

SUPPORTING INFORMATION

Influence of the substituents on the opening of silylepoxy alcohols: 5-*exo* cyclization towards tetrahydrofurans *vs* unexpected side reaction leading to tetrahydropyrans

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3.1 X-Ray Crystallographic Data of compound **4e**

The crystal structure has been deposited at the Cambridge Crystallographic Date Center and allocated the deposition number CCDC: 2118173. This data can be obtained free of charge from the Cambridge Crystallographic Date Center via www.ccdc.cam.ac.uk/data_request/ci

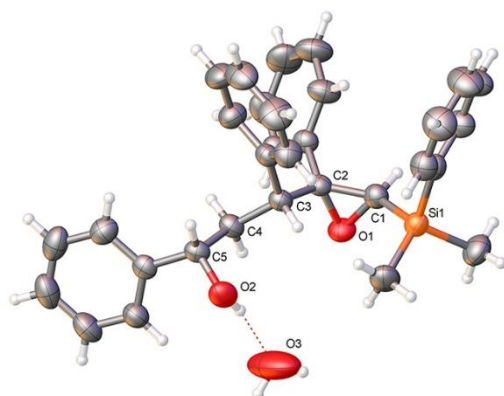


Figure 2. X-ray crystal structure of (1R*, 3S*, 4S*, 5S*)-4,5-epoxy-5-dimethyl(phenyl)silyl-1,3,4-triphenyl-pentan-1-ol **4e**

Experimental

Single crystals of $C_{31}H_{34}O_3Si$ (**4e**) were plates. A suitable crystal was selected and kept at 296.15 K during data collection. Using Olex2, the structure was solved with the ShelXT structure solution program using Direct Methods and refined with the ShelXL refinement package using Least Squares minimization.

X-Ray crystallographic data of **4e** (Figure 2). Crystal data and structure refinement for **4e**

Bond precision: C-C = 0.0029 Å Wavelength=1.54184

Cell: a=9.31052(11) b=18.41150(17) c=16.27509(14)
 alpha=90 beta=97.2884(9) gamma=90

Temperature: 296 K

	Calculated	Reported Volume
	2767.35(5)	2767.35(5)
Space group	P 21/n	P 1 21/n 1
Hall group	-P 2yn	-P 2yn
Moiety formula	C31 H32 O2 Si, H2 O	C31 H32 O2 Si, H2 O Sum
formula	C31 H34 O3 Si	C31 H34 O3 Si
Mr	482.67	482.67
Dx, g cm ⁻³	1.158	1.159
Z	4	4
Mu (mm ⁻¹)	0.967	0.967
F000	1032.0	1032.0
F000'	1035.73	
h, k, lmax	11, 23, 20	11, 23, 20
Nref	5686	5634
Tmin, Tmax	0.612, 0.722	0.818, 0.888
Tmin'	0.541	

Correction method= # Reported T Limits: Tmin=0.818 Tmax=0.888
AbsCorr = GAUSSIAN

Data completeness= 0.991 Theta(max)= 74.811

R(reflections)= 0.0577(4945) wR2(reflections)= 0.1696(5634)

S = 1.040 Npar= 323

3.2 X-Ray Crystallographic Data of compound 9

The crystal structure has been deposited at the Cambridge Crystallographic Data Center and allocated the deposition number CCDC: 2118174. This data can be obtained free of charge from the Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/ci

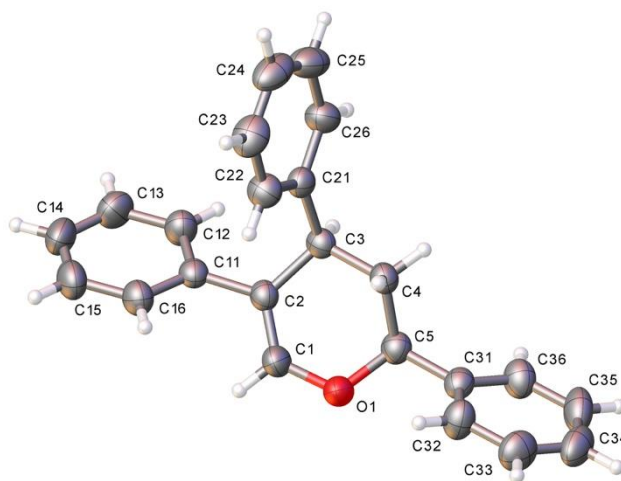


Figure 3. X-ray crystal structure of (2R*,4S*)-2,4,5-triphenyl-3,4-dihydro-2H-pyran **9**

Experimental

Single crystals of $C_{23}H_{20}O$ (**9**) were plates. A suitable crystal was selected and kept at 298.15 K during data collection. Using Olex2, the structure was solved with the ShelXT structure solution program using Direct Methods and refined with the ShelXL refinement package using Least Squares minimisation.

X-Ray crystallographic data of **9** (Figure 3). Crystal data and structure refinement for **9**

Bond precision: C-C = 0.0023 Å

Wavelength=0.71073

Cell: a=16.6186(11) b=6.2601(4) c=16.7987(12)
alpha=90 beta=100.025(7) gamma=90

Temperature: 298 K

	Calculated	Reported
Volume	1721.0(2)	1720.9(2)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C23 H20 O	C23 H20 O
Sum formula	C23 H20 O	C23 H20 O
Mr	312.39	312.39
Dx, g cm ⁻³	1.206	1.206
Z	4	4
Mu (mm ⁻¹)	0.072	0.072
F000	664.0	664.0
F000'	664.27	
h, k, lmax	23, 8, 23	22, 8, 23
Nref	4876	4026
Tmin, Tmax	0.976, 0.985	0.981, 0.987
Tmin'	0.975	

Correction method= # Reported T Limits: Tmin=0.981 Tmax=0.987 AbsCorr = GAUSSIAN

Data completeness= 0.826

Theta(max)= 29.698

R(reflections)= 0.0479(2752)

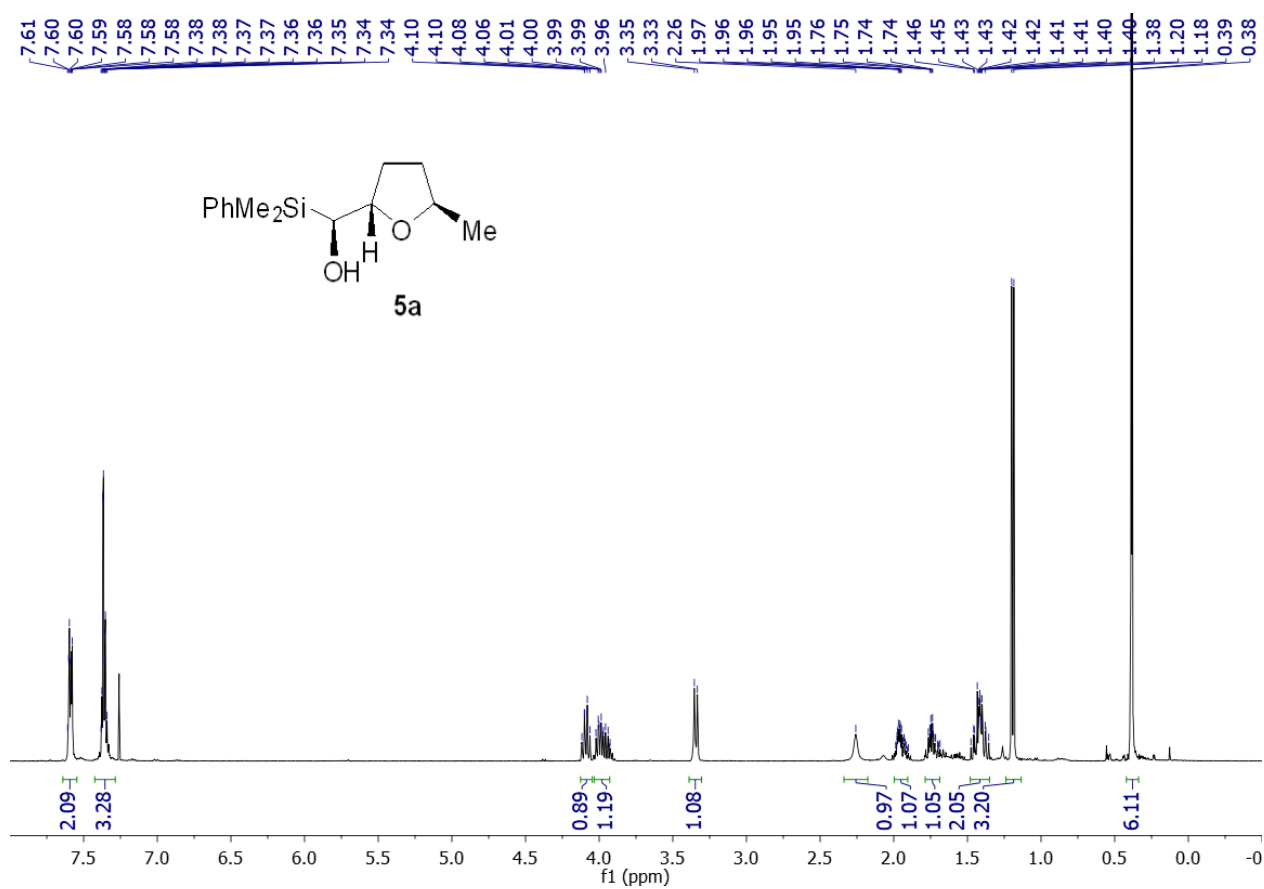
wR2(reflections)= 0.1192(4026)

