Electronic Supplementary Information (ESI)

Towards the Complete Synthetic *O*-Antigen of *Vibrio cholerae* O1, Serotype Inaba: Improved Synthesis of the Conjugation-ready Upstream Terminal Hexasaccharide Determinant

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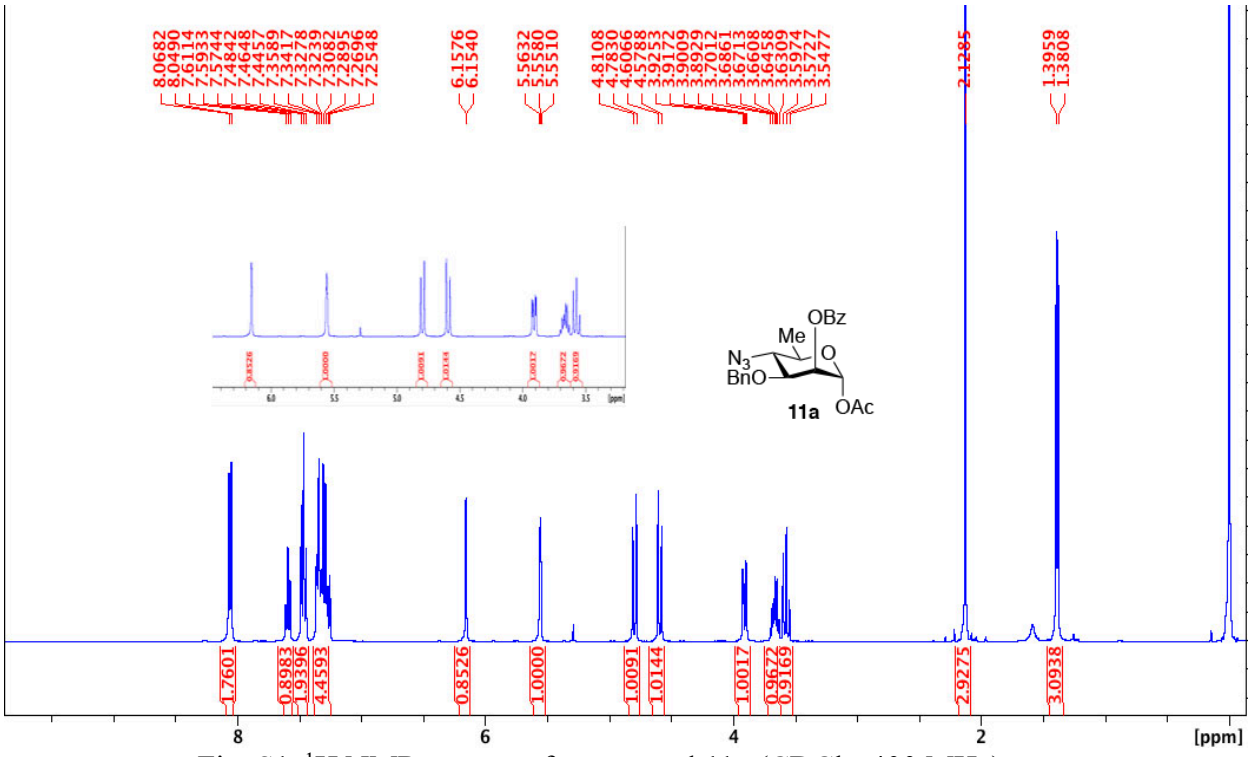

Fig. S1: ^{1}H NMR spectra of compound **11a** ($CDCl_3$, 400 MHz).

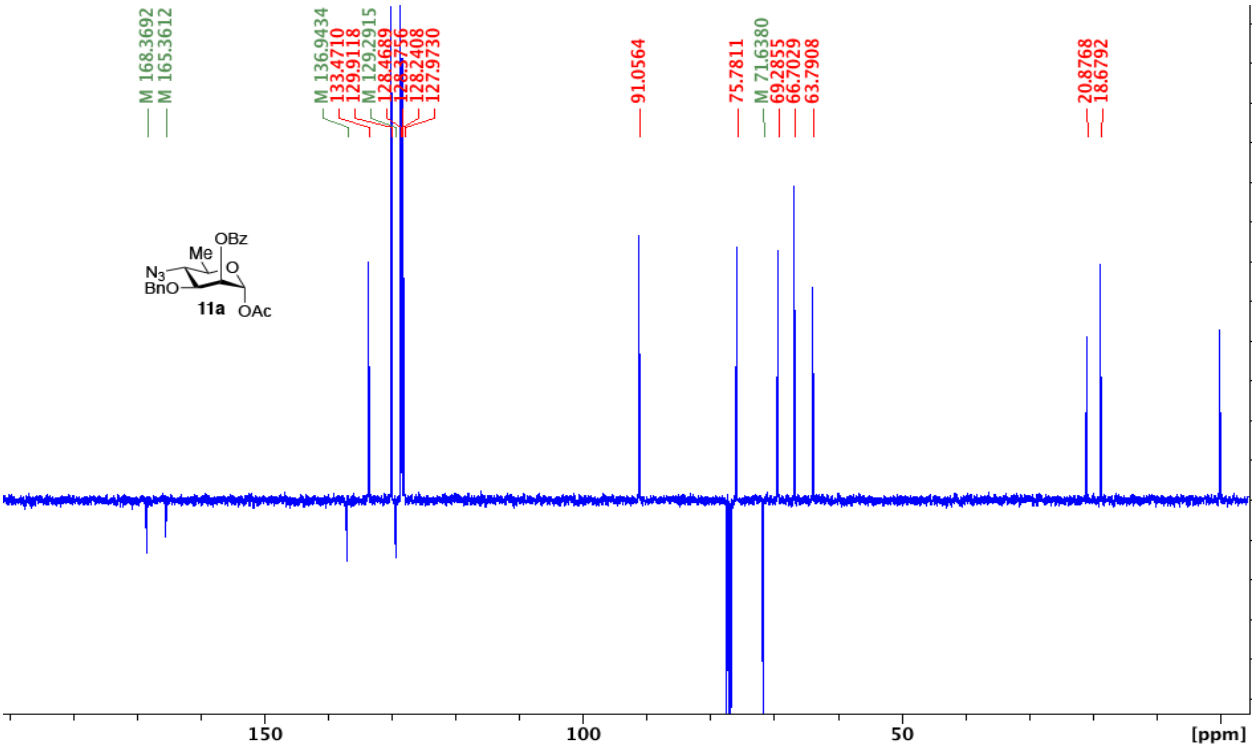

Fig. S2: ^{13}C{1H} NMR spectra of compound **11a** ($CDCl_3$, 100 MHz).

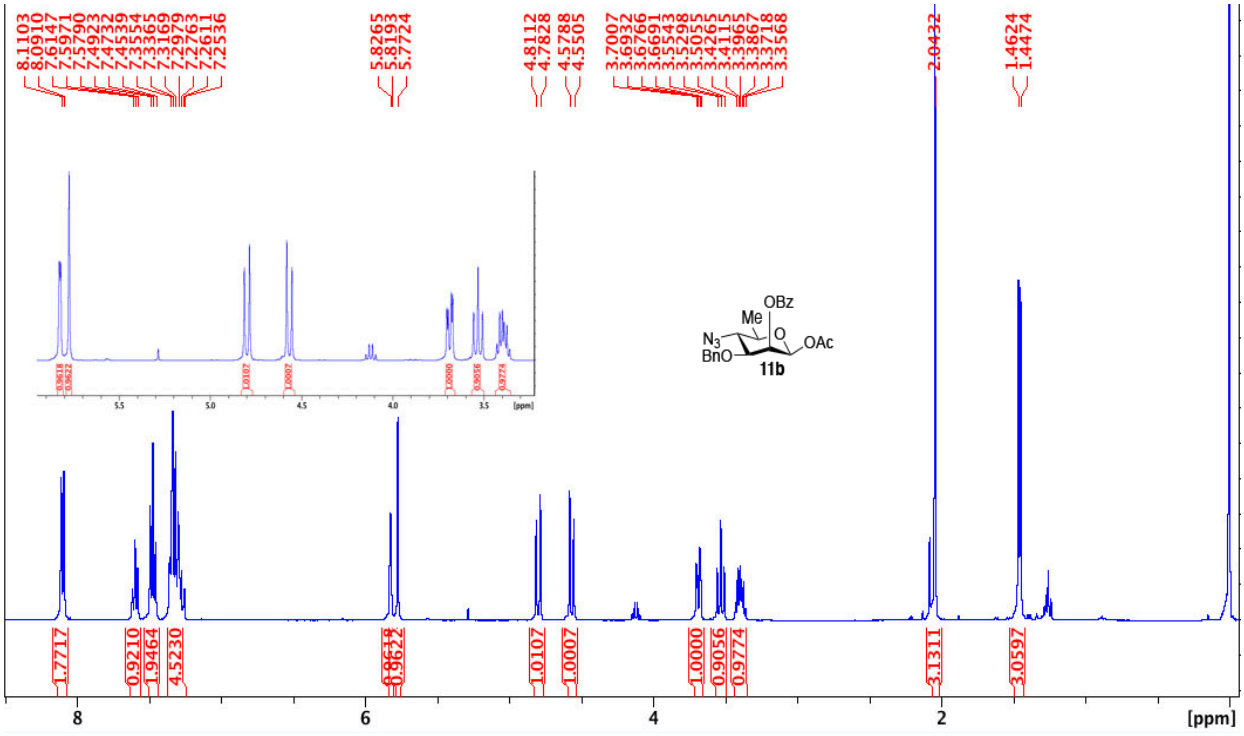

Fig. S3: ^{1}H NMR spectra of compound **11b** ($CDCl_3$, 400 MHz).

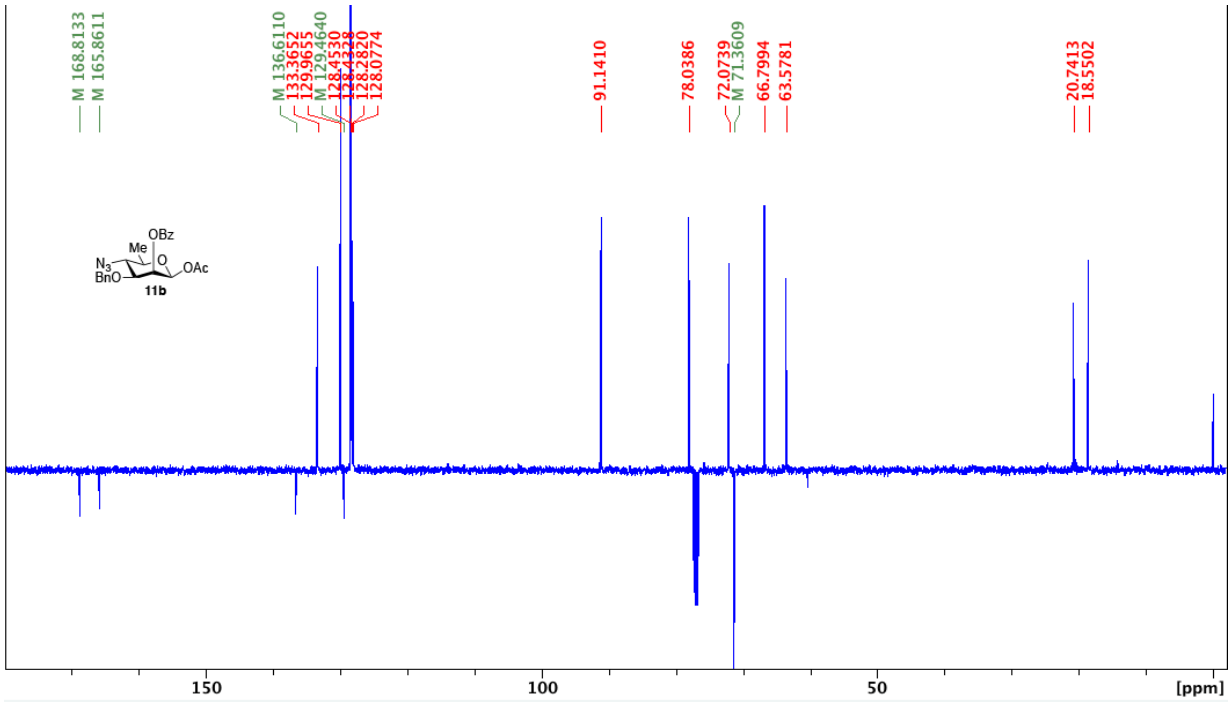

Fig. S4: ^{13}C{1H} NMR spectra of compound **11b** ($CDCl_3$, 100 MHz).

