

Supporting Information

Surfactant-Mediated Thioglycosylation of 1-Hydroxy Sugar in Water

Trichada Ratthachag,[‡] Supanat Buntasana,[†] Tirayut Vilaiwan,[‡] and Panuwat Padungros^{*†}

[†]Green Chemistry for Fine Chemical Productions STAR, Department of Chemistry, Faculty of Science, Chulalongkorn University, Phayathai Road, Pathumwan, Bangkok 10330, Thailand

[‡]Organic Synthesis Research Unit, Department of Chemistry, Faculty of Science, Chulalongkorn University, Phayathai Road, Pathumwan, Bangkok 10330, Thailand

*E-mail: panuwat.p@chula.ac.th

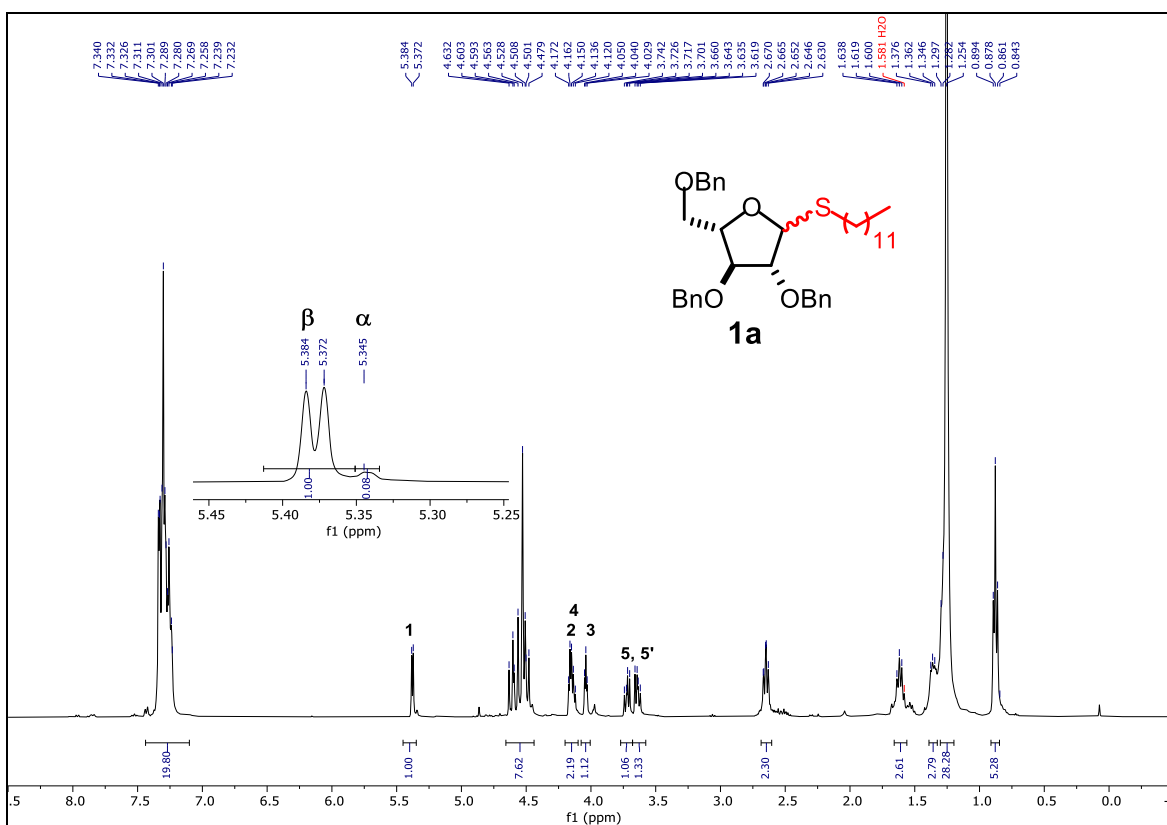


Figure S1. $^1\text{H NMR}$ spectrum of dodecyl 2,3,5-tri-O-benzyl-1-thio- α,β -L-arabinofuranoside (**1a**) (400 MHz, CDCl_3).

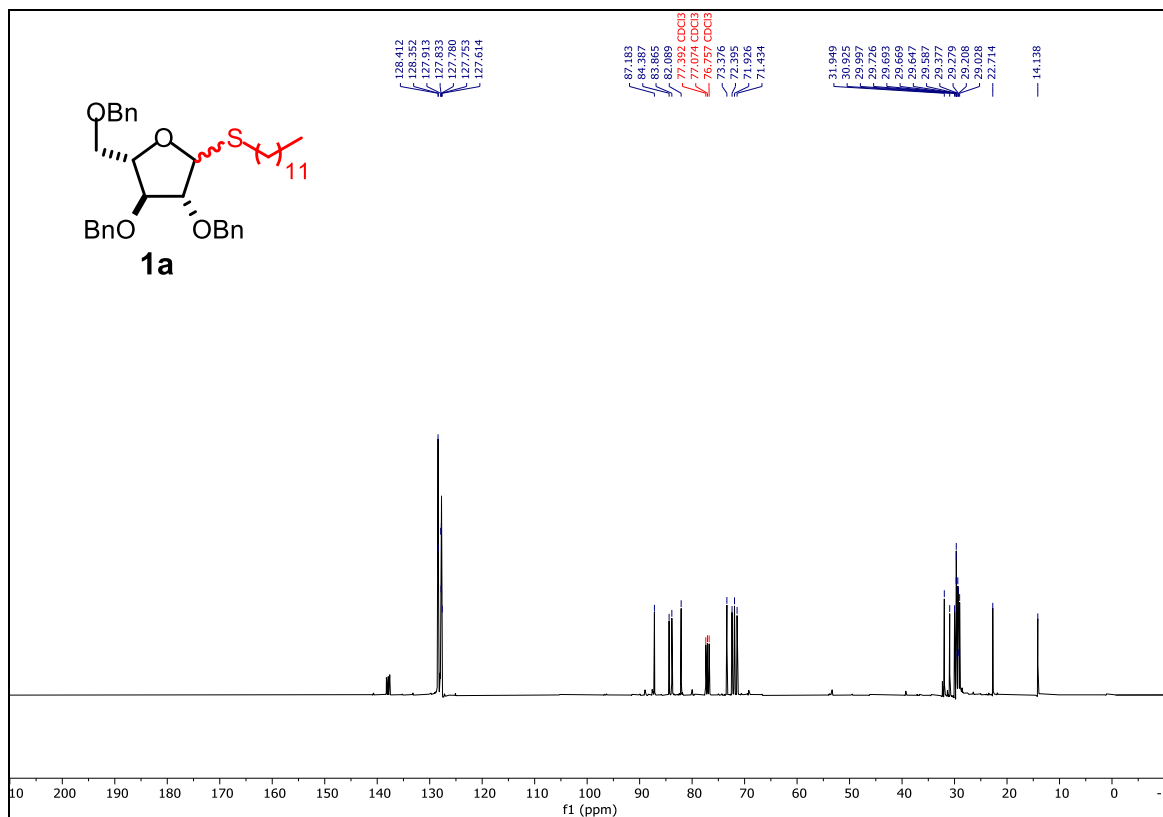


Figure S2. $^{13}\text{C NMR}$ spectrum of dodecyl 2,3,5-tri-O-benzyl-1-thio- α,β -L-arabinofuranoside (**1a**) (101 MHz, CDCl_3).

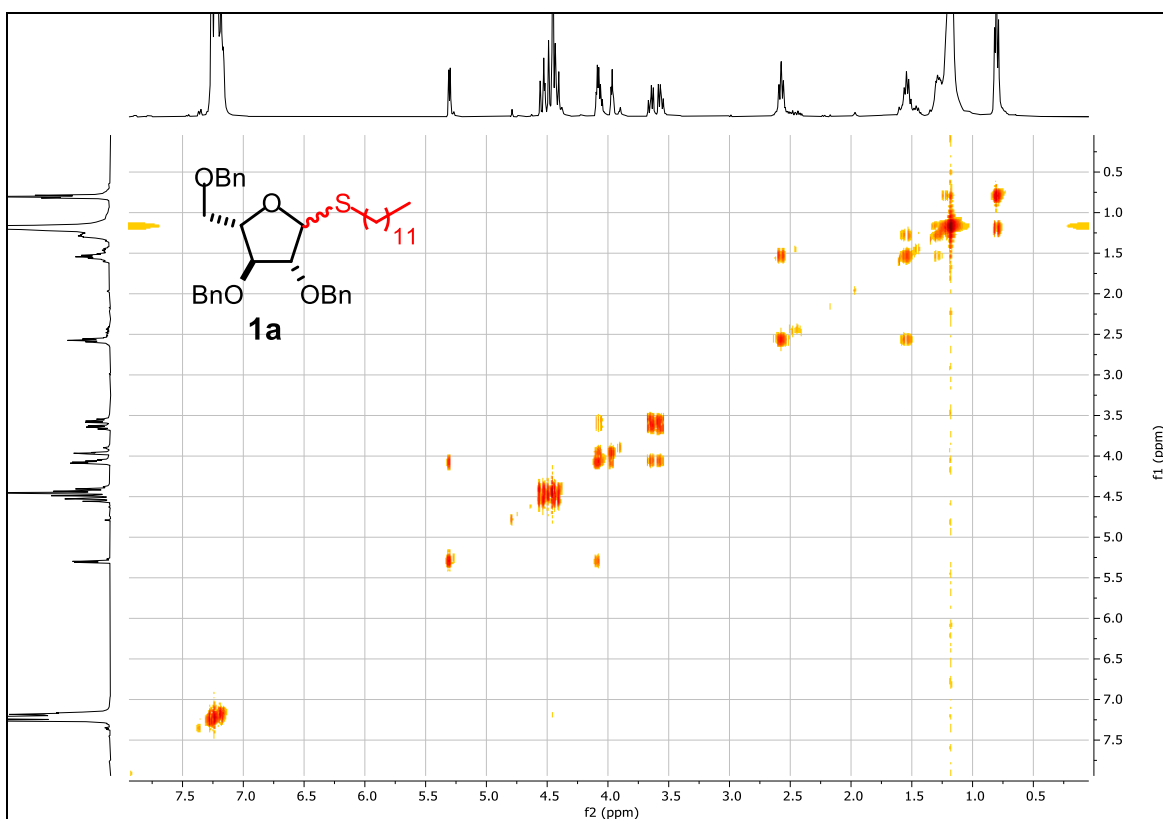


Figure S3. COSY spectrum of dodecyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**1a**) (400 MHz, CDCl₃).

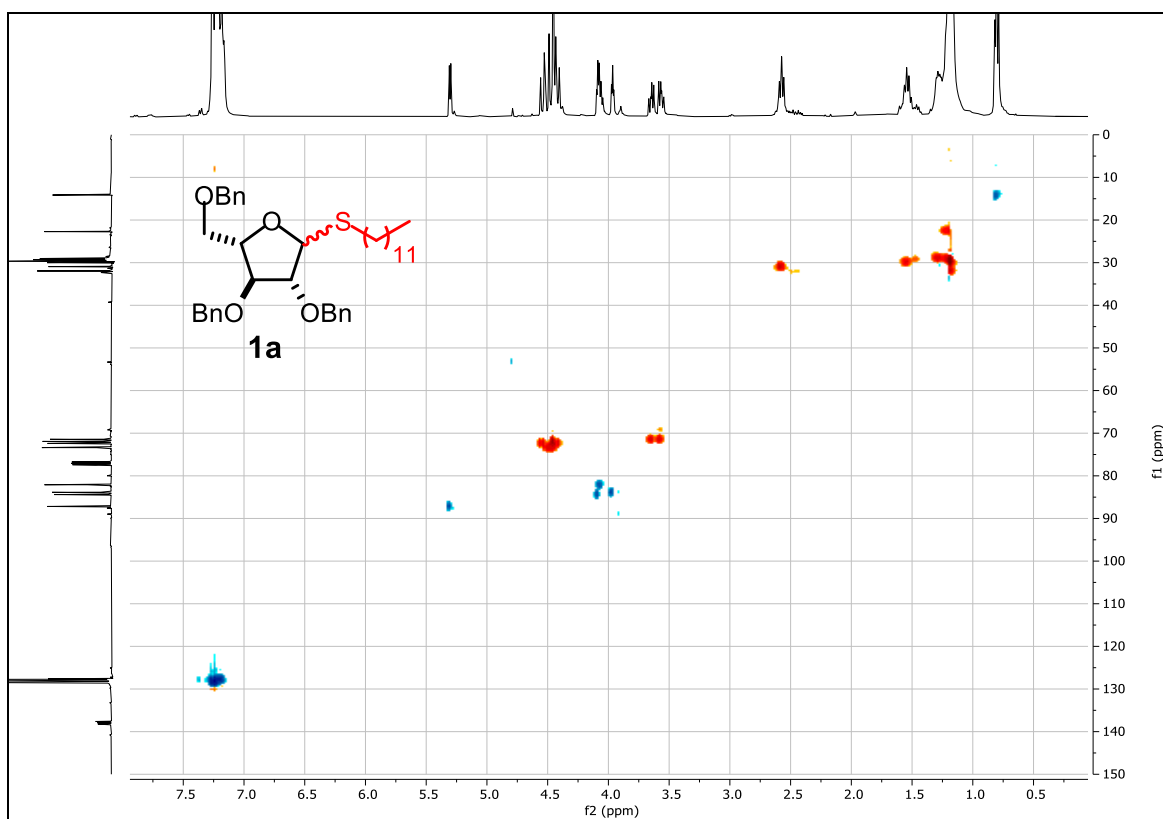


Figure S4. HSQC spectrum of dodecyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**1a**) (400 MHz, CDCl₃).

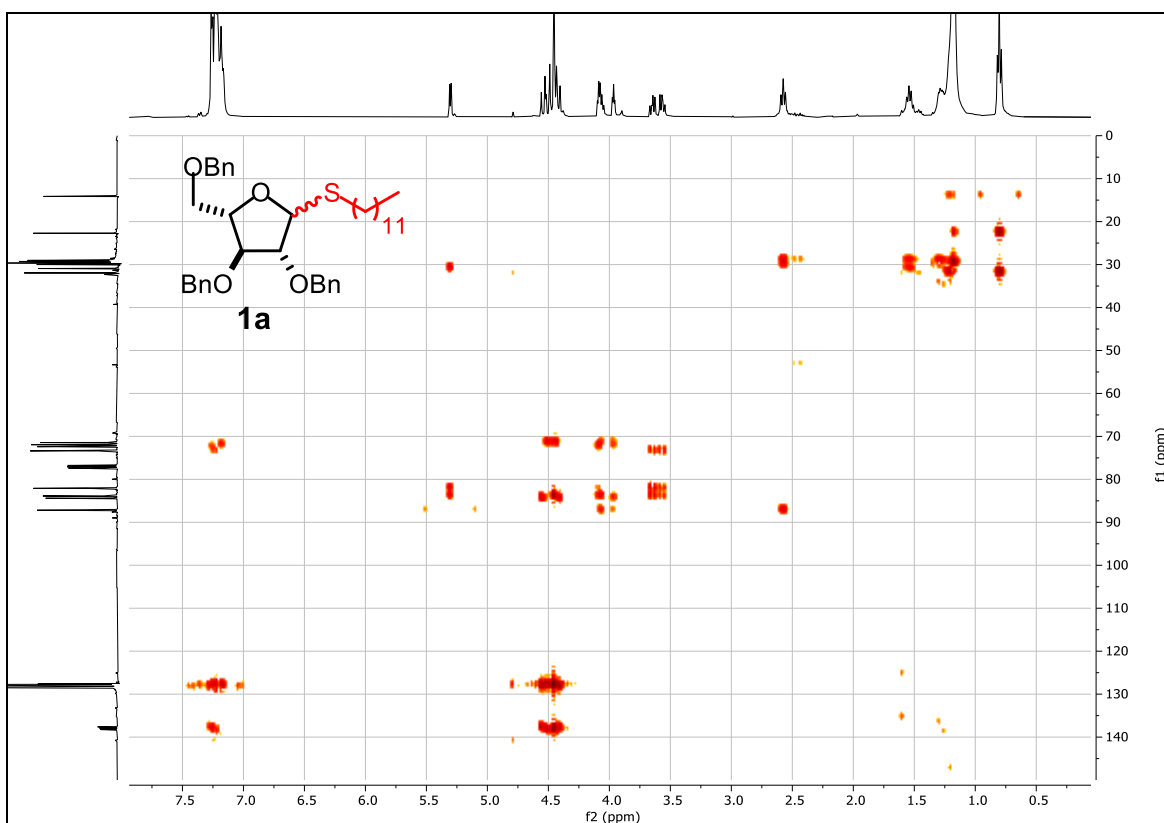


Figure S5. HMBC spectrum of dodecyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**1a**) (400 MHz, CDCl₃).

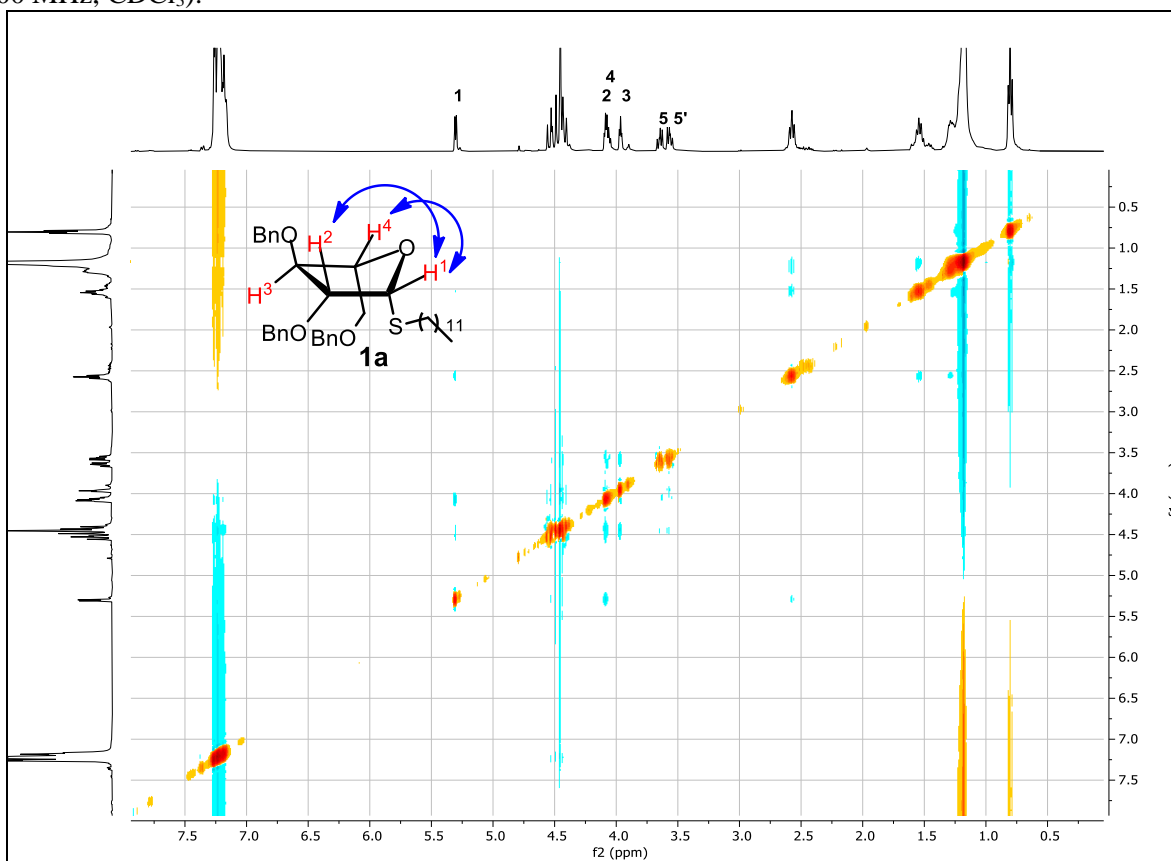


Figure S6. NOESY spectrum of dodecyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**1a**) (400 MHz, CDCl₃).

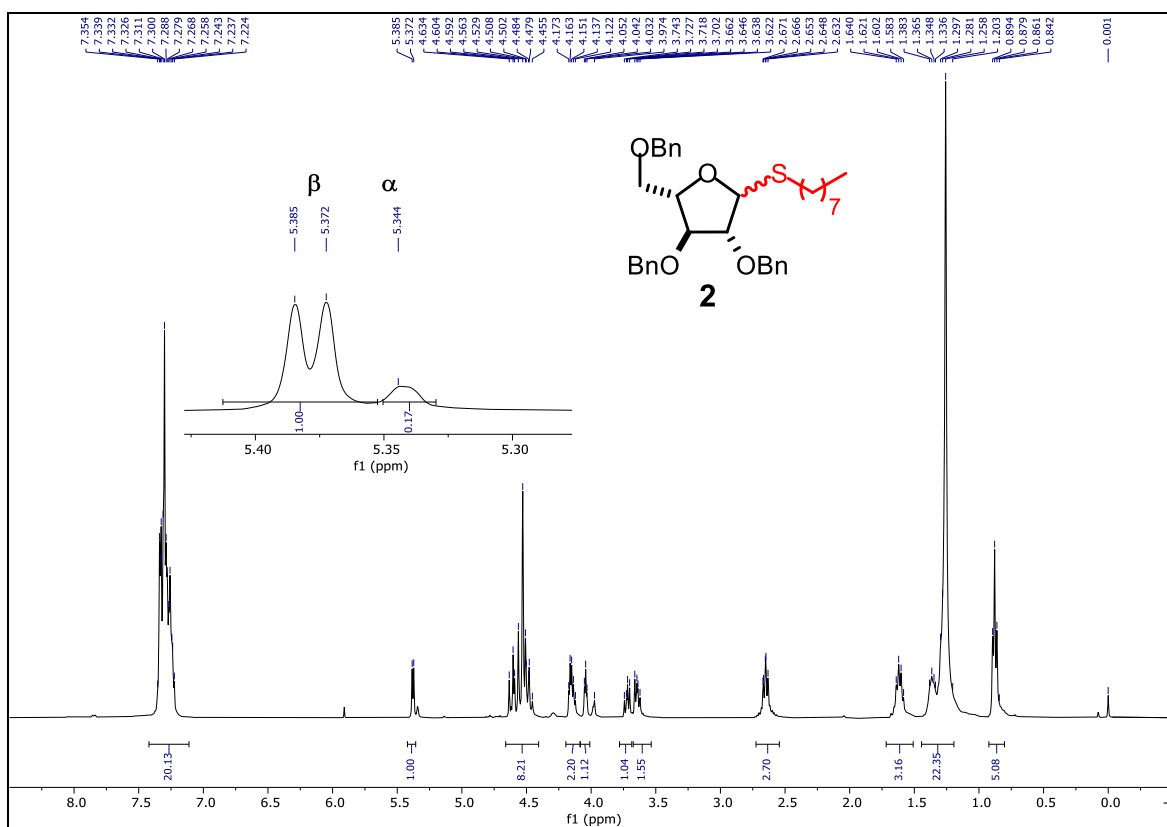


Figure S7. ¹H NMR spectrum of octyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**2**) (400 MHz, CDCl₃).

