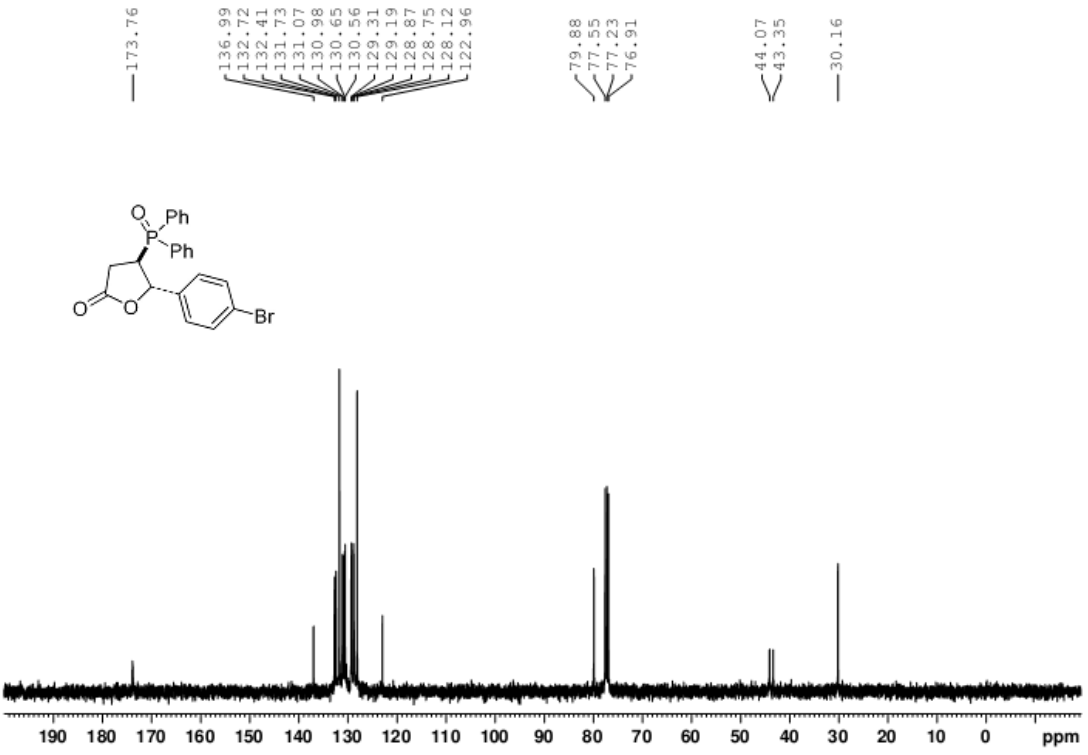
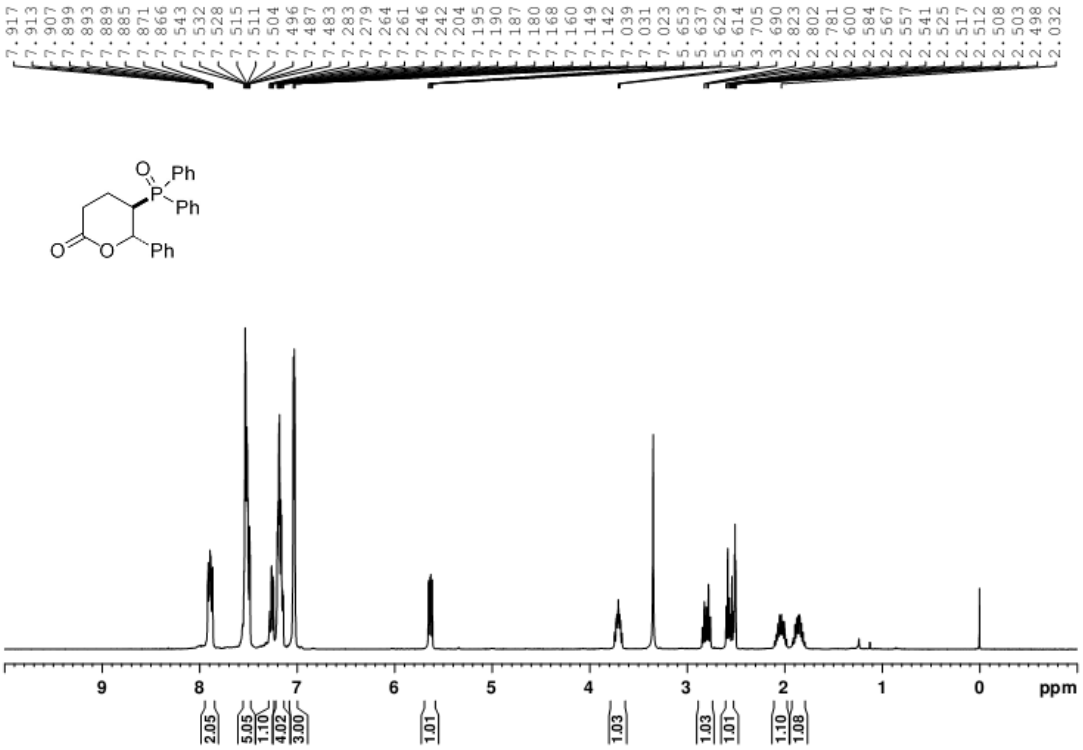
7.729
7.711
7.563
7.543
7.525
7.512
7.492
7.479
7.473
7.454
7.434
7.301
7.295
7.282
7.277
7.271
7.264
7.221
7.200
6.828
6.807
5.690
5.667
5.642
3.414
3.390
3.366
3.343
3.210
3.182
3.167
3.140
3.125
3.098
2.760
2.747
2.737
2.724
2.716
2.703
2.693
2.681
O
P
Ph
Ph
O
O
Br
9
8
7
6
5
4
3
2
1
0
ppm
2.07
6.10
2.13
2.01
2.02
1.00
1.04
1.07
1.11