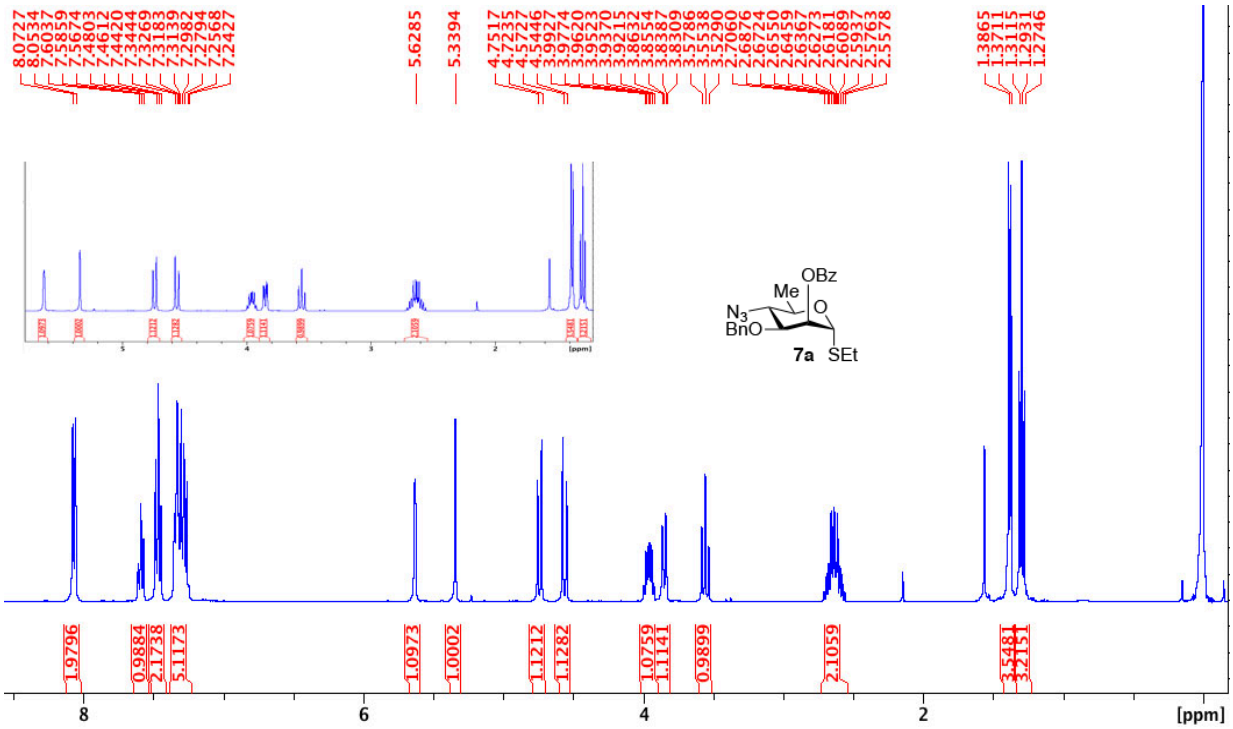

Fig. S5: ^{1}H NMR spectra of compound **7a** ($CDCl_3$, 400 MHz).

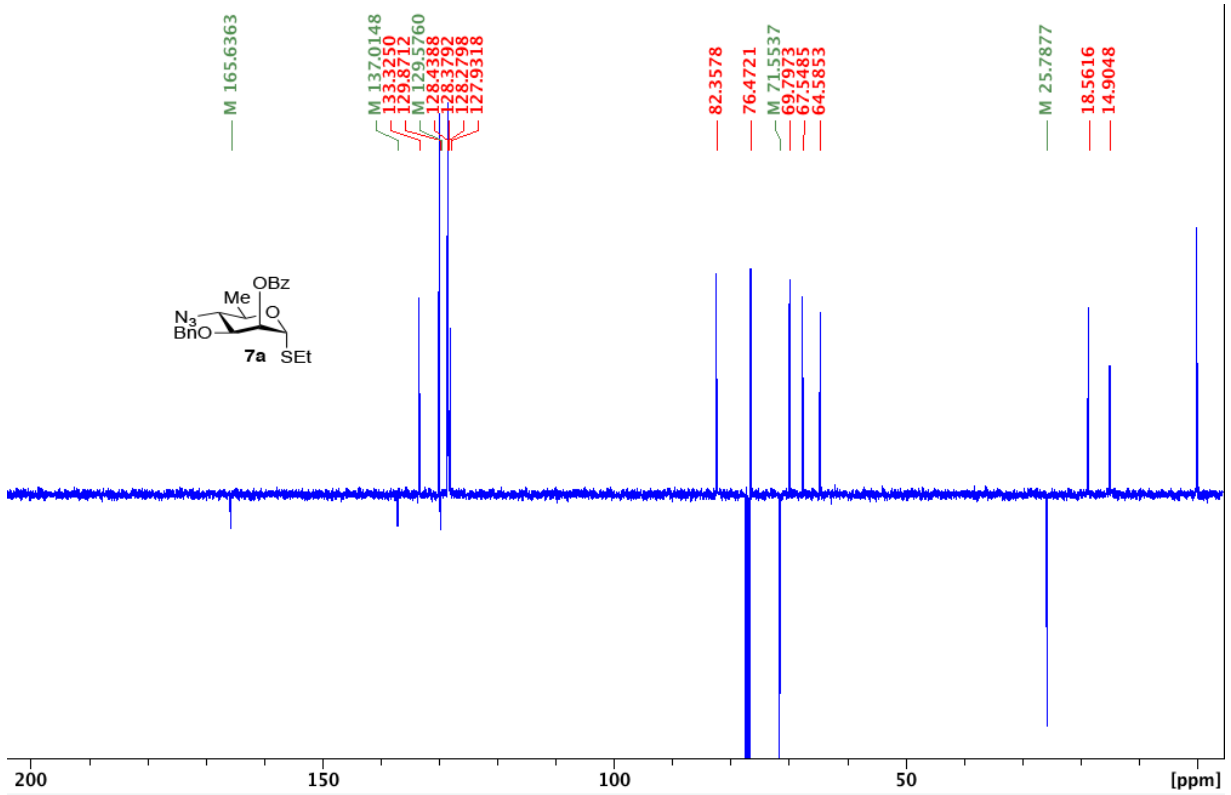

Fig. S6: ^{13}C{1H} NMR spectra of compound **7a** ($CDCl_3$, 100 MHz).

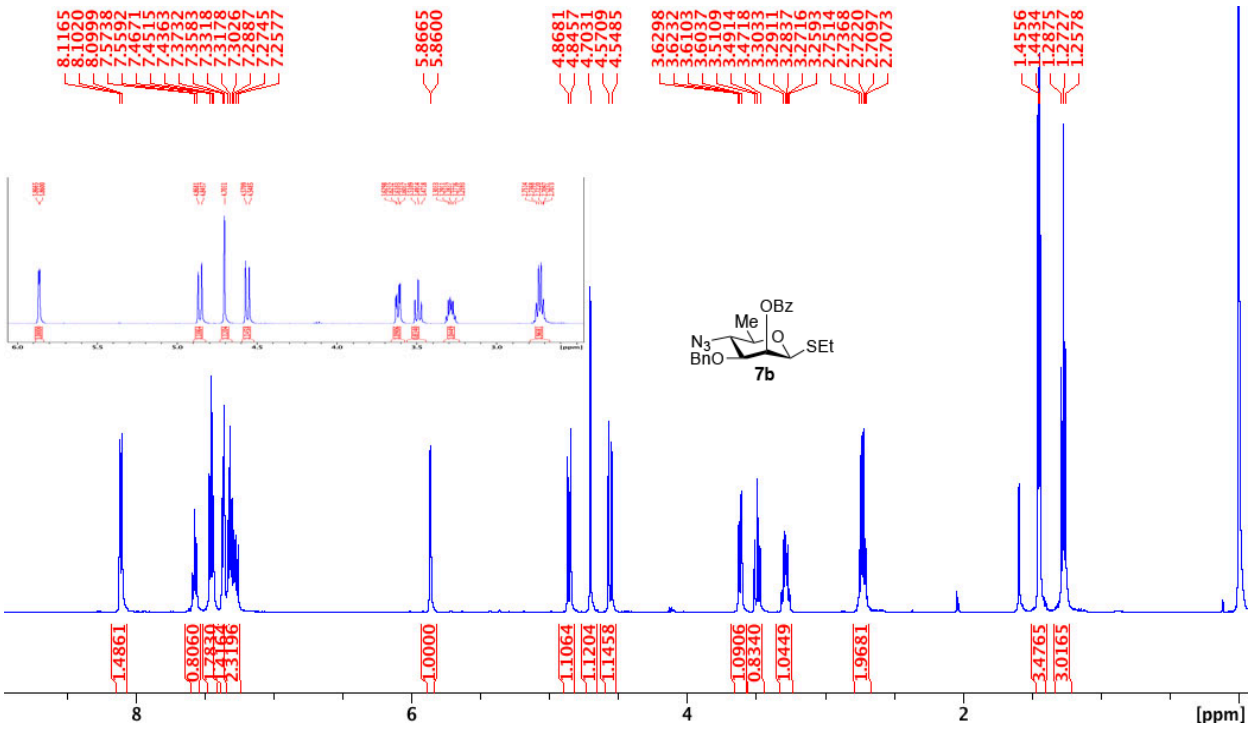

Fig. S7: ^{1}H NMR spectra of compound **7b** ($CDCl_3$, 500 MHz).

