

Total Synthesis of 2''', 5'''-Diepisilvestrol and its C1'''-Epimer: Key Structure Activity Relationships at C1''' and C2'''.

Jennifer M. Chambers,[†] David C. S. Huang,[‡] Lisa M. Lindqvist,[‡] G. Paul Savage,[¶] Jonathan M. White[†] and Mark A. Rizzacasa^{†*}
masr@unimelb.edu.au

[†]School of Chemistry, The Bio21 Institute, The University of Melbourne, Victoria 3010, Australia.

[‡]Walter and Eliza Hall Institute of Medical Research, 1G Royal Parade, Parkville, Victoria, 3052, Australia.

[¶]CSIRO Molecular and Health Technologies, Bayview Avenue, Victoria 3168, Australia.

Supporting Information

Figure 1: HPLC trace of separation of enantiomers (–)- 14 and (+)- 14	S2
Figure 2: HPLC trace of separation of isomers 4 and 15	S3
¹ H NMR spectrum of methyl ketal 9	S4
¹³ C NMR spectrum of methyl ketal 9	S5
¹ H NMR spectrum of alcohol 10	S6
¹³ C NMR spectrum of alcohol 10	S7
¹ H NMR spectrum of bis-TBS ether 11	S8
¹³ C NMR spectrum of bis-TBS ether 11	S9
¹ H NMR spectrum of lactol 6	S10
¹³ C NMR spectrum of lactol 6	S11
¹ H NMR spectrum of <i>endo</i> cyclopentabenzofuran (–)- 14	S12
¹ H NMR spectrum of phenol (–)- 7	S13
¹ H NMR spectrum of 2''', 5'''-diepisilvestrol (4)	S14
¹³ C NMR spectrum of 2''', 5'''-diepisilvestrol (4)	S15
¹ H NMR spectrum of 1''', 2''', 5'''-triepisilvestrol (15)	S16
¹³ C NMR spectrum of 1''', 2''', 5'''-triepisilvestrol (15)	S17

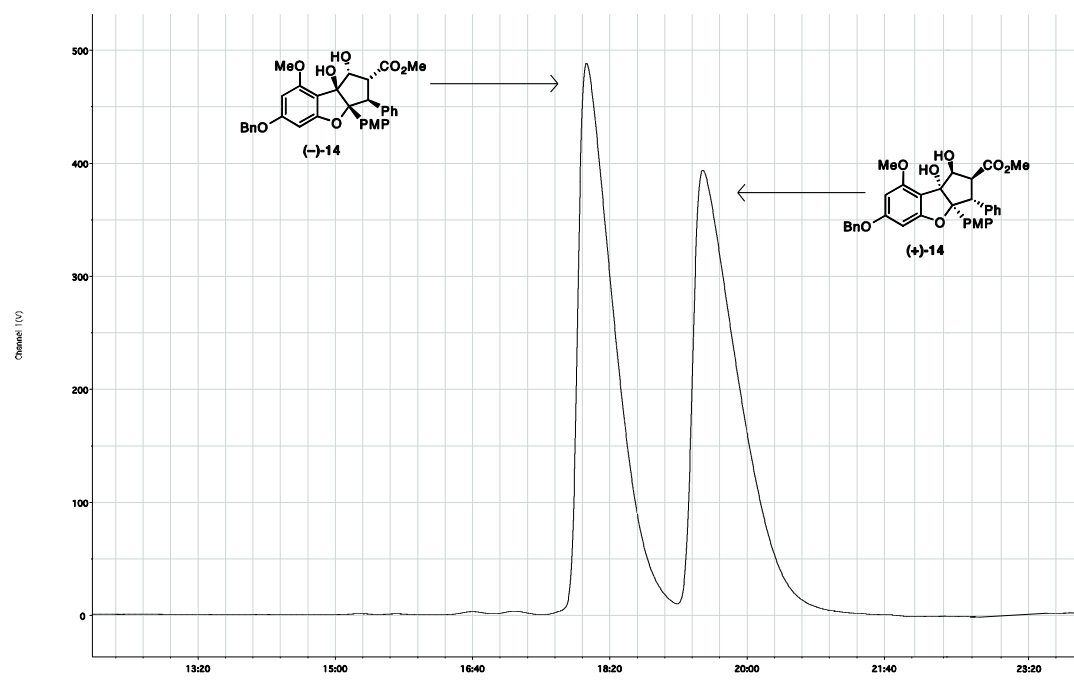


Figure 1: HPLC trace of separation of racemic *endo* **14**: (-)-**14** ($R_t = 18.03$ min); (+)-**14** ($R_t = 19.45$ min). 5 μ m Phenomenex Lux Cellulose-1 semi-preparative column: 10x250mm, 70% MeCN/H₂O eluent, flow rate: 2.00 mL/min.