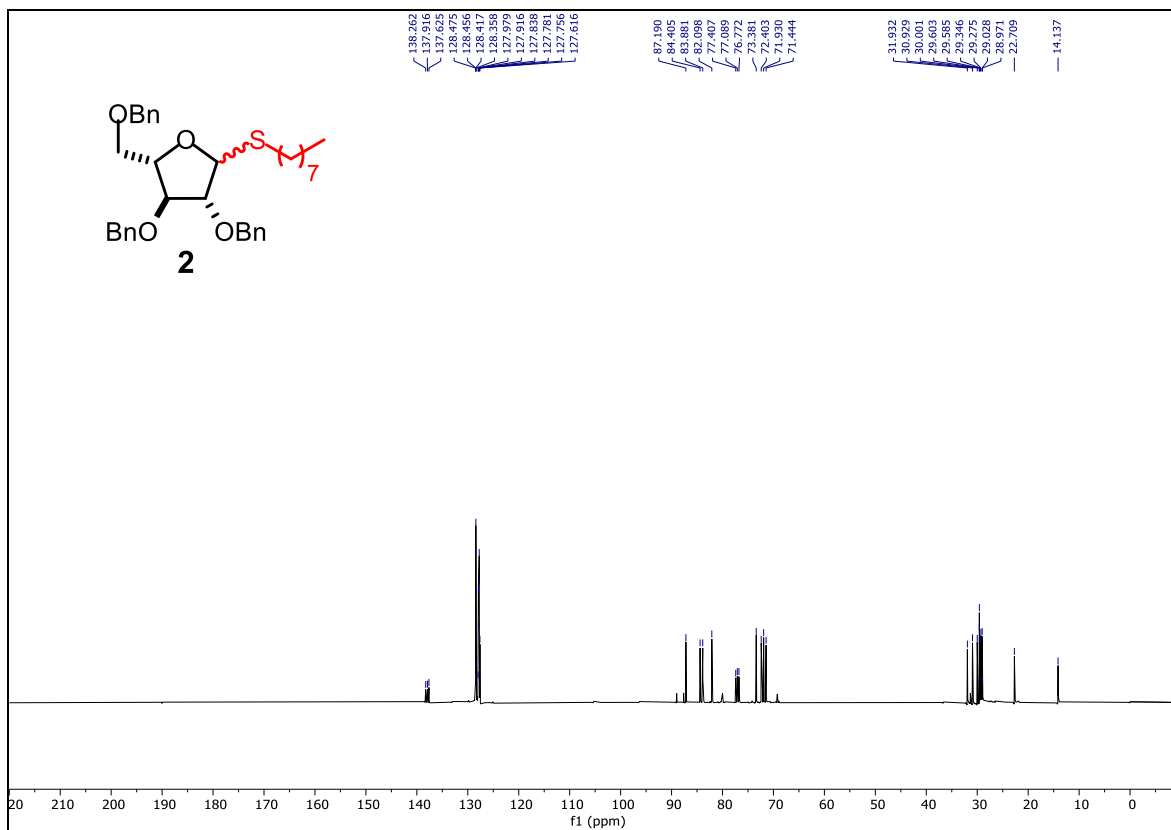


Figure S8. ¹³C NMR spectrum of octyl 2,3,5-tri-*O*-benzyl- α,β -L-thioarabinofuranoside (**2**) (101 MHz, CDCl₃).

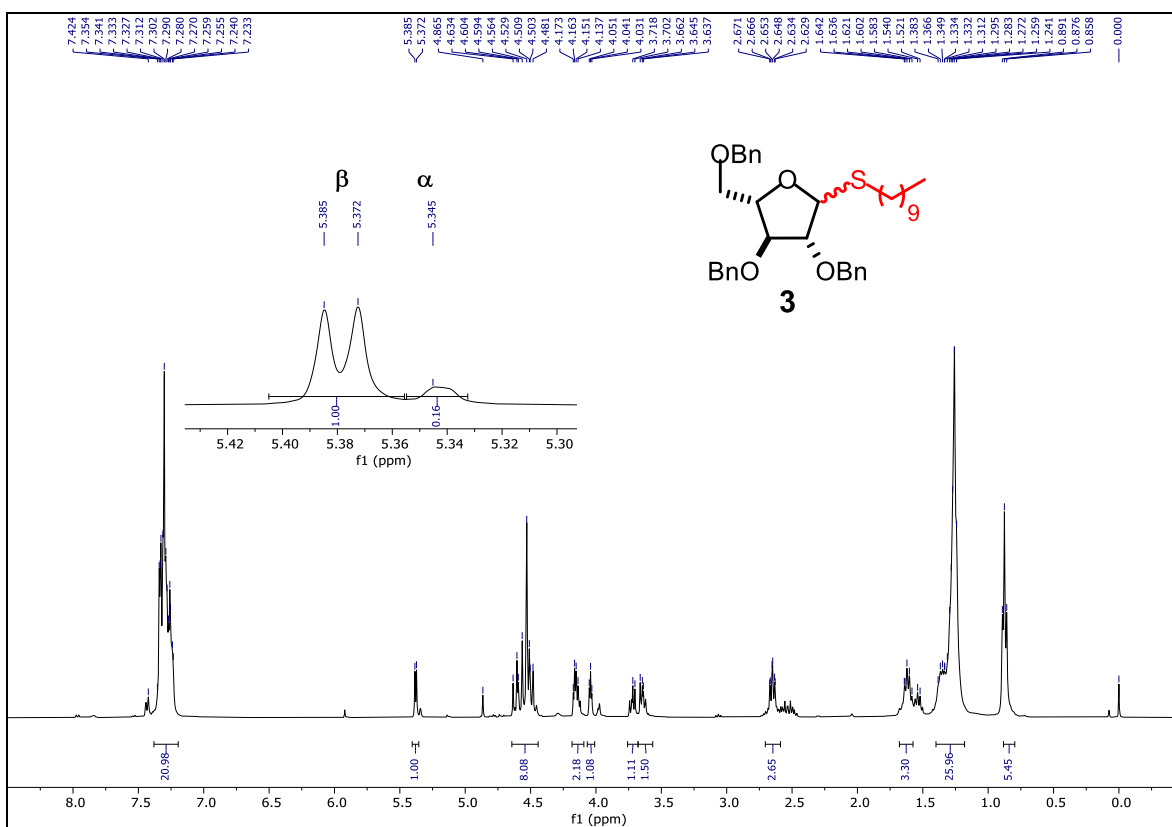


Figure S9. ^1H NMR spectrum of decyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**3**) (400 MHz, CDCl_3).

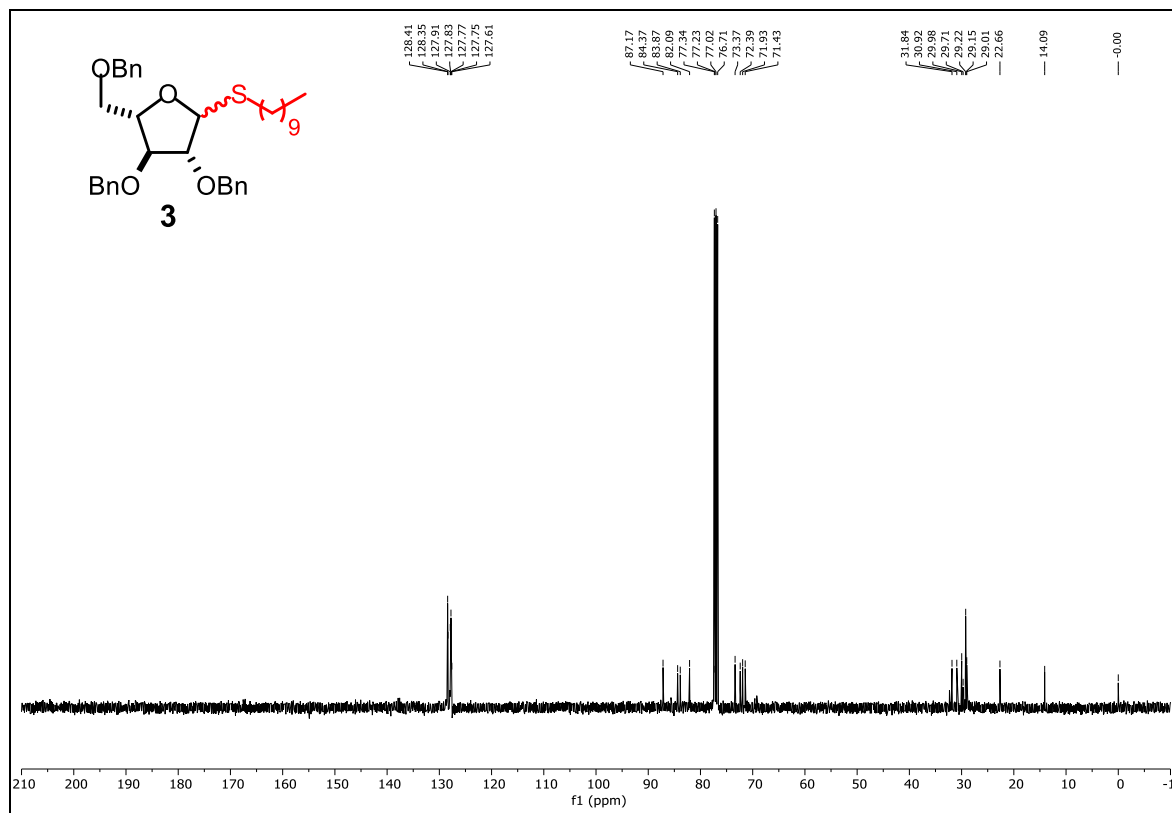


Figure S10. ^{13}C NMR spectrum of decyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**3**) (101 MHz, CDCl_3).

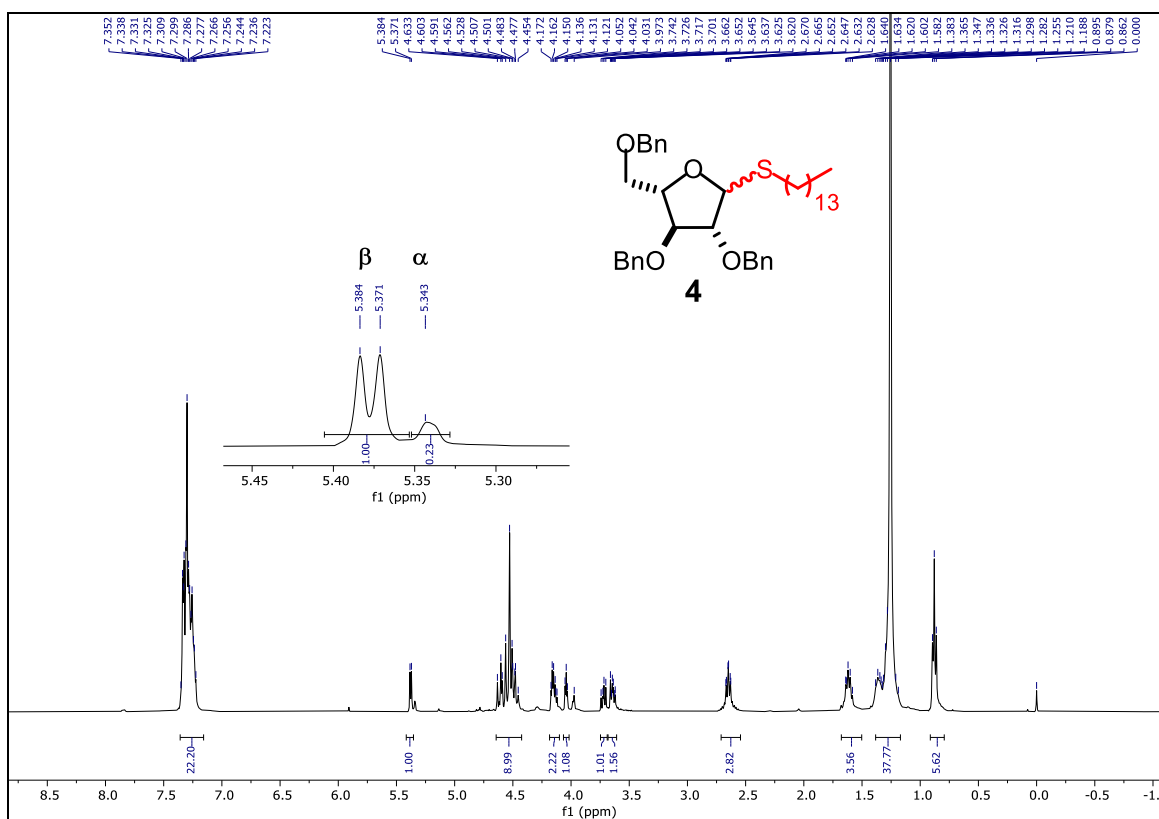


Figure S11. ^1H NMR spectrum of tetradecyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**4**) (400 MHz, CDCl_3).

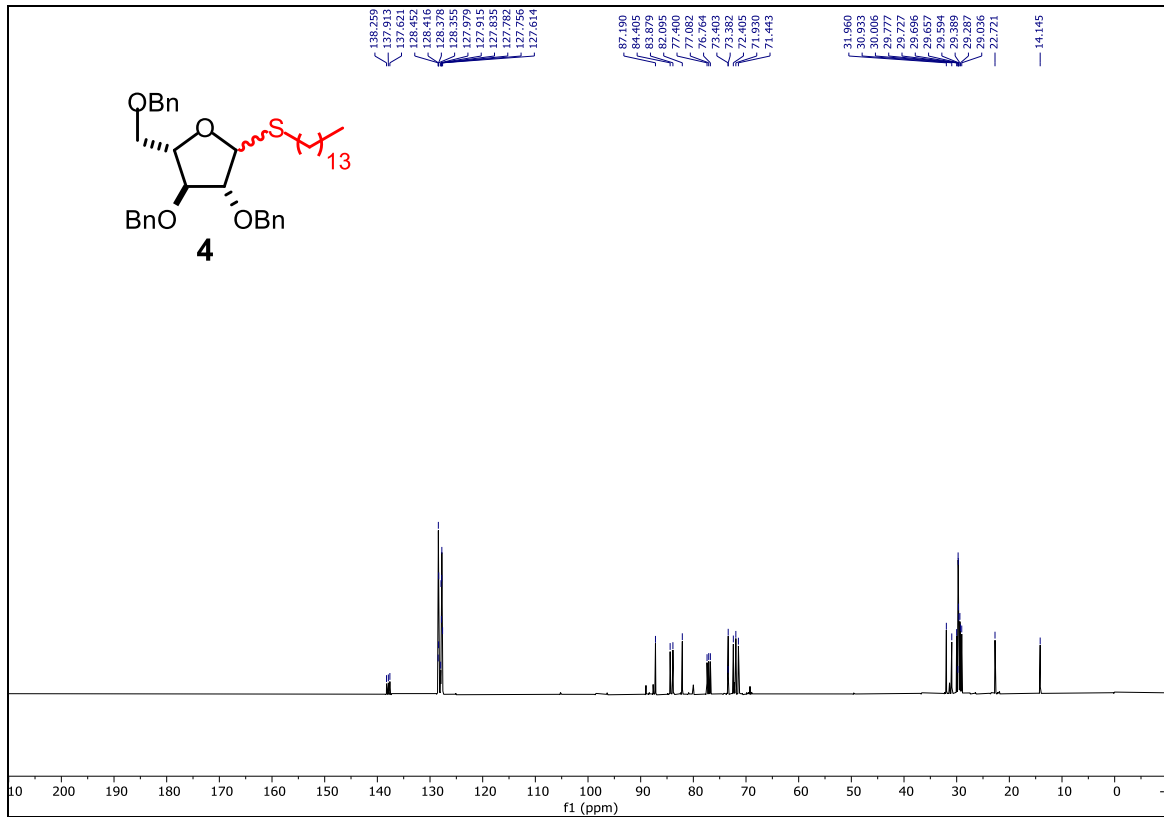


Figure S12. ^{13}C NMR spectrum of tetradecyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**4**) (101 MHz, CDCl_3).

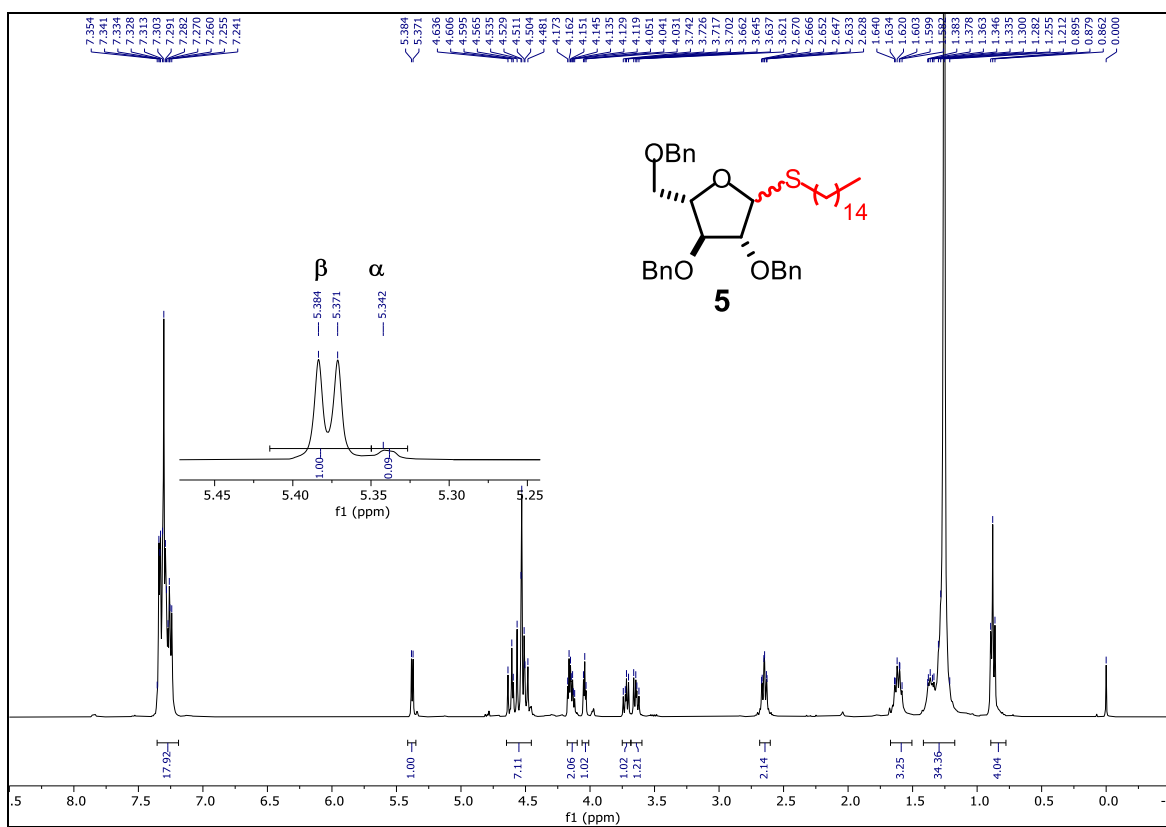


Figure S13. ^1H NMR spectrum of pentadecyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**5**) (400 MHz, CDCl_3).

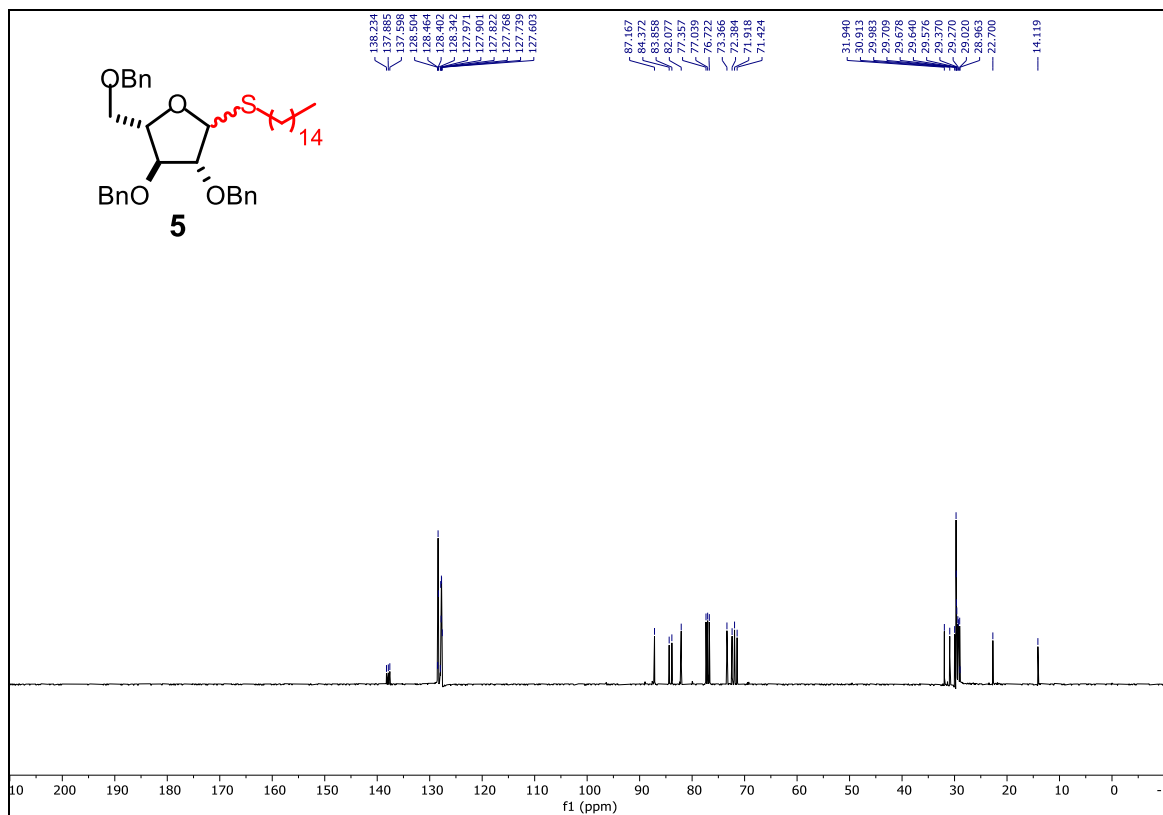


Figure S14. ^{13}C NMR spectrum of pentadecyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**5**) (101 MHz, CDCl_3).

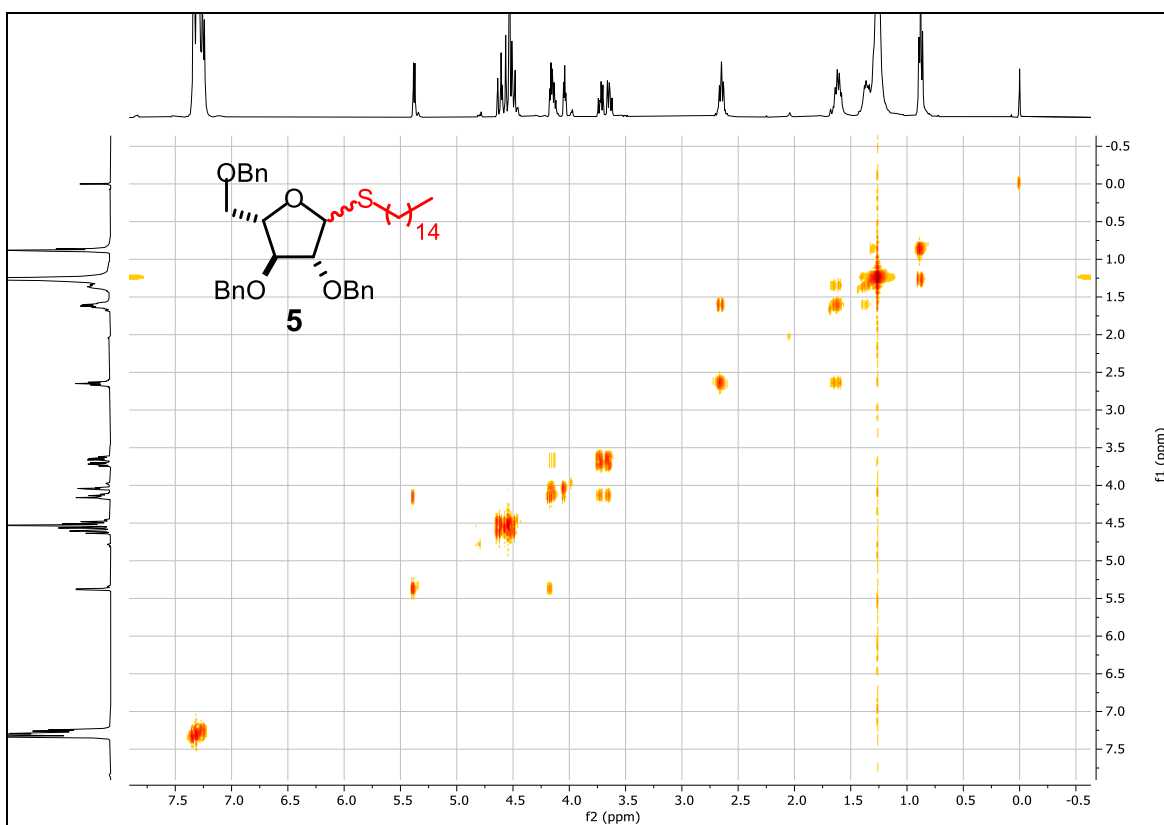


Figure S15. COSY spectrum of pentadecyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**5**) (400 MHz, CDCl_3).

