Straightforward chemical desymmetrisation of cis-(±)-4-*O*-protected-cyclopent-2-enol using resolving agents on column chromatography

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General conditions

^{1}H and ^{13}C NMR spectra were recorded at ambient temperature on a Bruker Avance III HD 400 and 500 MHz. Chemical shifts (δ) are quoted in parts per million (ppm) referenced to the residual solvent peak, ($CDCl_3$ fixed at 7.26 ppm and 77.16 ppm) relative to tetramethylsilane (TMS). Coupling constants, J, are reported in Hertz. ESI Mass and High Resolution Mass spectra were recorded in the positive ion mode on a Micromass Q-TOF. Thin-layer chromatography was performed on pre-coated aluminum sheets of Silica 60 F254 (Merck, Art. 5554), visualization of products being accomplished by UV absorbance and by charring with anisaldehyde solution and heating. Chromatography was performed on Merck Silica gel 60 (40-63 µm).

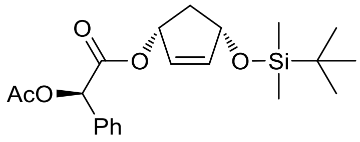
Flash purification general protocol

Biotage isolera flash purification system (Isolera Four) was used for the separation of the diastereoisomers. Buchi columns (FlashPure Silica) (40g, 80g and 120 g) were employed. The Column Volume (CV) was 50 mL, 110 mL and 155 mL, respectively. The flow rate was 30mL/min for column Buchi (40g) or 60 ml/min for column Buchi (80g and 120g).

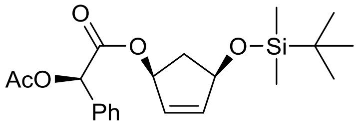
(±)-4-((*tert*-butyldimethylsilyl)oxycyclopent-2-enol. (±)-1

Compound **(±)-1** was obtained in three steps from commercially available furfuryl alcohol following a reported procedure.[1] The NMR spectra were identical to those previously reported.

Procedure for synthesis



F$_1$



F$_3$

To a mixture of racemic 4-((*tert*-butyldimethylsilyl)oxy)cyclopent-2-enol (1 g, 4.66 mmol), DCC (1.45g, 7 mmol) and DMAP (57 mg, 0.46 mmol) in dry DCM (15 mL) under argon was added (*R*)-*O*-acetyl mandelic acid (1.36 g, 7 mmol). After 2 hours, a total conversion was observed by TLC. After filtration and evaporation of solvent, a preliminary purification on silica gel column chromatograpy was made with PE/DCM (1/1 : v/v) to give the mixture of diastereoisomers (1.7 g, 93 %).

Purification of 500 mg of the mixture of diastereoisomers with Biotage flash purification with Buchi column (80 g, 40 µm) using as eluent petroleum ether and dichloromethane (2 CV at 0 %, 7 CV at 20 %, 10 CV at 30 % and 15 CV at 50 % of dichloromethane) gave three fractions (F$_1$: 245 mg; F$_2$ (mixture): 15 mg; F$_3$: 240 mg).

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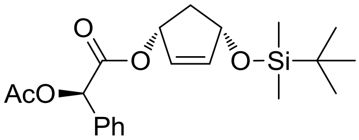
[1] T. T. Curran, D. A. Hay, C. P. Koegel, *Tetrahedron* **1997**, *53*, 1983-2004.



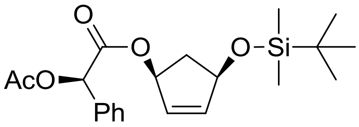
(–)-(*R*)-(1*R*,4*S*)-4-((*tert*-butyldimethylsilyl)oxy)cyclopent-2-en-1-yl 2-acetoxy-2-phenylacetate

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.46 (dd, *J* = 6.7, 2.9 Hz, 2H), 7.40-7.37 (m, 3H), 5.94 (dt, *J* = 5.7, 1.6 Hz, 1H), 5.89 (s, 1H), 5.72 (dt, *J* = 5.6, 1.6 Hz, 1H), 5.50 (ddt, *J* = 7.4, 5.0, 1.4 Hz, 1H), 4.72-4.68 (m, 1H), 2.80 (dt, *J* = 13.9, 6.9 Hz, 1H), 2.19 (s, 3H), 1.66 (dt, *J* = 13.9, 5.2 Hz, 1H), 0.89 (s, 9H), 0.08 (s, 6H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 170.5, 168.8, 139.5, 133.9, 130.6, 129.3, 128.9, 127.7, 78.3, 74.9, 74.8, 41.1, 26.0, 20.9, 18.3, -4.5, -4.6.

$[\alpha]_D^{20}$ = -21.1° (c = 1.00, $CHCl_3$).

MS (ESI^+) : m/z = 413.2 $(M+Na)^+$.
HRMS (ESI^+) : calculated for $C_{21}H_{31}O_5Si$ $[M+H]^+$: 391.1935 ; Found : 391.1939.



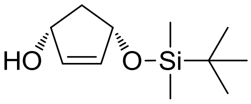
(–)-(*R*)-(1*S*,4*R*)-4-((*tert*-butyldimethylsilyl)oxy)cyclopent-2-en-1-yl 2-acetoxy-2-phenylacetate

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.48-7.45 (m, 2H), 7.41-7.36 (m, 3H), 5.97 (dt, *J* = 5.7, 1.6 Hz, 1H), 5.90-5.88 (m, 2H), 5.54-5.50 (m, 1H), 4.67 (dddd, *J* = 8.0, 4.5, 2.2, 1.1 Hz, 1H), 2.70 (dt, *J* = 13.8, 7.3 Hz, 1H), 2.19 (s, 3H), 1.41 (dt, *J* = 13.9, 5.0 Hz, 1H), 0.86 (s, 9H), 0.05 (s, 3H), 0.03 (s, 3H).

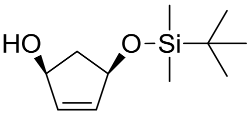
^{13}C NMR (101 MHz, Chloroform-*d*) δ 170.5, 168.7, 139.5, 133.8, 130.8, 129.3, 128.9, 127.7, 78.3, 74.9, 74.7, 40.8, 26.0, 20.9, 18.3, -4.5, -4.6.

$[\alpha]_D^{20}$ = -60.8° (c = 1.00, $CHCl_3$).

MS (ESI^+) : m/z = 413.2 $(M+Na)^+$.
HRMS (ESI^+) : calculated for $C_{21}H_{31}O_5Si$ $[M+H]^+$: 391.1935 ; Found : 391.1937.

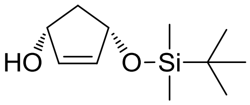


From **F_1**



From **F_3**

To a solution of diastereoisomer F_1 or F_3 (400 mg, 1.02 mmol) in THF/H_2O : 1/1 v/v was added, at room temperature, lithium hydroxide (123 mg, 5.12 mmol). After 1 hour under stirring, the completion of reaction was observed by TLC. A saturated solution of NH_4Cl (20 mL) and DCM (20 mL) were added, the layers were separated and the aqueous layer was washed with DCM (4x20 mL). The organic layers were combined, dried with Na_2SO_4 and the solvent was evaporated. After a short purification on silica gel (PE/EtOAc: 1/0 to 4/1), the products were obtained with 95 % (from F_1, 208 mg) and 95 % (from F_3, 208 mg).



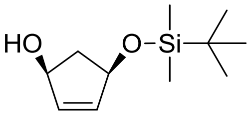
(–)-(1*R*,4*S*)-4-((*tert*-butyldimethylsilyl)oxy)cyclopent-2-enol

^1H NMR (400 MHz, Chloroform-*d*) δ 5.94 (dt, *J* = 5.6, 1.5 Hz, 1H), 5.88 (dt, *J* = 5.6, 1.6 Hz, 1H), 4.69 – 4.62 (m, 1H), 4.60-4.57 (m, 1H), 2.68 (dt, *J* = 13.9, 7.1 Hz, 1H), 1.51 (dt, *J* = 13.7, 4.5 Hz, 1H), 0.89 (s, 9H), 0.09 (s, 6H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 137.1, 135.7, 75.3, 75.3, 44.8, 26.0, 18.3, -4.5.

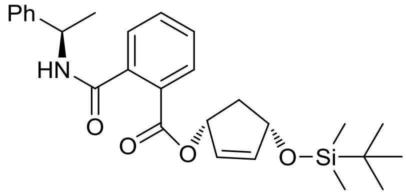
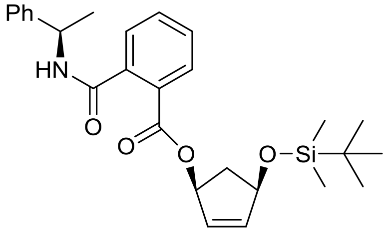
$[\alpha]_D^{20}$ = -21.1° (c = 1.00, $CHCl_3$).

MS (ESI^+) : m/z = 215.1 $(M+H)^+$.
HRMS (ESI^+) : calculated for $C_{11}H_{23}O_2Si$ $[M+H]^+$: 215.1469 ; Found : 215.1462.



