Development of Trityl-based Photolabile Hydroxyl Protecting Groups

Lei Zhou, Haishen Yang, and Pengfei Wang*

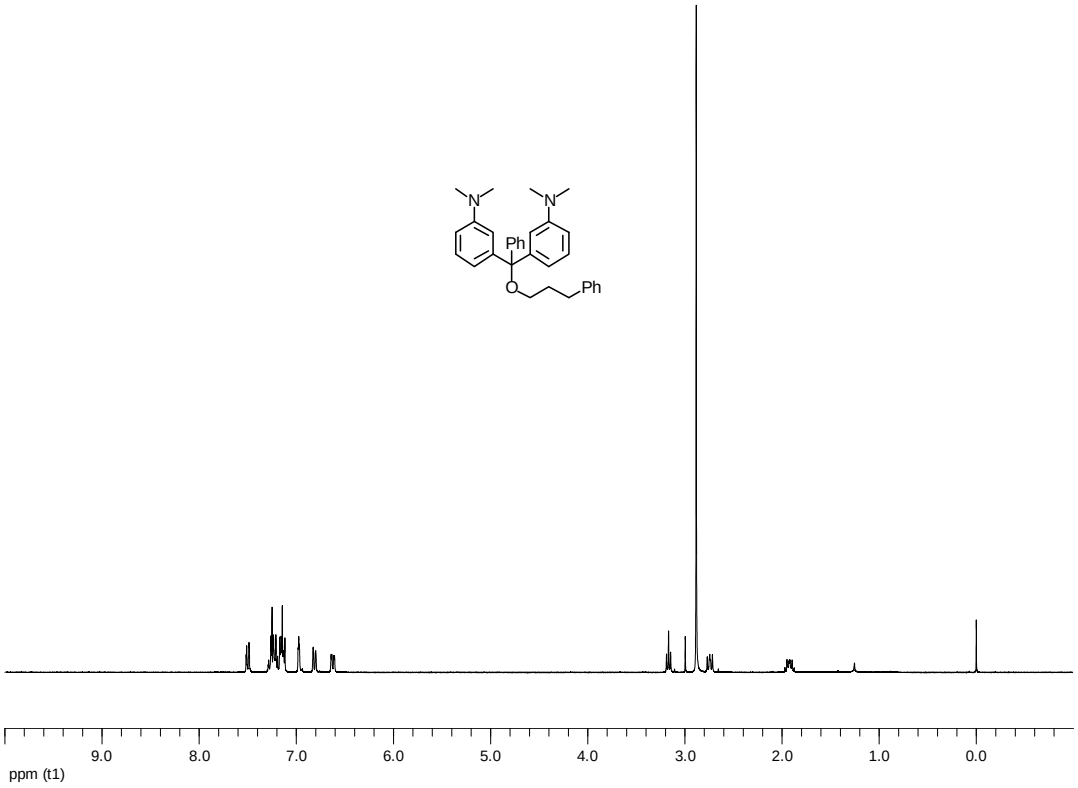
Department of Chemistry, University of Alabama at Birmingham, Birmingham, AL 35294

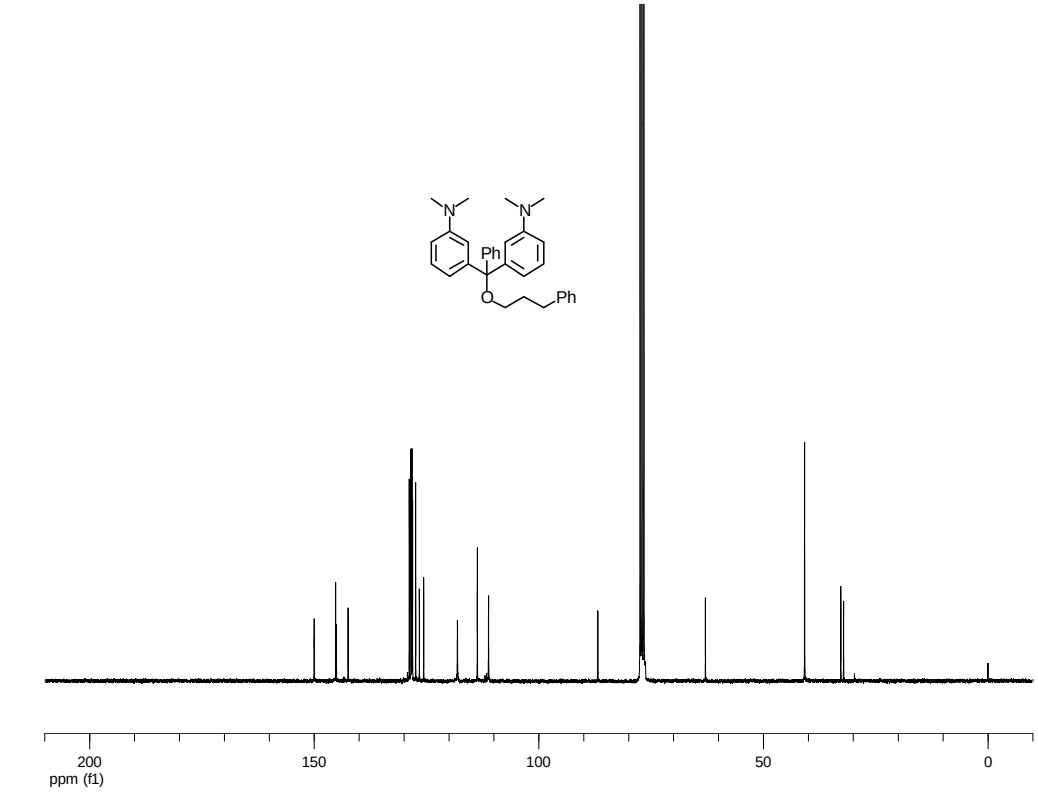
Table of Content

General. Organic solutions were concentrated by rotary evaporation at ca. 12 Torr. Flash column chromatography was performed employing 230-400 mesh silica gel. Thin-layer chromatography was performed using glass plates pre-coated to a depth of 0.25 mm with 230-400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Infrared (IR) data are presented as frequency of absorption (cm^{-1}). Proton and carbon-13 nuclear magnetic resonance (^{1}H NMR or ^{13}C NMR) spectra were recorded on a 300 and a 400 MHz NMR spectrometer; Chemical shifts are expressed in parts per million (δ scale) downfield from tetramethylsilane and are referenced to residual protium in the NMR solvent ($CHCl_3$: δ 7.26). Data are presented as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet and/or multiple resonances), coupling constant in Hertz (Hz), integration. UV-visible spectra were obtained with the sample concentration of 2.0×10^{-5} M.

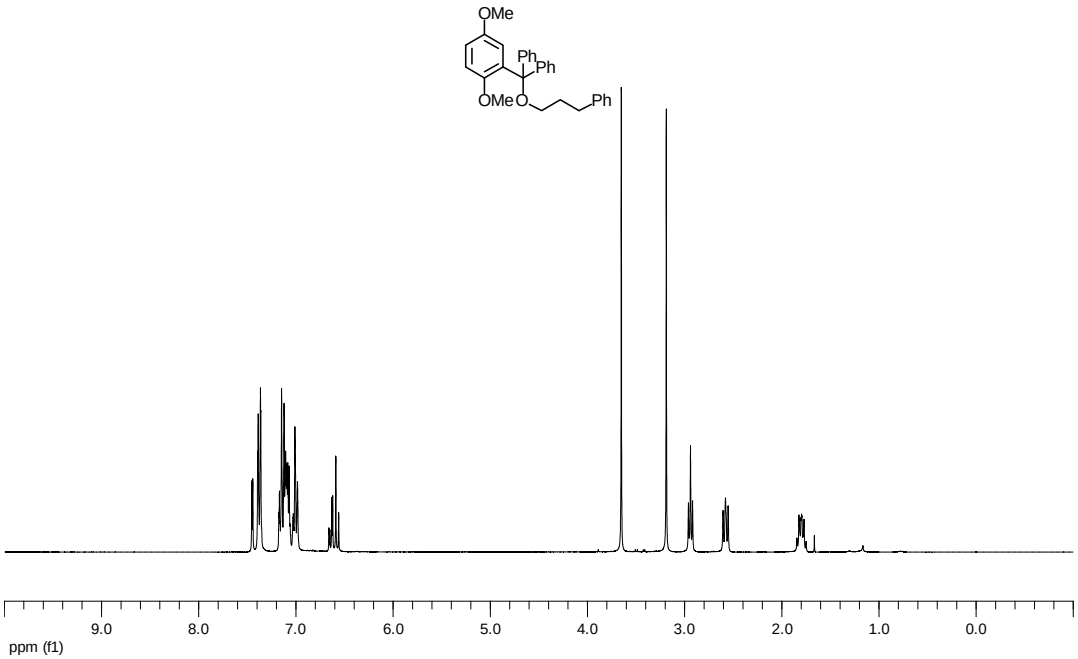
Materials. Tetrahydrofuran and toluene were distilled from appropriate drying reagents under a nitrogen atmosphere at 760 torr. Other chemicals were obtained from commercial vendors and used without further purification.

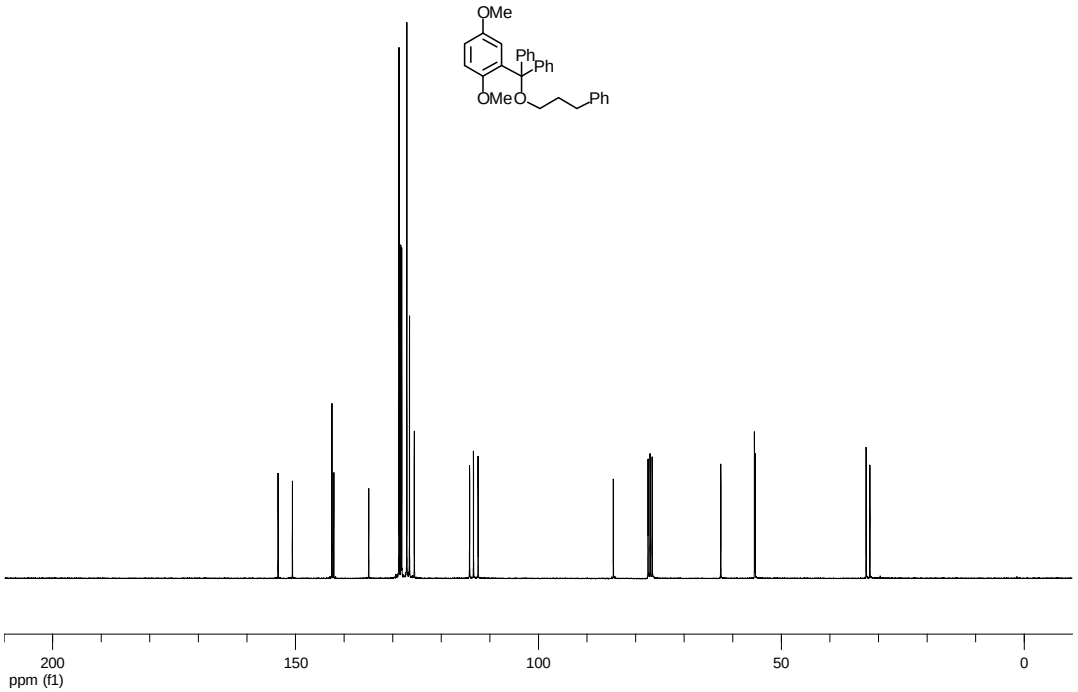
4


N
N
Ph
O
Ph
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

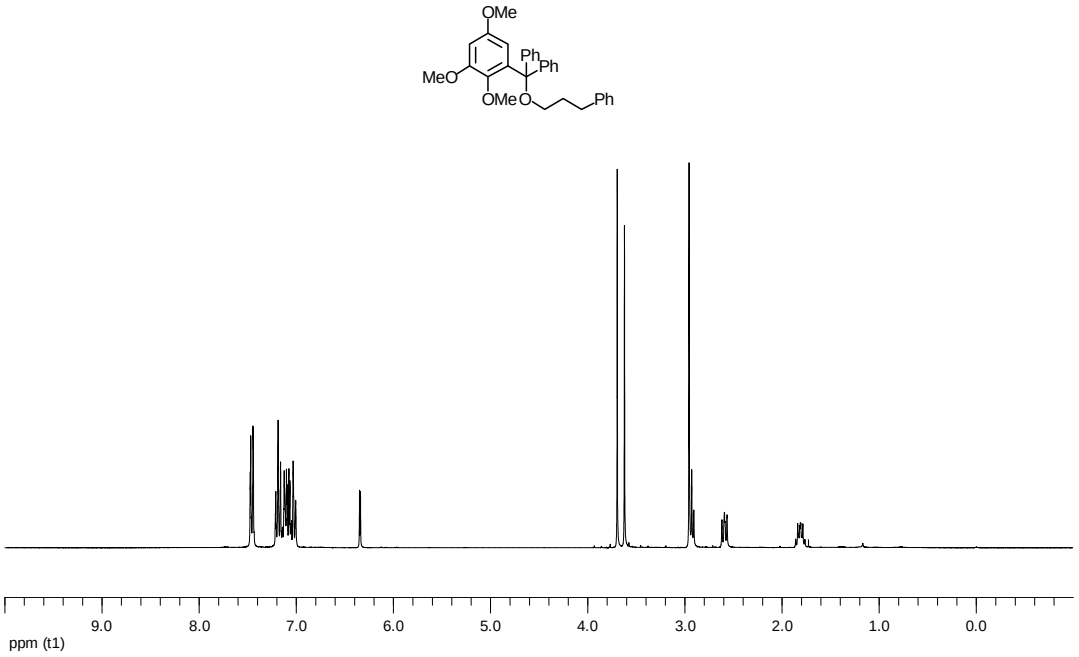

N
N
Ph
O
Ph
200
150
100
50
0
ppm (f1)

