

Supporting Information for

**The *o*-xylylene protecting group in carbohydrate chemistry:
application to the regioselective protection of a single *vic*-diol segment
in cyclodextrins**

**Patricia Balbuena,^a Rita Gonçalves-Pereira,^a José L. Jiménez Blanco,^{b,*} M. Isabel García-Moreno,^b
David Lesur,^c Carmen Ortiz Mellet,^b and José M. García Fernández^{a,*}**

List of Contents:

**.- S2 to S46 Figures S1 to S45 Showing the NMR Spectra of Compounds 6-8, 10-12,
14, 17-20, 23-27, 29, 31-33, 35-40, 44, 45, 49, 50, 41-43, 46-48 and 51-53.**

**.- S47 Figures S46 to S48 Showing the HPLC Chromatograms for Compounds
38-40, 44-45, 49, and 50.**

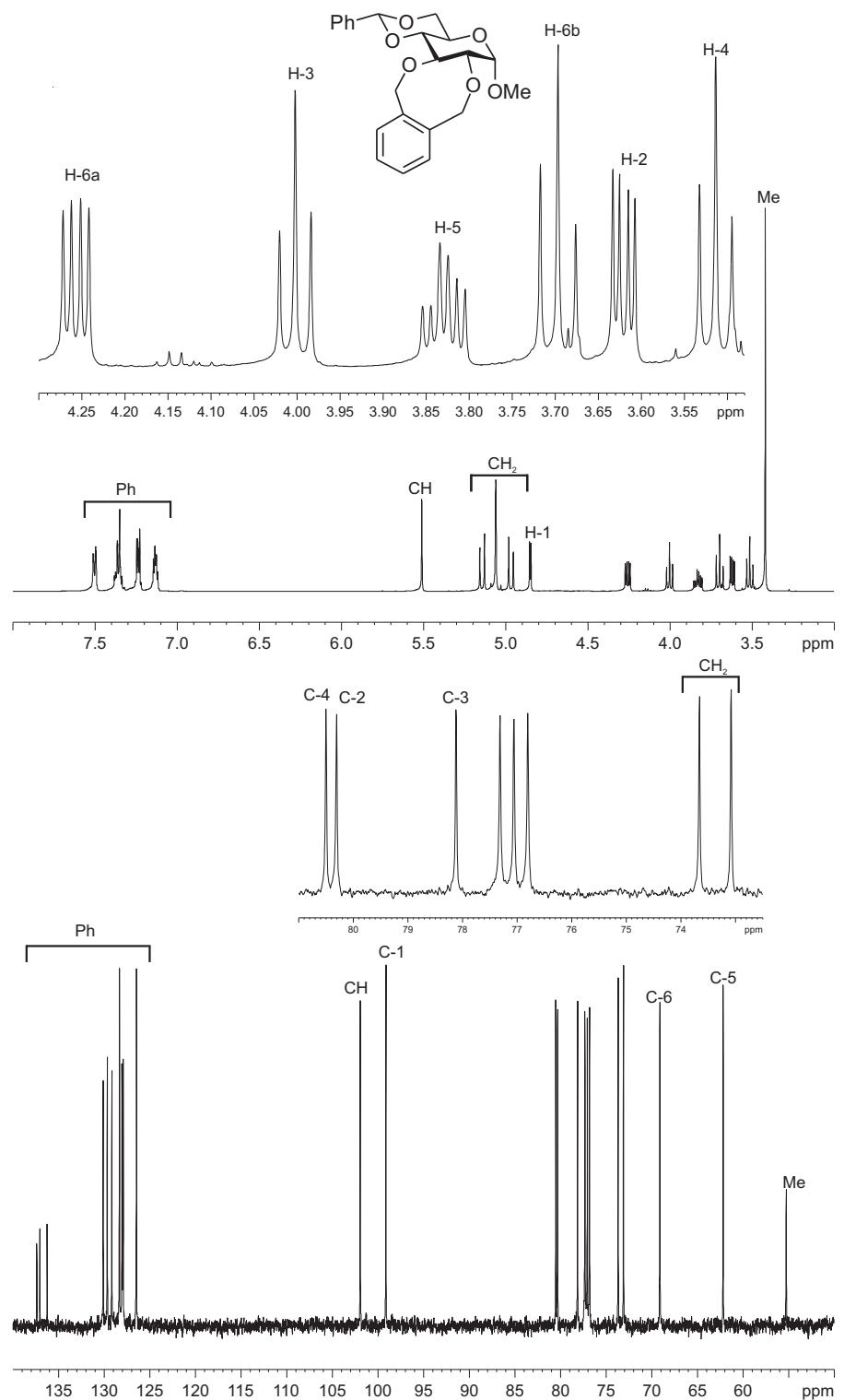


Figure S1. ^1H and ^{13}C NMR spectra (500 MHz, 125.7 MHz, CDCl_3) of **6**.

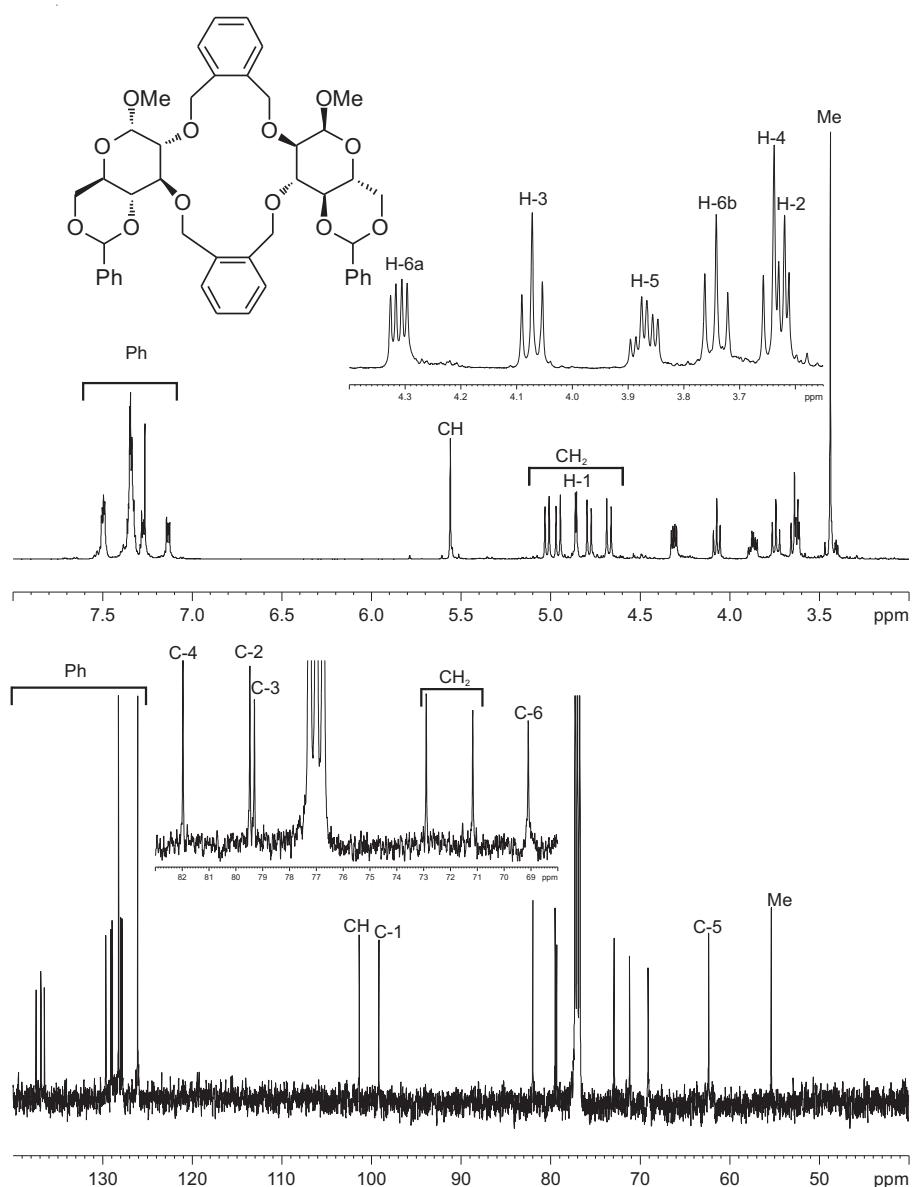


Figure S2. ¹H and ¹³C NMR spectra (500 MHz, 125.7 MHz, CDCl₃) of **7**.

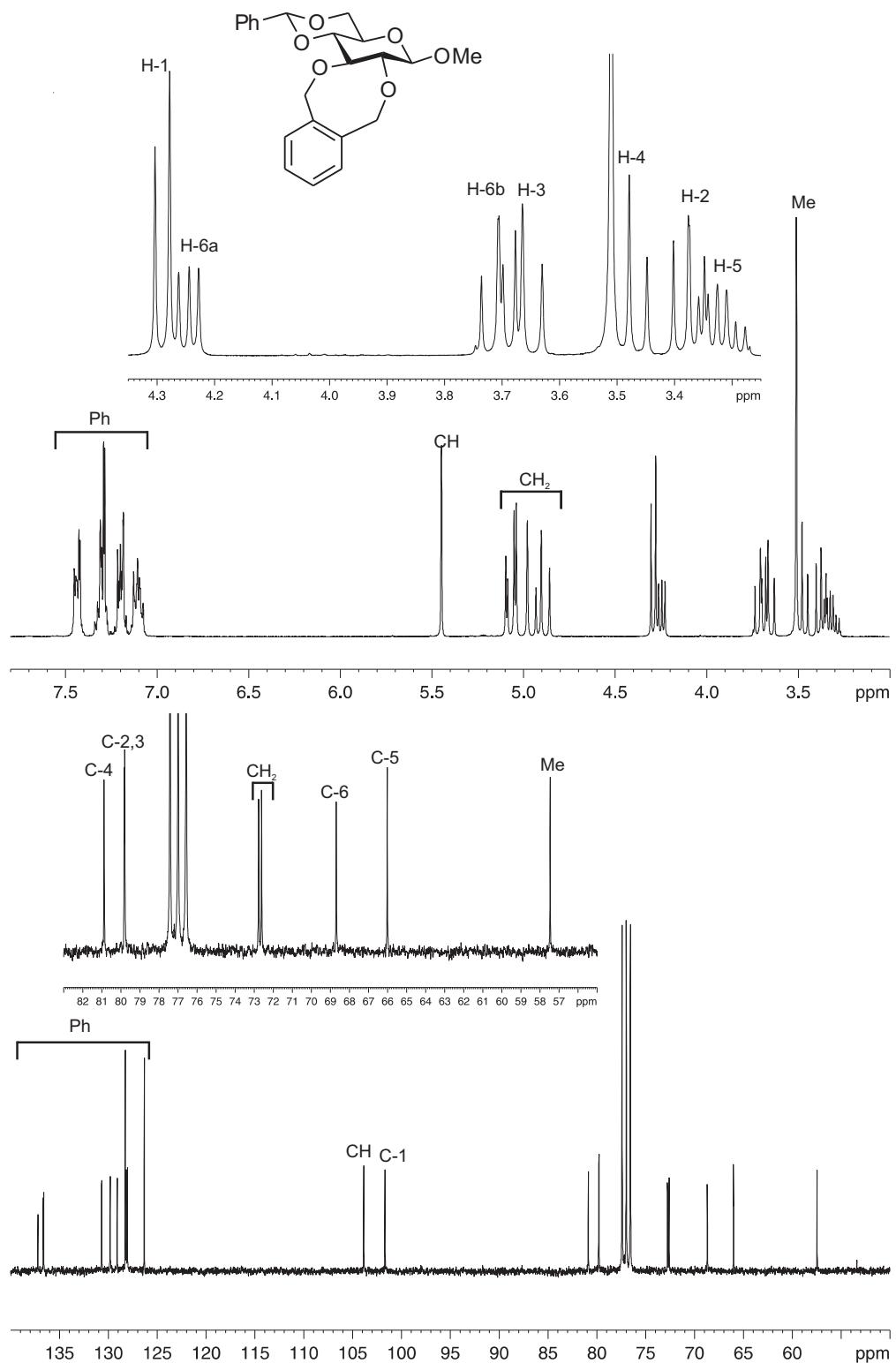


Figure S3. ^1H and ^{13}C NMR spectra (300 MHz, 75.5 MHz, CDCl₃) of **8**.

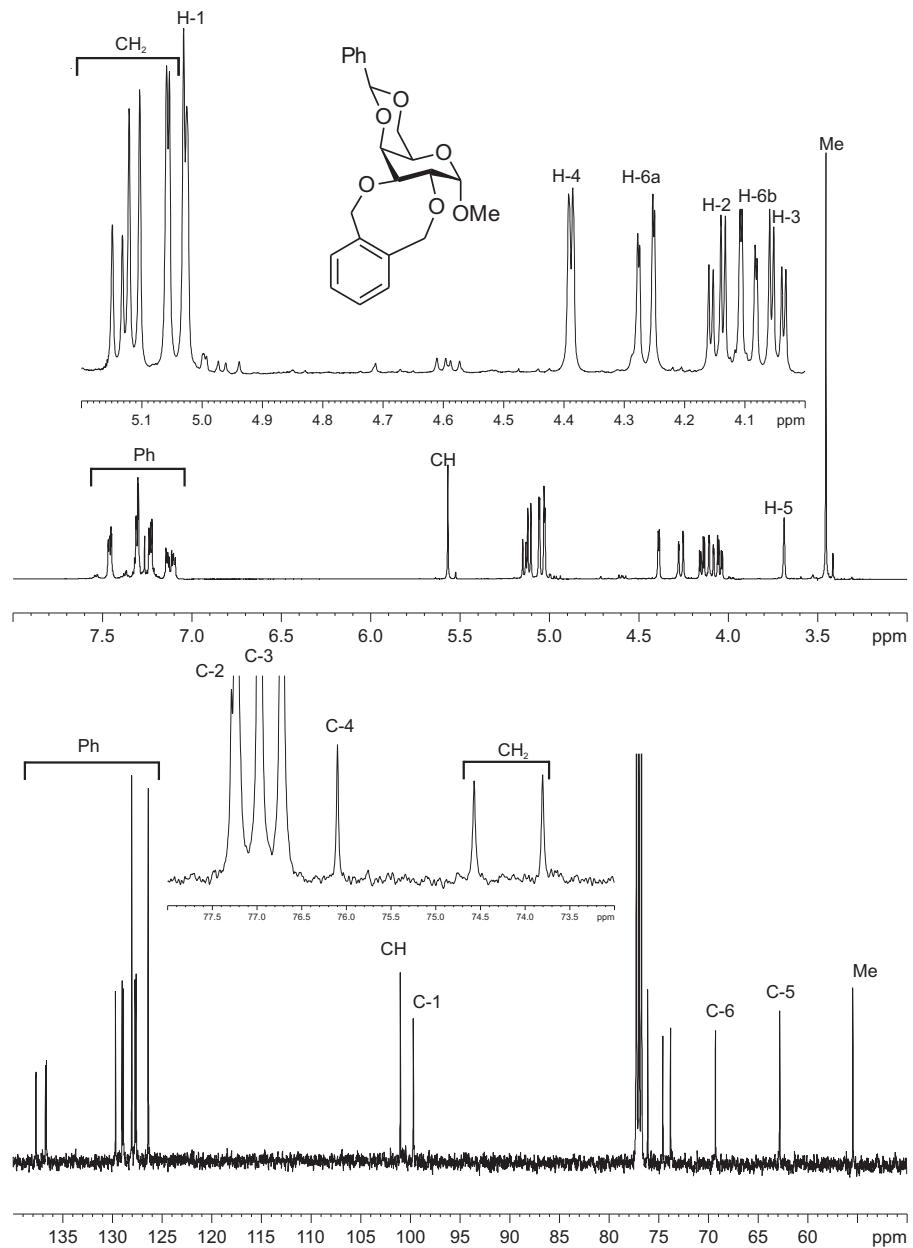


Figure S4. ^1H and ^{13}C NMR spectra (500 MHz, 125.7 MHz, CDCl_3) of **10**.

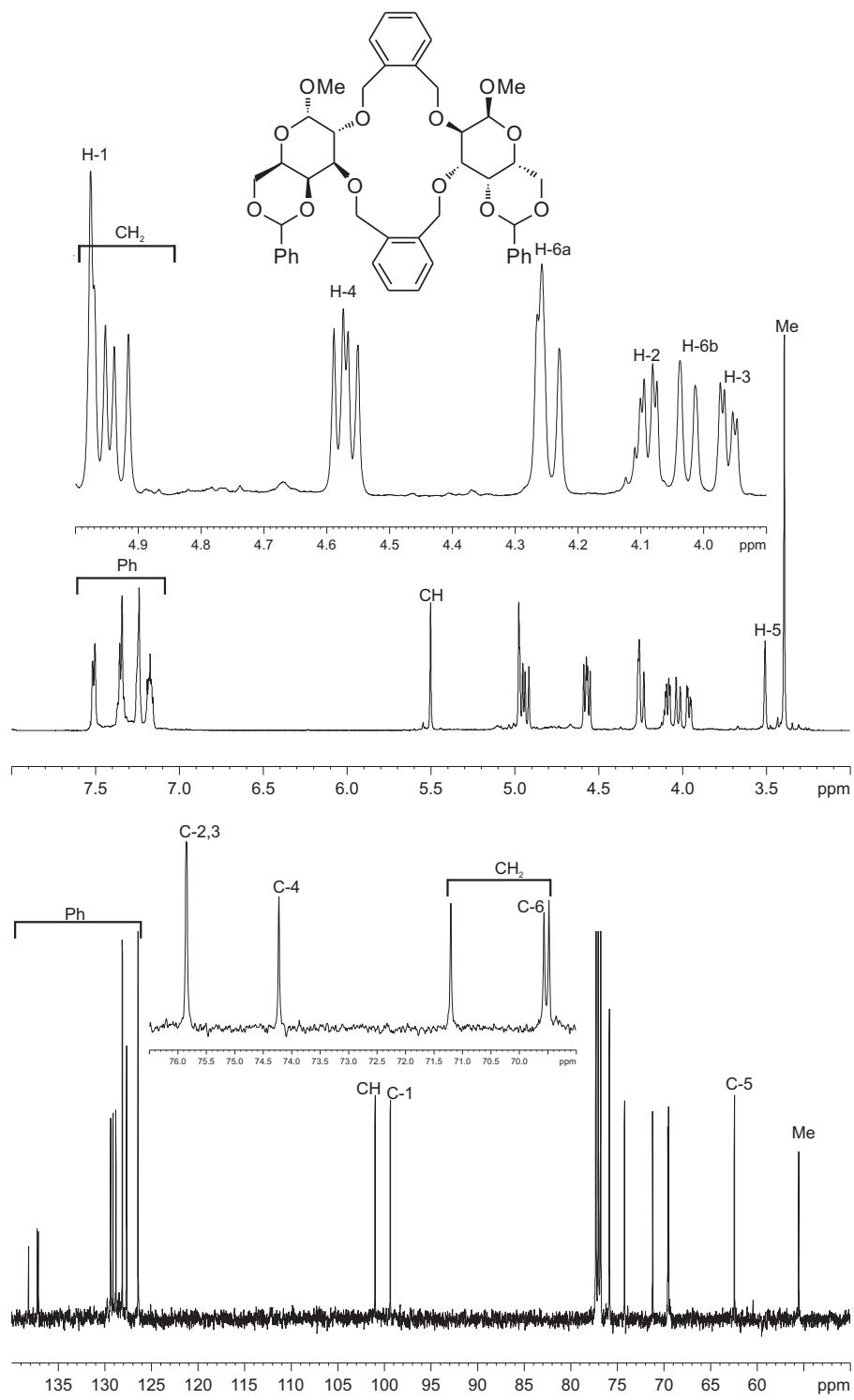


Figure S5. ^1H and ^{13}C NMR spectra (500 MHz, 125.7 MHz, CDCl_3) of **11**.