(+)-(**1*S*,4*R*)-4-((*tert*-butyldimethylsilyl)oxy)cyclopent-2-enol**

$[\alpha]_D^{20}$ = + 21.4° (c = 1.00, $CHCl_3$).

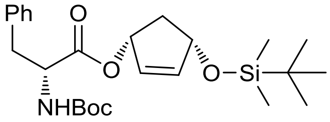
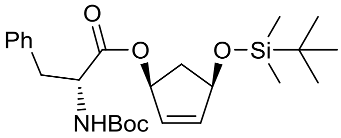
 **(1*R*,4*S*)-4-((*tert*-butyldimethylsilyl)oxy)cyclopent-2-en-1-yl 2-(((*R*)-1-phenylethyl)carbamoyl)benzoate**	 **(1*S*,4*R*)-4-((*tert*-butyldimethylsilyl)oxy)cyclopent-2-en-1-yl 2-(((*R*)-1-phenylethyl)carbamoyl)benzoate**

To a mixture of racemic 4-((*tert*-butyldimethylsilyl)oxy)cyclopent-2-enol (300 mg, 1.4 mmol), DCC (434 mg, 2.1 mmol) and DMAP (17 mg, 0.14 mmol) in dry DCM (5 mL) under atmosphere of argon was added Phthalamic acid derivative (565 mg, 2.1 mmol). After 6 days of reaction, the total conversion was not observed. After filtration and evaporation of solvent, purification on silica gel column chromatography with DCM/MeOH (1/0 to 98/2) gave the mixture of diastereoisomers (270 mg, 41 %).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.90-7.85 (m, 2H, Dia1 + Dia2), 7.53-7.47 (m, 2H, Dia1 + Dia2), 7.47-7.39 (m, 8H, Dia1 + Dia2), 7.36 (ddd, *J* = 8.1, 6.8, 2.0 Hz, 4H, Dia1 + Dia2), 7.28 (dt, *J* = 7.7, 1.7 Hz, 2H, Dia1 + Dia2), 6.11 (t, *J* = 8.6 Hz, 2H, Dia1 + Dia2), 6.01 (dt, *J* = 5.6, 1.7 Hz, 1H, Dia1 or Dia2), 5.99 (dt, *J* = 5.6, 1.7 Hz, 1H, Dia1 or Dia2), 5.97 (dt, *J* = 5.6, 1.6 Hz, 1H, Dia1 or Dia2), 5.91 (dt, *J* = 5.6, 1.6 Hz, 1H, Dia1 or Dia2), 5.69 (dddd, *J* = 8.7, 5.9, 2.9, 1.3 Hz, 2H, Dia1 + Dia2), 5.33 (dtd, *J* = 14.4, 7.1, 4.1 Hz, 2H, Dia1 + Dia2), 4.76 (tdt, *J* = 5.7, 2.6, 1.4 Hz, 2H, Dia1 + Dia2), 2.90 (dtd, *J* = 13.7, 7.3, 2.8 Hz, 2H, Dia1 + Dia2), 1.75 (ddt, *J* = 13.8, 6.8, 5.3 Hz, 2H, Dia1 + Dia2), 1.61 (d, *J* = 6.9 Hz, 6H, Dia1 + Dia2), 0.91 (s, 9H, Dia1 or Dia2), 0.90 (s, 9H, Dia1 or Dia2), 0.10 (t, *J* = 2.4 Hz, 12H, Dia1 + Dia2).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 168.4, 168.3, 166.5, 166.5, 143.1, 143.1, 139.3, 138.4, 138.3, 132.0, 131.9, 131.1, 131.1, 130.3, 130.3, 129.7, 129.7, 129.6, 129.4, 128.8, 127.7, 127.7, 127.5, 126.5, 126.5, 78.2, 75.0, 49.4, 49.4, 41.2, 41.1, 26.0, 26.0, 21.7, 21.7, 18.3, 18.3, -4.5, -4.6.

MS (ESI$^+$) : m/z = 466.2 (M+H)$^+$.
HRMS (ESI$^+$) : calculated for $C_{27}H_{36}NO_4Si$ [M+H]$^+$: 466.2408 ; Found : 466.2412.

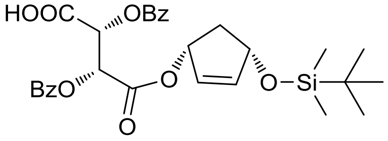
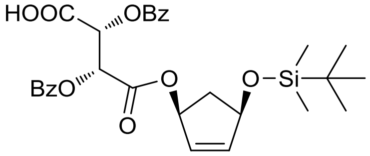
 **(*R*)-(1*R*,4*S*)-4-((*tert*-butyldimethylsilyl)oxy)cyclopent-2-en-1-yl 2-((*tert*-butoxycarbonyl)amino)-3-phenylpropanoate**	 **(*R*)-(1*S*,4*R*)-4-((*tert*-butyldimethylsilyl)oxy)cyclopent-2-en-1-yl 2-((*tert*-butoxycarbonyl)amino)-3-phenylpropanoate**

To a mixture of racemic 4-((*tert*-butyldimethylsilyl)oxy)cyclopent-2-enol (300 mg, 1.4 mmol), DCC (434 mg, 2.1 mmol) and DMAP (17 mg, 0.14 mmol) in dry DCM (5 mL) under atmosphere of argon was added phenylalanine derivative (557 mg, 2.1 mmol). After 3 h of reaction, the total conversion was observed with TLC. After filtration and evaporation of solvent, purification on silica gel column chromatography with DCM/MeOH (1/0 to 98/2) gave the mixture of diastereoisomers (637 mg, 96 %).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.35-7.19 (m, 6H, Dia1 + Dia2), 7.15 (dt, *J* = 6.9, 2.7 Hz, 4H, Dia1 + Dia2), 5.99 (ddd, *J* = 6.3, 4.0, 2.3 Hz, 2H, Dia1 + Dia2), 5.83 (dt, *J* = 5.6, 1.6 Hz, 1H, Dia1 or Dia2), 5.79 (dt, *J* = 5.8, 1.7 Hz, 1H, Dia1 or Dia2), 5.48 (tt, *J* = 7.5, 3.4 Hz, 2H, Dia1 + Dia2), 4.97 (dd, *J* = 8.3, 5.4 Hz, 2H, Dia1 + Dia2), 4.80-4.64 (m, 2H, Dia1 + Dia2), 4.56 (h, *J* = 5.8 Hz, 2H, Dia1 + Dia2), 3.14-2.02 (m, 4H, Dia1 + Dia2), 2.76 (dq, *J* = 13.9, 7.7 Hz, 2H, Dia1 + Dia2), 1.60 – 1.50 (m, 2H, Dia1 + Dia2), 1.42 (d, *J* = 2.6 Hz, 18H, Dia1 + Dia2), 0.90 (s, 18H, Dia1 + Dia2), 0.09 (s, 6H, Dia1), 0.09 (s, 6H, Dia2).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 171.7, 171.6, 155.2, 139.5, 139.4, 136.1, 130.9, 130.8, 129.6, 129.6, 128.6, 127.1, 80.0, 78.1, 78.0, 74.9, 54.5, 41.1, 38.3, 28.4, 26.0, 18.3, -4.5, -4.5.

MS (ESI$^+$) : m/z = 462.3 (M+H)$^+$.
HRMS (ESI$^+$) : calculated for $C_{25}H_{40}NO_5Si$ [M+H]$^+$: 462.2670 ; Found : 466.2677.