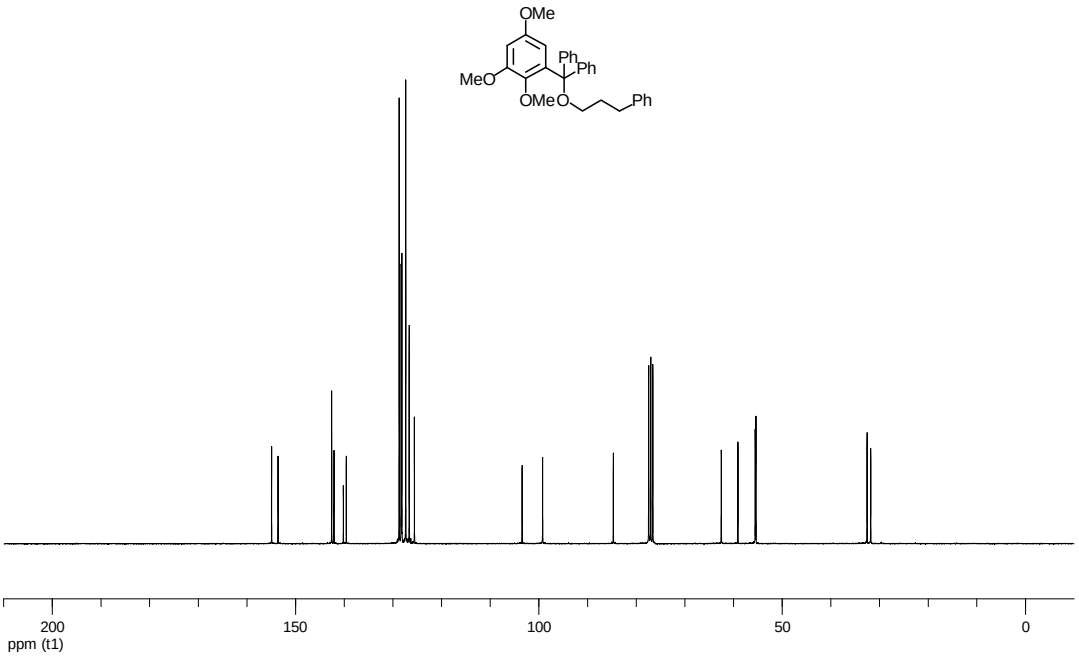
6


OMe
Ph
Ph
OMe
O
Ph
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)

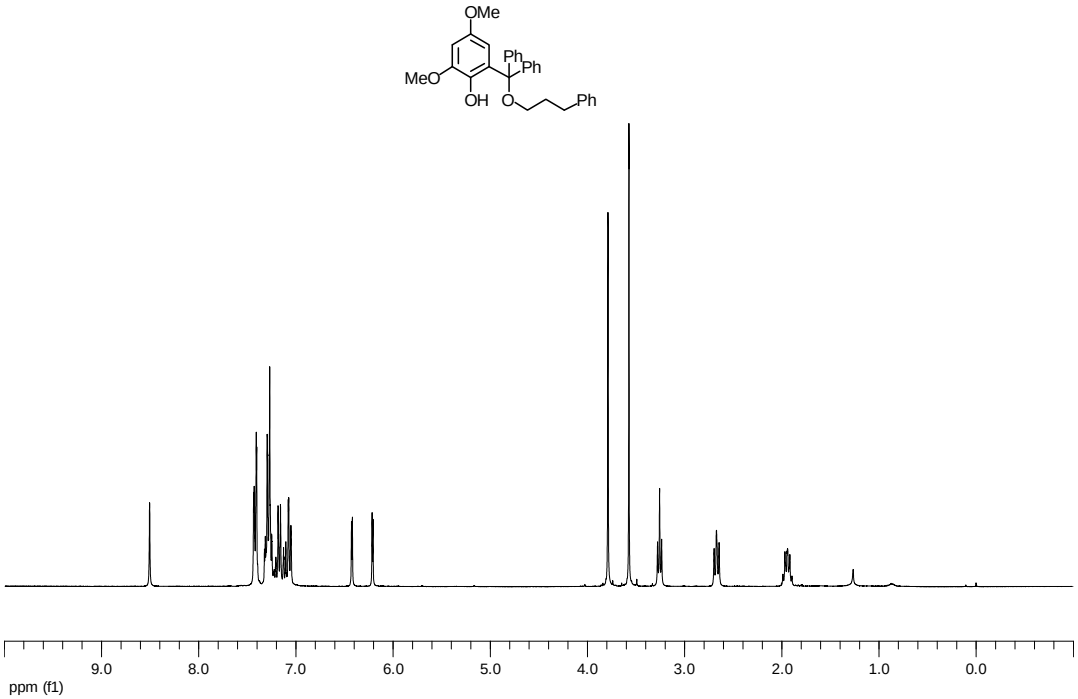

OMe
Ph
Ph
OMe
O
Ph
200
150
100
50
0
ppm (f1)

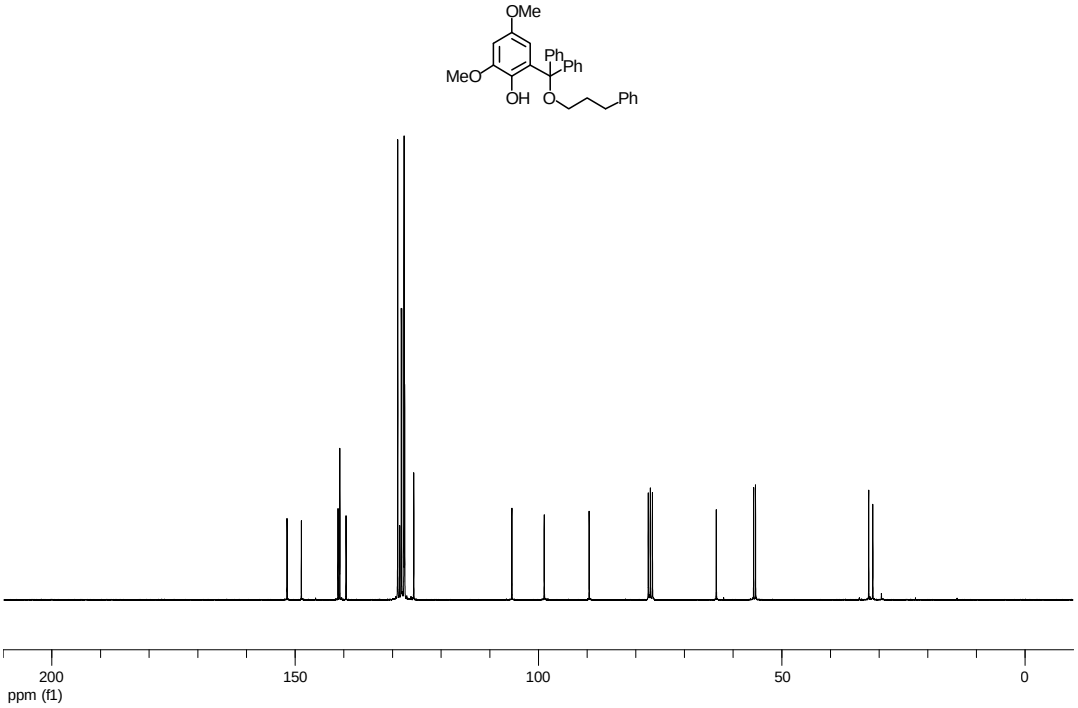
7


OMe
Ph
Ph
MeO
OMe
O
Ph
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

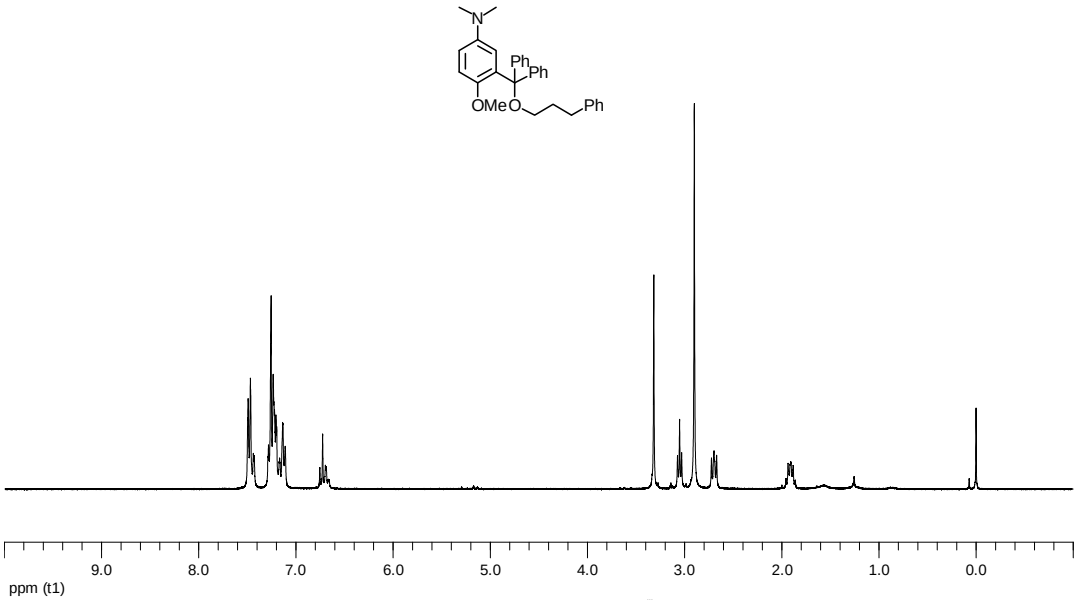

OMe
Ph
Ph
MeO
OMe
O
Ph
200
150
100
50
0
ppm (t1)

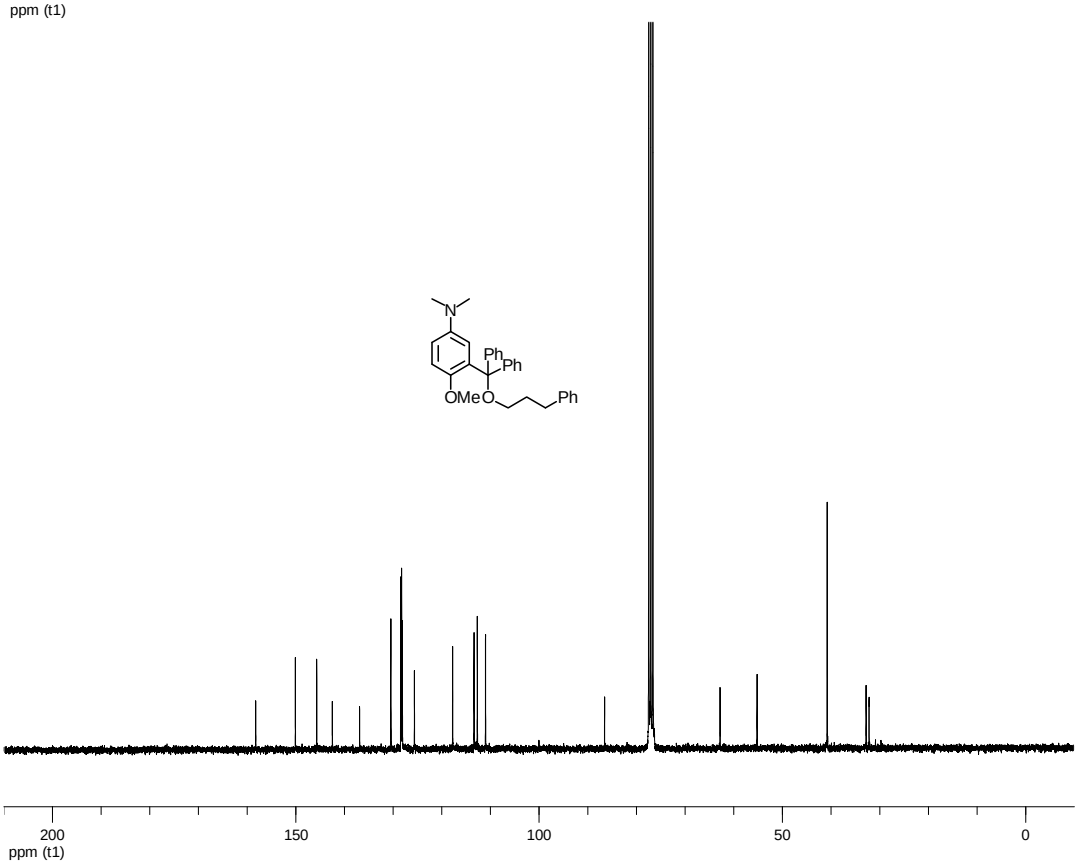
8


OMe
MeO
OH
Ph
Ph
O
Ph
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)