S = 1.023

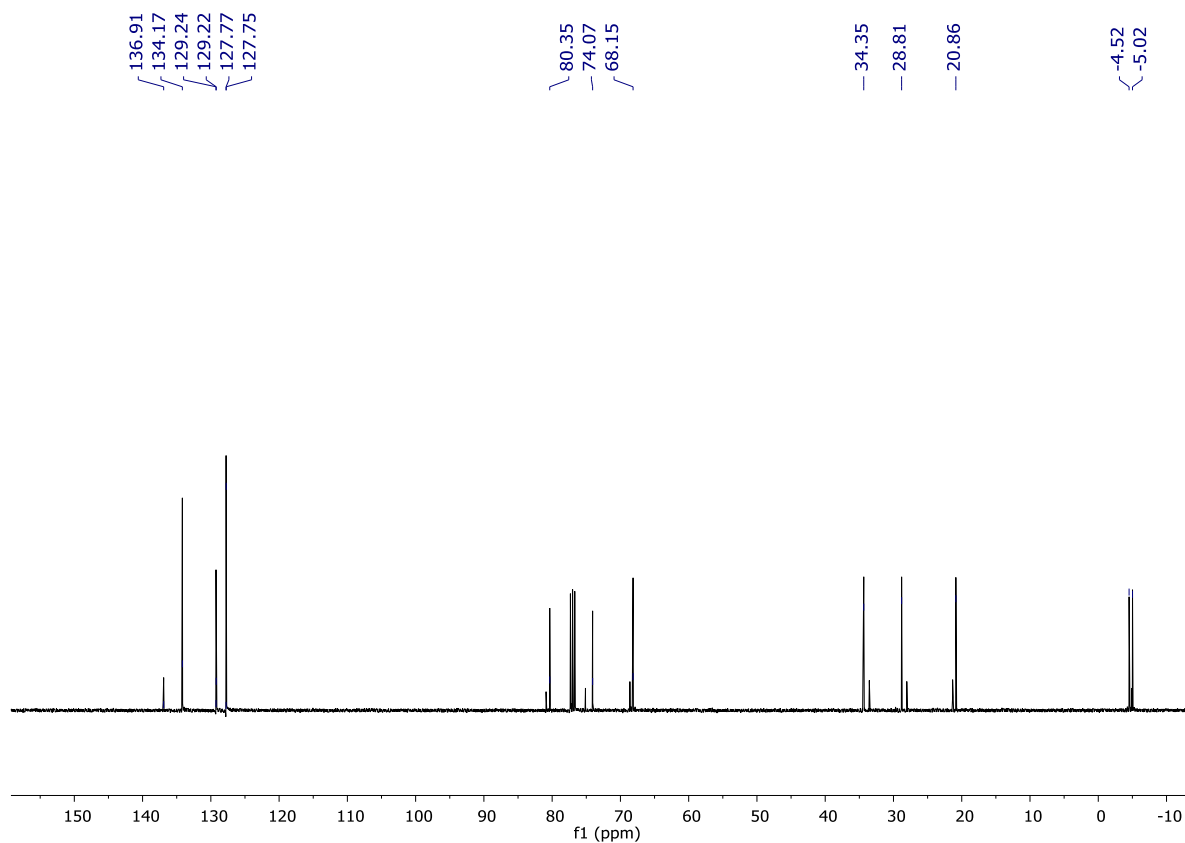
Npar= 218

4. NMR spectra of compounds

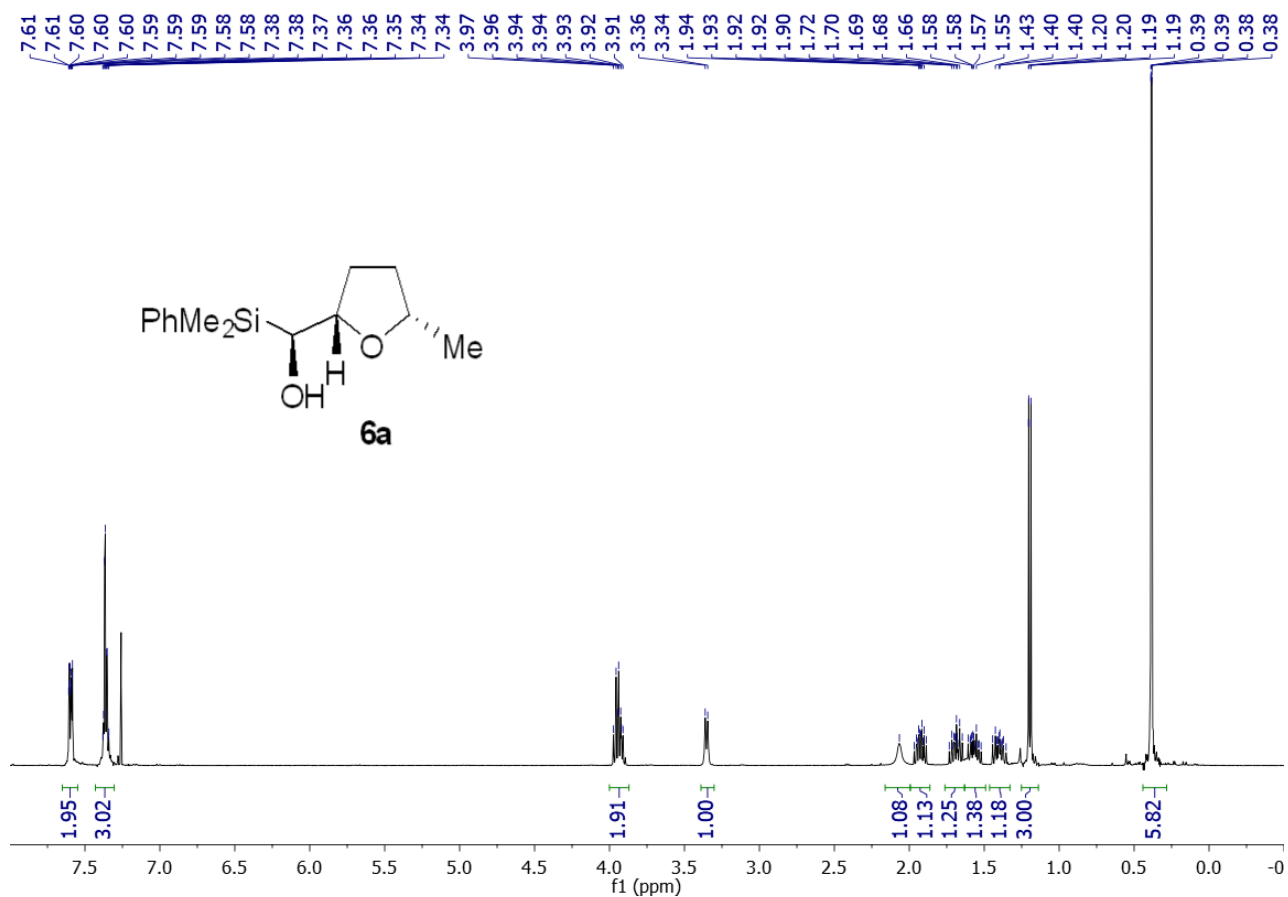
^1H NMR (400 MHz, CDCl_3)



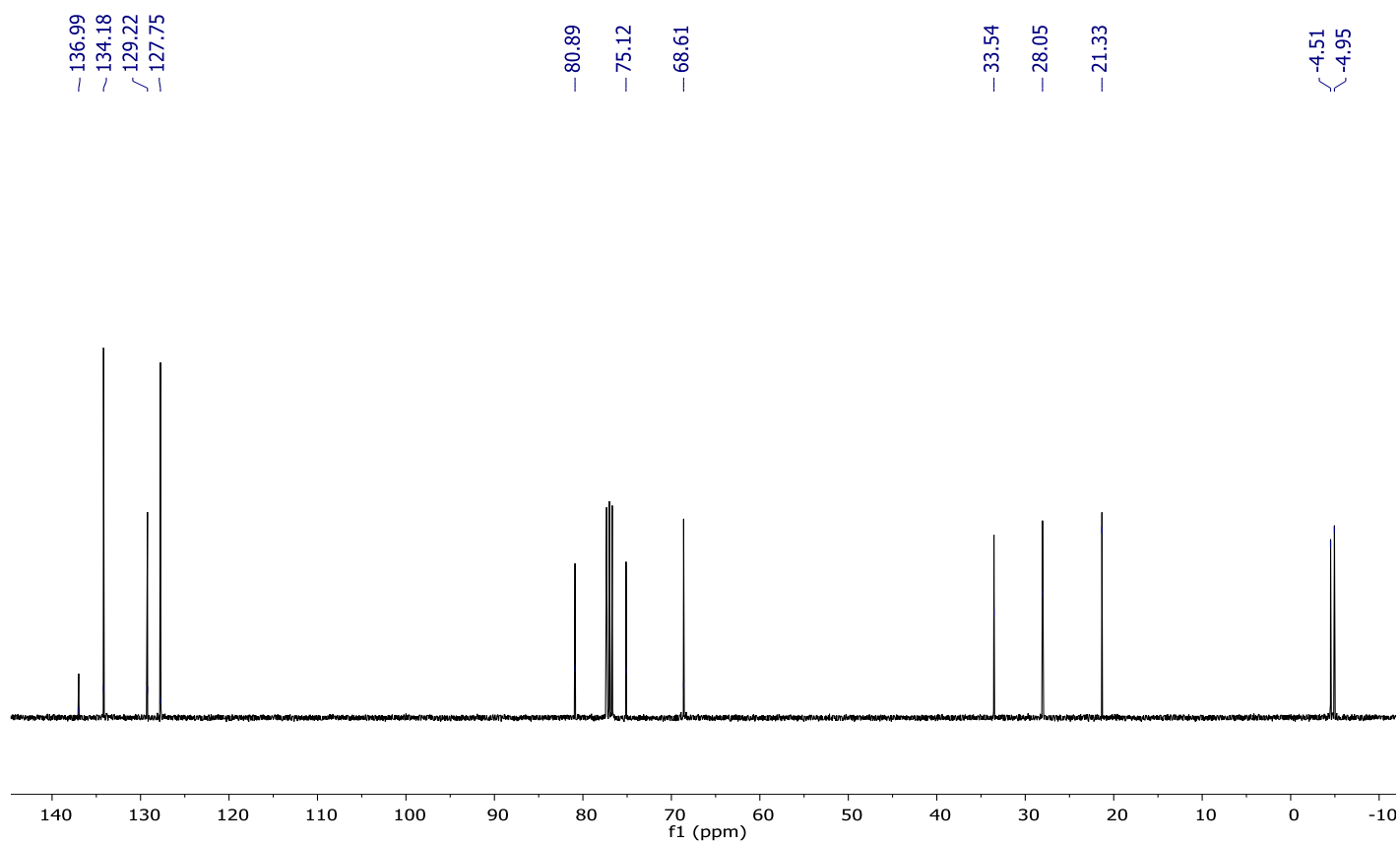
^{13}C NMR (101 MHz, CDCl_3)



¹H NMR (400 MHz, CDCl₃)



¹³C NMR (101 MHz, CDCl₃)

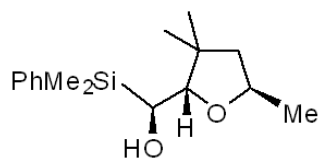


¹H NMR (400 MHz, CDCl₃)