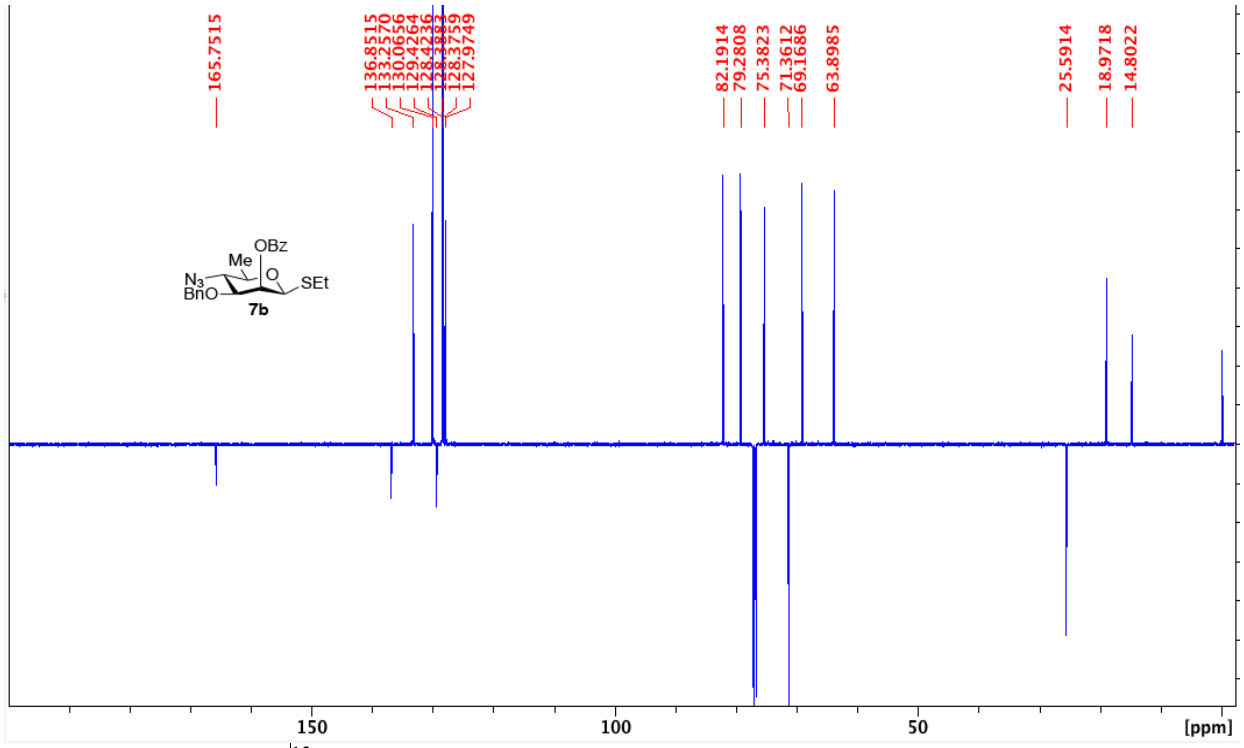

Fig. S8: ^{13}C{1H} NMR spectra of compound **7b** ($CDCl_3$, 125 MHz).

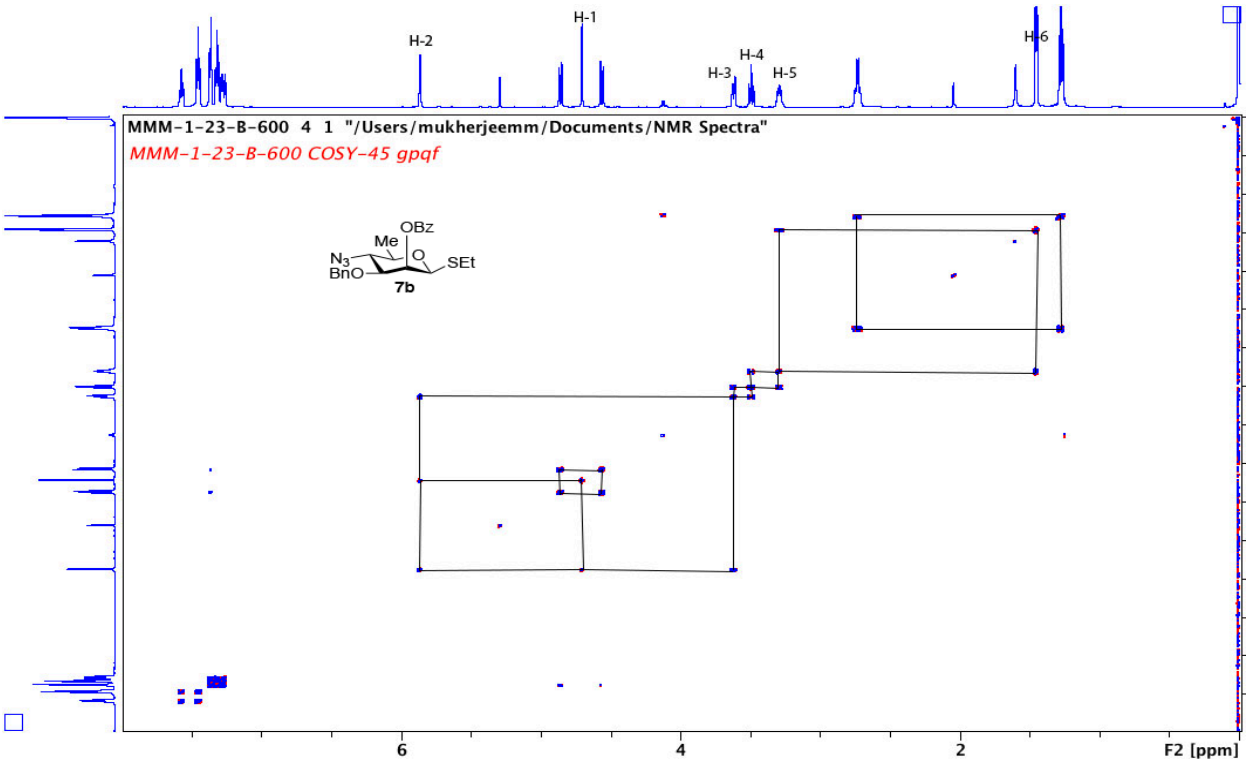

Fig. S9: COSY NMR spectra of compound **7b** ($CDCl_3$, 500 MHz).

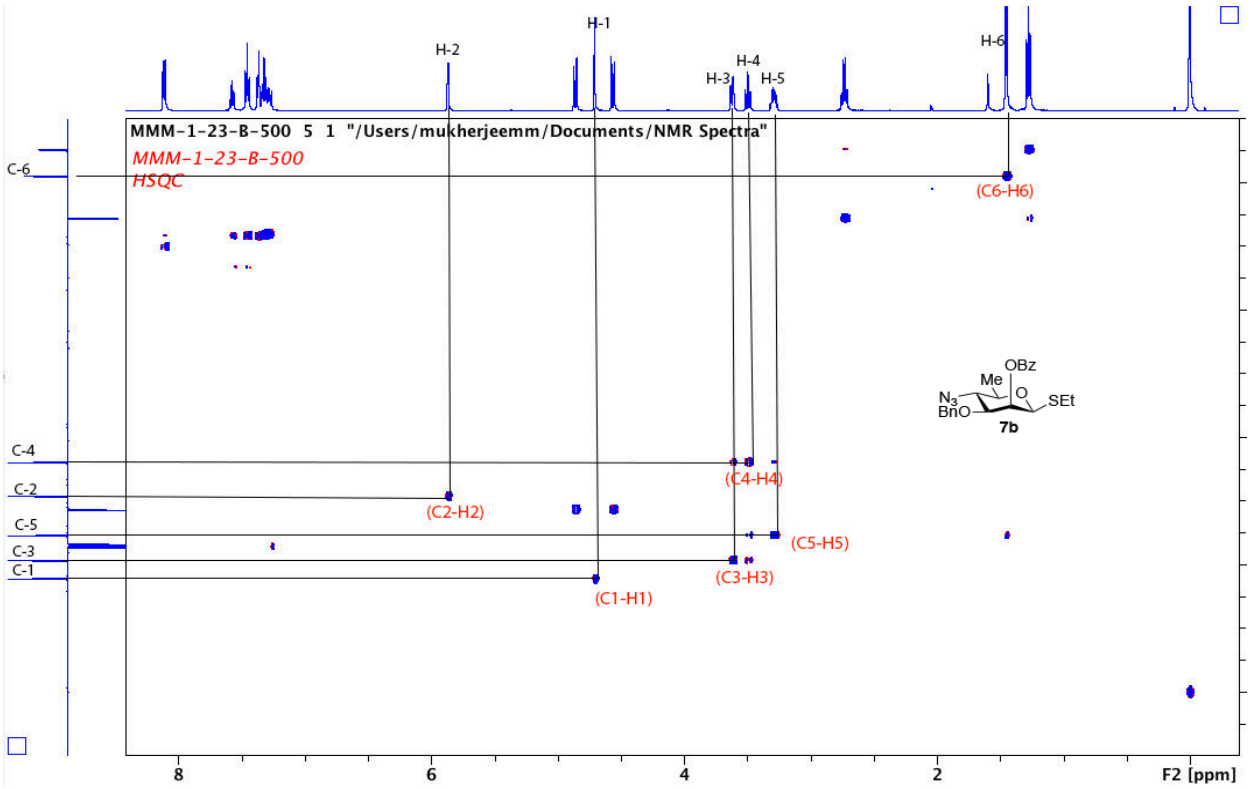

Fig. S10: HSQC NMR spectra of compound **7b** ($CDCl_3$).

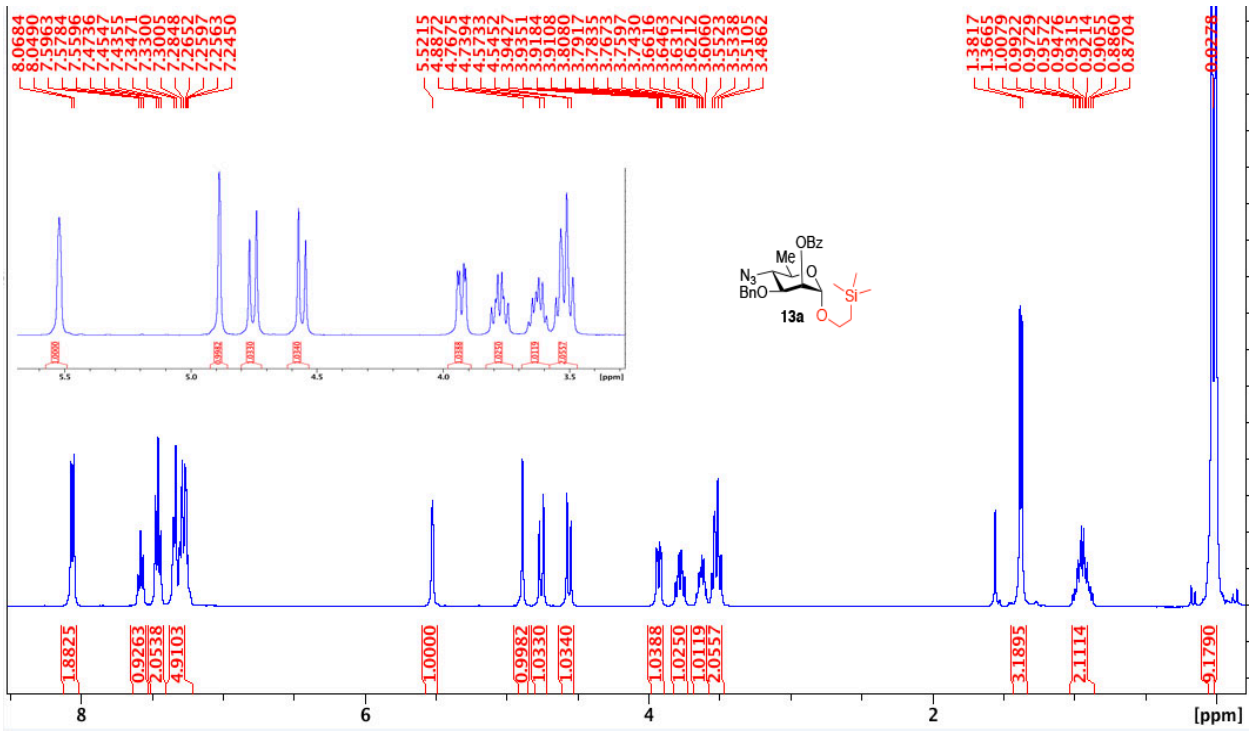

Fig. S11: ^{1}H NMR spectra of compound **13a** ($CDCl_3$, 400 MHz).