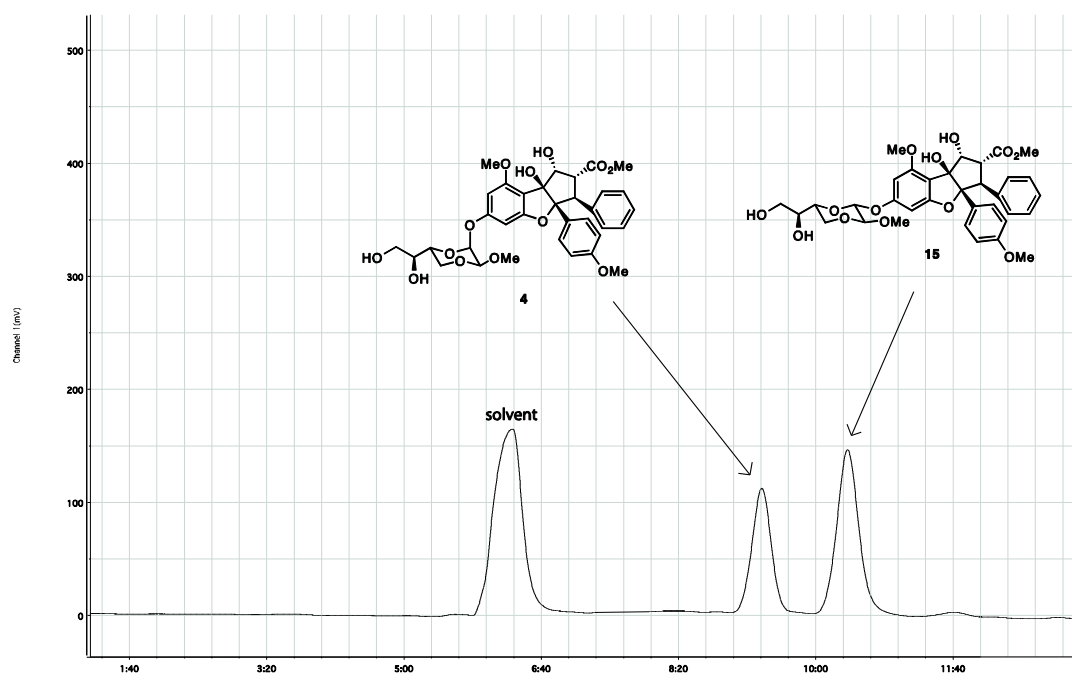
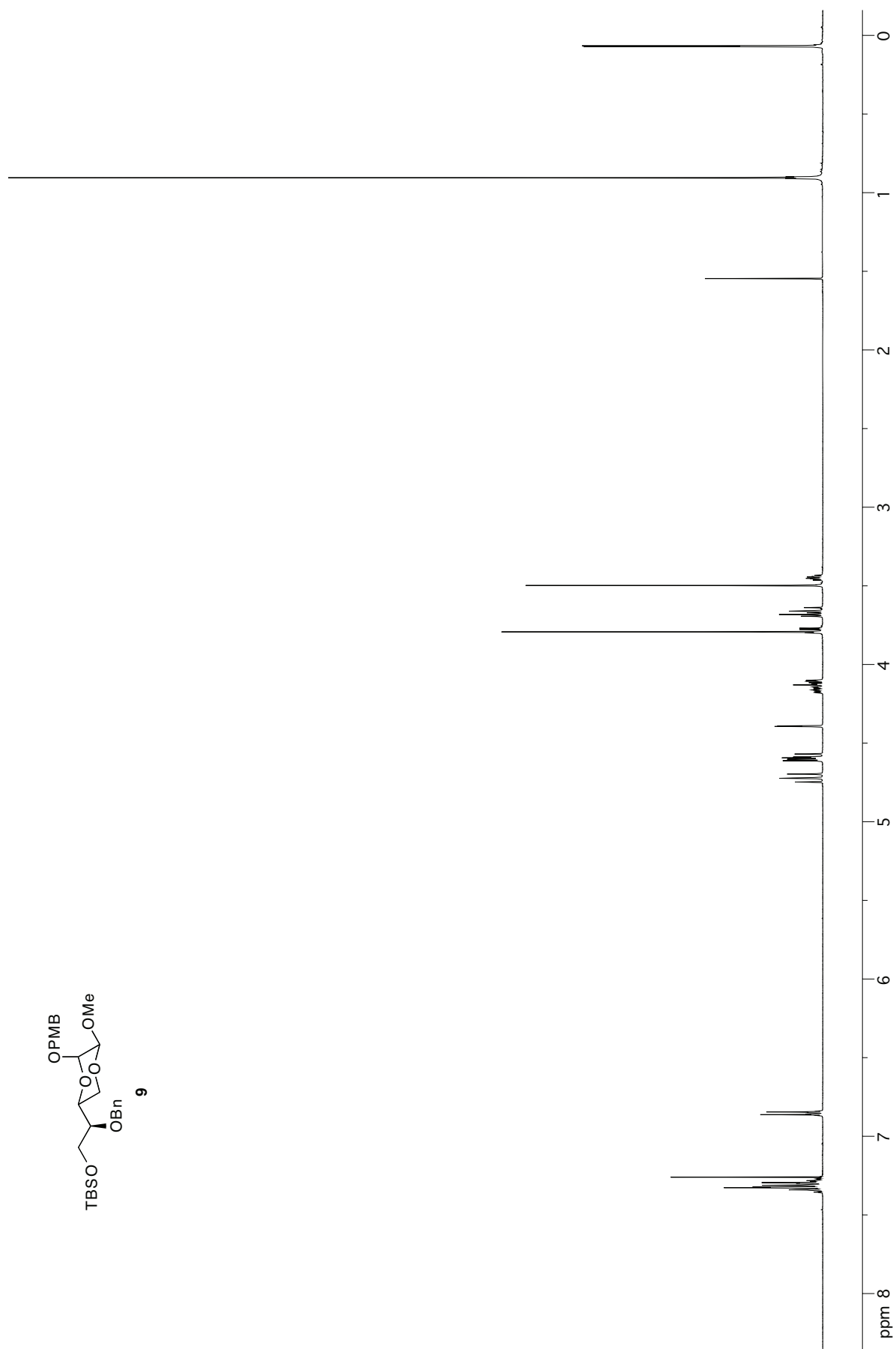
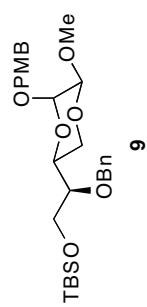
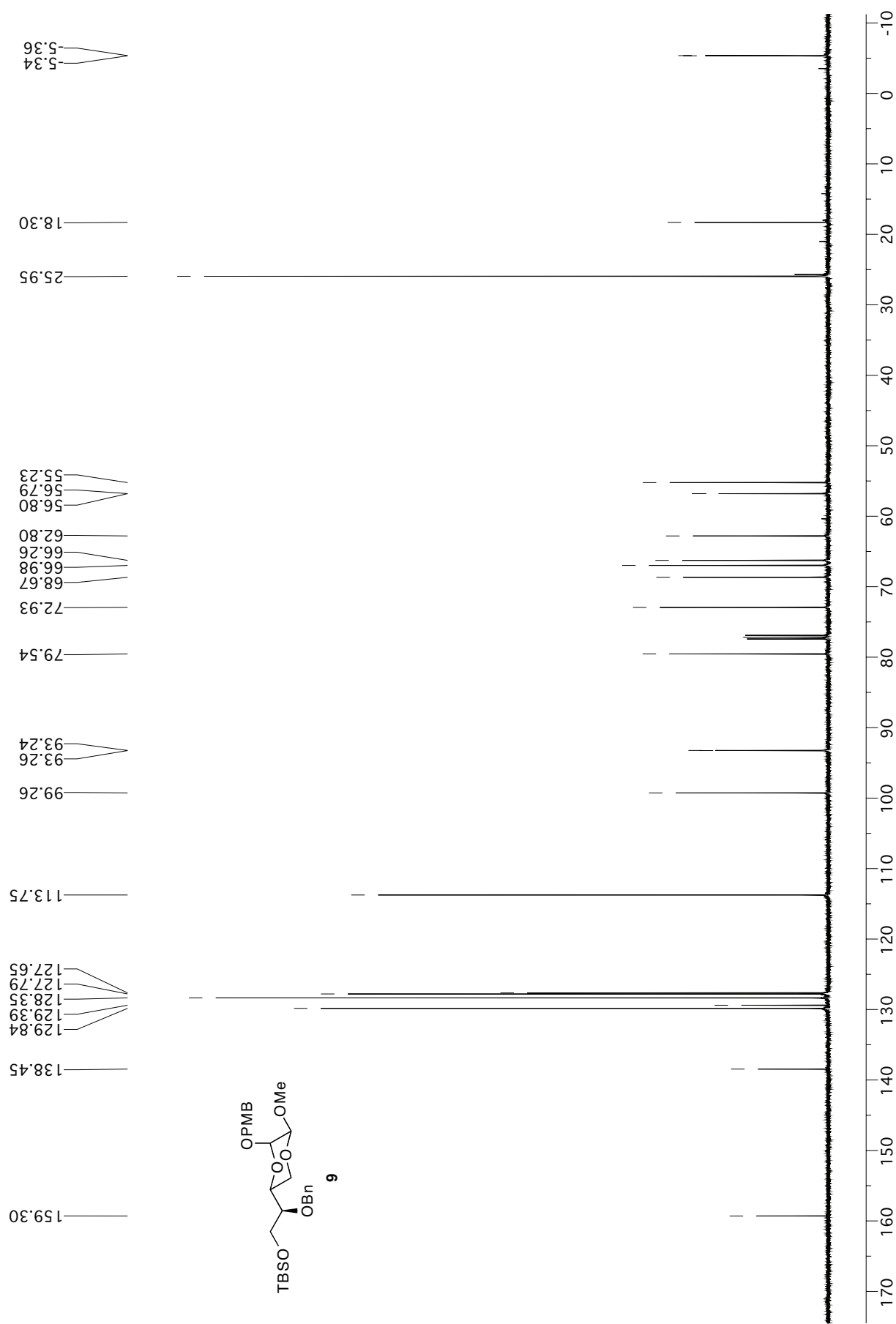