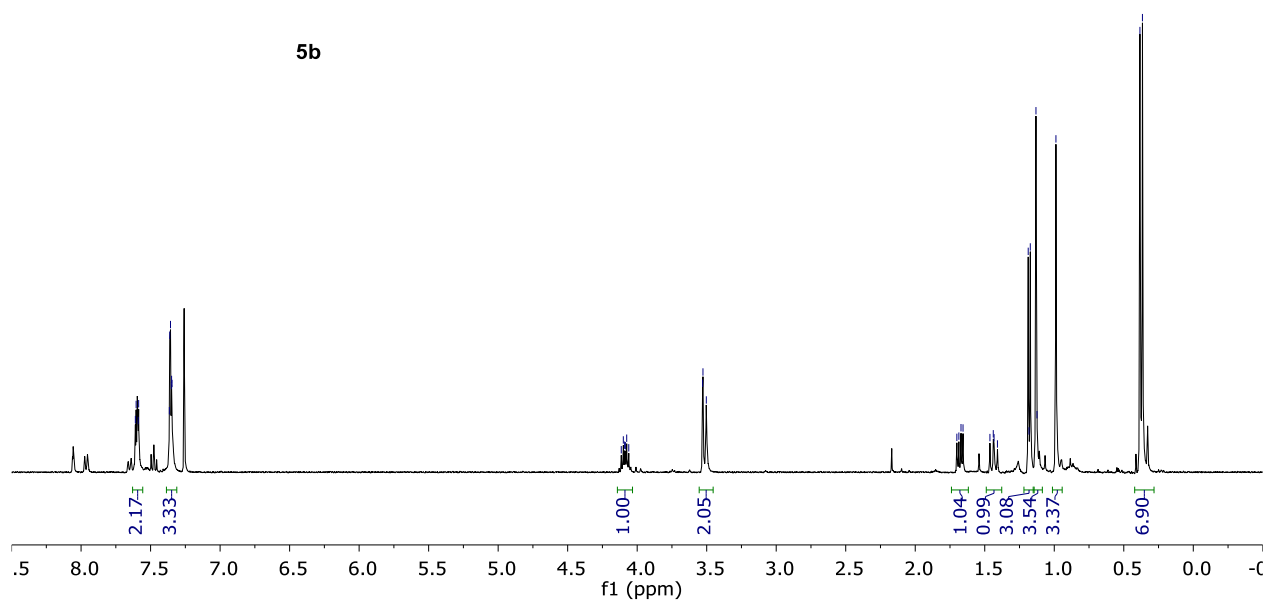
7.61
7.60
7.59
7.59
7.36
7.36
7.35
7.35

4.11
4.10
4.09
4.08
4.08
4.06
3.53
3.53
3.50

1.70
1.69
1.67
1.66
1.46
1.44
1.43
1.41
1.19
1.18
1.17
1.13
1.12
0.99
0.38
0.37



5b



¹³C NMR (101 MHz, CDCl₃)