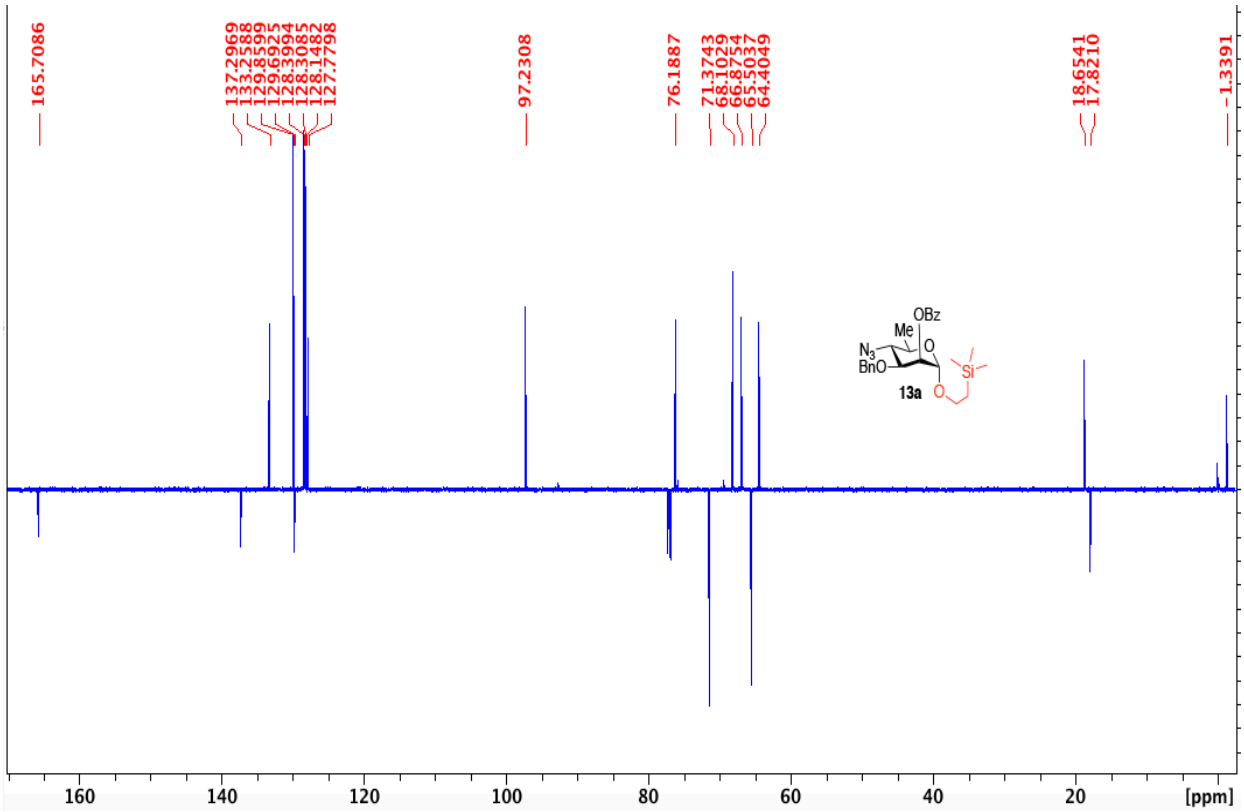

Fig. S12: ^{13}C{1H} NMR spectra of compound **13a** ($CDCl_3$, 100 MHz).

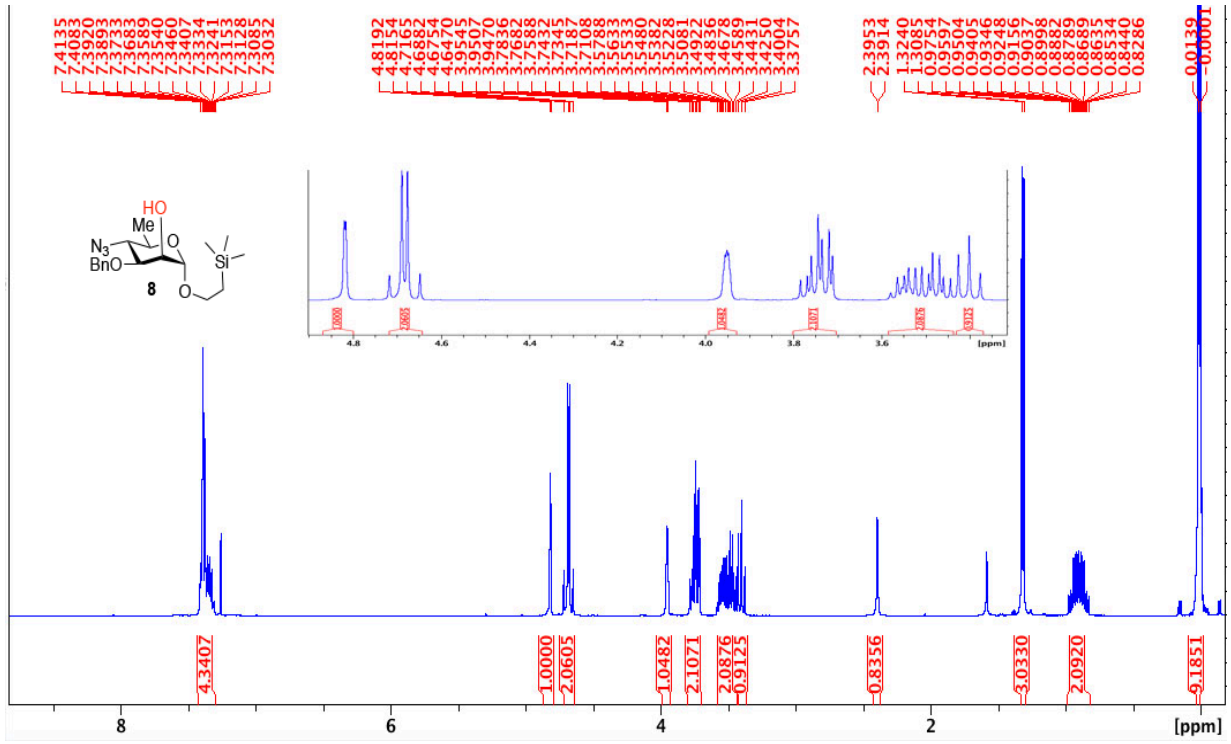

Fig. S13: ^{1}H NMR spectra of compound **8** ($CDCl_3$, 400 MHz).

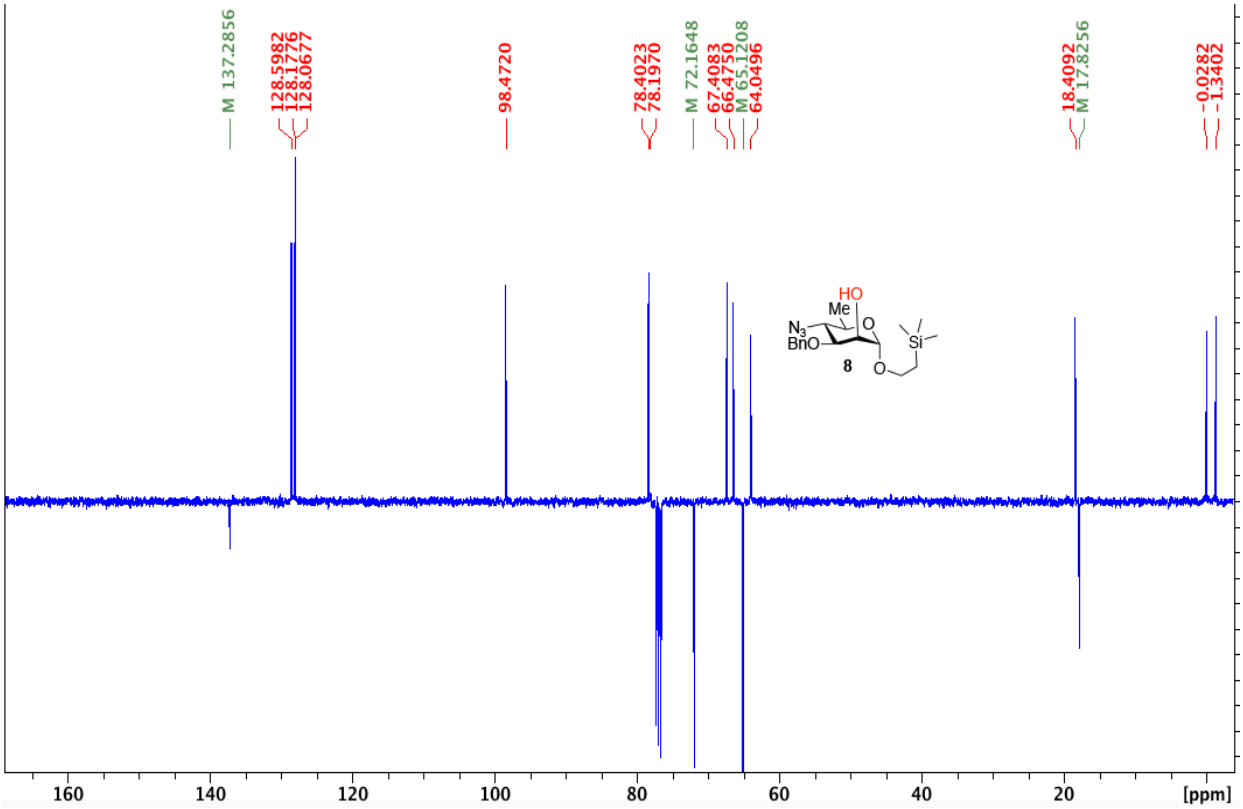

Fig. S14: $^{13}C\{1H\}$ NMR spectra of compound **8** ($CDCl_3$, 100 MHz).