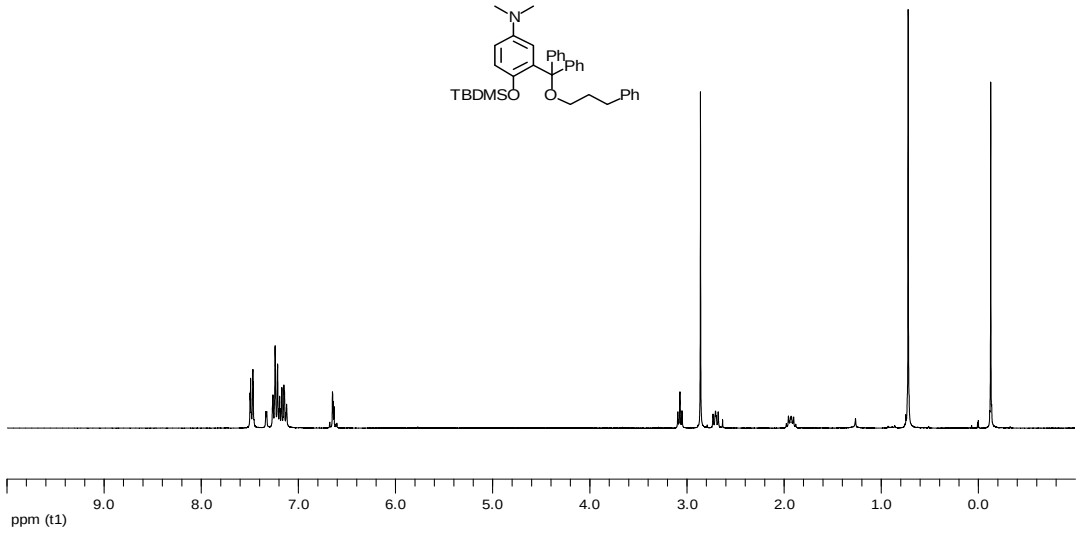

OMe
MeO
OH
Ph
Ph
O
Ph
200
150
100
50
0
ppm (f1)

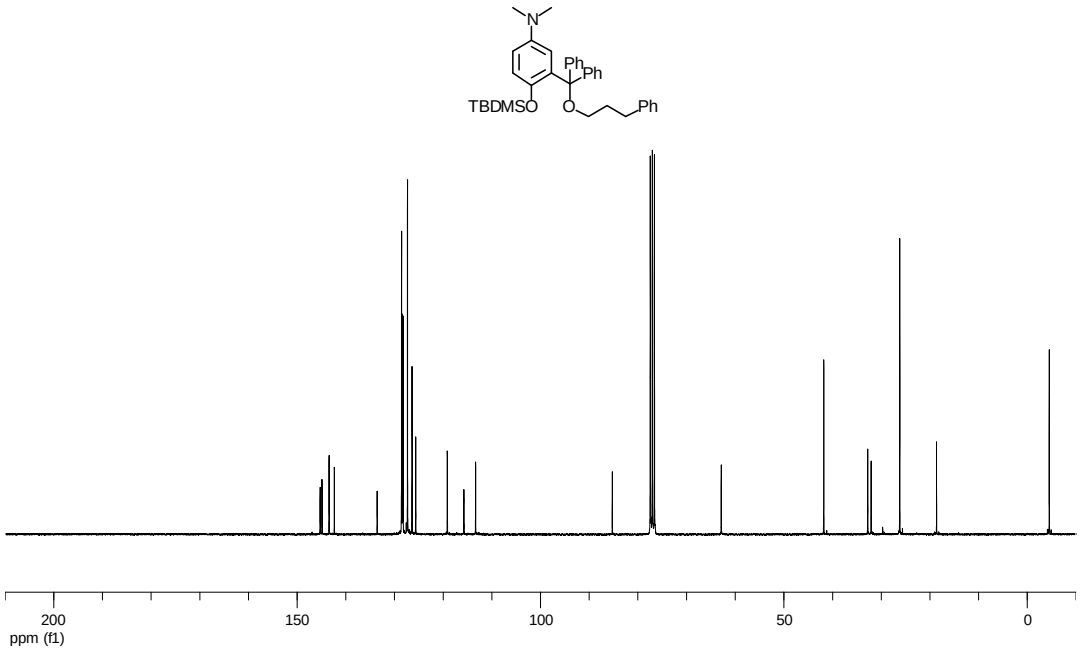
9


N
Ph
Ph
OMe
O
Ph
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

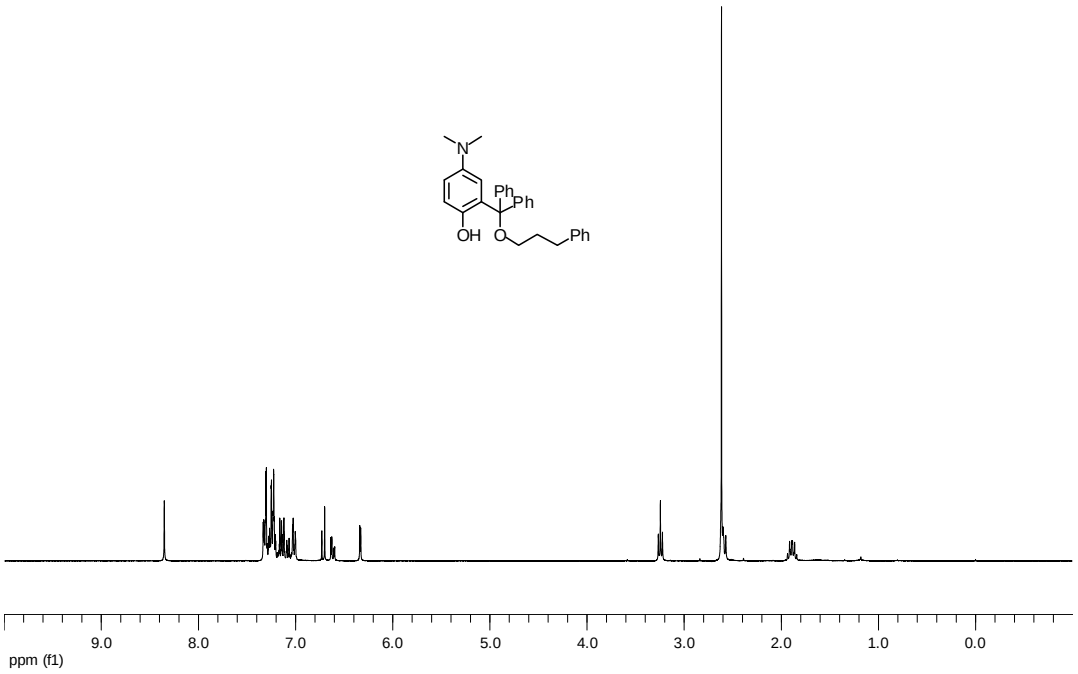

N
Ph
Ph
OMe
O
Ph
200
150
100
50
0
ppm (t1)

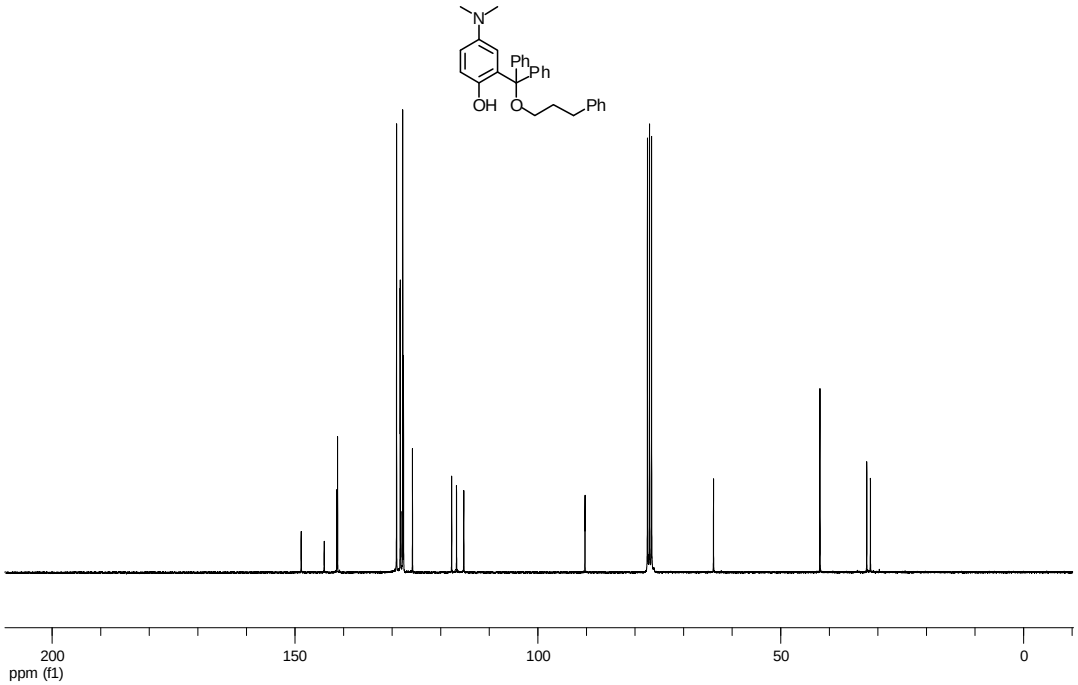
10


N
Ph
Ph
TBDMSO
O
Ph
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

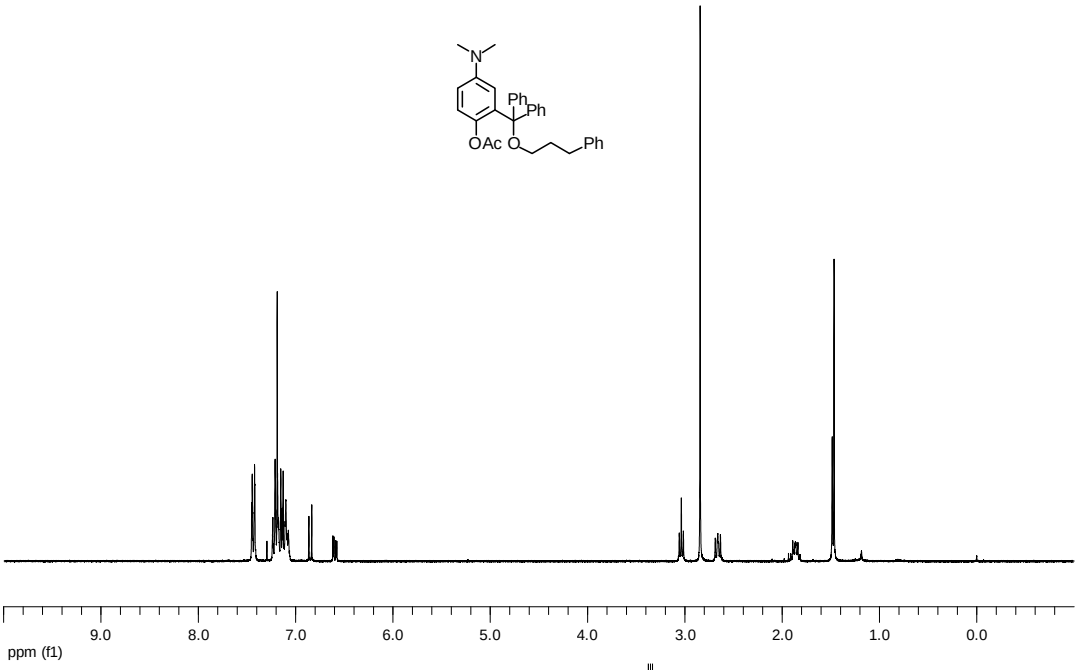

N
Ph
Ph
TBDMSO
O
Ph
200
150
100
50
0
ppm (f1)