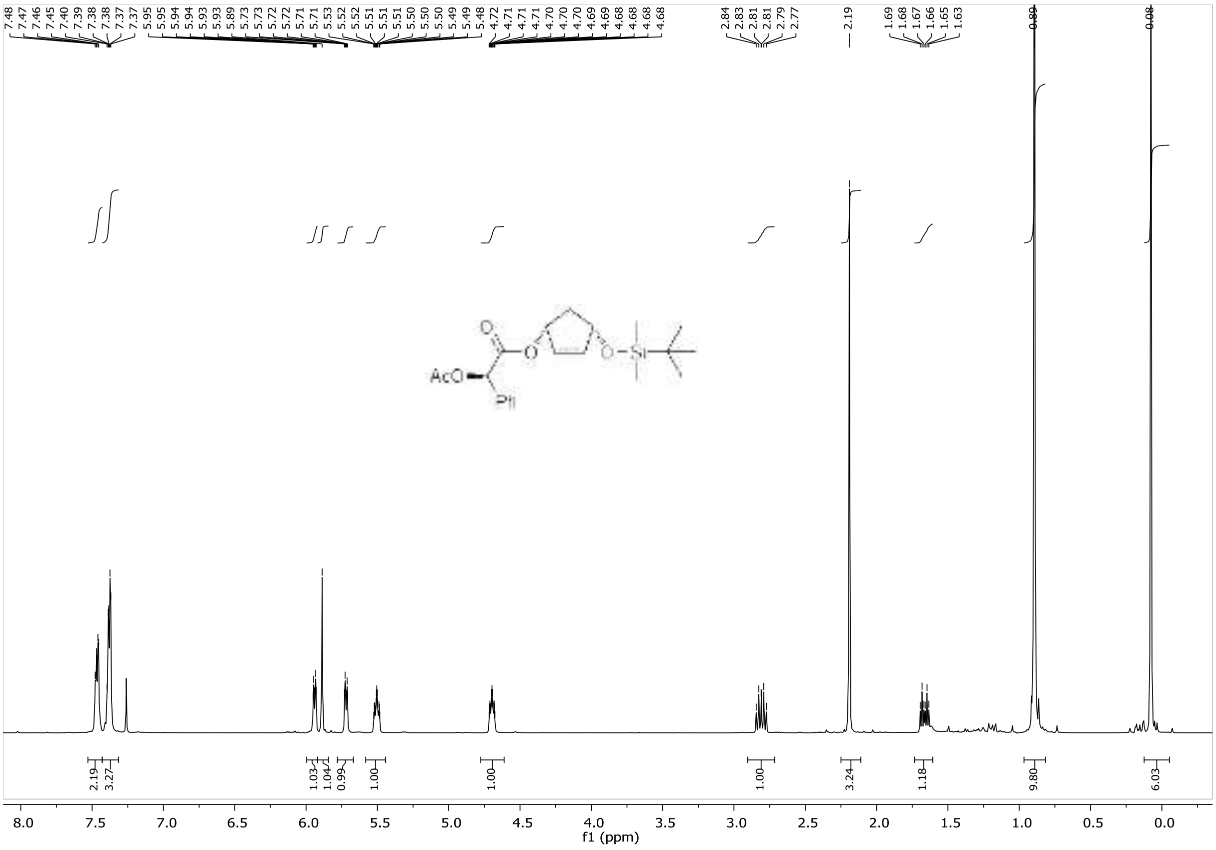
 **(2*R*,3*R*)-2,3-bis(benzoyloxy)-4-(((1*R*,4*S*)-4-((*tert*-butyldimethylsilyl)oxy)cyclopent-2-en-1-yl)oxy)-4-oxobutanoic acid**	 **(2*R*,3*R*)-2,3-bis(benzoyloxy)-4-(((1*S*,4*R*)-4-((*tert*-butyldimethylsilyl)oxy)cyclopent-2-en-1-yl)oxy)-4-oxobutanoic acid**

To a mixture of racemic 4-((*tert*-butyldimethylsilyl)oxy)cyclopent-2-enol (300 mg, 1.4 mmol), DCC (434 mg, 2.1 mmol) and DMAP (17 mg, 0.14 mmol) in dry DCM (5 mL) under atmosphere of argon was added tartric acid derivative (527 mg, 2.1 mmol). After 5 days of reaction, the total conversion was not observed. After filtration and evaporation of solvent, purification on silica gel column chromatography with DCM/MeOH (1/0 to 98/2) gave the mixture of diastereoisomers (488 mg, 63 %).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.15-8.05 (m, 8H, Dia1 + Dia2), 7.58 (tdd, *J* = 8.3, 6.2, 1.5 Hz, 4H, Dia1 + Dia2), 7.44 (qd, *J* = 8.0, 3.0 Hz, 8H, Dia1 + Dia2), 6.01-5.95 (m, 4H, Dia1 + Dia2), 5.93 (dt, *J* = 5.6, 1.7 Hz, 1H, Dia1 or Dia2), 5.87 (dt, *J* = 5.6, 1.7 Hz, 1H, Dia1 or Dia2), 5.82 (dt, *J* = 5.6, 1.6 Hz, 1H, Dia1 or Dia2), 5.67 (dt, *J* = 5.7, 1.6 Hz, 1H, Dia1 or Dia2), 5.60-5.46 (m, 2H, Dia1 + Dia2), 4.65 (ddt, *J* = 6.9, 4.6, 1.4 Hz, 1H, Dia1 or Dia2), 4.63-4.56 (m, 1H, Dia1 + Dia2), 2.77 (dt, *J* = 13.7, 7.3 Hz, 1H, Dia1 or Dia2), 2.65 (dt, *J* = 14.3, 7.3 Hz, 1H, Dia1 or Dia2), 1.66 (dt, *J* = 13.8, 5.2 Hz, 1H, Dia1 or Dia2), 1.44 (dt, *J* = 13.9, 5.1 Hz, 1H, Dia1 or Dia2), 0.86 (s, 9H, Dia1 or Dia2), 0.81 (s, 9H, Dia1 or Dia2), 0.04 (d, *J* = 3.3 Hz, 6H, Dia1 or Dia2), -0.03 (d, *J* = 21.1 Hz, 6H, Dia1 or Dia2).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 176.6, 168.9, 168.8, 165.8, 165.8, 165.4, 165.4, 165.2, 165.1, 139.6, 133.7, 133.6, 130.3, 130.2, 130.2, 130.1, 128.8, 128.7, 128.7, 128.6, 128.5, 128.5, 78.8, 74.8, 74.7, 71.8, 71.4, 71.4, 40.8, 40.7, 25.9, 25.8, 18.2, 18.1, -4.7, -4.7, -4.8, -4.8.