137.30
134.46
134.22
130.23
129.79
129.09
127.87
127.69

88.31

73.28

65.02

49.29

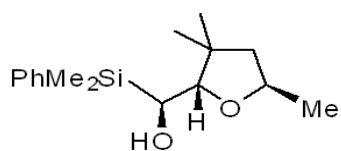
42.28

29.19

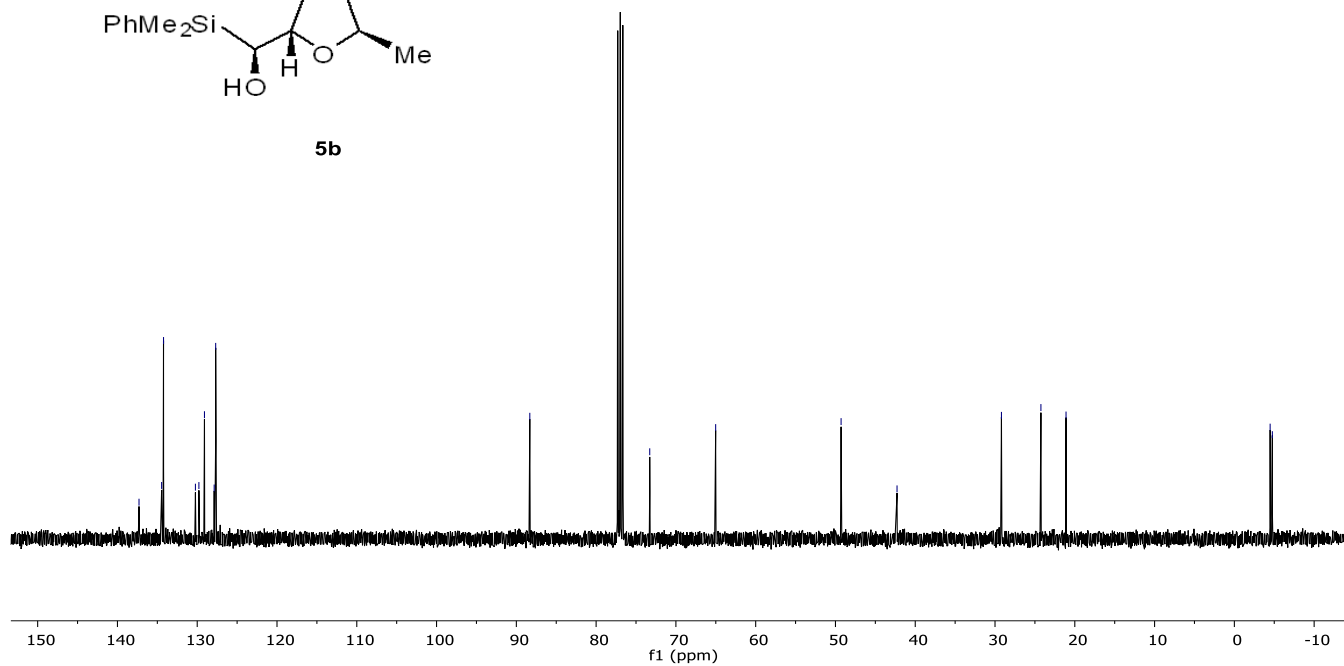
24.25

21.10

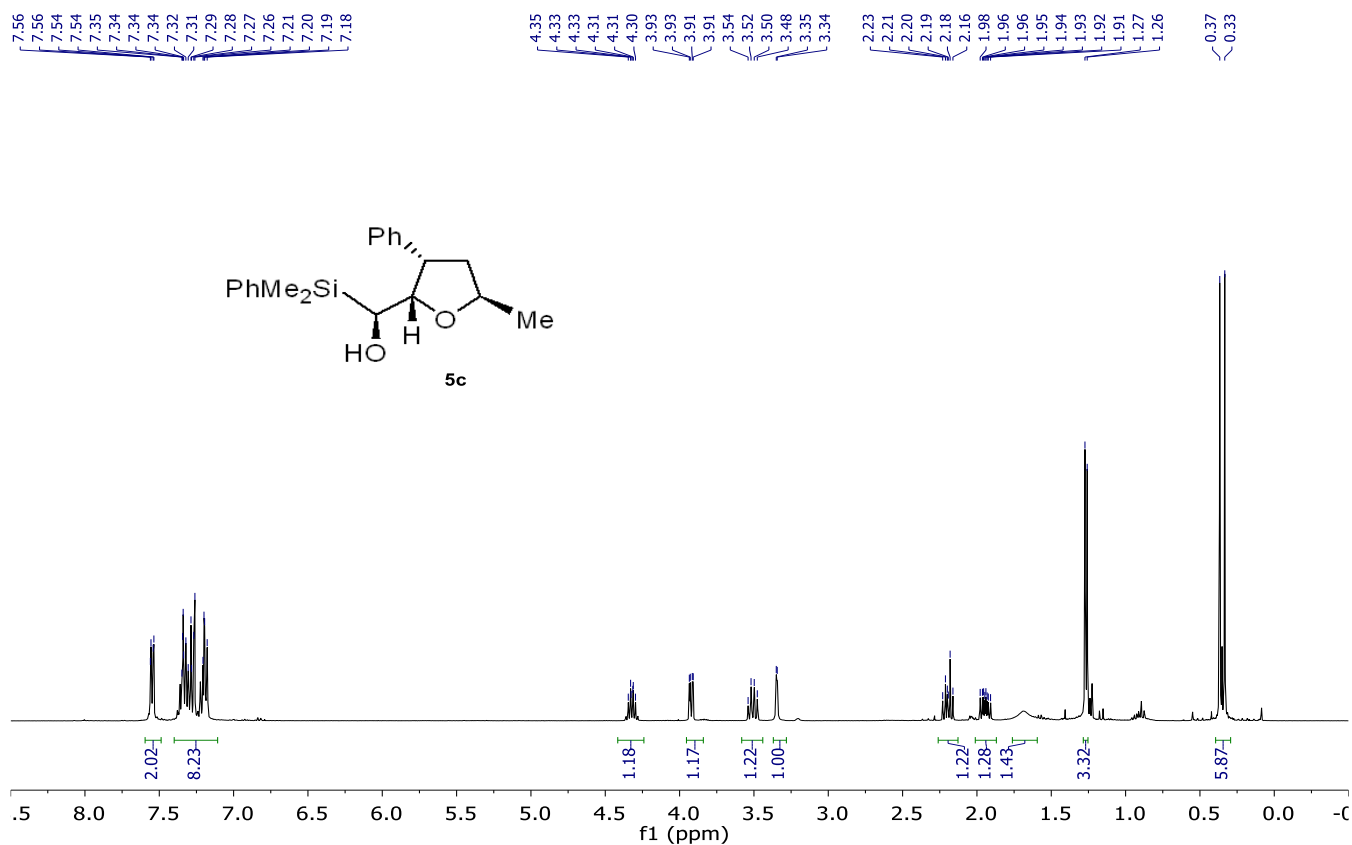
4.49
4.76



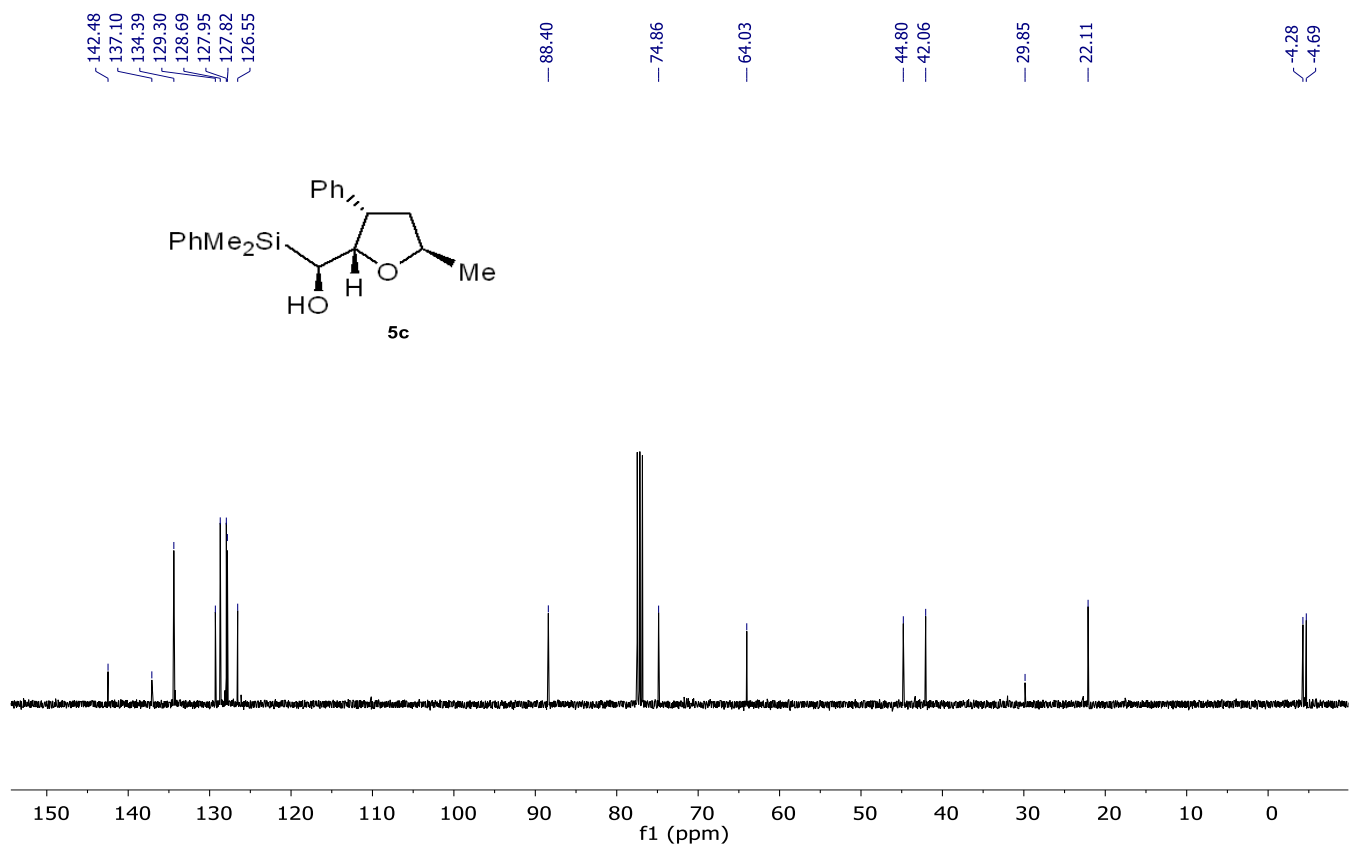
5b



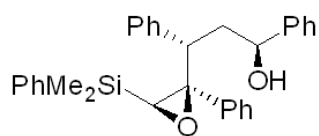
¹H NMR (400 MHz, CDCl₃)



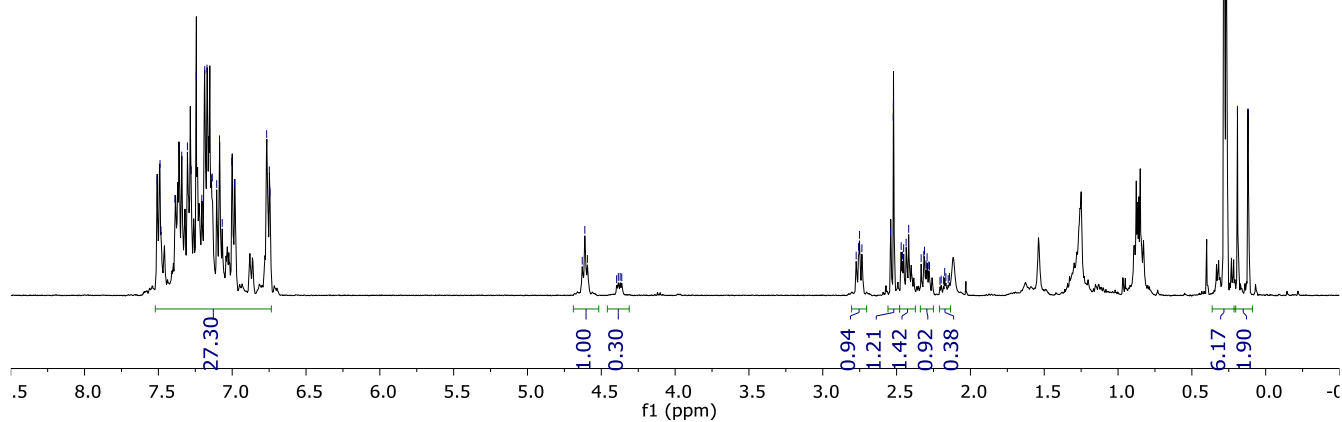
¹³C NMR (101 MHz, CDCl₃)



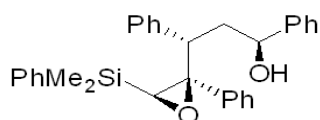
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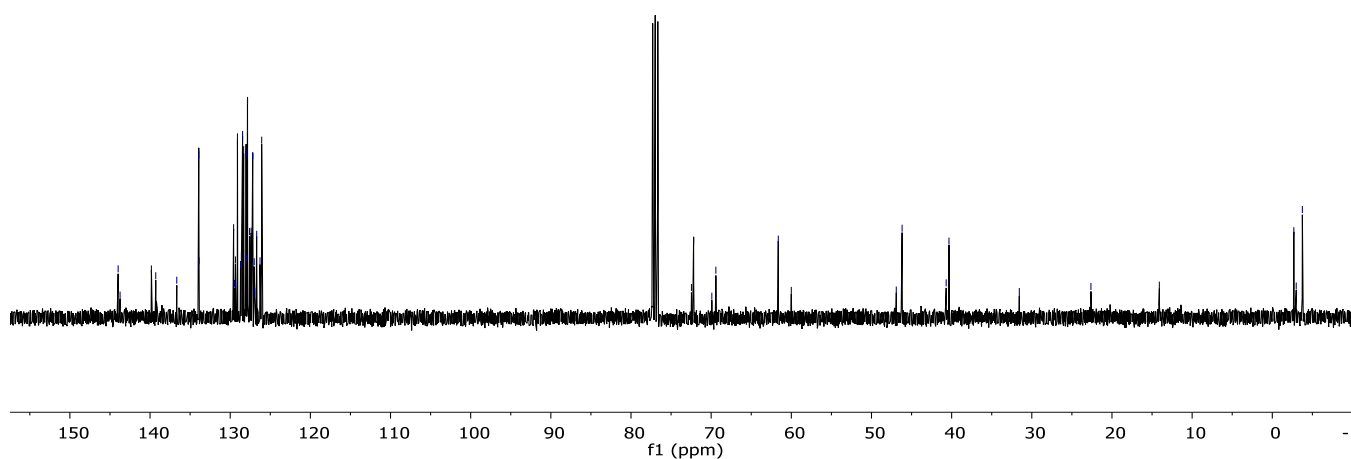
4d (+ minor)



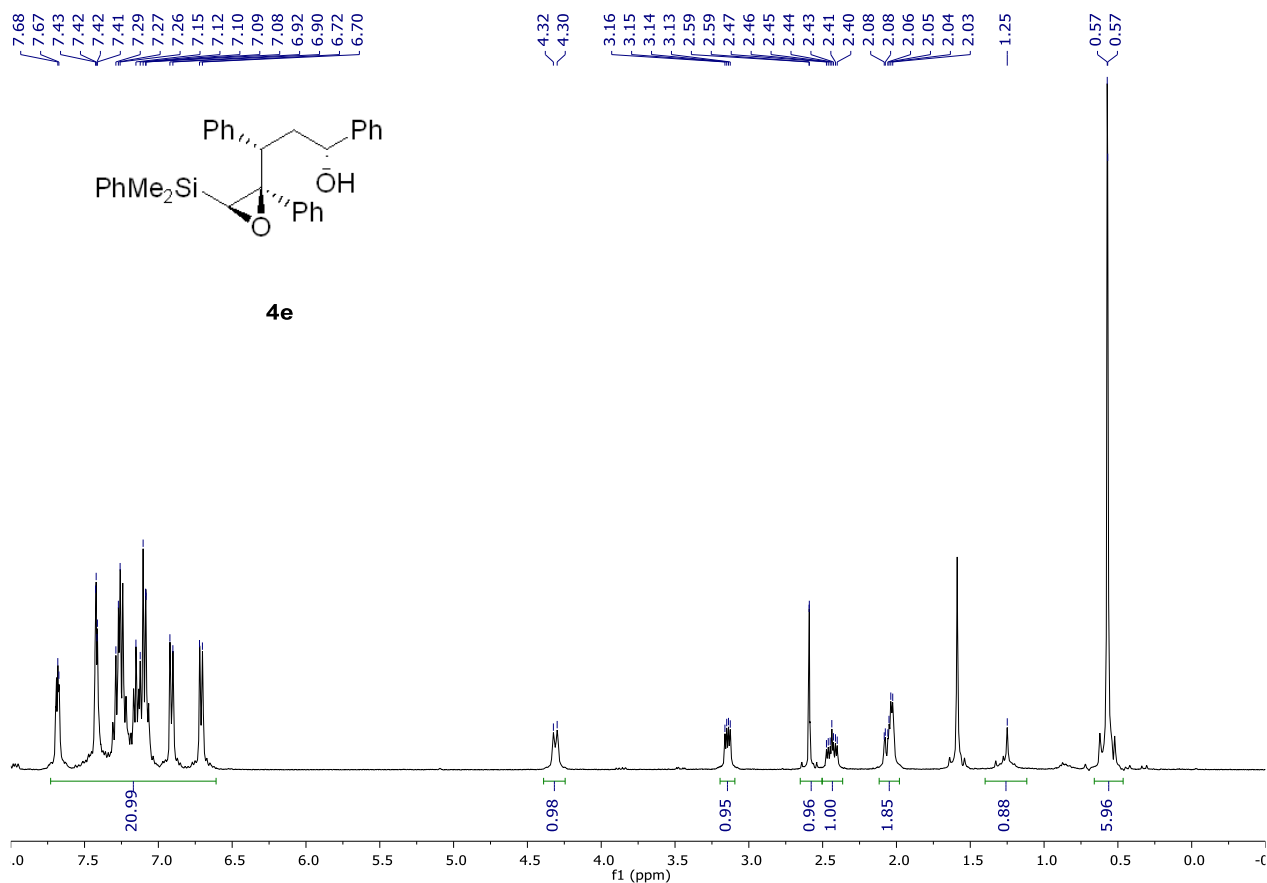
¹³C NMR (101 MHz, CDCl₃)