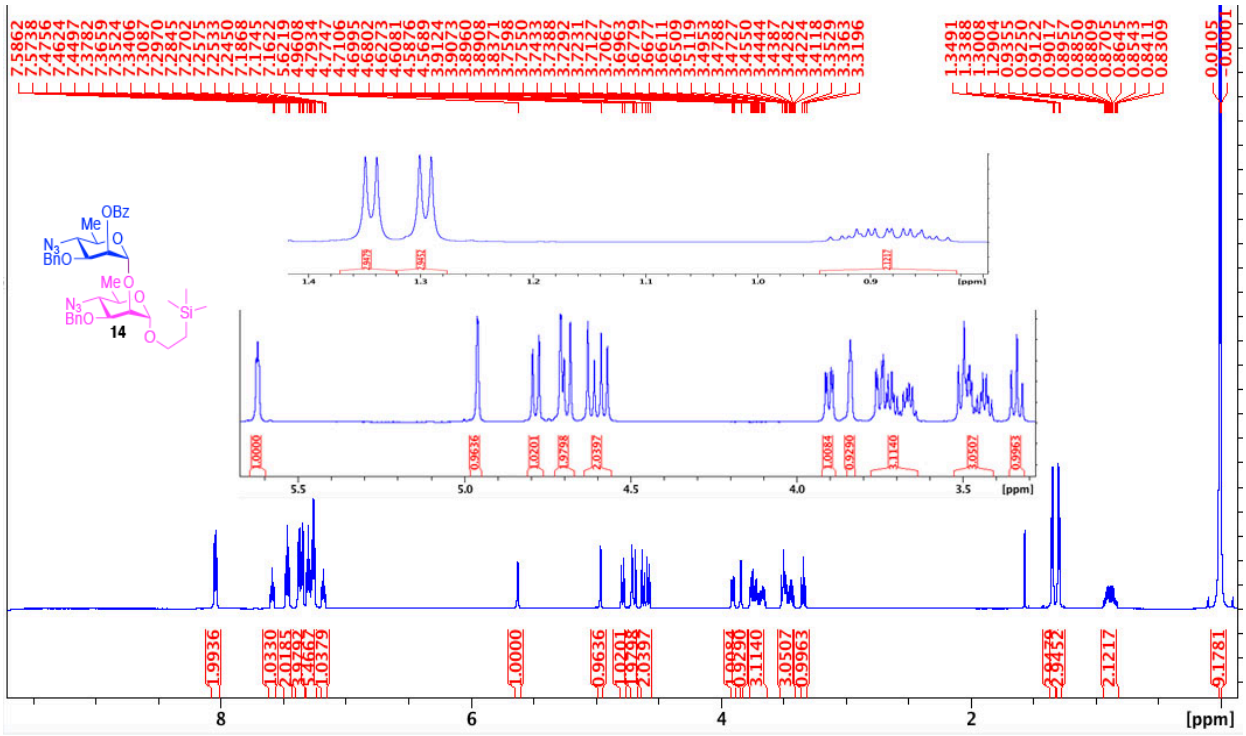

Fig. S15: ^{1}H NMR spectra of compound **14** ($CDCl_3$, 600 MHz).

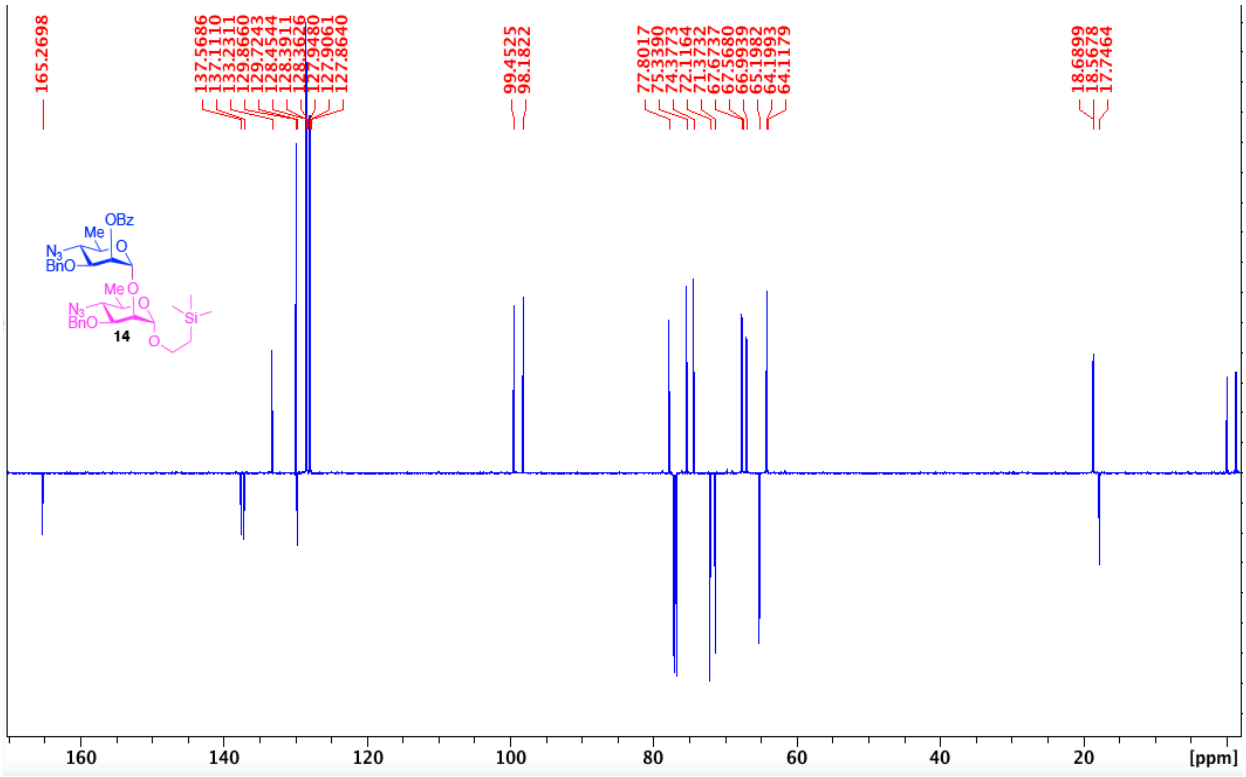

Fig. S16: $^{13}C\{1H\}$ NMR spectra of compound **14** ($CDCl_3$, 150 MHz).

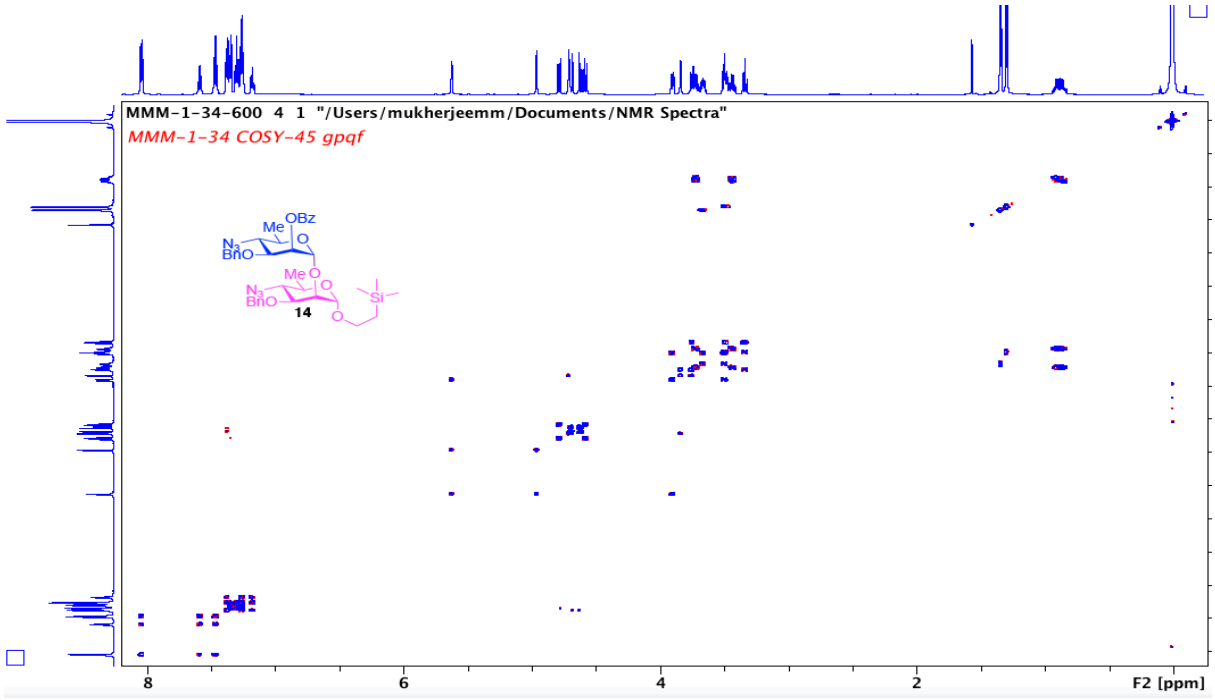

Fig. S17: COSY NMR spectra of compound **14** ($CDCl_3$, 600 MHz).

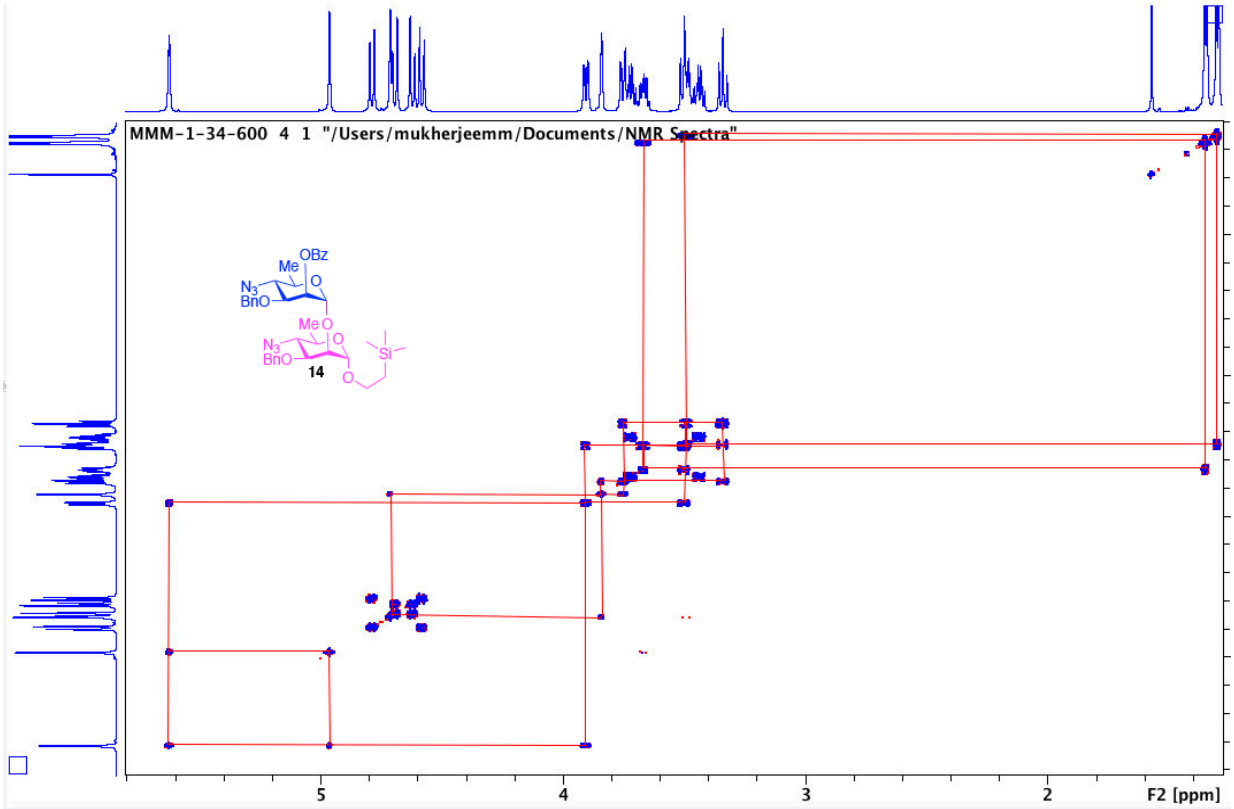

Fig. S18: COSY expansion (1.5 ppm to 5.5 ppm) NMR spectra of compound **14** ($CDCl_3$, 600 MHz).