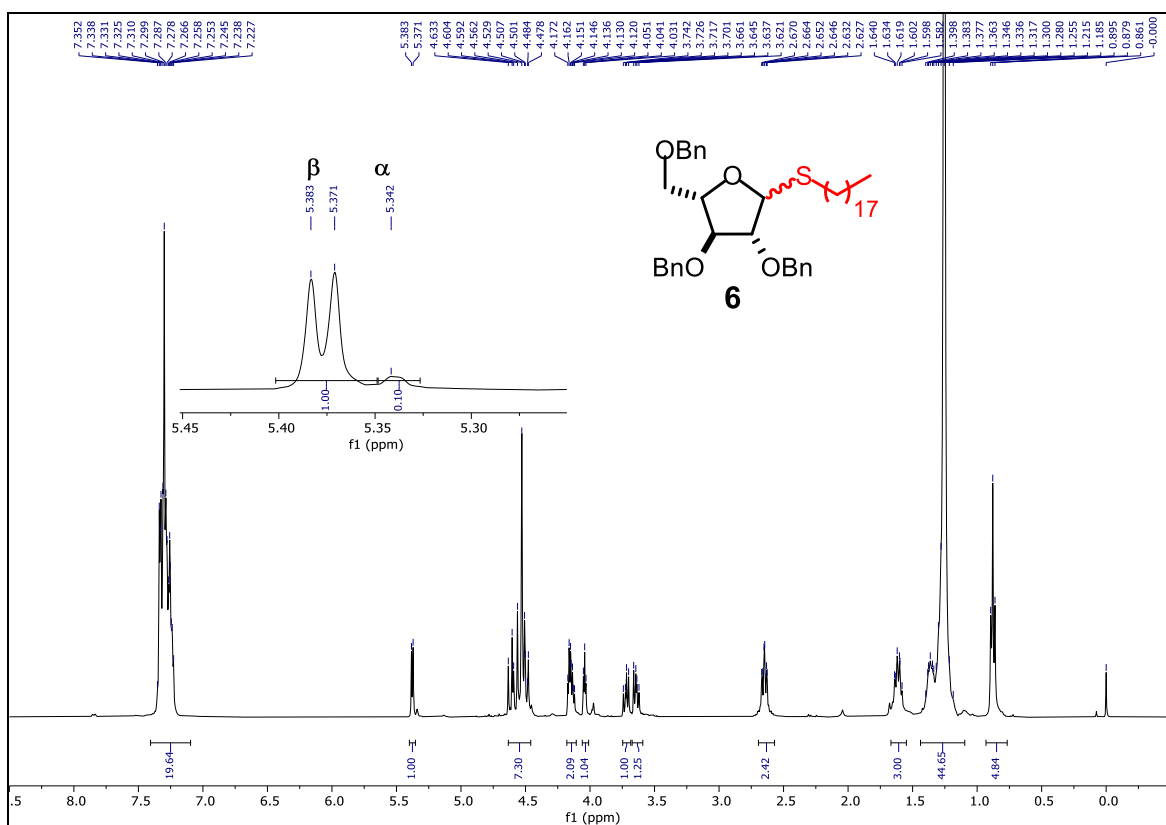


Figure S16. ¹H NMR spectrum of octadecyl 2,3,5-tri-O-benzyl-1-thio- α,β -L-arabinofuranoside (**6**) (400 MHz, CDCl₃).

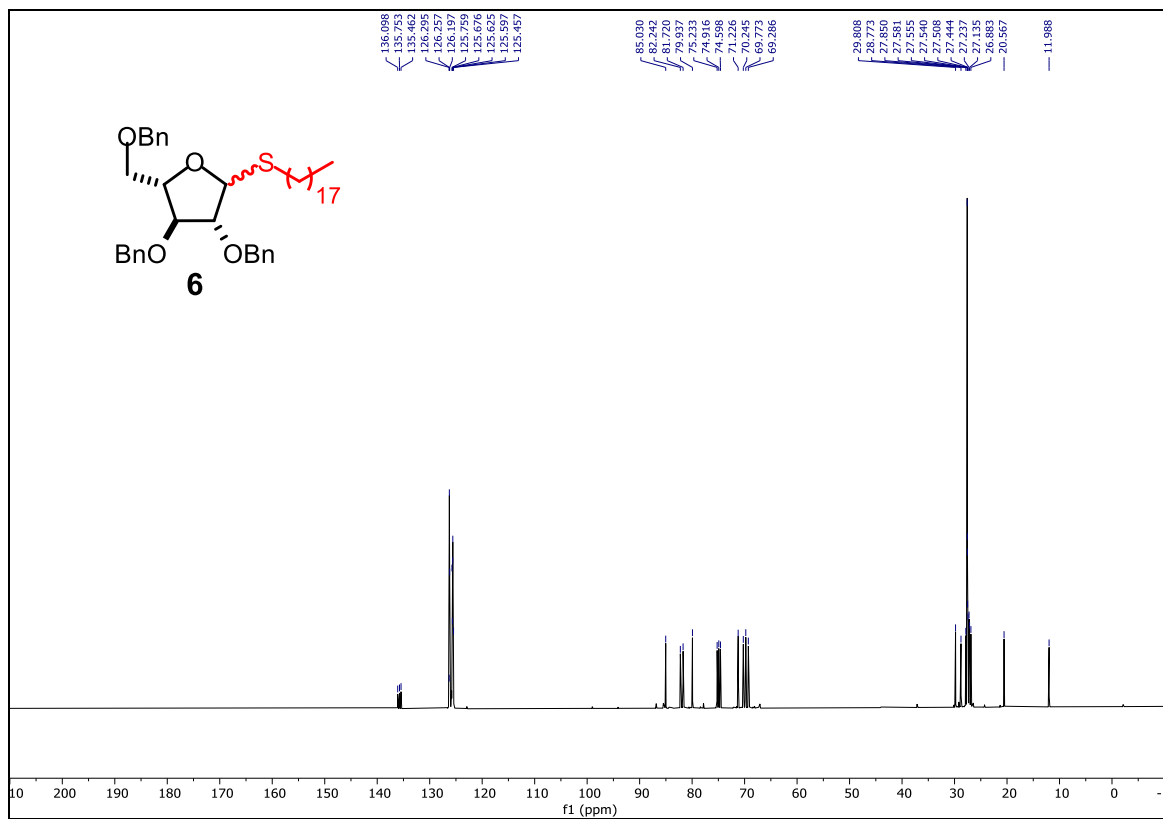


Figure S17. ¹³C NMR spectrum of octadecyl 2,3,5-tri-O-benzyl-1-thio- α,β -L-arabinofuranoside (**6**) (101 MHz, CDCl₃).

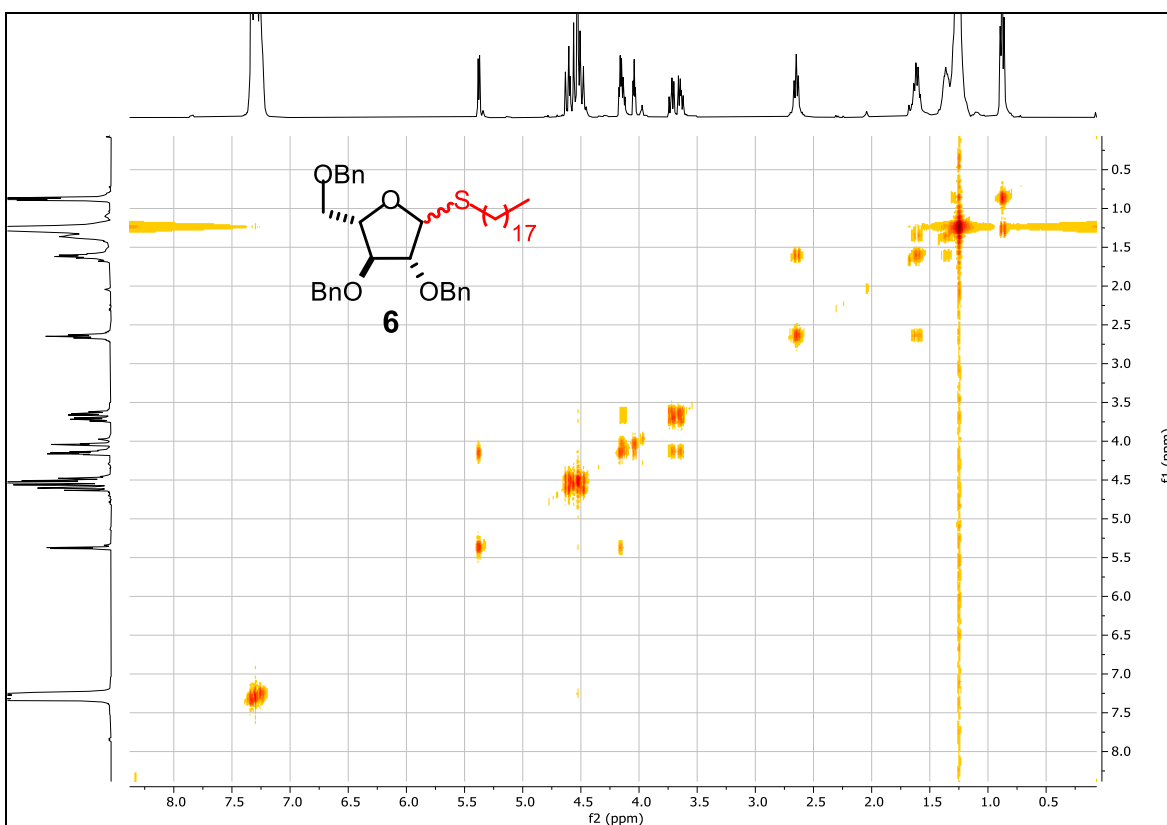


Figure S18. COSY spectrum of octadecyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**6**) (400 MHz, CDCl_3).

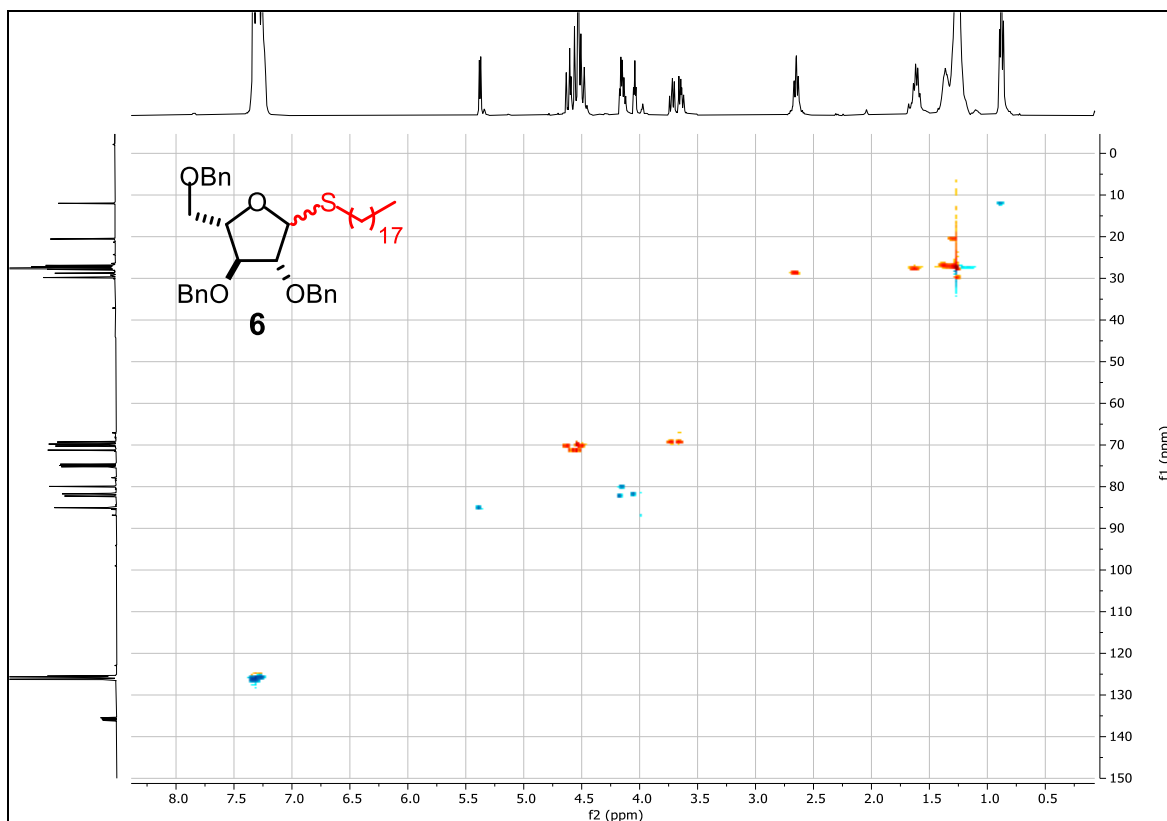


Figure S19. HSQC spectrum of octadecyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**6**) (400 MHz, CDCl_3).

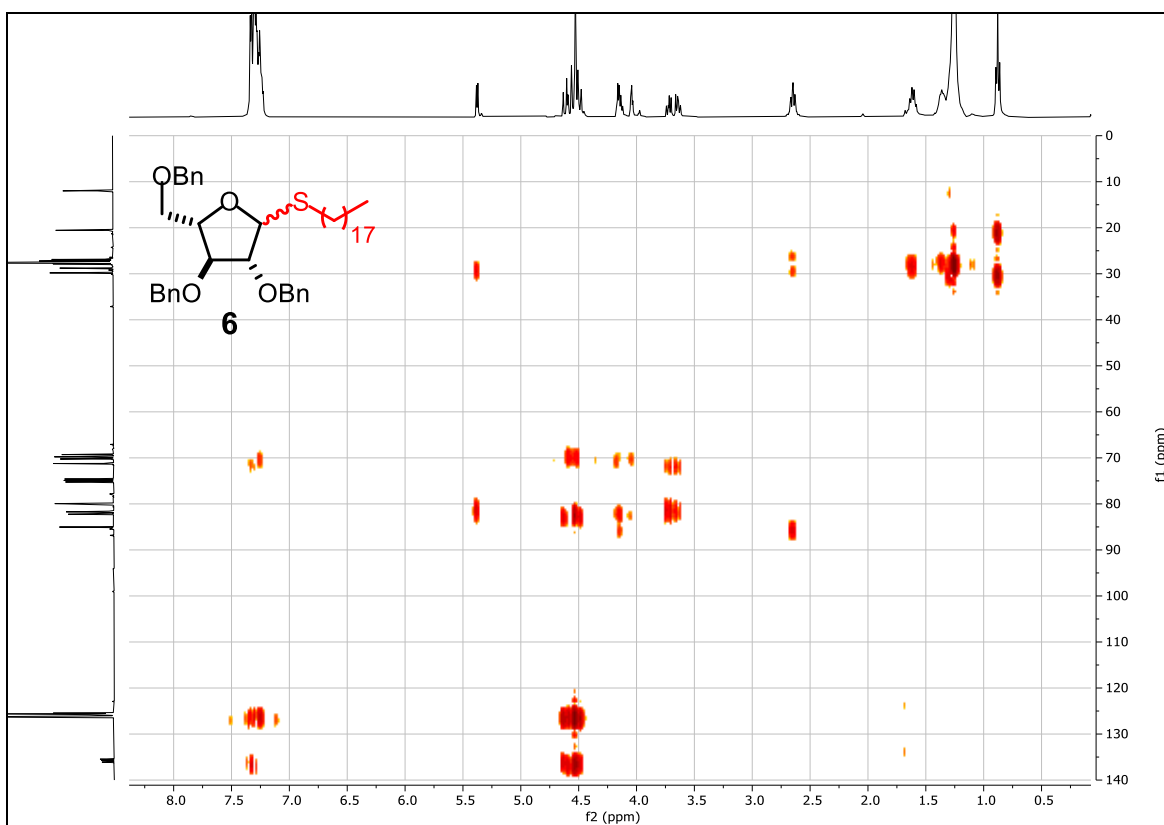


Figure S20. HMBC spectrum of octadecyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**6**) (400 MHz, CDCl₃).

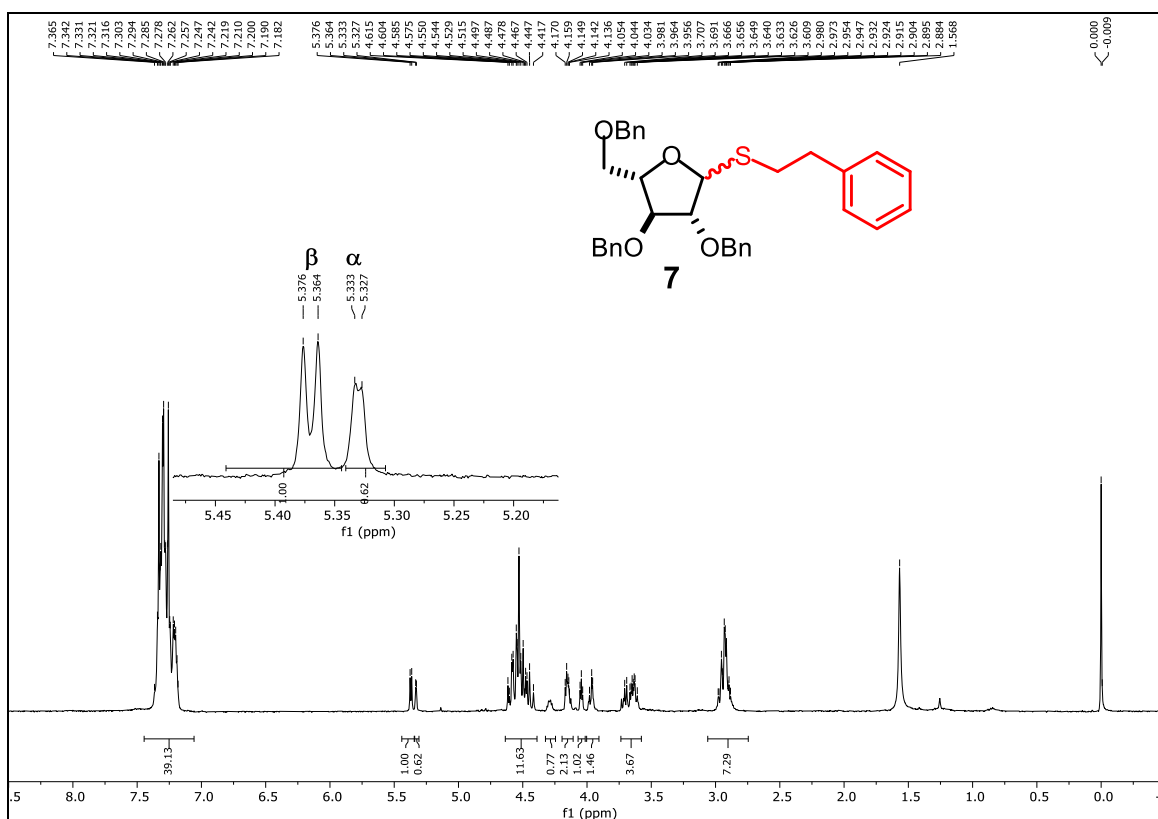


Figure S21. ^1H spectrum of 2-phenylethyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**7**) (400 MHz, CDCl_3).

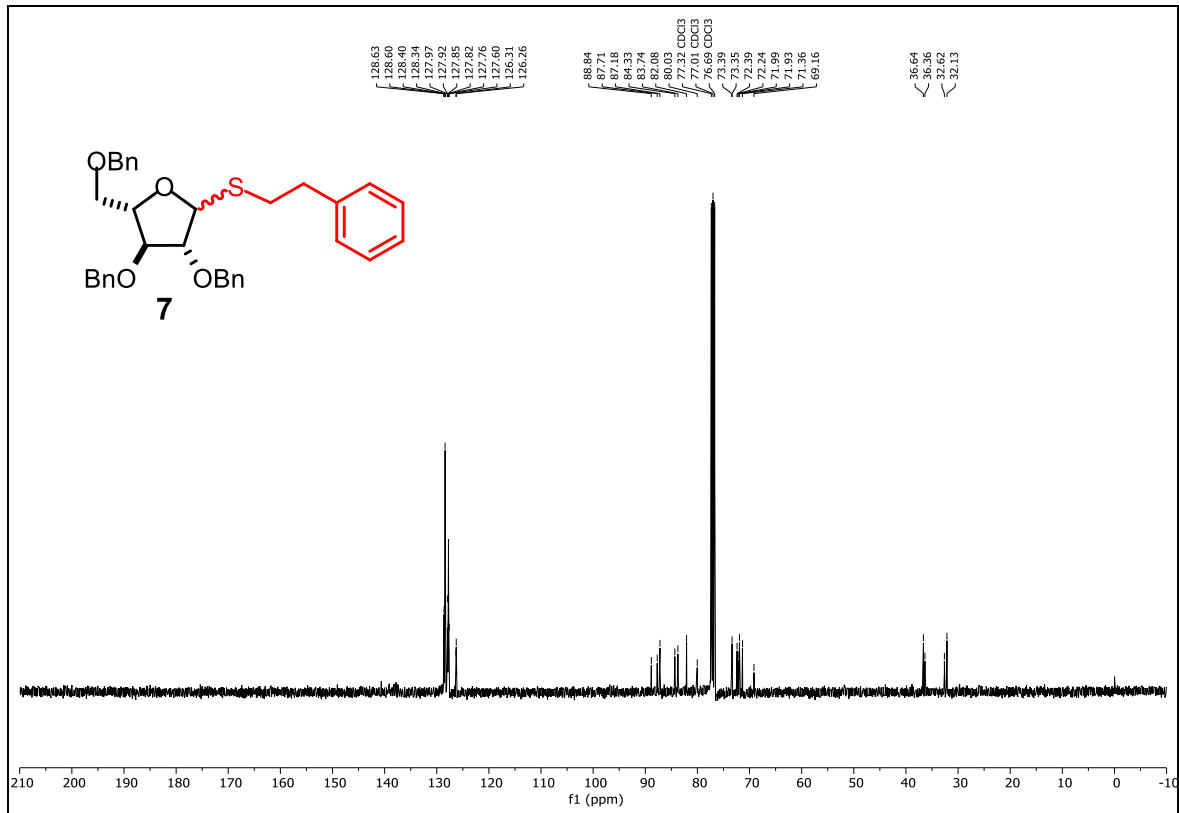


Figure S22. ^{13}C spectrum of 2-phenylethyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**7**) (101 MHz, CDCl_3).

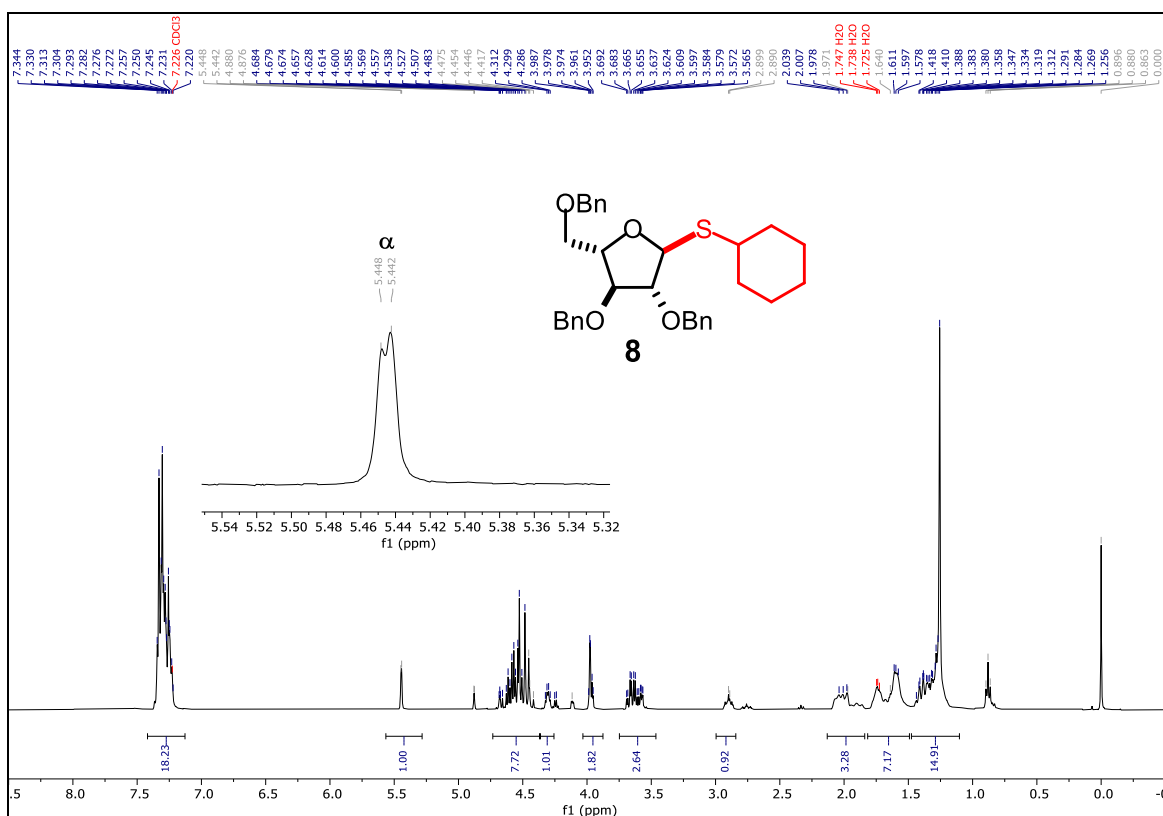


Figure S23. ¹H spectrum of cyclohexyl 2,3,5-tri-*O*-benzyl-1-thio- α -L-arabinofuranoside (**8**) (400 MHz, CDCl₃).