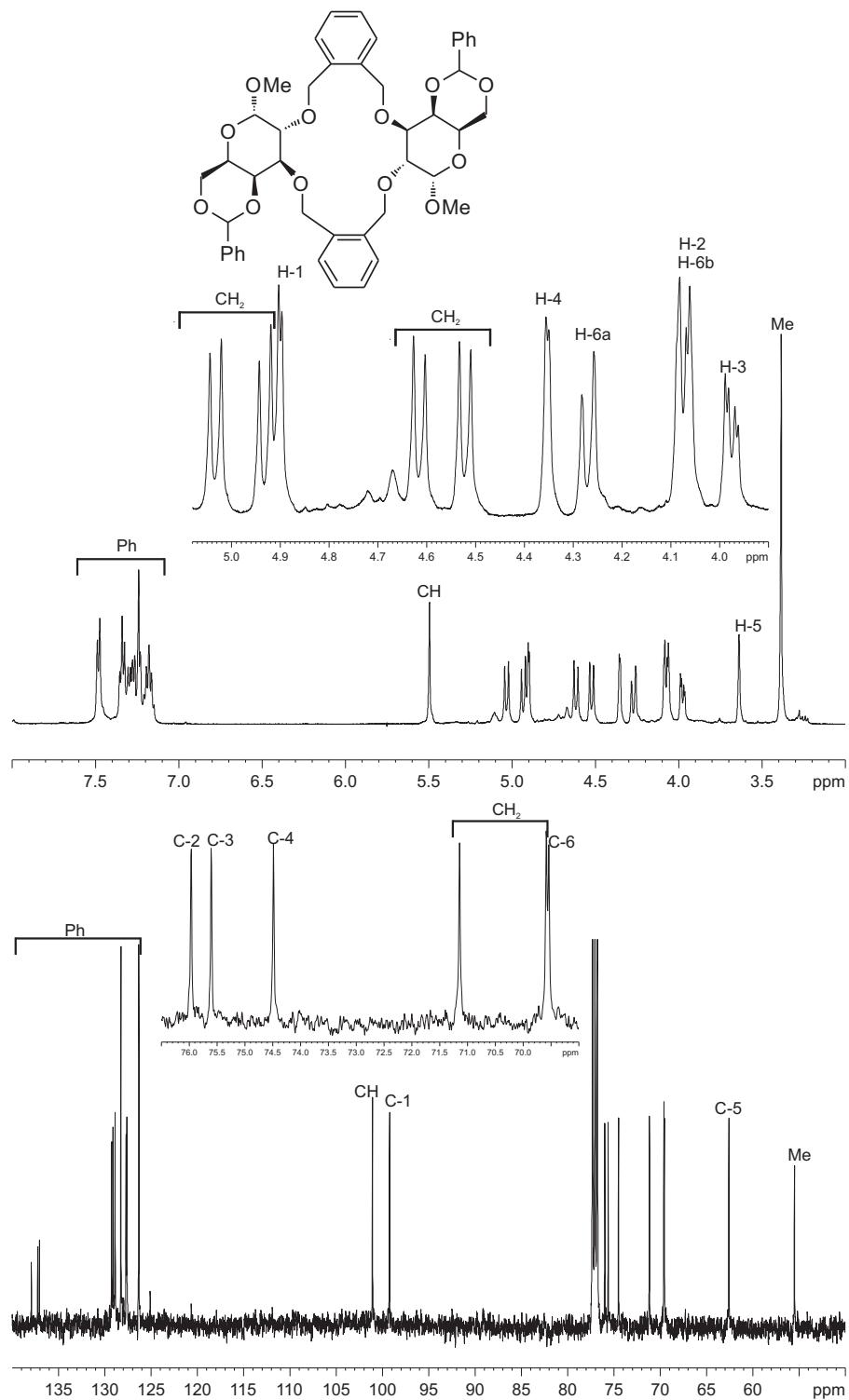


Figure S6. ^1H and ^{13}C NMR spectra (500 MHz, 125.7 MHz, CDCl_3) of **12**.

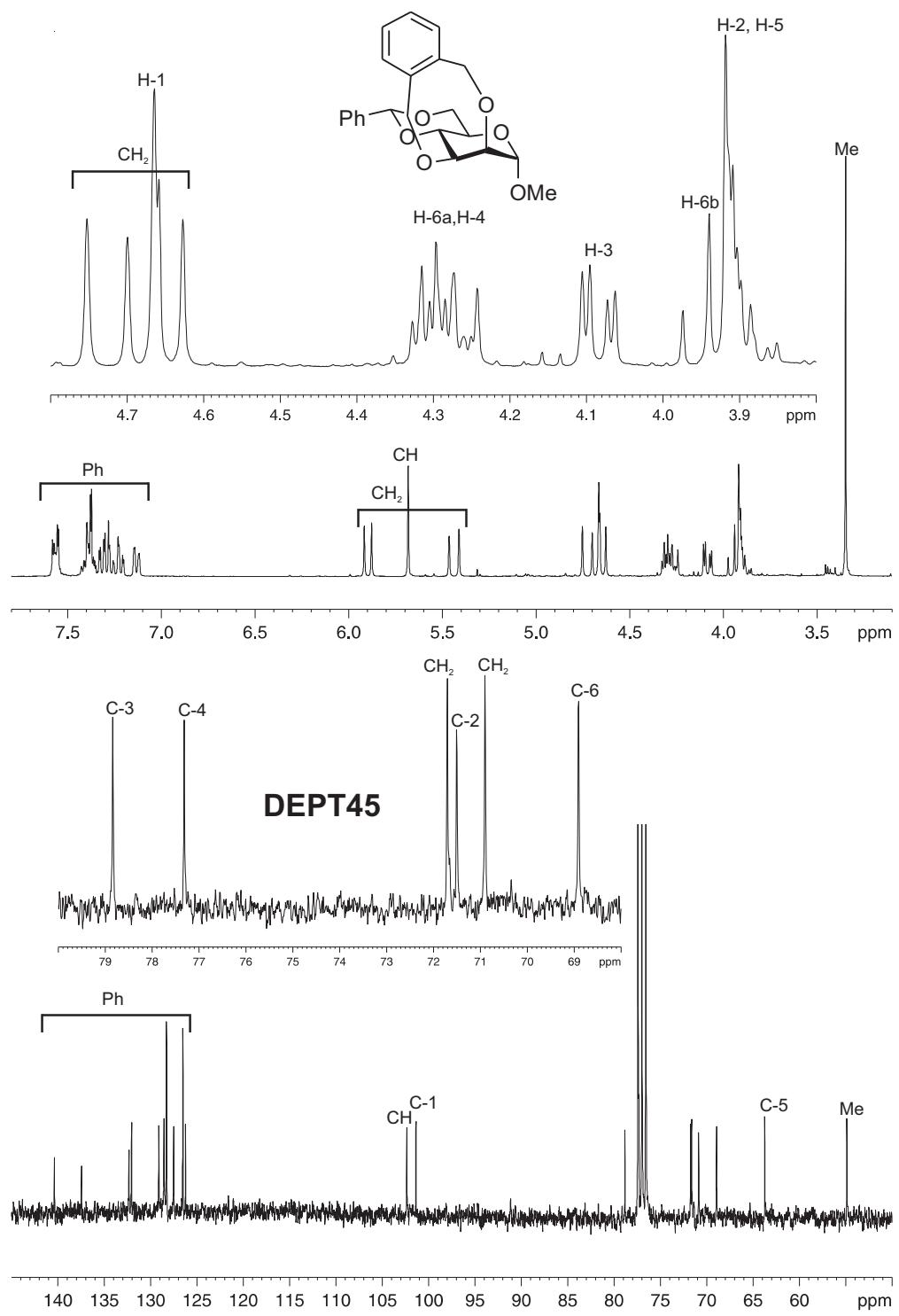


Figure S7. ^1H , ^{13}C -decoupled and DEPT45 NMR spectra (300 MHz, 75.5 MHz, CDCl₃) of **14**.

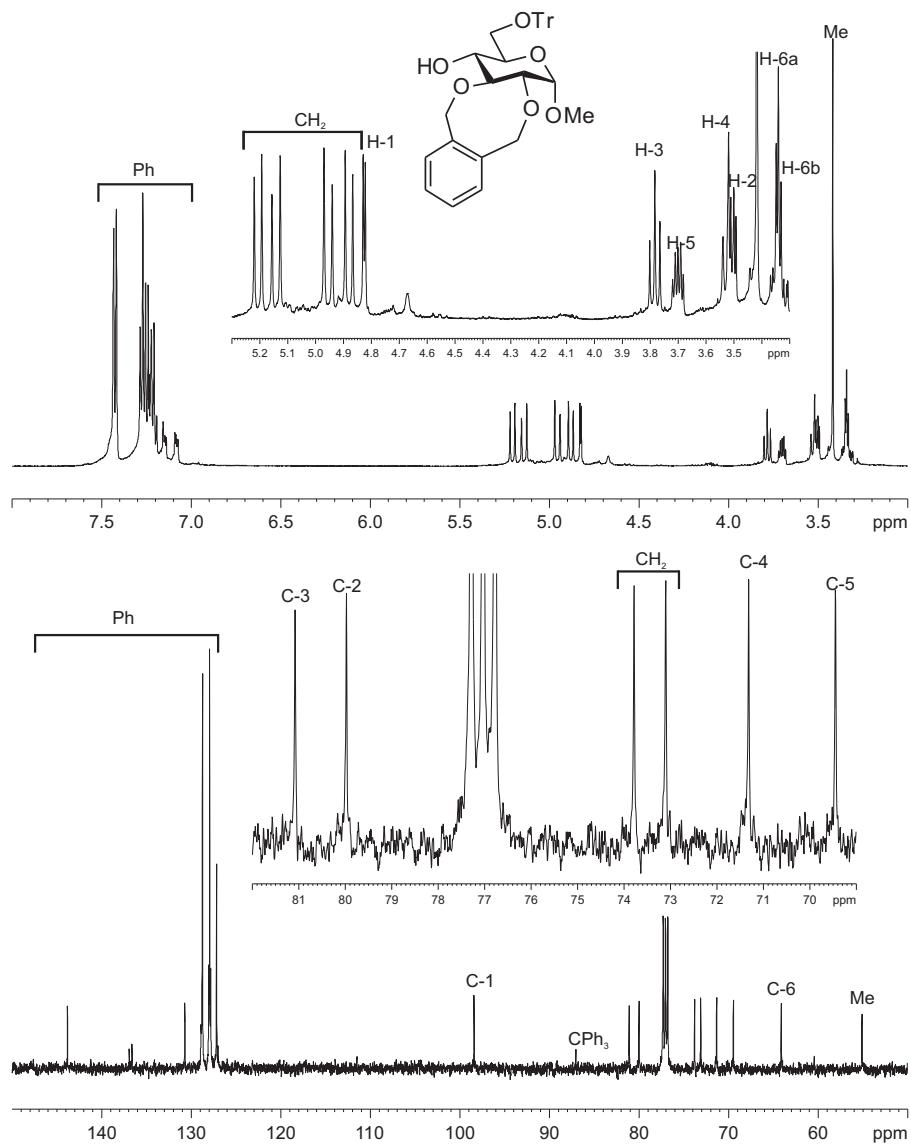


Figure S8. ^1H and ^{13}C NMR spectra (500 MHz, 125.7 MHz, CDCl_3) of **17**.

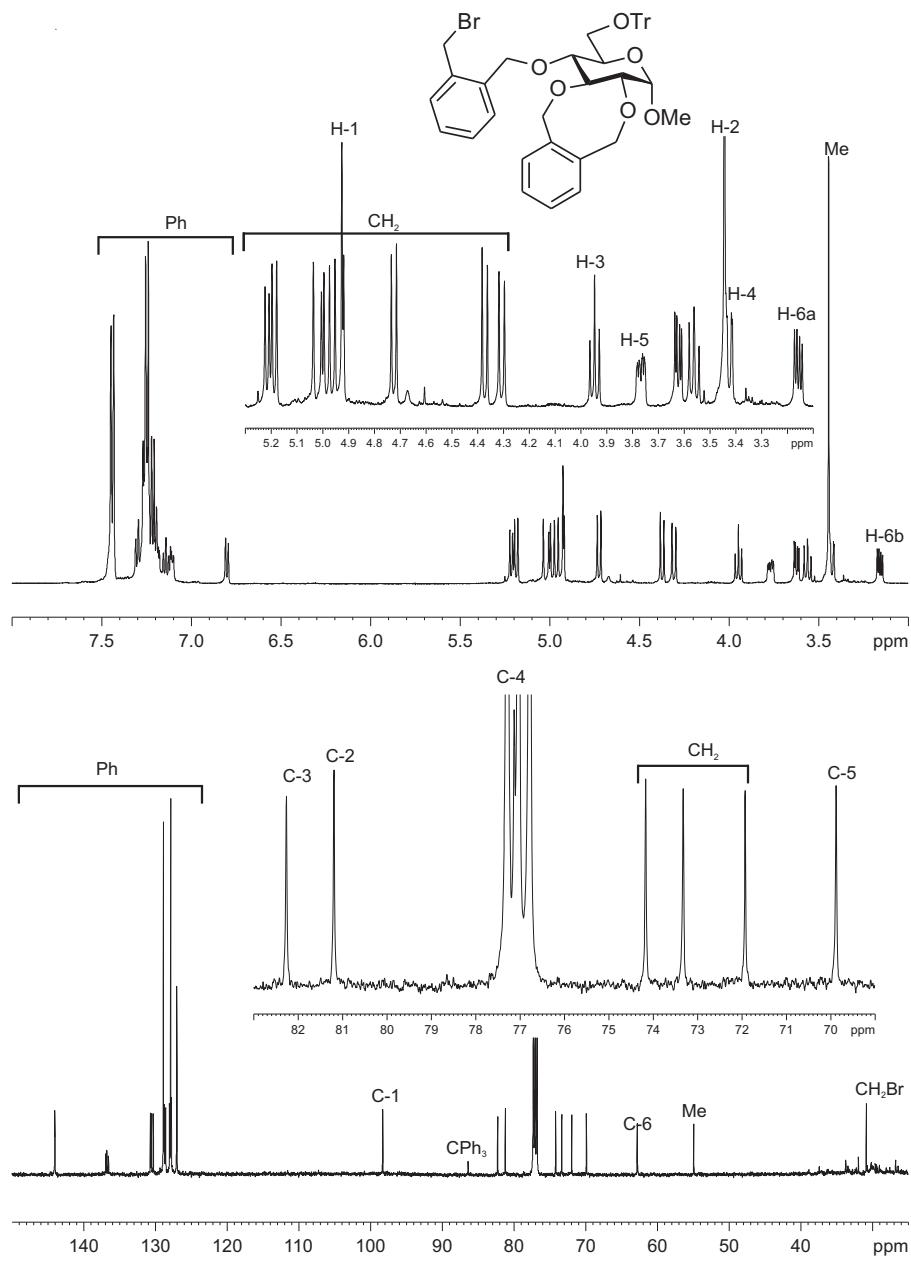


Figure S9. ^1H and ^{13}C NMR spectra (500 MHz, 125.7 MHz, CDCl_3) of **18**.

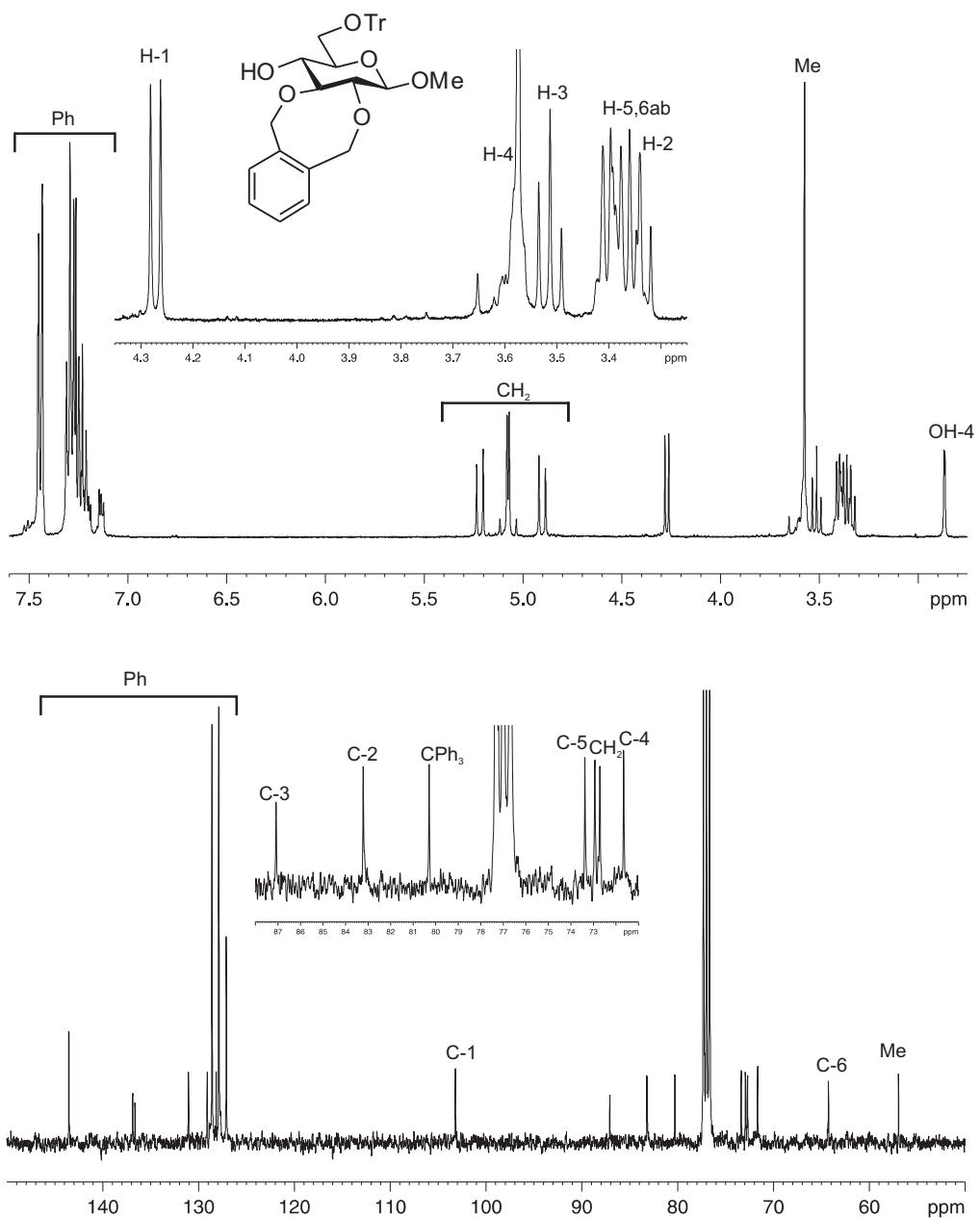


Figure S10. ^1H and ^{13}C NMR spectra (400 MHz, 100.6 MHz, CDCl_3) of **19**.

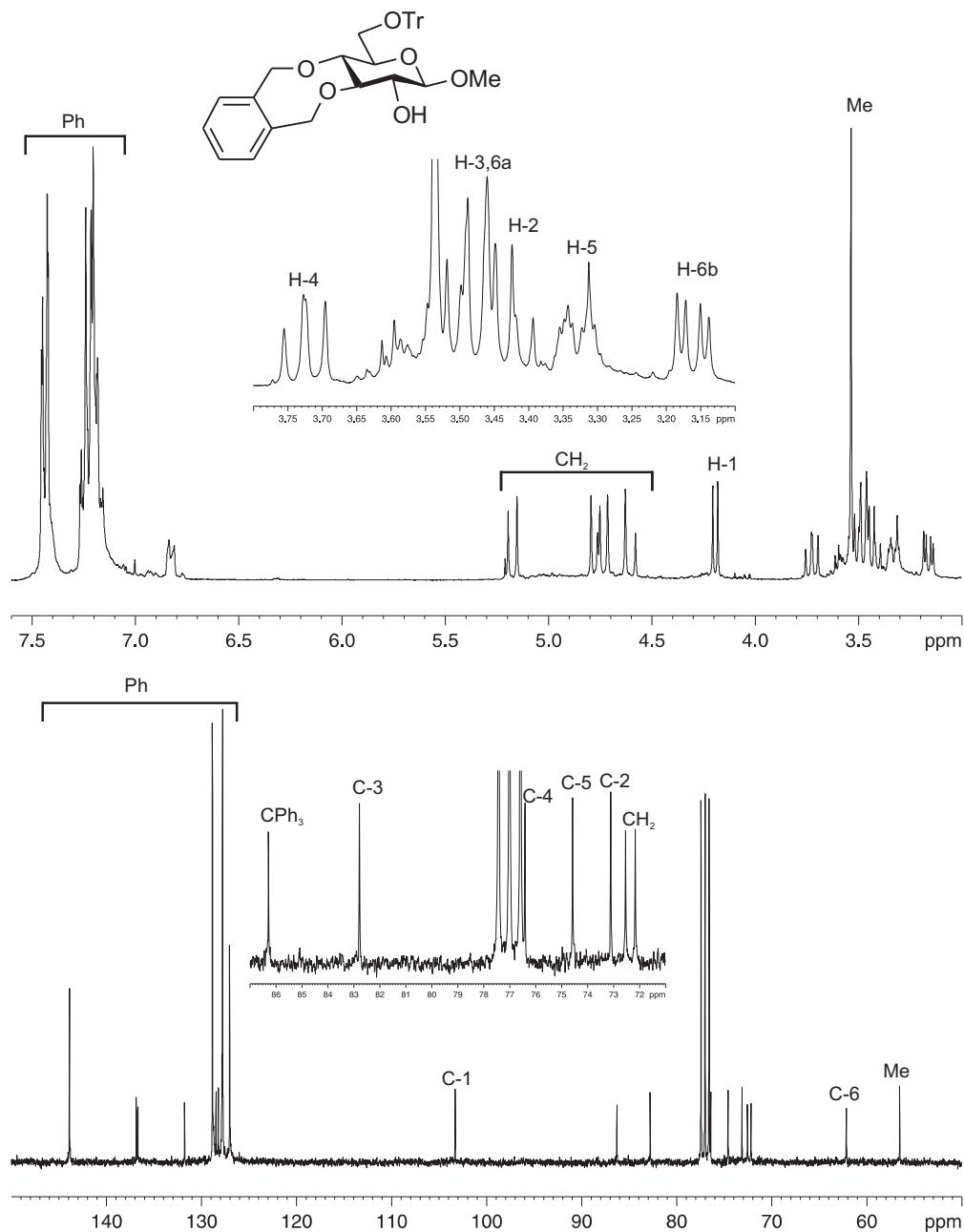


Figure S11. ¹H and ¹³C NMR spectra (300 MHz, 75.5 MHz, CDCl₃) of **20**.