MS (ESI$^-$) : m/z = 553.2 (M-H)$^-$.
HRMS (ESI$^-$) : calculated for $C_{29}H_{33}O_9Si$ [M-H]$^-$: 553.1899 ; Found : 553.1905.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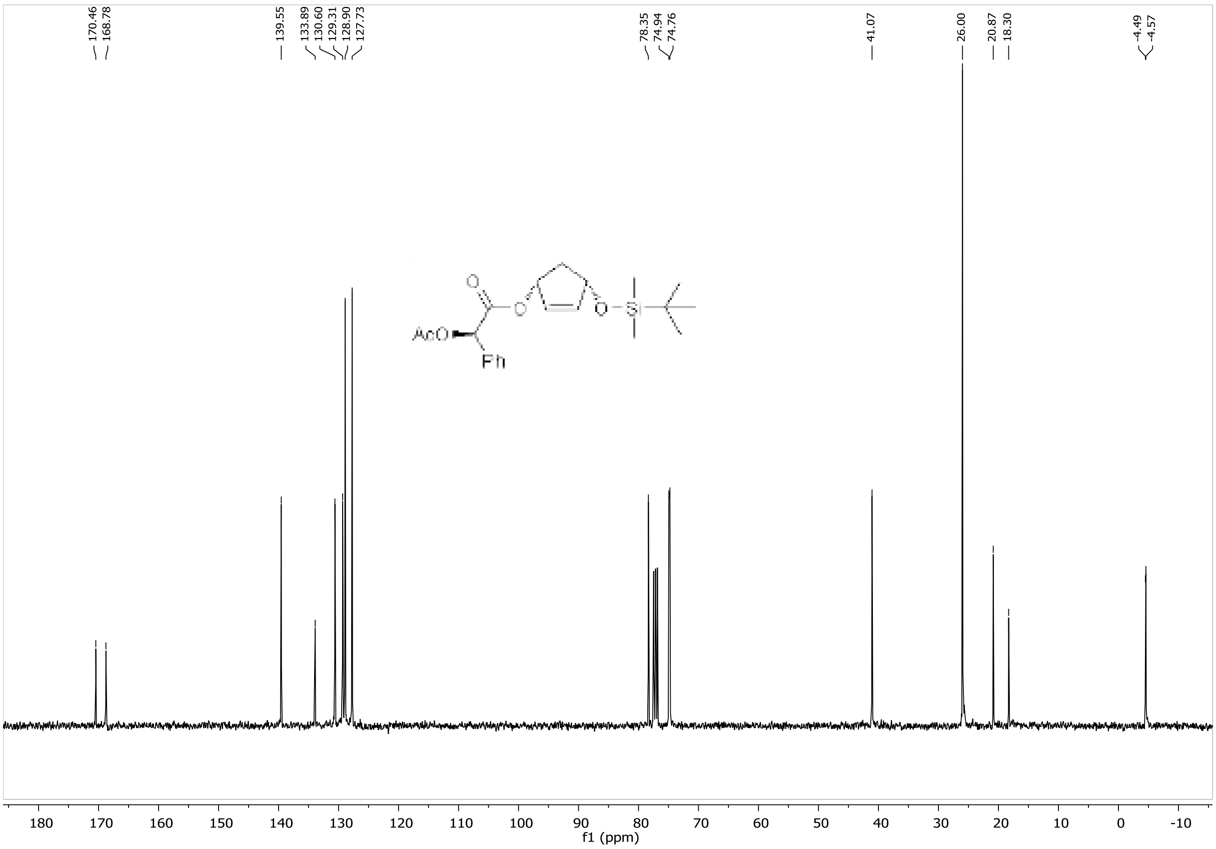
f1 (ppm)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Ph
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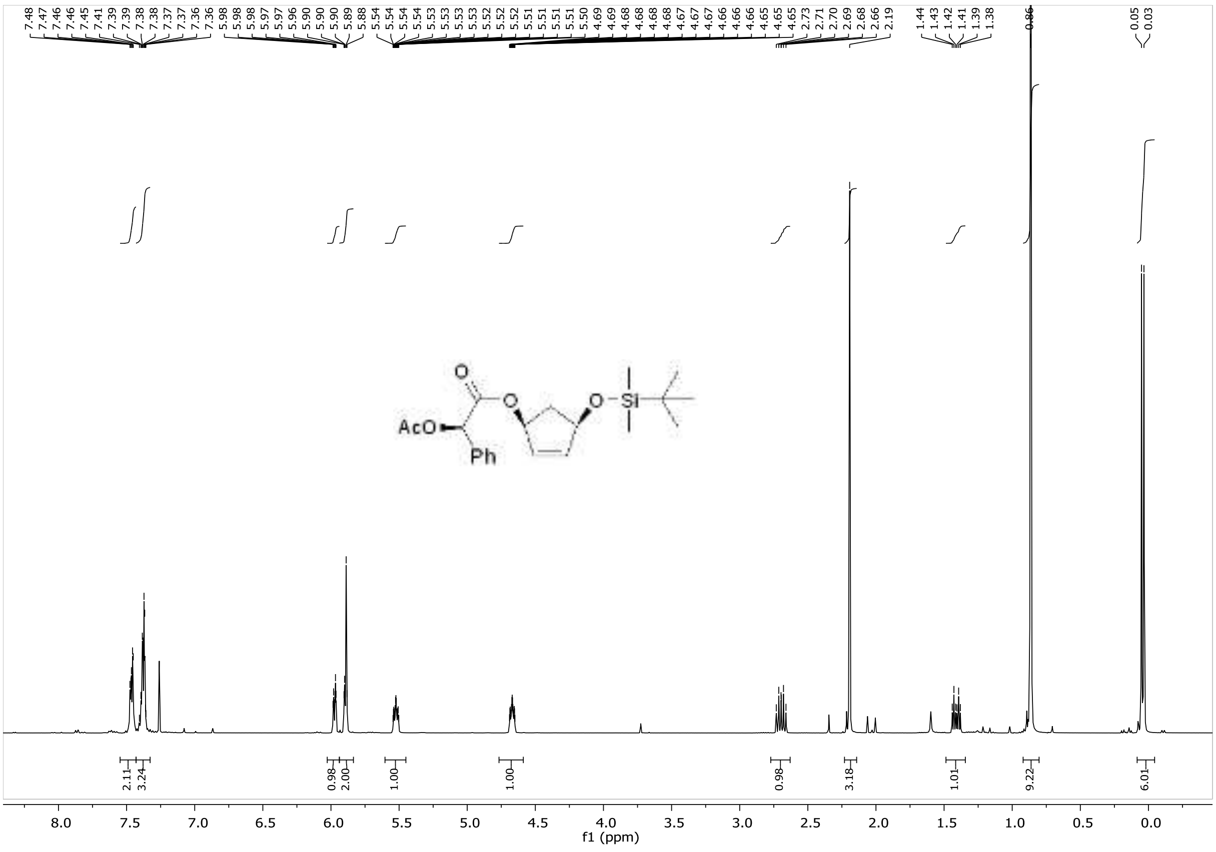
f1 (ppm)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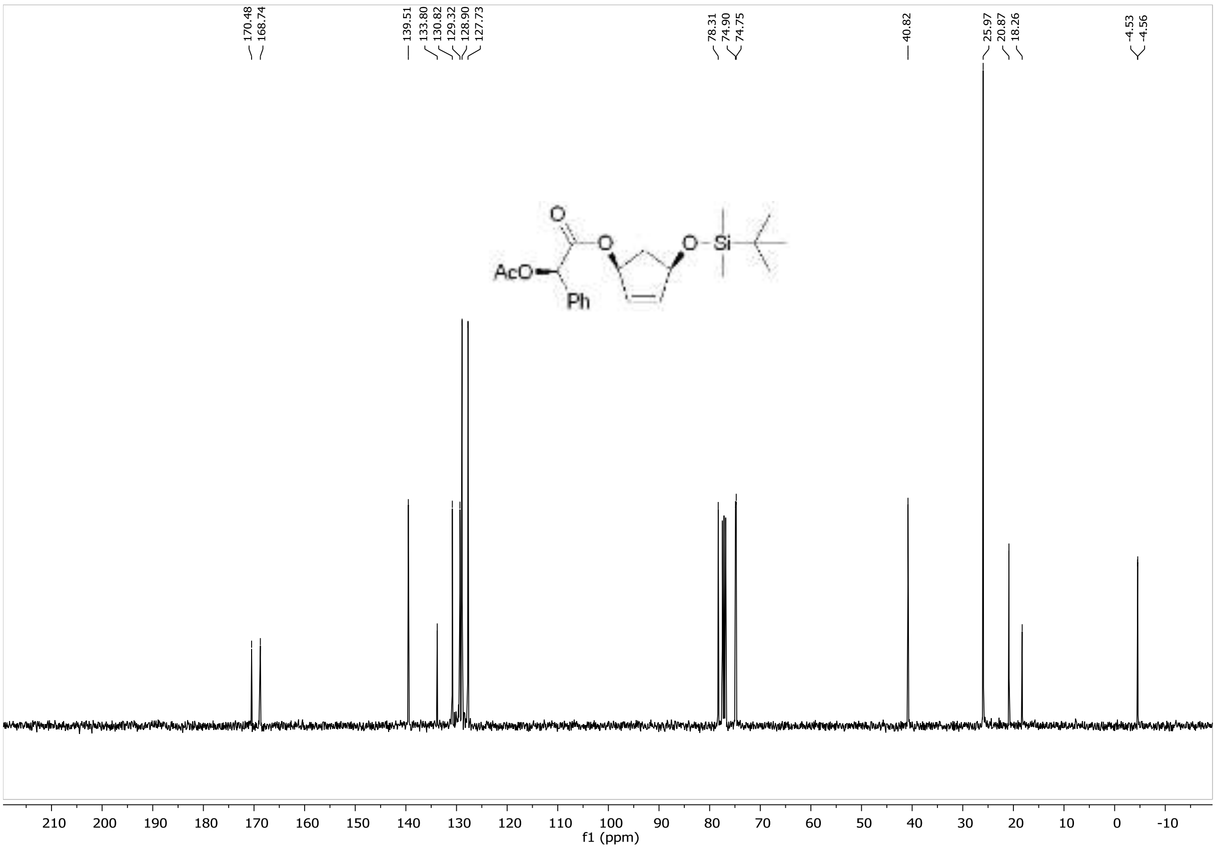
0.86
0.05
0.03