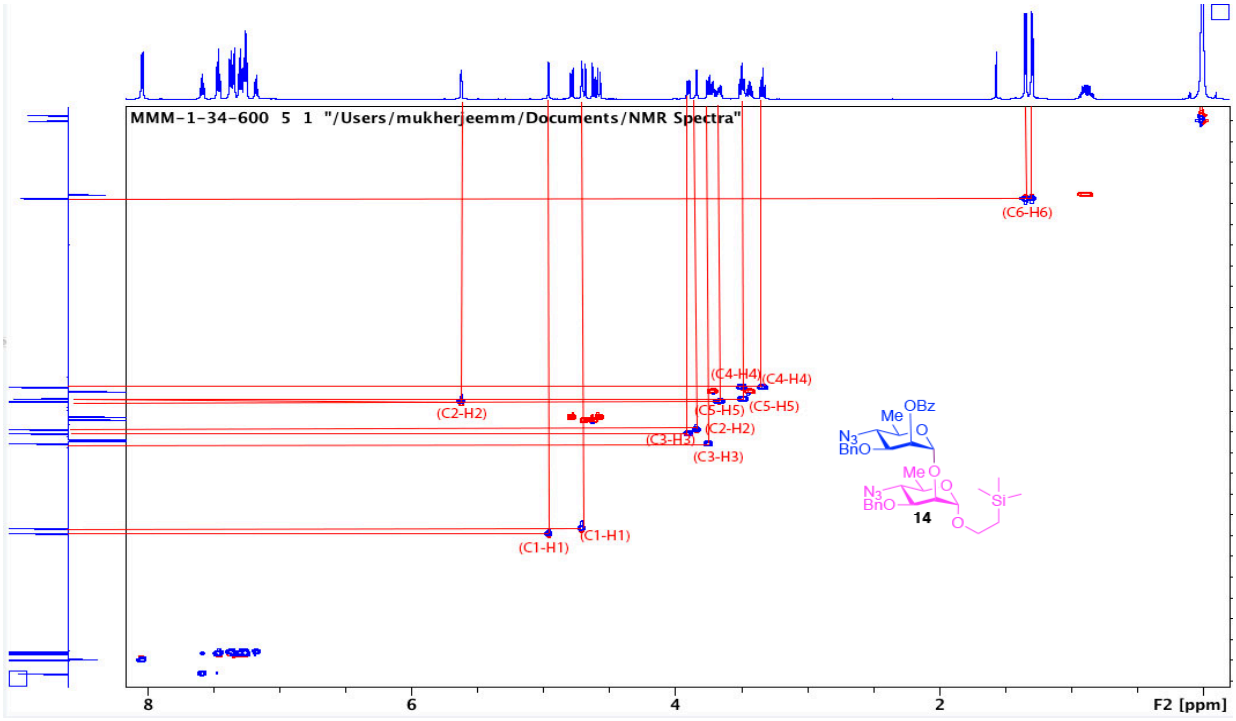

Fig. S19: HSQC NMR spectra of compound **14** ($CDCl_3$).

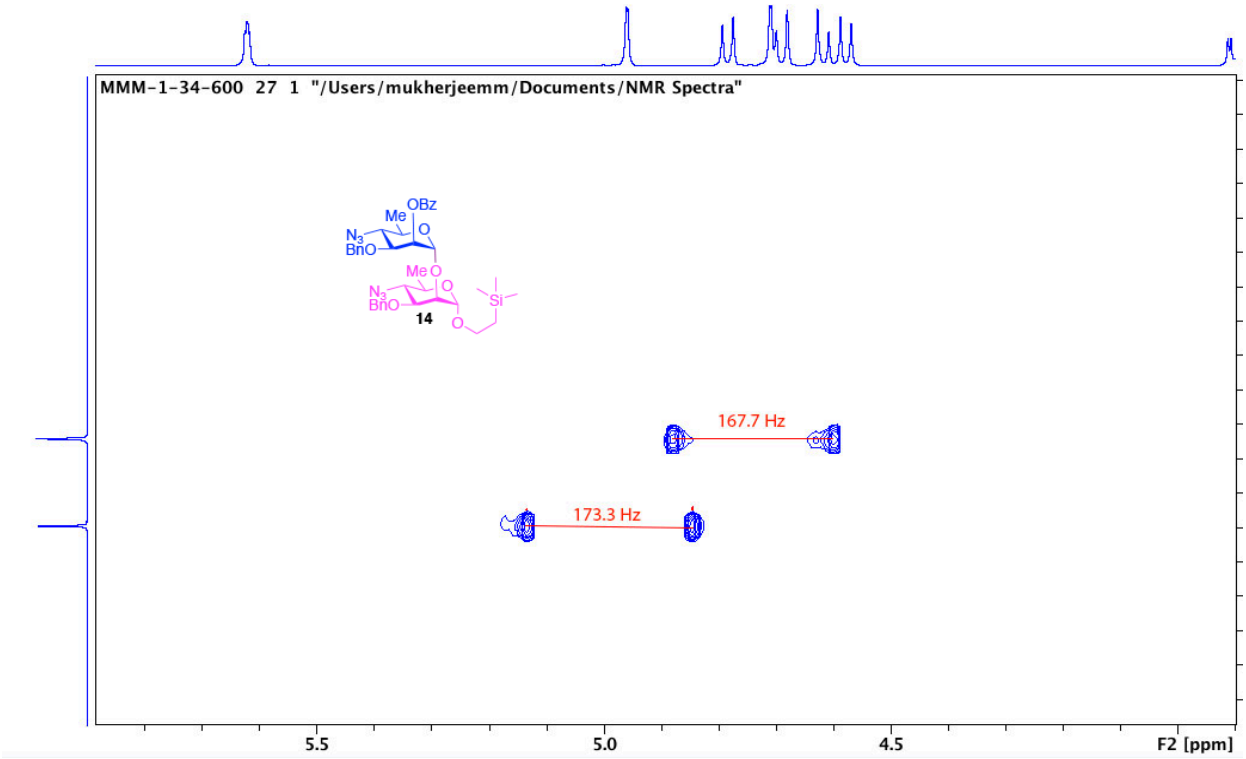

Fig. S20: ^{1}H-13C coupled NMR spectra of compound **14 (**$CDCl_3$).

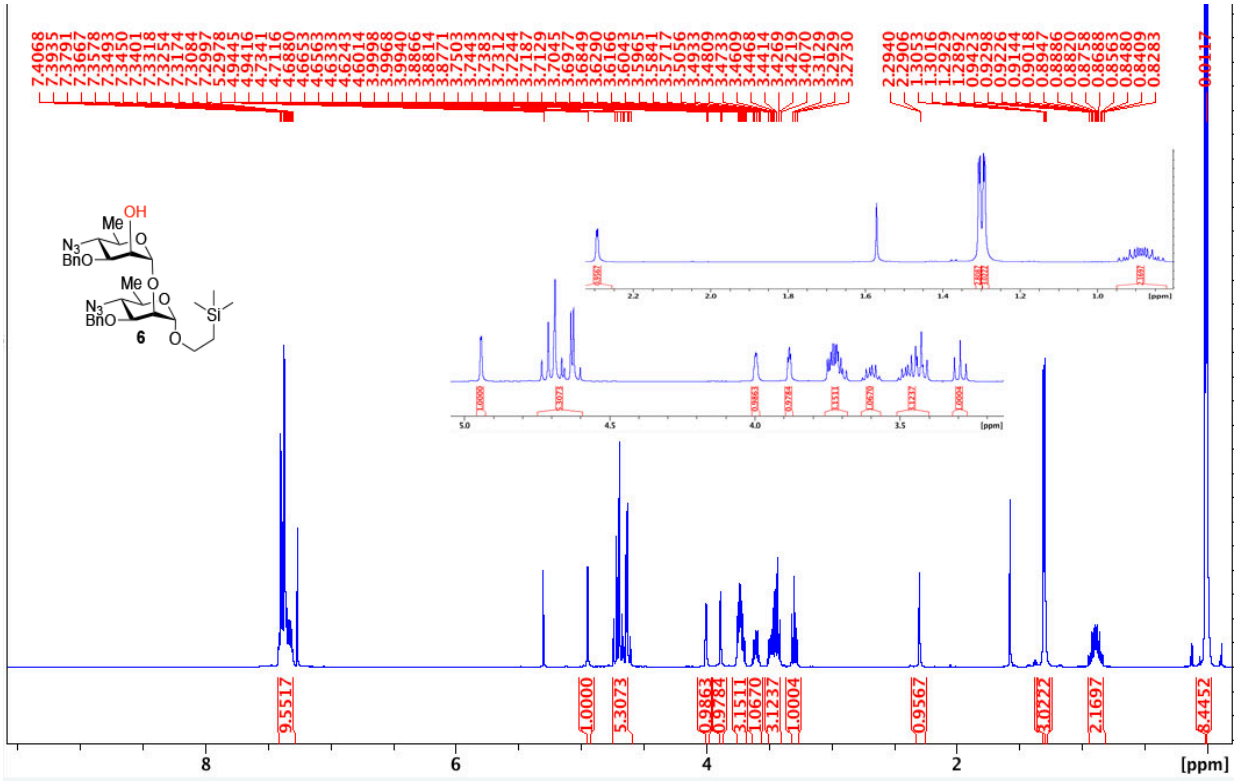

Fig. S21: ^{1}H NMR spectra of compound **6** ($CDCl_3$, 500 MHz).

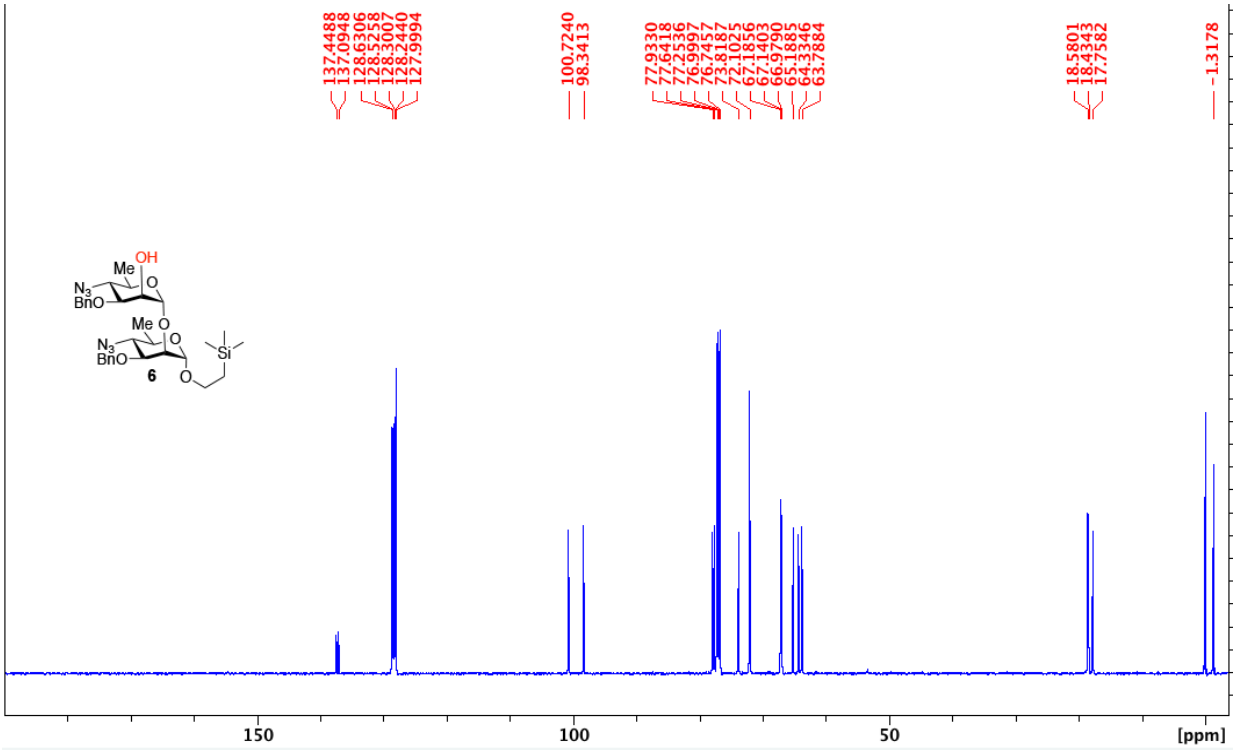

Fig. S22: ^{13}C{1H} NMR spectra of compound **6** ($CDCl_3$, 125 MHz).