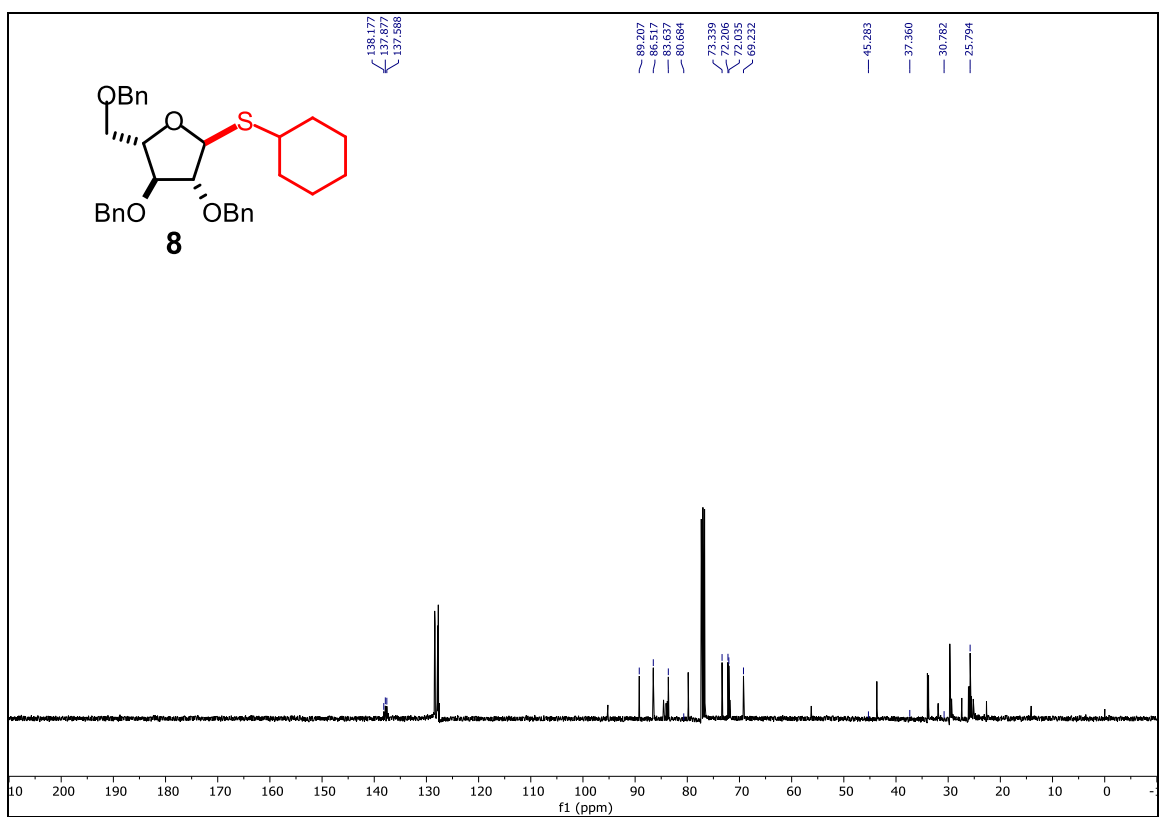


Figure S24. ¹³C spectrum of cyclohexyl 2,3,5-tri-*O*-benzyl-1-thio- α -L-arabinofuranoside (**8**) (101 MHz, CDCl₃).

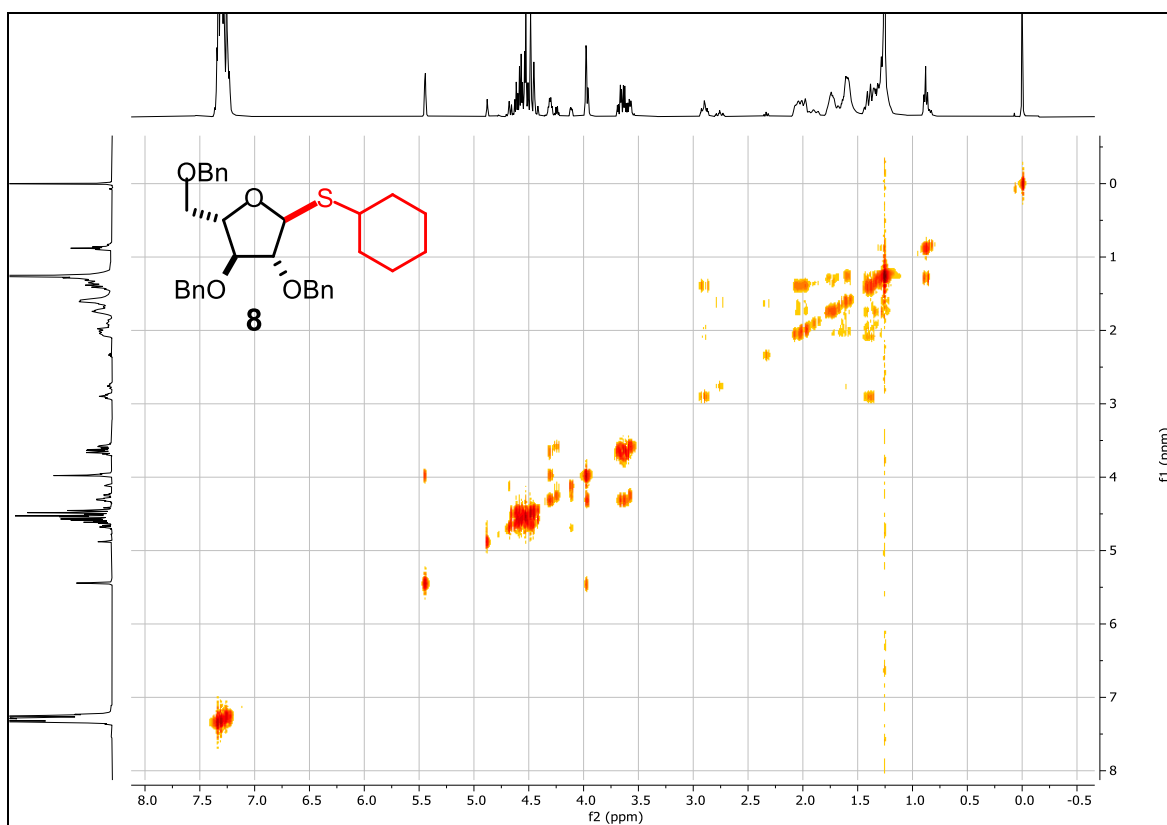


Figure S25. COSY spectrum of cyclohexyl 2,3,5-tri-*O*-benzyl-1-thio- α -L-arabinofuranoside (**8**) (400 MHz, CDCl₃).

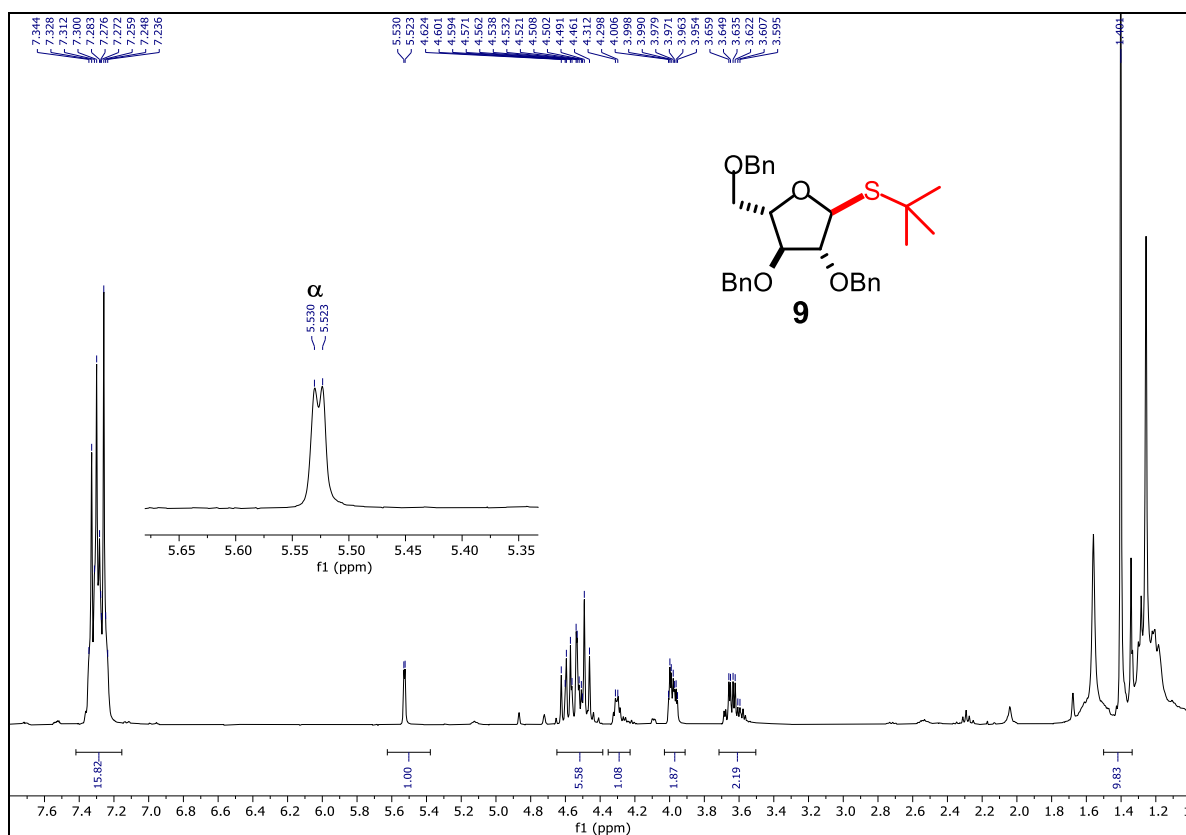


Figure S26. ¹H spectrum of *tert*-butyl 2,3,5-tri-*O*-benzyl-1-thio- α -L-arabinofuranoside (**9**) (400 MHz, CDCl₃).

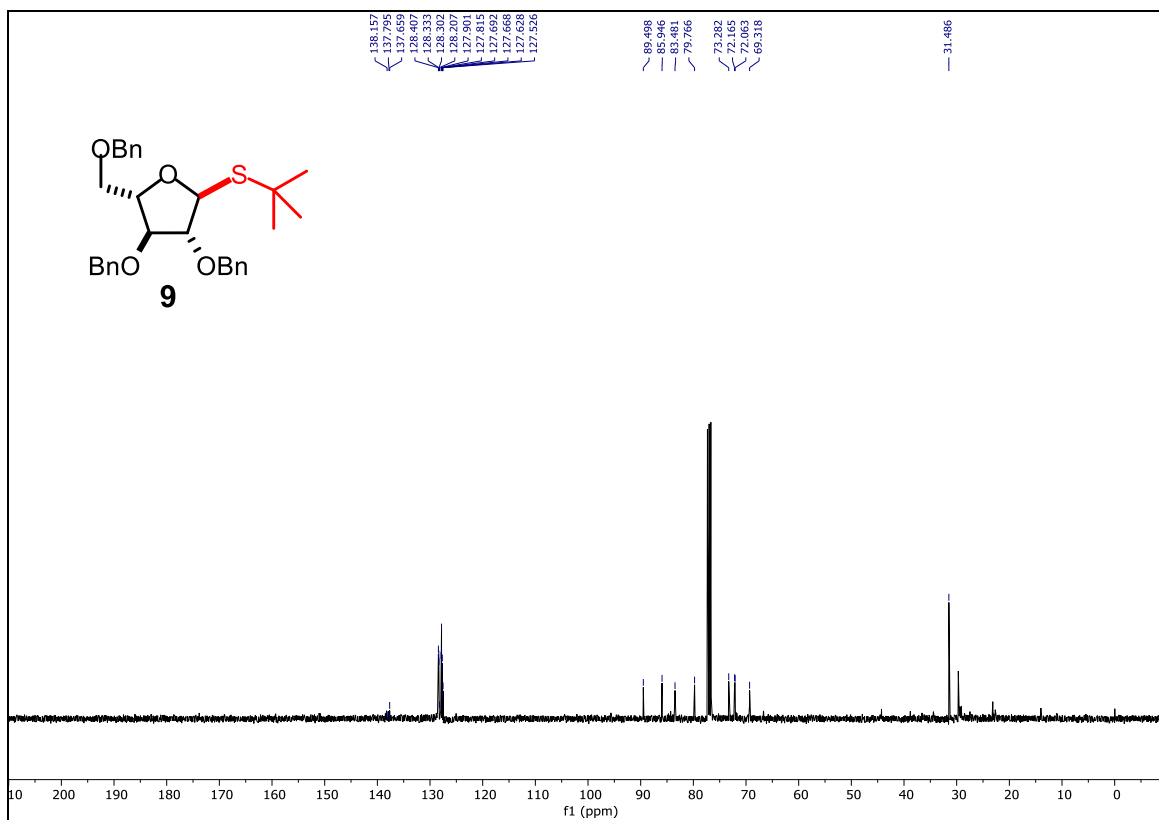


Figure S27. ¹³C spectrum of *tert*-butyl 2,3,5-tri-*O*-benzyl-1-thio- α -L-arabinofuranoside (**9**) (101 MHz, CDCl₃).

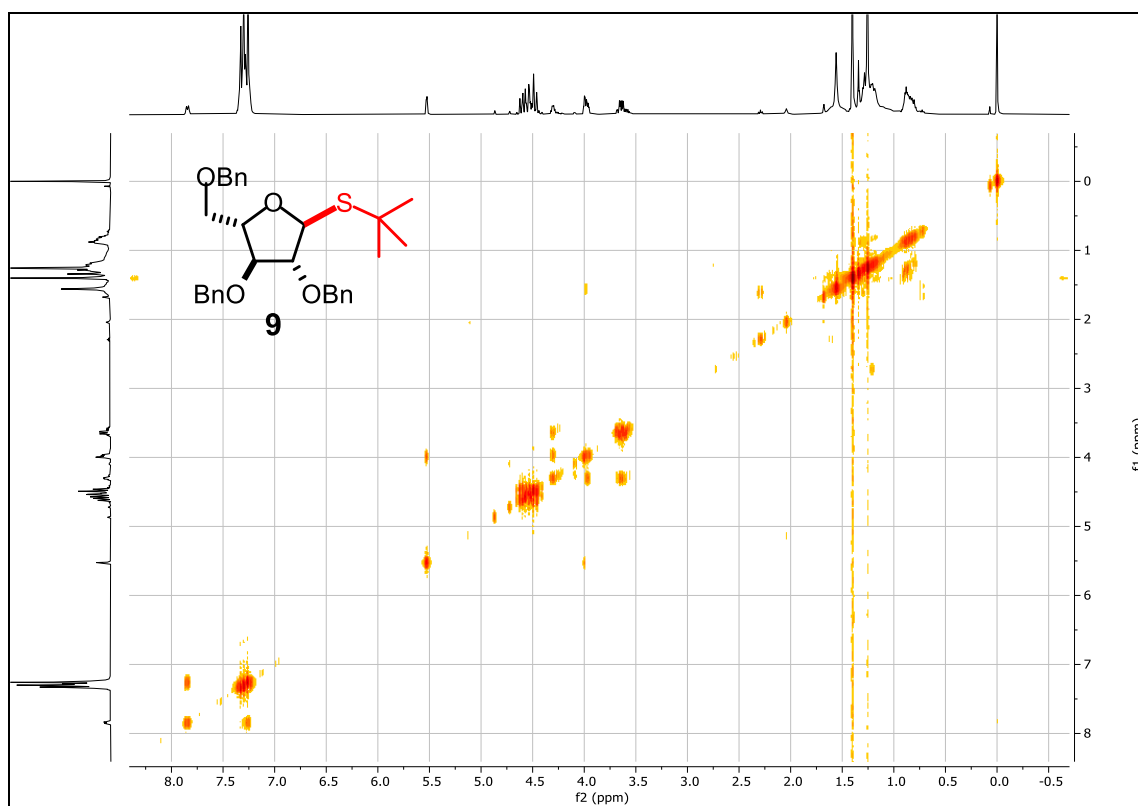


Figure S28. COSY spectrum of *tert*-butyl 2,3,5-tri-*O*-benzyl-1-thio- α -L-arabinofuranoside (**9**) (400 MHz, CDCl₃).

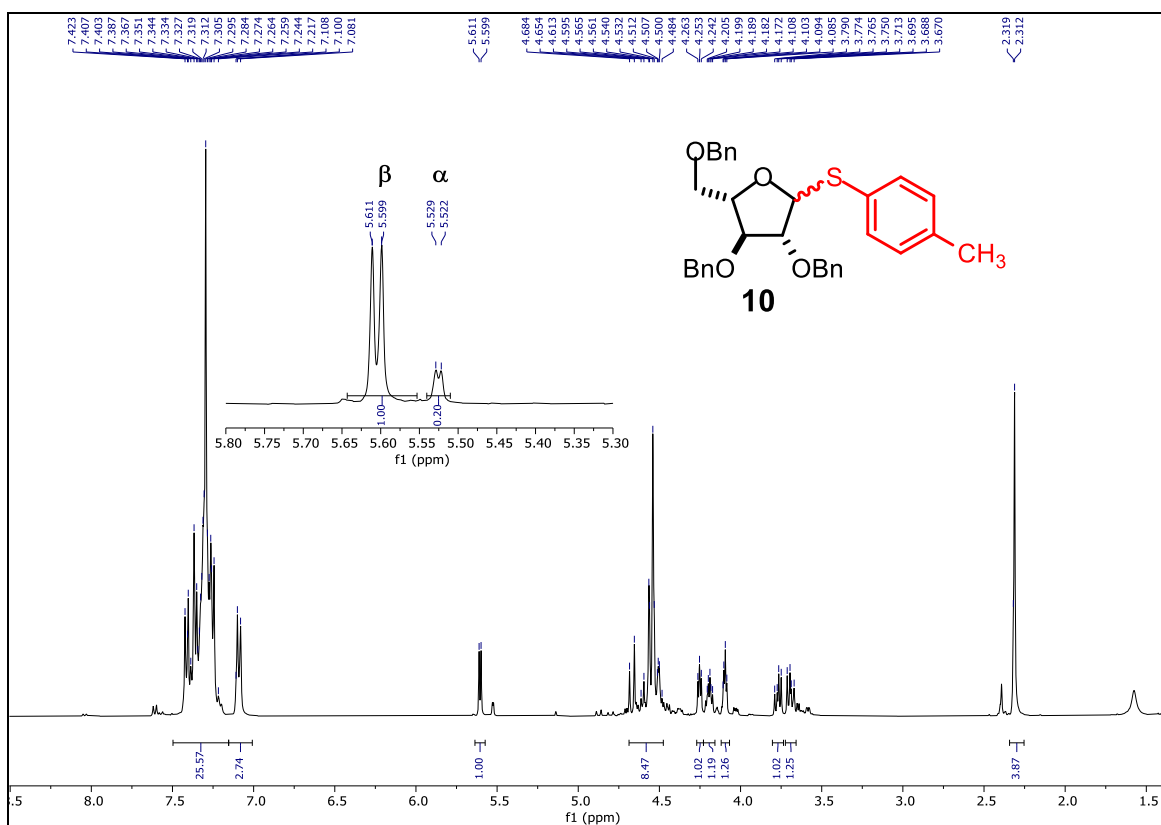


Figure S29. ^1H spectrum of 4-methylphenyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**10**) (400 MHz, CDCl_3).

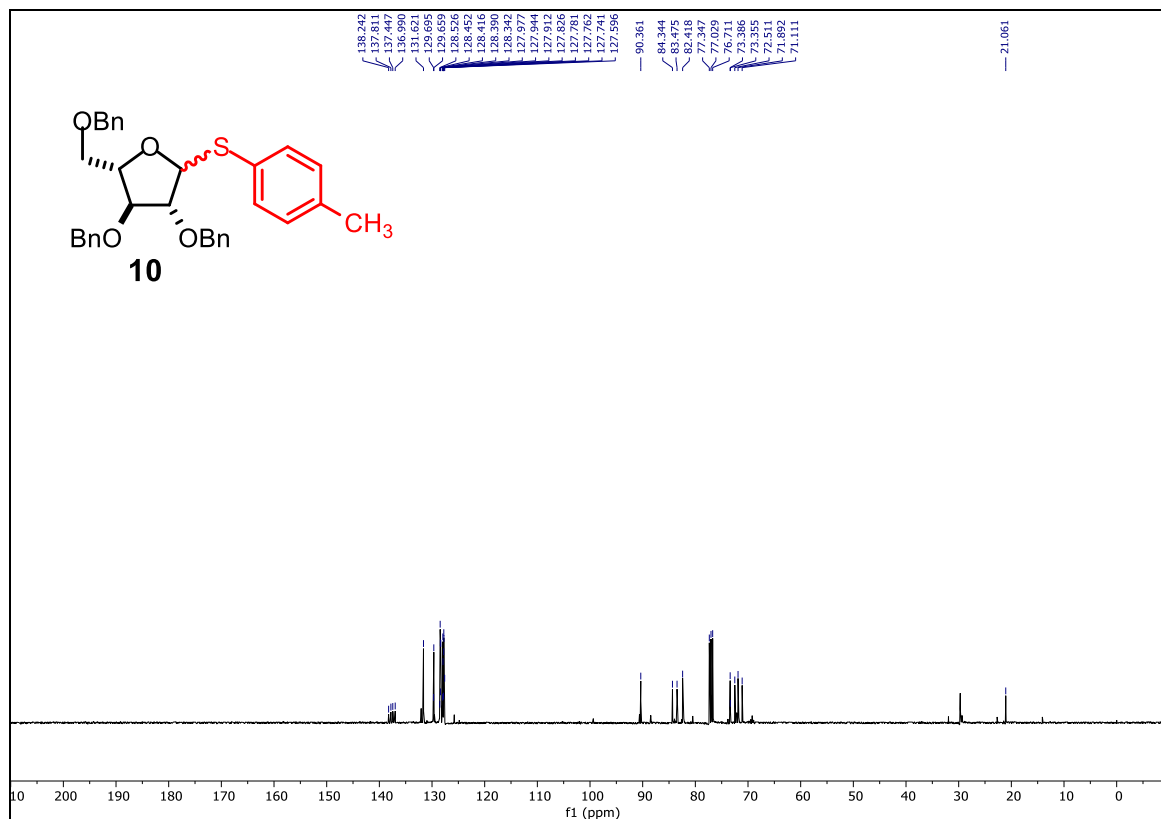


Figure S30. ^{13}C spectrum of 4-methylphenyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**10**) (101 MHz, CDCl_3).

