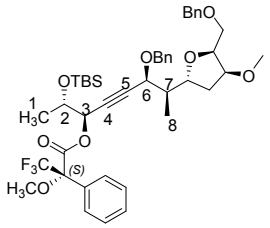
Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

A facile approach for the synthesis of C13-C24 fragment of maltepolides A, C and D

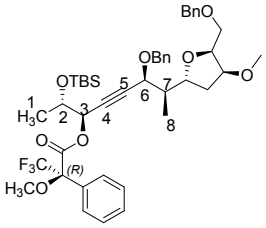
P. Sankara Rao[a] and P. Srihari*[a]

Supporting Information

1) Mosher ester analysis
2) ^{1}H and ^{13}C NMR of new compounds



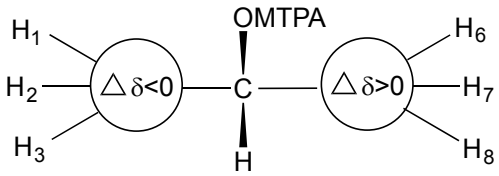
(*S*)-Mosher ester



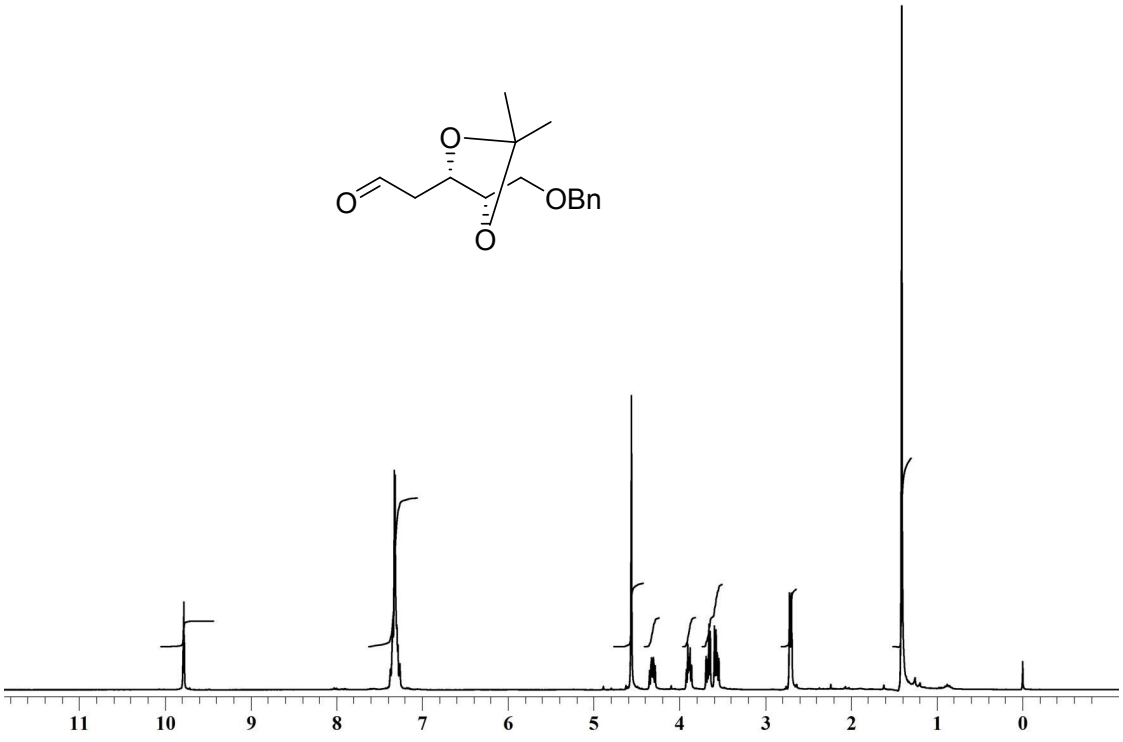
(*R*)-Mosher ester

(*R*)-MTPA ester : To a stirred solution of alcohol **8** (10 mg, 0.01mmol), DCC (14 mg, 0.06 mmol) and DMAP (4 mg, 0.003 mmol) in CH_2Cl_2 (1.0 mL) at rt was added (+)-(*R*)-α-methoxy-α-(trifluoromethyl)-phenylacetic acid (16 mg, 0.03 mmol) in one portion. After 10 h, the crude reaction mixture was purified directly by flash column chromatography (10% EtOAc/Hexanes) to provide (*R*)-MTPA ester (5 mg, 40%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36─7.28 (m, 15H), 5.65─5.64 (dd, *J* = 2.8, 1.3 Hz, 1H), 4.69─4.66 (d, *J* = 11.7 Hz, 1H), 4.62─4.59 (d, *J* =12 Hz, 1H), 4.53─4.50 (m, 3H), 4.20─4.15 (m, 1H), 4.10─4.04 (m, 1H), 3.91─3.88 (m, 1H), 3.92─3.89 (t, *J* = 3.5 Hz, 1H), 3.71─3.65 (dd, *J* = 10, 5.3 Hz, 1H), 3.59 (s, 3H), 3.29 (s, 3H), 2.16─2.13 (m, 1H), 1.82─1.78 (dd, *J* = 9.7, 6.7, 2.7 Hz, 1H), 1.52─1.50 (m, 1H), 1.22─1.20 (d, *J* = 6.1 Hz, 3H), 0.96─0.94 (d, *J* = 7.0 Hz, 3H), 0.86 (s, 9H), 0.08─0.07 (d, *J* = 4.5 Hz, 6H).

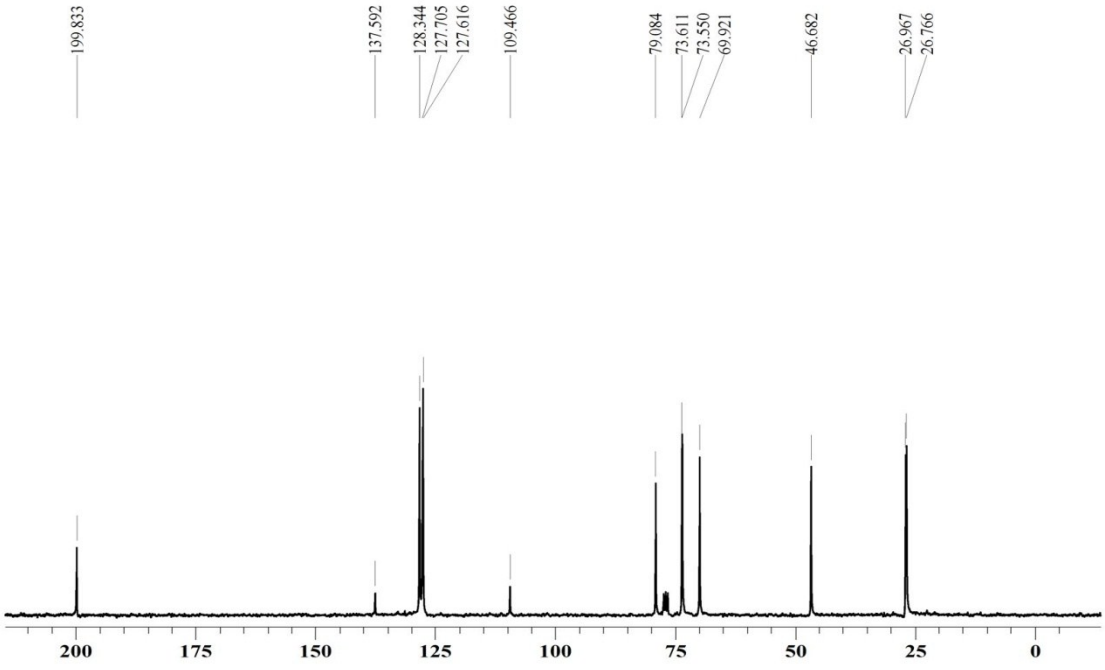
(*S*)-MTPA ester: To a stirred solution of alcohol **8** (10 mg, 0.01mmol), DCC (14 mg, 0.06 mmol) and DMAP (4 mg, 0.003 mmol) in CH_2Cl_2 (1.0 mL) at rt was added (-)-(*S*)-α-methoxy-α-(trifluoromethyl)- phenylacetic acid (16 mg, 0.03 mmol) in one portion. After 10 h, the crude reaction mixture was purified directly by flash column chromatography (10% EtOAc/Hexanes) to provide (*S*)-MTPA ester (4 mg, 26%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35─7.27 (m, 15H), 5.58─5.57 (dd, *J* = 3.3, 1.2 Hz, 1H), 4.74─4.71 (d, *J* = 11.9 Hz, 1H), 4.62─4.52 (d, *J* =12 Hz, 1H), 4.54─4.50 (m, 3H), 4.21─4.16 (m, 1H), 4.11─4.07 (m, 1H), 4.00─3.95 (m, 1H), 3.92─3.89 (t, *J* = 3.9 Hz, 1H), 3.71─3.67 (dd, *J* = 10, 5.1 Hz, 1H), 3.57 (s, 3H), 3.29 (s, 3H), 2.18─2.14 (m, 1H), 1.85─1.80 (dd, *J* = 8.3, 6.9, 3.0 Hz, 1H), 1.53─1.50 (m, 1H), 1.19─1.17 (d, *J* = 6.2 Hz, 3H), 0.99─0.97 (d, *J* = 7.0 Hz, 3H), 0.84 (s, 9H), 0.00─ -0.01(d, *J* = 3.8 Hz, 6H).



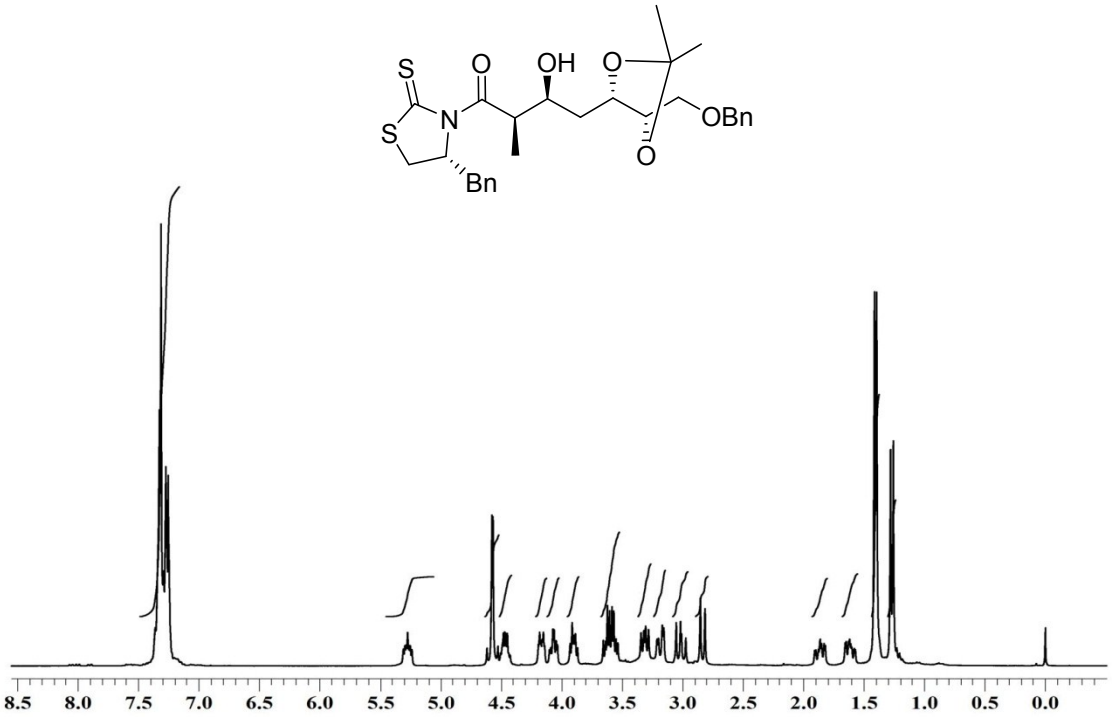
	δ (ppm) *(S)*-Mosher ester	δ (ppm) *(R)*-Mosher ester	$\Delta\delta_{SR}$ (= $\delta_S - \delta_R$) (500 MHz)
1 ^{1}H	1.19	1.22	-0.03
2 ^{1}H	4.00	4.06	-0.06
3 ^{1}H	5.58	5.65	-0.07
6 ^{1}H	4.54	4.52	+0.02
7 ^{1}H	2.18	2.16	+0.02
8 ^{1}H	0.99	0.96	+0.03