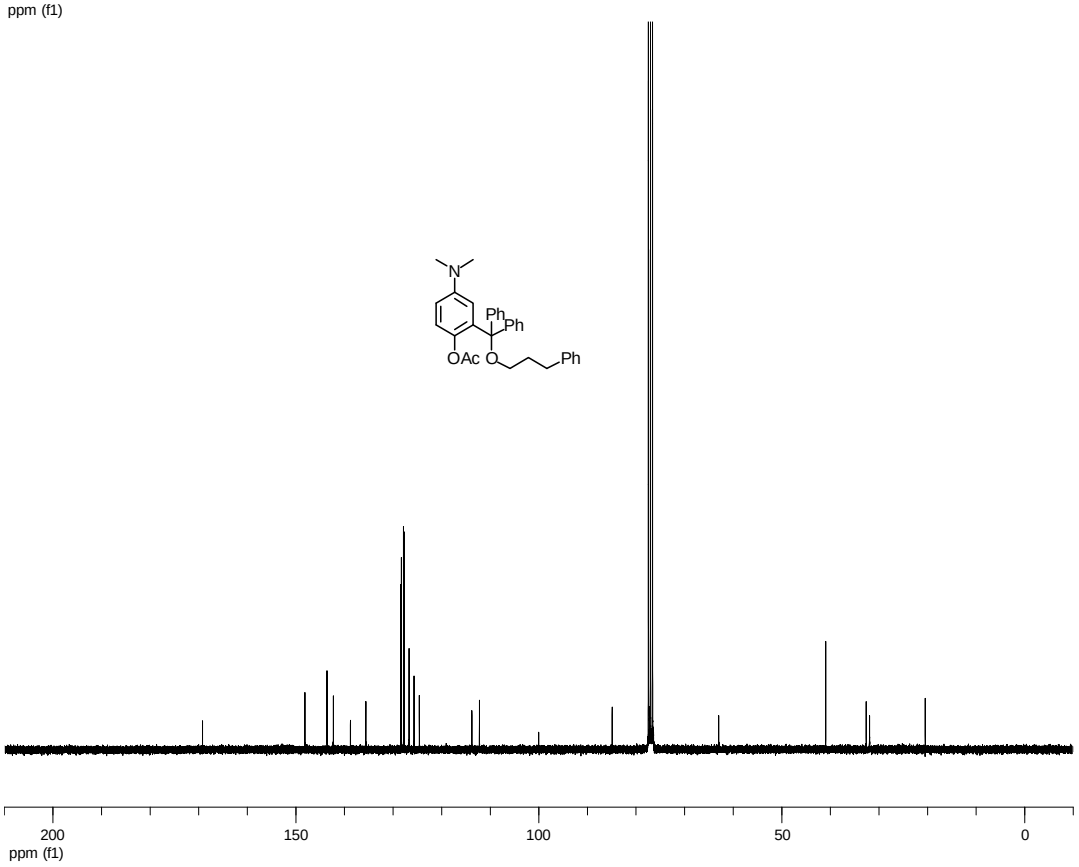
11


N
Ph
Ph
Ph
OH
O
Ph
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)

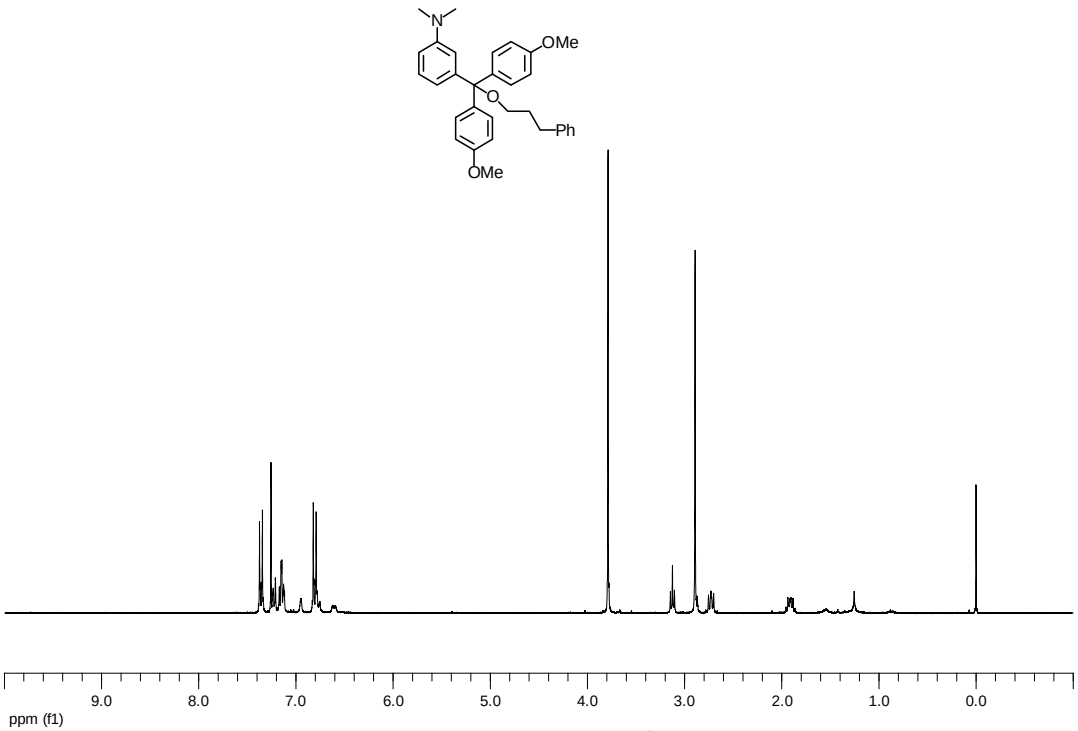

N
Ph
Ph
Ph
OH
O
Ph
200
150
100
50
0
ppm (f1)

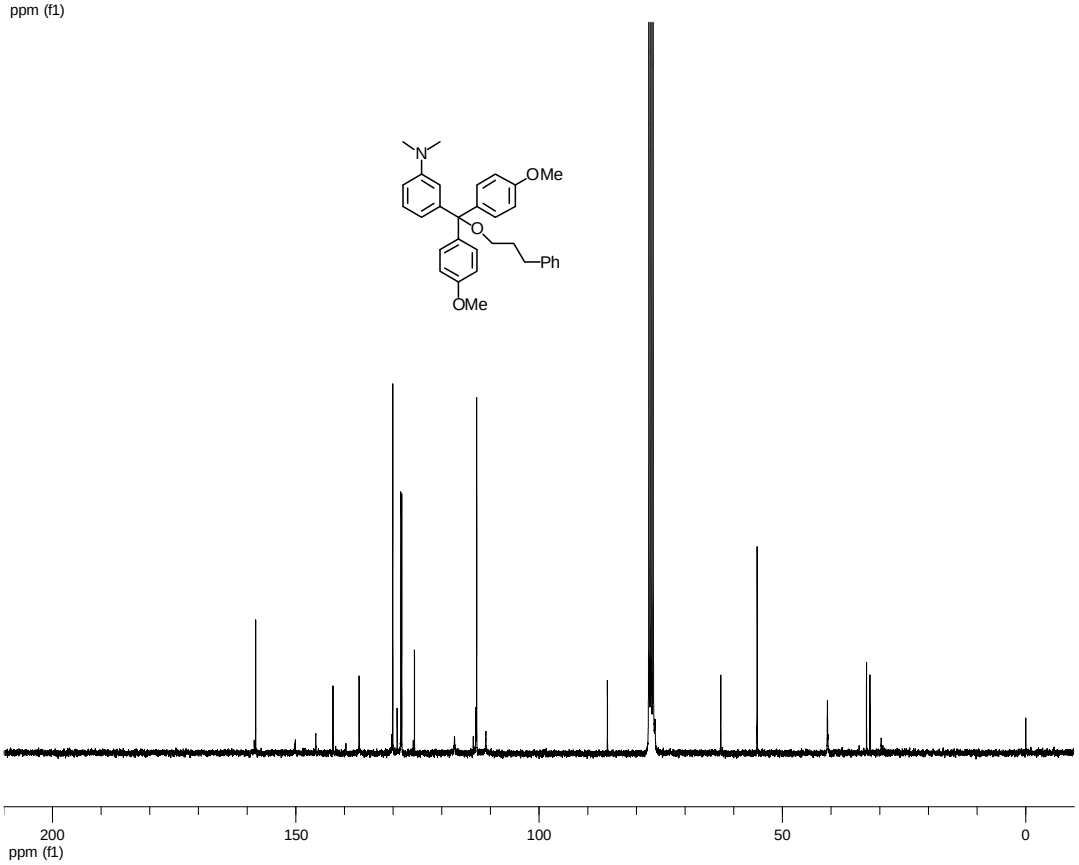
12


N
Ph
Ph
OAc
O
Ph
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)

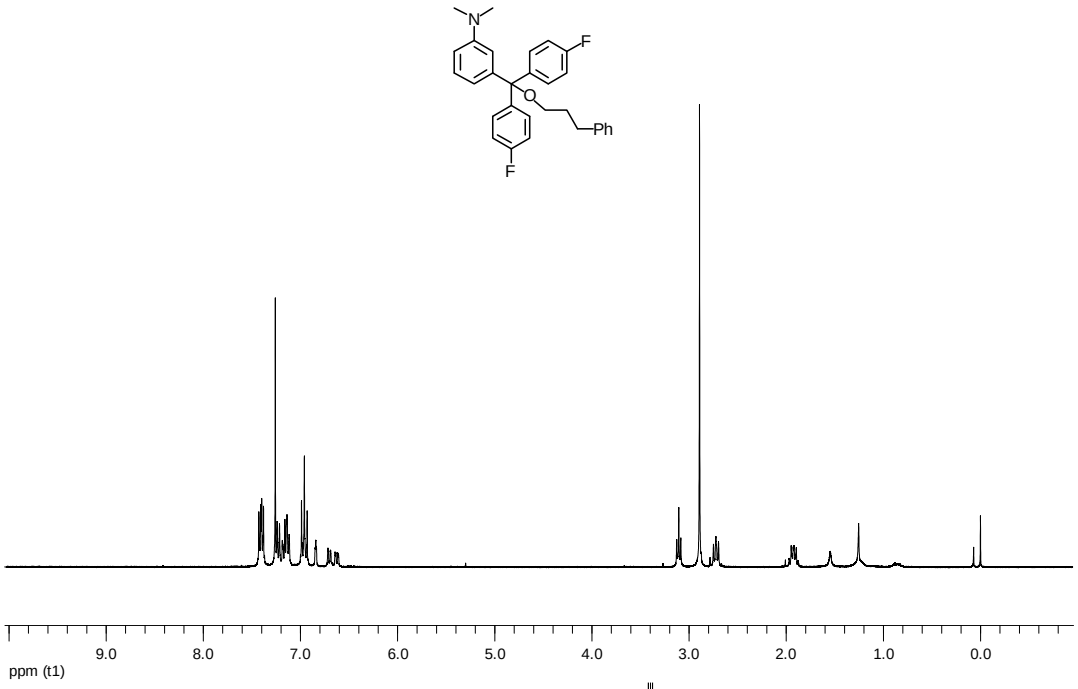

N
Ph
Ph
OAc
O
Ph
200
150
100
50
0
ppm (f1)

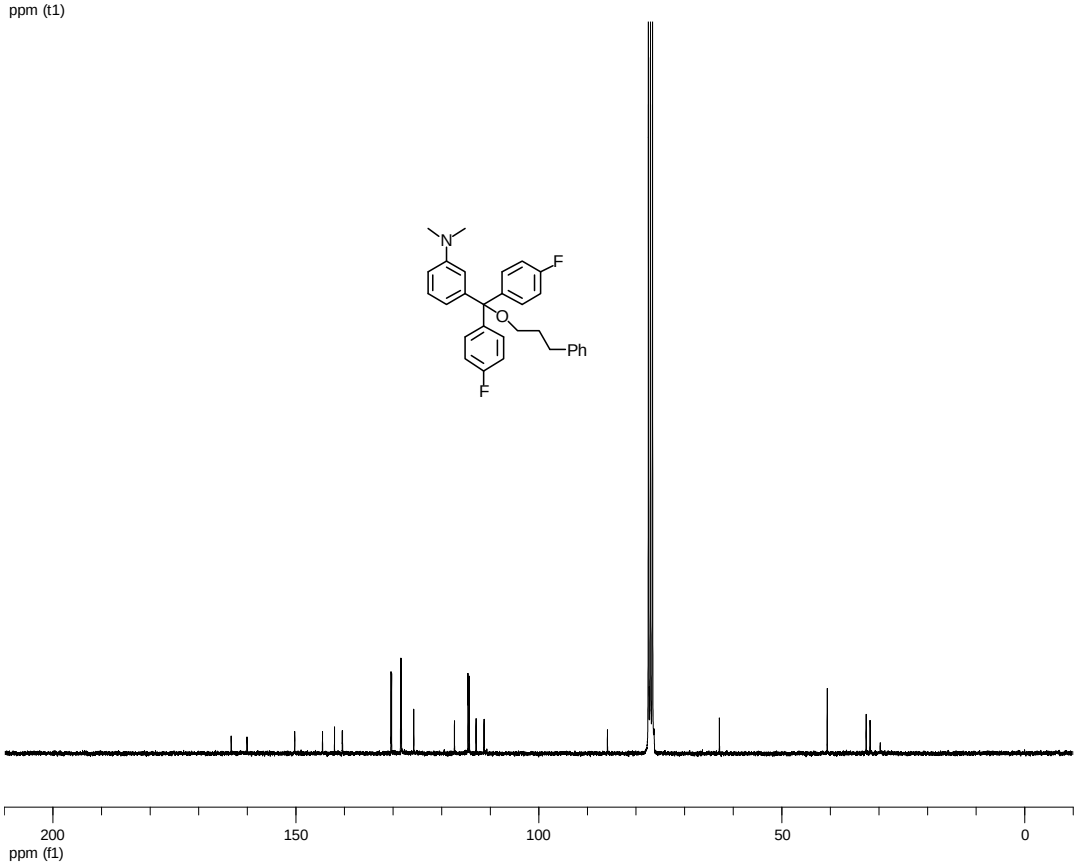
13


N
OMe
O
Ph
OMe
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)