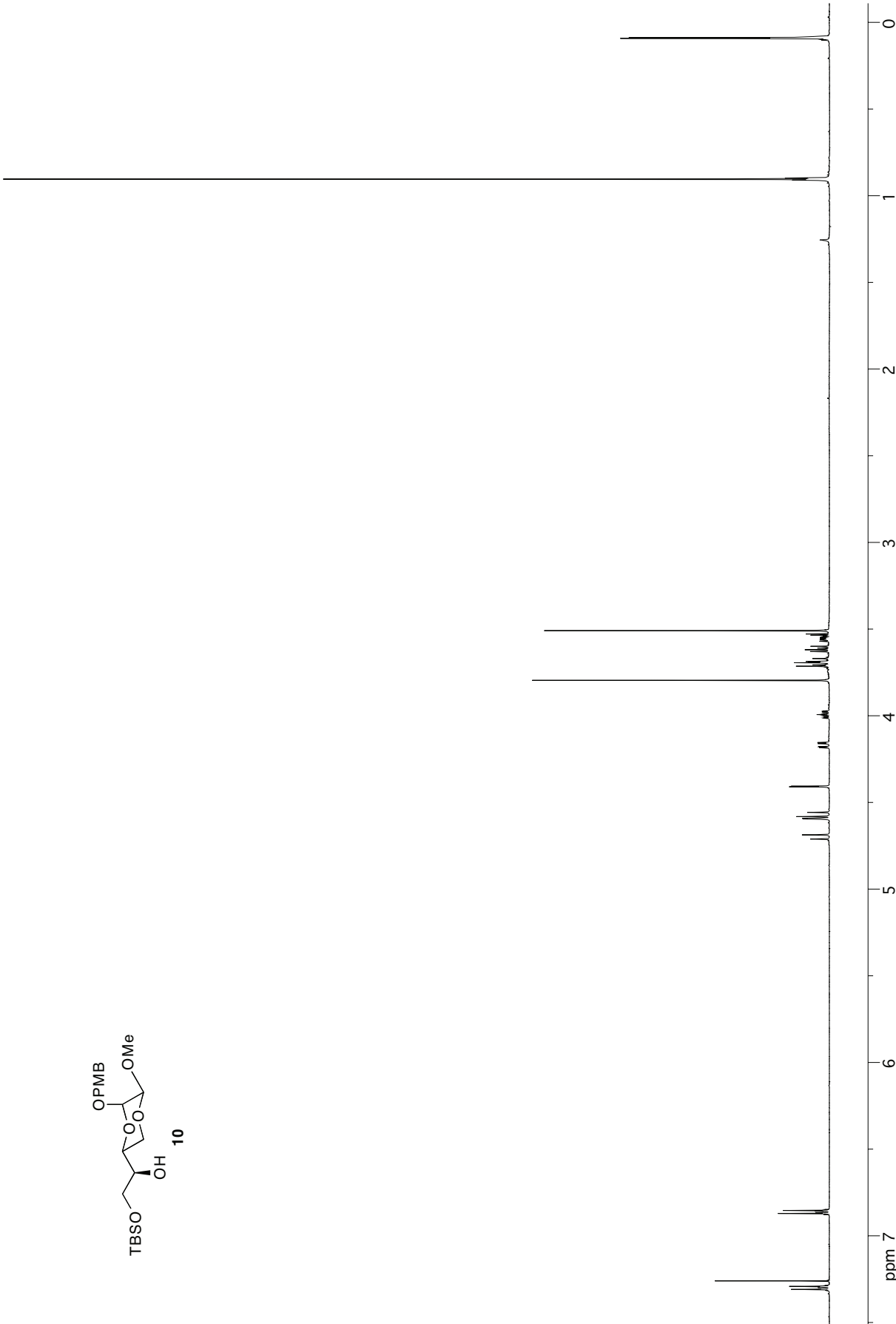
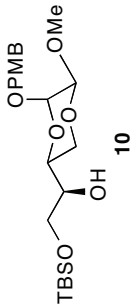
Figure 2: HPLC trace of separation of 2''', 5'''-diepisilvestrol (**4**) ($R_t = 9.33$ min) and 1''', 2''', 5'''-triepisilvestrol (**15**) ($R_t = 10.38$ min). 5 μ m Phenomenex Lux Cellulose-1 semi-preparative column: 10x250mm, 70% MeCN/H₂O eluent, flow rate: 2.00 mL/min.