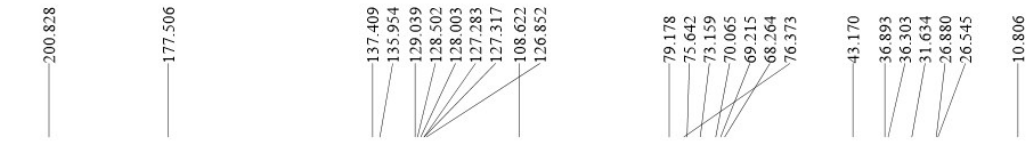
^{1}H NMR OF COMPOUND 15 (300 MHz, $CDCl_3$)

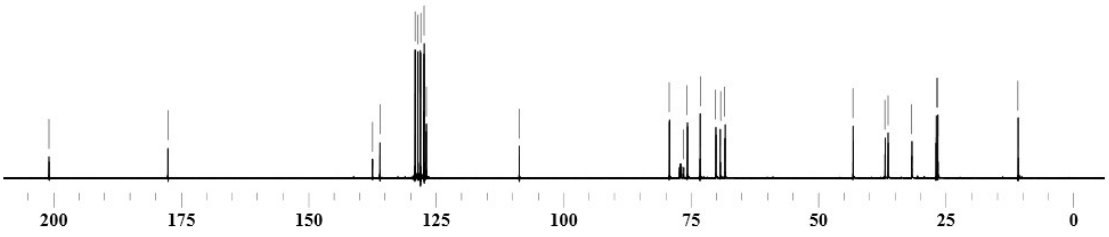


^{13}C NMR OF COMPOUND 15 (75 MHz, $CDCl_3$)

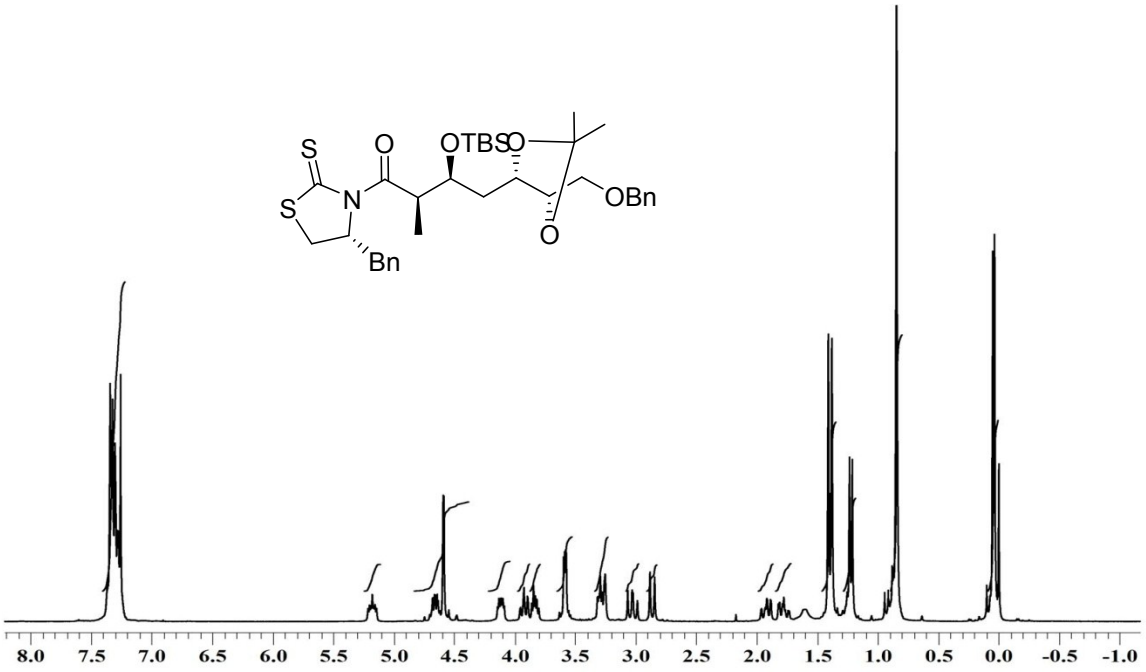


^{1}H NMR OF COMPOUND 19 (500 MHz, $CDCl_3$)

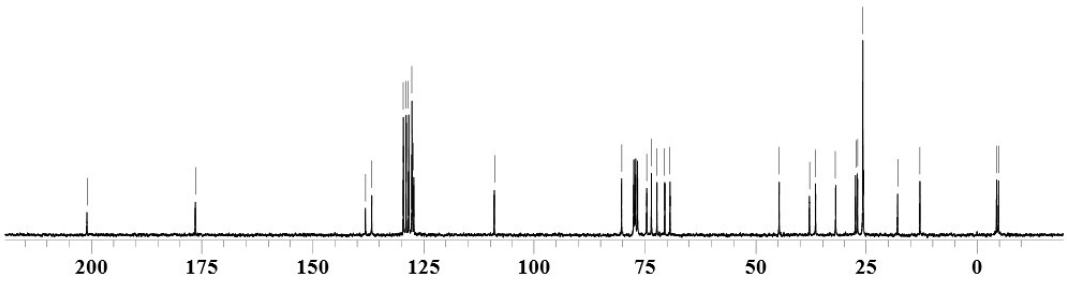




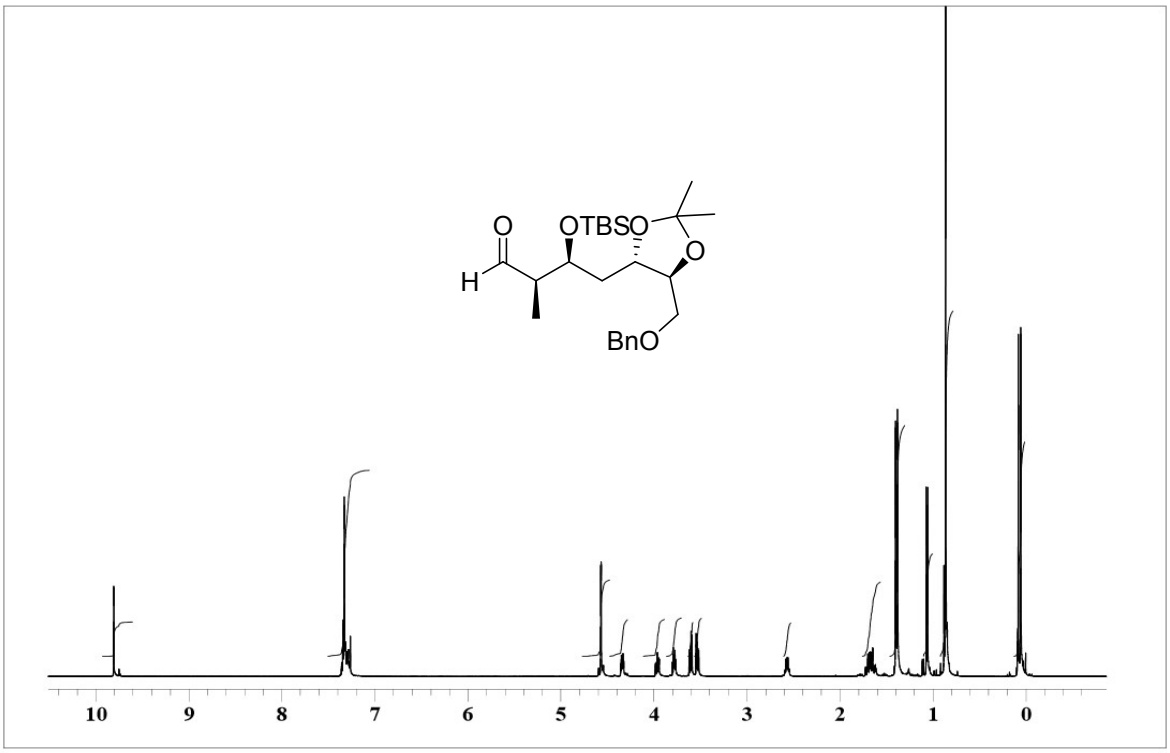
^{13}C NMR OF COMPOUND 19 (125 MHz, $CDCl_3$)



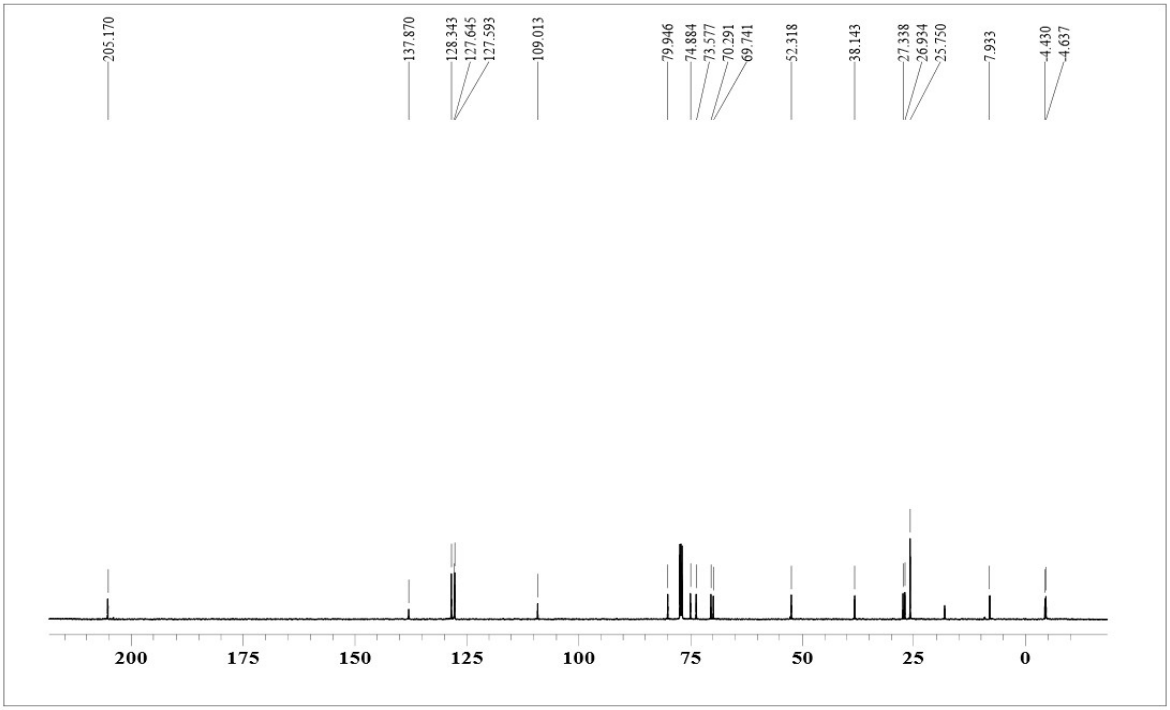
^{1}H NMR OF COMPOUND 14 (300 MHz, $CDCl_3$)