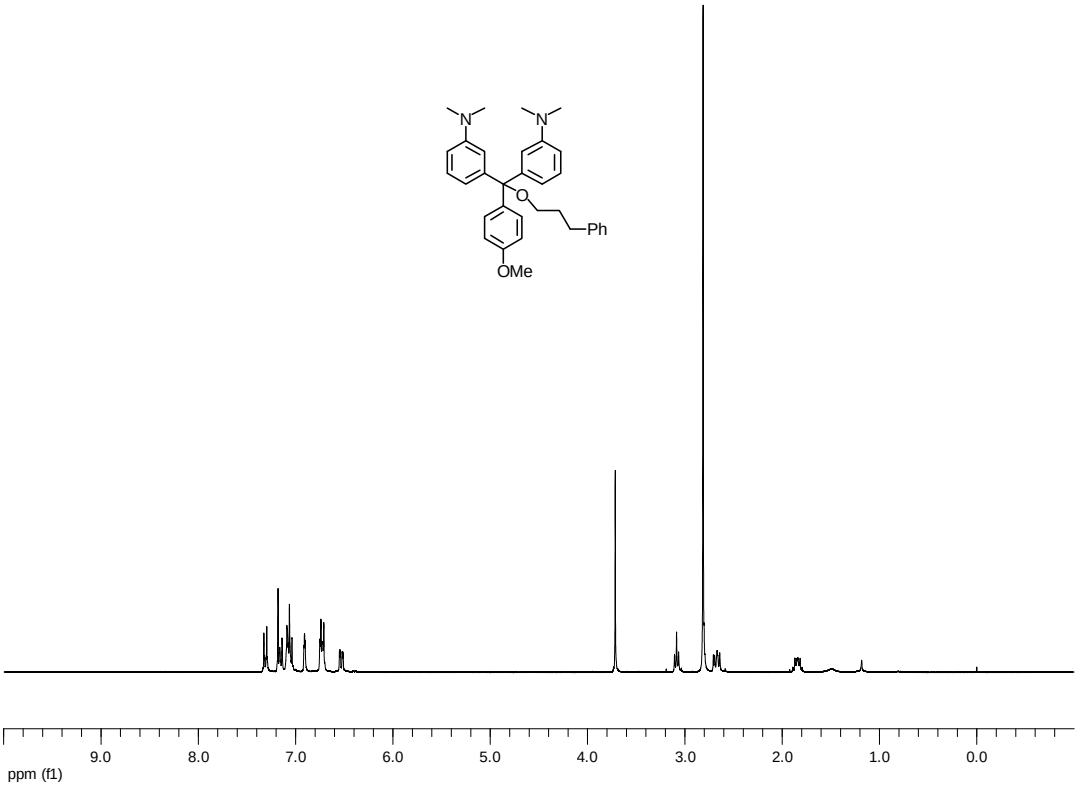

N
OMe
O
Ph
OMe
200
150
100
50
0
ppm (f1)

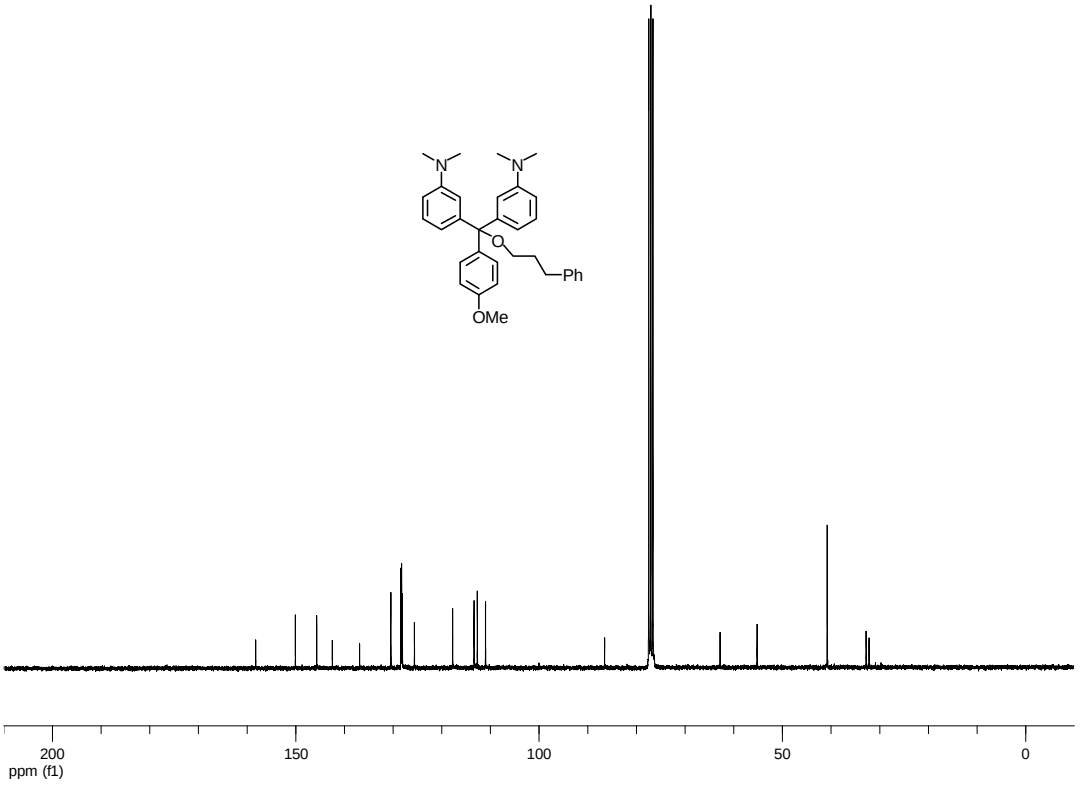
14

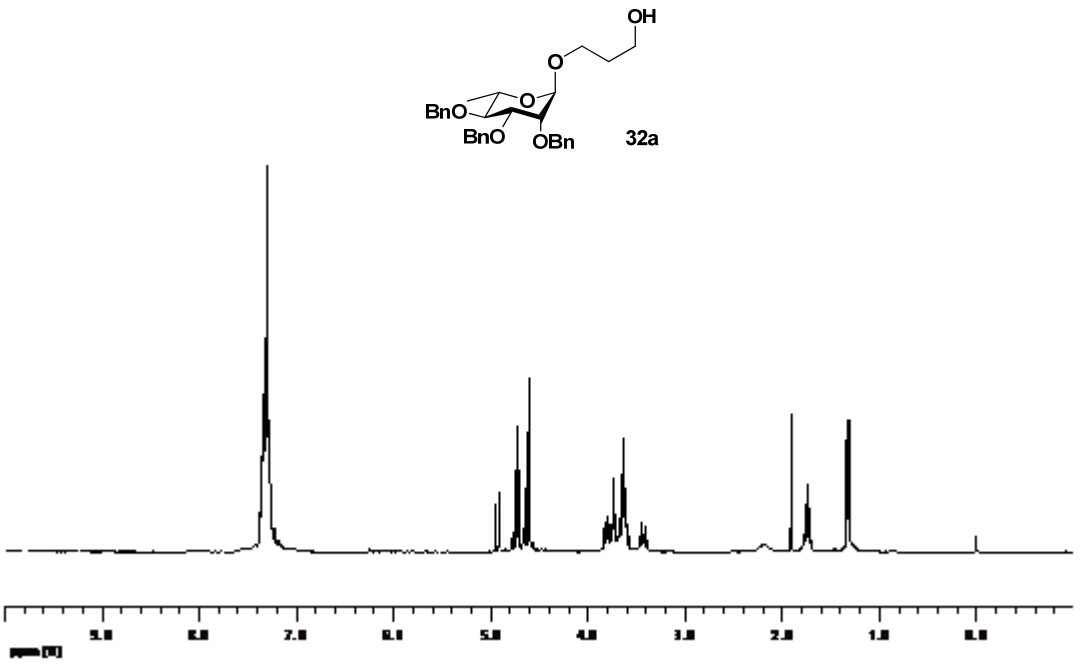

N
F
O
Ph
F
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

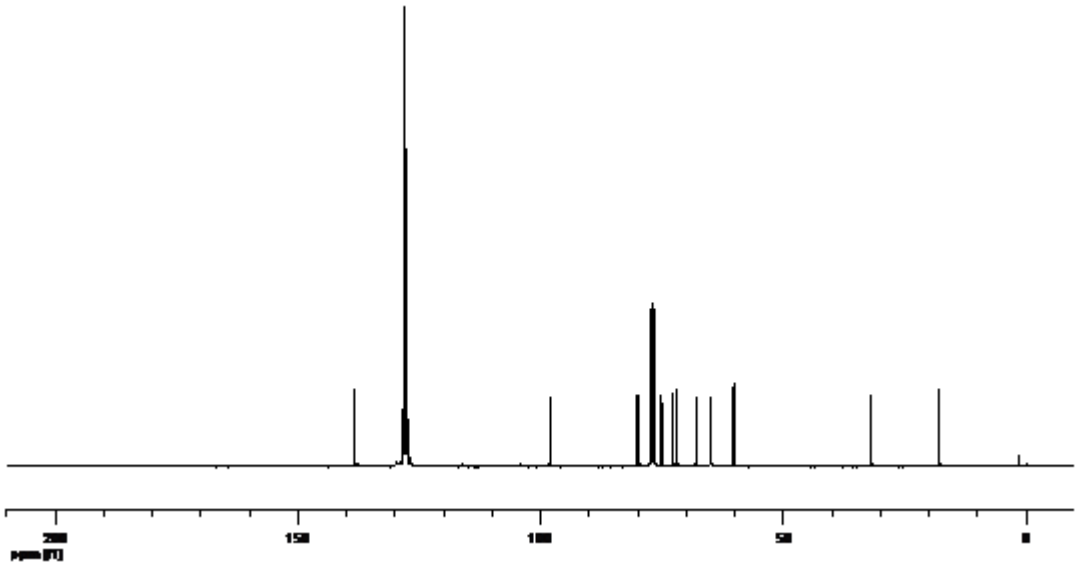

N
F
O
Ph
F
200
150
100
50
0
ppm (f1)

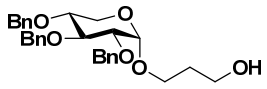
15


N
N
O
Ph
OMe
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)

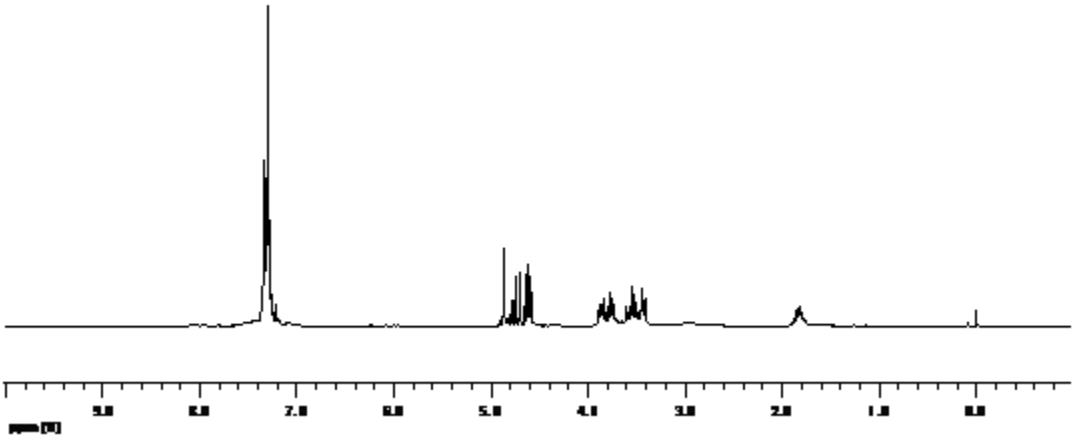

N
N
O
Ph
OMe
200
150
100
50
0
ppm (f1)