^1H NMR (500 MHz, CDCl_3)

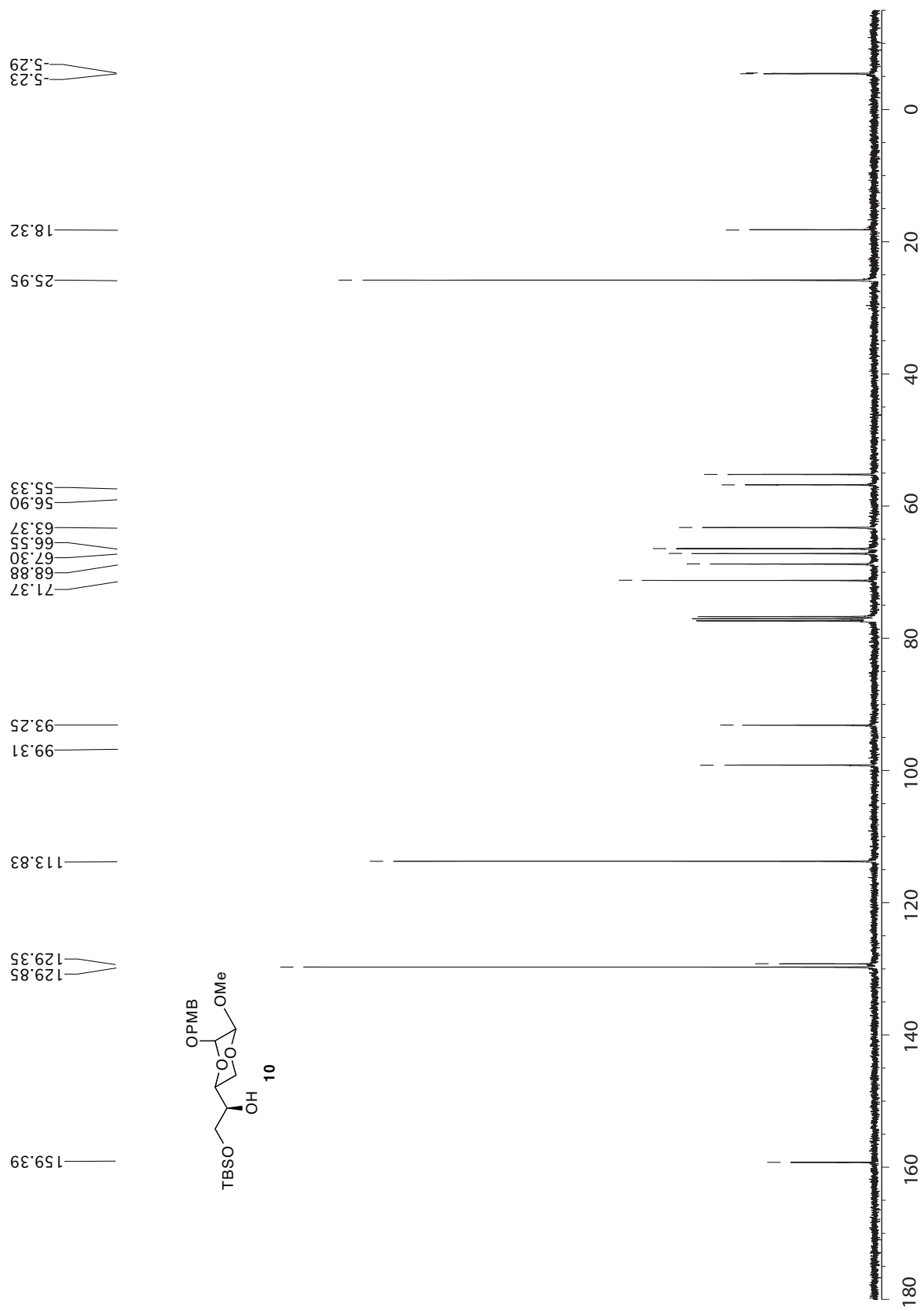


¹³C NMR (125 MHz, CDCl₃)

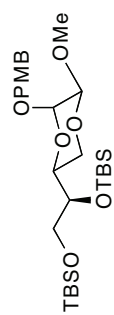


¹H NMR (500 MHz, CDCl₃)

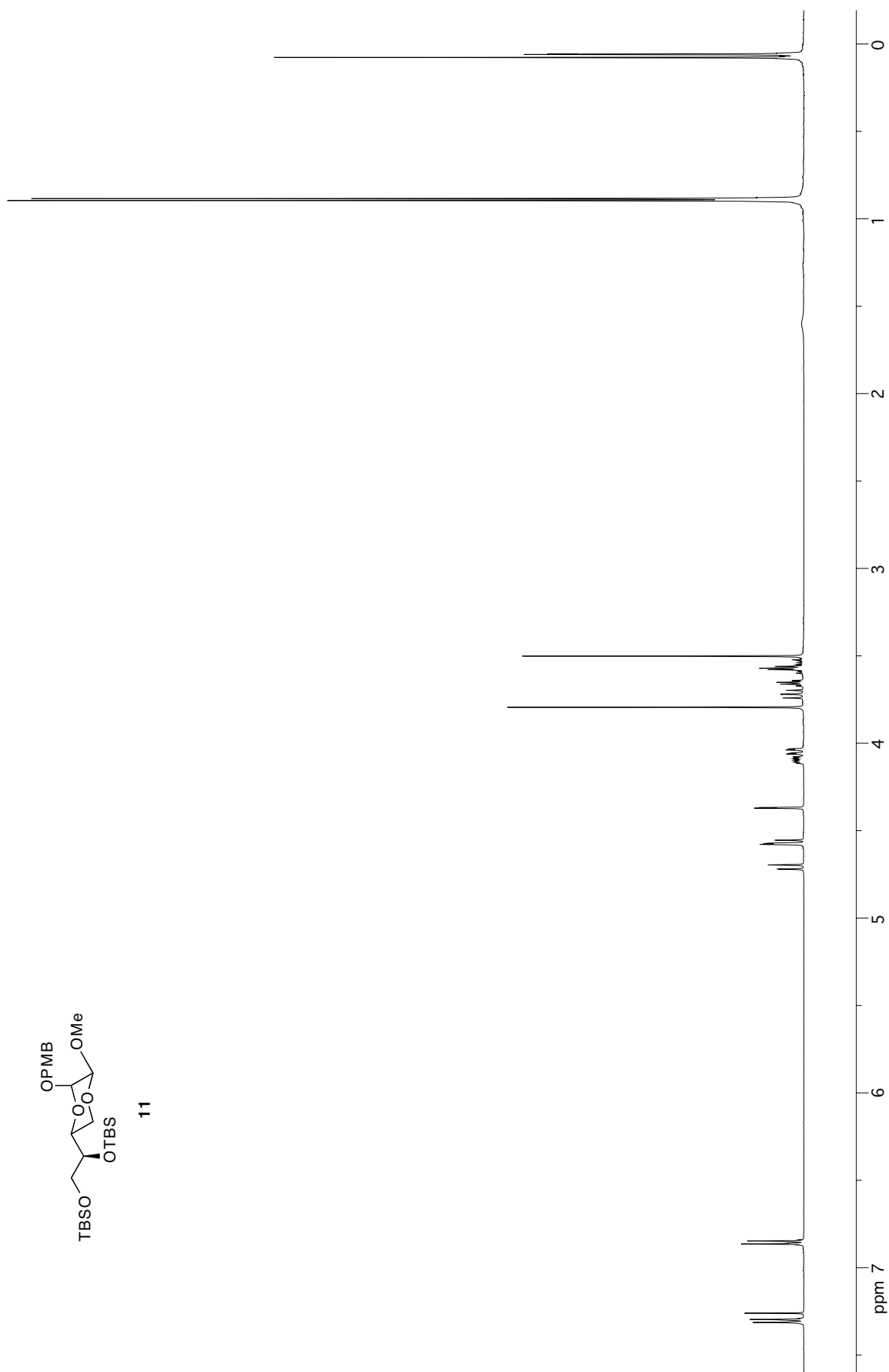
^{13}C NMR (100 MHz, CDCl_3)