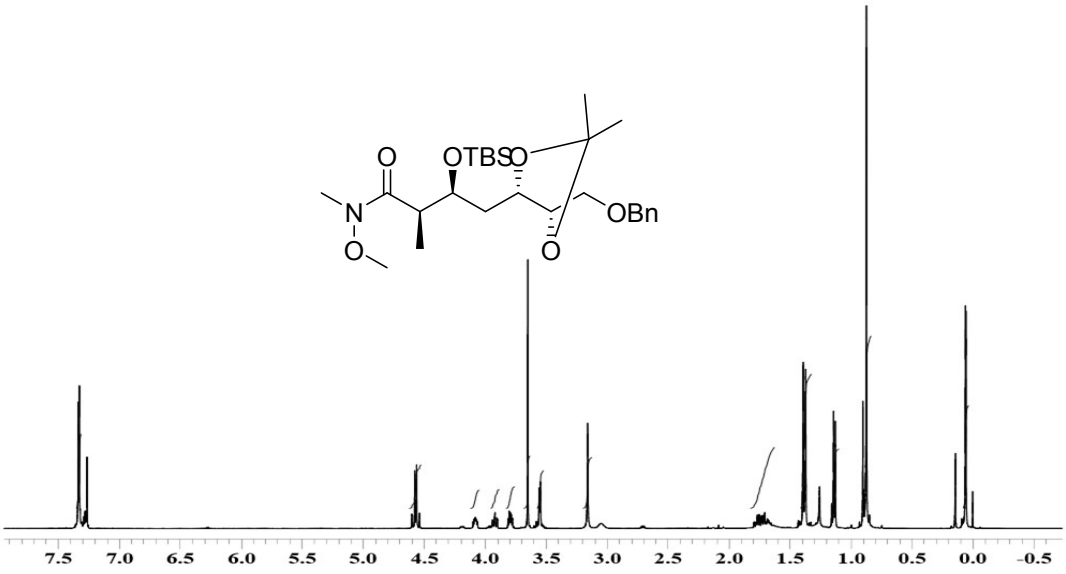
^{13}C NMR OF COMPOUND 14 (75 MHz, $CDCl_3$)



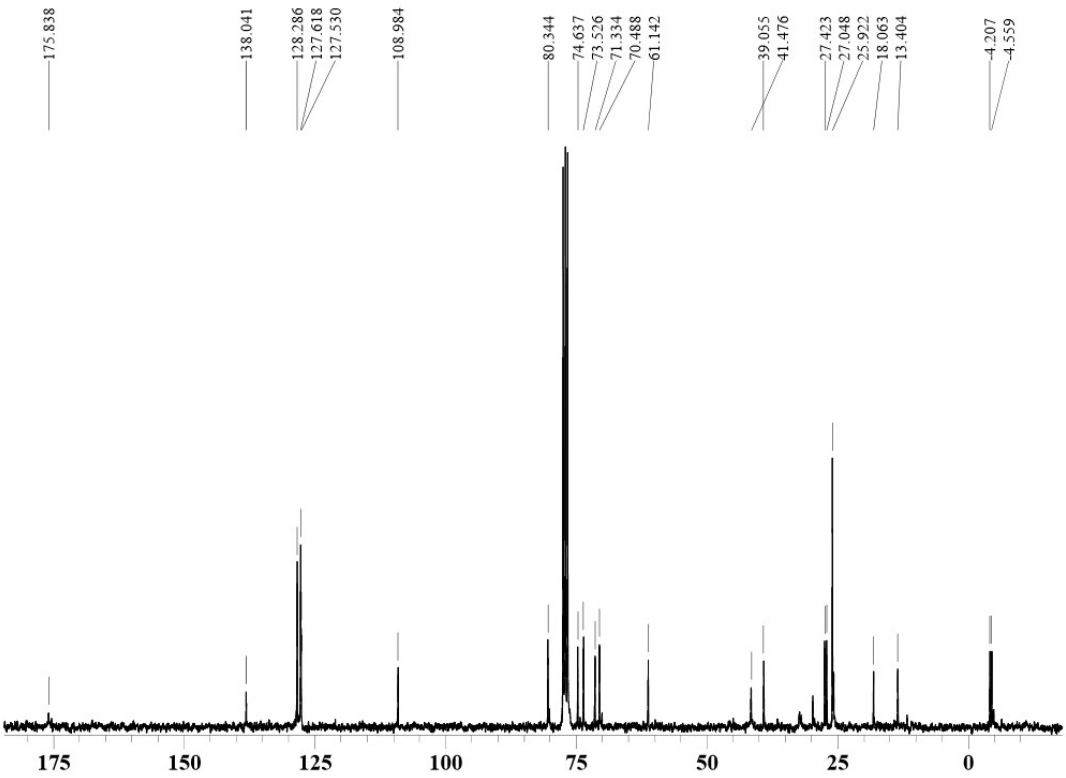
^{1}H NMR OF COMPOUND 22 (500 MHz, $CDCl_3$)



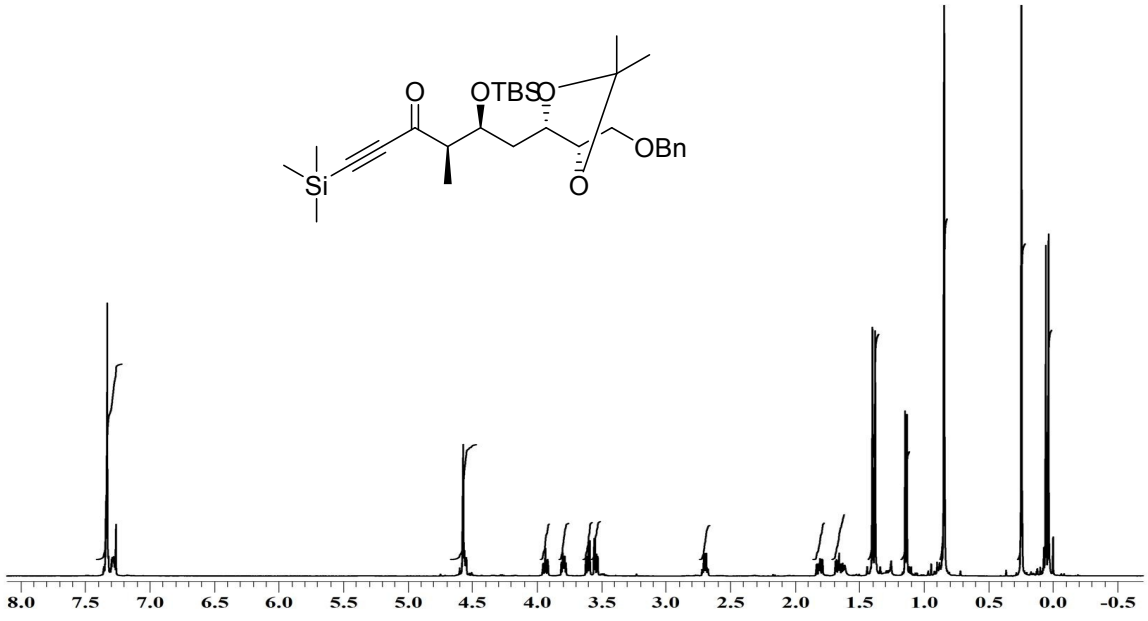
^{13}C NMR OF COMPOUND 22 (125 MHz, $CDCl_3$)



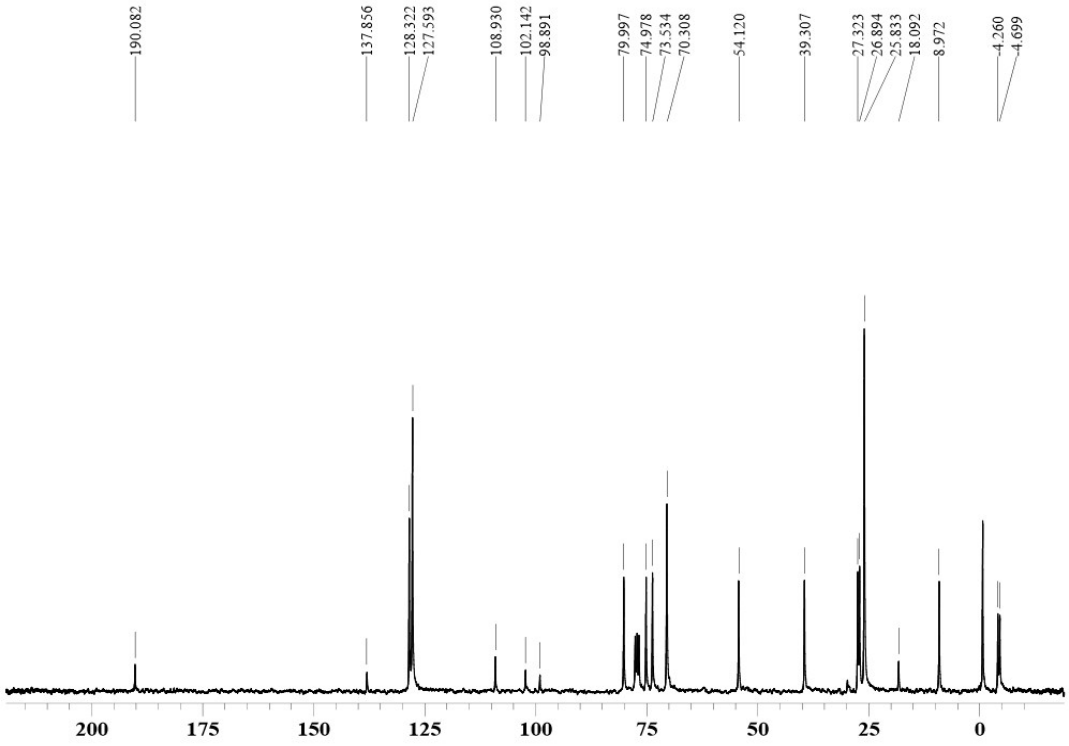
^{1}H NMR OF COMPOUND 20 (500 MHz, $CDCl_3$)