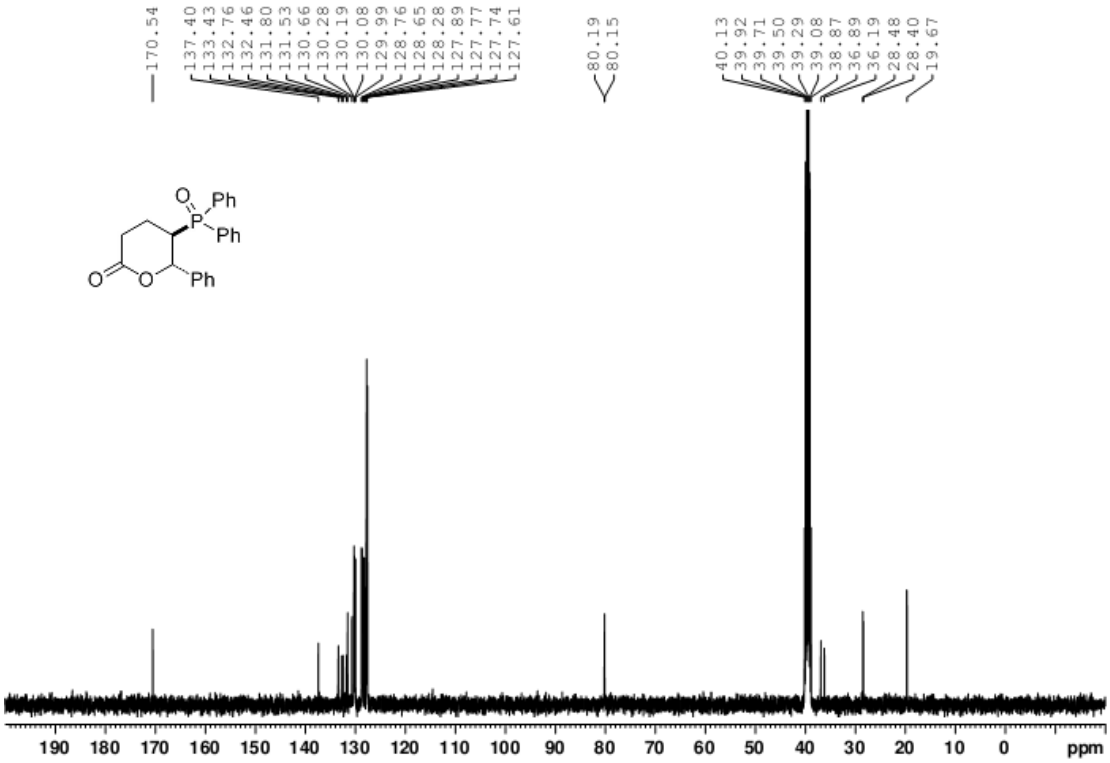
5p


173.76
136.99
132.72
132.41
131.73
131.07
130.98
130.65
130.56
129.31
129.19
128.87
128.75
128.12
122.96
79.88
77.55
77.23
76.91
44.07
43.35
30.16
O
P
Ph
Ph
O
O
Br
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

5q


7.917
7.913
7.907
7.899
7.893
7.889
7.885
7.871
7.866
7.543
7.532
7.528
7.515
7.511
7.504
7.496
7.487
7.483
7.283
7.279
7.264
7.261
7.246
7.242
7.204
7.195
7.190
7.187
7.180
7.168
7.160
7.149
7.142
7.039
7.031
7.023
5.653
5.637
5.629
5.614
3.705
3.690
2.823
2.802
2.781
2.600
2.584
2.567
2.557
2.541
2.525
2.517
2.512
2.508
2.503
2.498
2.032
O
P
Ph
Ph
O
O
Ph
9
8
7
6
5
4
3
2
1
0
ppm
2.05
5.05
1.10
4.02
3.00
1.01
1.03
1.03
1.01
1.10
1.08