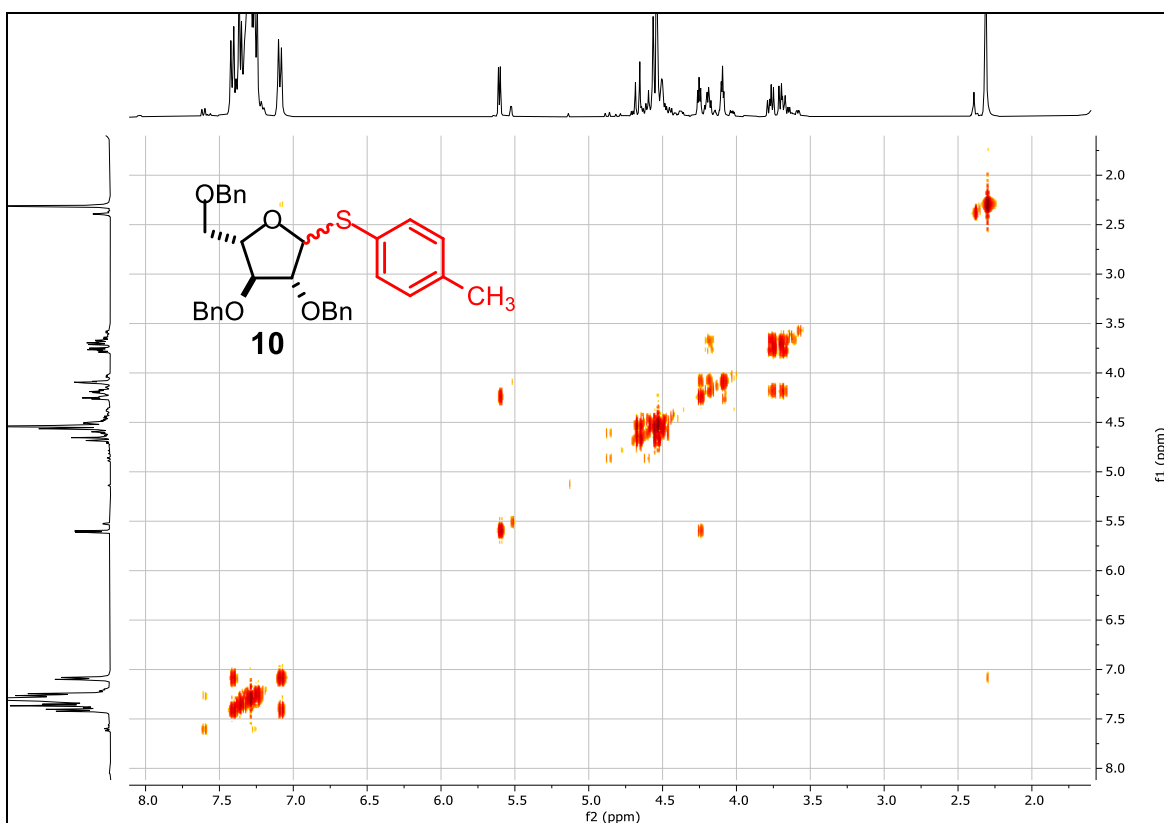


Figure S31. COSY spectrum of 4-methylphenyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**10**) (400 MHz, CDCl₃).

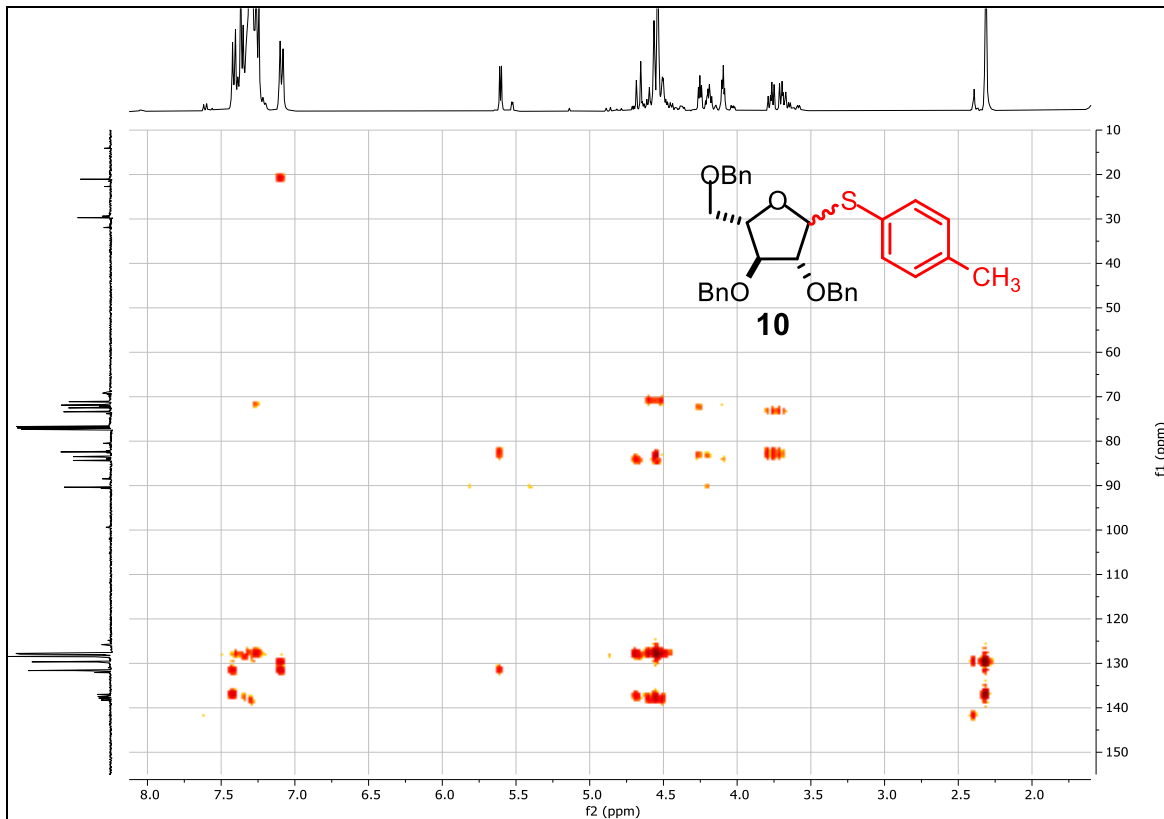
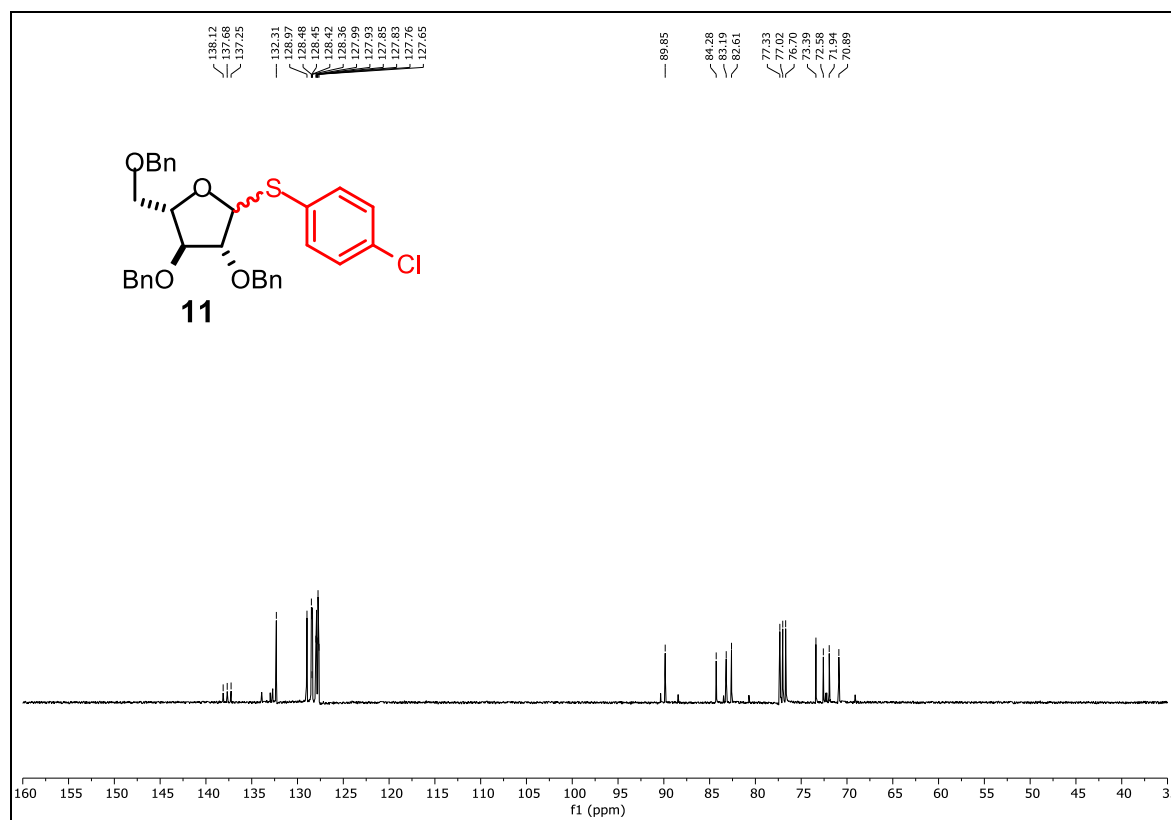
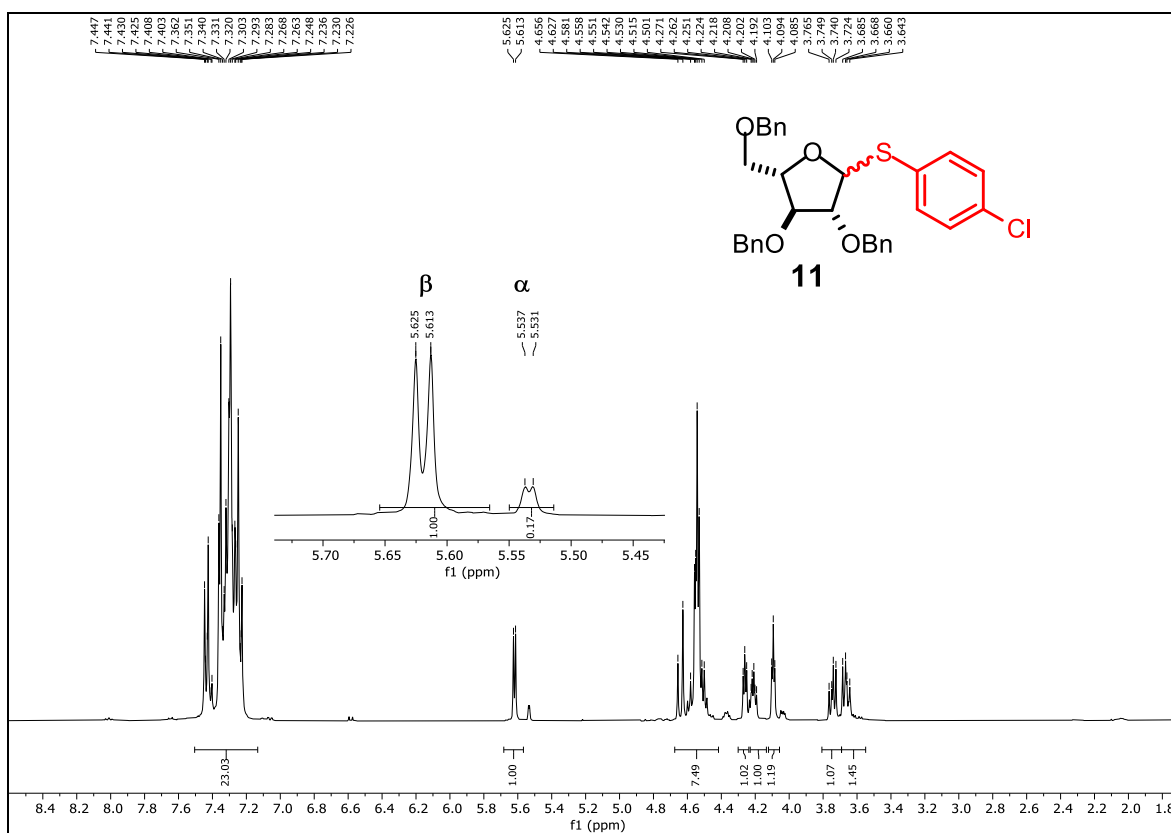


Figure S32. HMBC spectrum of 4-methylphenyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**10**) (400 MHz, CDCl₃).



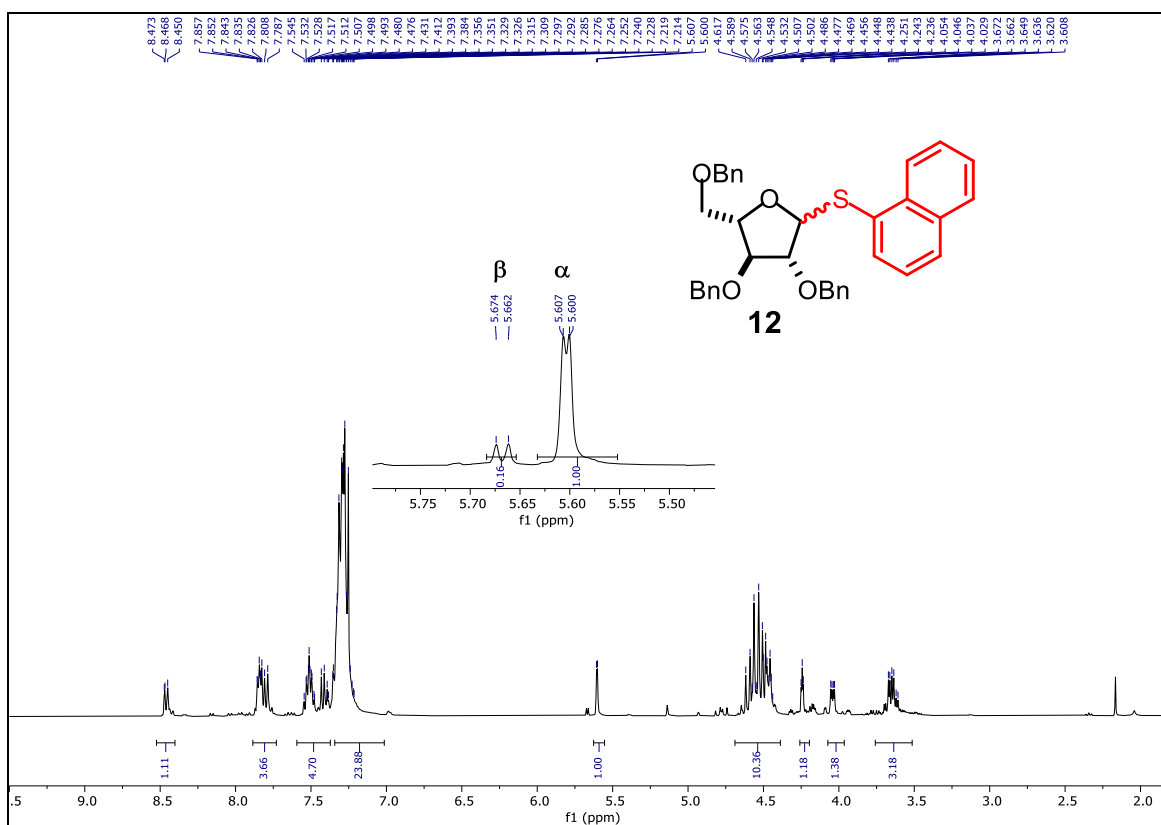


Figure S35. ^1H spectrum of 1-naphthyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**12**) (400 MHz, CDCl_3).

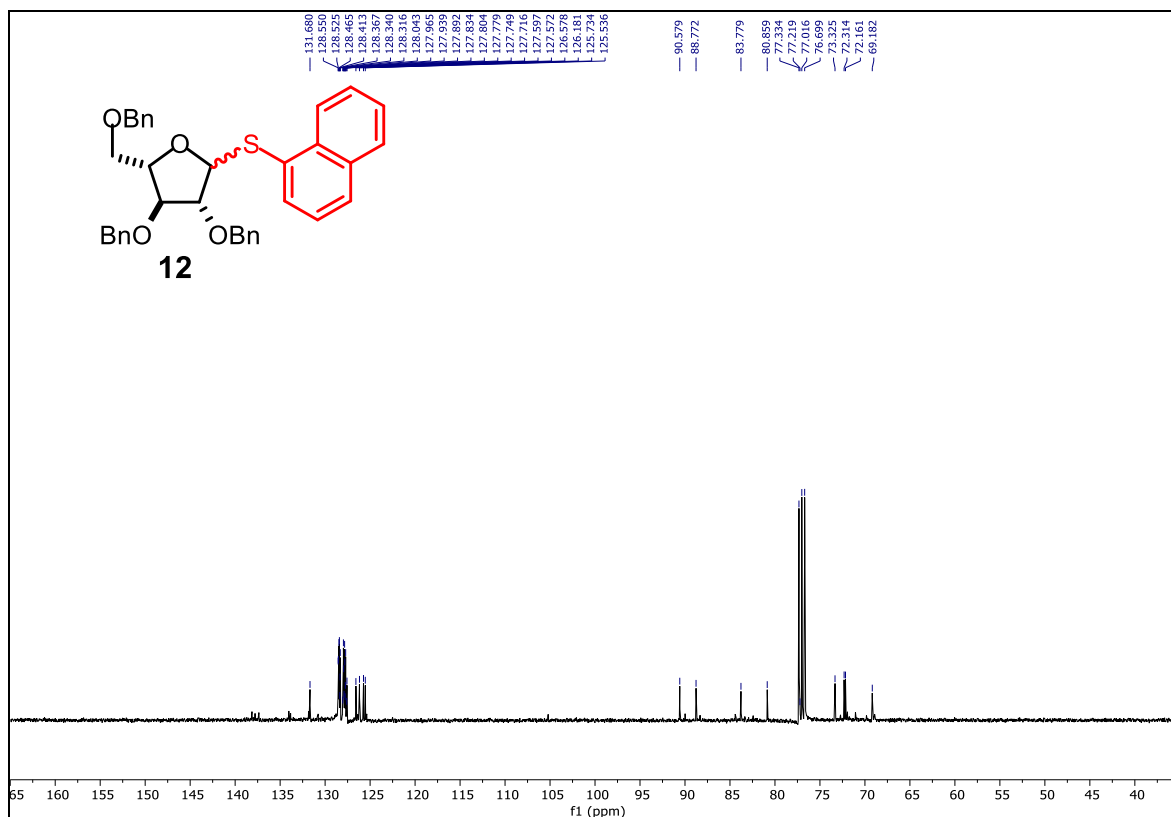


Figure S36. ^{13}C spectrum of 1-naphthyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**12**) (101 MHz, CDCl_3).

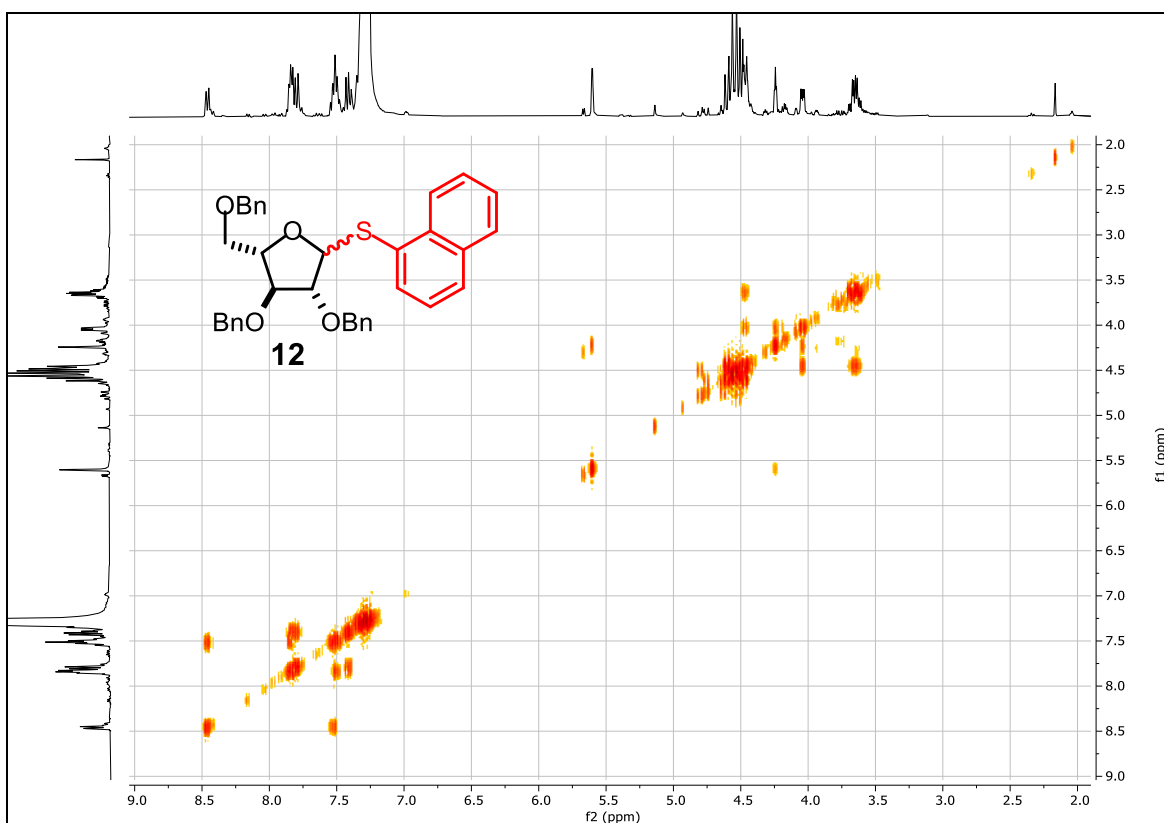


Figure S37. COSY spectrum of 1-naphthyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**12**) (400 MHz, CDCl₃).

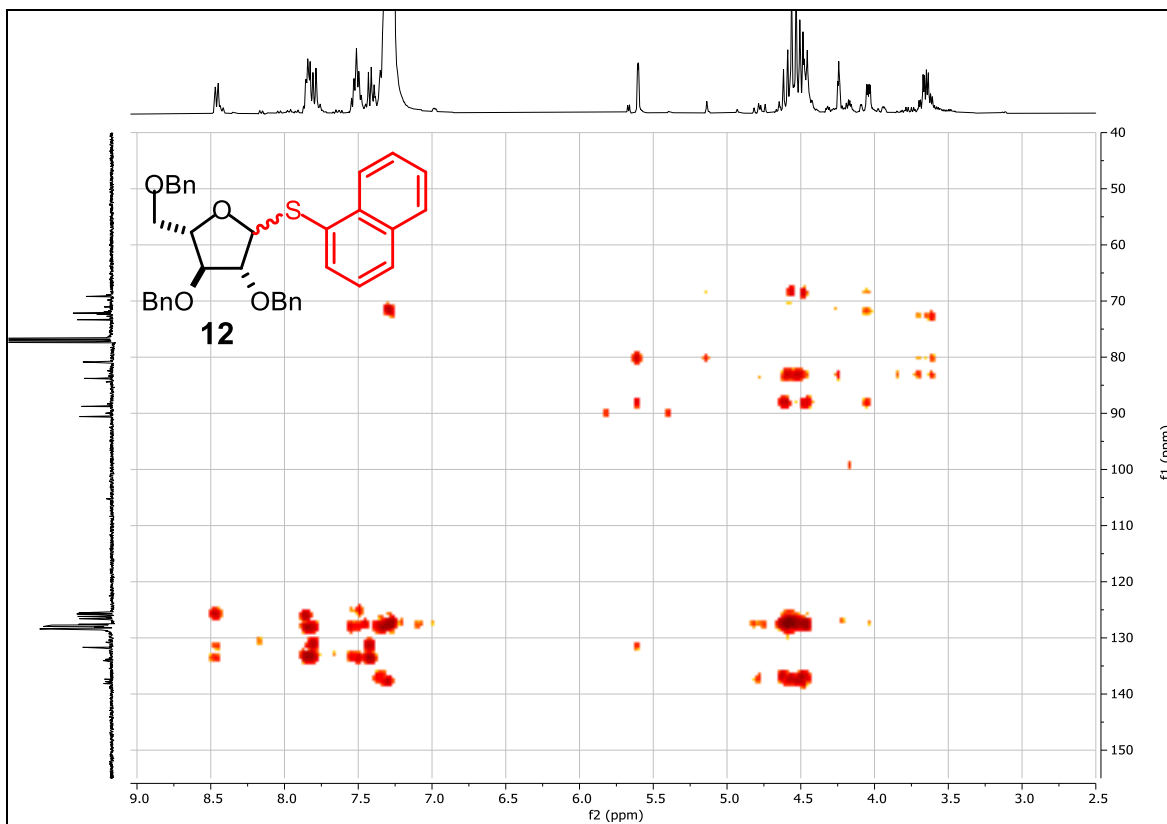


Figure S38. HMBC spectrum of 1-naphthyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -L-arabinofuranoside (**12**) (400 MHz, CDCl₃).