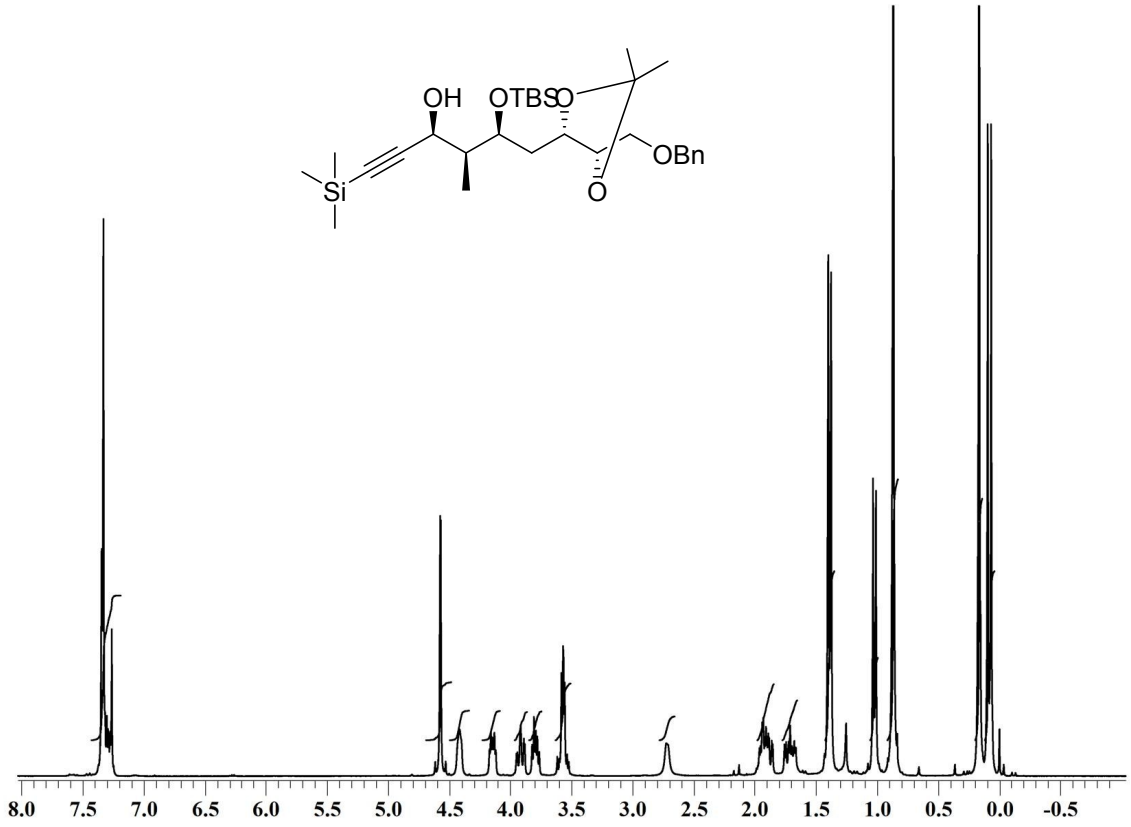
^{13}C NMR OF COMPOUND 20 (125 MHz, $CDCl_3$)



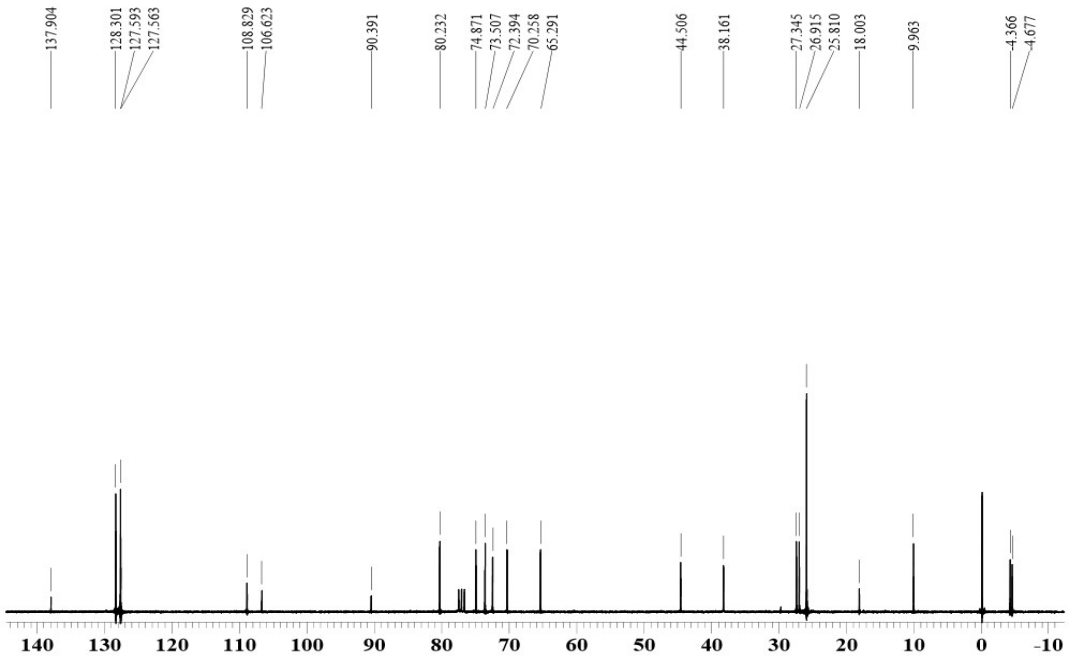
^{1}H NMR OF COMPOUND 13 (500 MHz, $CDCl_3$)



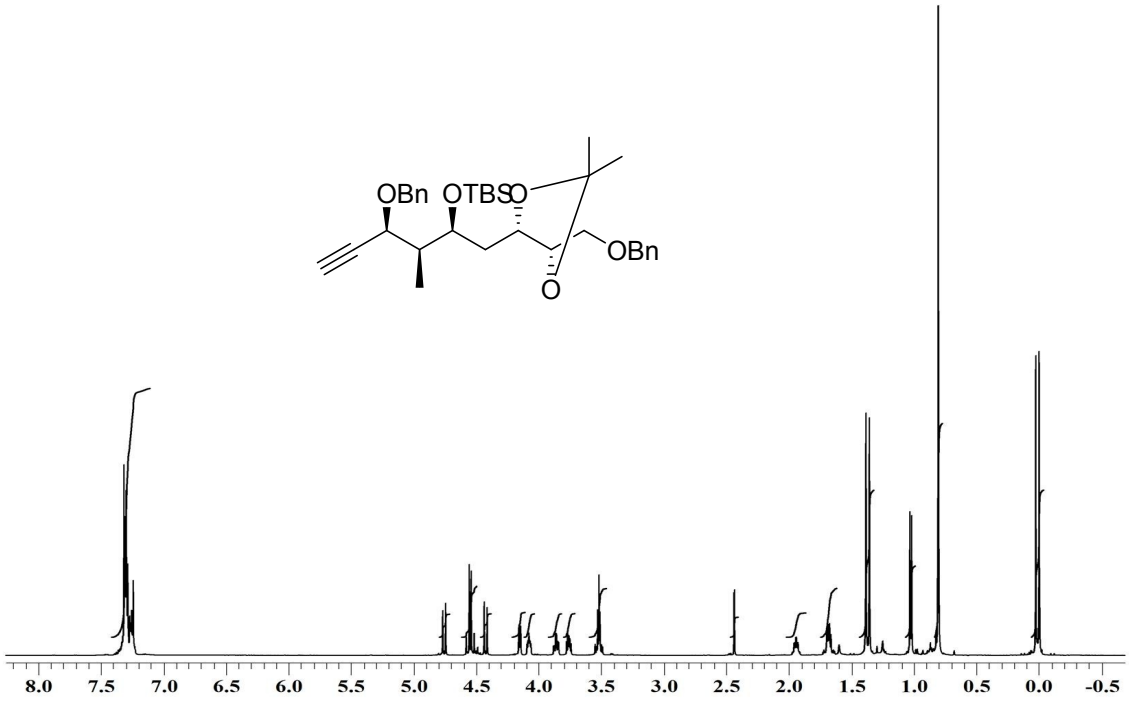
^{13}C NMR OF COMPOUND 13 (125 MHz, $CDCl_3$)



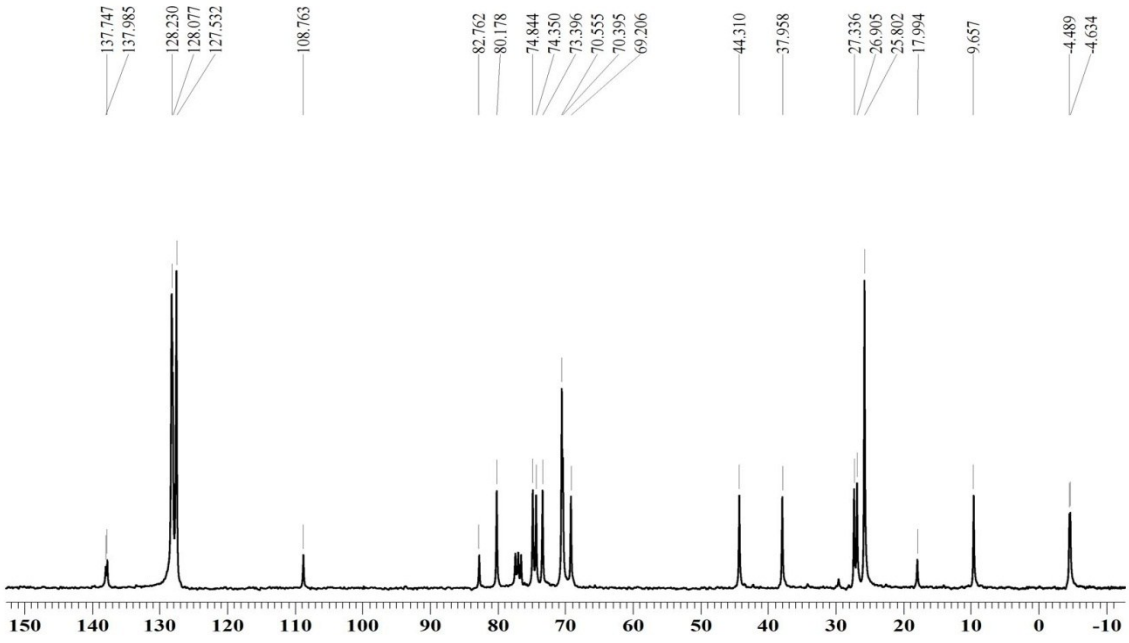
^{1}H NMR OF COMPOUND 23 (300 MHz, $CDCl_3$)