5q


170.54
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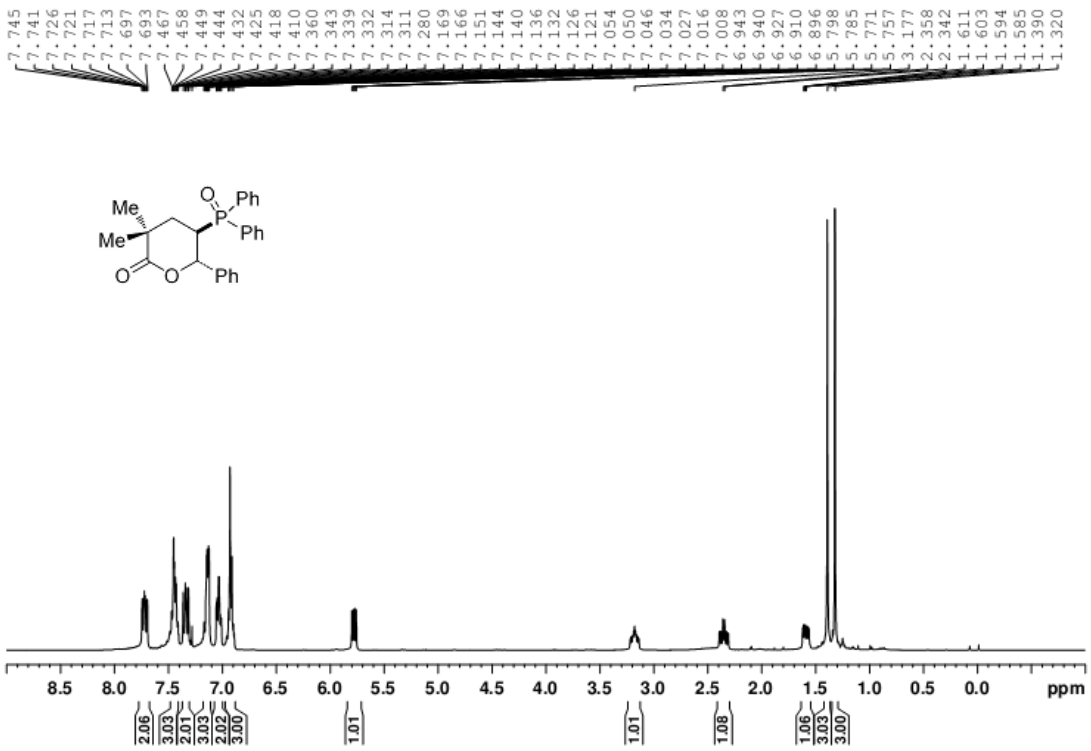
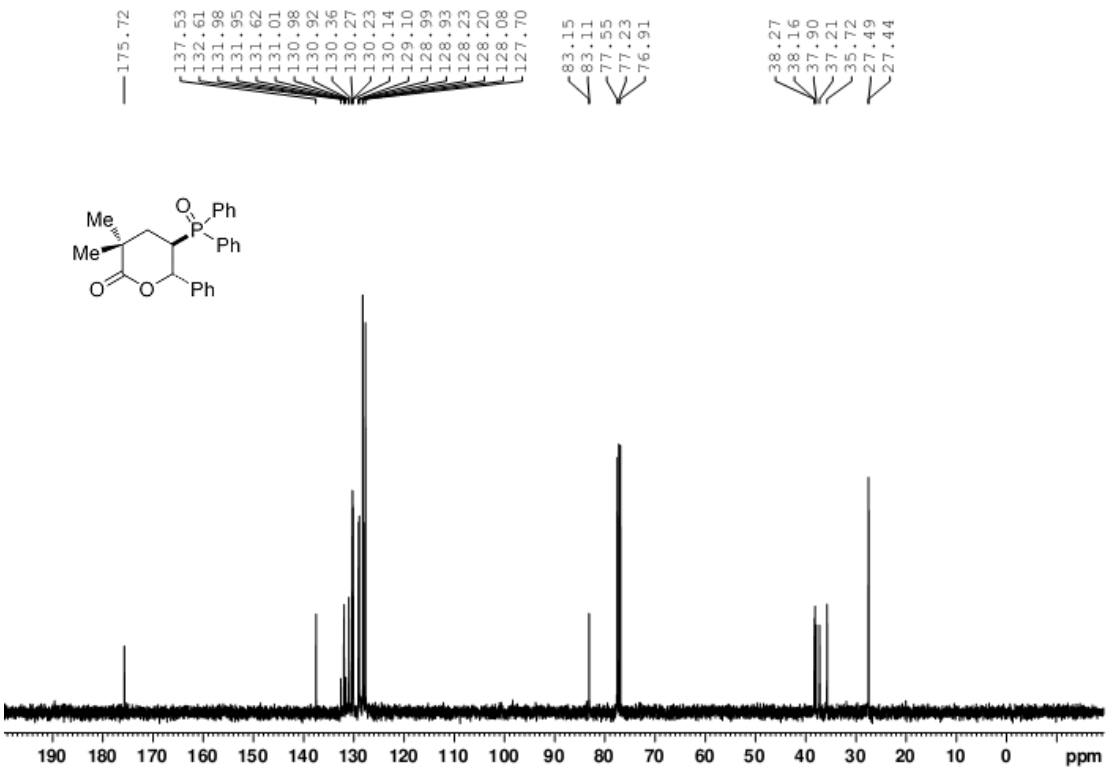
133.43
132.76
132.46
131.80
131.53
130.66
130.28
130.19
130.08
129.99
128.76
128.65
128.28
127.89
127.77
127.74
127.61
80.19
80.15
40.13
39.92
39.71
39.50
39.29
39.08
38.87
36.89
36.19
28.48
28.40
19.67
O
P
Ph
Ph
O
O
Ph
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

5r


7.745
7.741
7.726
7.721
7.717
7.713
7.697
7.693
7.467
7.458
7.449
7.444
7.432
7.425
7.418
7.410
7.360
7.343
7.339
7.332
7.314
7.311
7.280
7.169
7.166
7.151
7.144
7.140
7.136
7.132
7.126
7.121
7.054
7.050
7.046
7.034
7.027
7.016
7.008
6.943
6.940
6.927
6.910
6.896
5.798
5.785
5.771
5.757
3.177
2.358
2.342
1.611
1.603
1.594
1.585
1.390
1.320
Me
Me
O
P
Ph
Ph
O
O
Ph
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
ppm
2.06
3.03
2.01
3.03
2.02
3.00
1.01
1.01
1.08
1.06
3.03
3.00