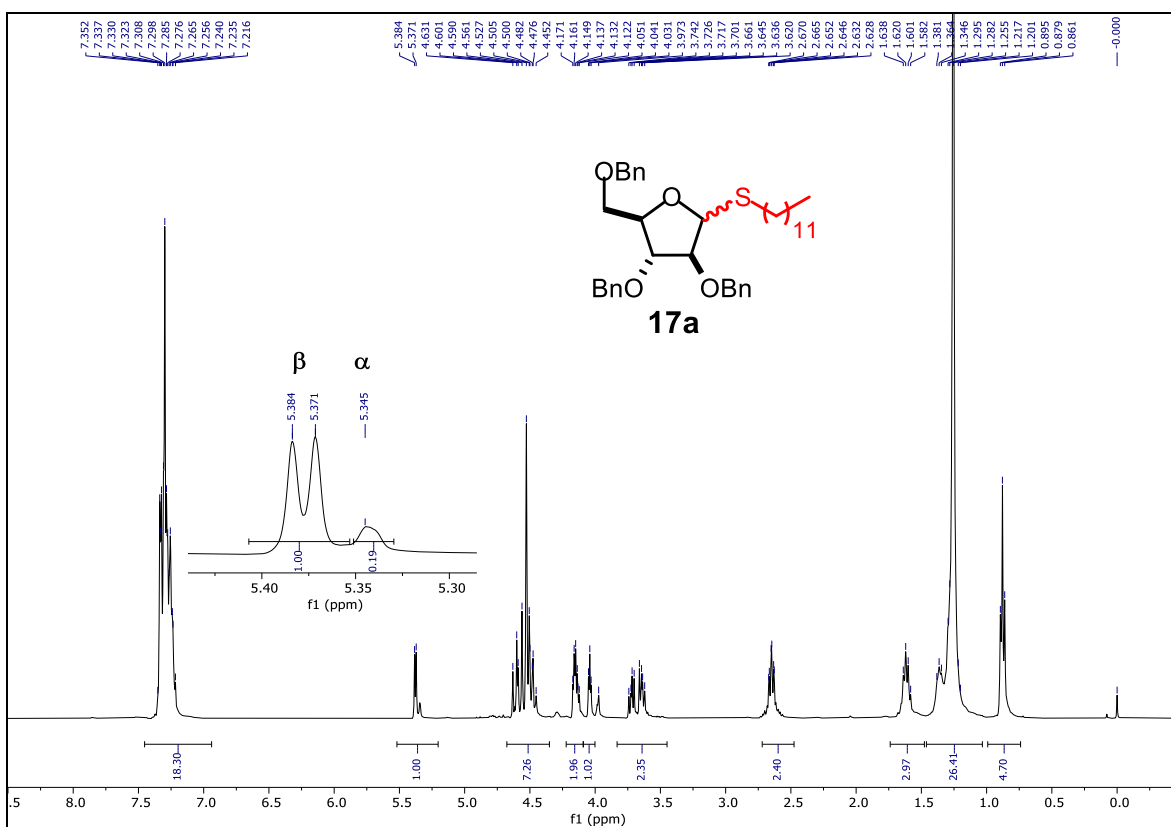


Figure S39. ^1H NMR spectrum of dodecyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -D-arabinofuranoside (**17a**) (400 MHz, CDCl_3).

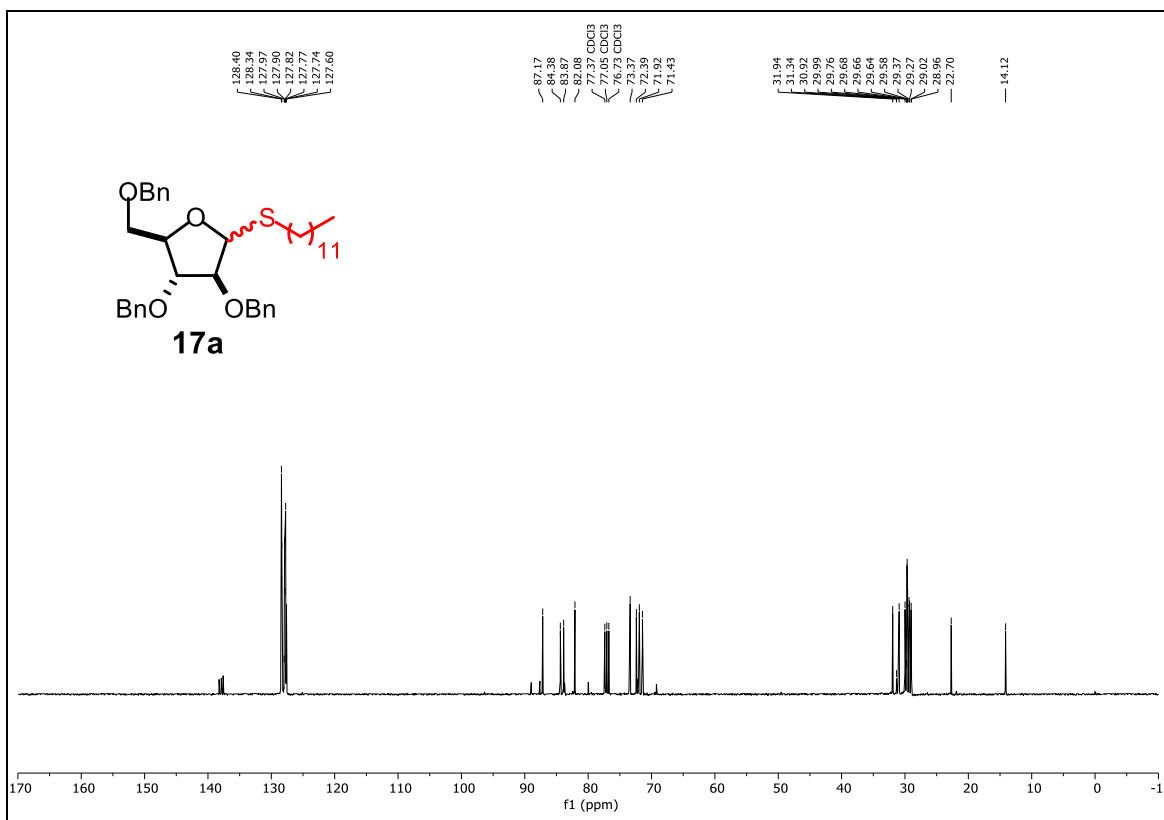


Figure S40. ^{13}C NMR spectrum of dodecyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -D-arabinofuranoside (**17a**) (101 MHz, CDCl_3).

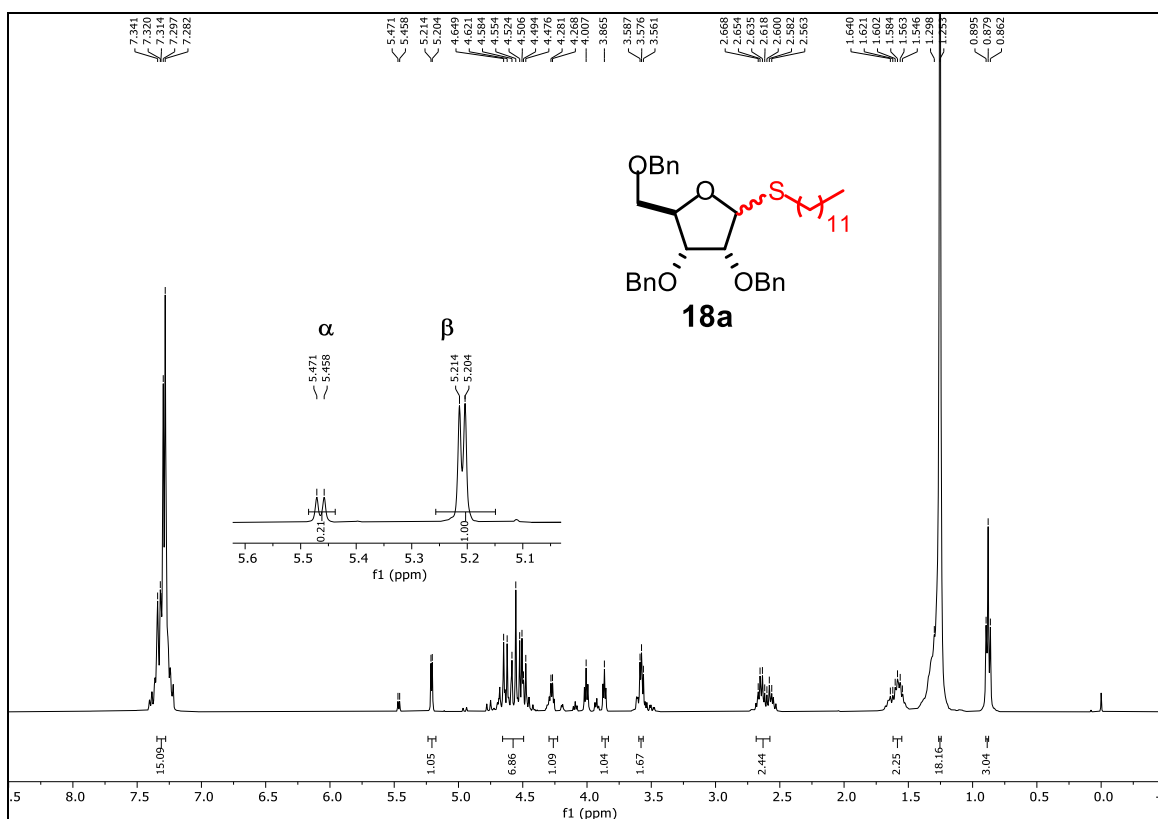


Figure S41. ¹H NMR spectrum of dodecyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -D-ribofuranoside (**18a**) (400 MHz, CDCl₃).^{1,2}

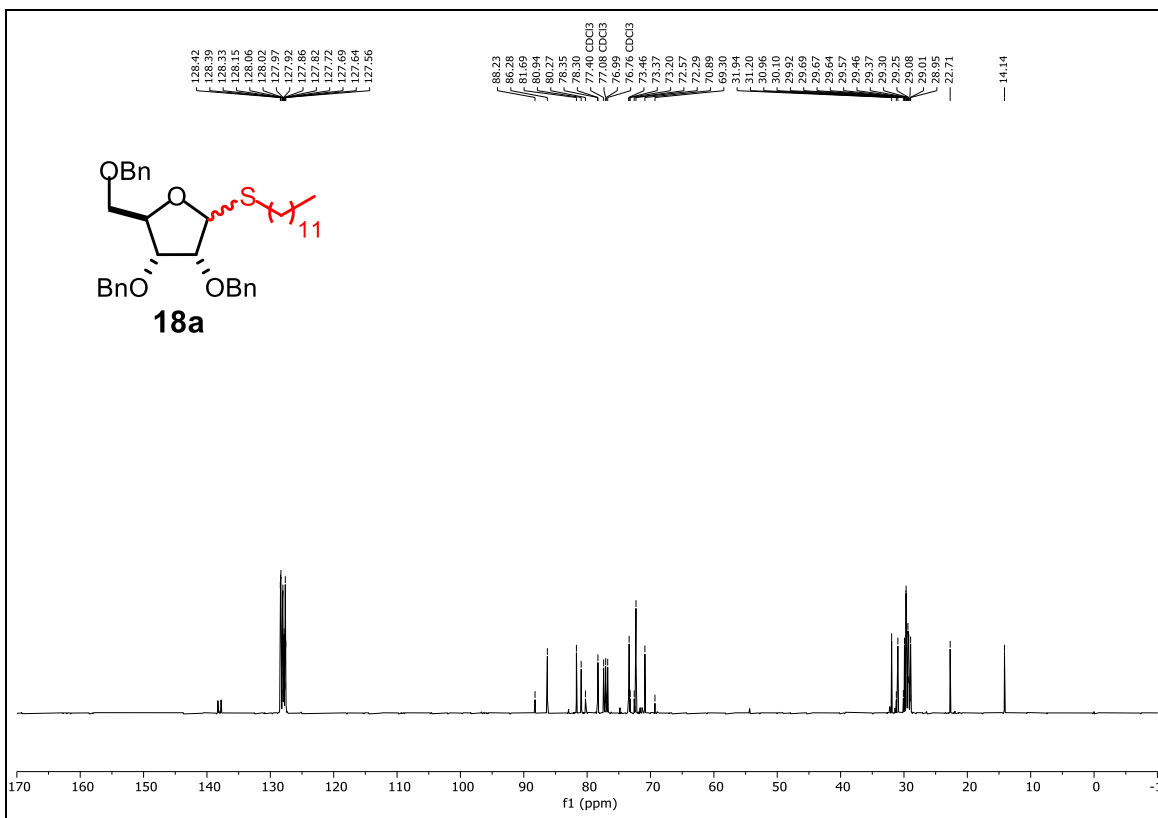


Figure S42. ¹³C NMR spectrum of dodecyl 2,3,5-tri-*O*-benzyl-1-thio- α,β -D-ribofuranoside (**18a**) (101 MHz, CDCl₃).

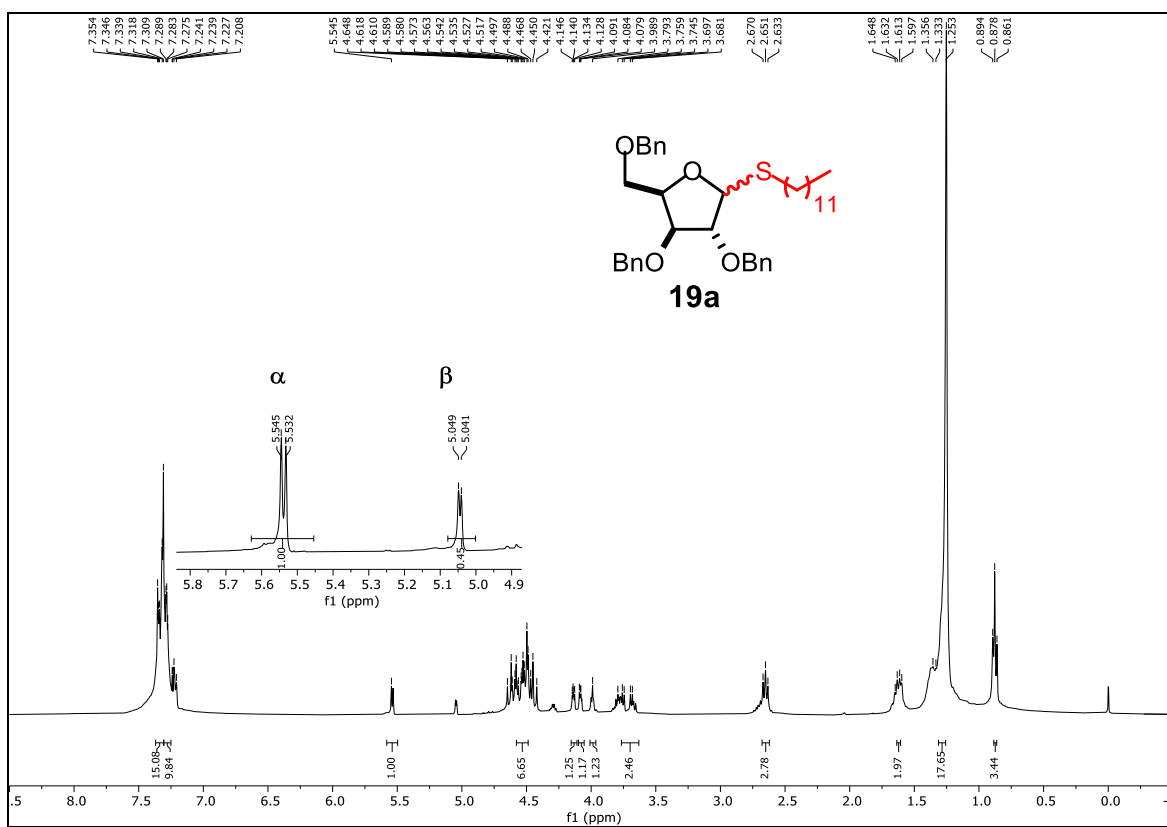


Figure S43. ¹H NMR spectrum of dodecyl 2,3,5-tri-O-benzyl-1-thio- α,β -D-xylofuranoside (**19a**) (400 MHz, CDCl₃).³

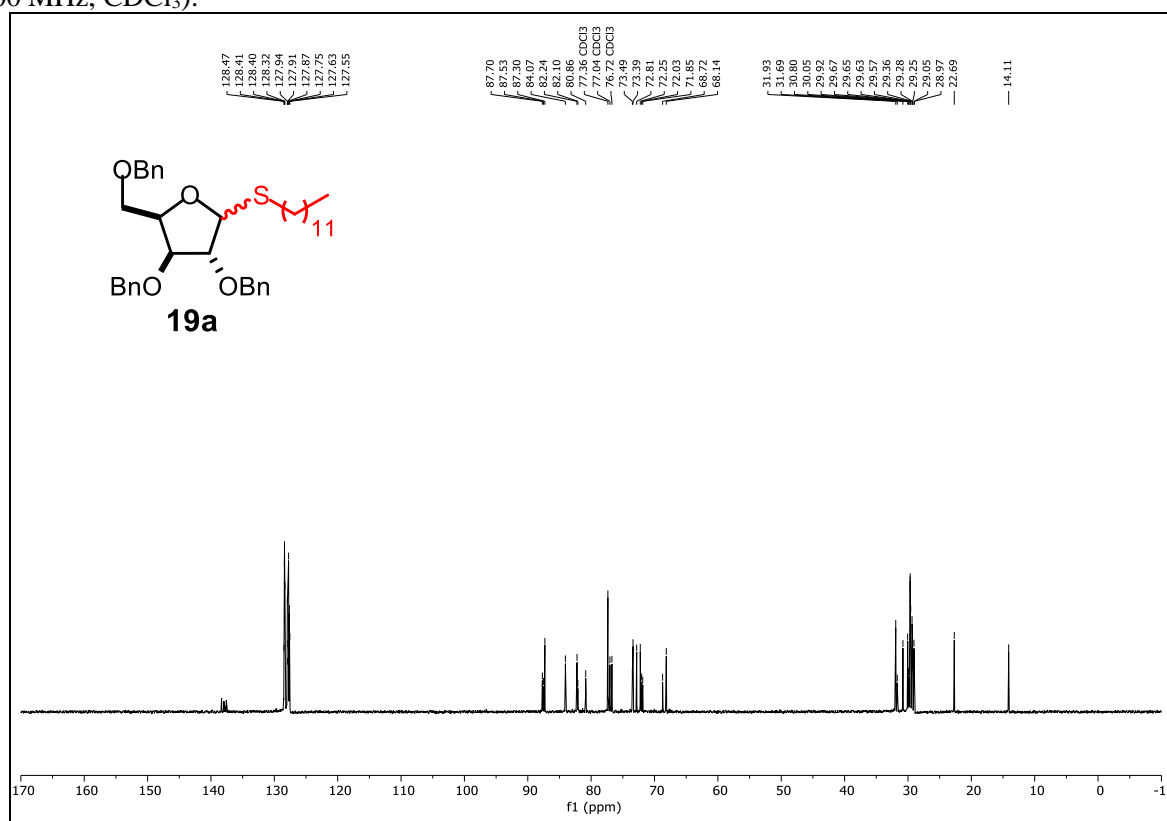


Figure S44. ¹³C NMR spectrum of dodecyl 2,3,5-tri-O-benzyl-1-thio- α,β -D-xylofuranoside (**19a**) (101 MHz, CDCl₃).

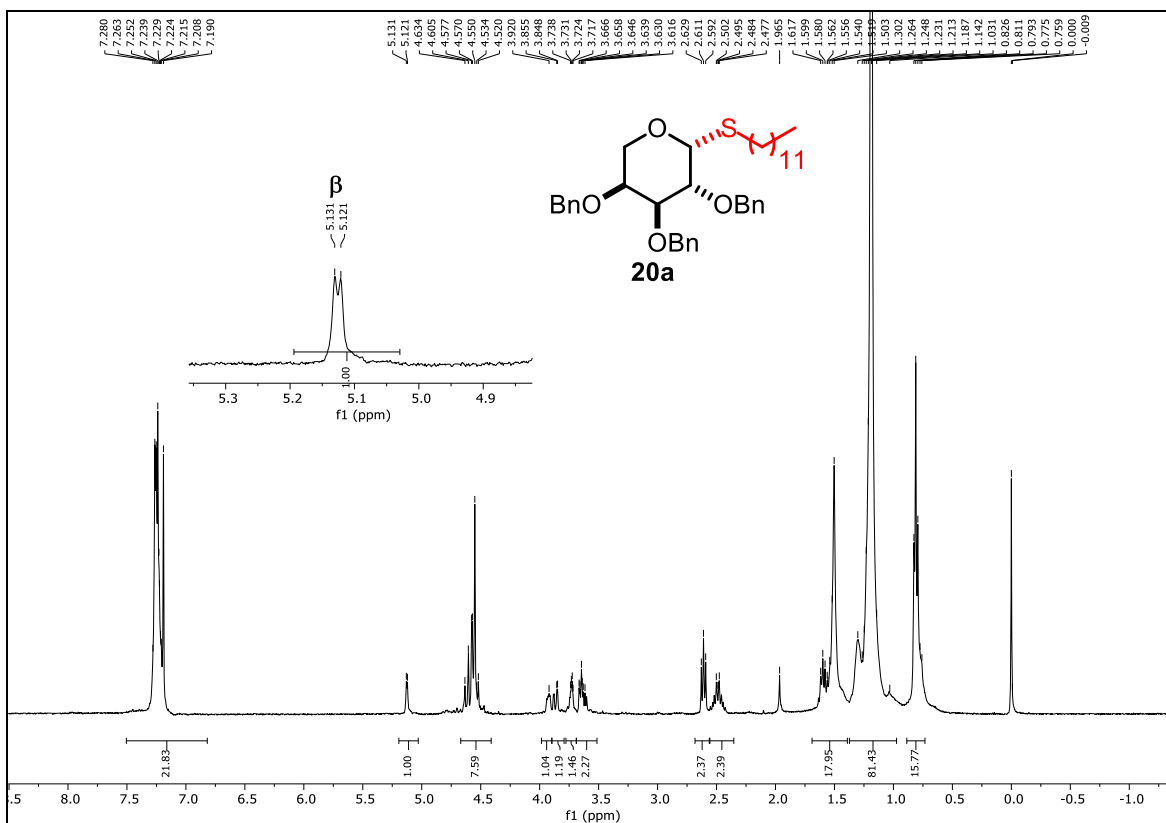


Figure S45. ¹H NMR spectrum of dodecyl 2,3,4-tri-*O*-benzyl-1-thio-β-L-arabinopyranoside (**20a**) (400 MHz, CDCl₃).⁴