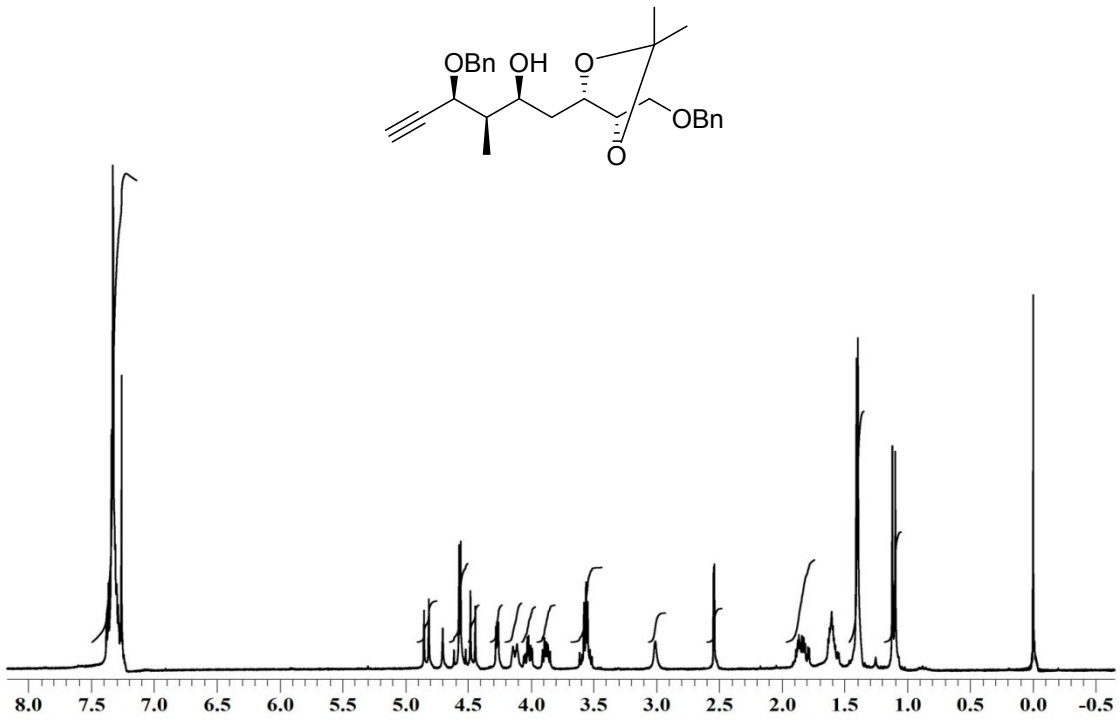
^{13}C NMR OF COMPOUND 23 (75 MHz, $CDCl_3$)



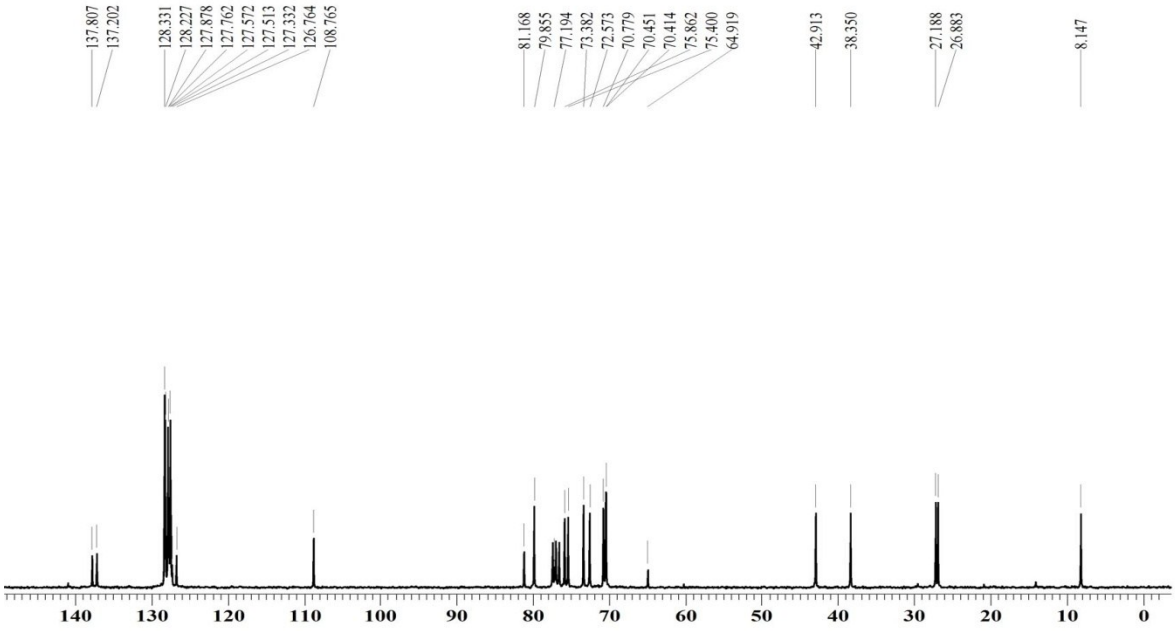
^{1}H NMR OF COMPOUND 26 (500 MHz, $CDCl_3$)



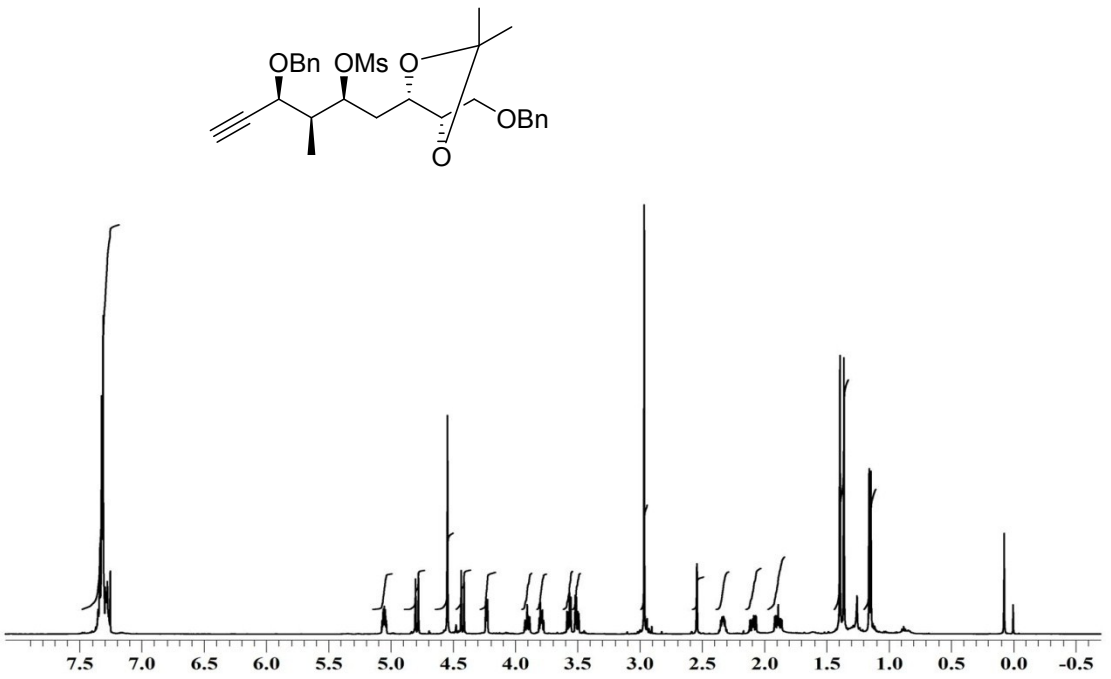
^{13}C NMR OF COMPOUND 26 (125 MHz, $CDCl_3$)



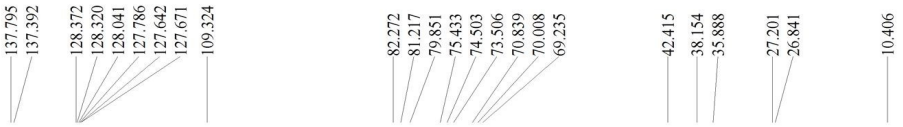
^{1}H NMR OF COMPOUND 27 (500 MHz, $CDCl_3$)

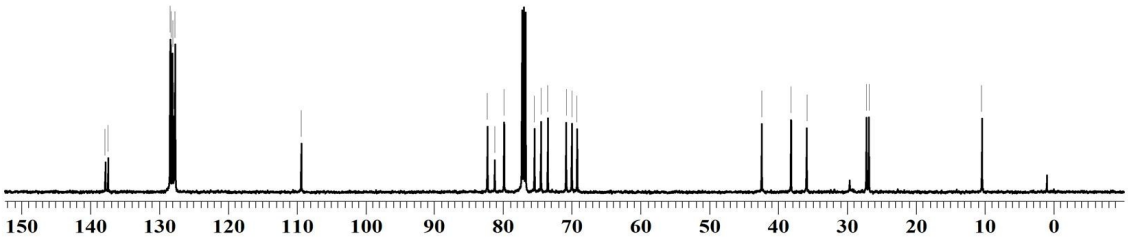


^{13}C NMR OF COMPOUND 27 (125 MHz, $CDCl_3$)

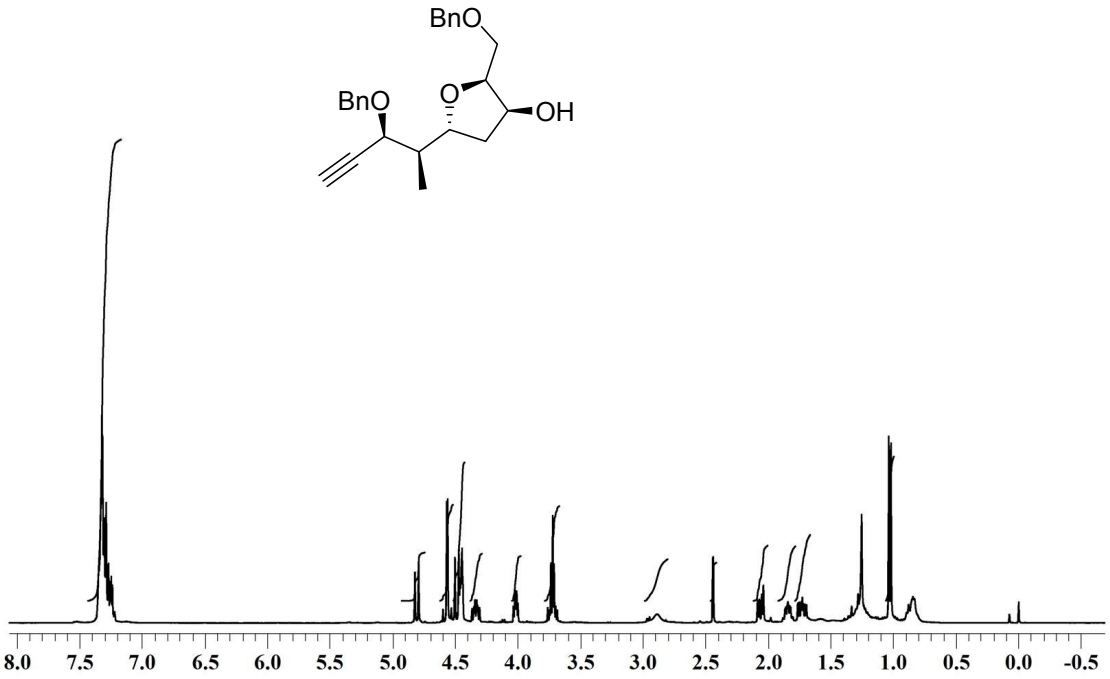


^{1}H NMR OF COMPOUND 12 (500 MHz, $CDCl_3$)