5r


175.72
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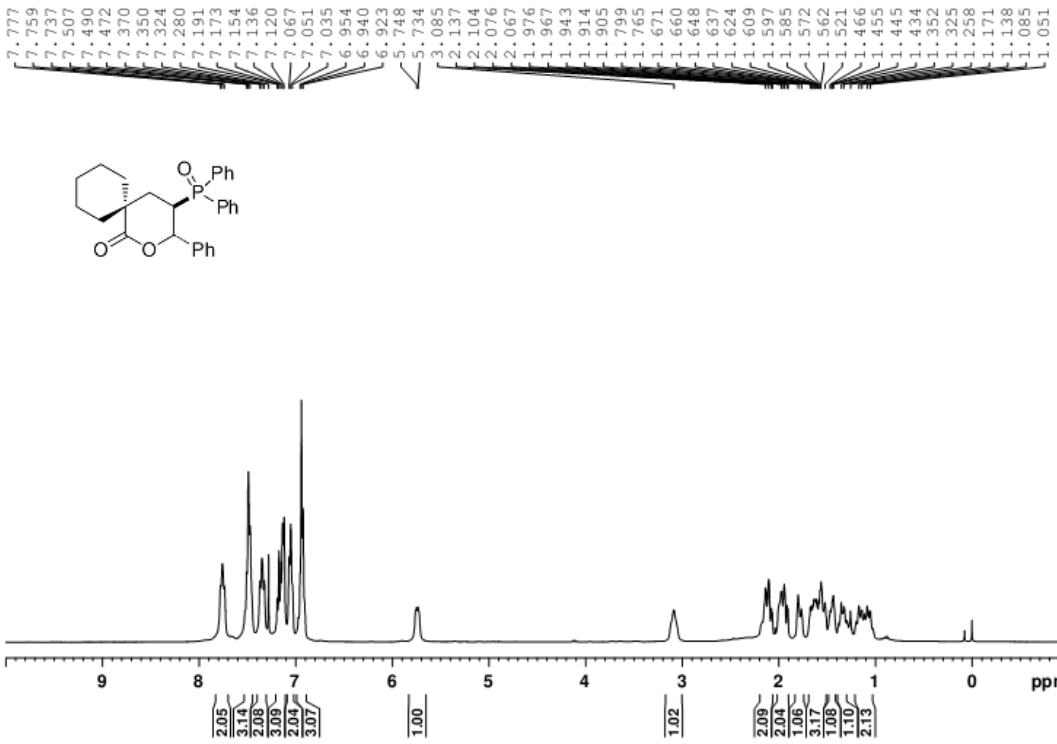
132.61
131.98
131.95
131.62
131.01
130.98
130.92
130.36
130.27
130.23
130.14
129.10
128.99
128.93
128.23
128.20
128.08
127.70
83.15
83.11
77.55
77.23
76.91
38.27
38.16
37.90
37.21
35.72
27.49
27.44
Me
Me
O
P
Ph
Ph
O
O
Ph
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