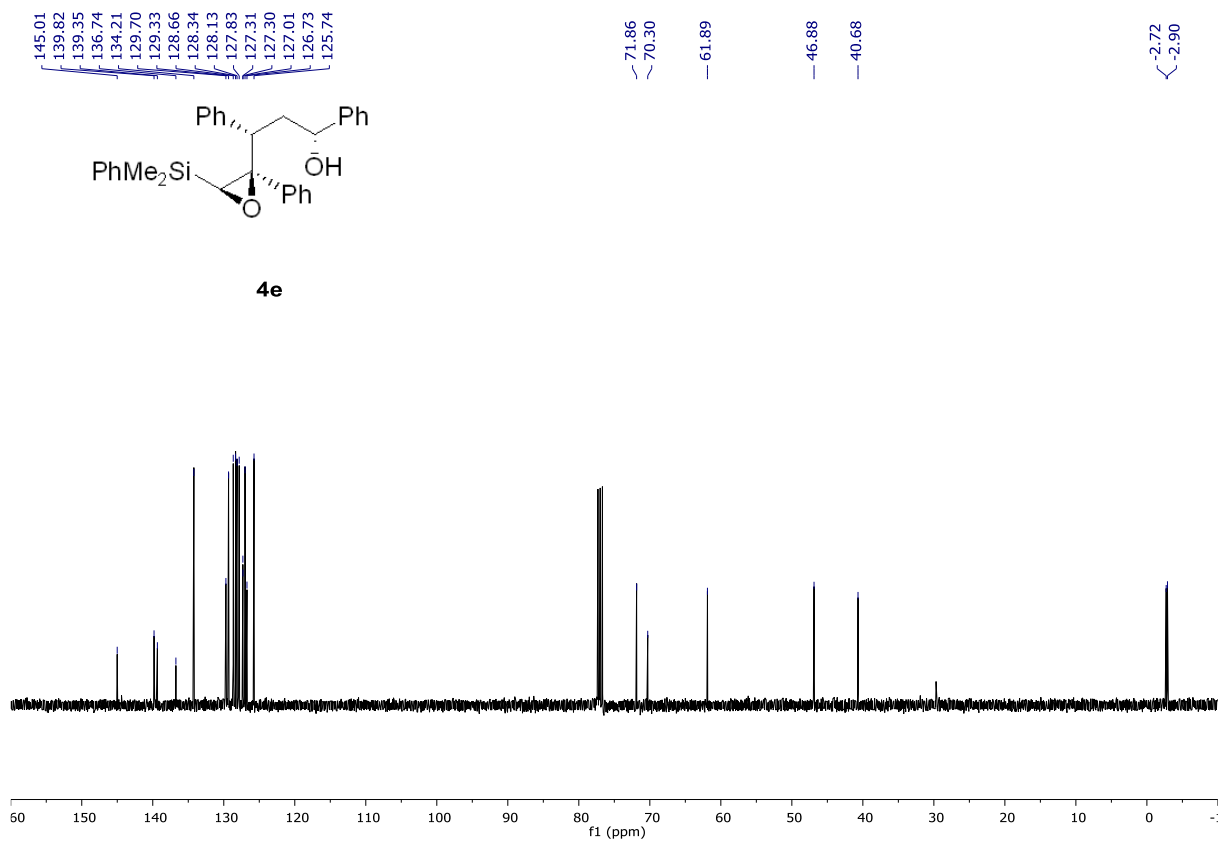
4d (+ minor)



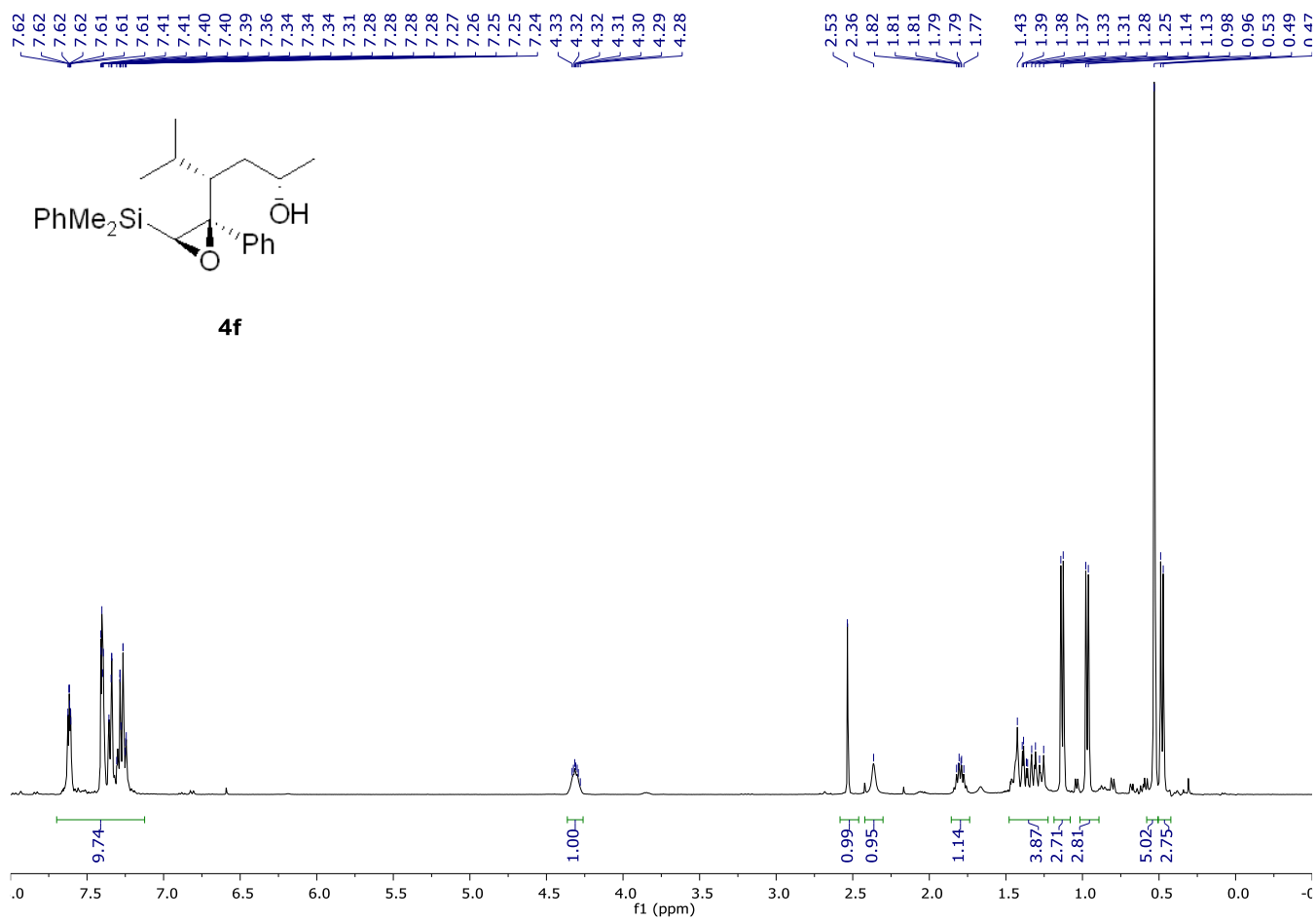
¹H NMR (400 MHz, CDCl₃)



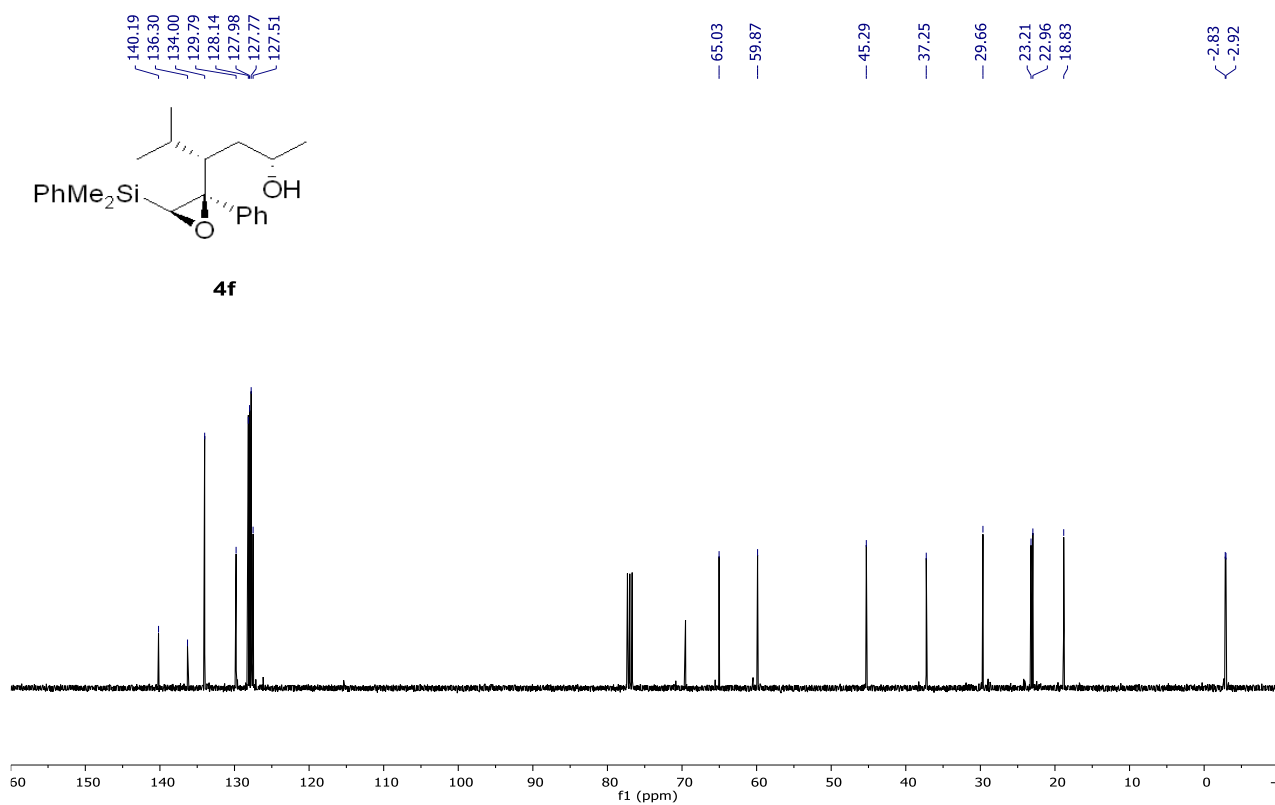
¹³C NMR (101 MHz, CDCl₃)