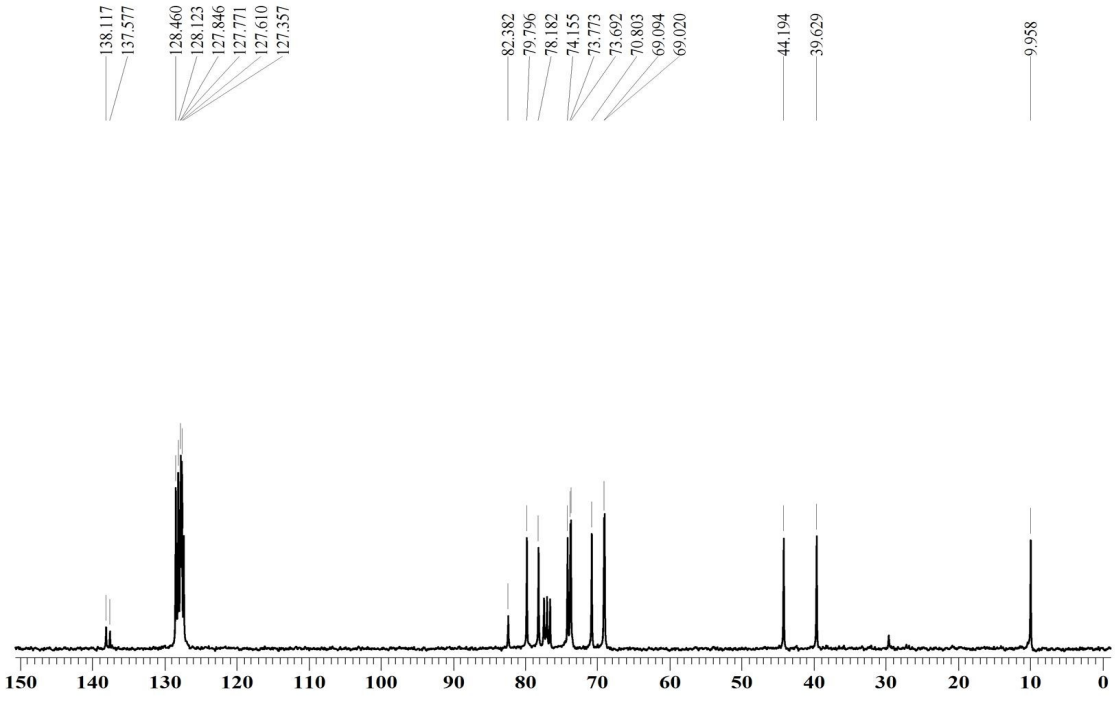




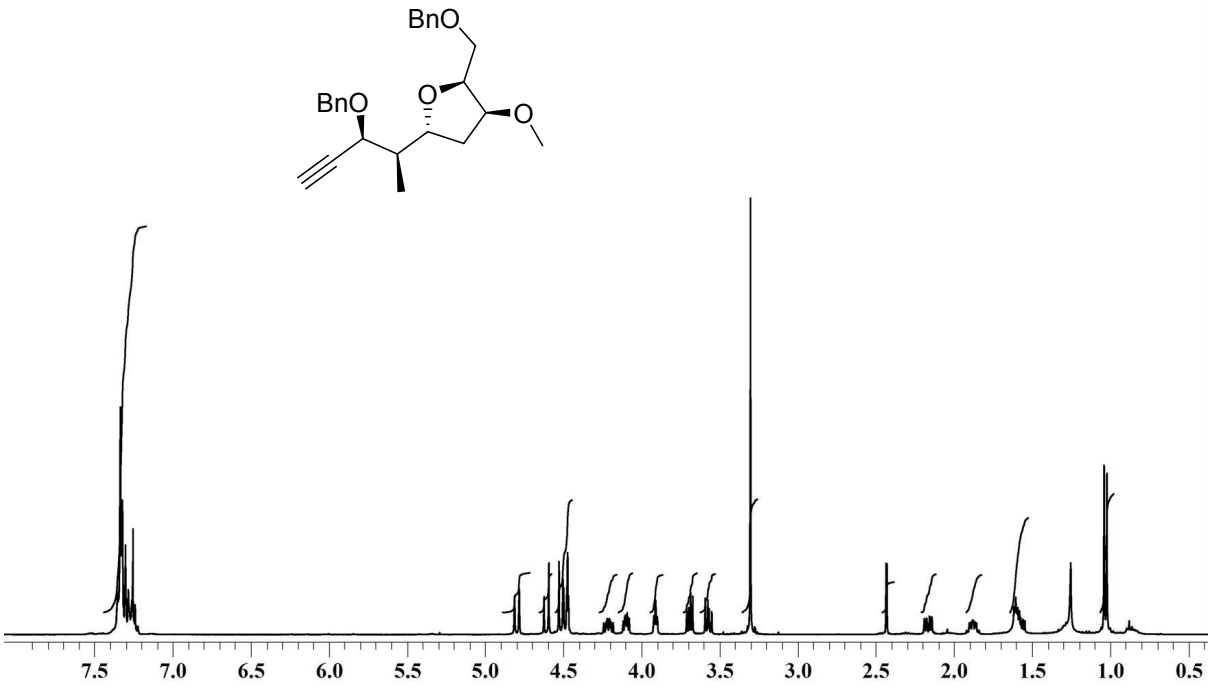
^{13}C NMR OF COMPOUND 12 (125 MHz, $CDCl_3$)



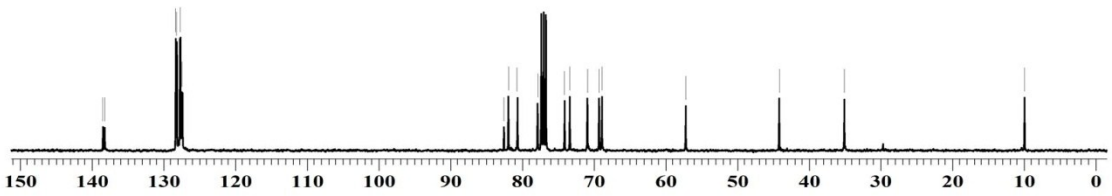
^{1}H NMR OF COMPOUND 28 (500 MHz, $CDCl_3$)



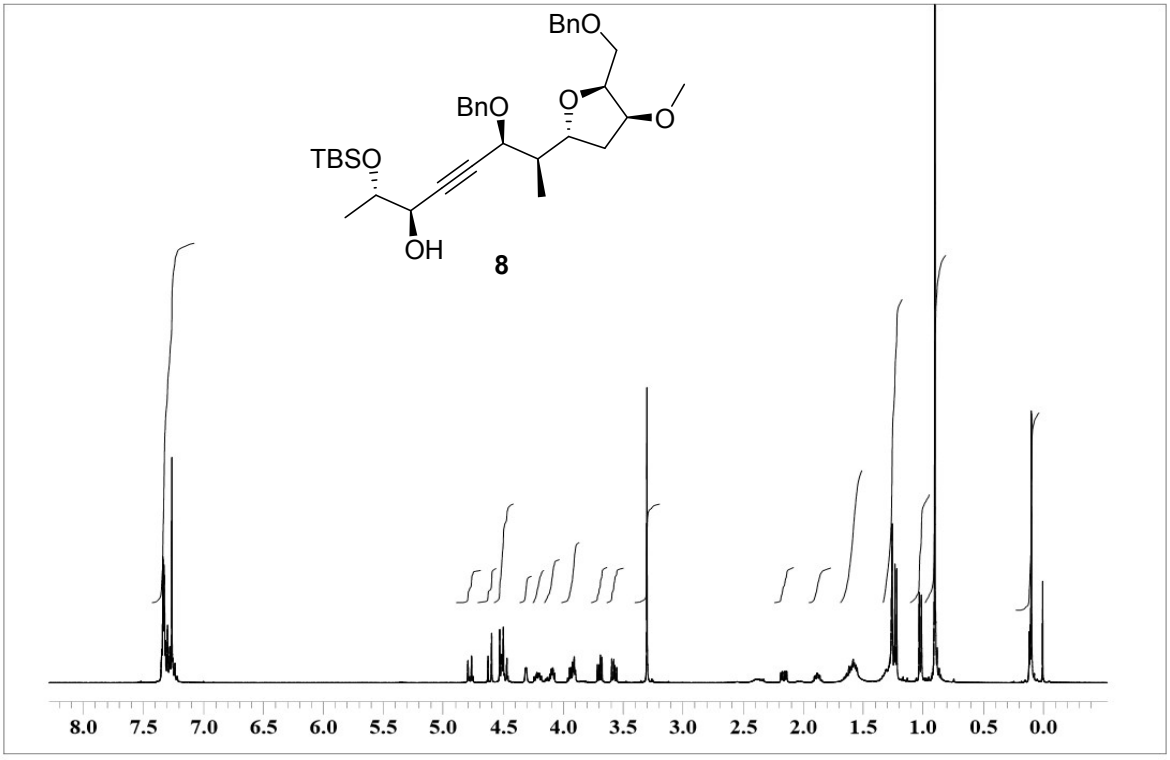
^{13}C NMR OF COMPOUND 28 (125 MHz, $CDCl_3$)



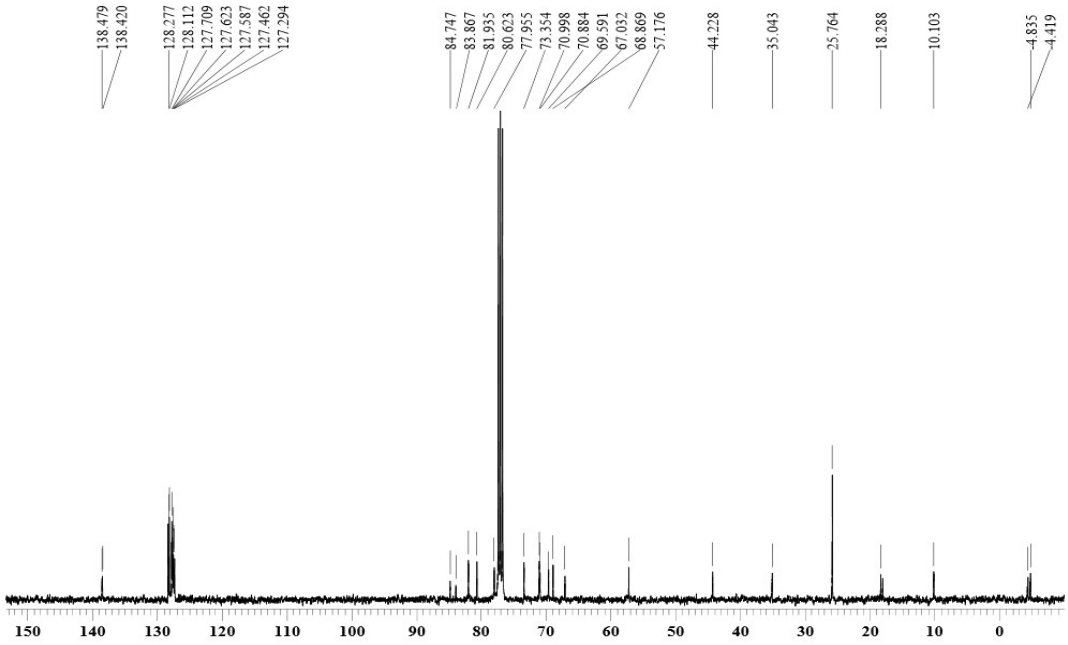
^{1}H NMR OF COMPOUND 10 (400 MHz, $CDCl_3$)