5s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7.737
7.507
7.490
7.472
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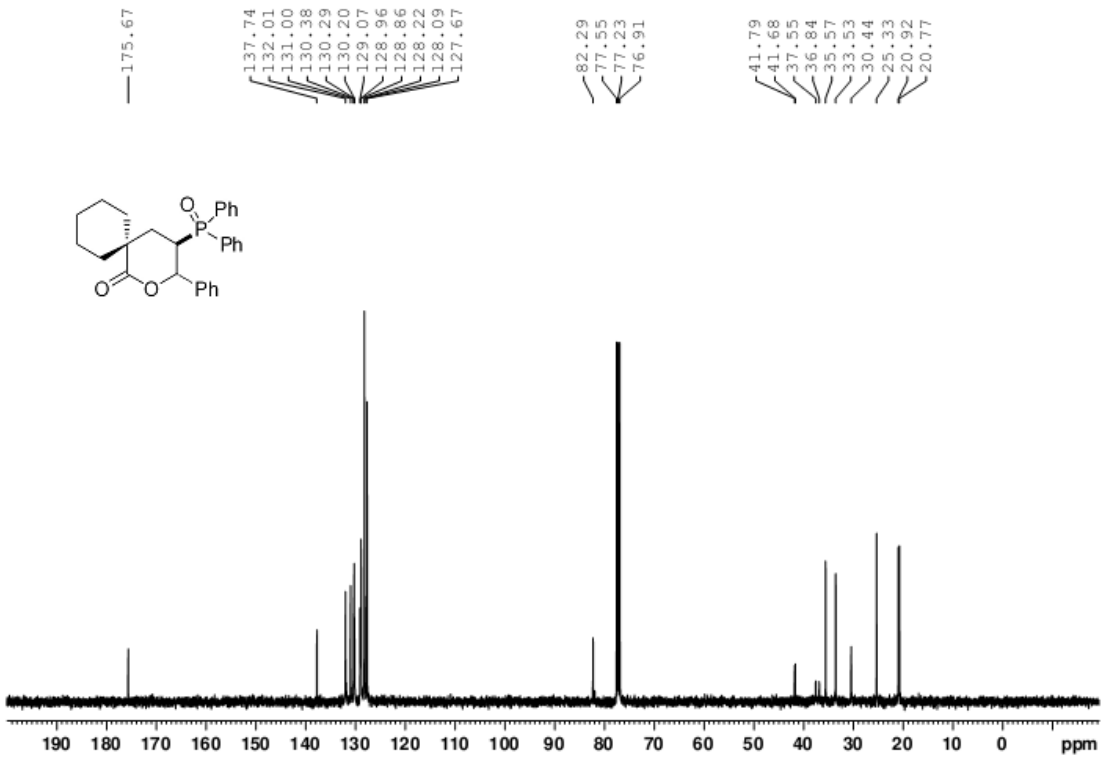
7.350
7.324
7.280
7.191
7.173
7.154
7.136
7.120
7.067
7.051
7.035
6.954
6.940
6.923
5.748
5.734
3.085
2.137
2.104
2.076
2.067
1.976
1.967
1.943
1.914
1.905
1.799
1.765
1.671
1.660
1.648
1.637
1.624
1.609
1.597
1.585
1.572
1.562
1.521
1.466
1.455
1.445
1.434
1.352
1.325
1.258
1.171
1.138
1.085
1.051
O
P
Ph
Ph
O
O
Ph
9
8
7
6
5
4
3
2
1
0
ppm
2.05
3.14
2.08
3.09
2.04
3.07
1.00
1.02
2.09
2.04
1.06
3.17
1.08
1.10
2.13