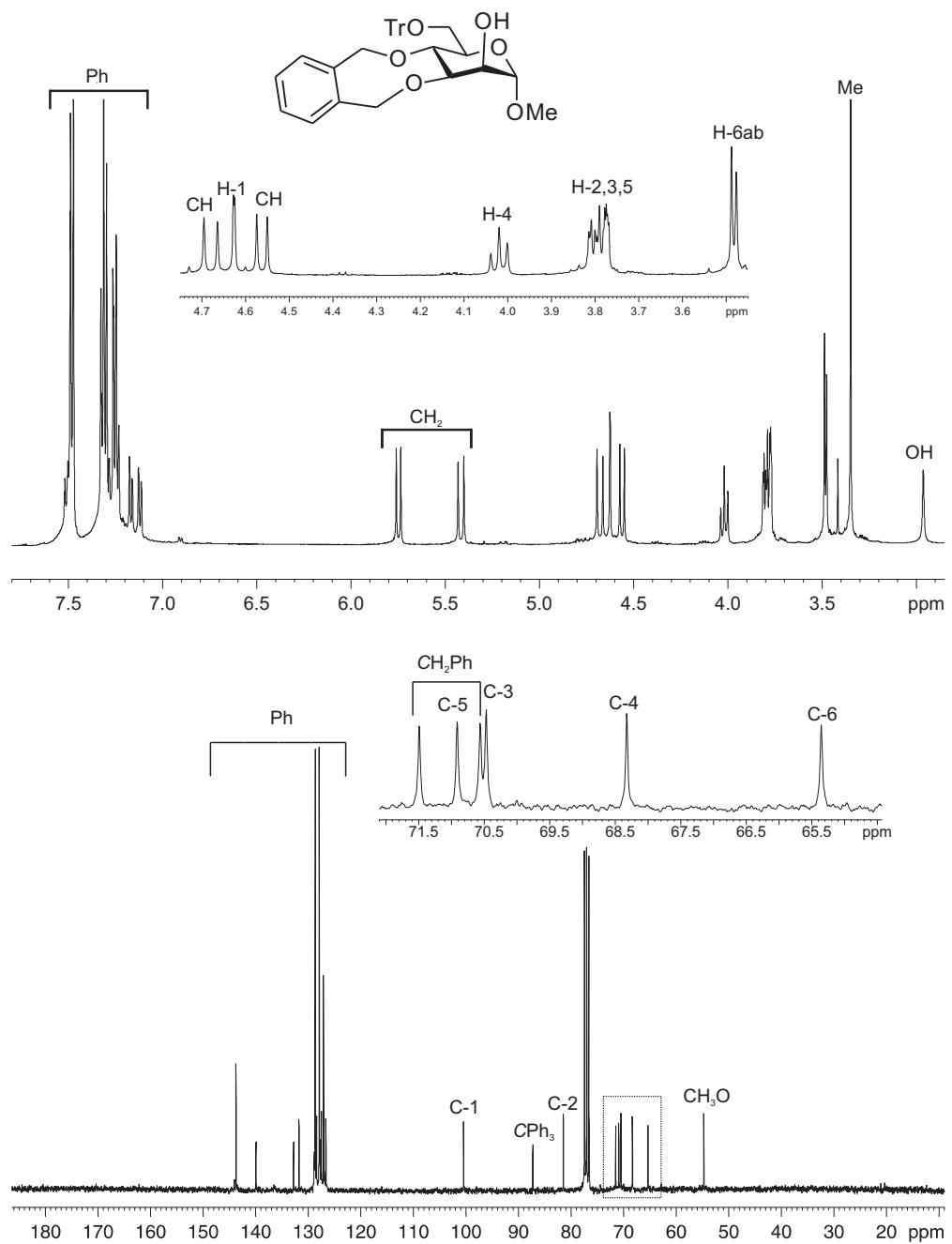


Figure S12. ¹H and ¹³C NMR spectra (500 MHz, 125.7 MHz, CDCl₃) of **23**.

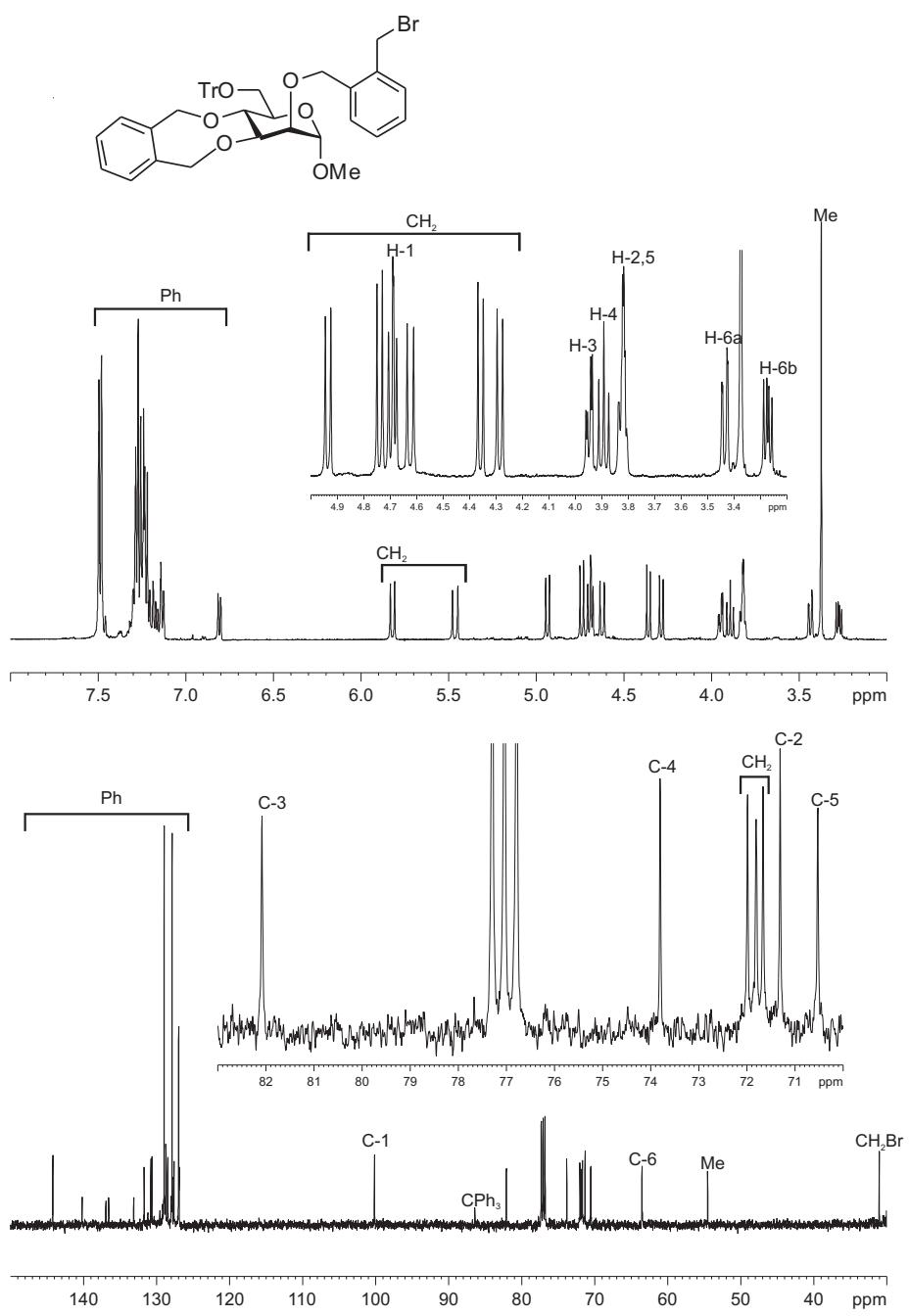


Figure S13. ¹H and ¹³C NMR spectra (500 MHz, 125.7 MHz, CDCl₃) of **24**.

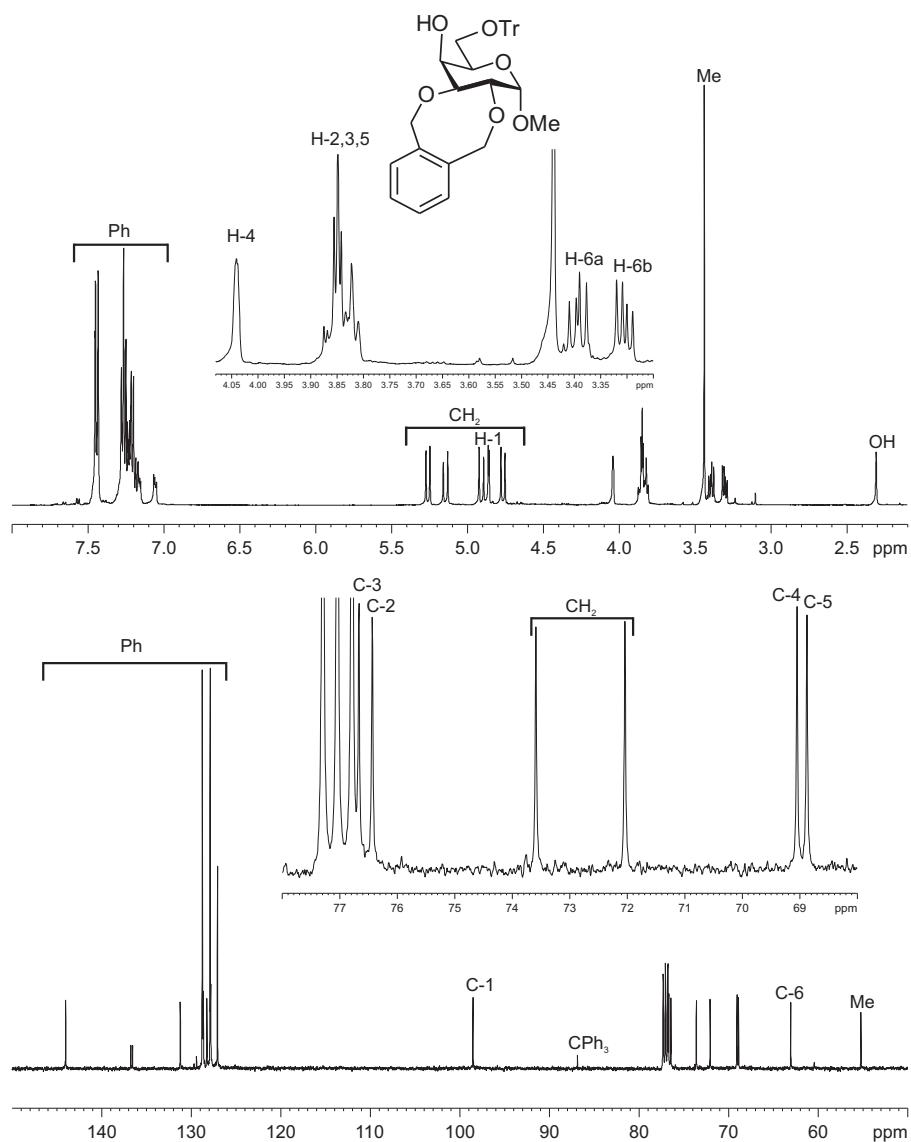


Figure S14. ¹H and ¹³C NMR spectra (500 MHz, 125.7 MHz, CDCl₃) of **25**.

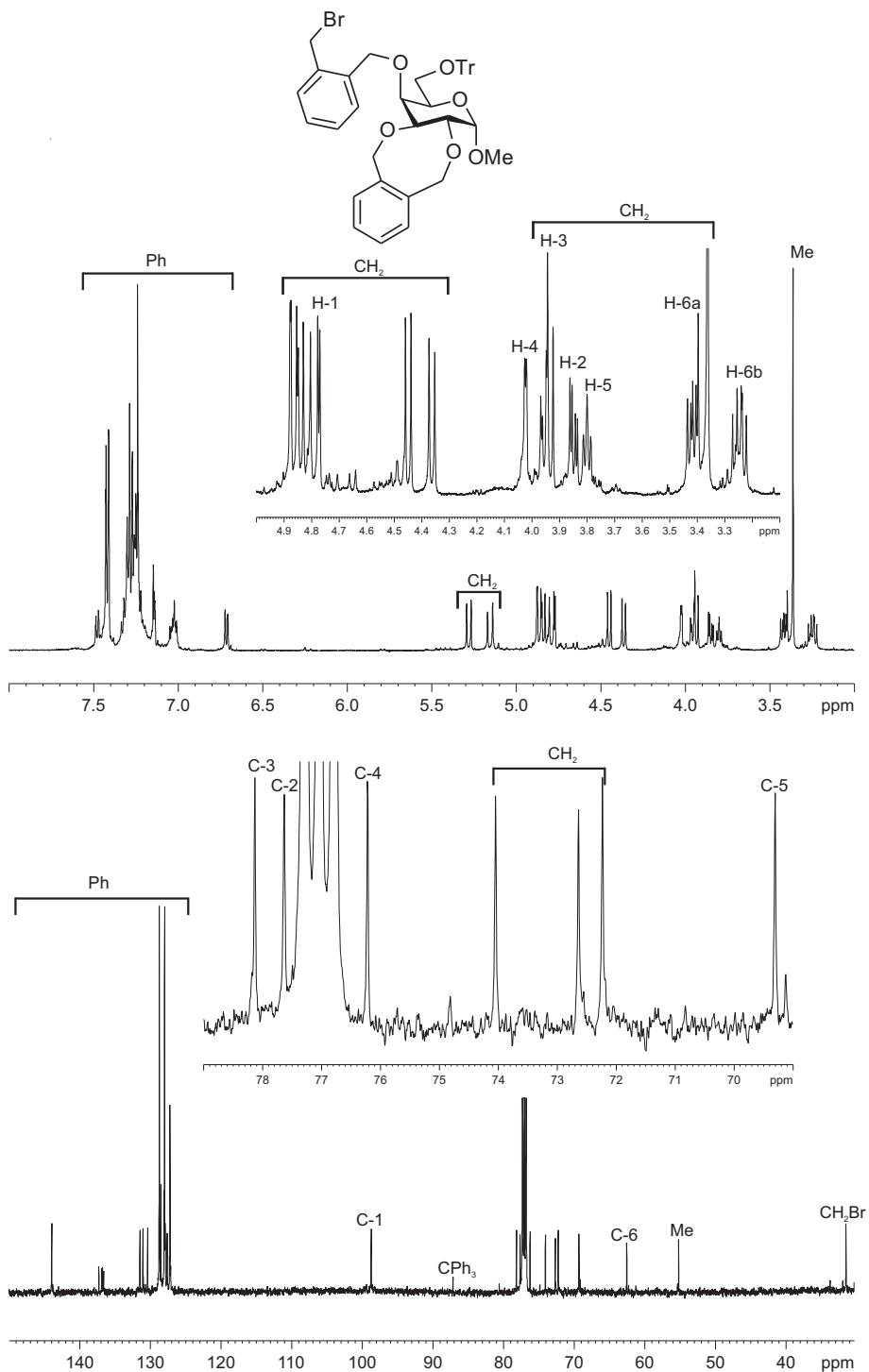


Figure S15. ^1H and ^{13}C NMR spectra (500 MHz, 125.7 MHz, CDCl₃) of **26**.

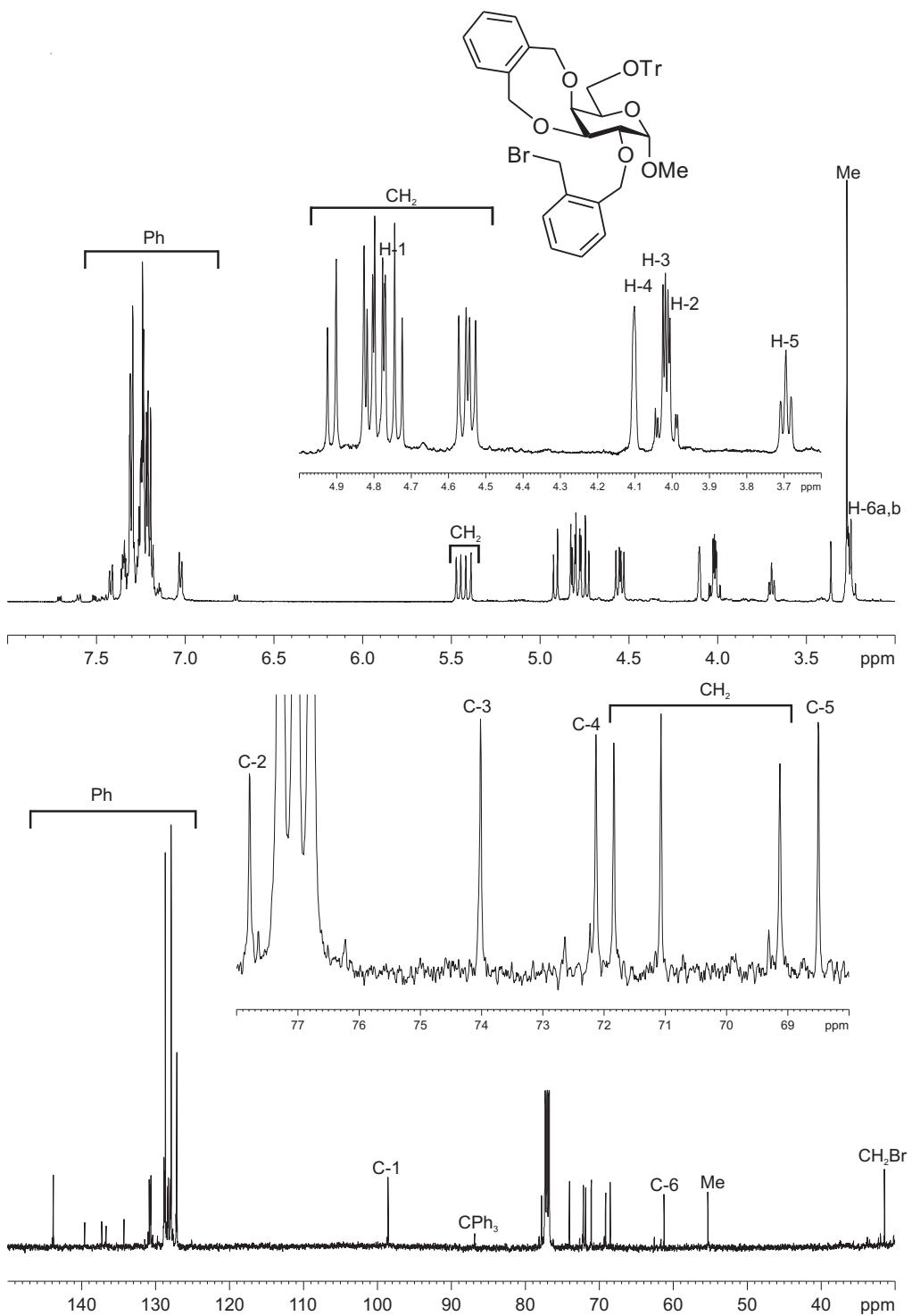


Figure S16. ^1H and ^{13}C NMR spectra (500 MHz, 125.7 MHz, CDCl_3) of **27**.