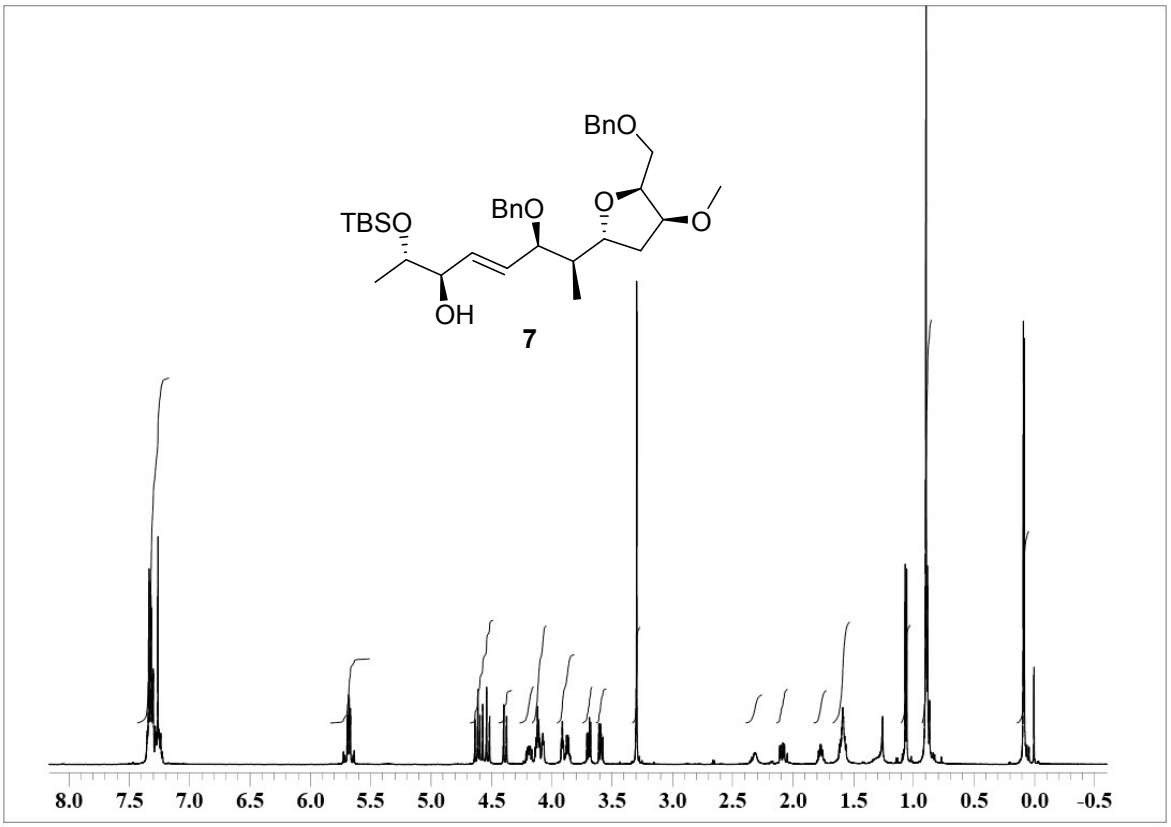
^{13}C NMR OF COMPOUND 10 (100 MHz, $CDCl_3$)



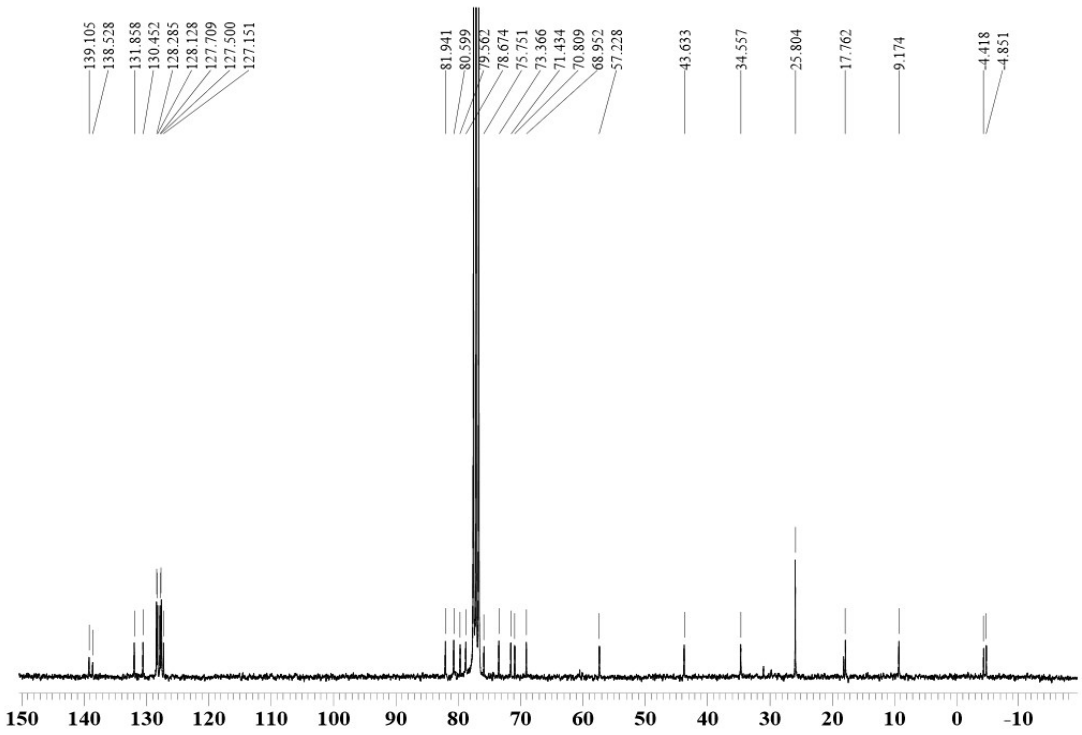
^{1}H NMR OF COMPOUND 8 (400 MHz, $CDCl_3$)



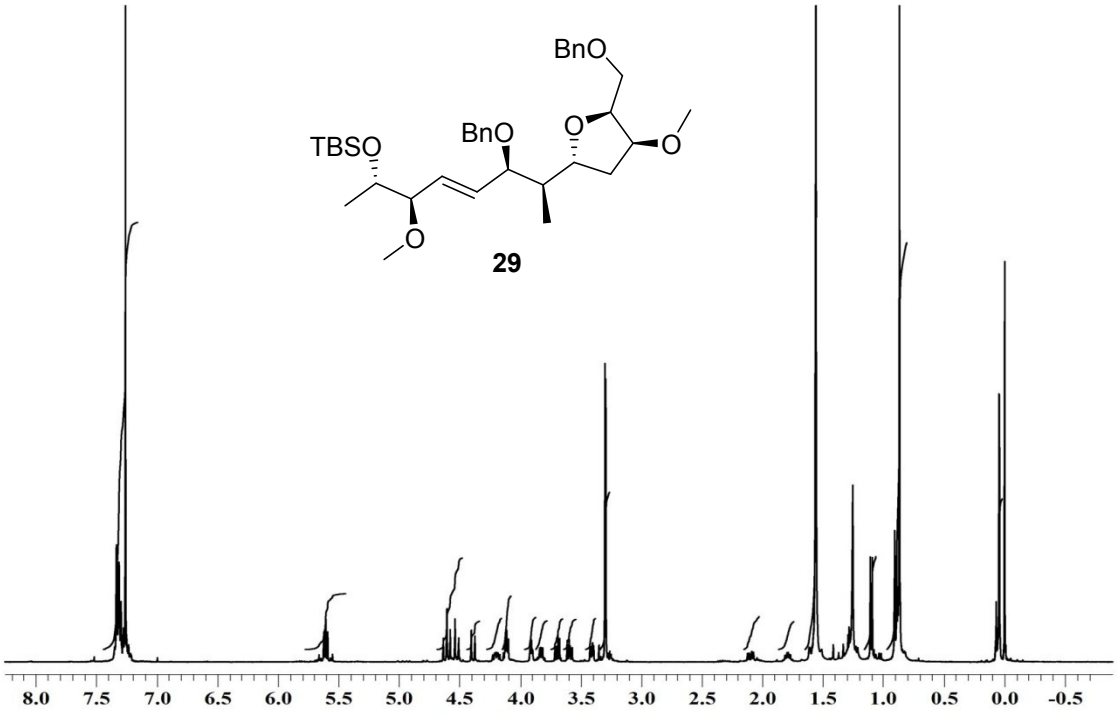
^{13}C NMR OF COMPOUND 8 (100 MHz, $CDCl_3$)



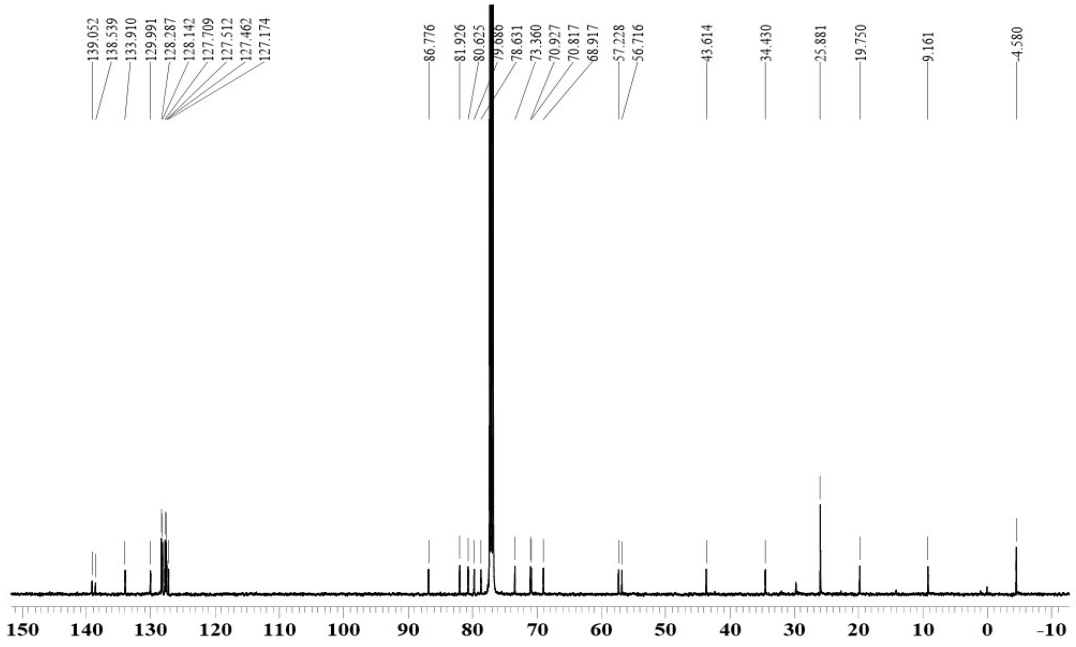
^{1}H NMR OF COMPOUND 7 (500 MHz, $CDCl_3$)