AcO
Ph
O
O
O
O
Si
2.11
3.24
0.98
2.00
1.00
1.00
0.98
3.18
1.01
9.22
6.01
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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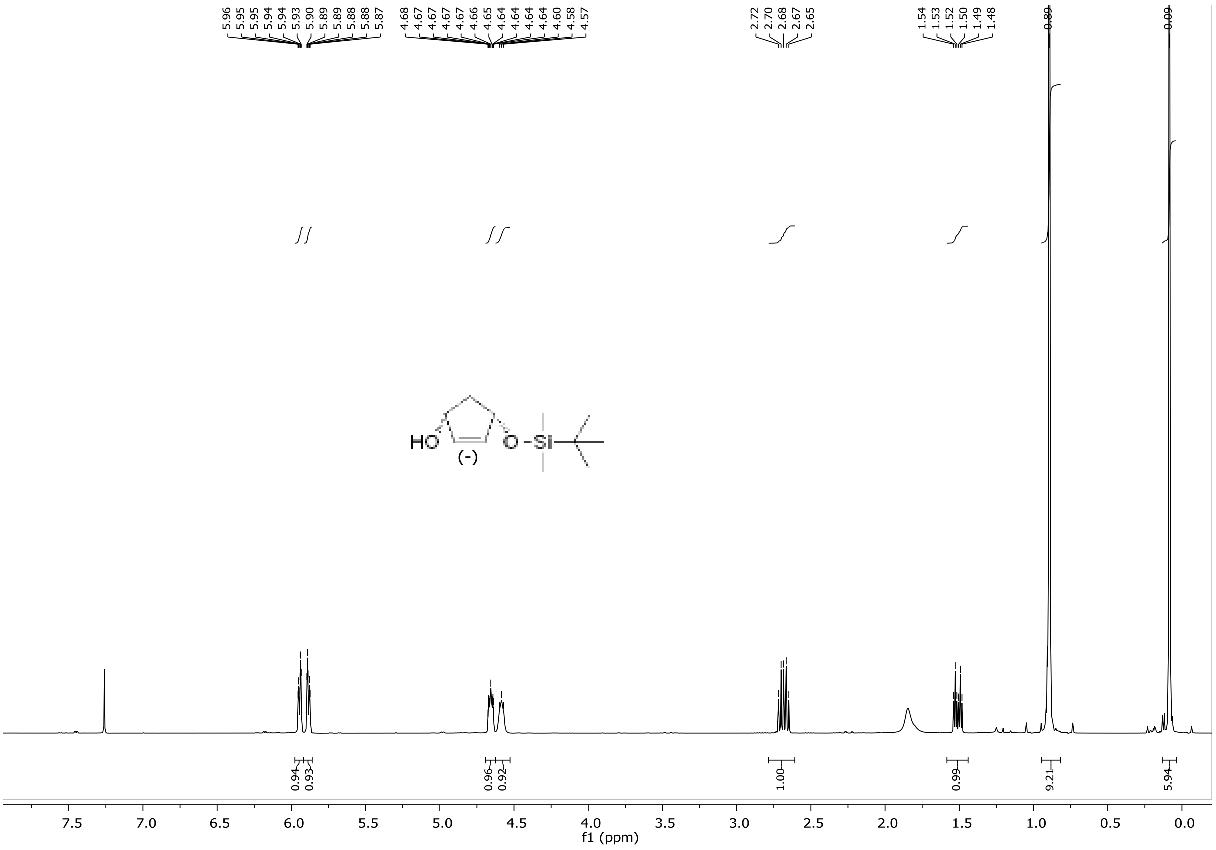
168.74
139.51
133.80
130.82
129.32
128.90
127.73
78.31
74.90
74.75
40.82
25.97
20.87
18.26
-4.53
-4.56
AcO
Ph
O
O
O
O
Si
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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5.95
5.95
5.94
5.94
5.93
5.90
5.89
5.89