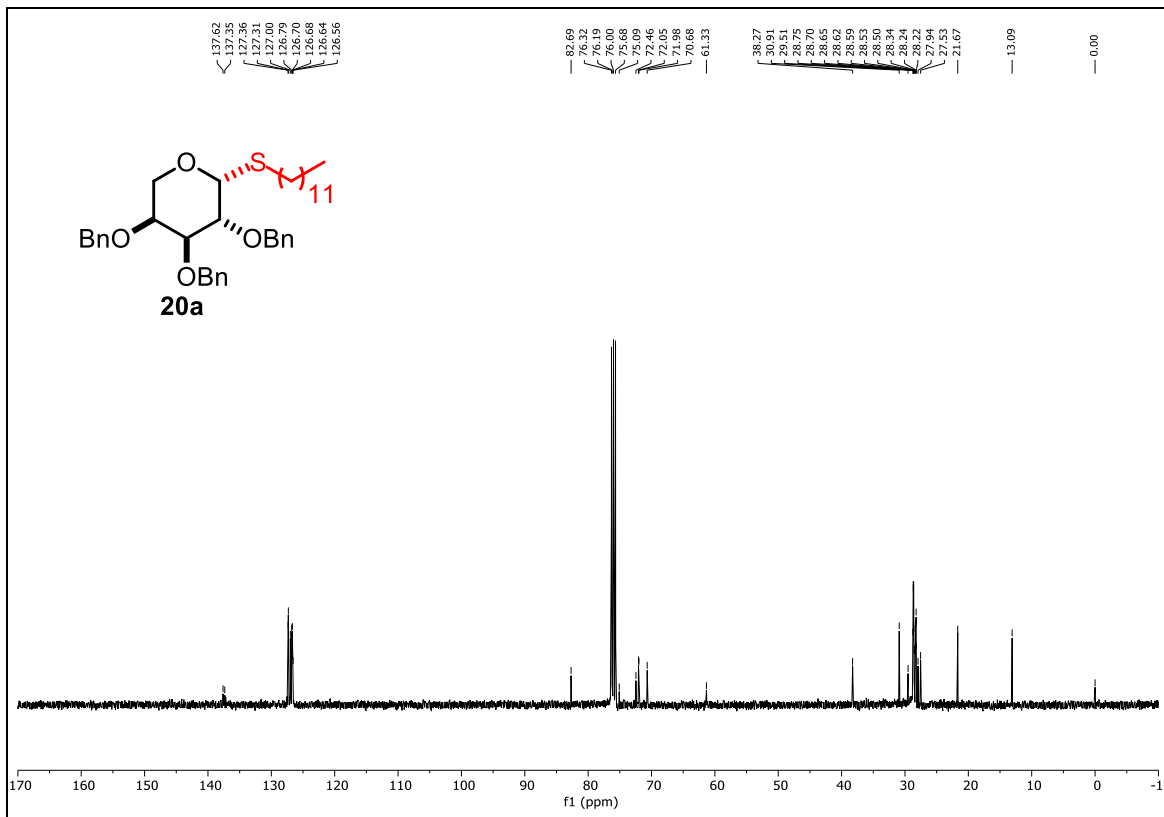
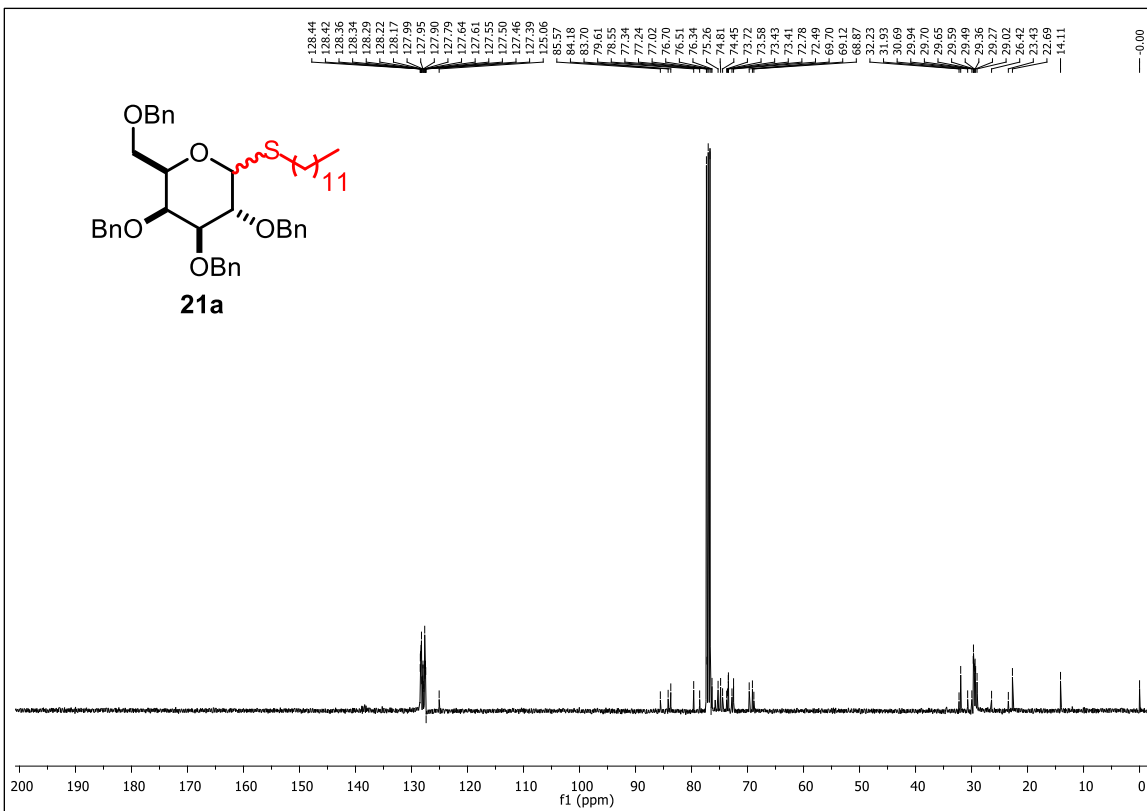
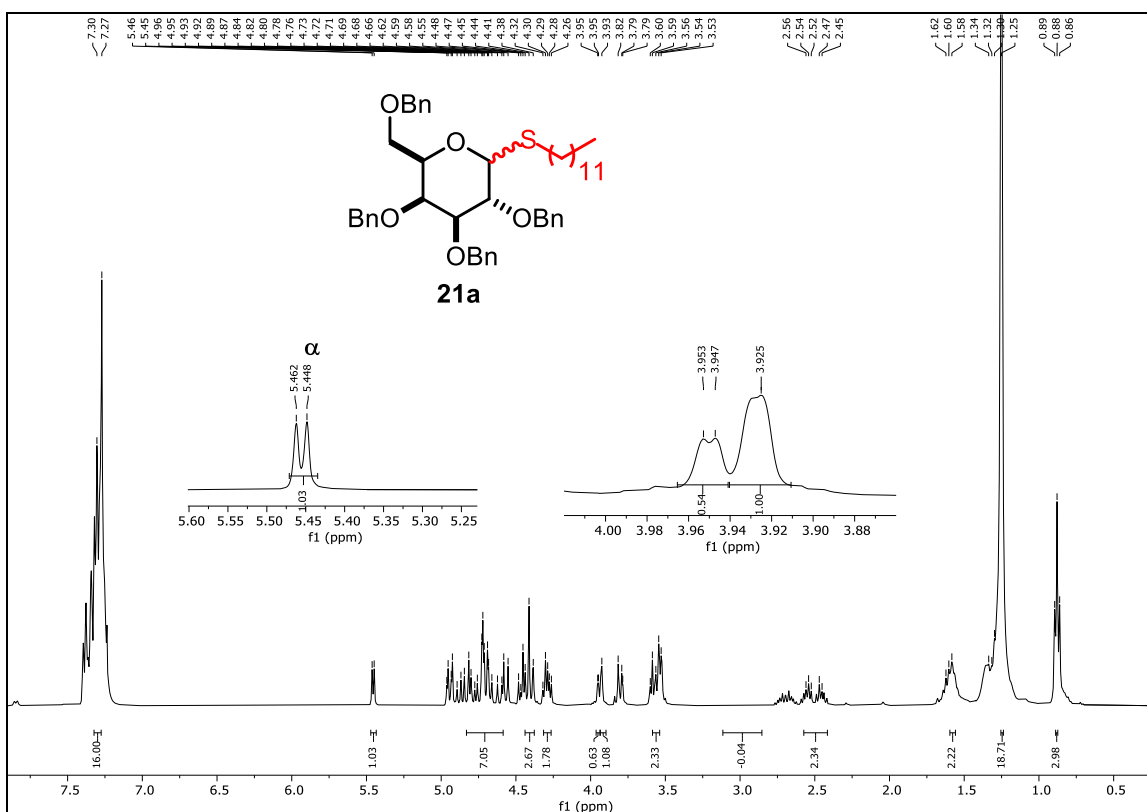


Figure S46. ¹³C NMR spectrum of dodecyl 2,3,4-tri-*O*-benzyl-1-thio-β-L-arabinopyranoside (**20a**) (101 MHz, CDCl₃).



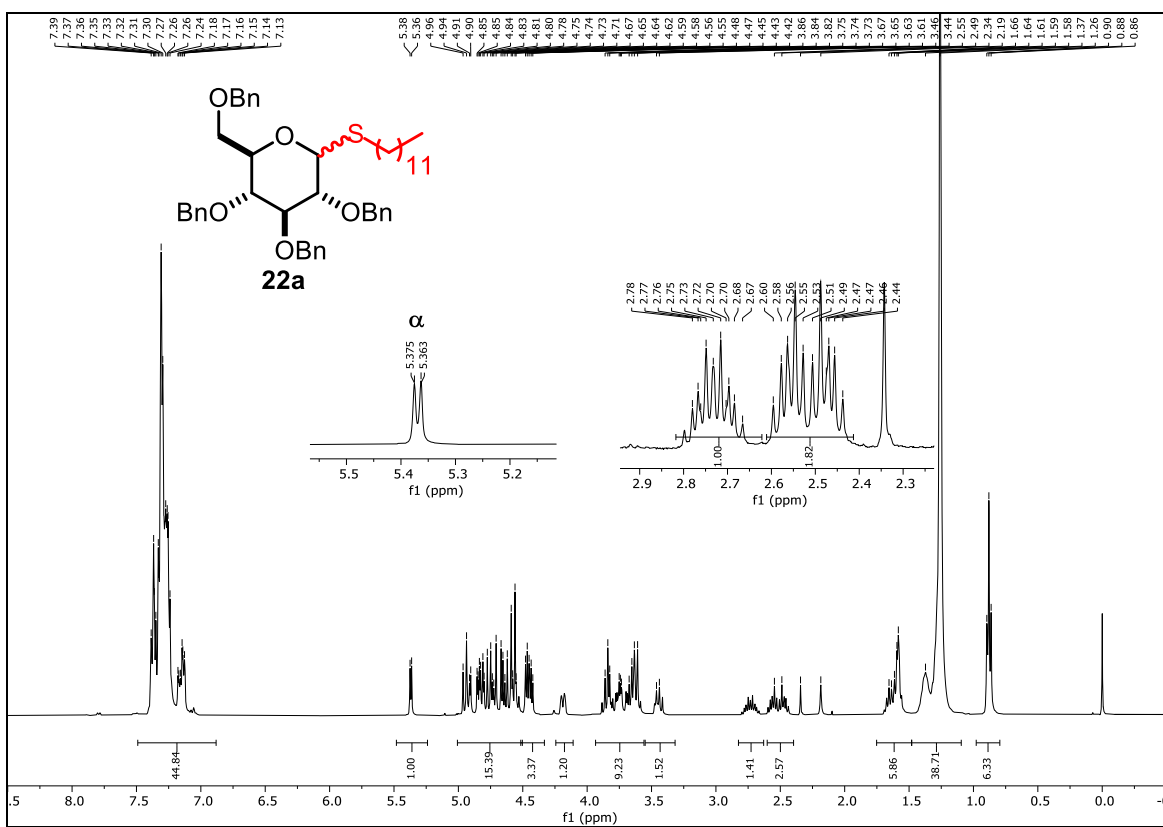


Figure S49. ¹H NMR spectrum of dodecyl 2,3,4,6-tetra-*O*-benzyl-1-thio- α,β -D-glucopyranoside (**22a**) (400 MHz, CDCl₃).⁶

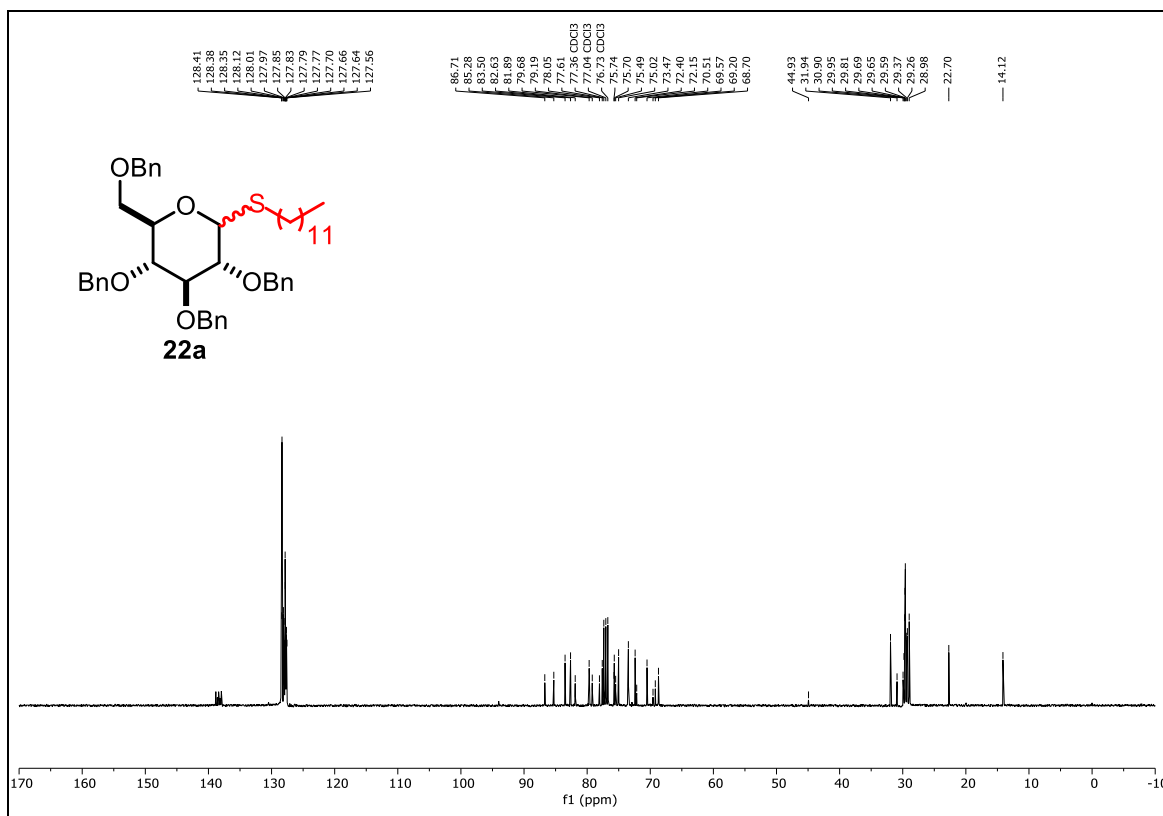


Figure S50. ¹³C NMR spectrum of dodecyl 2,3,4,6-tetra-*O*-benzyl-1-thio- α,β -D-glucopyranoside (**22a**) (101 MHz, CDCl₃).

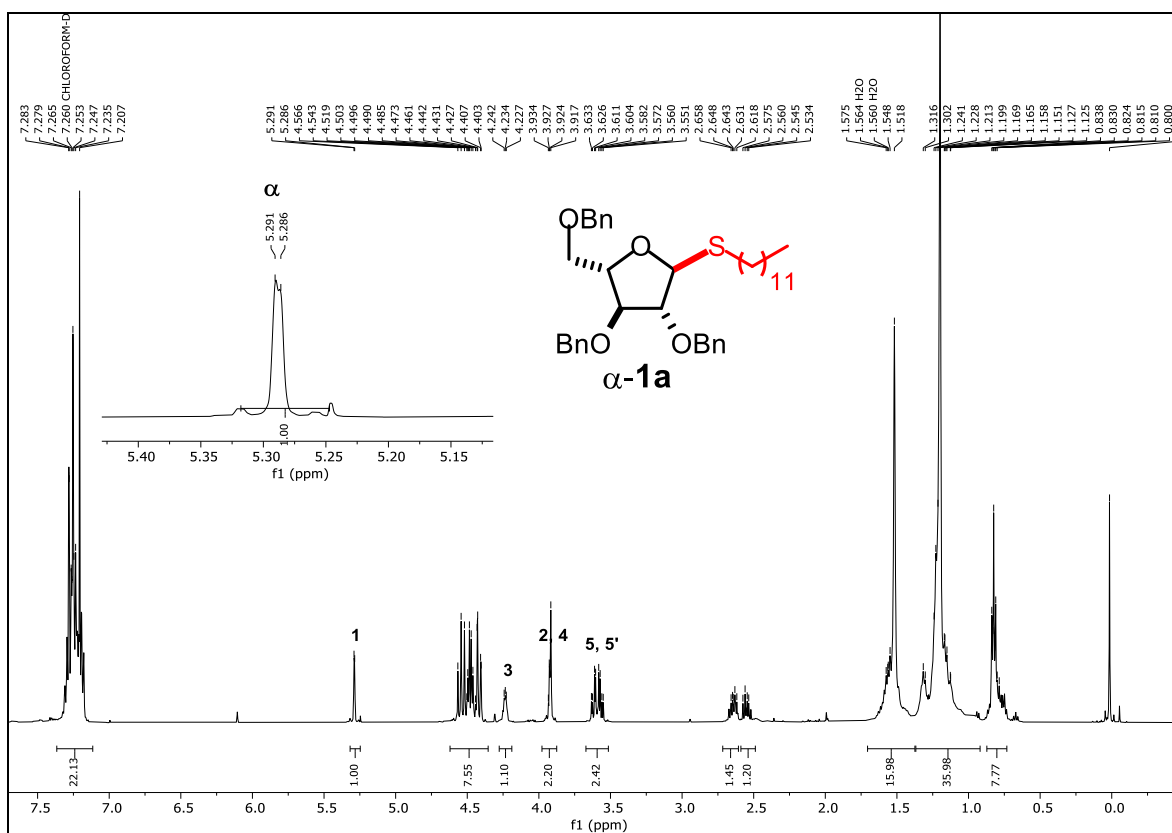


Figure S51. ¹H NMR spectrum of dodecyl 2,3,5-tri-*O*-benzyl-1-thio- α -L-arabinofuranoside (**1a**) (500 MHz, CDCl₃).

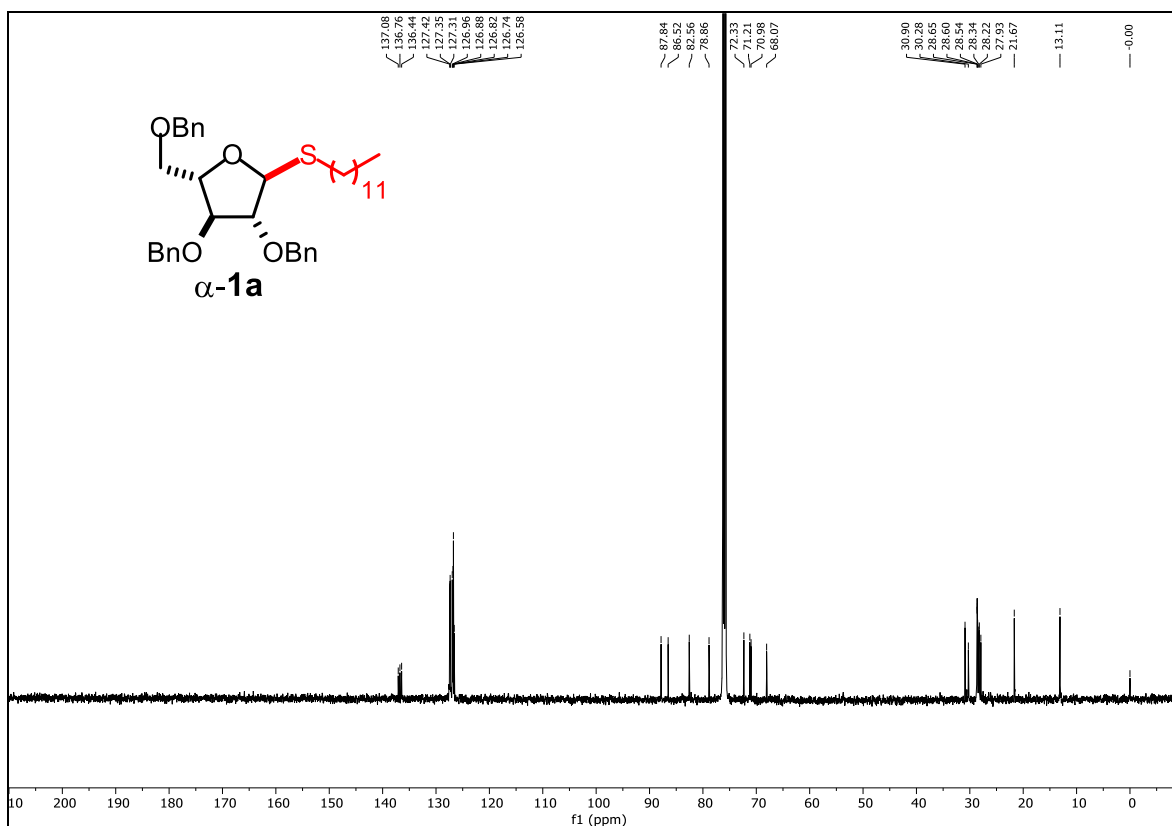


Figure S52. ¹³C NMR spectrum of dodecyl 2,3,5-tri-*O*-benzyl-1-thio- α -L-arabinofuranoside (**1a**) (126 MHz, CDCl₃).

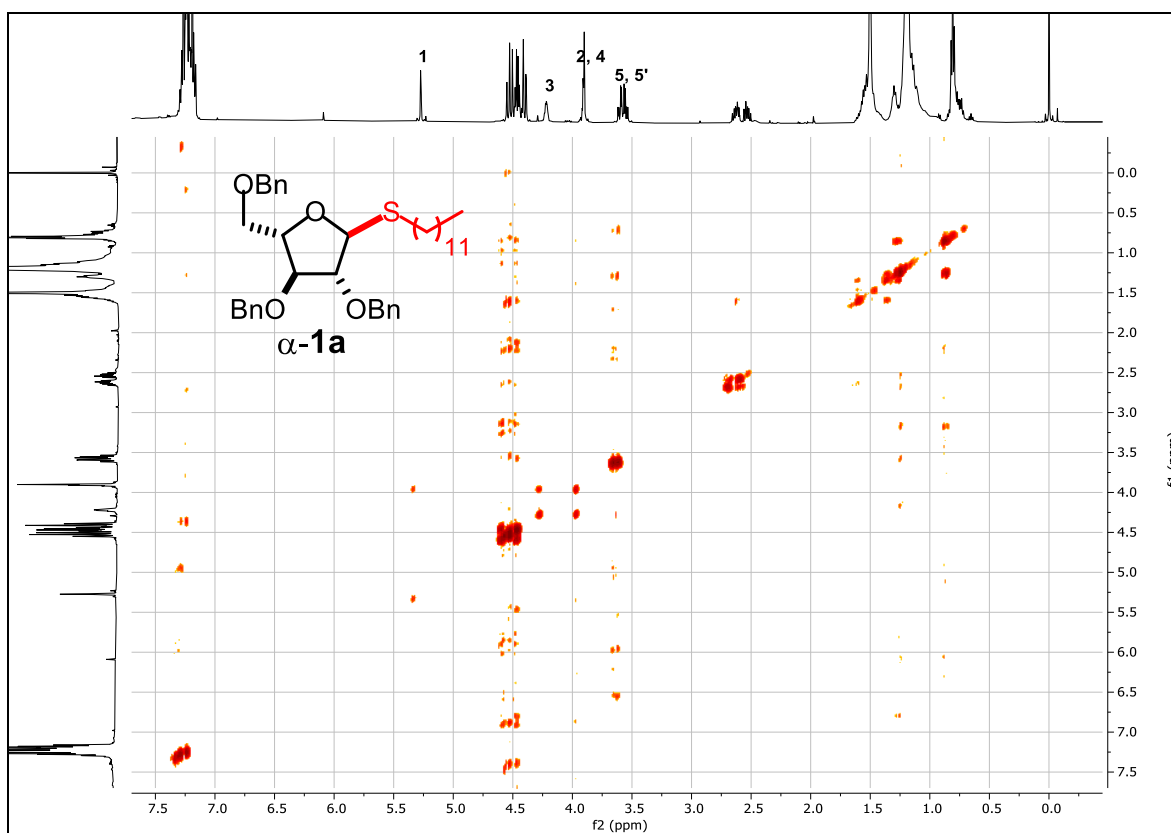


Figure S53. COSY spectrum of dodecyl 2,3,5-tri-*O*-benzyl-1-thio- α -L-arabinofuranoside (**1a**) (500 MHz, CDCl_3).