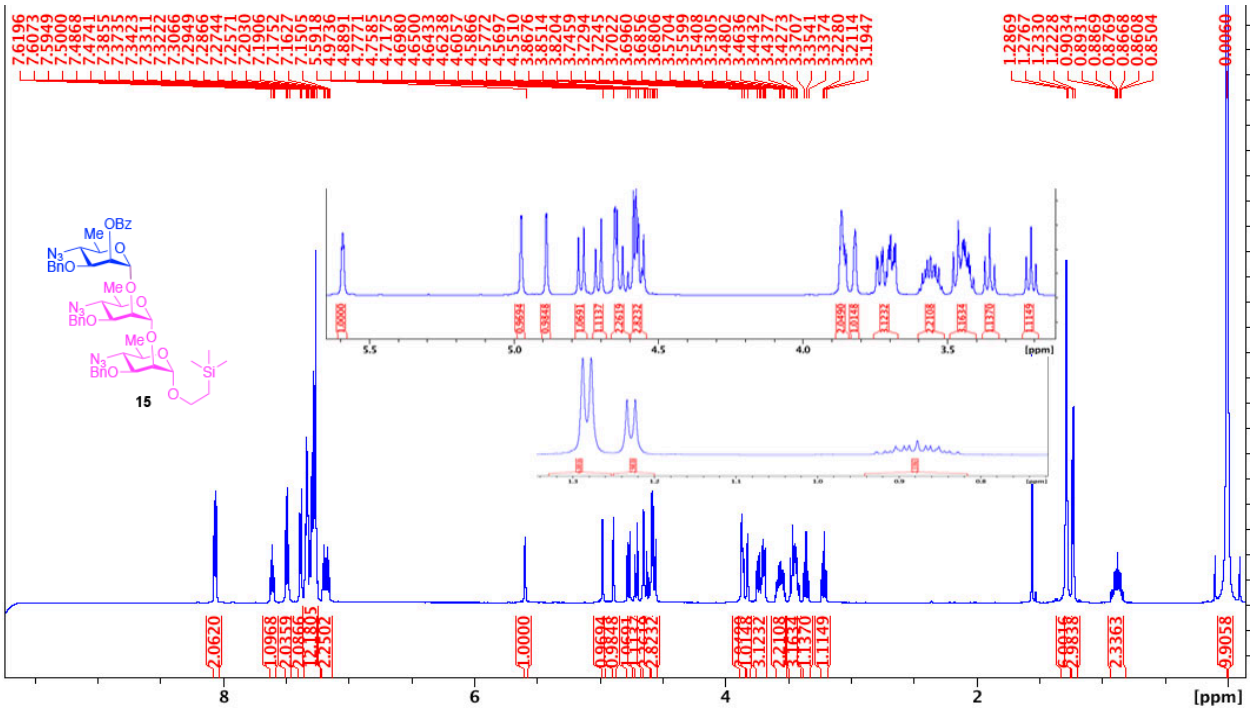

Fig. S23: ^{1}H NMR spectra of compound **15** ($CDCl_3$, 600 MHz).

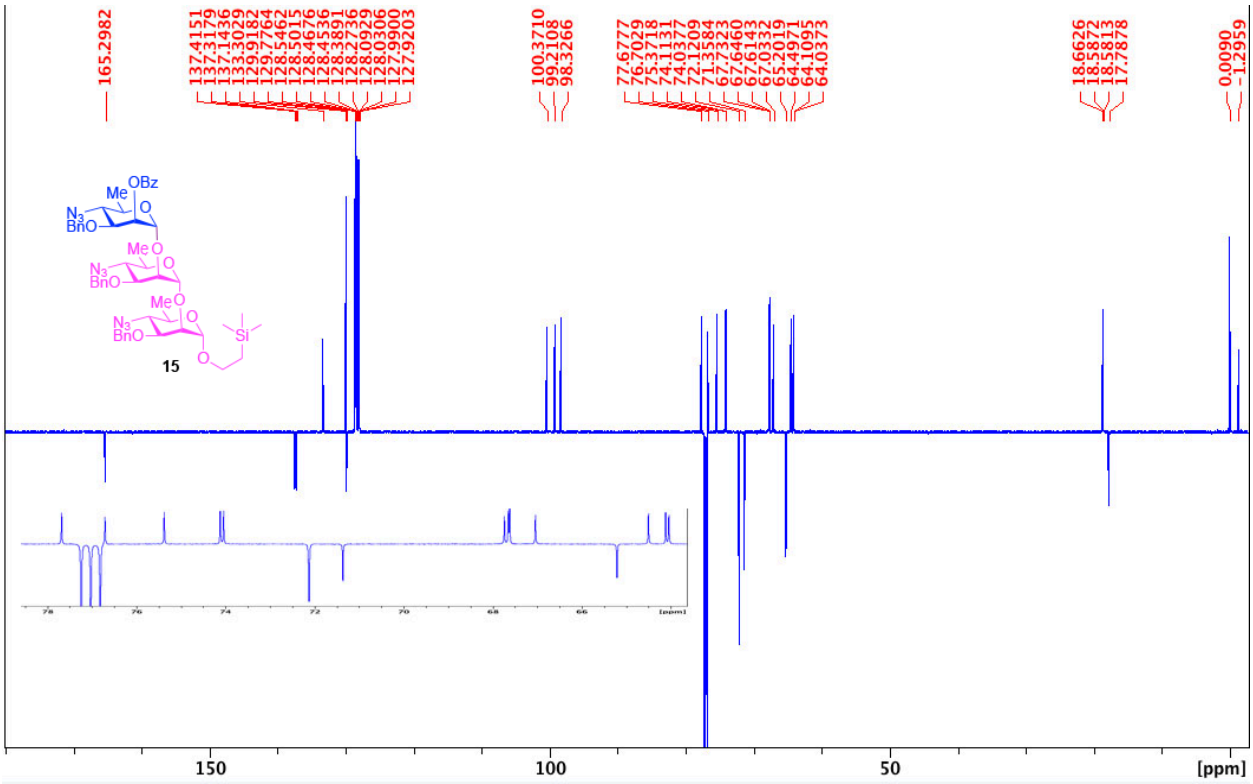

Fig. S24: ^{13}C{1H} NMR spectra of compound **15** ($CDCl_3$, 150 MHz).

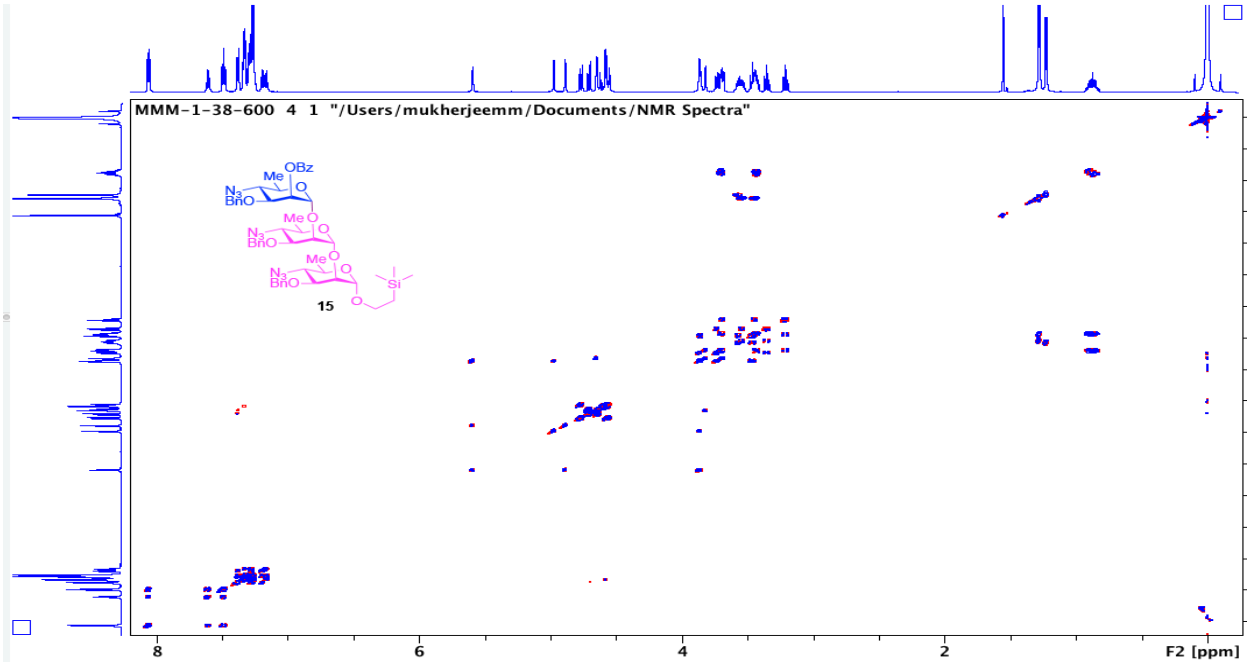

Fig. S25: COSY NMR spectra of compound **15** ($CDCl_3$, 600 MHz).