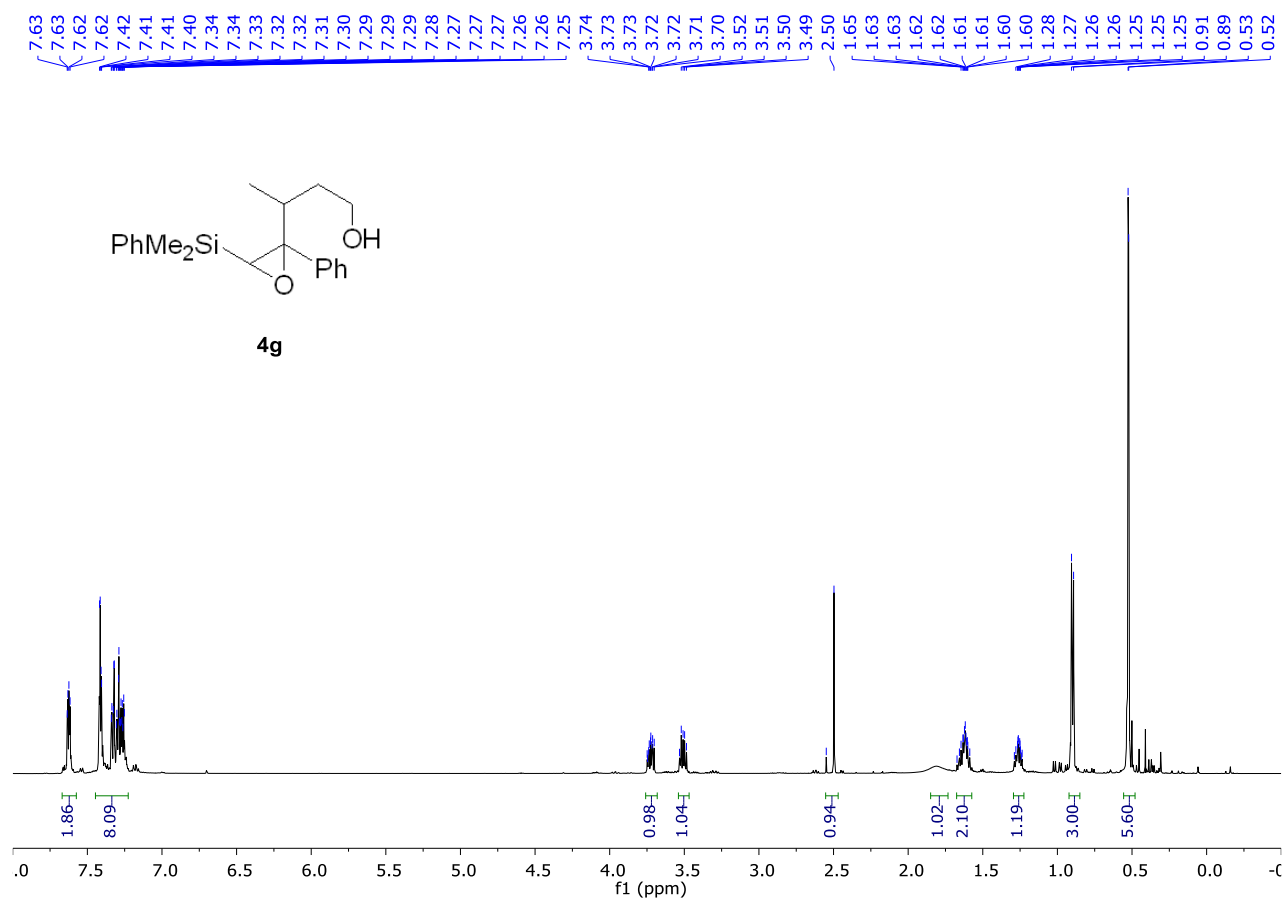
¹H NMR (400 MHz, CDCl₃)



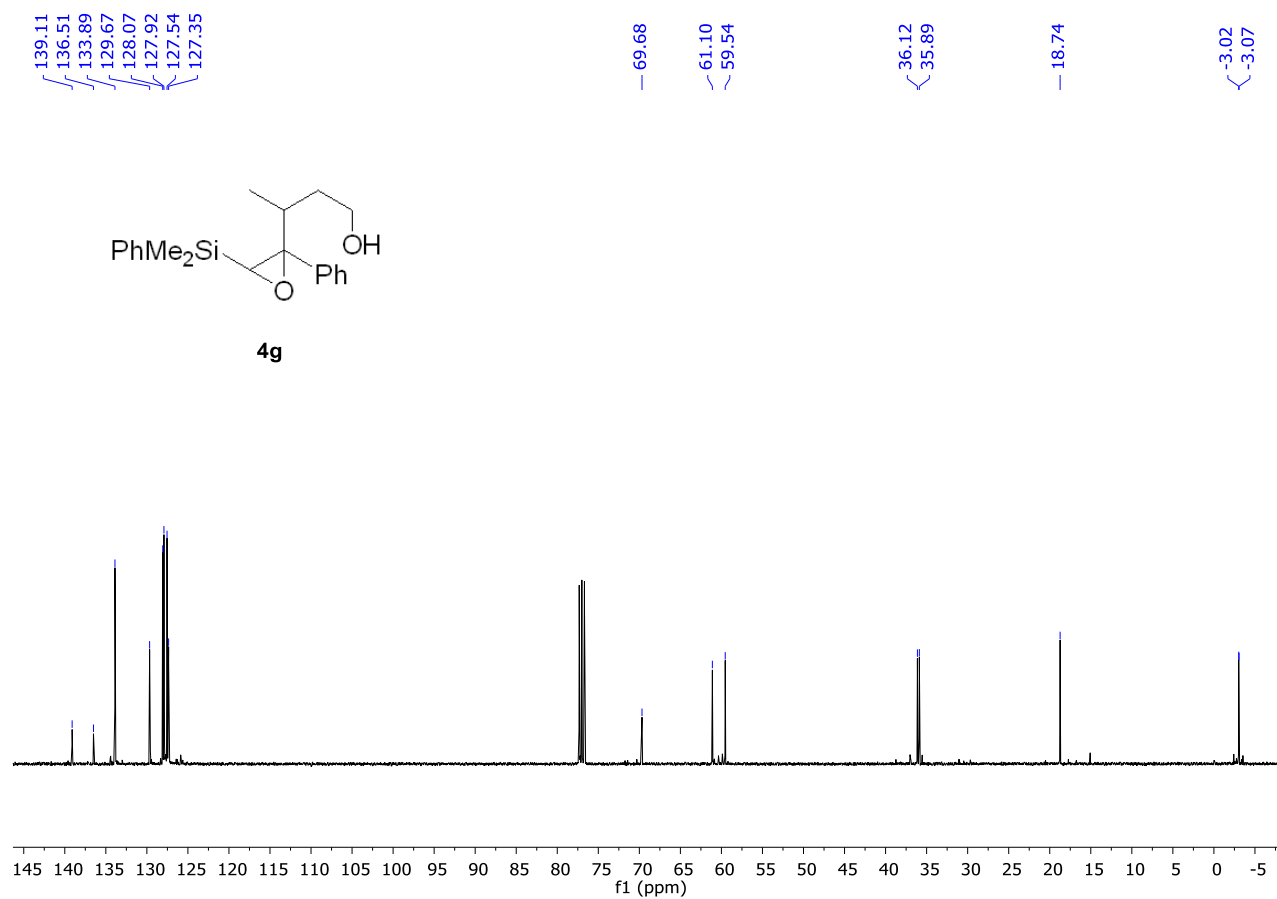
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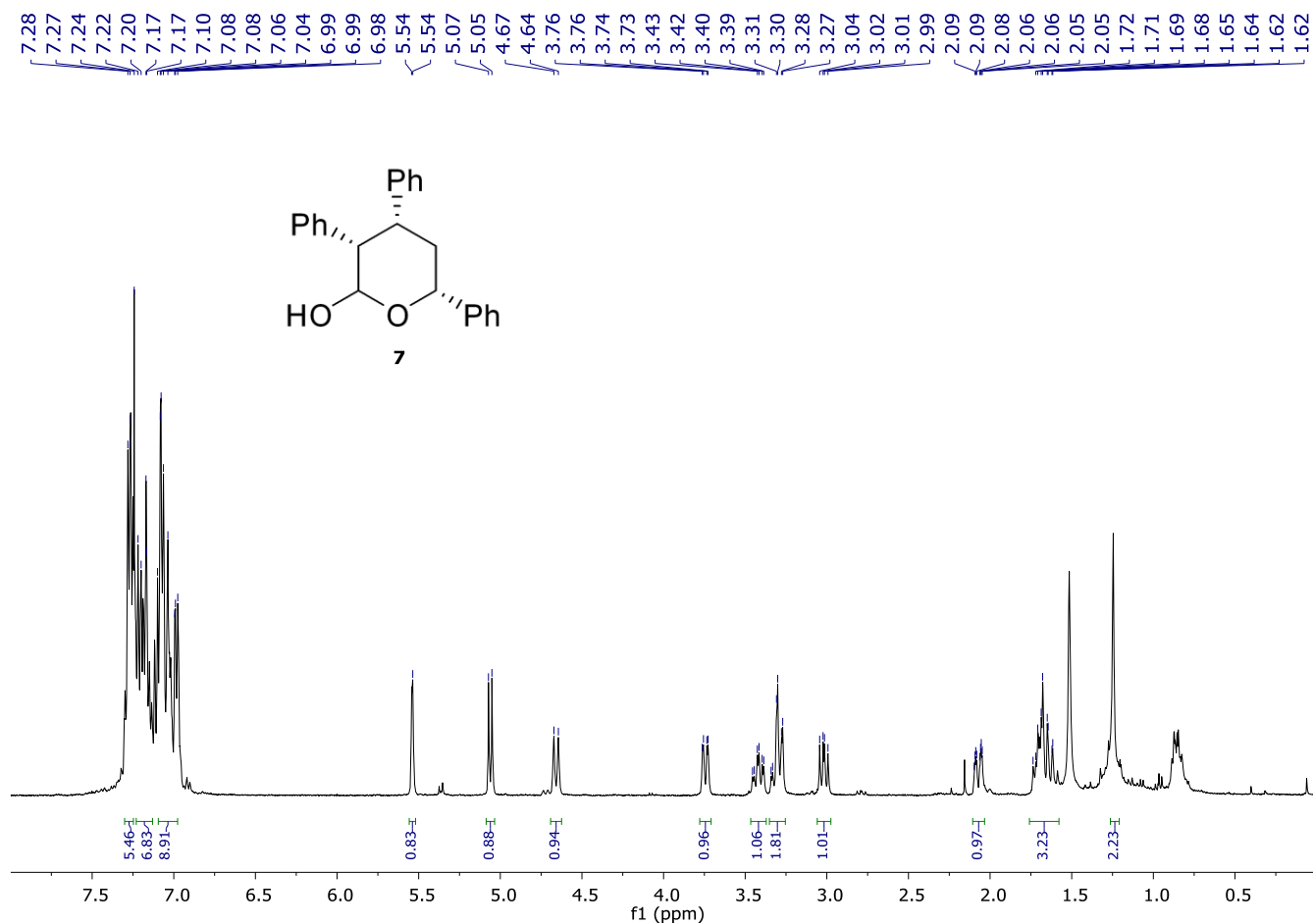
¹H NMR (400 MHz, CDCl₃)