5s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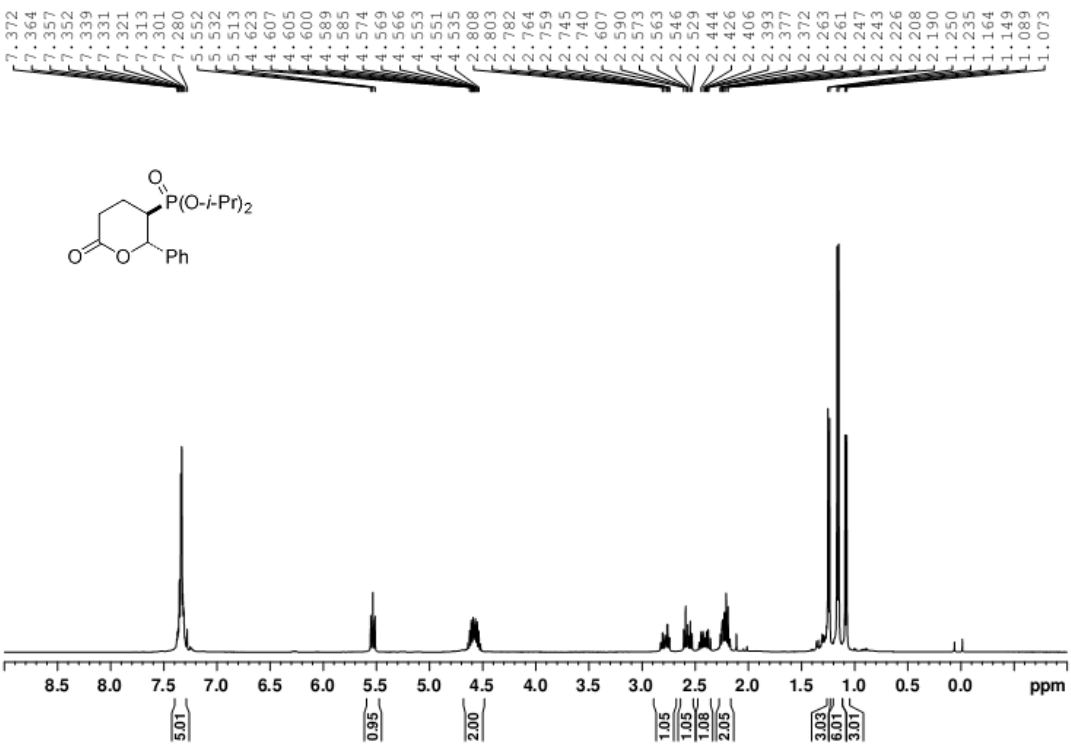
137.74
132.01
131.00
130.38
130.29
130.20
129.07
128.96
128.86
128.22
128.09
127.67
82.29
77.55
77.23
76.91
41.79
41.68
37.55
36.84
35.57
33.53
30.44
25.33
20.92
20.77
O
P
Ph
Ph
O
O
Ph
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

5t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7.301
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5.513
4.623
4.607
4.605
4.600
4.589
4.585
4.574
4.569
4.566
4.553
4.551
4.535
2.808
2.803
2.782
2.764
2.759
2.745
2.740
2.607
2.590
2.573
2.563
2.546
2.529
2.444
2.426
2.406
2.393
2.377
2.372
2.263
2.261
2.247
2.243
2.226
2.208
2.190
1.250
1.235
1.164
1.149
1.089
1.073
O
P(O-i-Pr)2
O
O
Ph
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 ppm
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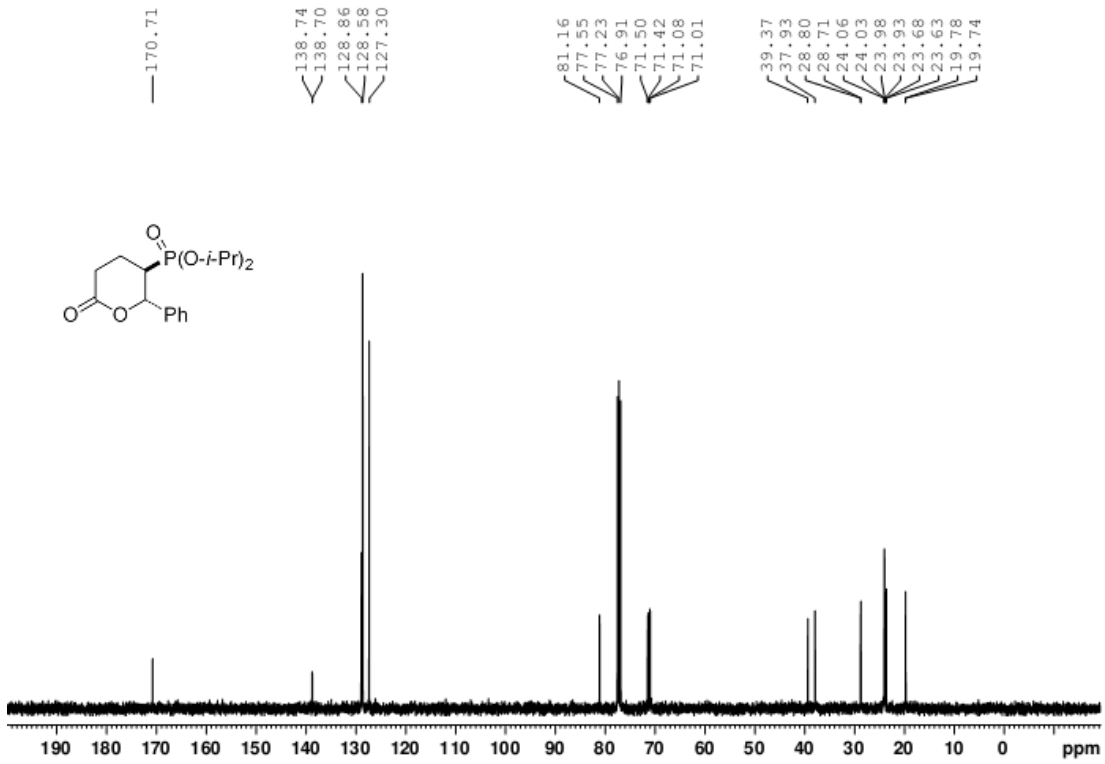
0.95
2.00
1.05
1.05
1.08
2.05
3.03
6.01
3.01