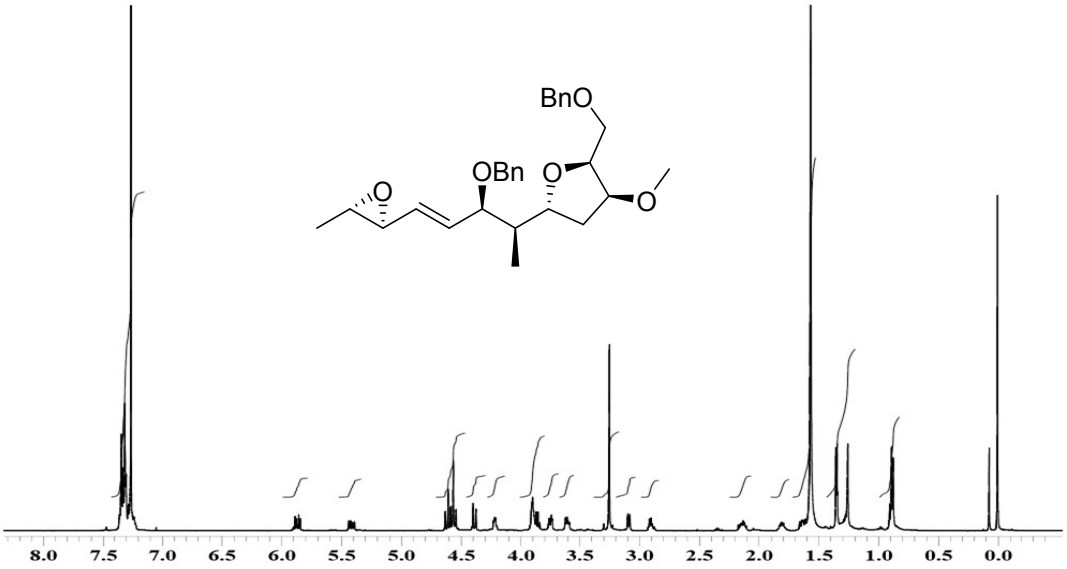
^{13}C NMR OF COMPOUND 7 (125 MHz, $CDCl_3$)



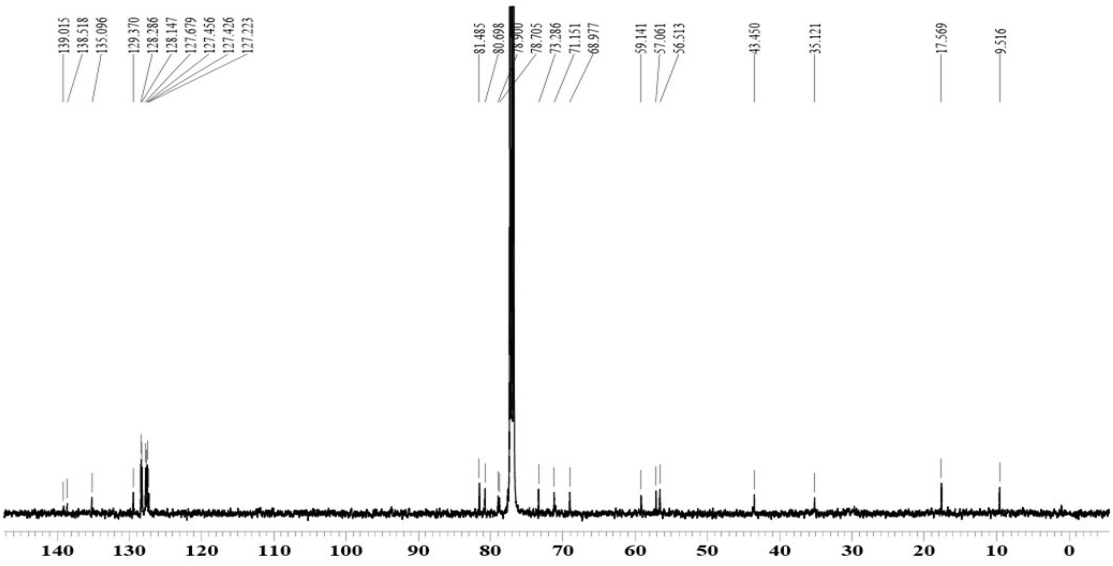
^{1}H NMR OF COMPOUND 29 (500 MHz, $CDCl_3$)



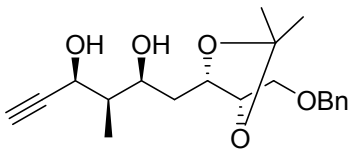
^{13}C NMR OF COMPOUND 29 (125 MHz, $CDCl_3$)

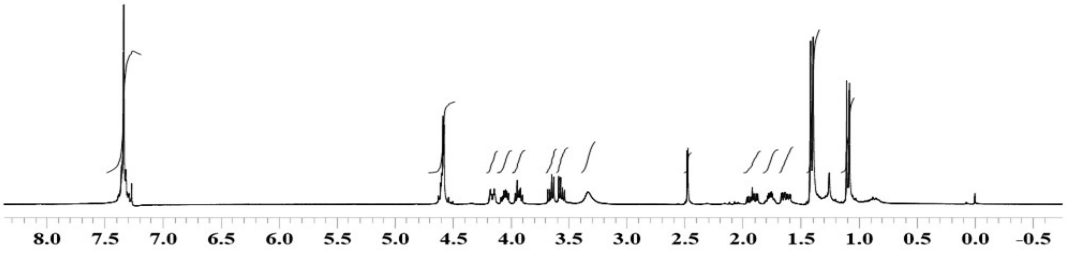


^{1}H NMR OF COMPOUND 30 (500 MHz, $CDCl_3$)

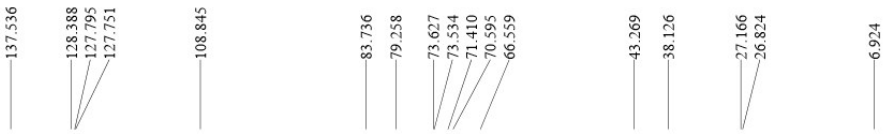


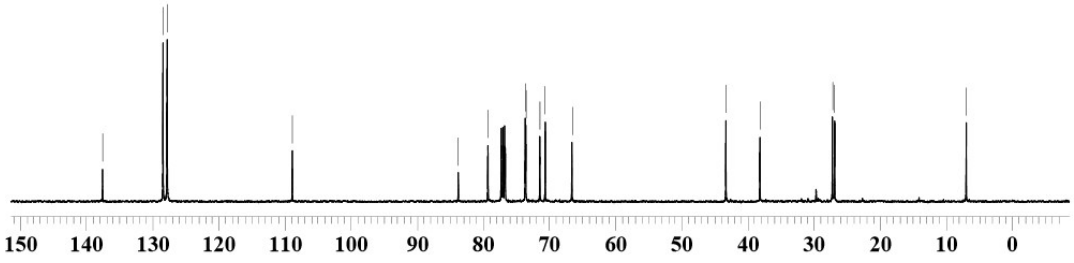
^{13}C NMR OF COMPOUND 30 (125 MHz, $CDCl_3$)



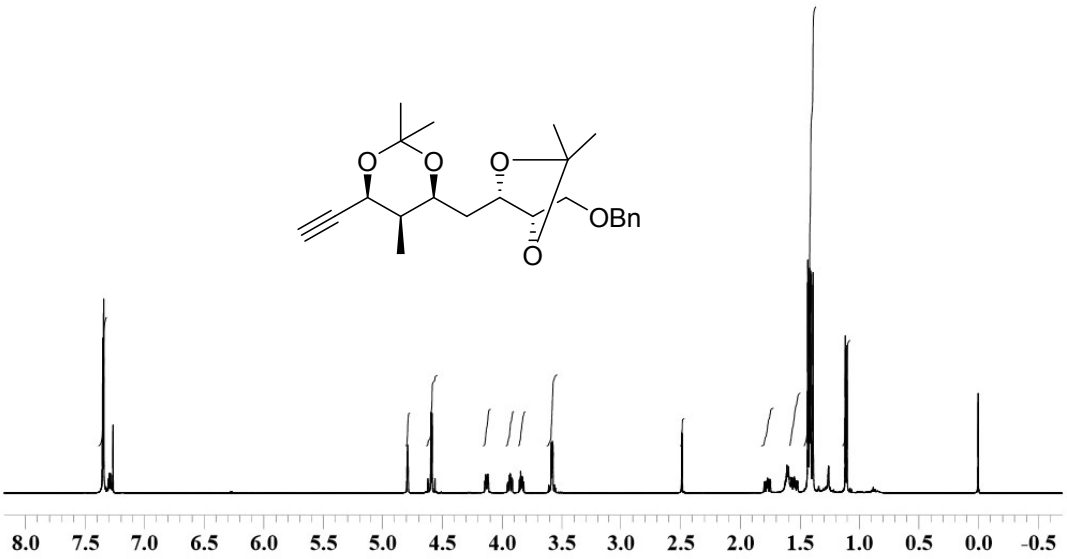


^{1}H NMR OF COMPOUND 24 (300 MHz, $CDCl_3$)

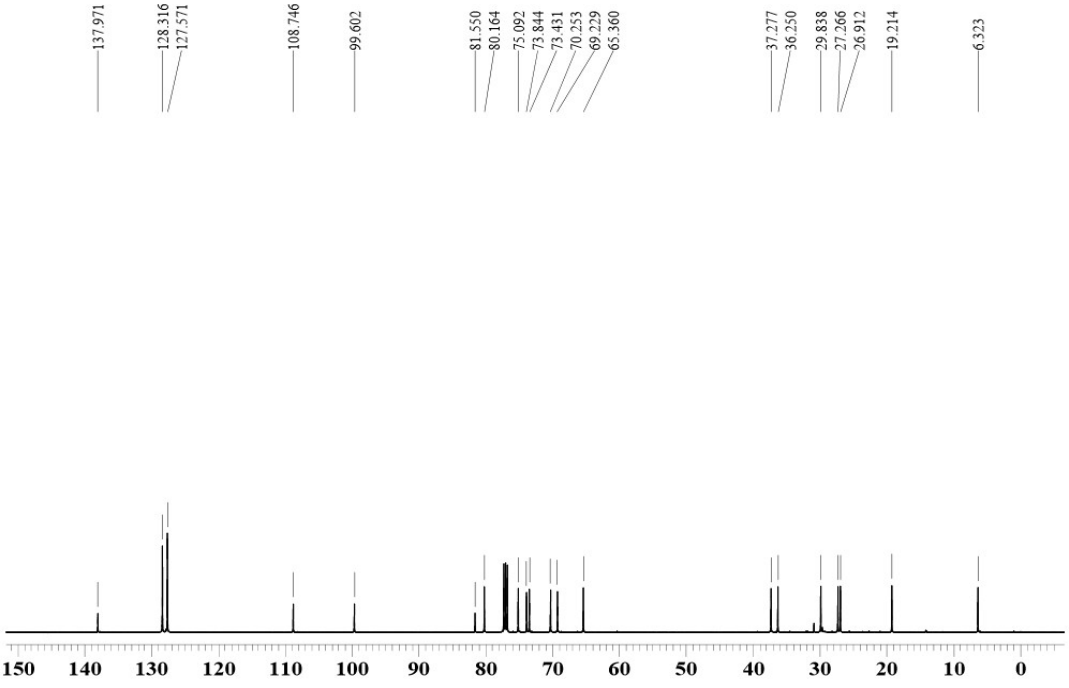




^{13}C NMR OF COMPOUND 24 (75 MHz, $CDCl_3$)



^{1}H NMR OF COMPOUND 25 (500 MHz, $CDCl_3$)



^{13}C NMR OF COMPOUND 25 (125 MHz, $CDCl_3$)