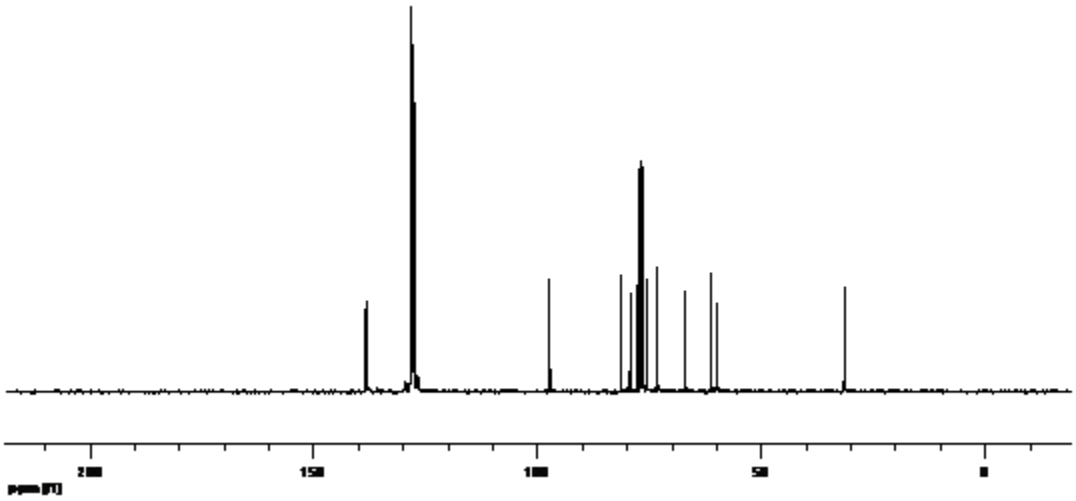

OH
O
BnO
O
BnO
OBn
32a

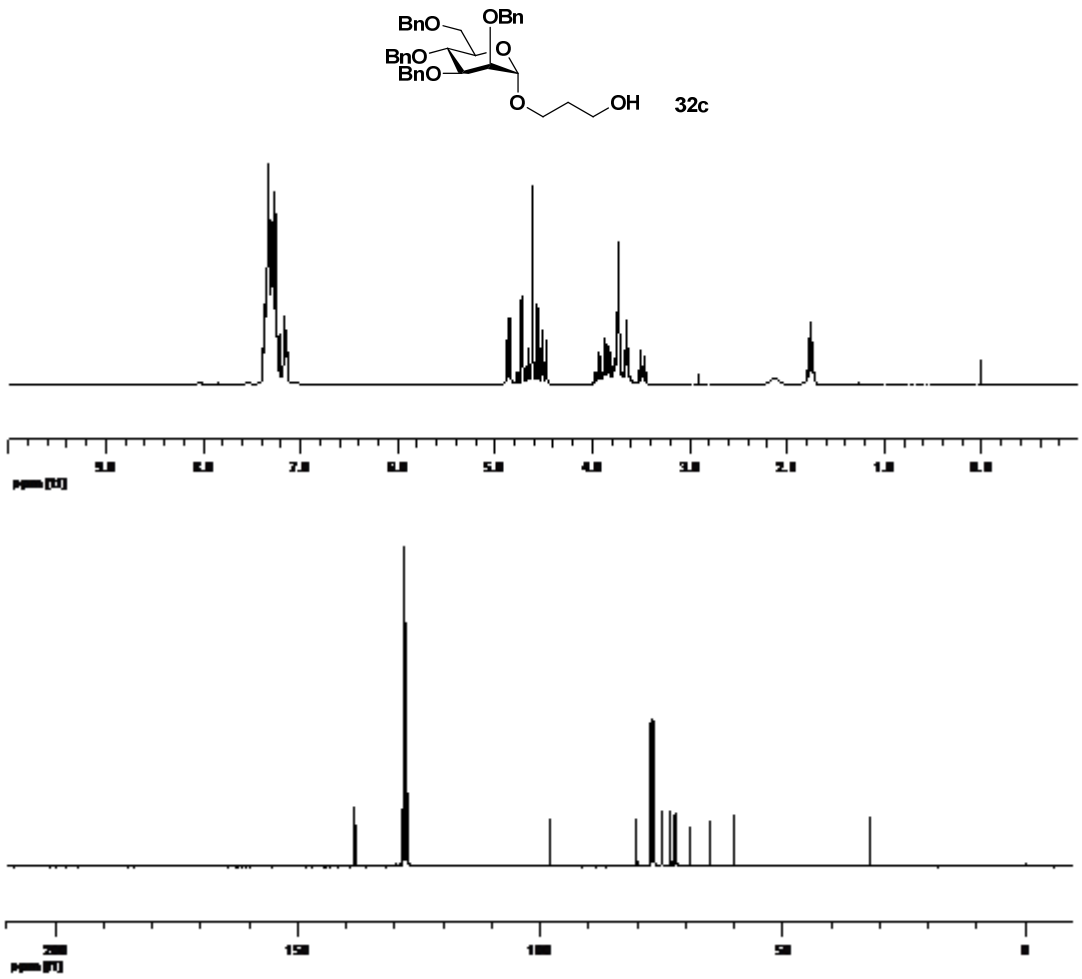


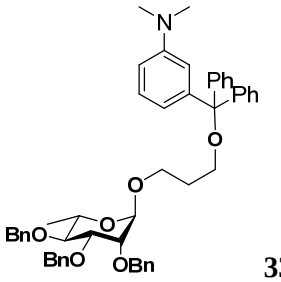


32b

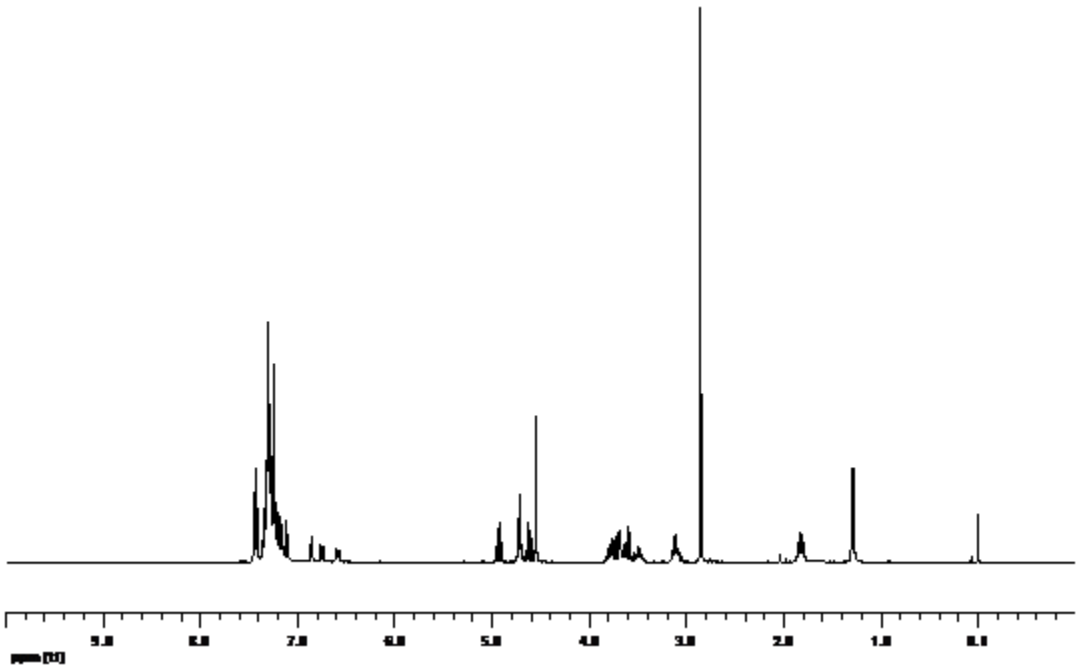


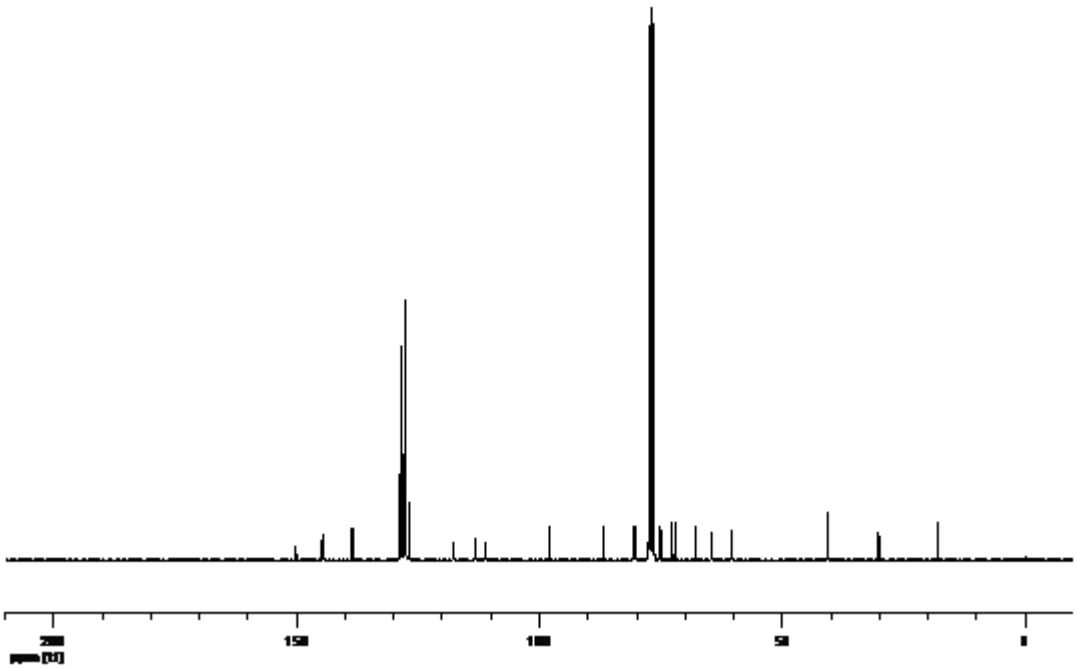


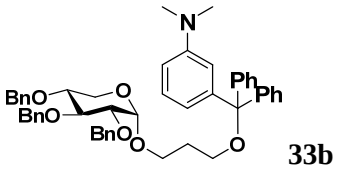

BnO
OBn
BnO
O
BnO
O
OH
32c



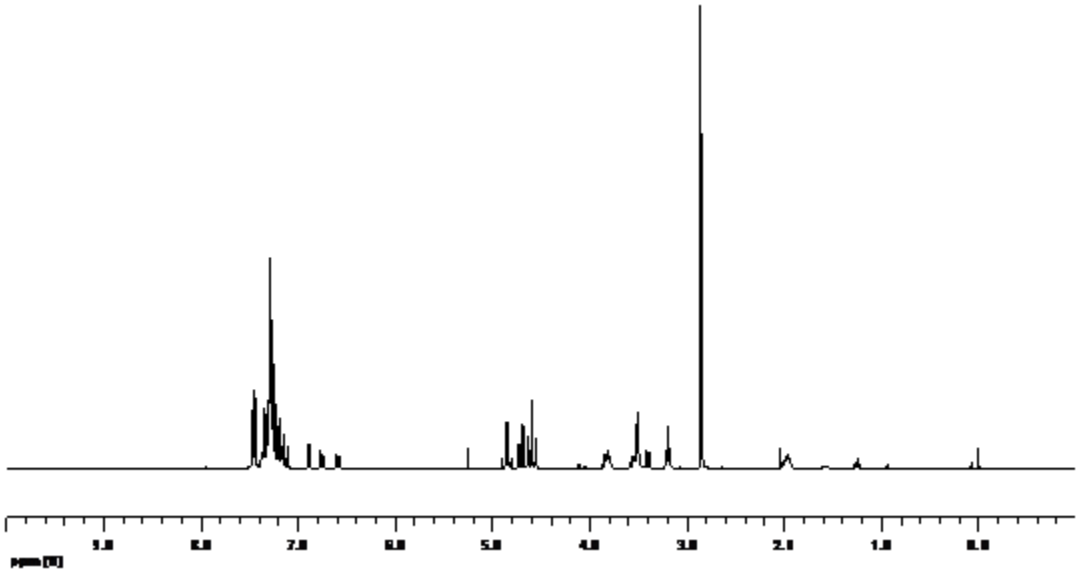
33a

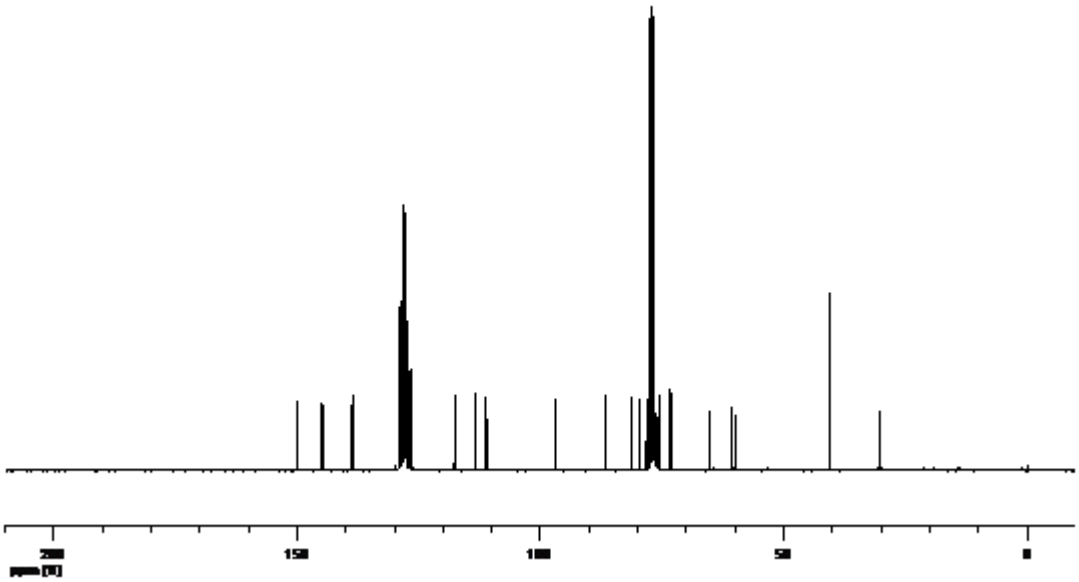