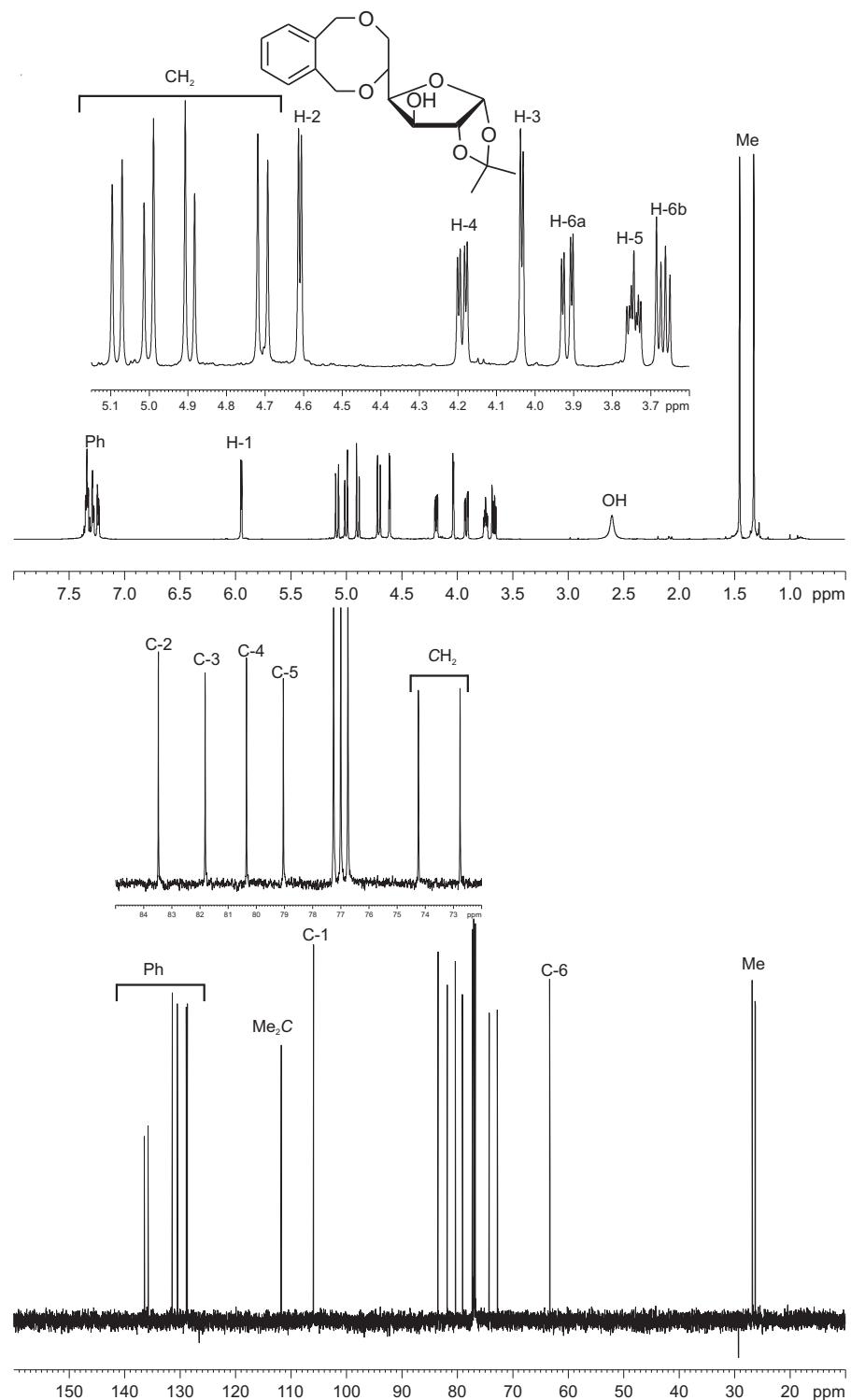


Figure S17. ¹H and ¹³C NMR spectra (500 MHz, 125.7 MHz, CDCl₃) of **29**.

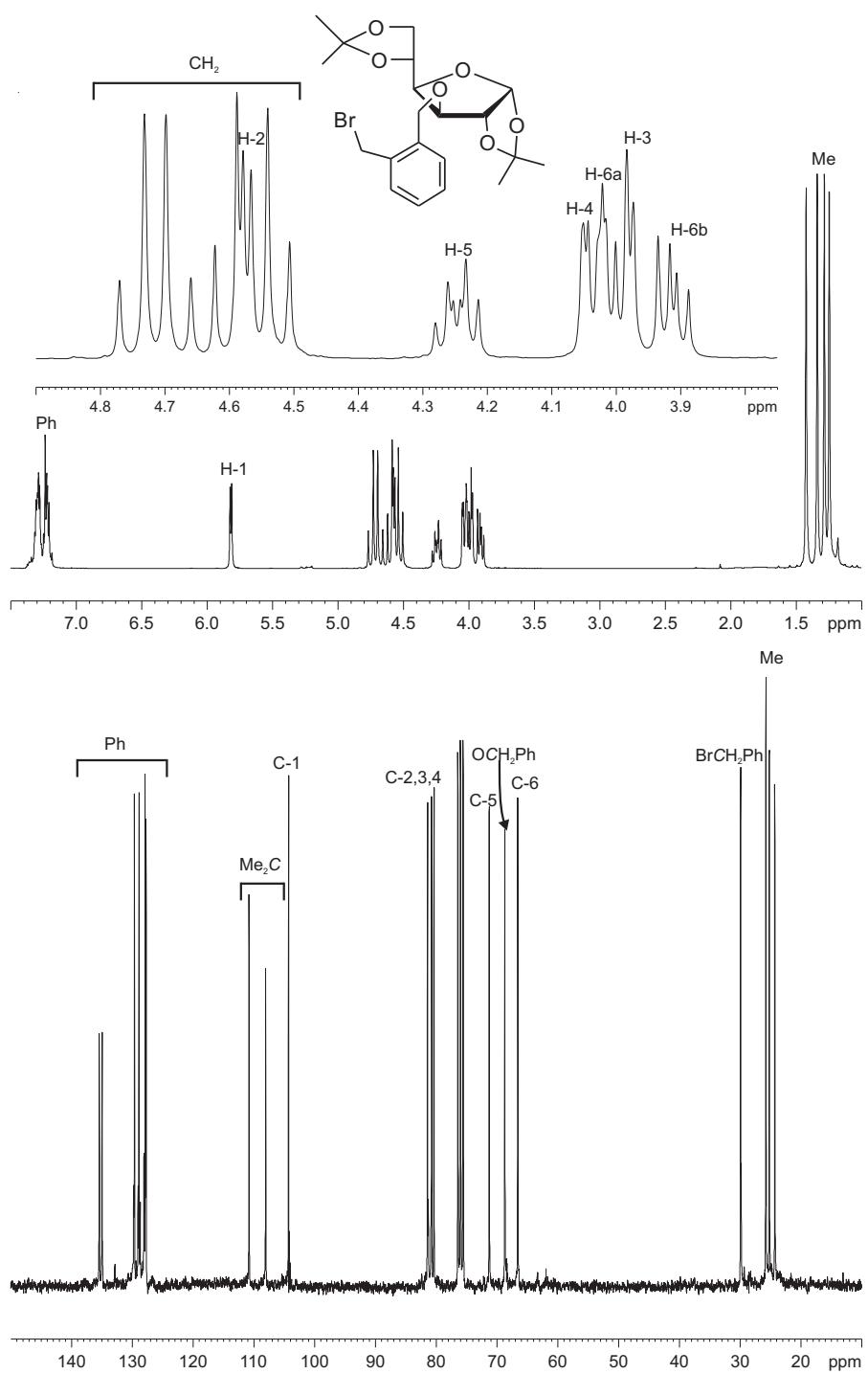


Figure S18. ^1H and ^{13}C NMR spectra (500 MHz, 125.7 MHz, CDCl_3) of **31**.

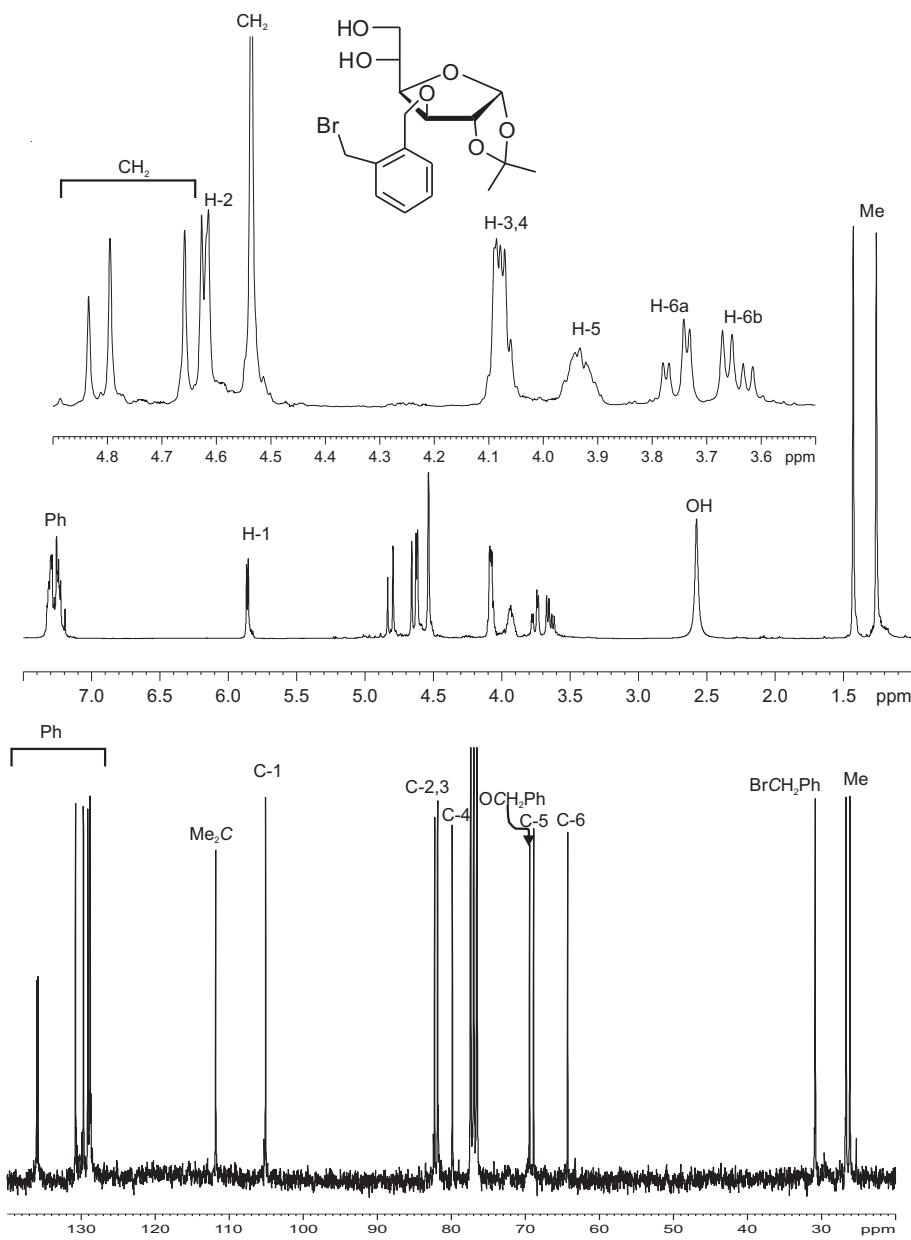


Figure S19. ^1H and ^{13}C NMR spectra (500 MHz, 125.7 MHz, CDCl_3) of **32**.

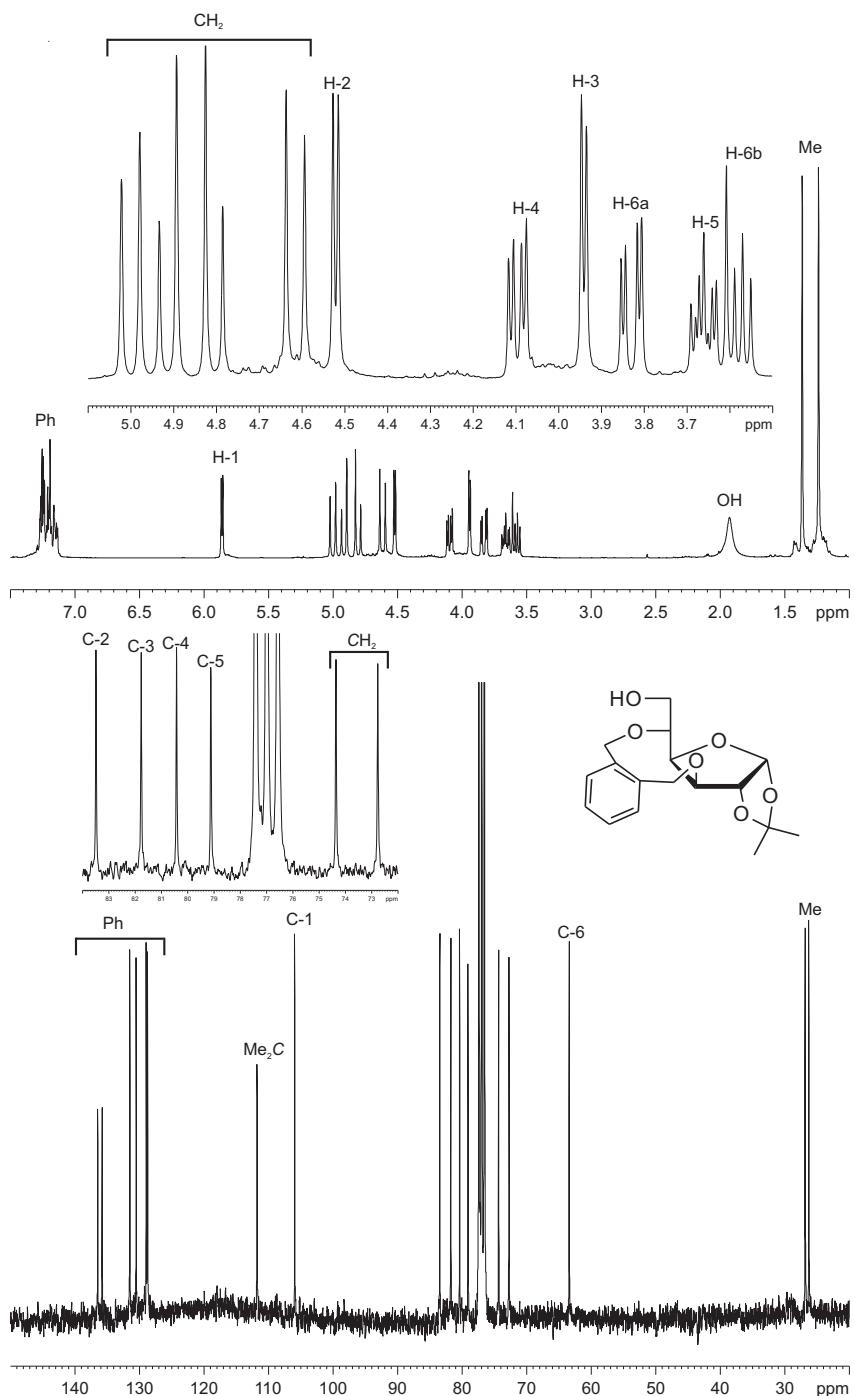


Figure S20. ^1H and ^{13}C NMR spectra (500 MHz, 125.7 MHz, CDCl_3) of **33**.

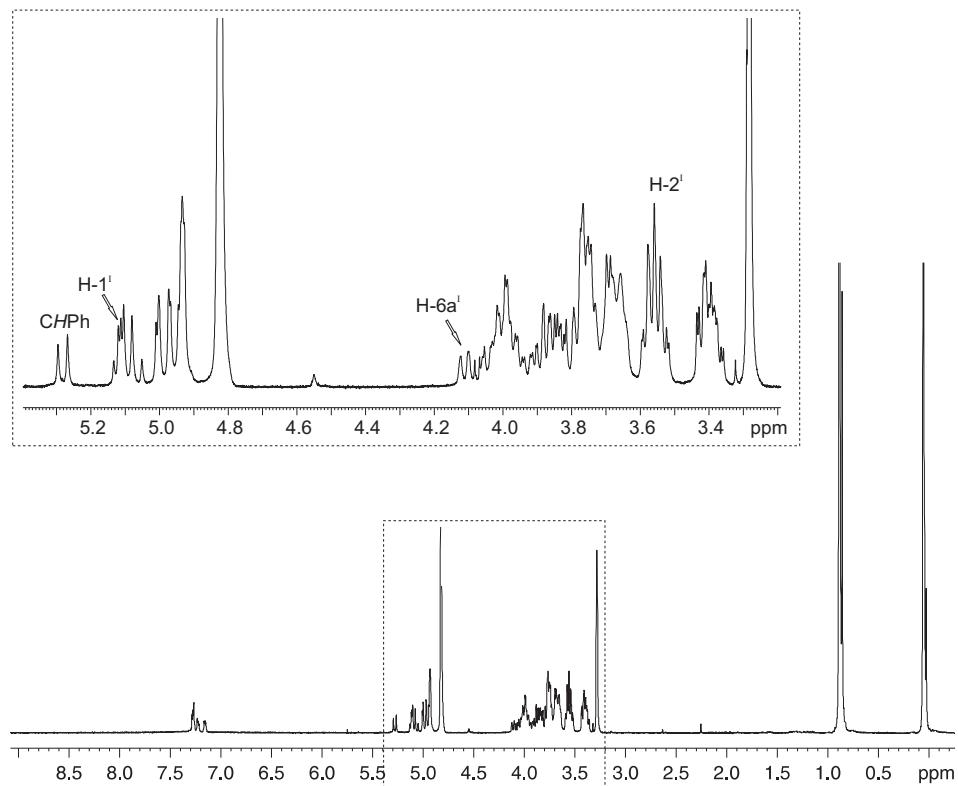
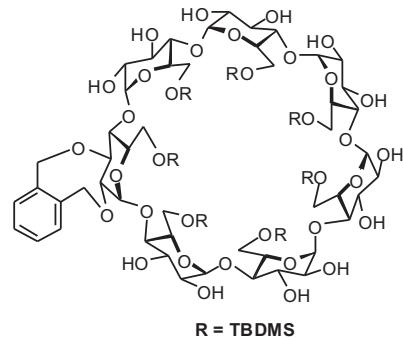


Figure S21. ¹H NMR spectrum (500 MHz, CD₃OD) of **35**.

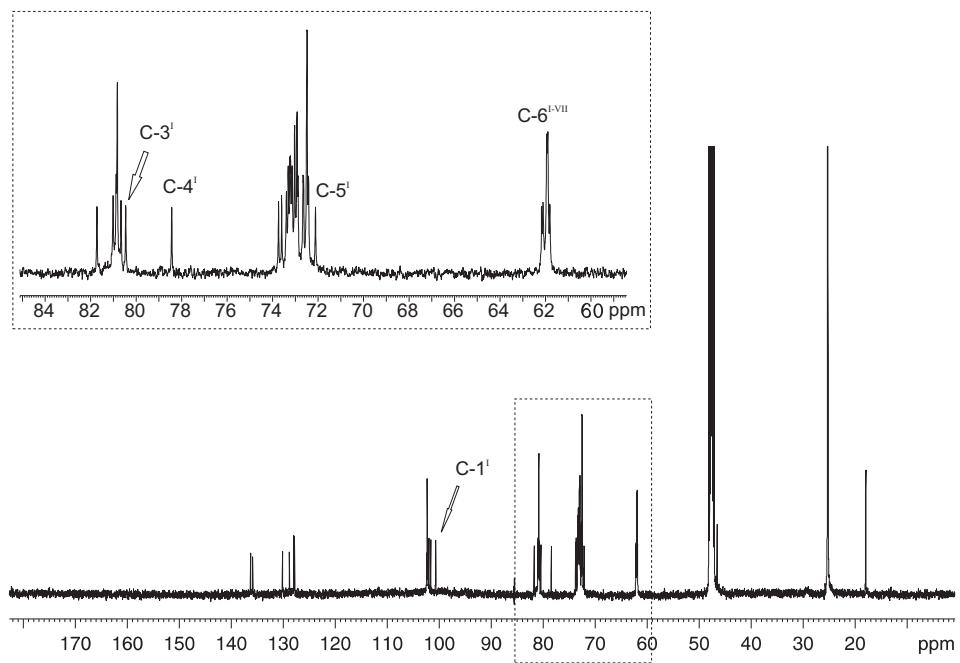
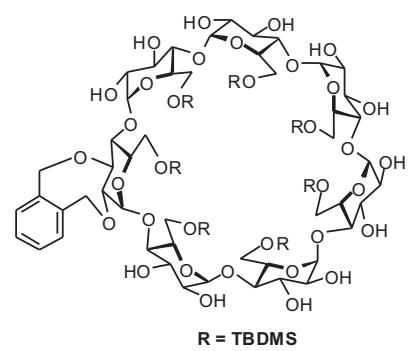


Figure S22. ^{13}C NMR spectrum (125.7 MHz, CD_3OD) of 35.