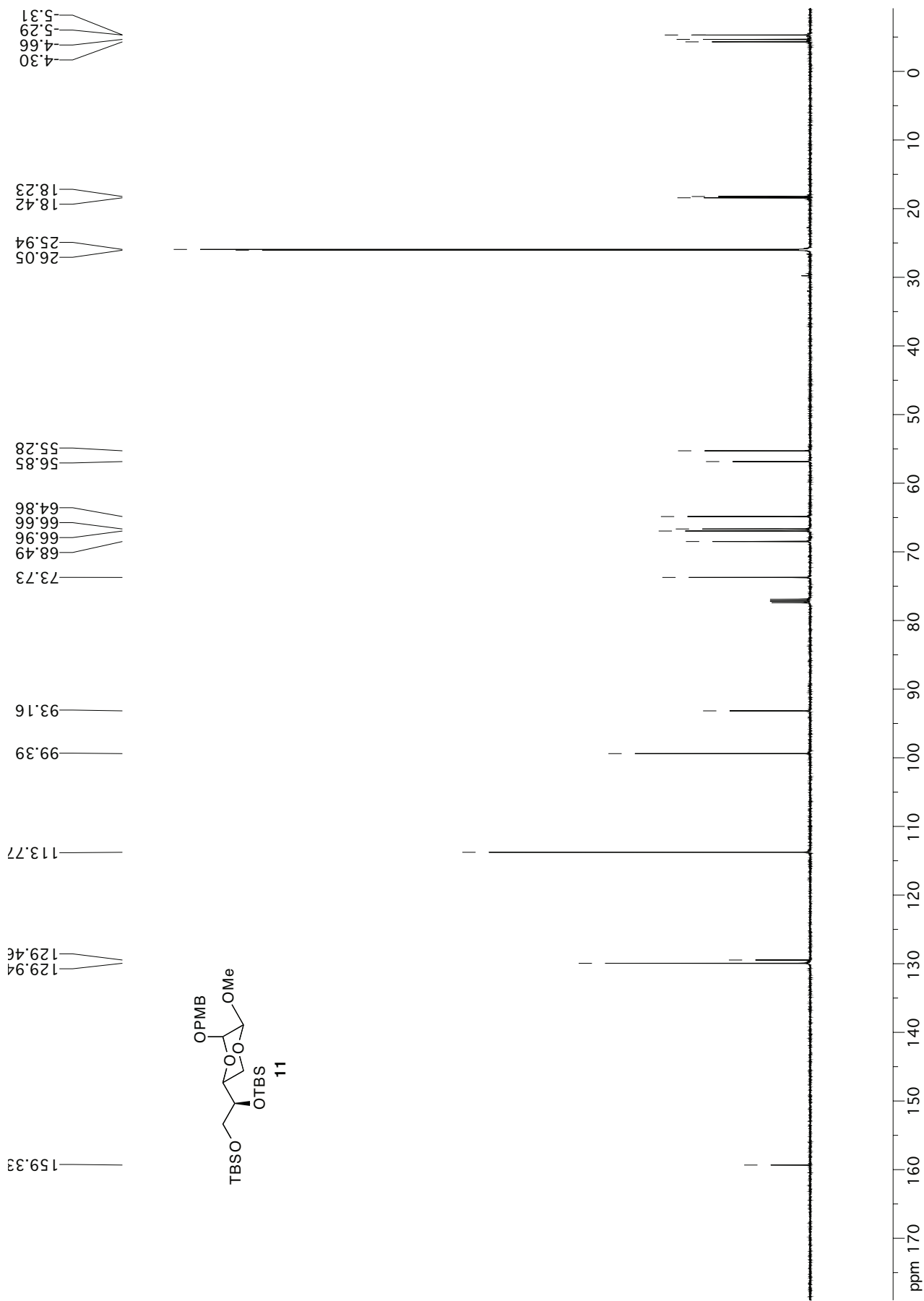
^1H NMR (500 MHz, CDCl_3)