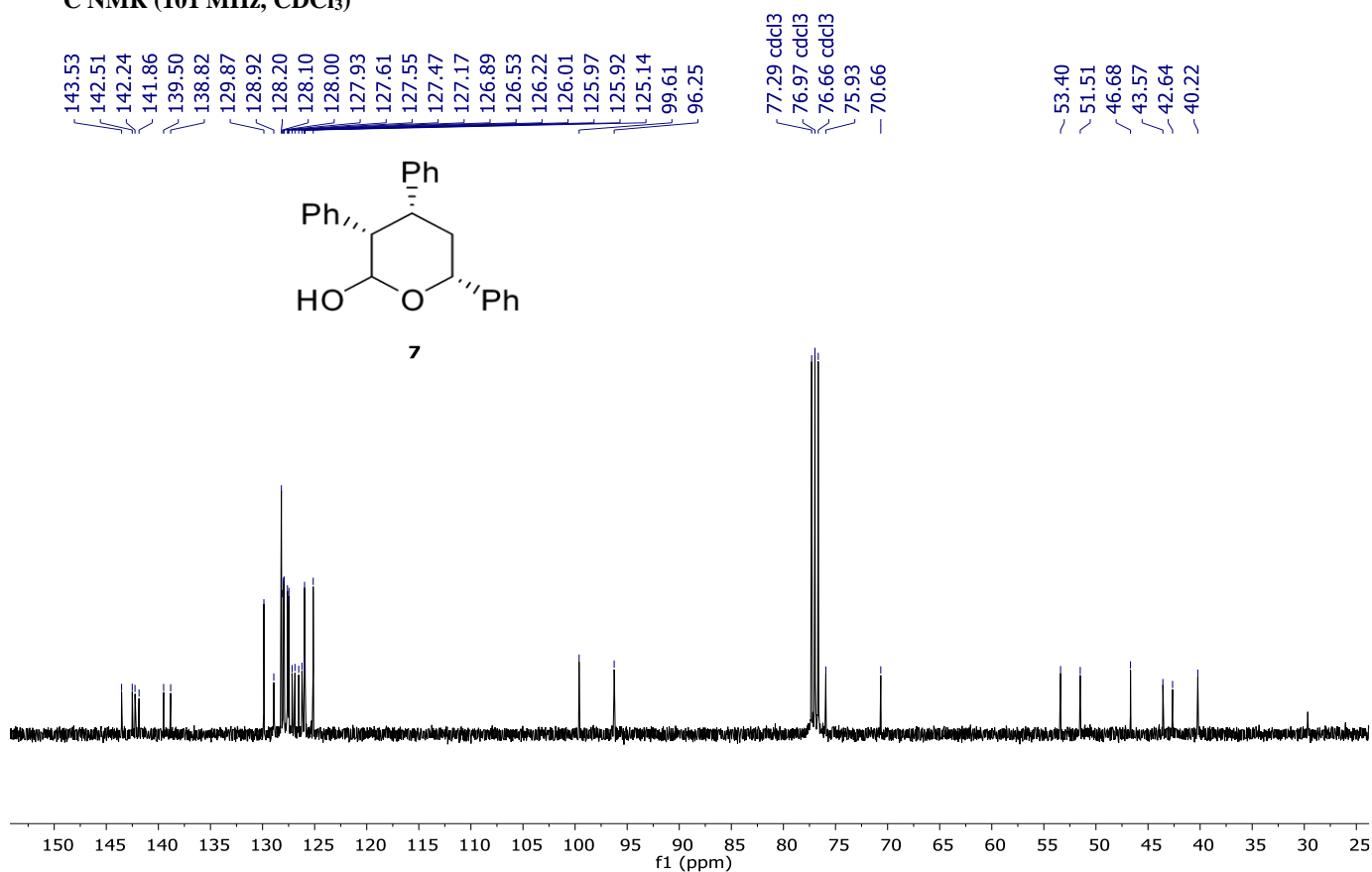
¹³C NMR (101 MHz, CDCl₃)



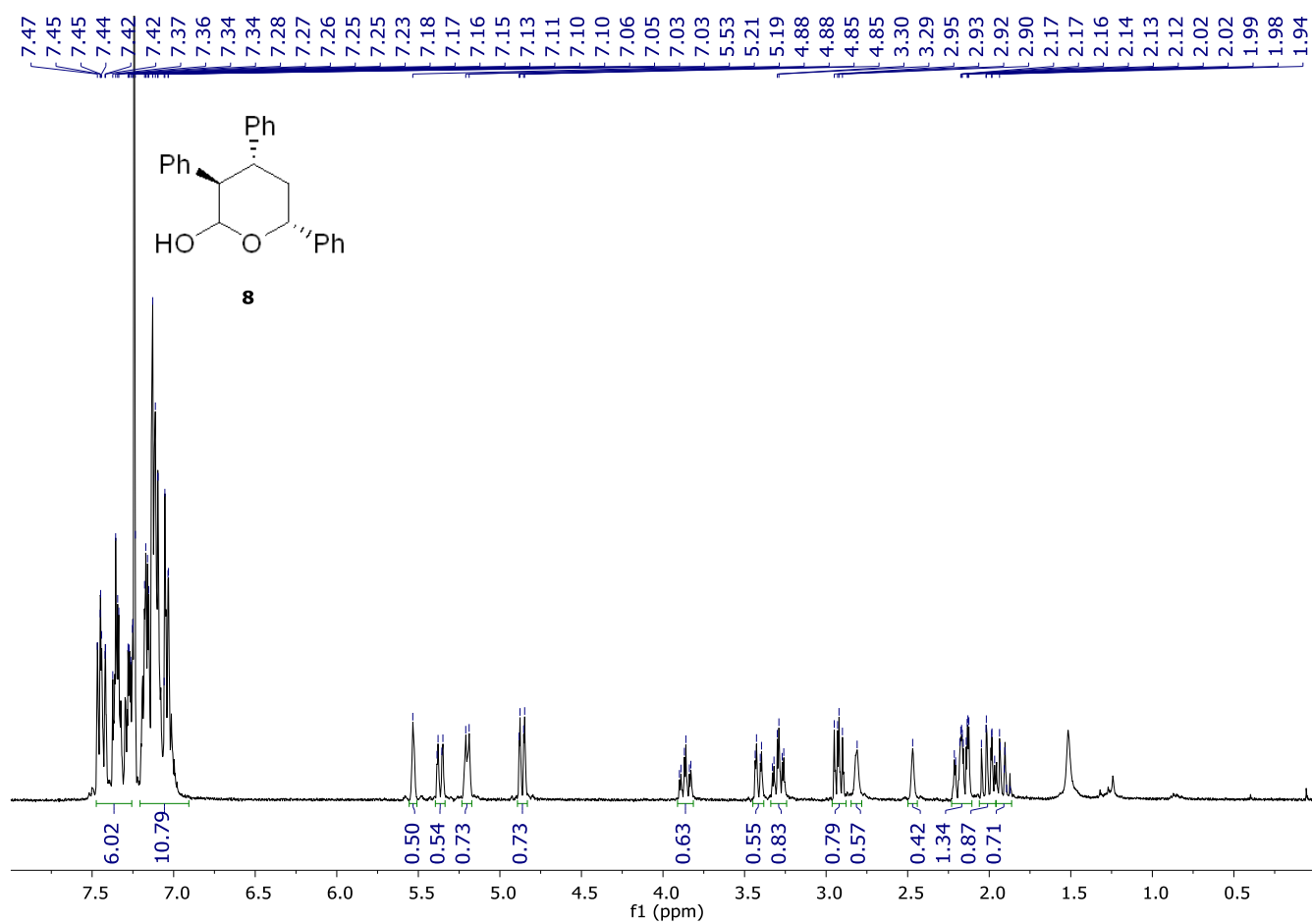
¹H NMR (400 MHz, CDCl₃)