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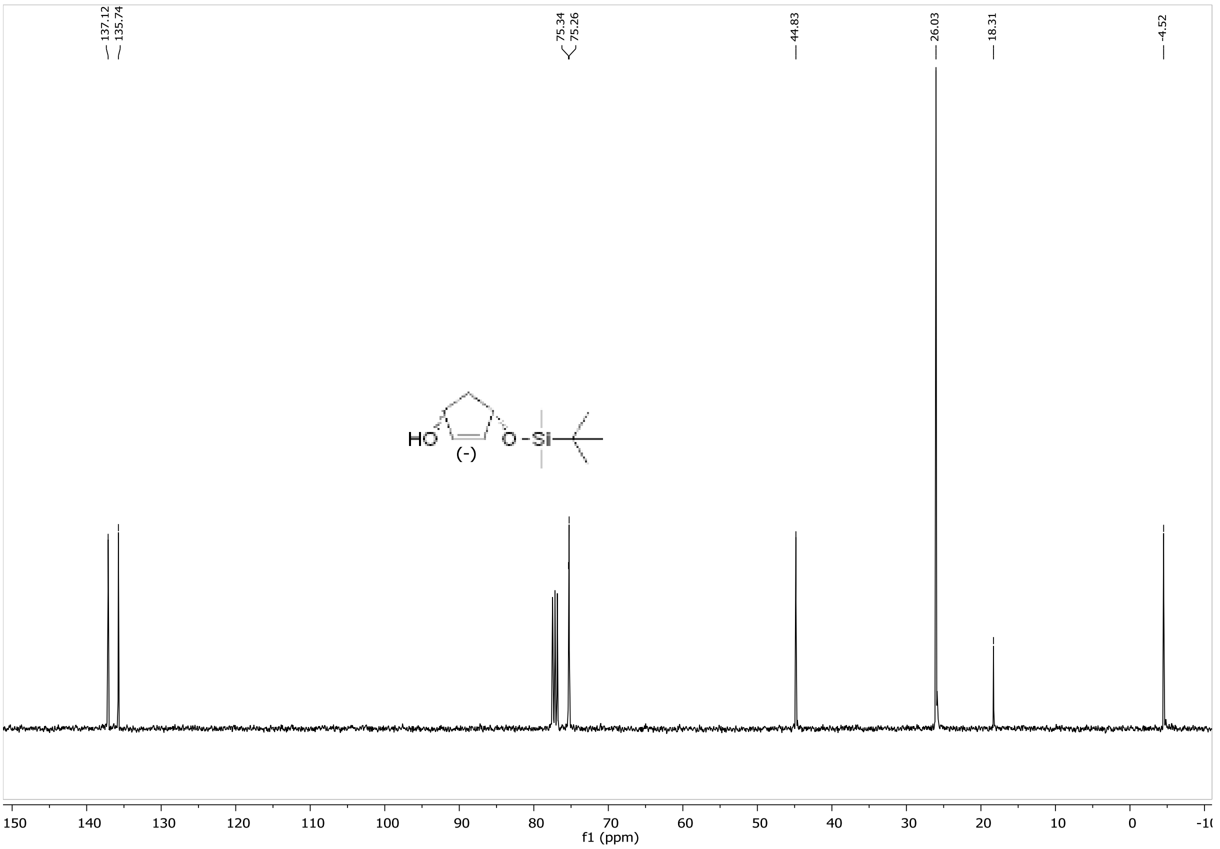
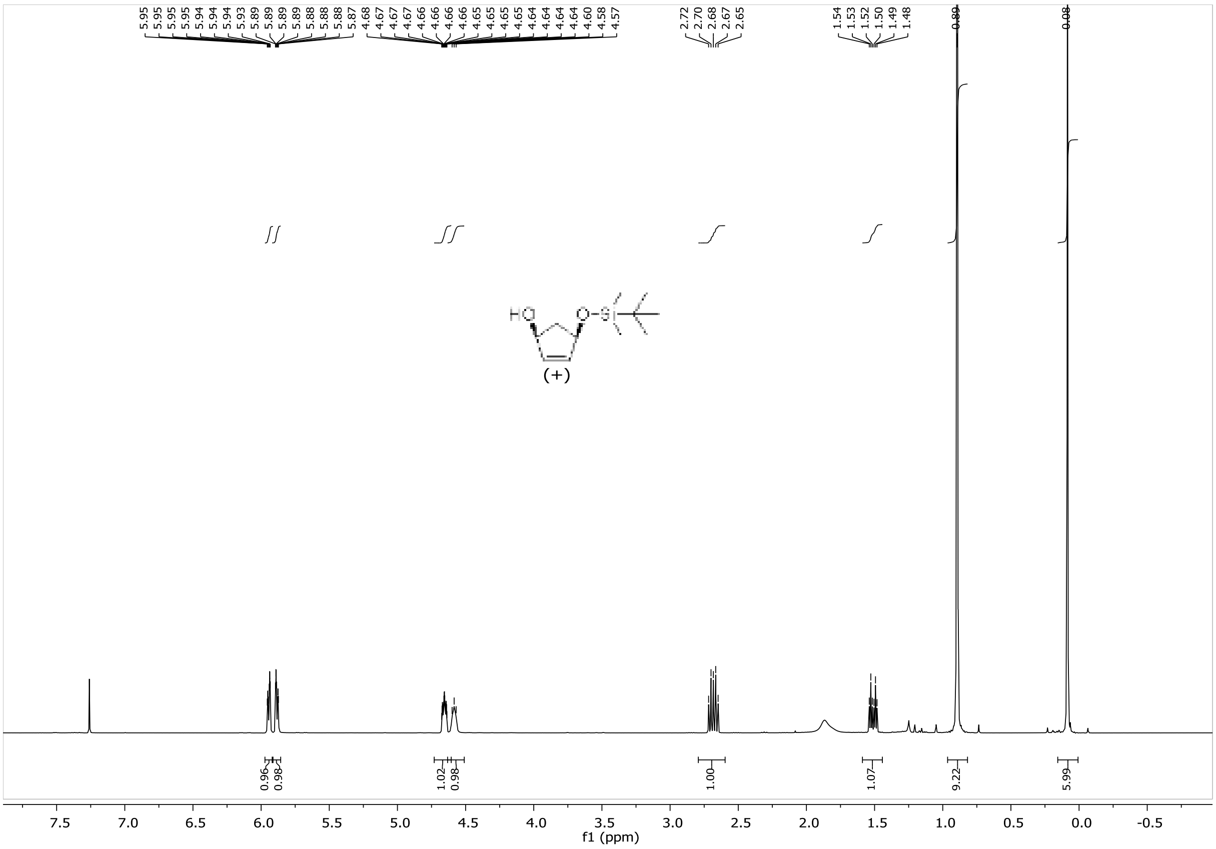
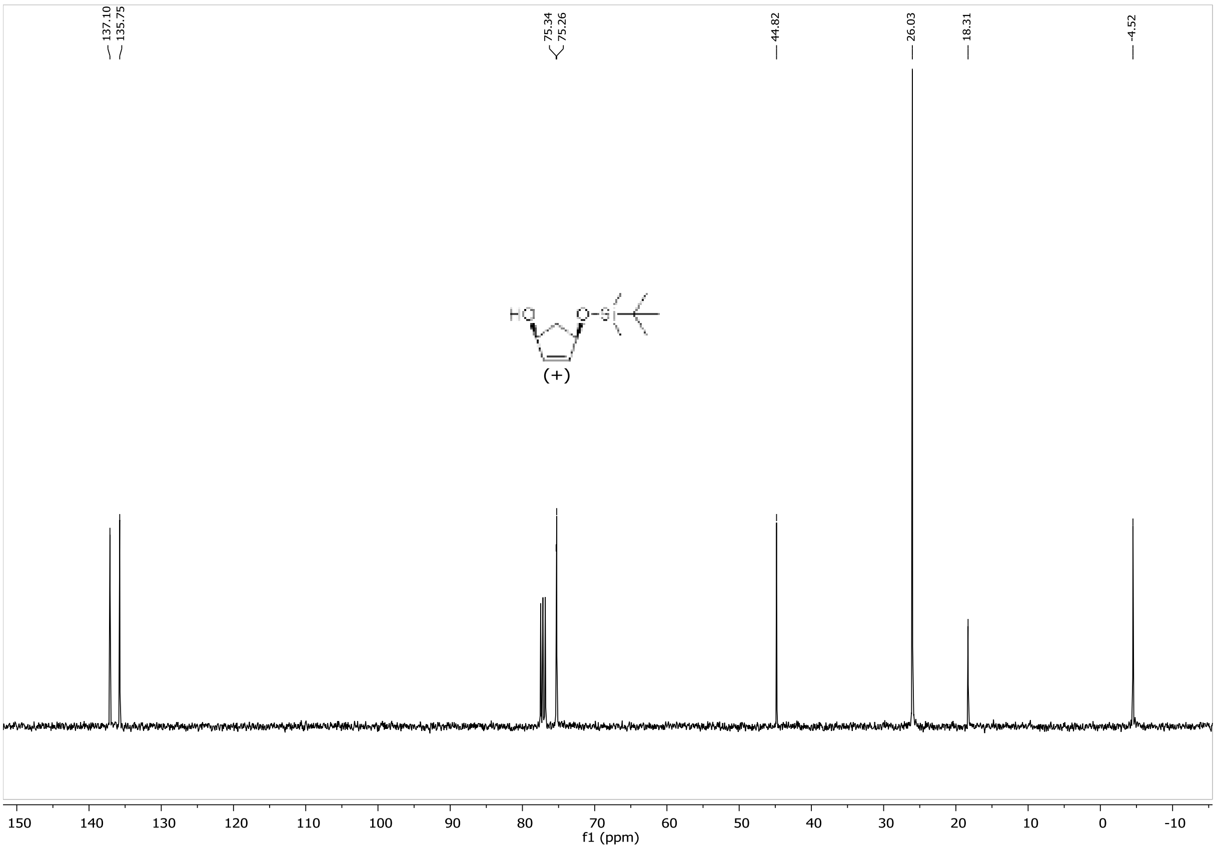
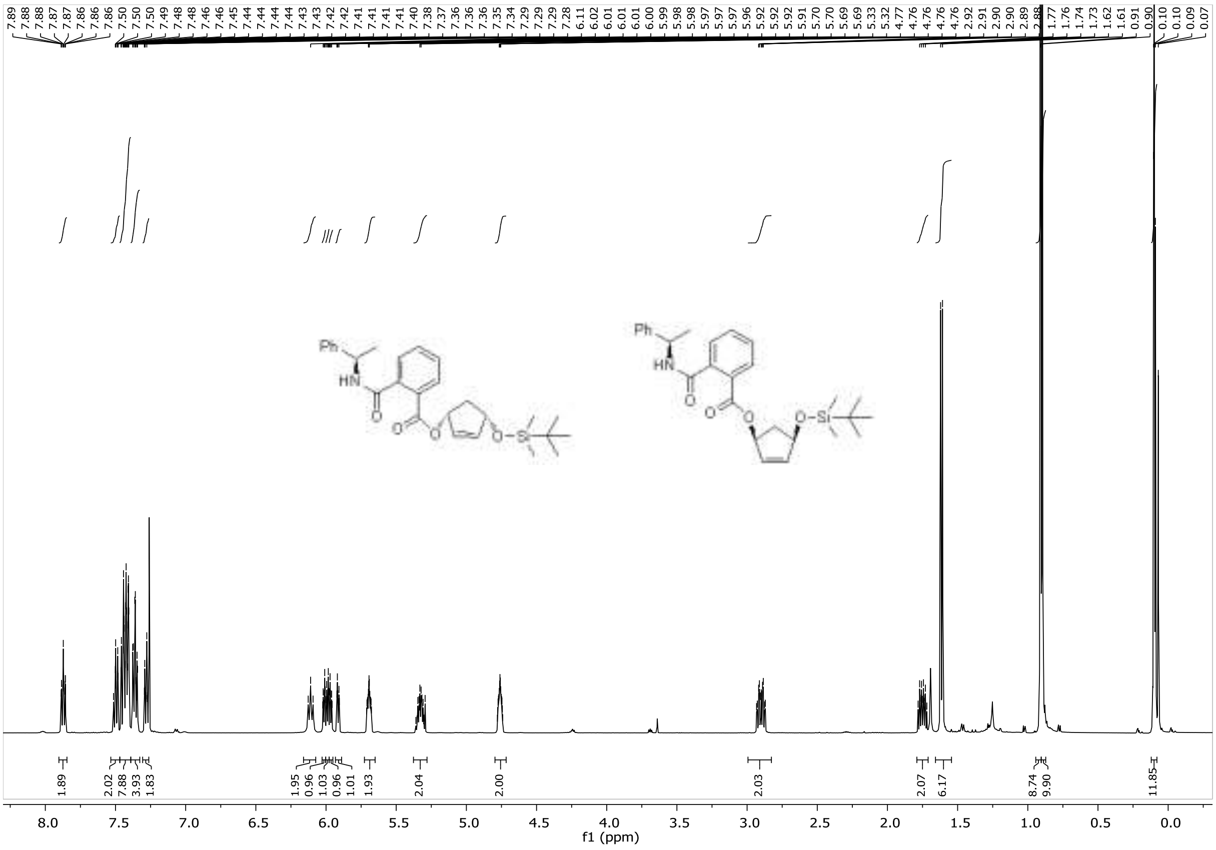
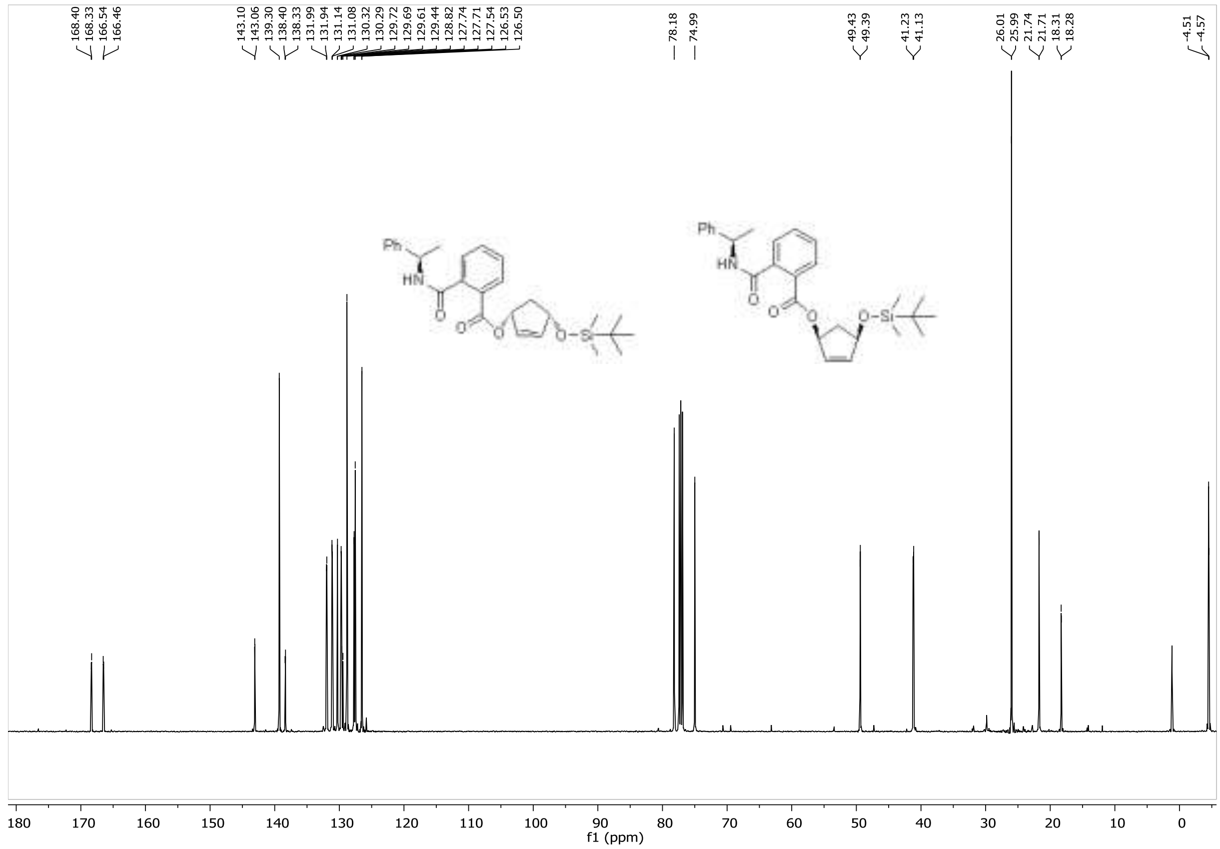
5.88
5.87
4.68
4.67
4.67
4.67
4.67
4.66
4.65
4.64
4.64
4.64
4.64
4.60
4.58
4.57
2.72
2.70
2.68
2.67
2.65
1.54
1.53
1.52
1.50
1.49
1.48
0.89
0.09
HO
(-)
O-Si
0.94
0.93
0.96
0.92
1.00
0.99
9.21
5.94
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