5t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138.74
138.70
128.86
128.58
127.30
81.16
77.55
77.23
76.91
71.50
71.42
71.08
71.01
39.37
37.93
28.80
28.71
24.06
24.03
23.98
23.93
23.68
23.63
19.78
19.74
O
P(O-i-Pr)2
O
O
Ph
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

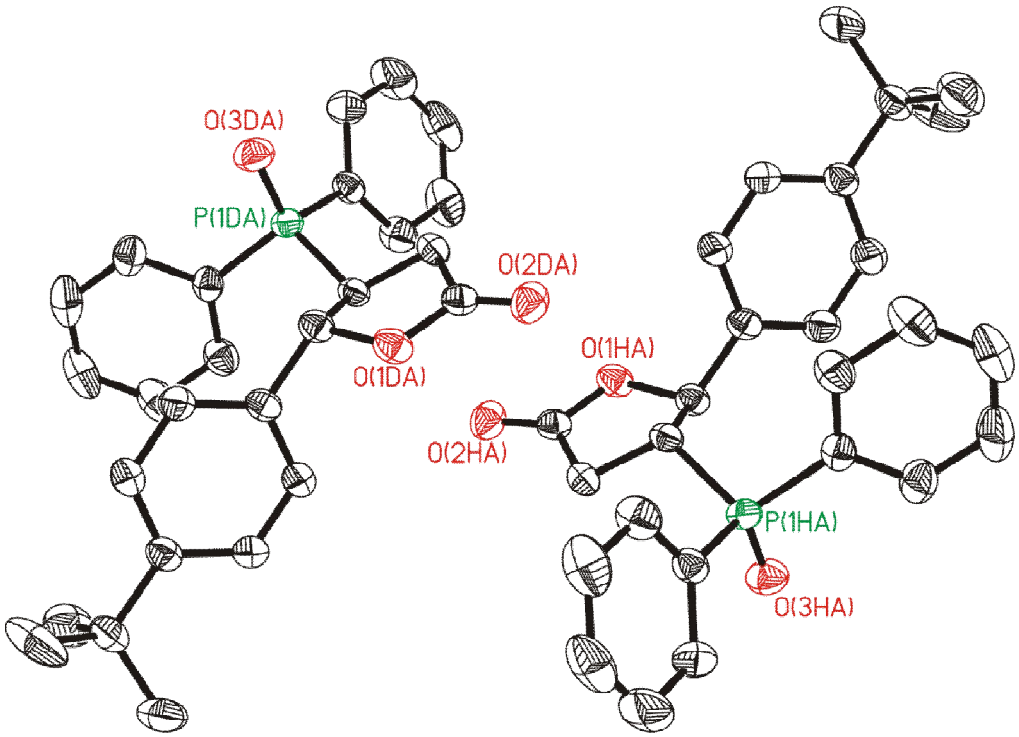

Figure 1. Single-crystal X-ray analysis of **5o**.