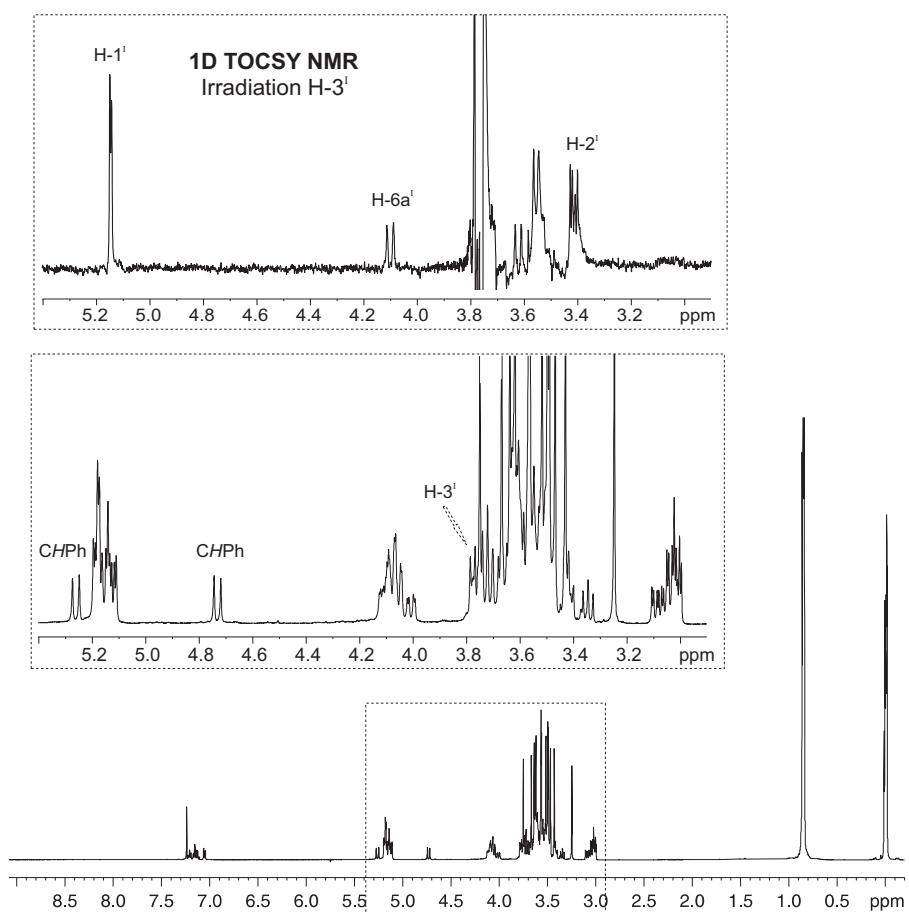
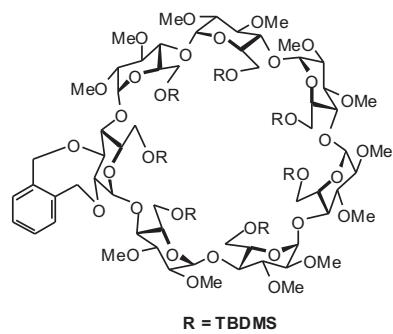


Figure S23. ^1H and 1D TOCSY NMR spectra (500 MHz, CDCl_3) of **36**.

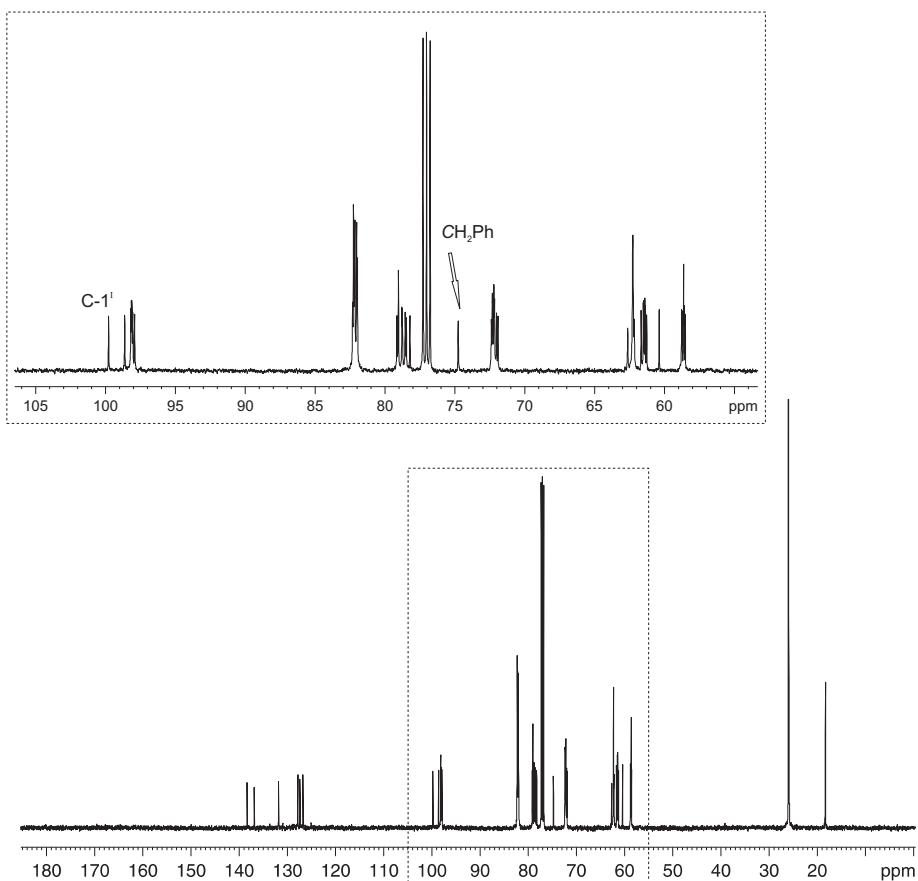
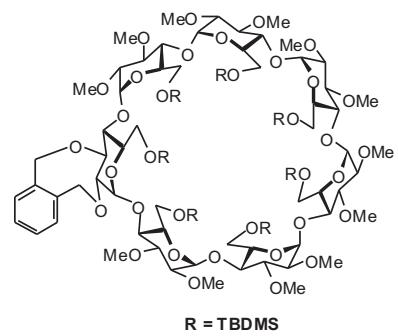


Figure S24. ¹³C NMR spectrum (125.7 MHz, CDCl₃) of **36**.

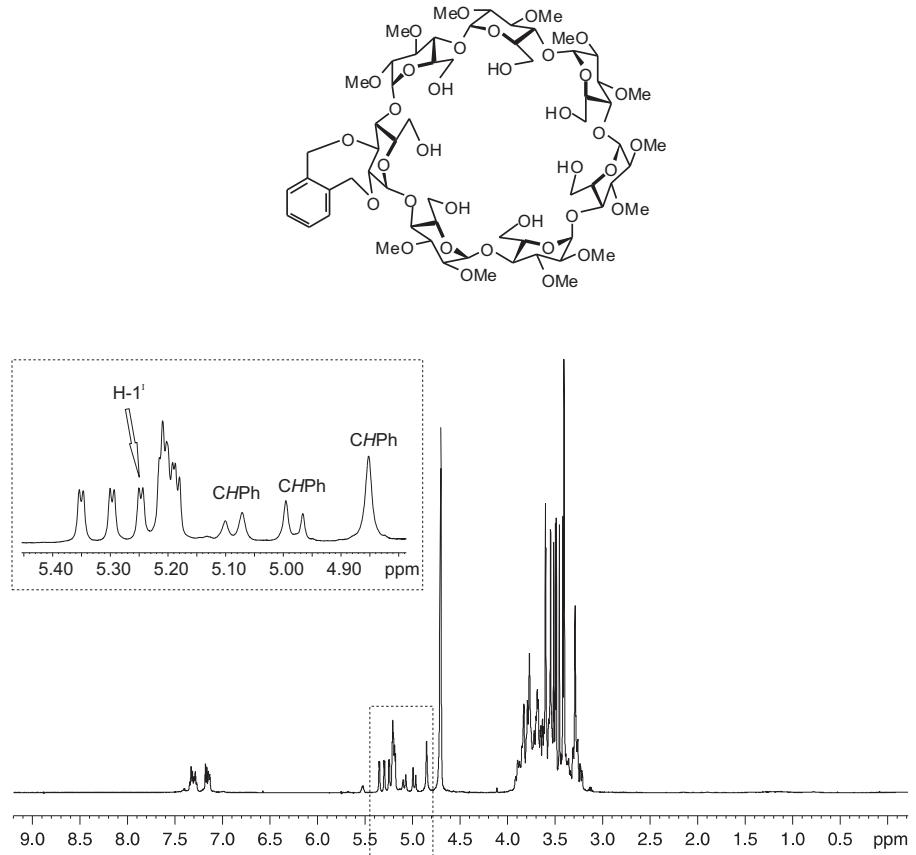


Figure S25. ^1H NMR spectra (500 MHz, D_2O) of **37**.

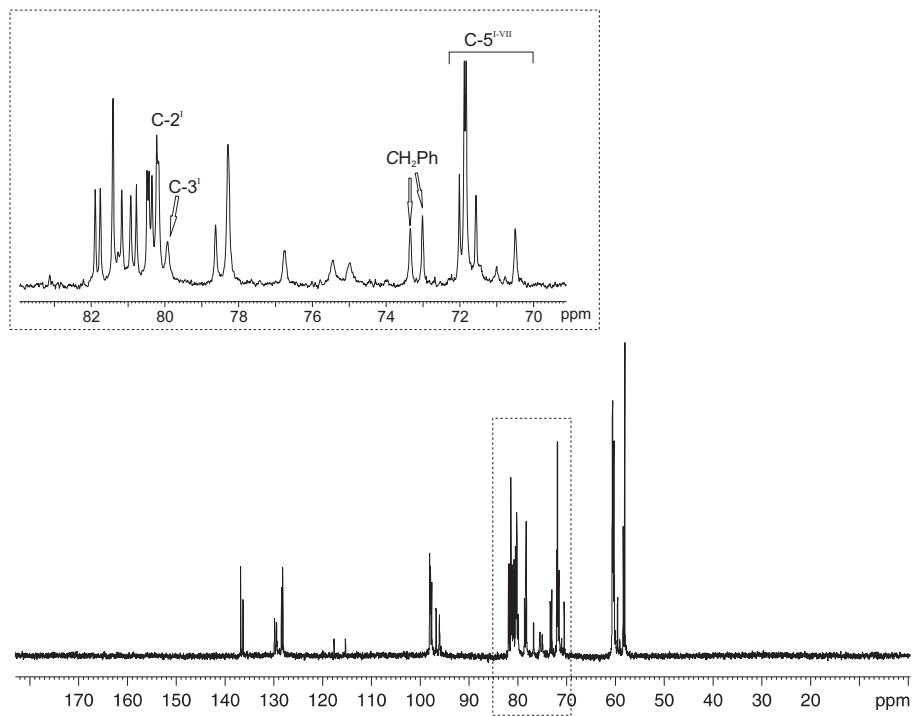
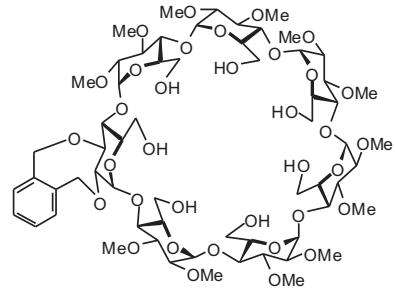


Figure S26. ^{13}C NMR spectra (125.7 MHz, D_2O) of **37**.

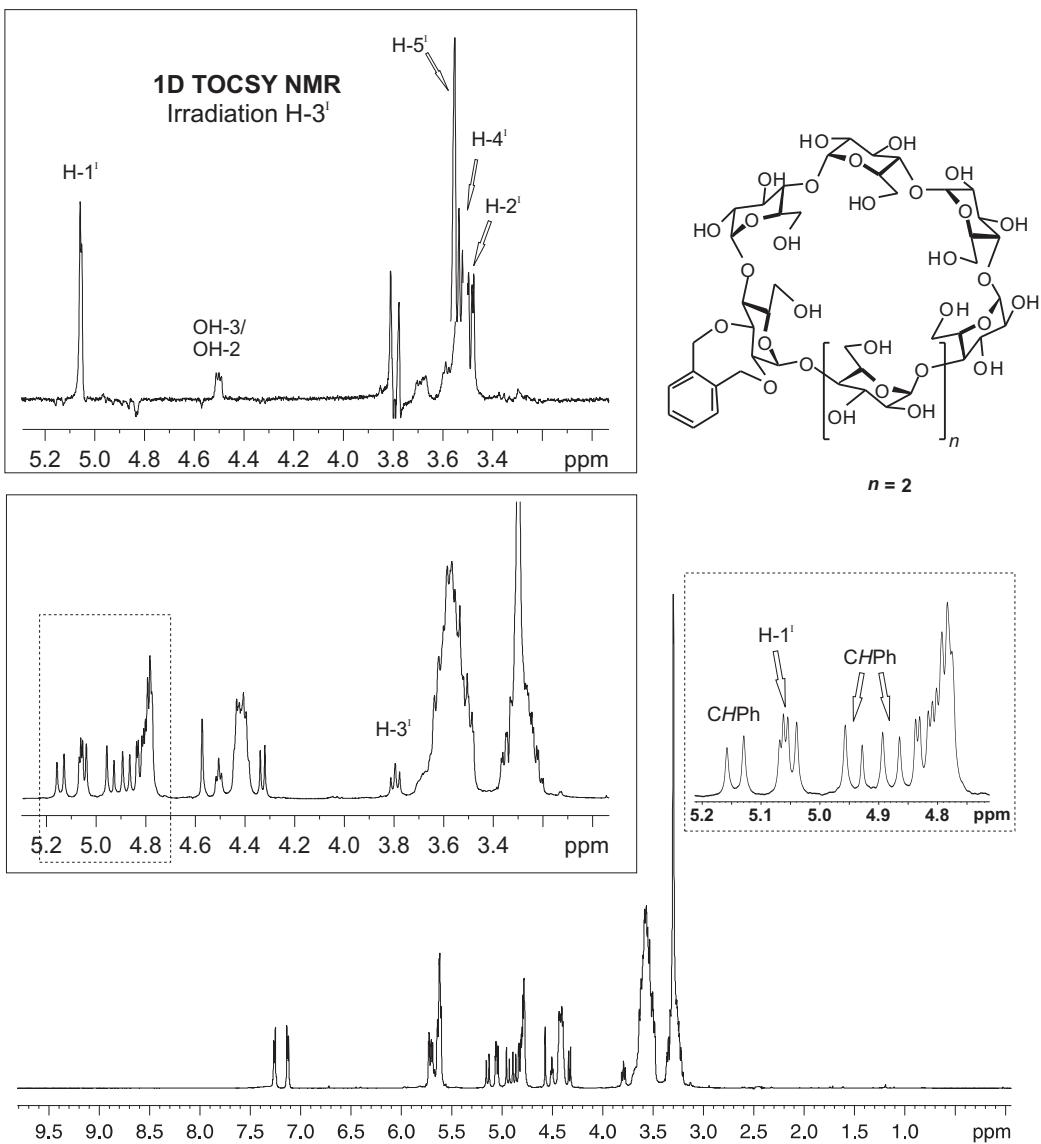
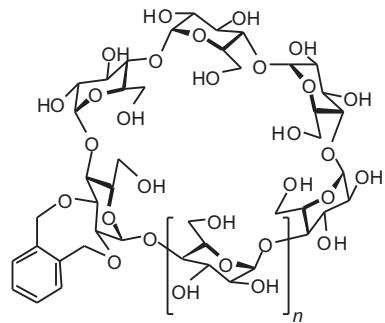


Figure S27. ¹H and 1D TOCSY NMR spectra (500 MHz, DMSO-*d*₆) of **38**.



n = 2

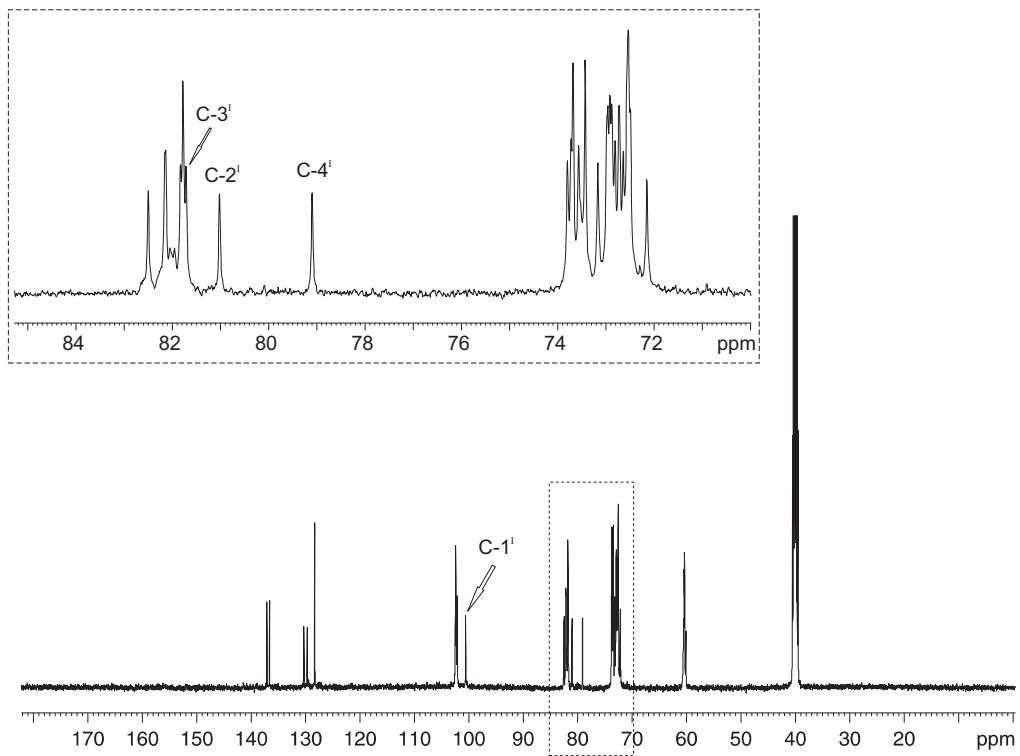


Figure S28. ^{13}C NMR spectrum (125.7 MHz, $\text{DMSO}-d_6$) of **38**.

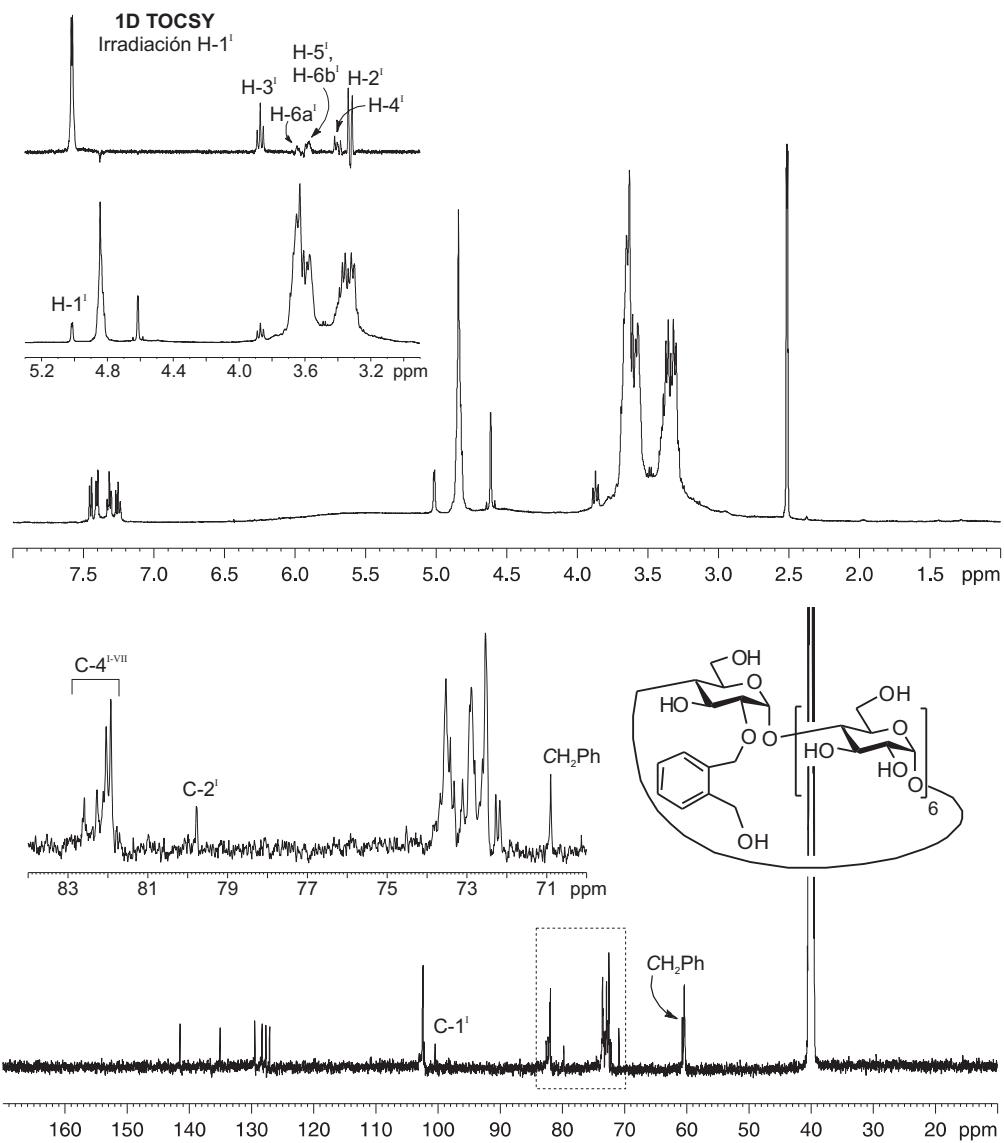


Figure S29. ¹H, 1D TOCSY and ¹³C RMN spectra (500 MHz, 125.7 MHz, DMSO-*d*₆) of **39**.