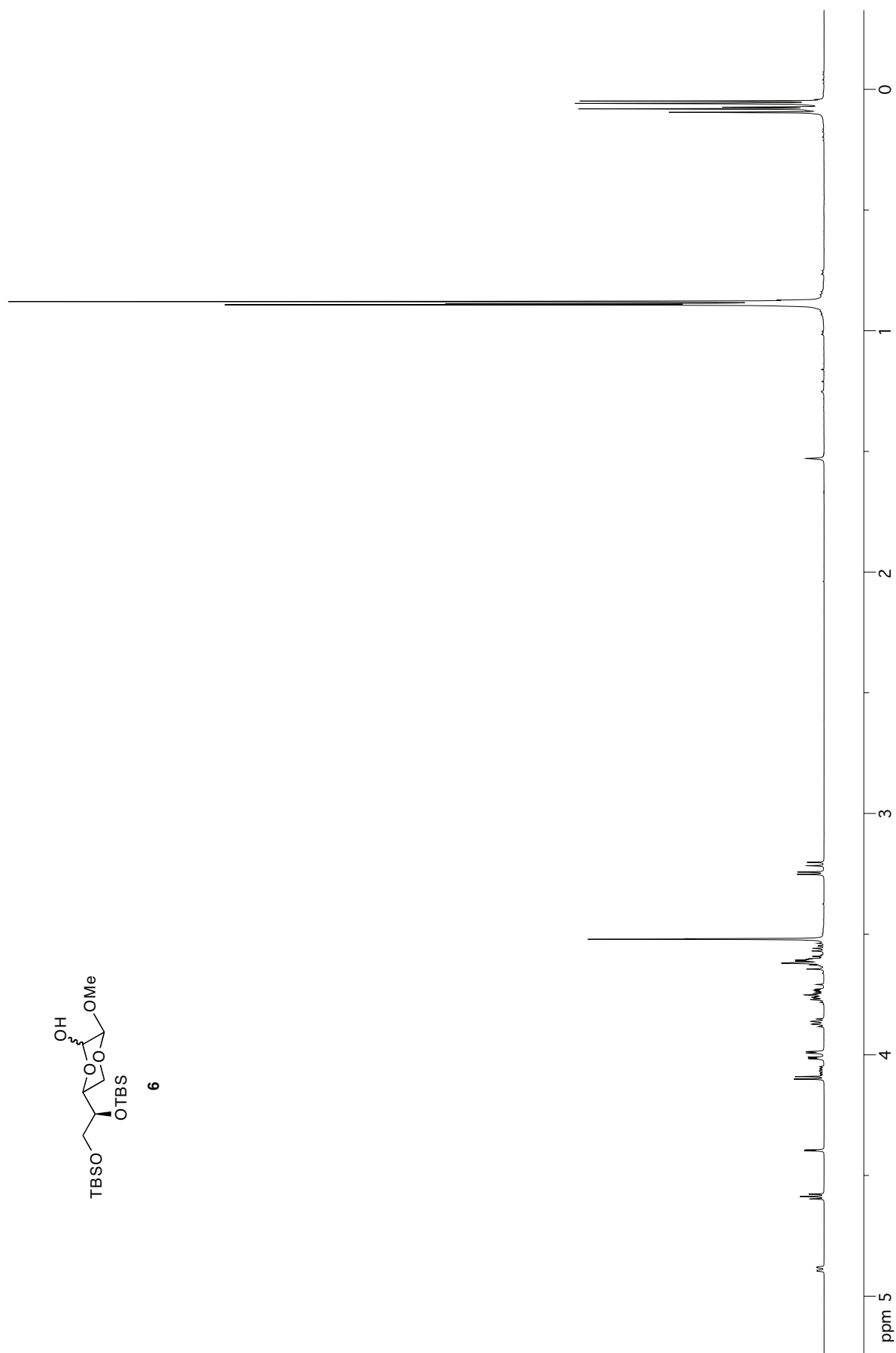
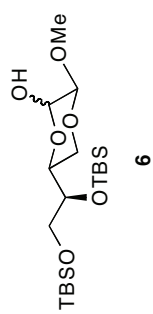
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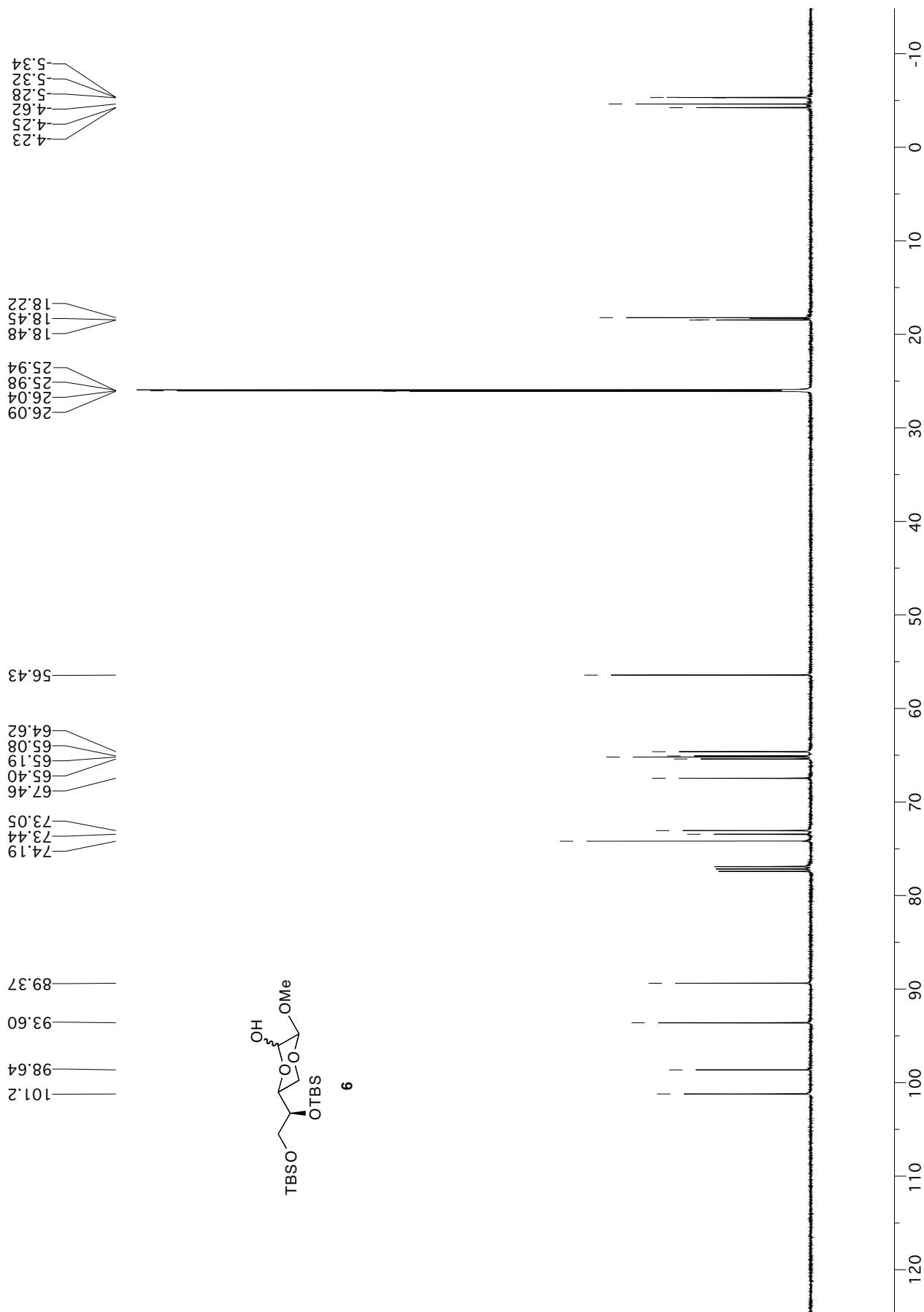
^{13}C NMR (125 MHz, CDCl_3)