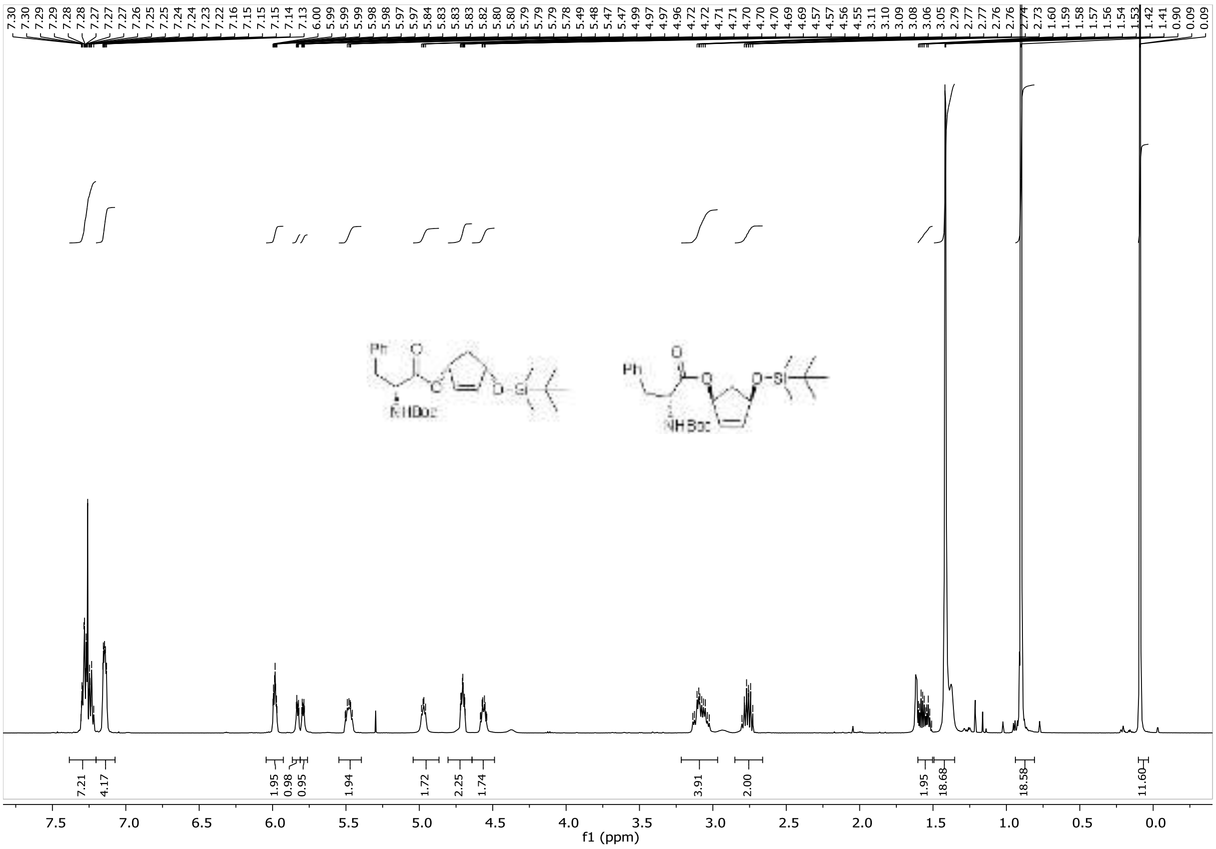

137.12
135.74
75.34
75.26
44.83
26.03
18.31
-4.52
HO
(-)
O-Si
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

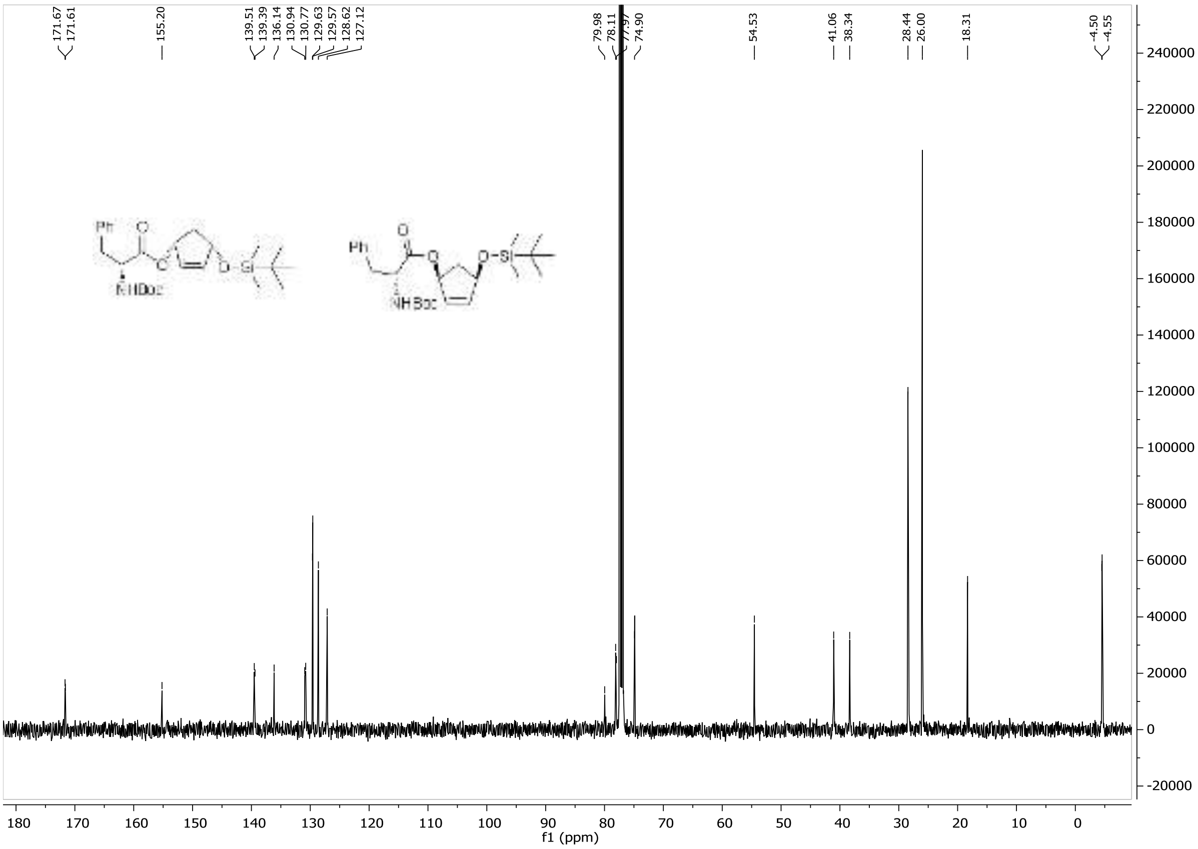

5.95
5.95
5.95
5.95
5.94
5.94
5.94
5.93
5.89
5.89
5.89
5.89
5.88
5.88
5.88
5.87
4.68
4.67
4.67
4.67
4.66
4.66
4.66
4.65
4.65
4.65
4.65
4.64
4.64
4.64
4.64
4.60
4.58
4.57
2.72
2.70
2.68
2.67
2.65
1.54
1.53
1.52
1.50
1.49
1.48
0.89
0.08
HO
O-Si
(+)
0.96
0.98
1.02
0.98
1.00
1.07
9.22
5.99
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