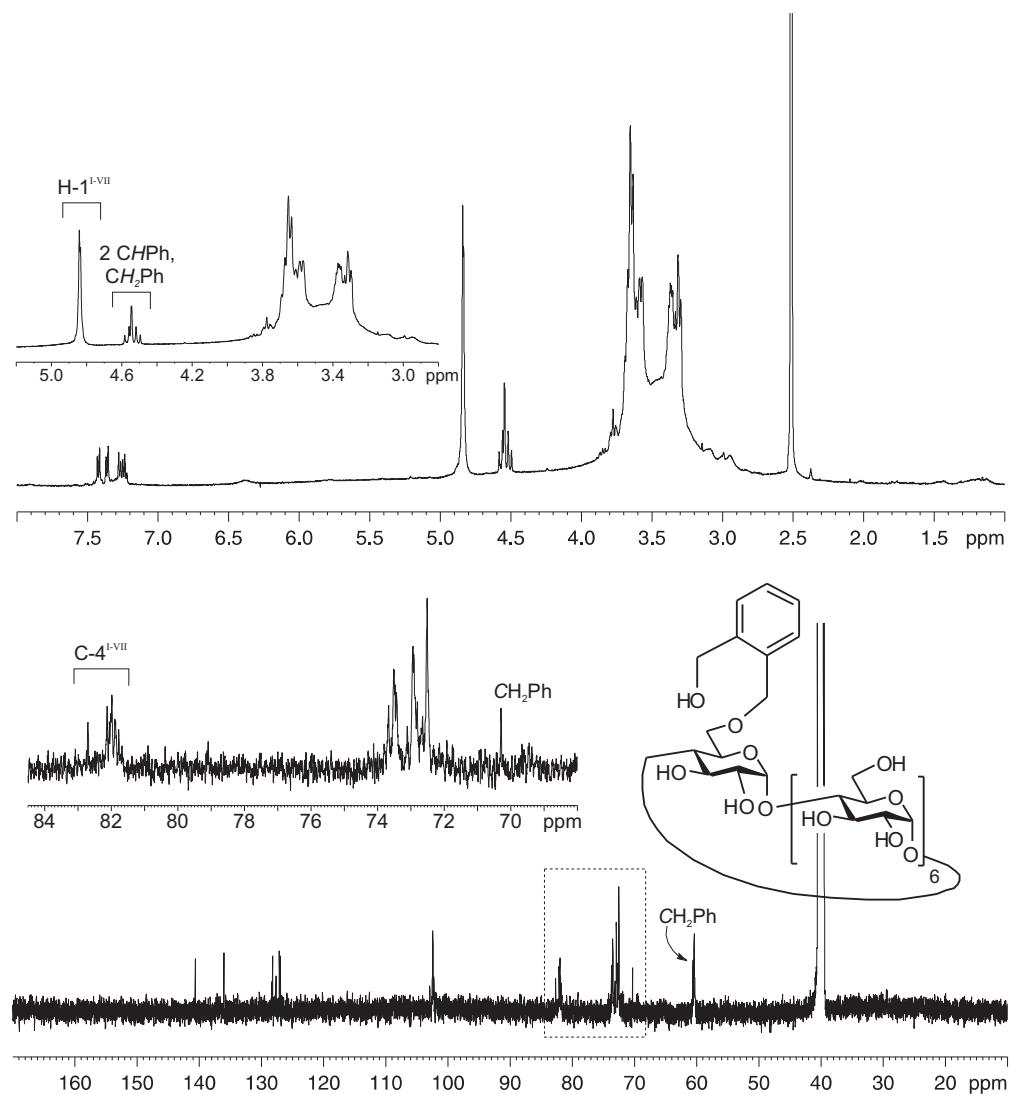


Figure S30. ^1H and ^{13}C NMR spectra (500 MHz, 125.7 MHz, $\text{DMSO}-d_6$) of **40**.

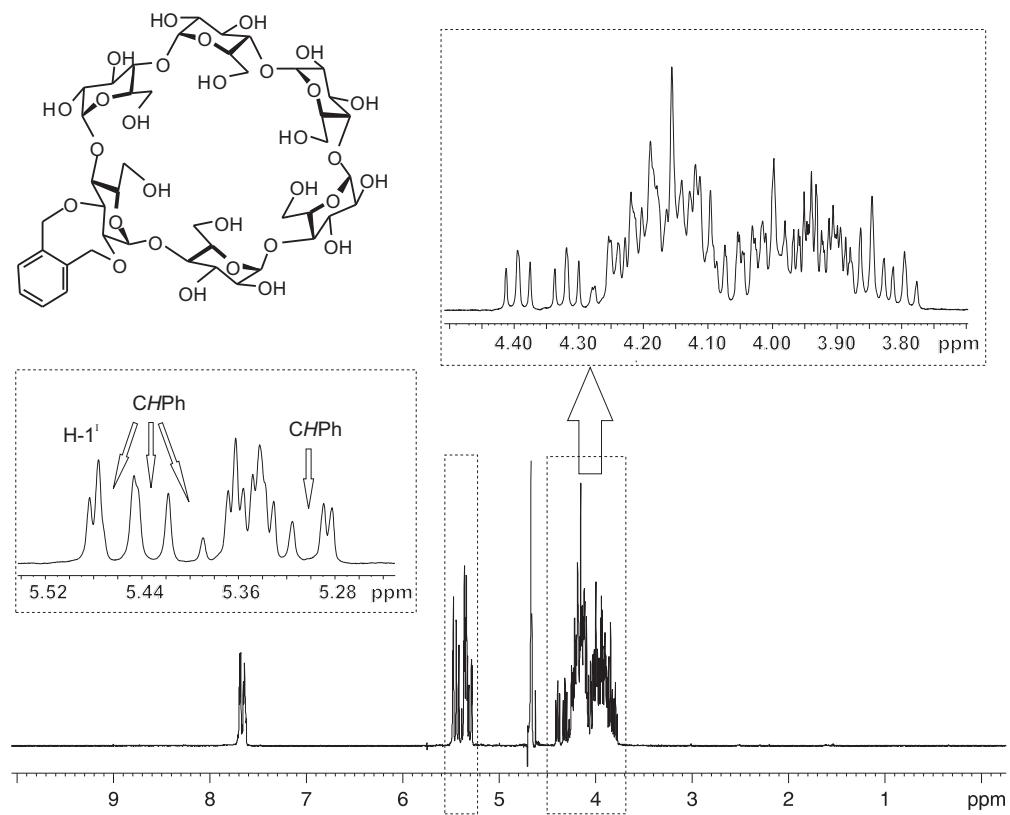


Figure S31. ¹H NMR spectrum (500 MHz, D₂O, 333 K) of **44**

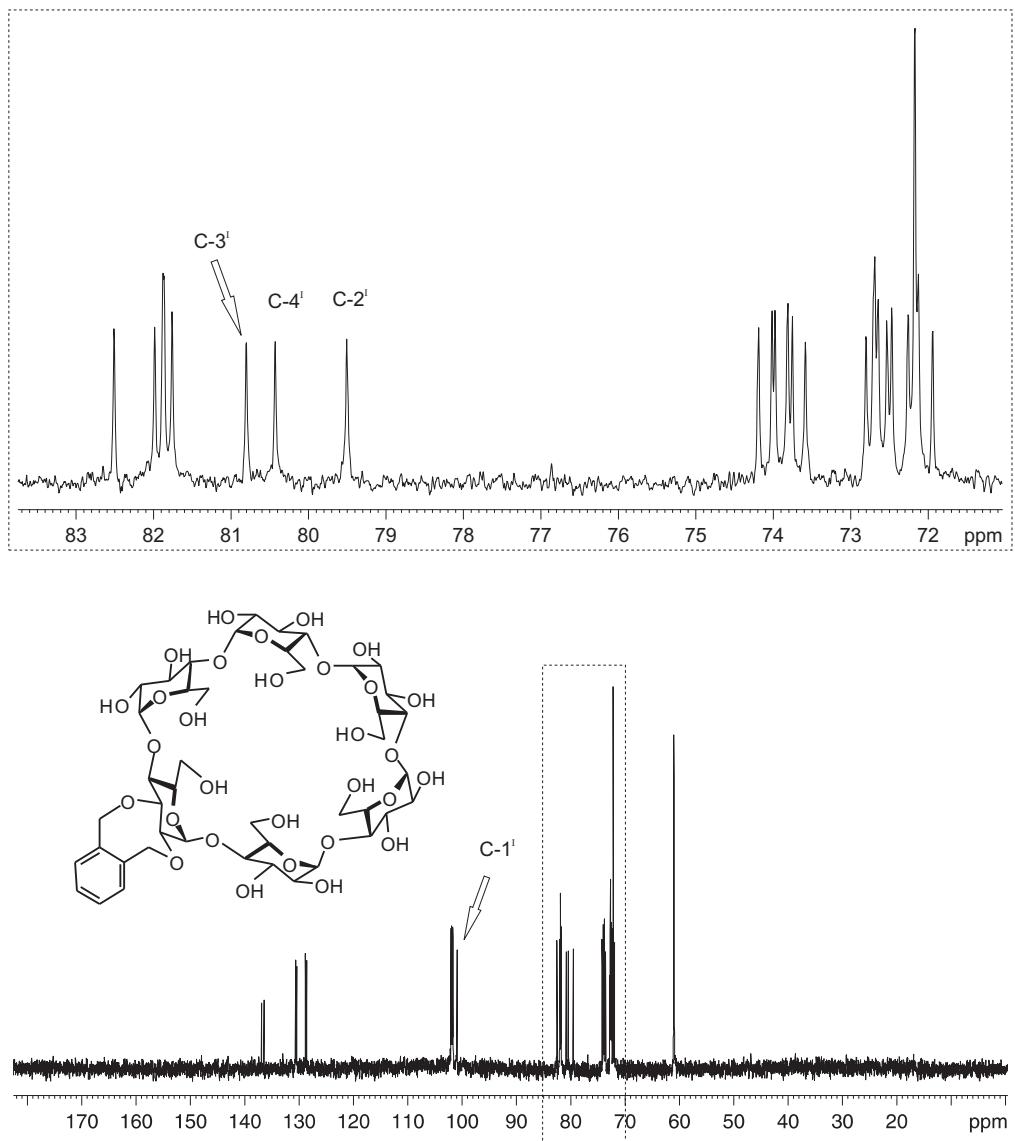


Figure S32. ^{13}C NMR spectrum (125.7 MHz, D_2O , 333 K) of **44**.

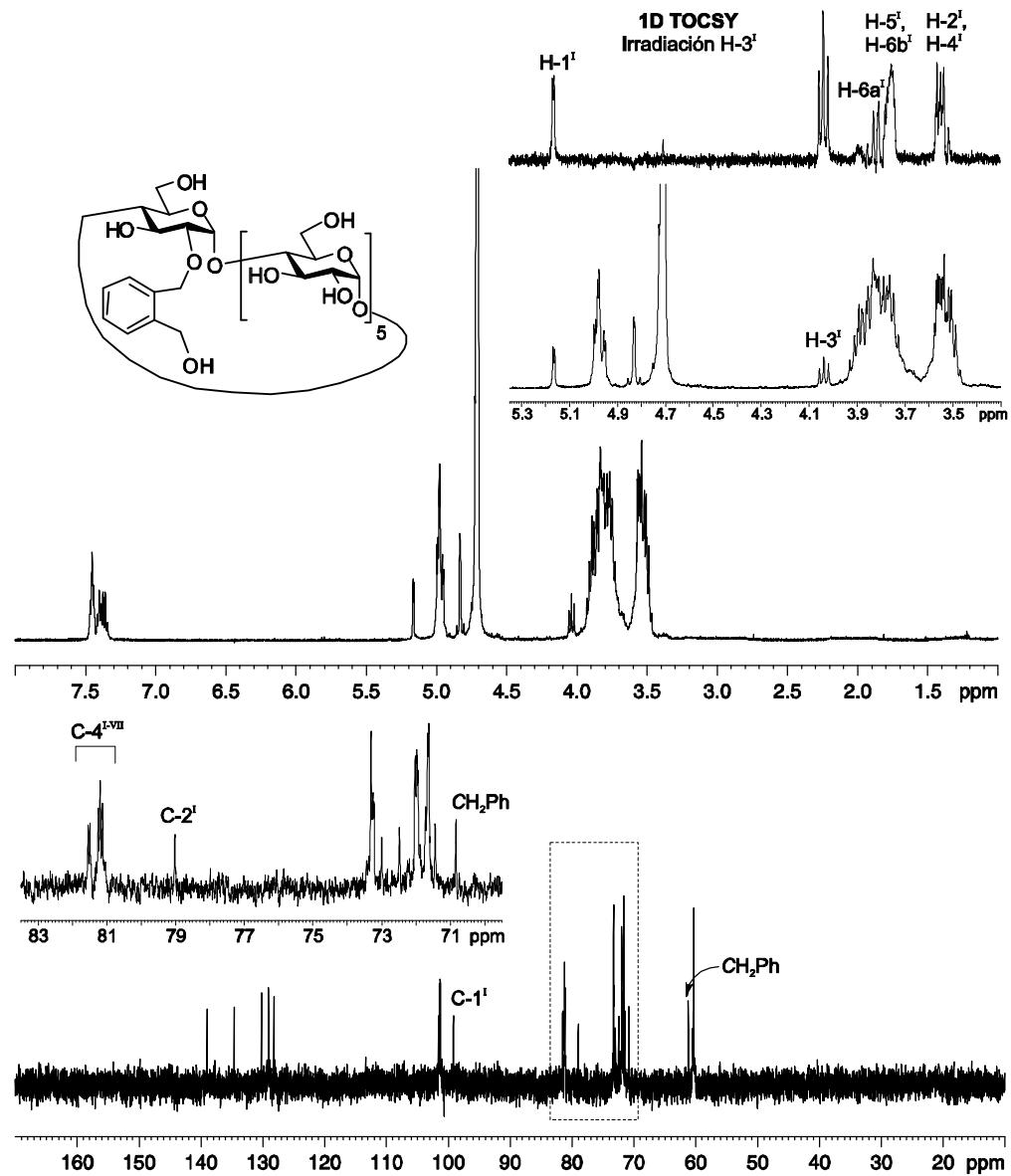


Figure S33. ¹H, 1D TOCSY and ¹³C NMR spectra (500 MHz, 125.7 MHz, D₂O) of **45**.

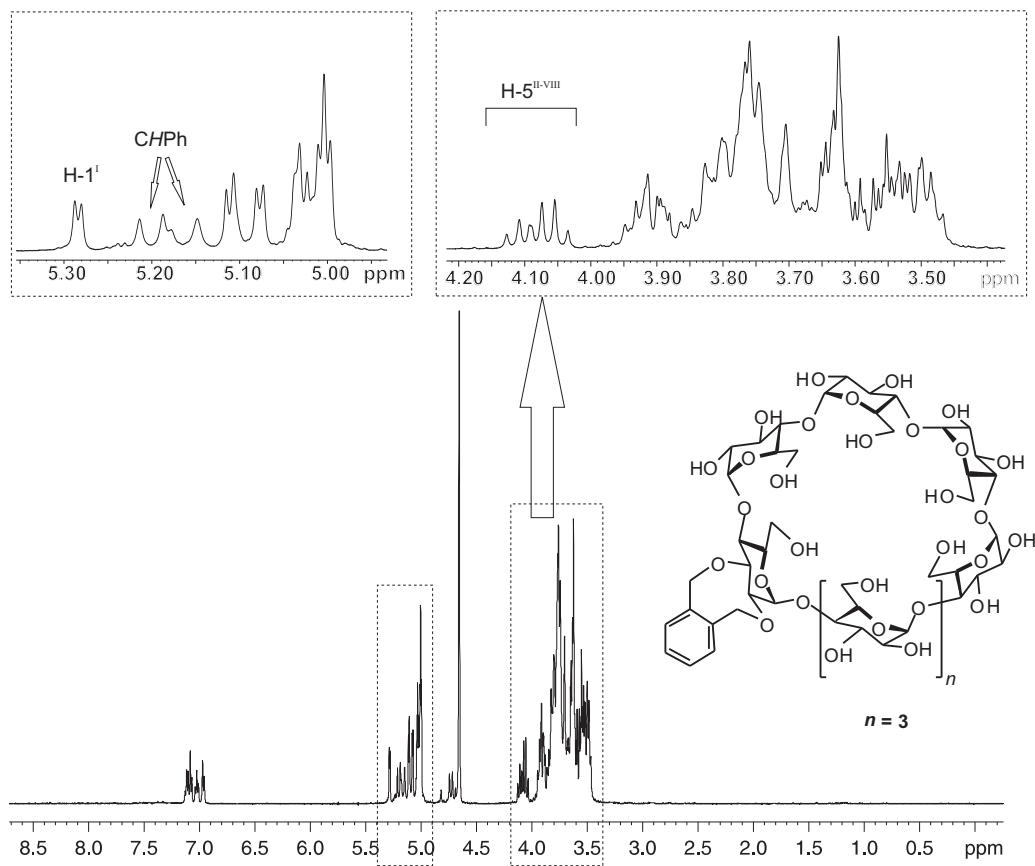


Figure S34. ^1H NMR spectrum (500 MHz, D_2O) of **49**.

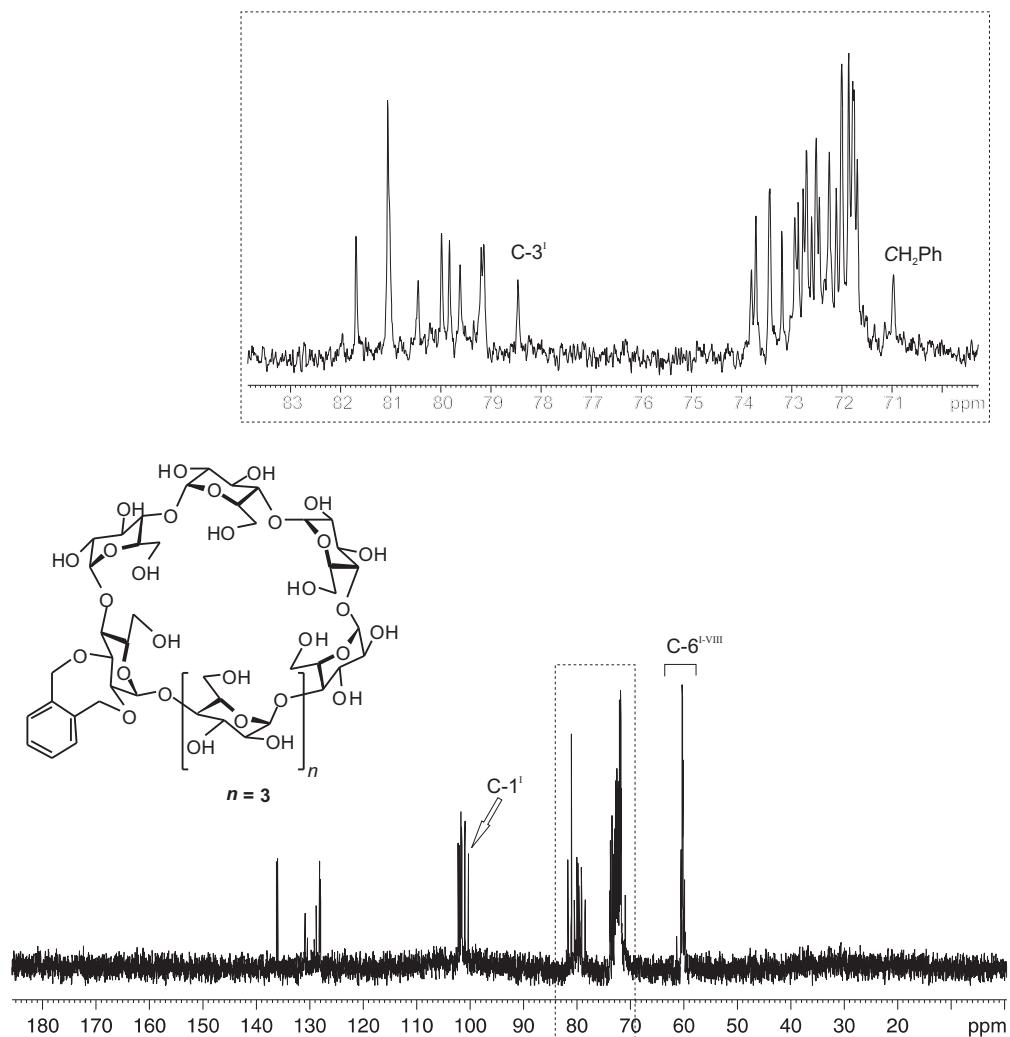


Figure S35. ^{13}C NMR spectrum (125.7 MHz, D_2O) of **49**.