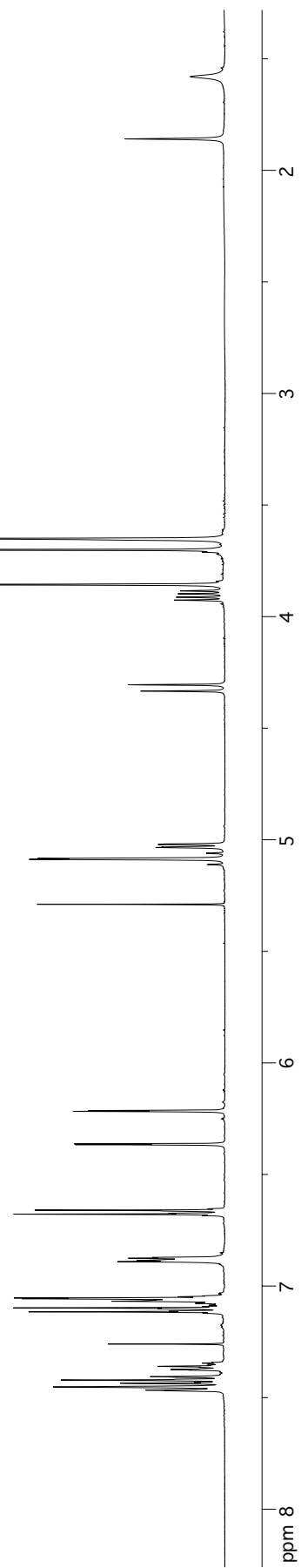
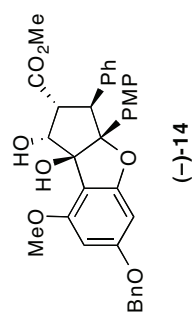
^1H NMR (500 MHz, CDCl_3)



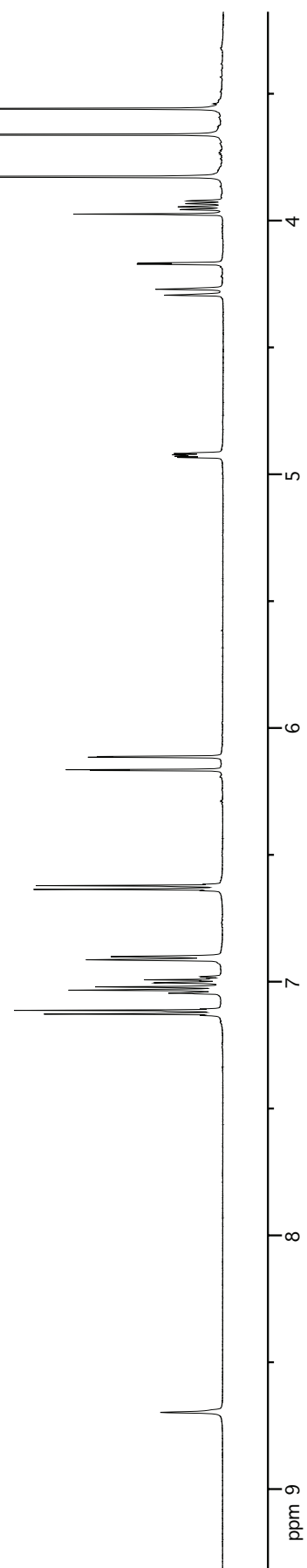
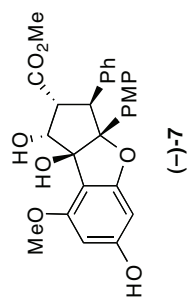
^{13}C NMR (125 MHz, CDCl_3)