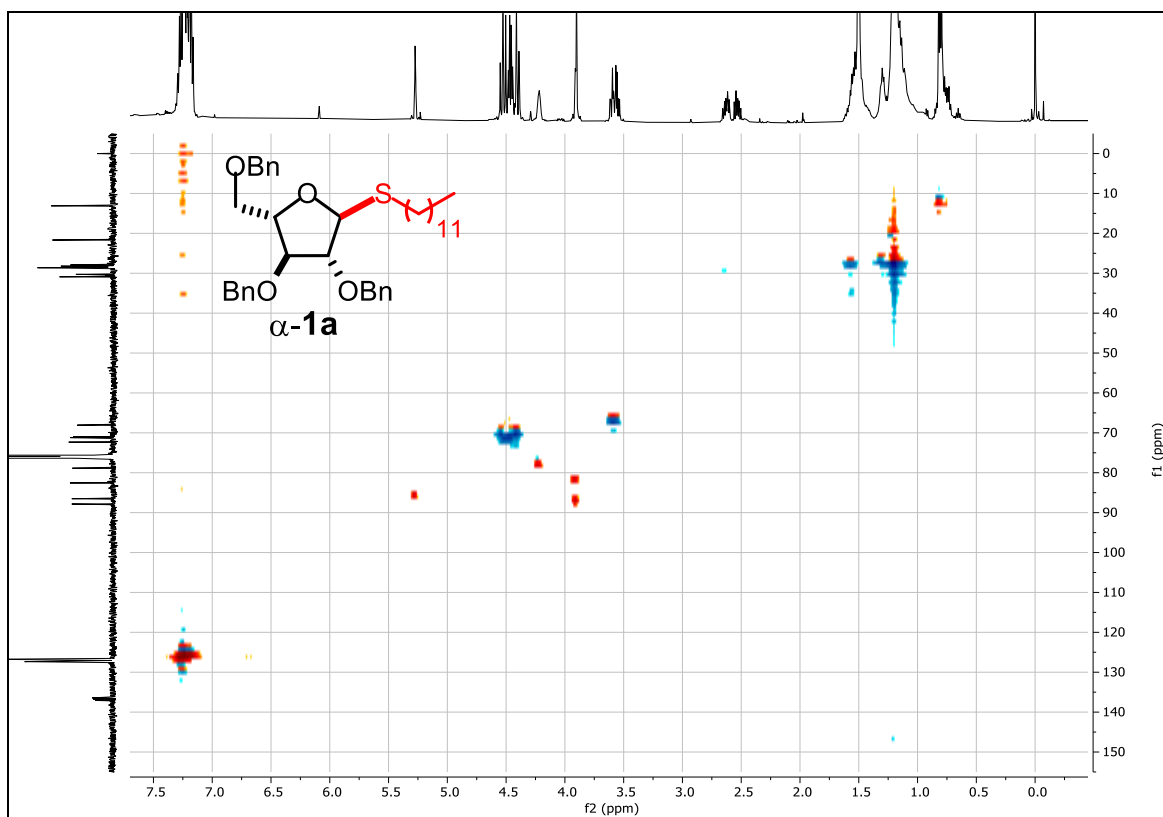


Figure S54. HSQC spectrum of dodecyl 2,3,5-tri-*O*-benzyl-1-thio- α -L-arabinofuranoside (**1a**) (500 MHz, CDCl_3).

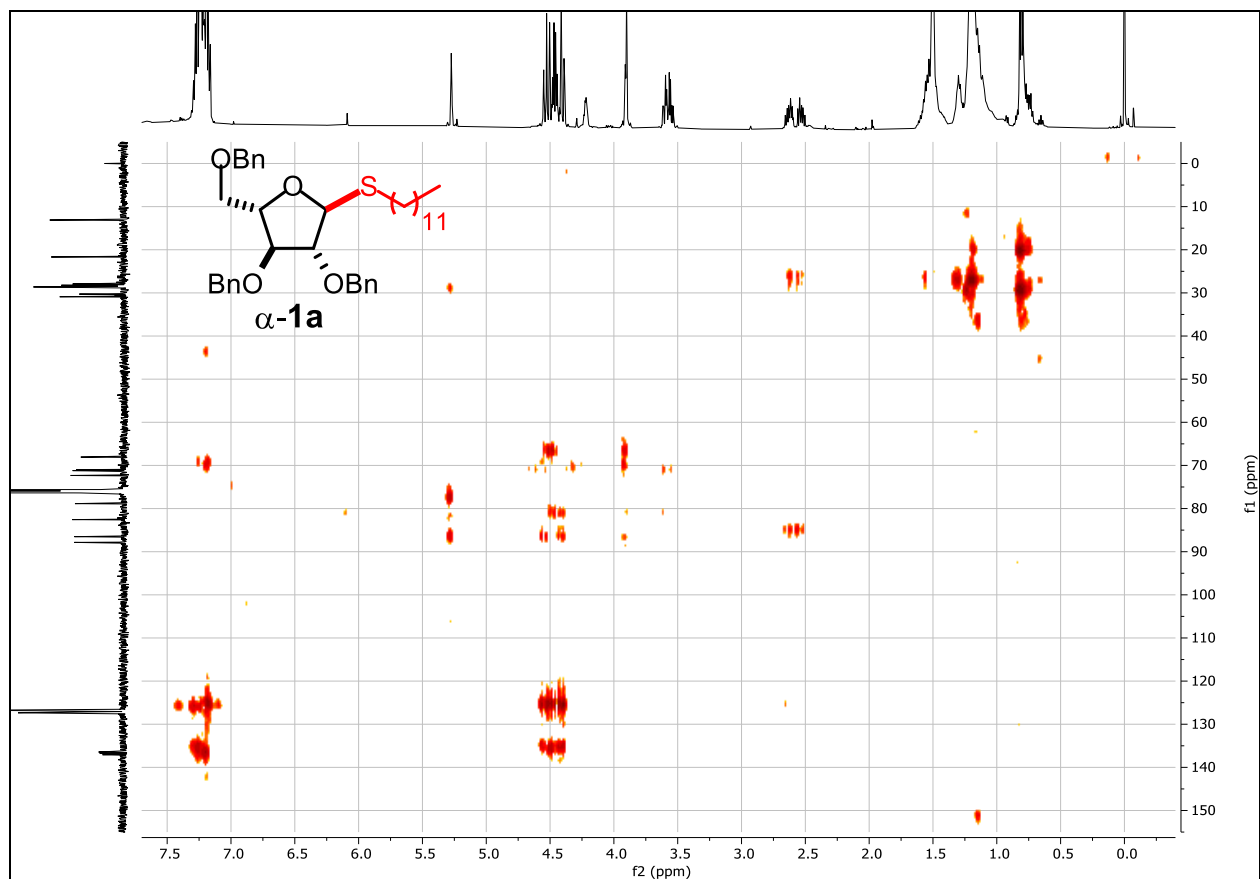


Figure S55. HMBC spectrum of dodecyl 2,3,5-tri-*O*-benzyl-1-thio- α -L-arabinofuranoside (**1a**) (500 MHz, CDCl_3).

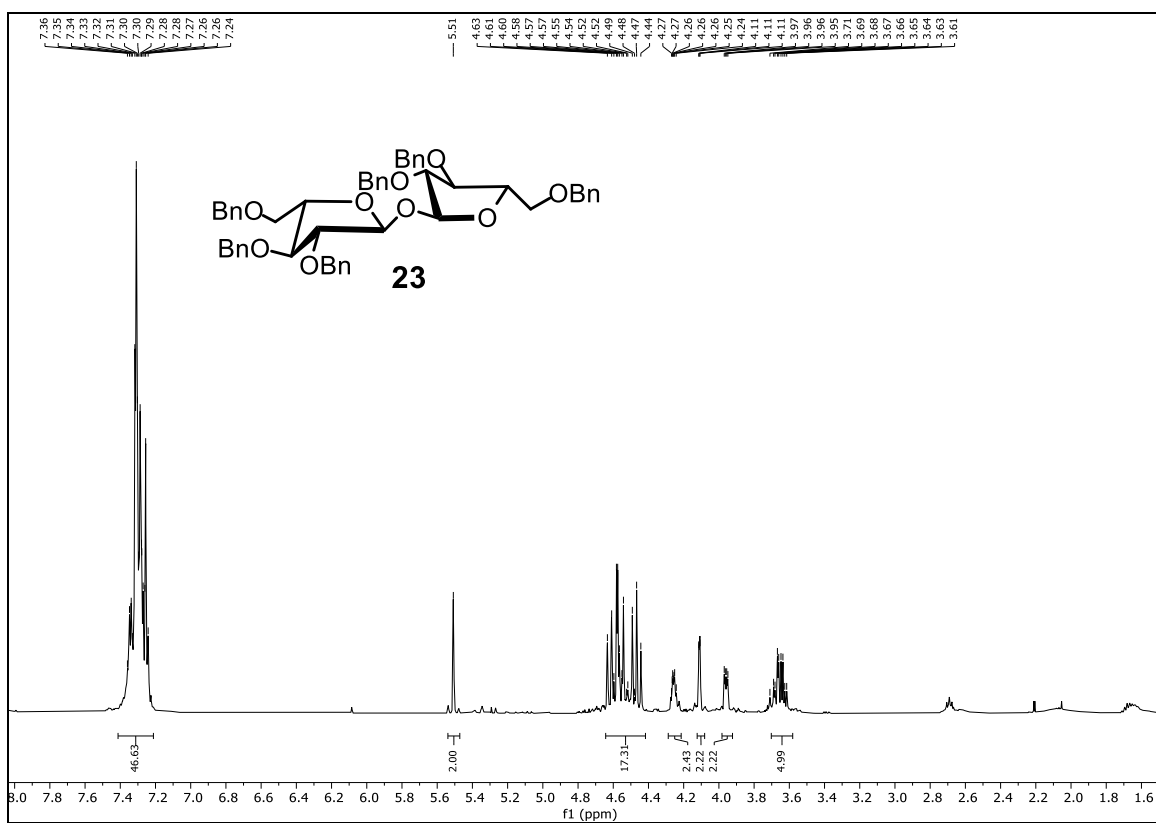


Figure S56. ^1H NMR spectrum of 2,3,5,2',3',5'-hexa-*O*-benzyl- α,α - L-arabinofuranoside (**23**) (500 MHz, CDCl_3).

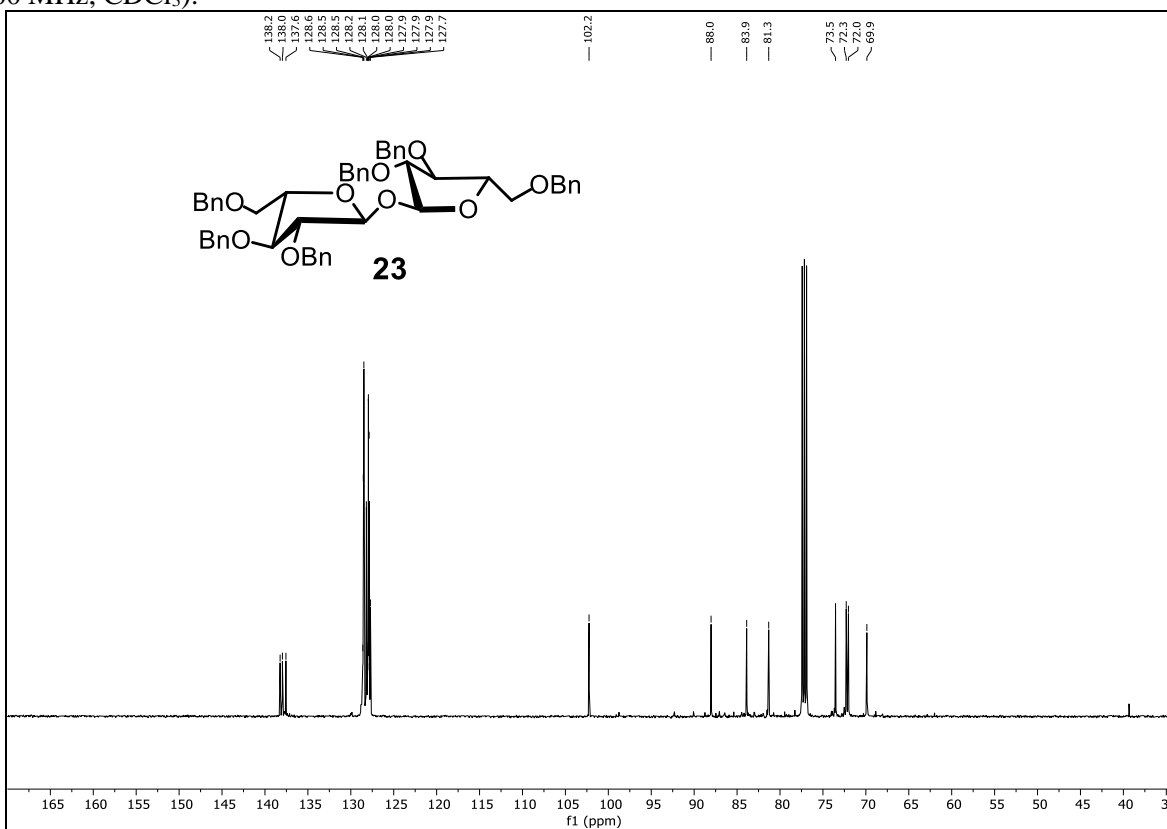


Figure S57. ^{13}C NMR spectrum of 2,3,5,2',3',5'-hexa-*O*-benzyl- α,α - L-arabinofuranoside (**23**) (126 MHz, CDCl_3).

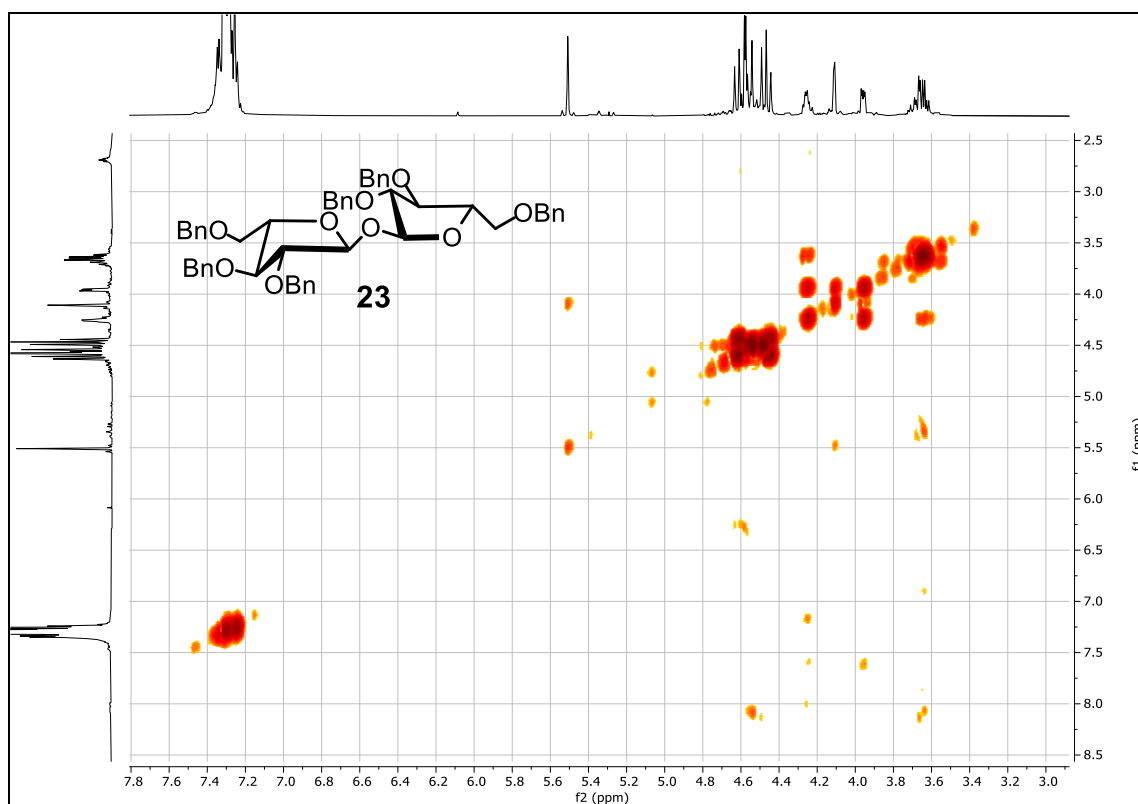


Figure S58. COSY spectrum of 2,3,5,2',3',5'-hexa-*O*-benzyl- α,α -L-arabinofuranoside (**23**) (500 MHz, CDCl_3).

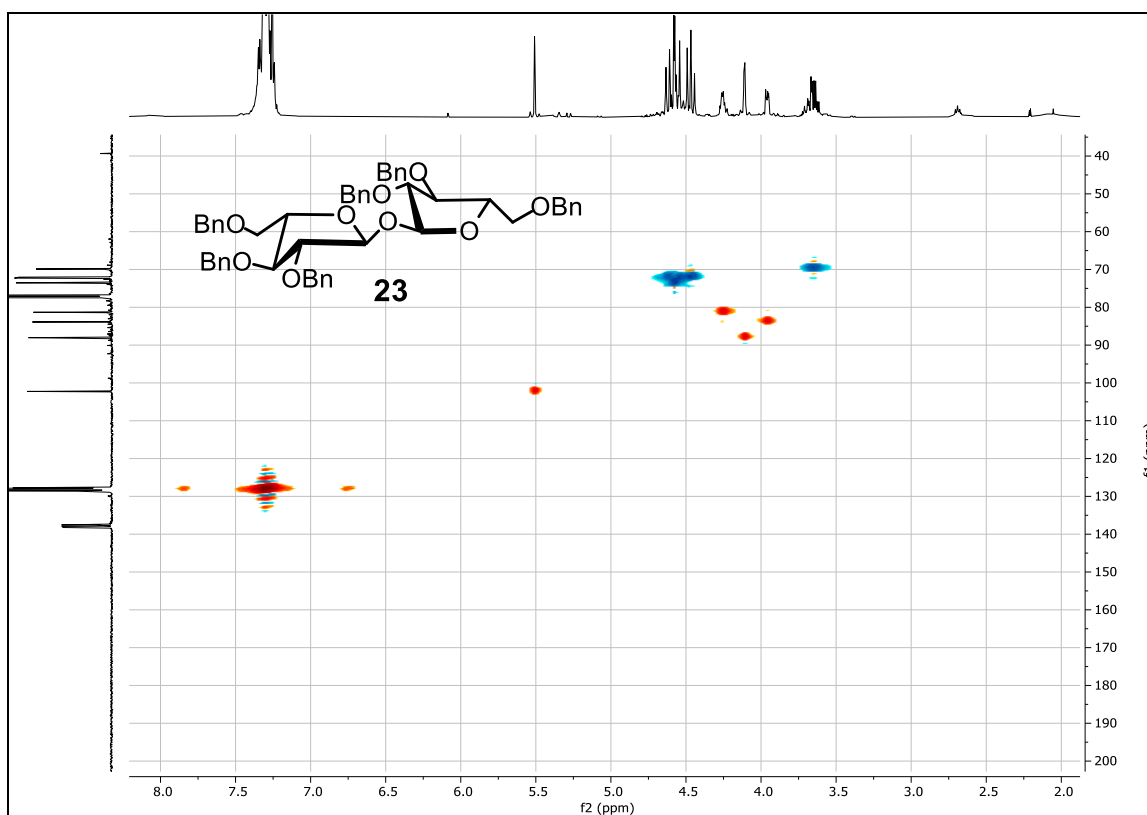


Figure S59. HSQC spectrum of 2,3,5,2',3',5'-hexa-*O*-benzyl- α,α -L-arabinofuranoside (**23**) (500 MHz, CDCl_3).

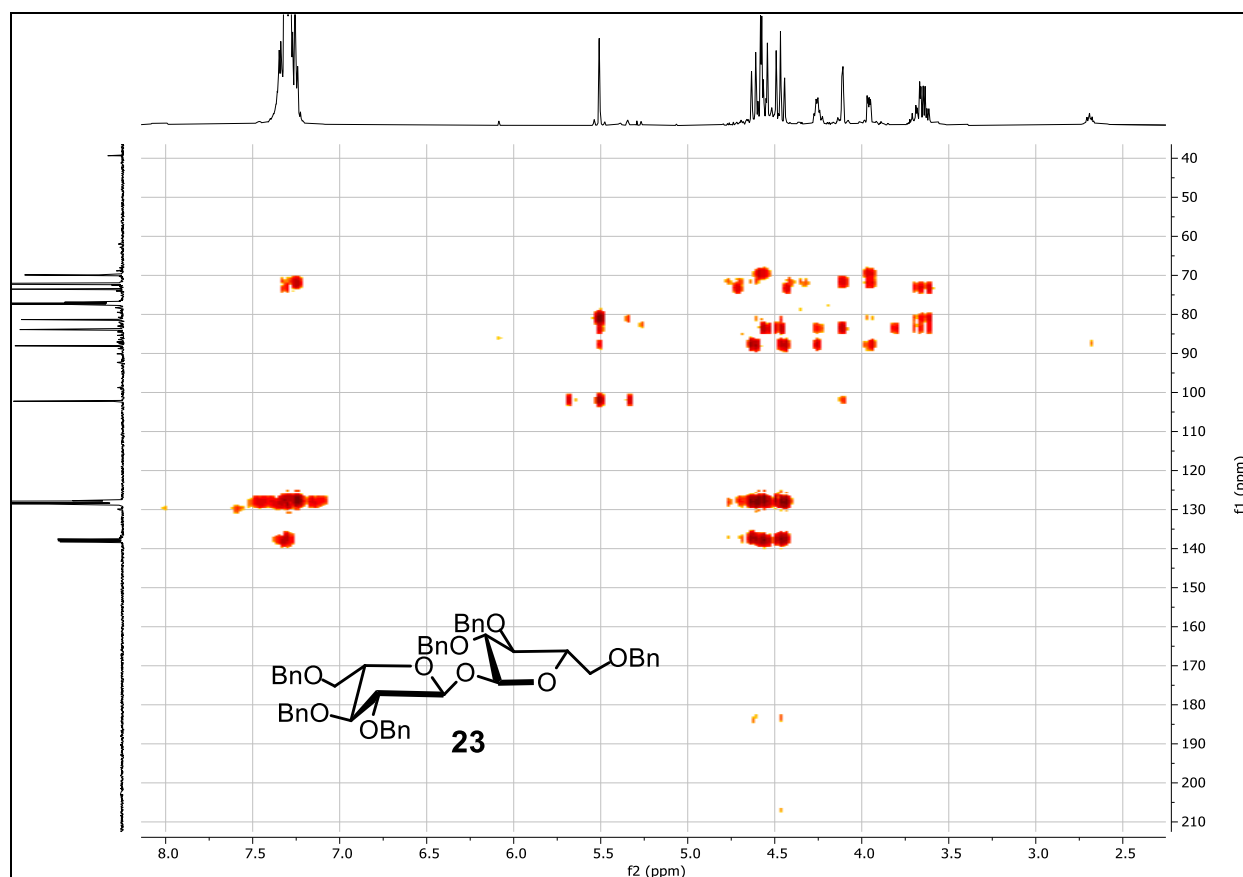


Figure S60. HMBC spectrum of 2,3,5,2',3',5'-hexa-*O*-benzyl- α,α -L-arabinofuranoside (**23**) (500 MHz, CDCl₃).

Reference

1. Kametani, T.; Kawamura, K.; Honda, T., New entry to the C-glycosidation by means of carbenoid displacement reaction. Its application to the synthesis of showdomycin. *J. Am. Chem. Soc.* **1987**, *109* (10), 3010-3017.
2. Oka, N.; Mori, A.; Ando, K., Stereoselective Synthesis of 1-Thio- α -d-Ribofuranosides Using Ribofuranosyl Iodides as Glycosyl Donors. *Eur. J. Org. Chem.* **2018**, *2018* (45), 6355-6362.
3. Vidadala, S. R.; Gayatri, G.; Sastry, G. N.; Hotha, S., Propargyl/methyl furanosides as potential glycosyl donors. *Chem. Commun.* **2011**, *47* (35), 9906-9908.
4. ZHAO, G.-L.; YU, Z.-Y.; LI, Y.; PANG, L.-N.; WANG, J.-W., Total Synthesis of 4''-O-Acetylmananthoside B Part II: Synthesis of the Disaccharide Fragment. *Chinese Journal of Chemistry* **2008**, *26* (1), 158-164.
5. Weïwer, M.; Sherwood, T.; Linhardt, R. J., Synthesis of Floridoside. *Journal of Carbohydrate Chemistry* **2008**, *27* (7), 420-427.
6. Kaeothip, S.; Pornsuriyasak, P.; Demchenko, A. V., Silver(I) tetrafluoroborate as a potent promoter for chemical glycosylation. *Tetrahedron Lett.* **2008**, *49* (9), 1542-1545.