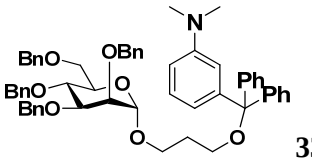




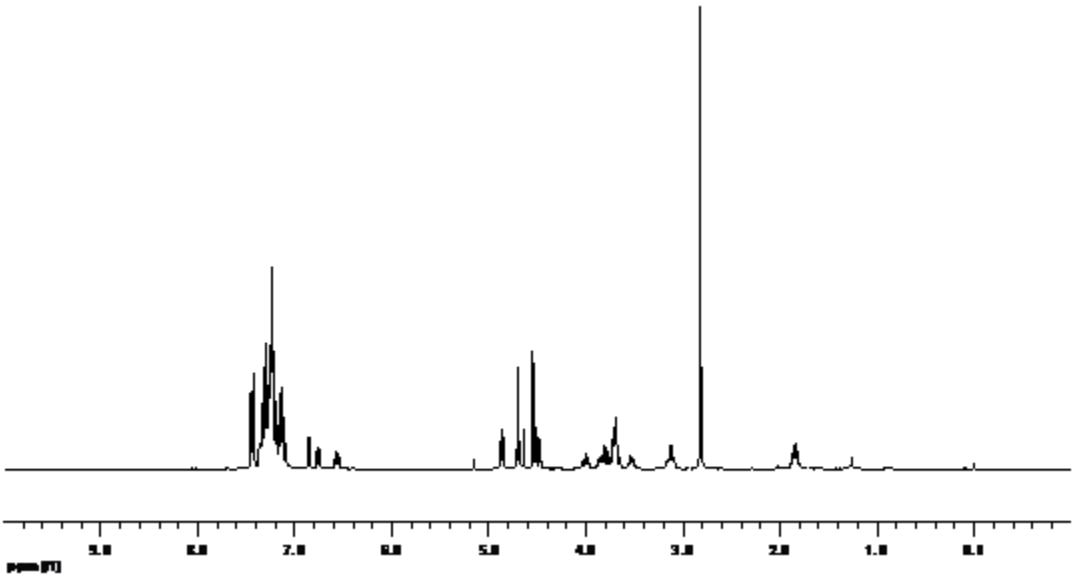
33b

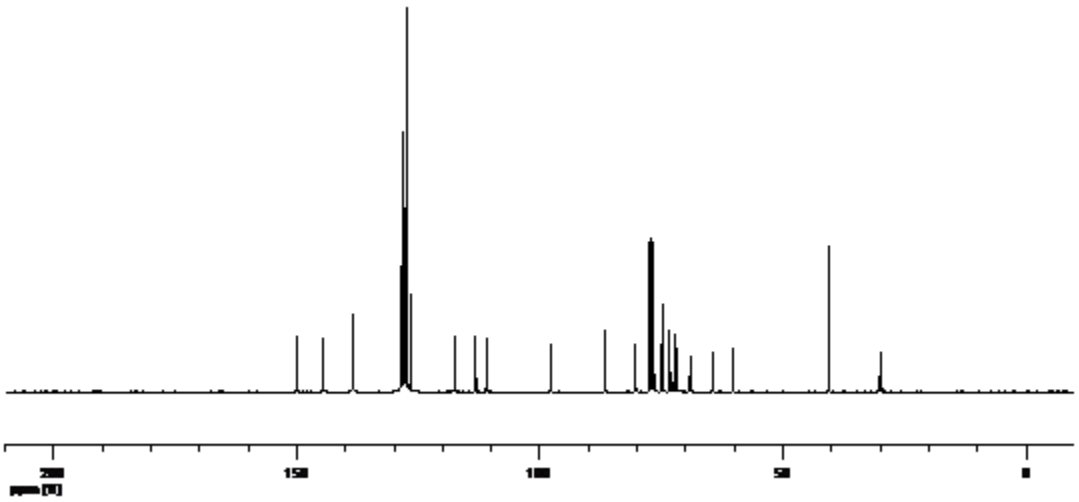


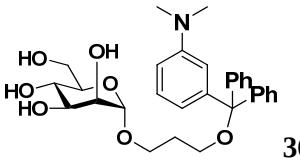




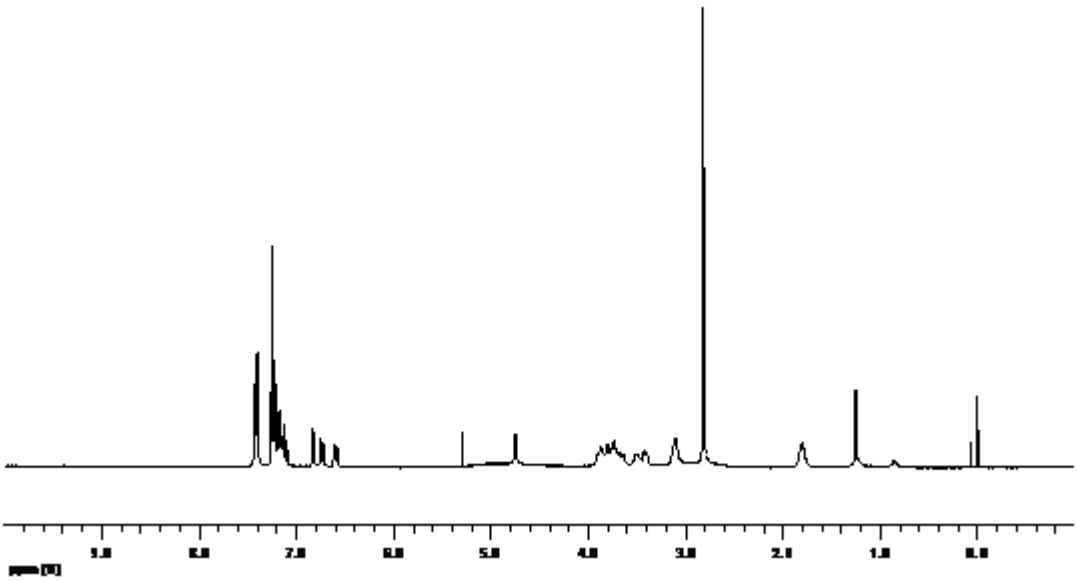
33c

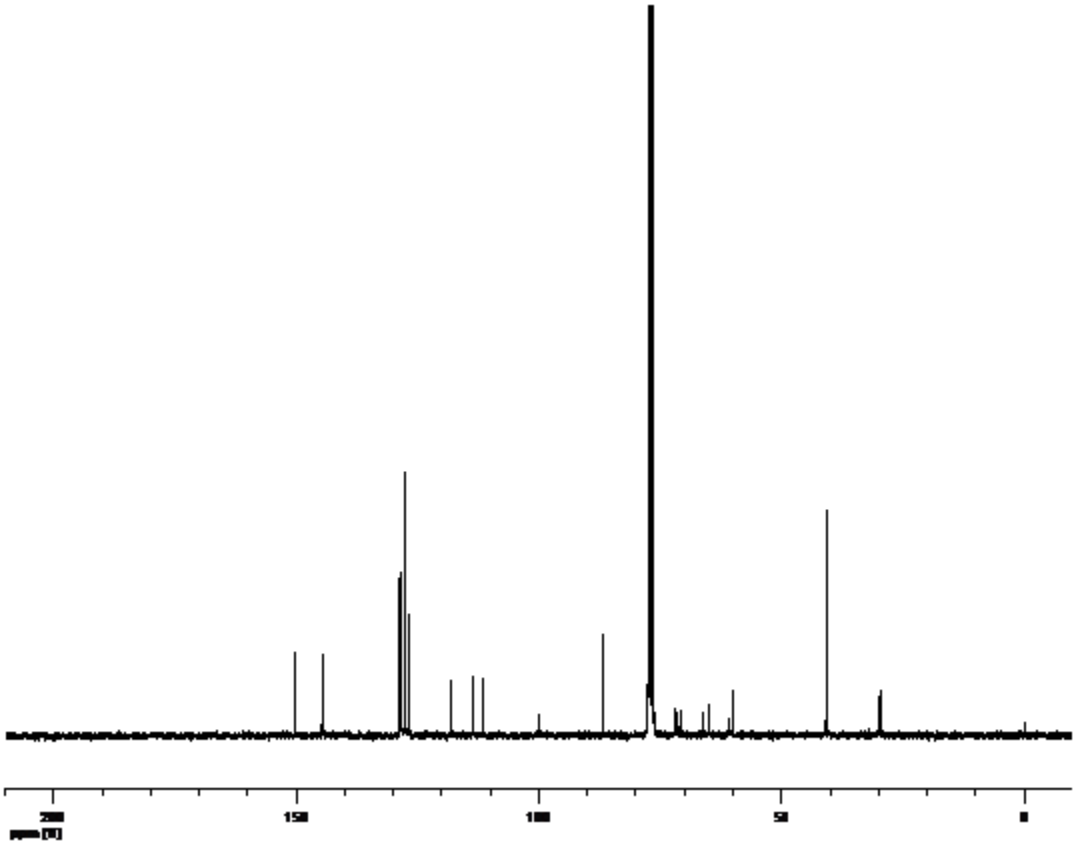




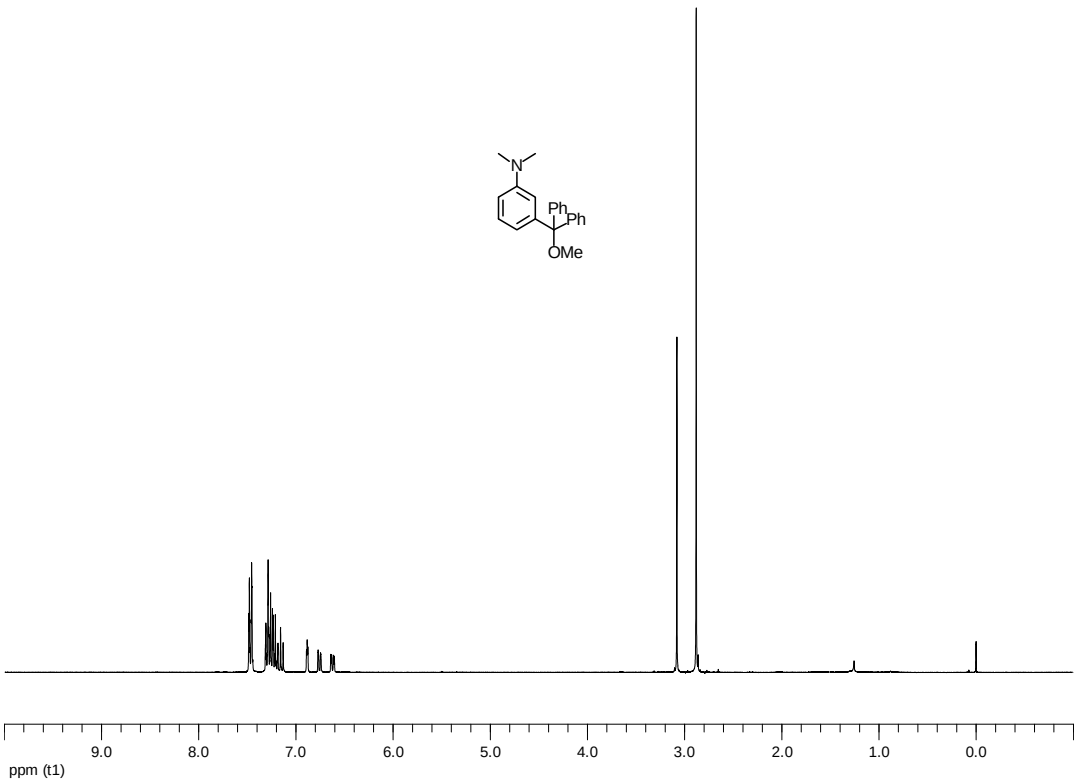


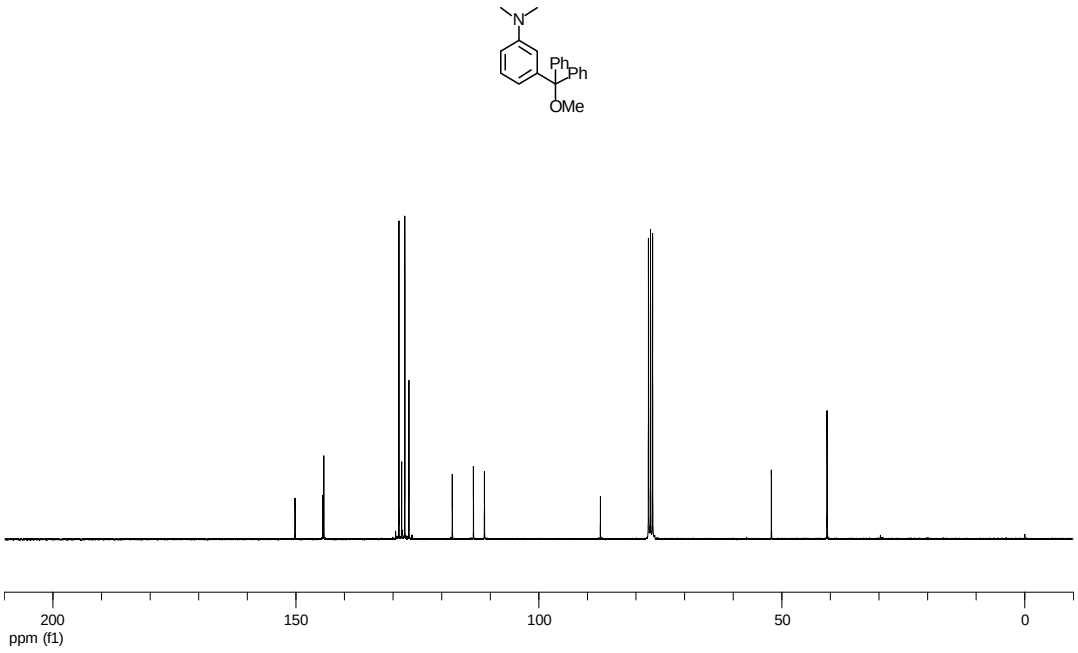
36



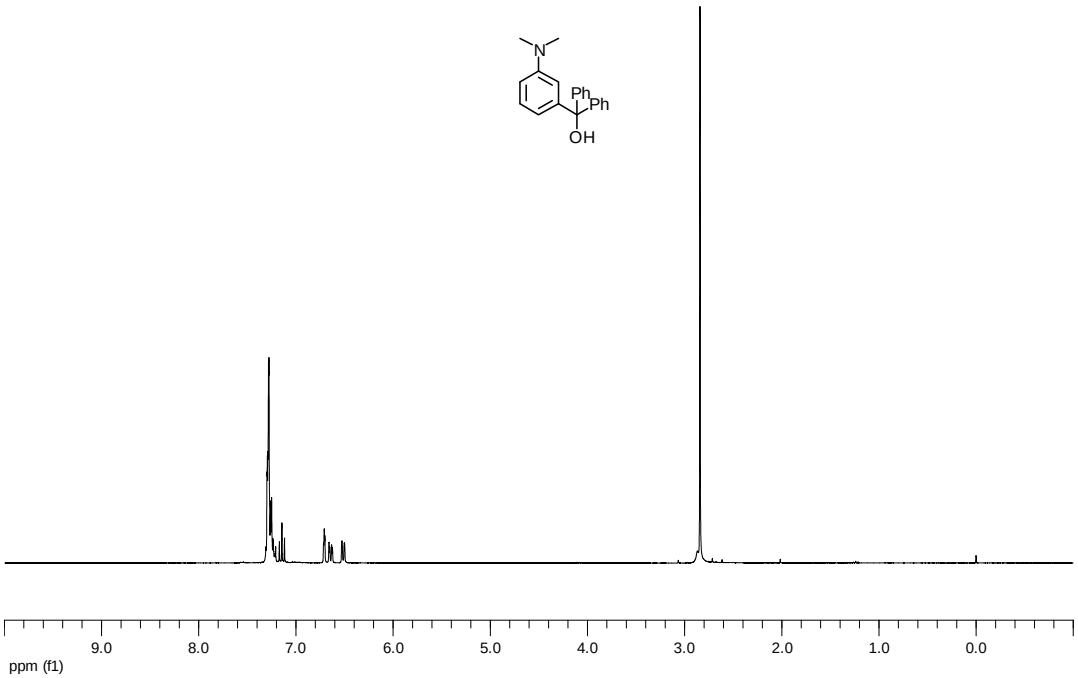


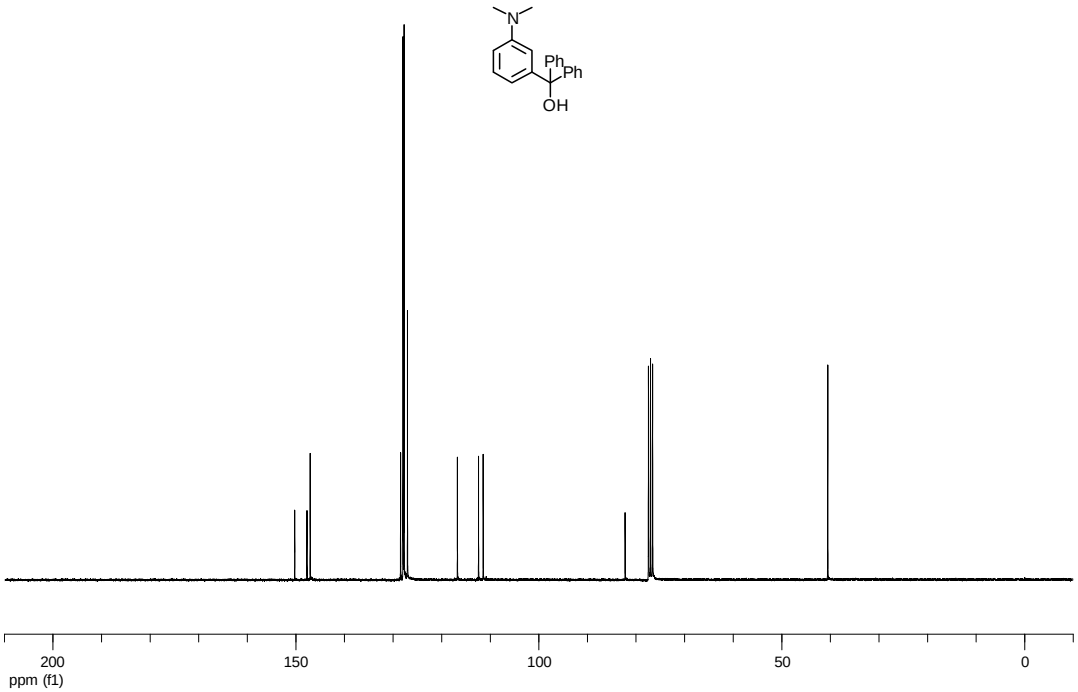