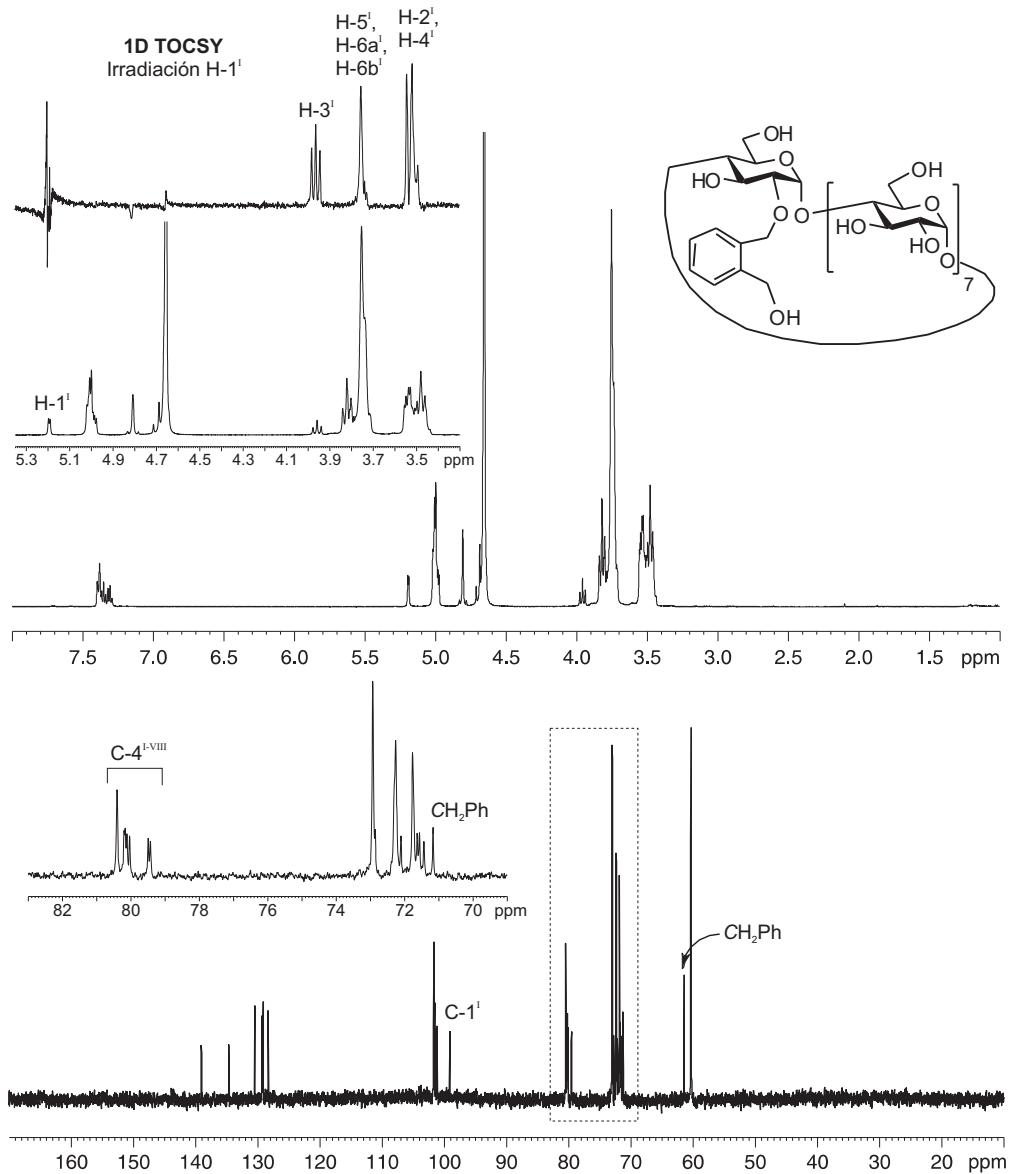


Figure S36. ^1H , 1D TOCSY and ^{13}C NMR spectra (500 MHz, 125.7 MHz, D_2O) of **50**.

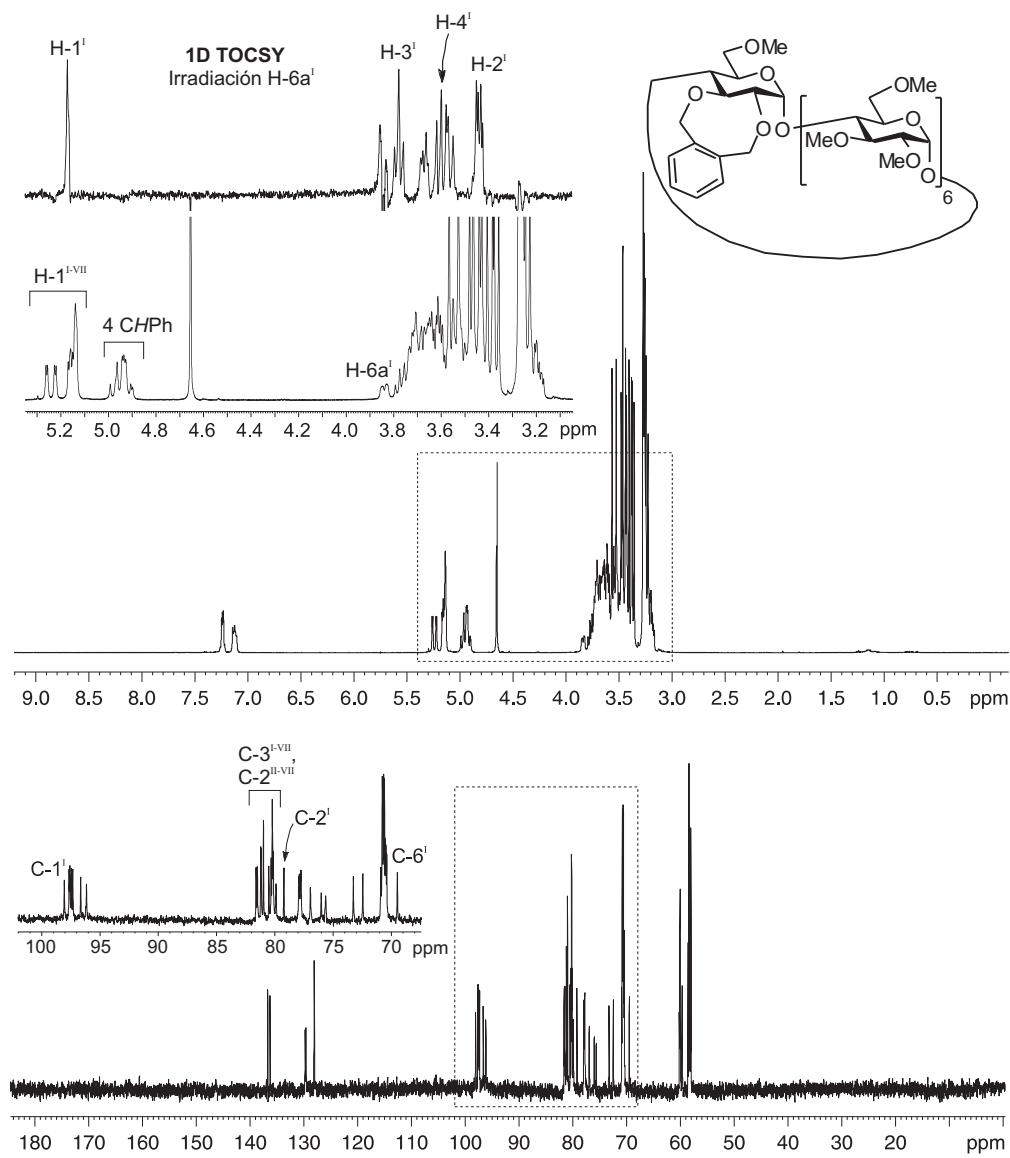


Figure S37. ^1H , 1D TOCSY and ^{13}C NMR spectra (500 MHz, 125.7 MHz, D_2O) of **41**.

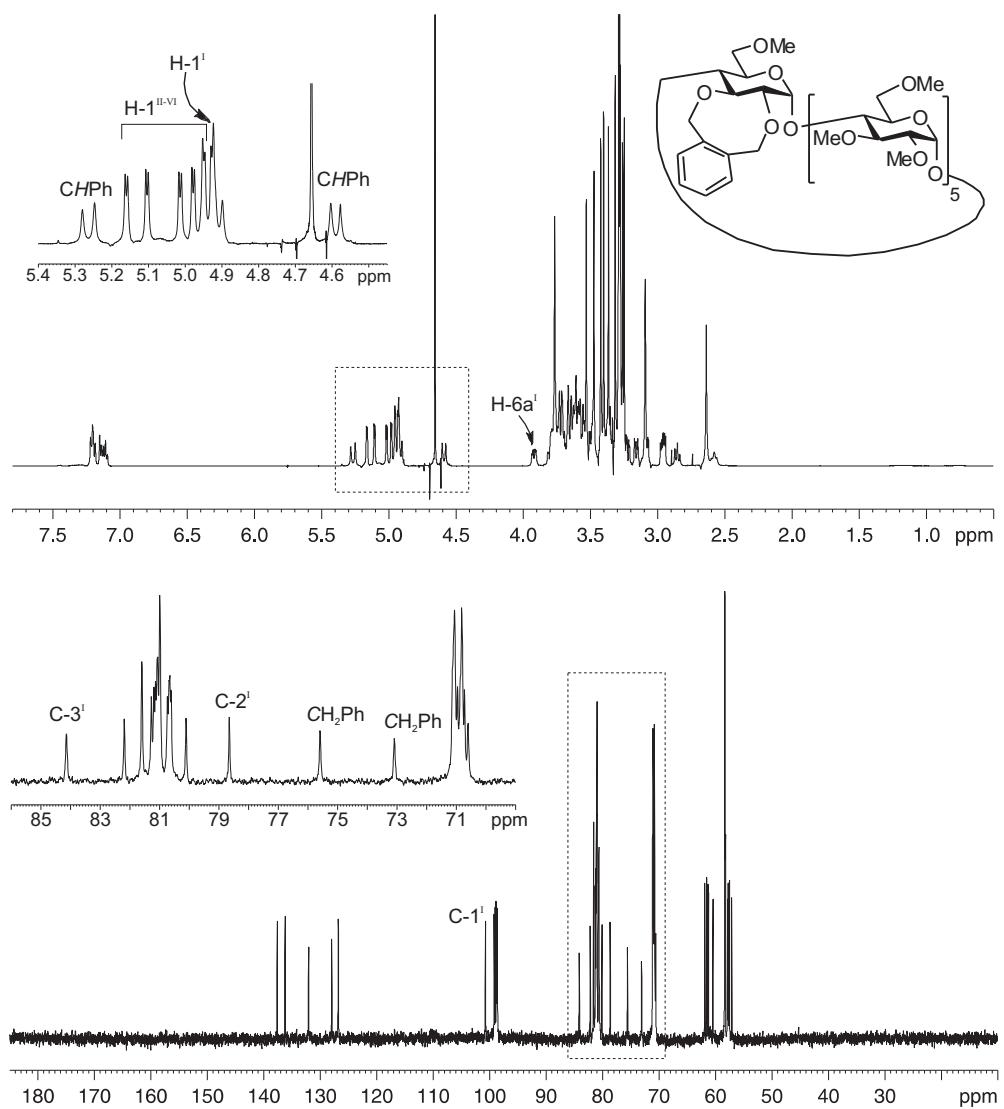


Figure S38. ¹H and ¹³C NMR spectra (500 MHz, 125.7 MHz, D₂O) of **46**.

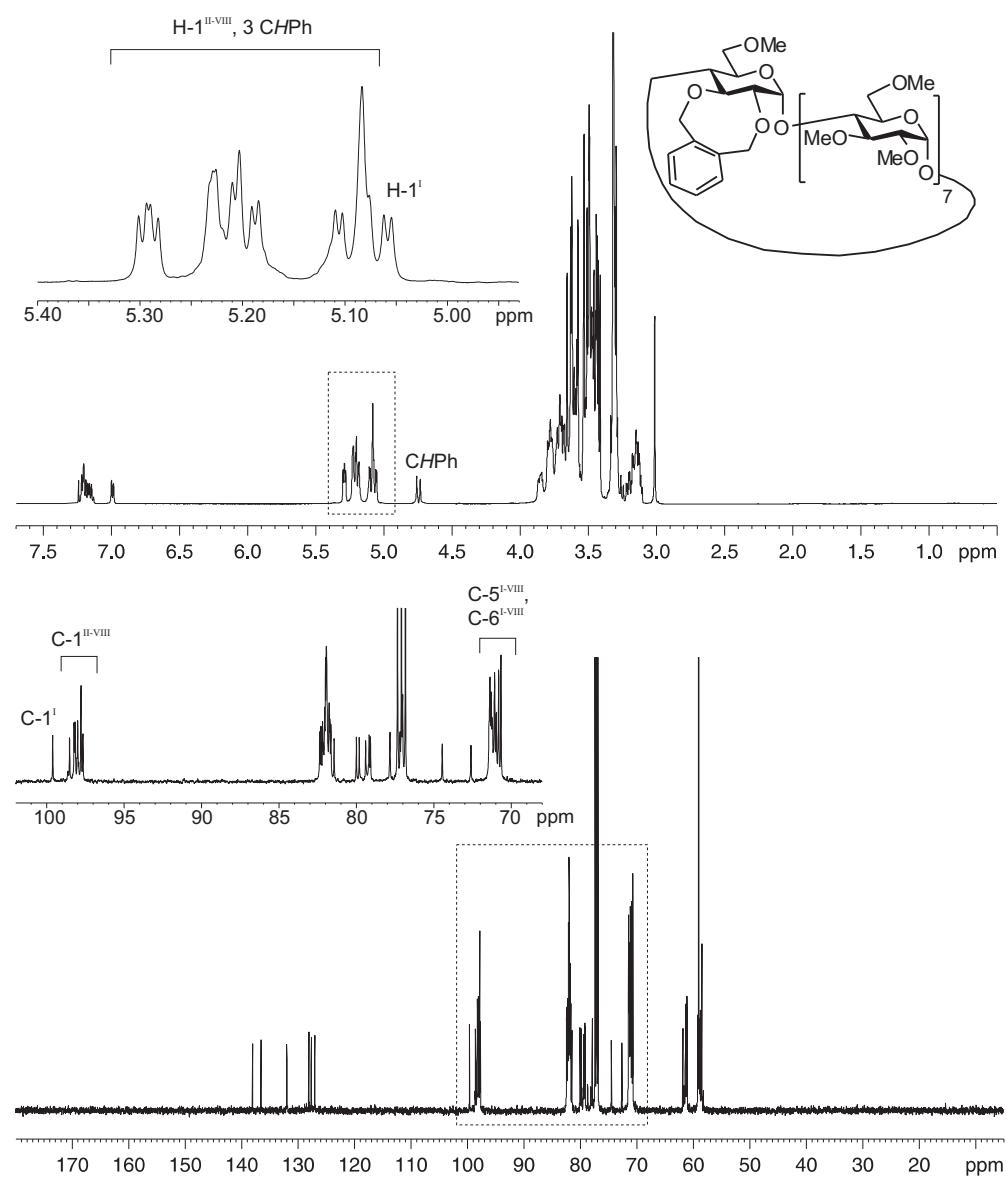


Figure S39. ^1H and ^{13}C NMR spectra (500 MHz, 125.7 MHz, CDCl_3) of **51**.

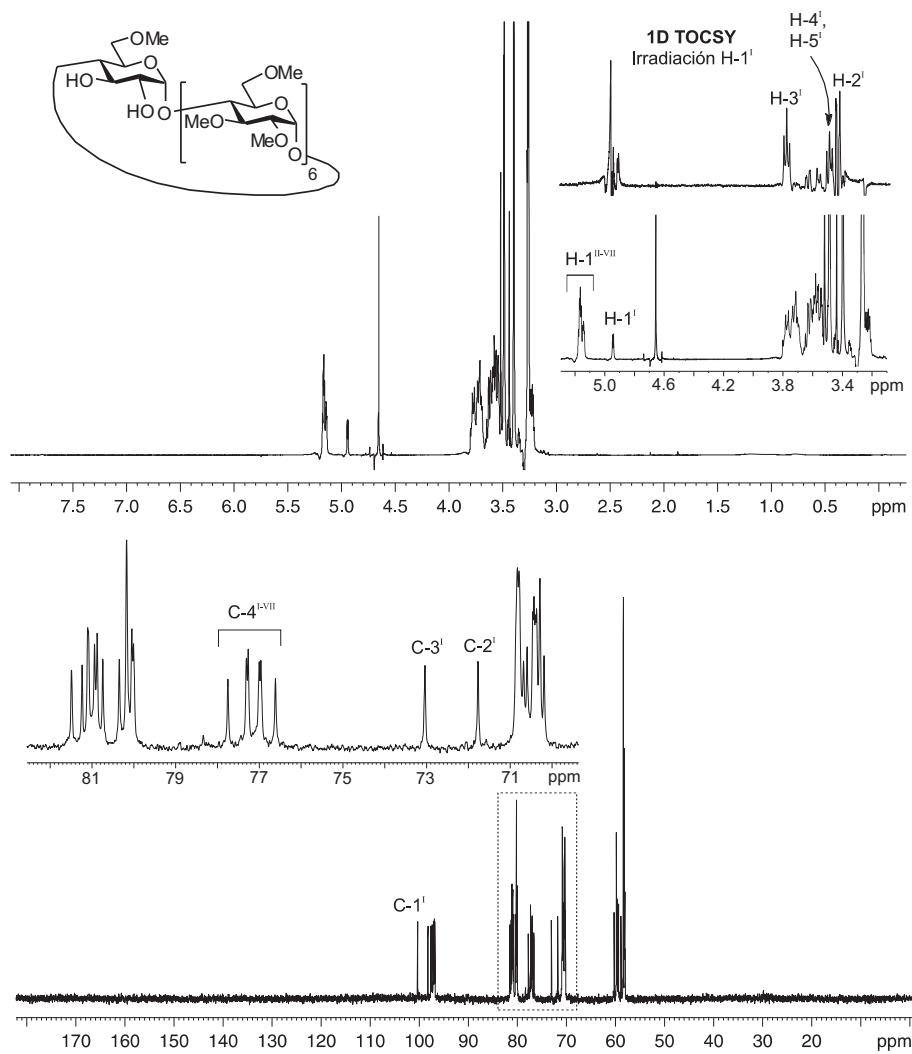


Figure S40. ¹H, 1D TOCSY and ¹³C NMR spectra (500 MHz, 125.7 MHz, D₂O) of **42**.

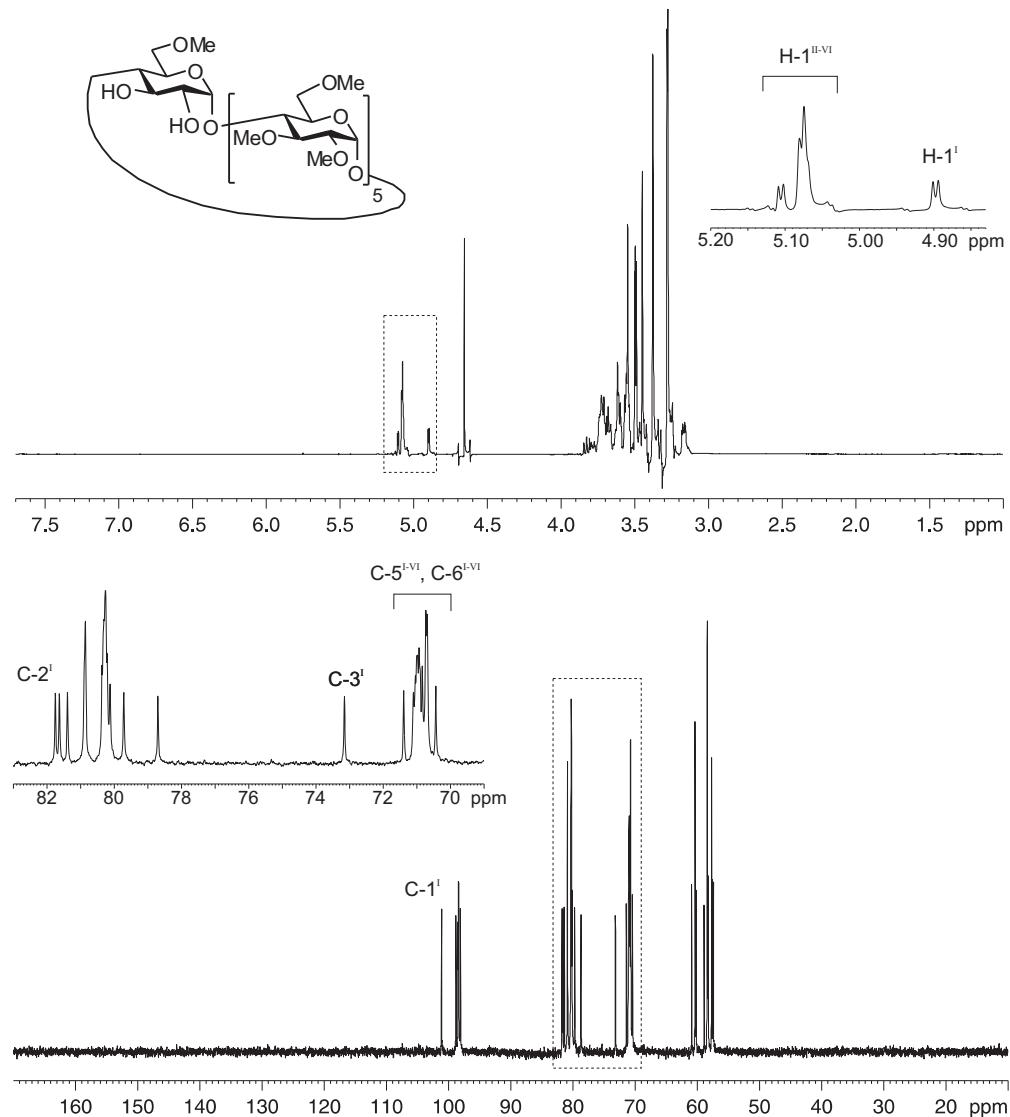


Figure S41. ¹H and ¹³C NMR spectra (500 MHz, 125.7 MHz, D₂O) of compound 47.