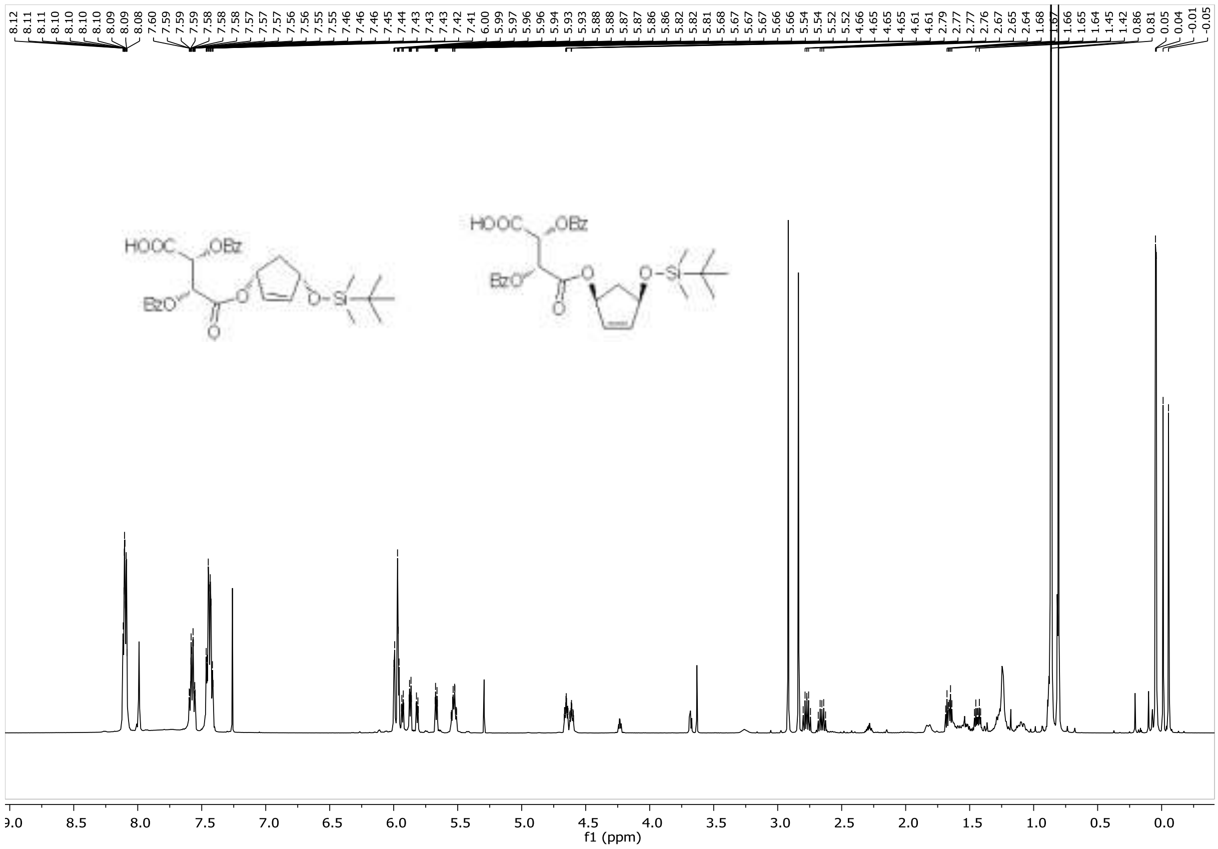

137.10
135.75
75.34
75.26
44.82
26.03
18.31
-4.52
HO
O-Si
(+)
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.89 7.88 7.88 7.87 7.87 7.86 7.86 7.86 7.50 7.50 7.50 7.50 7.49 7.48 7.48 7.46 7.46 7.45 7.44 7.44 7.44 7.44 7.43 7.43 7.42 7.42 7.41 7.41 7.41 7.41 7.40 7.38 7.37 7.36 7.36 7.36 7.35 7.34 7.29 7.29 7.29 7.29 7.28 6.11 6.02 6.01 6.01 6.01 6.00 5.99 5.98 5.98 5.97 5.97 5.97 5.96 5.92 5.92 5.92 5.91 5.91 5.70 5.70 5.69 5.69 5.33 5.33 5.32 4.77 4.76 4.76 4.76 2.92 2.92 2.91 2.91 2.90 2.90 2.89 2.88 1.77 1.76 1.74 1.73 1.62 1.61 0.91 0.90 0.10 0.10 0.09 0.07
Ph
HN
O
O
O
O-Si
1.89
2.02
7.88
3.93
1.83
1.95
0.96
1.03
0.96
1.01
1.93
2.04
2.00
2.03
2.07
6.17
8.74
9.90
11.85
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)


168.40 168.33 166.54 166.46
143.10 143.06 139.30 138.40 138.33 131.99 131.94 131.14 131.08 130.32 130.29 129.72 129.69 129.61 129.44 128.82 127.74 127.71 127.54 126.53 126.50
78.18
74.99
49.43 49.39
41.23 41.13
26.01 25.99 21.74 21.71 18.31 18.28
-4.51 -4.57
Ph
HN
O
O
O
O-Si
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.30 7.30 7.29 7.29 7.28 7.28 7.27 7.27 7.27 7.26 7.25 7.25 7.24 7.24 7.23 7.22 7.16 7.15 7.15 7.14 7.13 6.00 5.99 5.99 5.99 5.98 5.98 5.97 5.97 5.84 5.83 5.83 5.82 5.80 5.80 5.79 5.79 5.78 5.49 5.48 5.47 5.47 4.99 4.97 4.97 4.96 4.72 4.72 4.71 4.71 4.70 4.70 4.69 4.57 4.57 4.56 4.55 3.11 3.10 3.09 3.08 3.06 3.05 2.79 2.77 2.77 2.76 2.74 2.73 1.60 1.59 1.58 1.57 1.56 1.54 1.52 1.42 1.41 0.90 0.09 0.09
Ph
O
NHBoc
O–Si
7.21
4.17
1.95
0.98
0.95
1.94
1.72
2.25
1.74
3.91
2.00
1.95
18.68
18.58
11.60
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)


171.67
171.61
155.20
139.51
139.39
136.14
130.94
130.77
129.63
129.57
128.62
127.12
79.98
78.11
77.97
74.90
54.53
41.06
38.34
28.44
26.00
18.31
-4.50
-4.55
Ph
O
NHBoc
O–Si
240000
220000
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
-20000
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.12
8.11
8.11
8.10
8.10
8.10
8.09
8.08
7.60
7.59
7.59
7.58
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7.58
7.57
7.57
7.57
7.56
7.55
7.55
7.46
7.46
7.46
7.45
7.44
7.43
7.43
7.42
7.41
6.00
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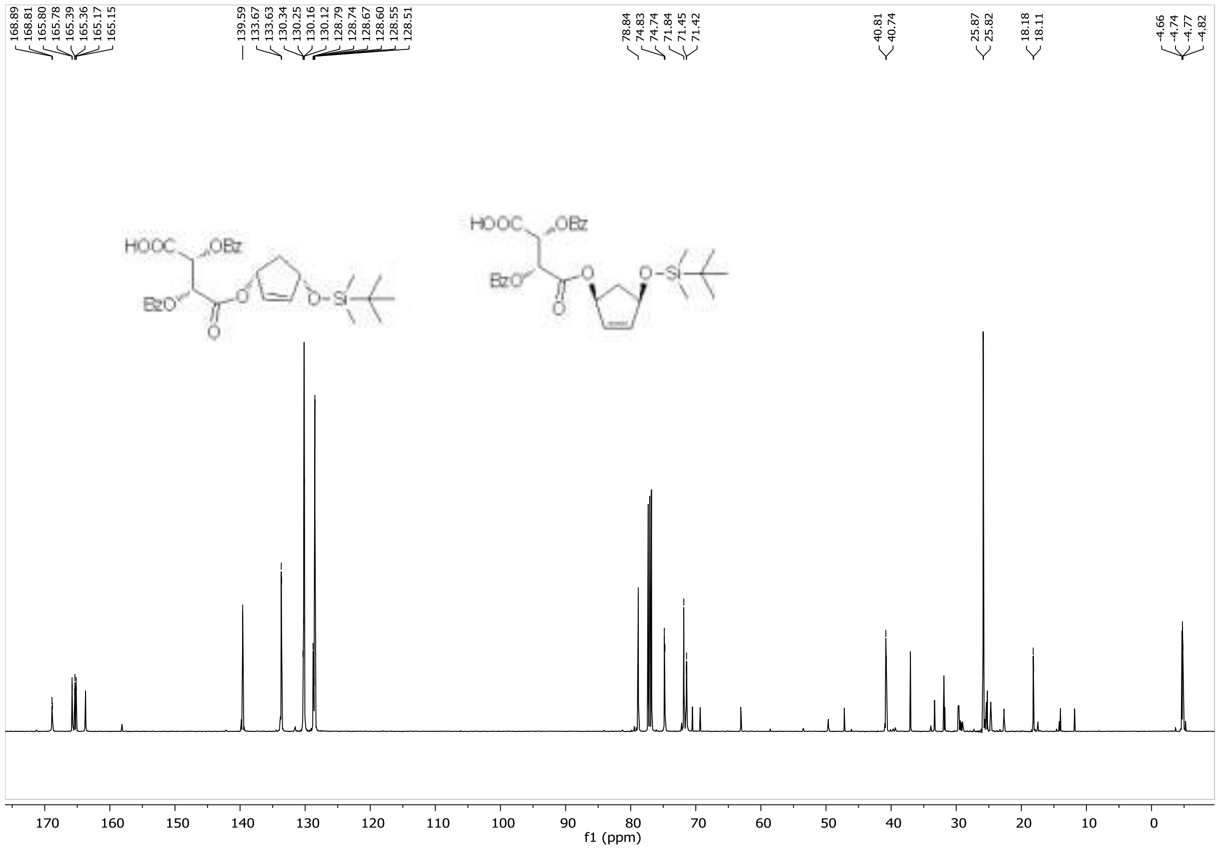
5.97
5.96
5.96
5.94
5.93
5.93
5.88
5.88
5.87
5.87
5.86
5.86
5.82
5.82
5.81
5.68
5.67
5.67
5.67
5.66
5.66
5.54
5.54
5.52
5.52
4.66
4.65
4.65
4.65
4.61
4.61
2.79
2.77
2.77
2.76
2.67
2.65
2.64
1.68
1.67
1.66
1.65
1.64
1.45
1.42
0.86
0.81
0.05
0.04
-0.01
-0.05
HOOC
OBz
BzO
O
Si
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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)


168.89
168.81
165.80
165.78
165.39
165.36
165.17
165.15
139.59
133.67
133.63
130.34
130.25
130.16
130.12
128.79
128.74
128.67
128.60
128.55
128.51
78.84
74.83
74.74
71.84
71.45
71.42
40.81
40.74
25.87
25.82
18.18
18.11
-4.66
-4.74
-4.77
-4.82
HOOC
OBz
BzO
O
Si
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)