38


N
Ph
Ph
OMe
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

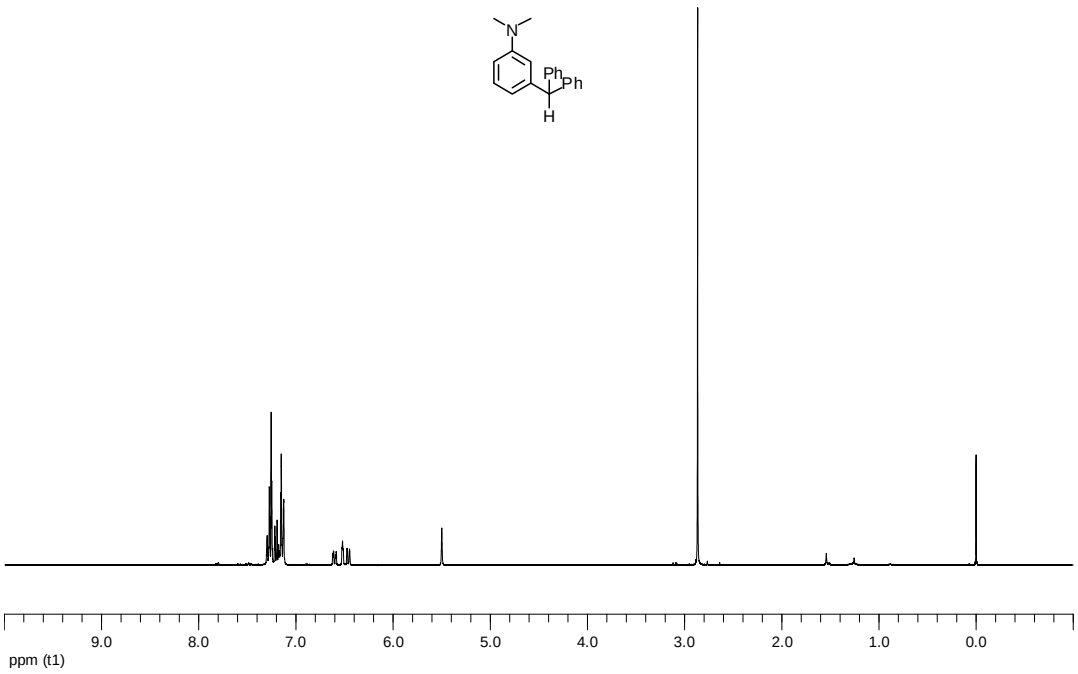

N
Ph
Ph
OMe
200
150
100
50
0
ppm (f1)

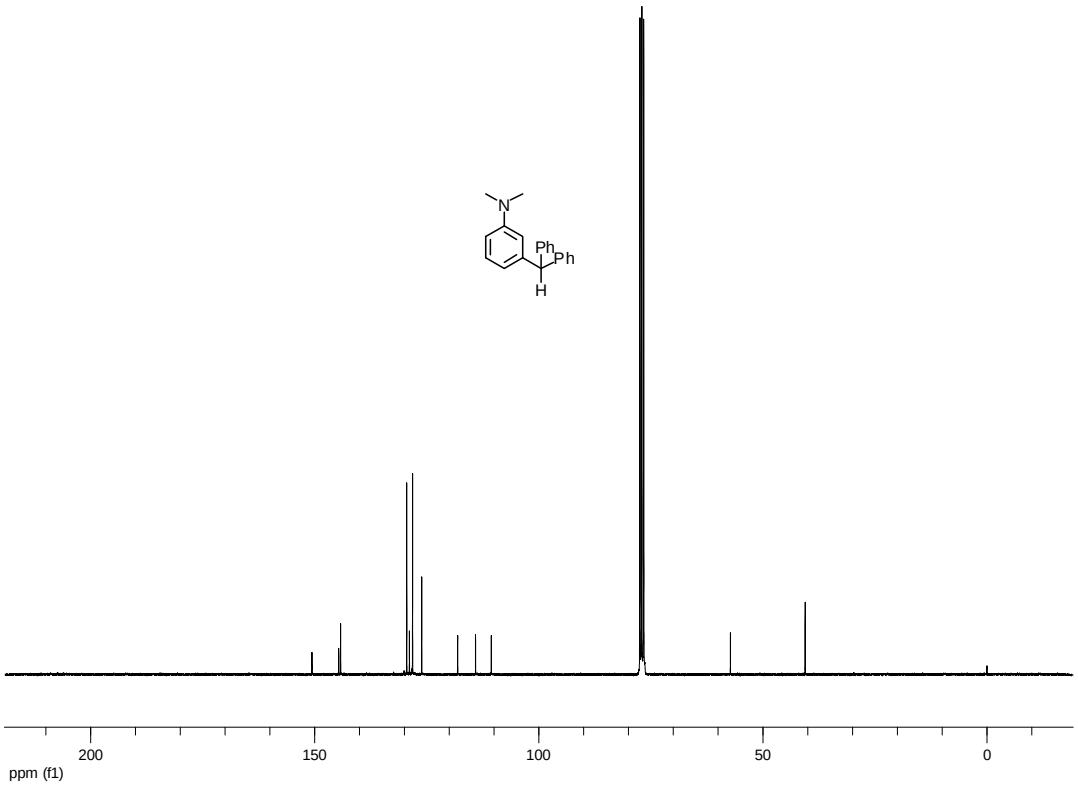
39


N
Ph
Ph
OH
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)


N
Ph
Ph
OH
200
150
100
50
0
ppm (f1)

40


N
Ph
Ph
H
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


N
Ph
Ph
H
200
150
100
50
0
ppm (f1)