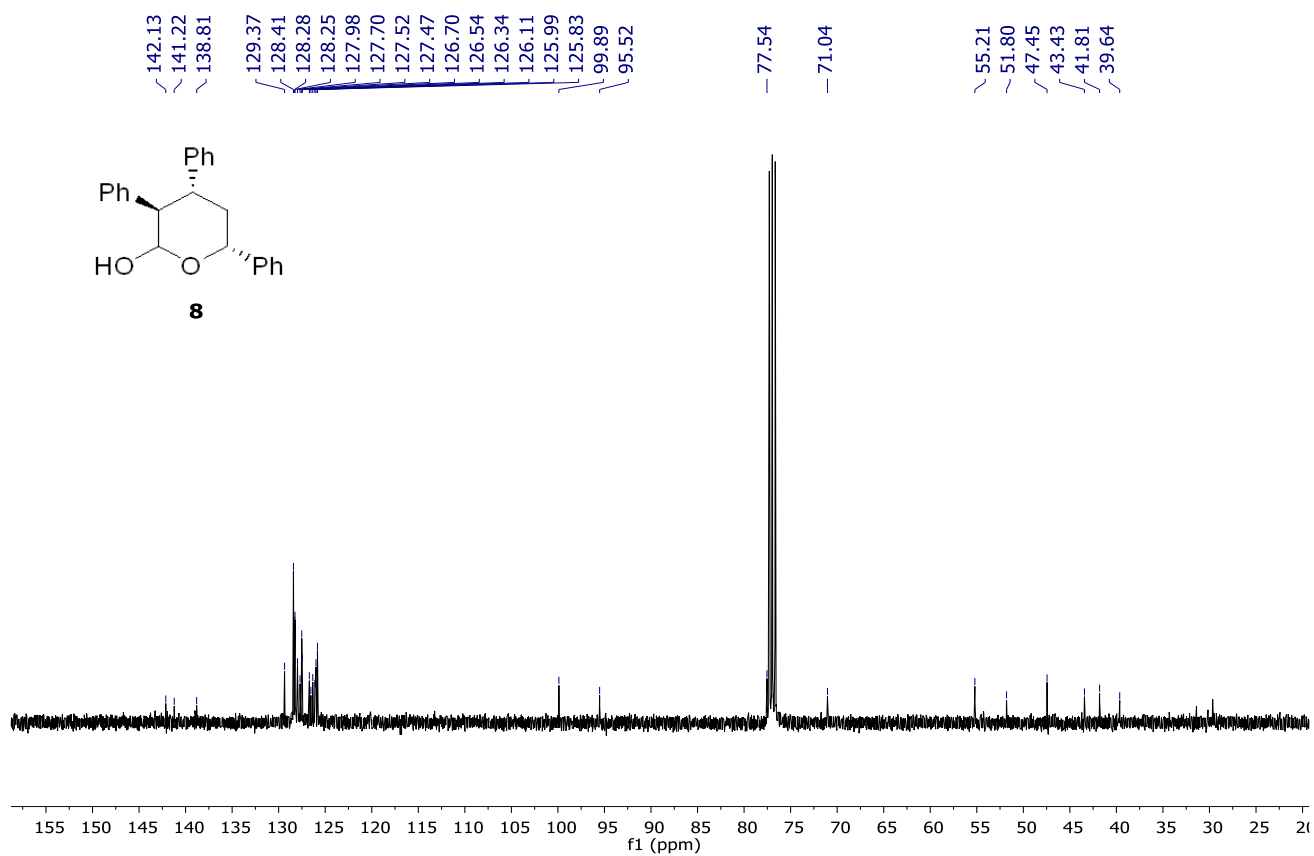
¹³C NMR (101 MHz, CDCl₃)



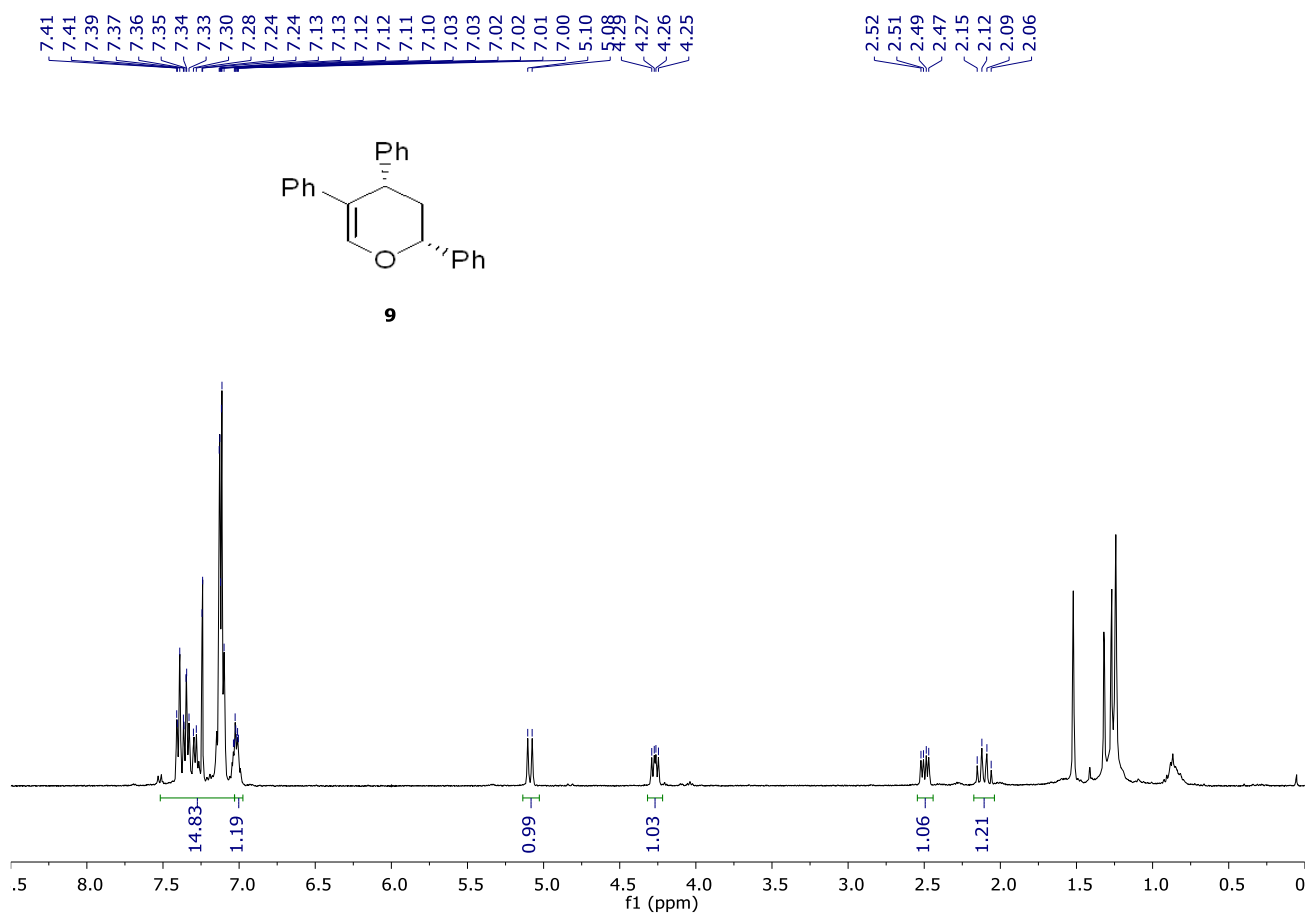
¹H NMR (400 MHz, CDCl₃)



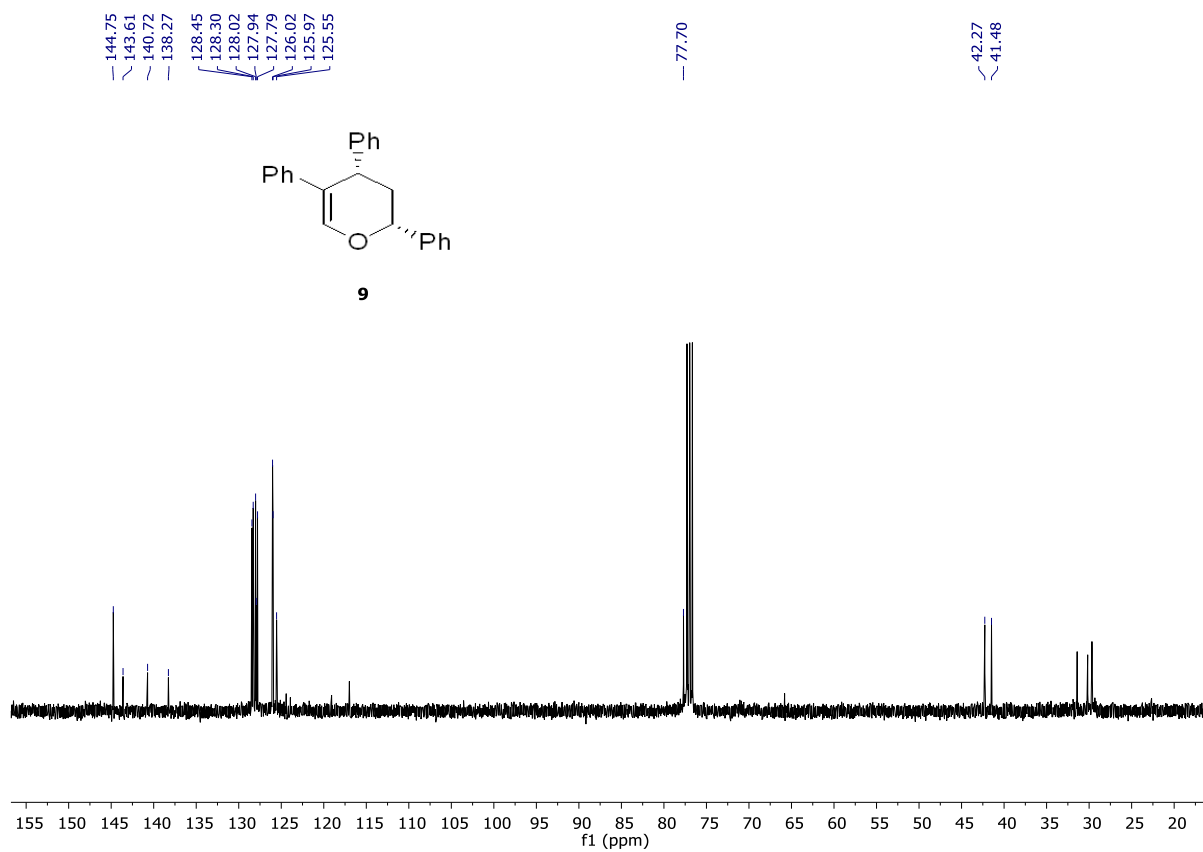
¹³C NMR (101 MHz, CDCl₃)



¹H NMR (400 MHz, CDCl₃)



¹³C NMR (101 MHz, CDCl₃)



Chemical structure of **10**: Cc1ccc(Cc2ccccc2)c3cc(Cc4ccccc4)cc(Cc5ccccc5)c3O

¹H NMR spectrum (CDCl₃) of compound **10**. The x-axis represents the chemical shift in ppm (f1), ranging from 0.00 to 8.00. The spectrum shows several multiplets in the aromatic region (6.5-7.5 ppm) and a large peak for the dimethylphenylsilyl group (around 2.5 ppm). Integration values are shown below the baseline.

Integration values (from left to right): 1.95, 8.95, 8.64, 1.93, 1.00, 1.03, 1.03, 1.05, 6.24.

¹³C NMR (101 MHz, CDCl₃)

Chemical structure of **10** is shown above the spectrum.

Chemical shift values (ppm) are listed above the spectrum:

- 133.74, 130.94, 128.63, 128.54, 128.23, 128.02, 127.48, 127.26, 127.09, 126.33, 126.13, 125.64
- 77.28 cdcl3, 76.97 cdcl3, 76.65 cdcl3
- 46.74, 41.30
- 2.28, -2.72

10

f1 (ppm)