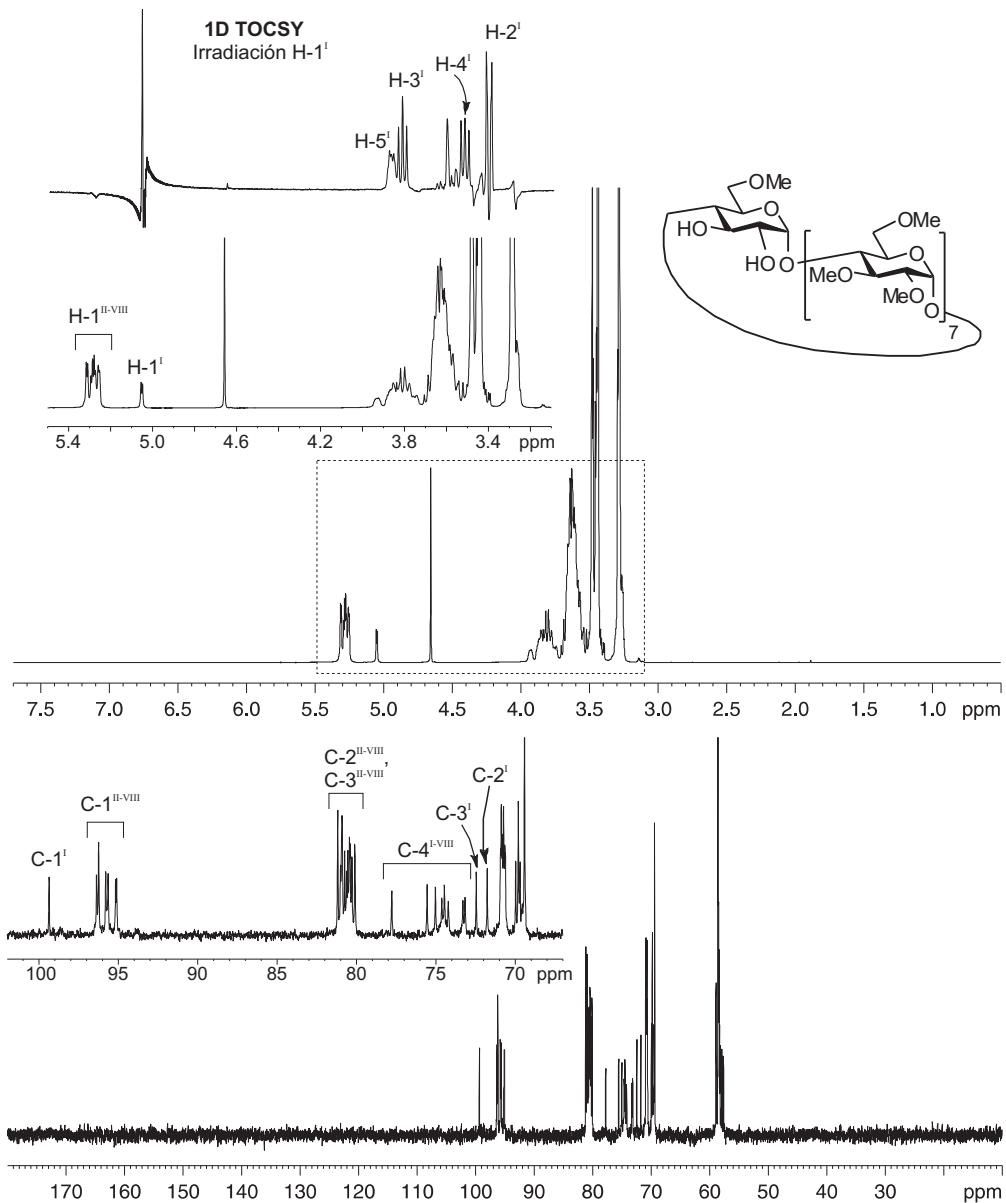


Figure S42. ^1H , 1D TOCSY and ^{13}C NMR spectra (500 MHz, 125.7 MHz, D_2O) of compound **52**.

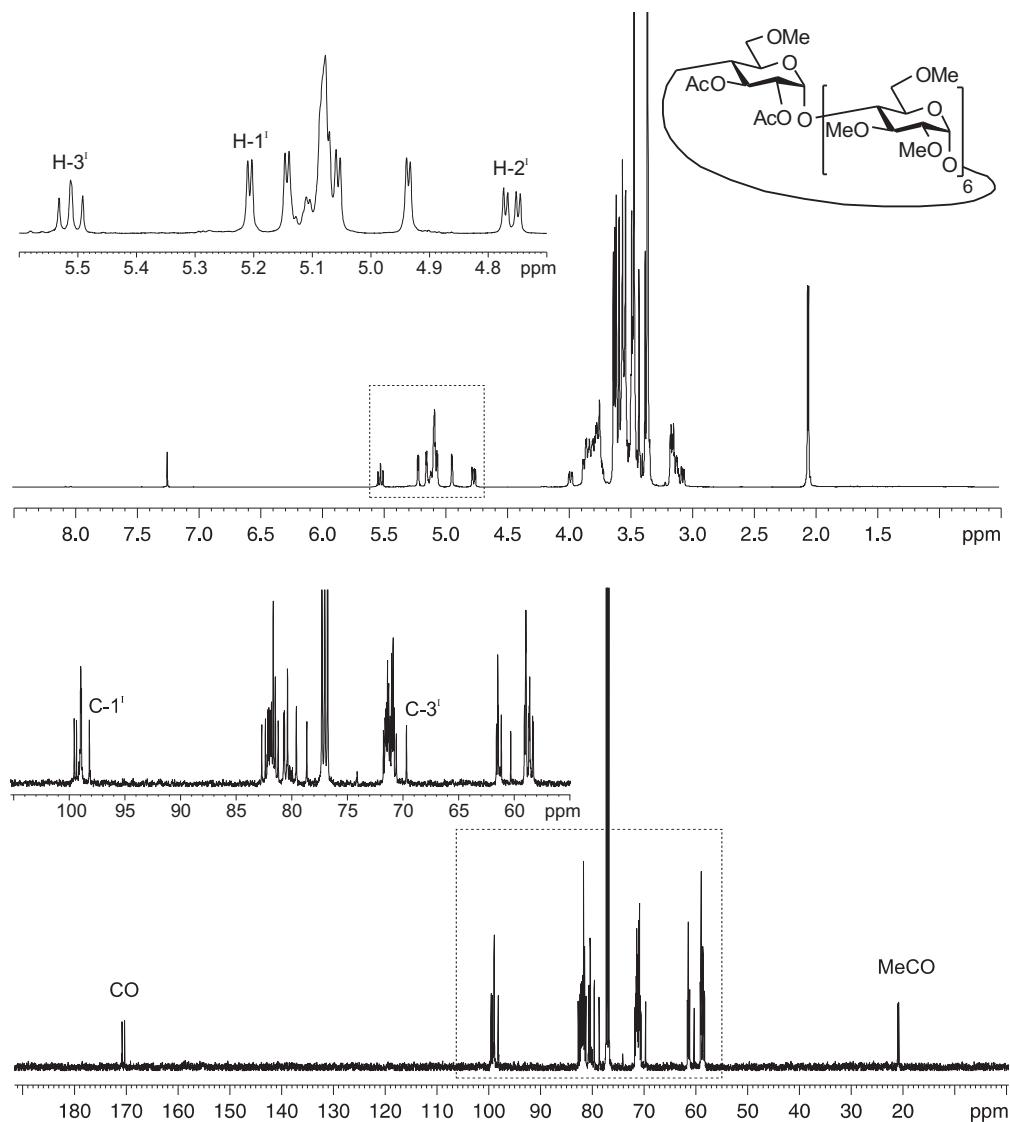


Figure S43. ¹H and ¹³C NMR spectra (500 MHz, 125.7 MHz CDCl₃) of **43**.

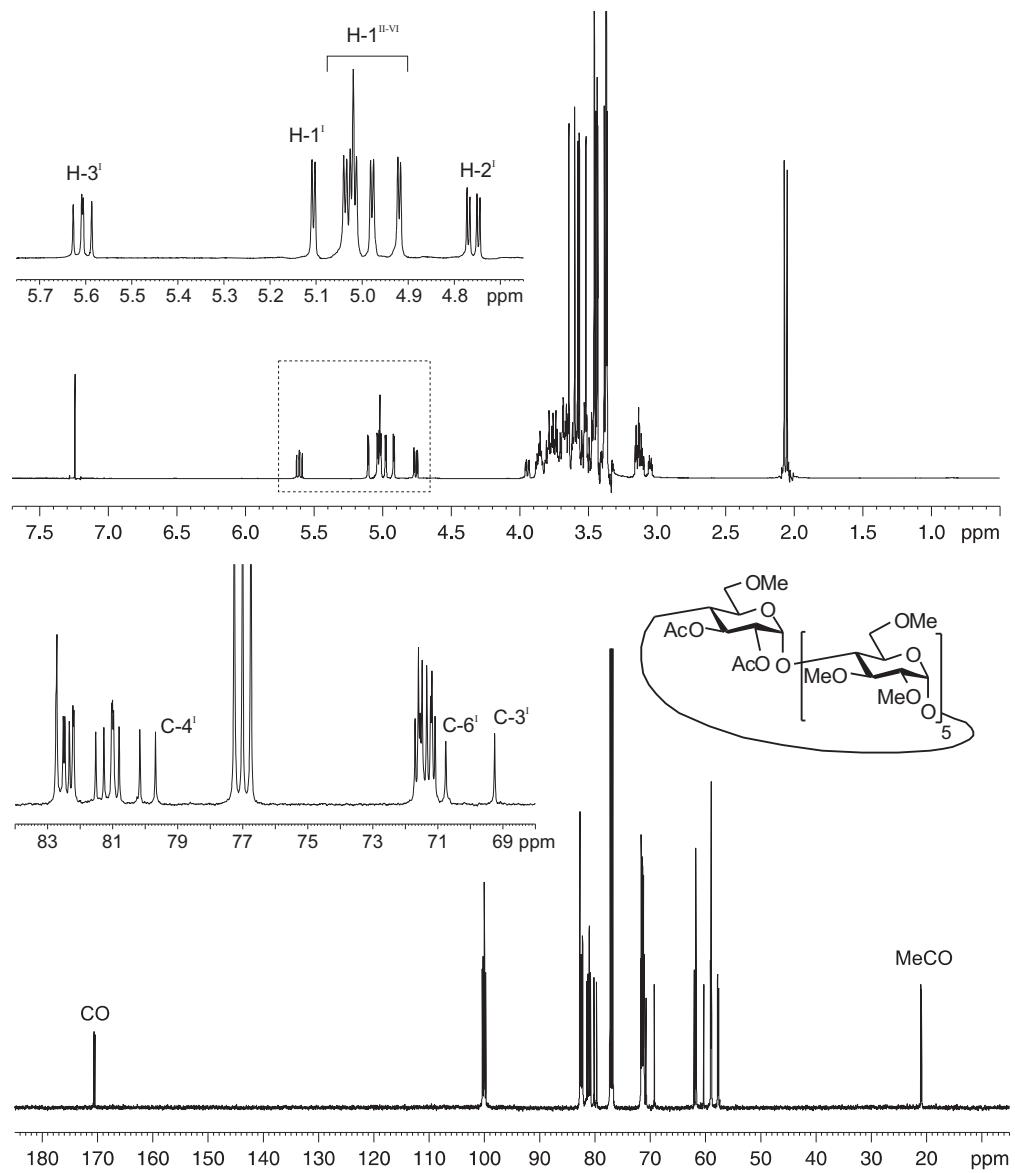


Figure S44. ^1H and ^{13}C NMR spectra (500 MHz, 125.7 MHz, CD_3Cl) of **48**.

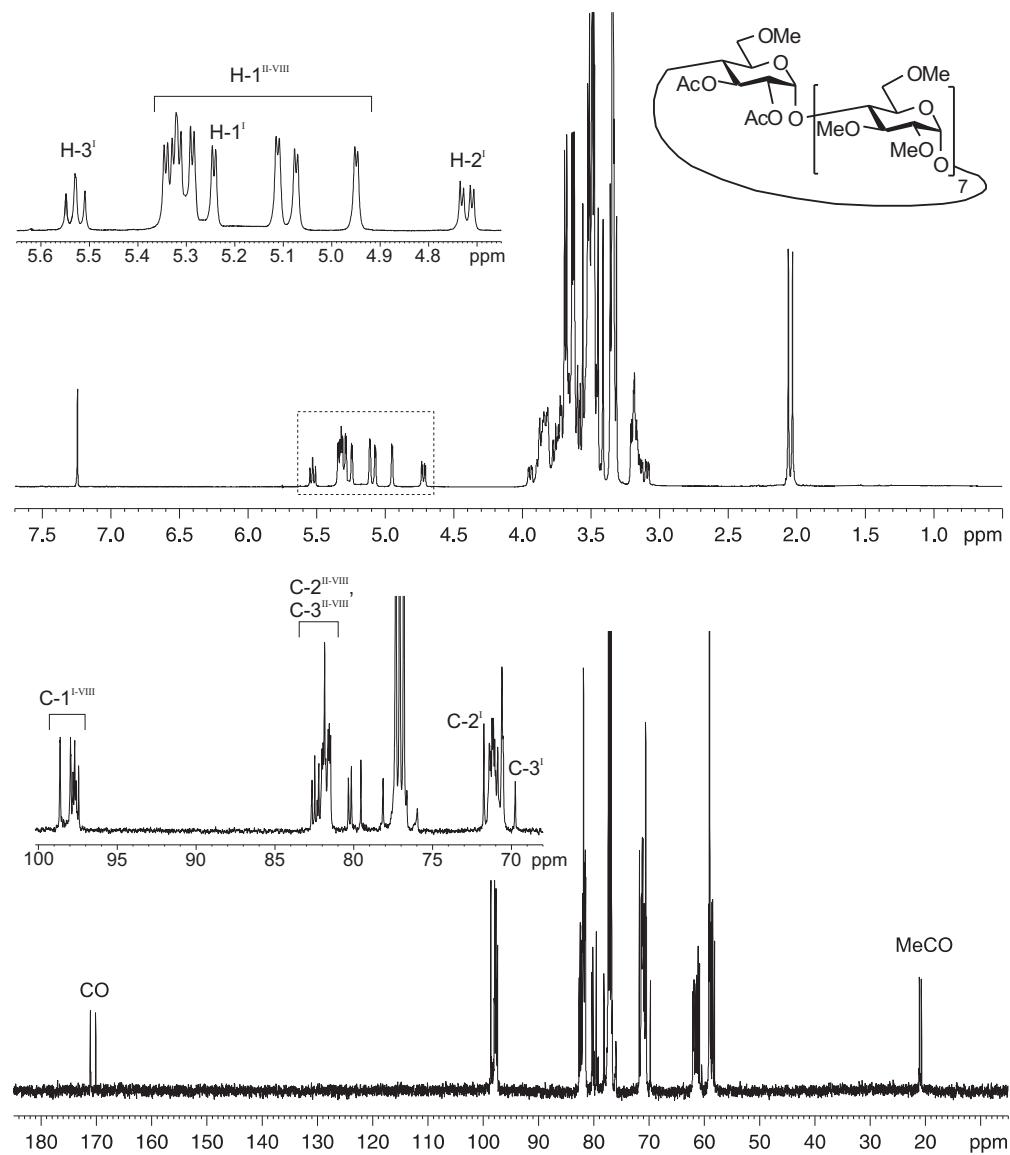


Figure S45. ¹H, 1D TOCSY and ¹³C NMR spectra (500 MHz, 125.7 MHz, CDCl₃) of compound 53.

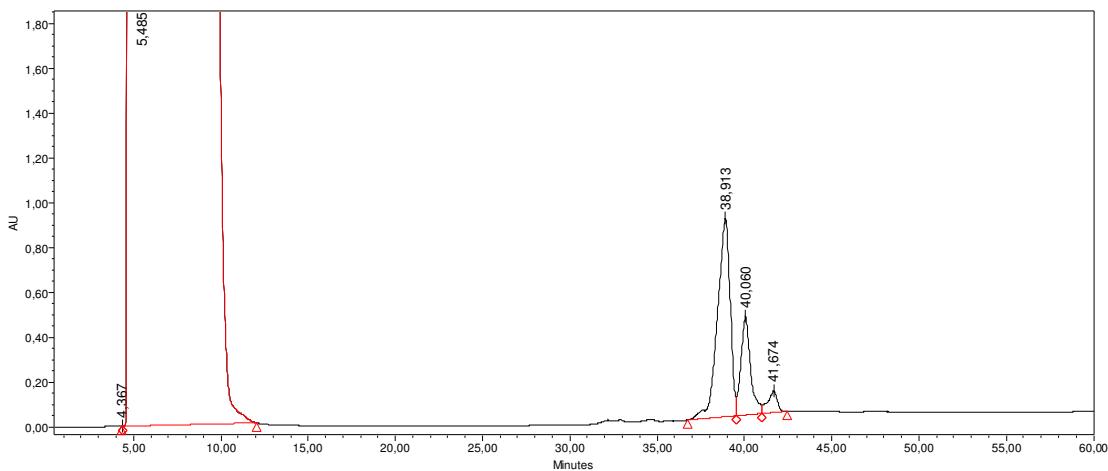


Figure S46. HPLC chromatogram of compounds **38** (t_R 36.9 min), **39** (t_R 40.0 min) and **40** (t_R 41.7 min).

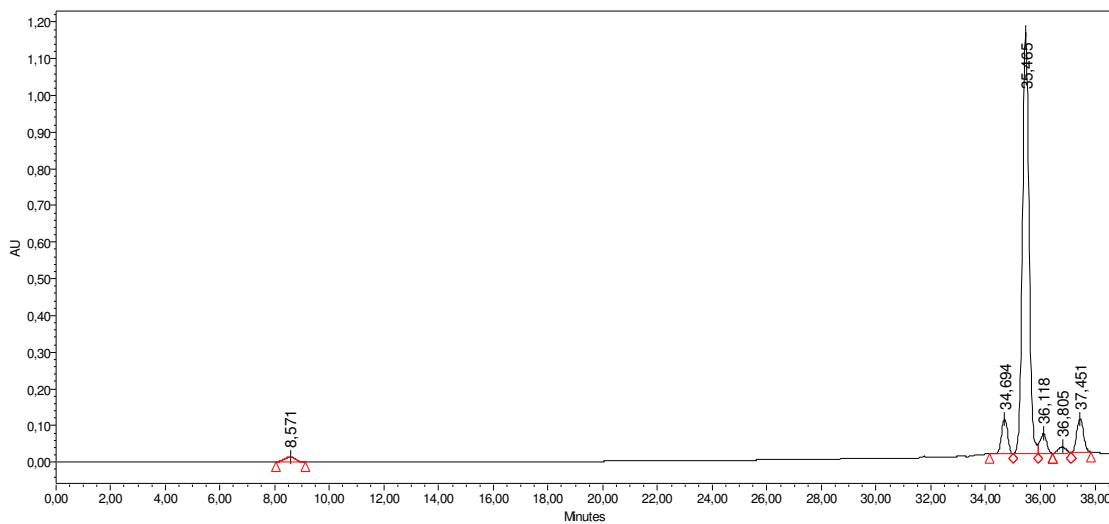


Figure S47. HPLC chromatogram of compounds **44** (t_R 35.4 min) and **45** (t_R 37.4 mi n).

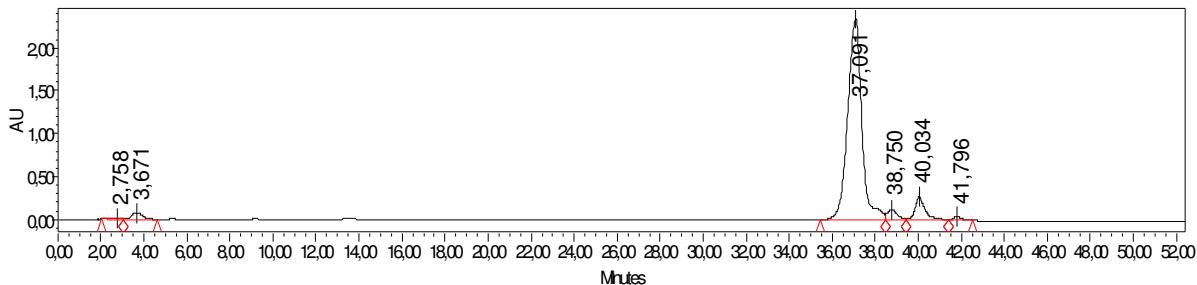


Figure S48. HPLC chromatogram of compounds **49** (t_R 37.1 min) and **50** (t_R 40.0 min).