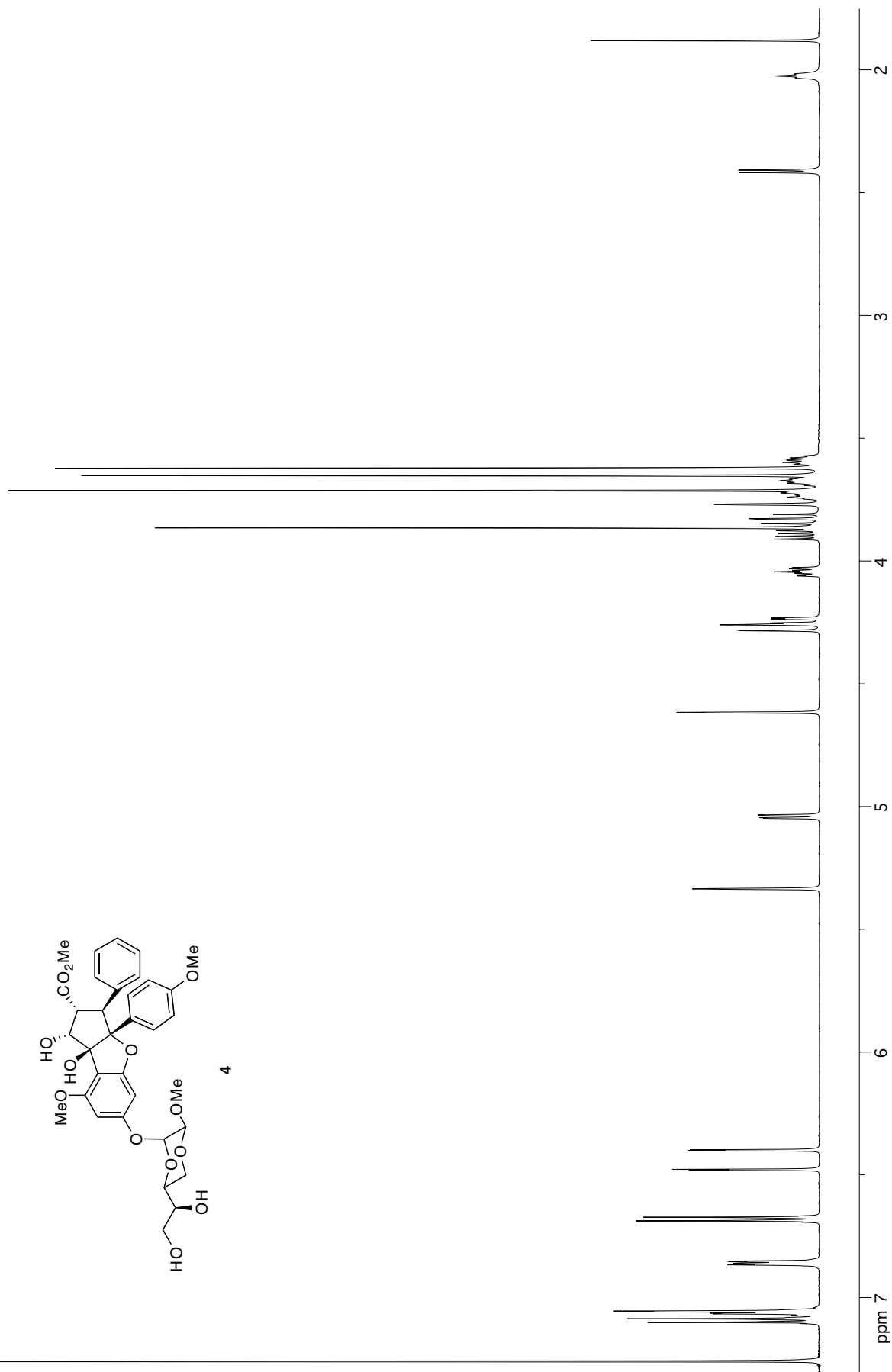
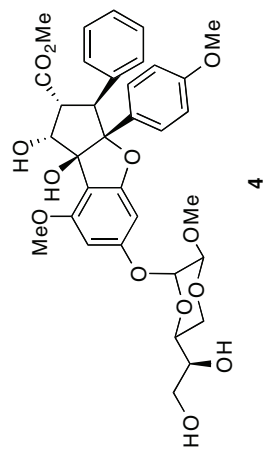
^1H NMR (500 MHz, CDCl_3)



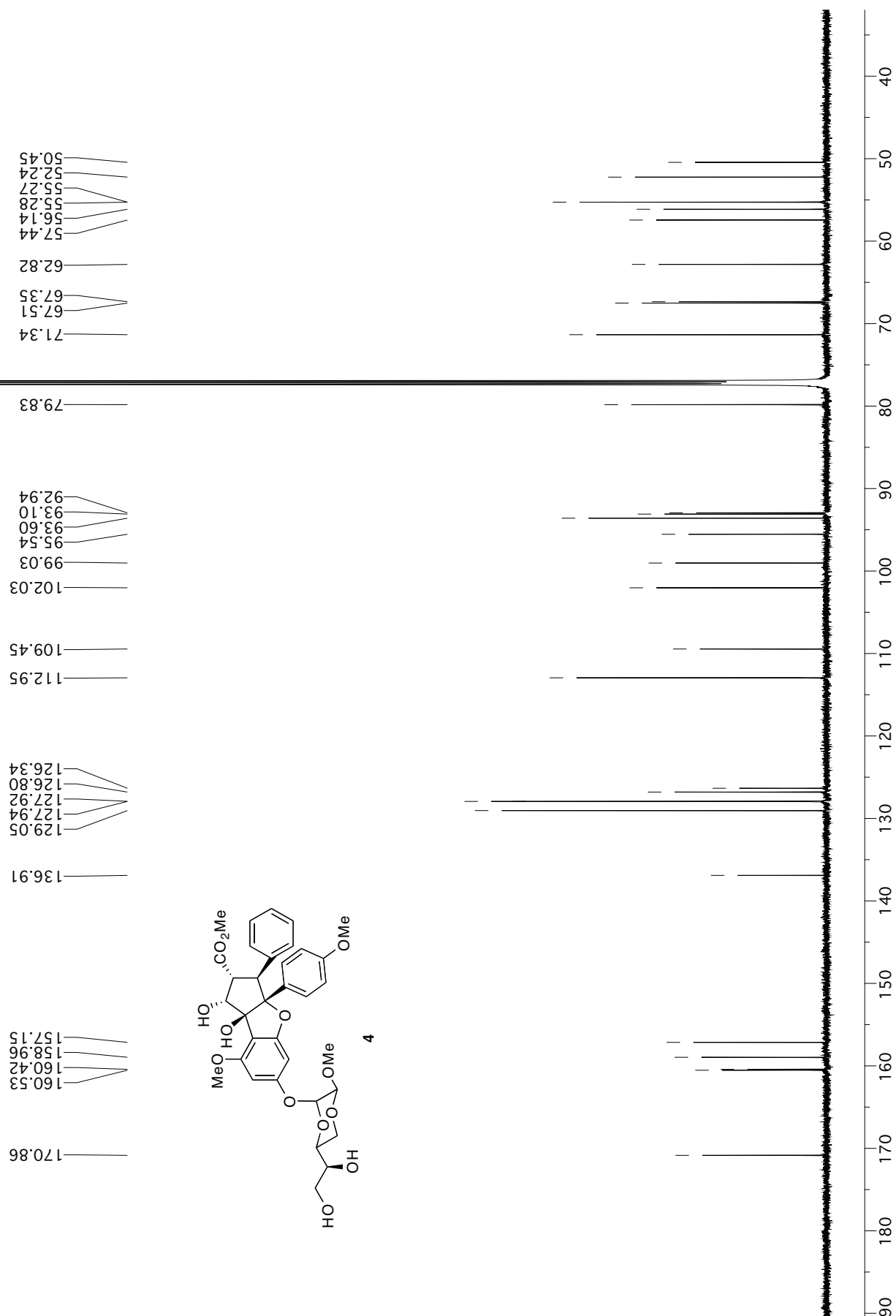
^1H NMR (500 MHz, CDCl_3)



¹H NMR (600 MHz, CDCl₃)



¹³C NMR (150 MHz, CDCl₃)



¹³C NMR (150 MHz, CDCl₃)