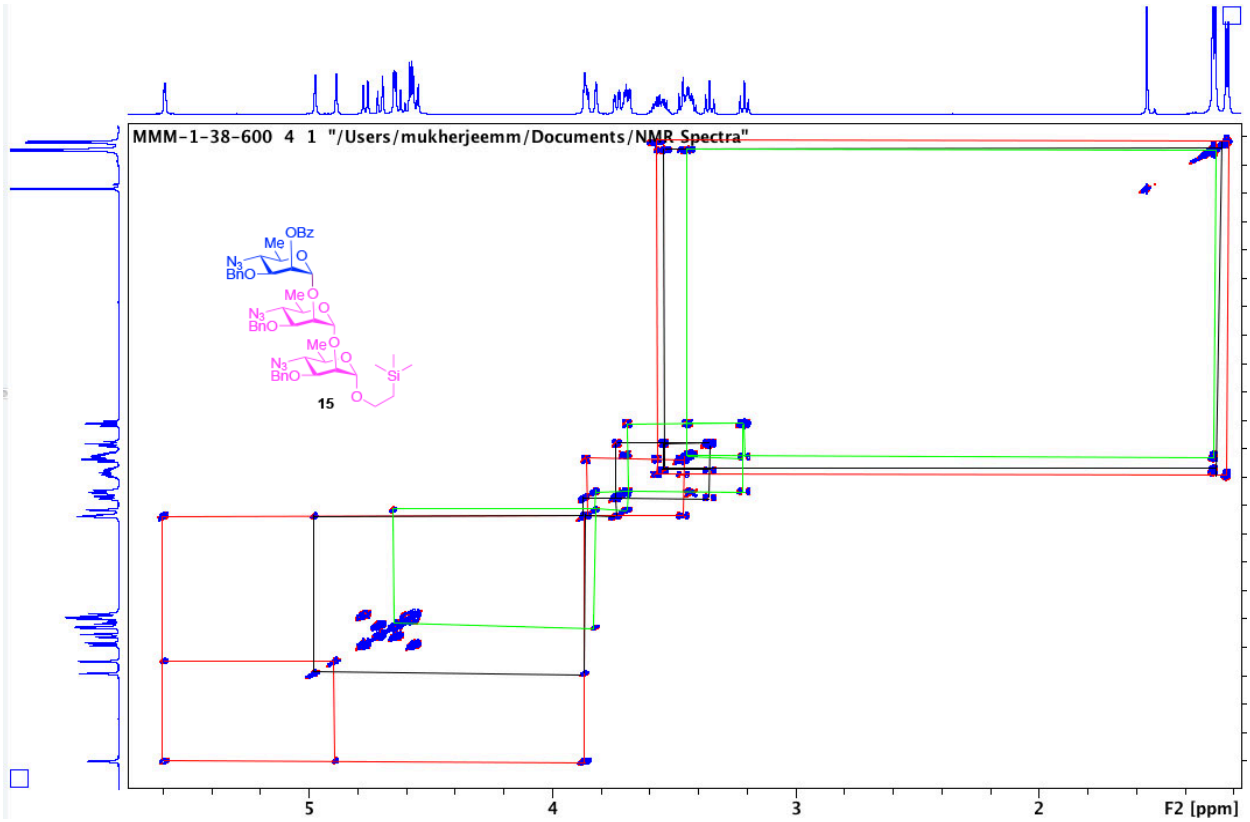

Fig. S26: COSY expansion (1.5 ppm to 5.5 ppm) NMR spectra of compound **15** ($CDCl_3$, 600 MHz).

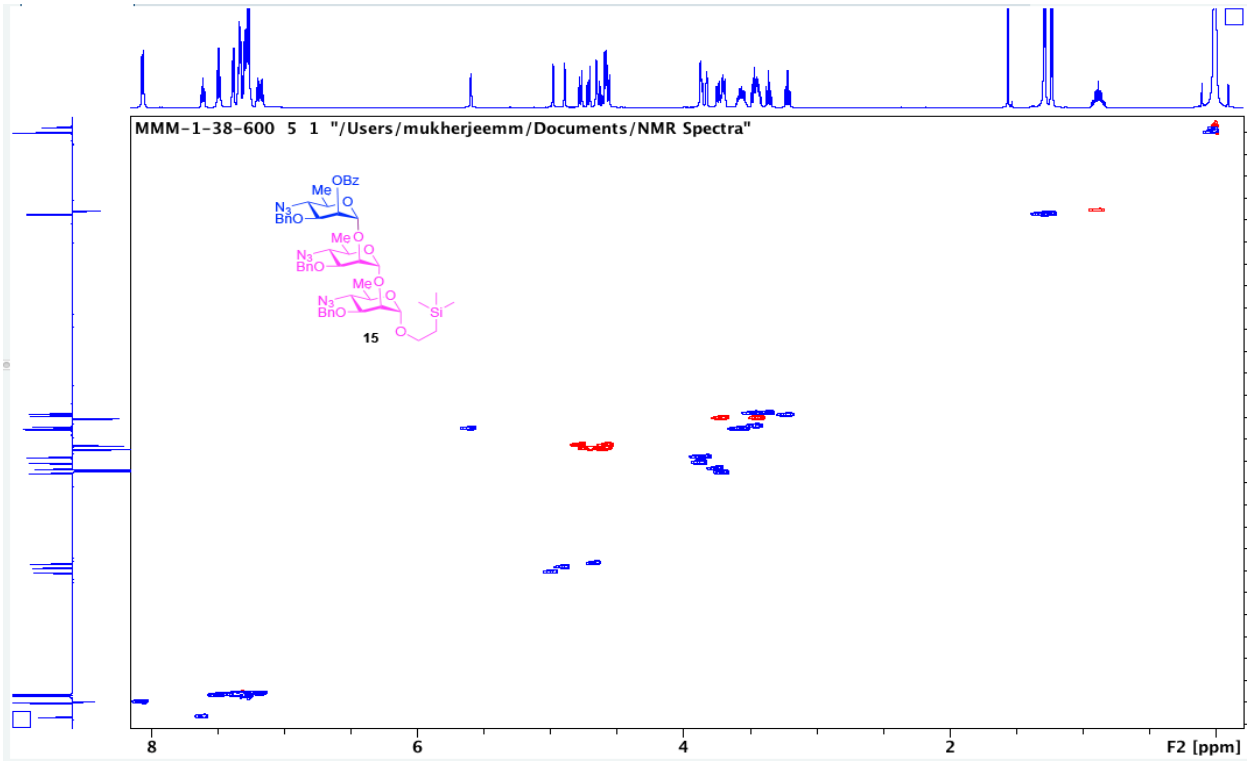

Fig. S27: HSQC NMR spectra of compound **15** ($CDCl_3$).

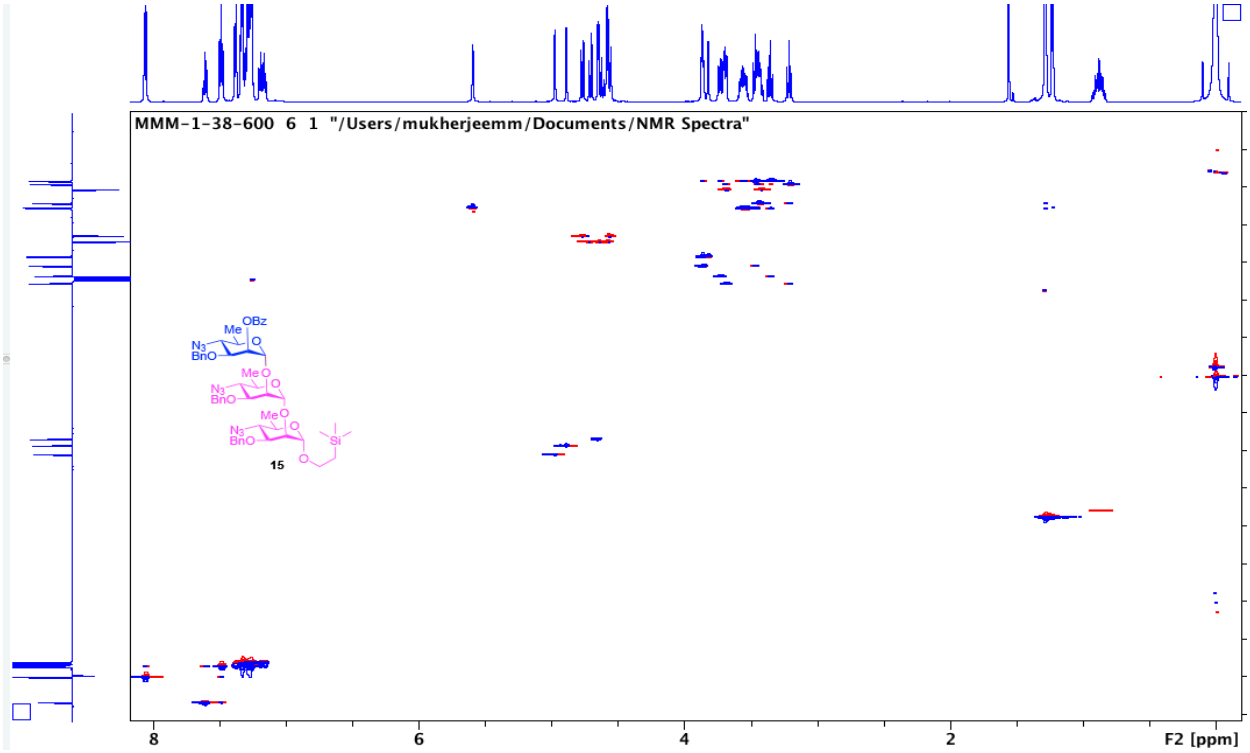

Fig. S28: HMBC NMR spectra of compound **15** ($CDCl_3$).

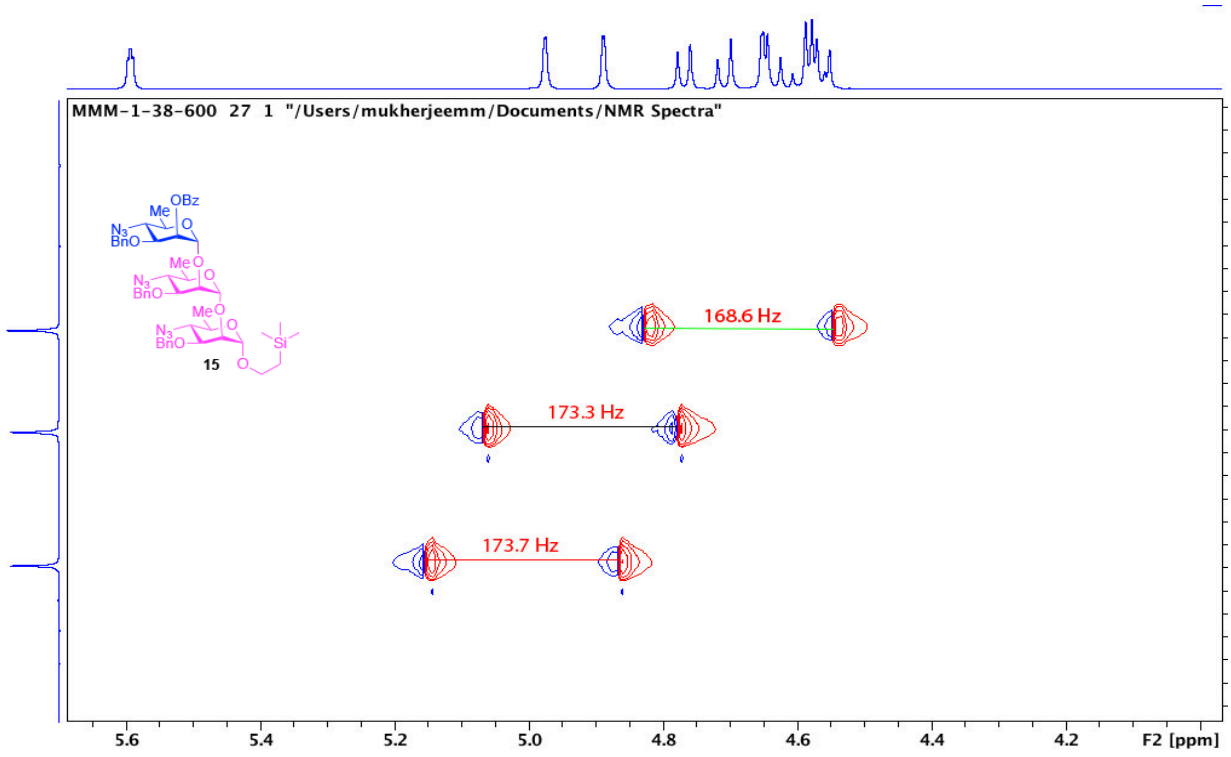

Fig. S29: ^{1}H-^{13}C Coupled NMR spectra of compound **15** ($CDCl_3$).

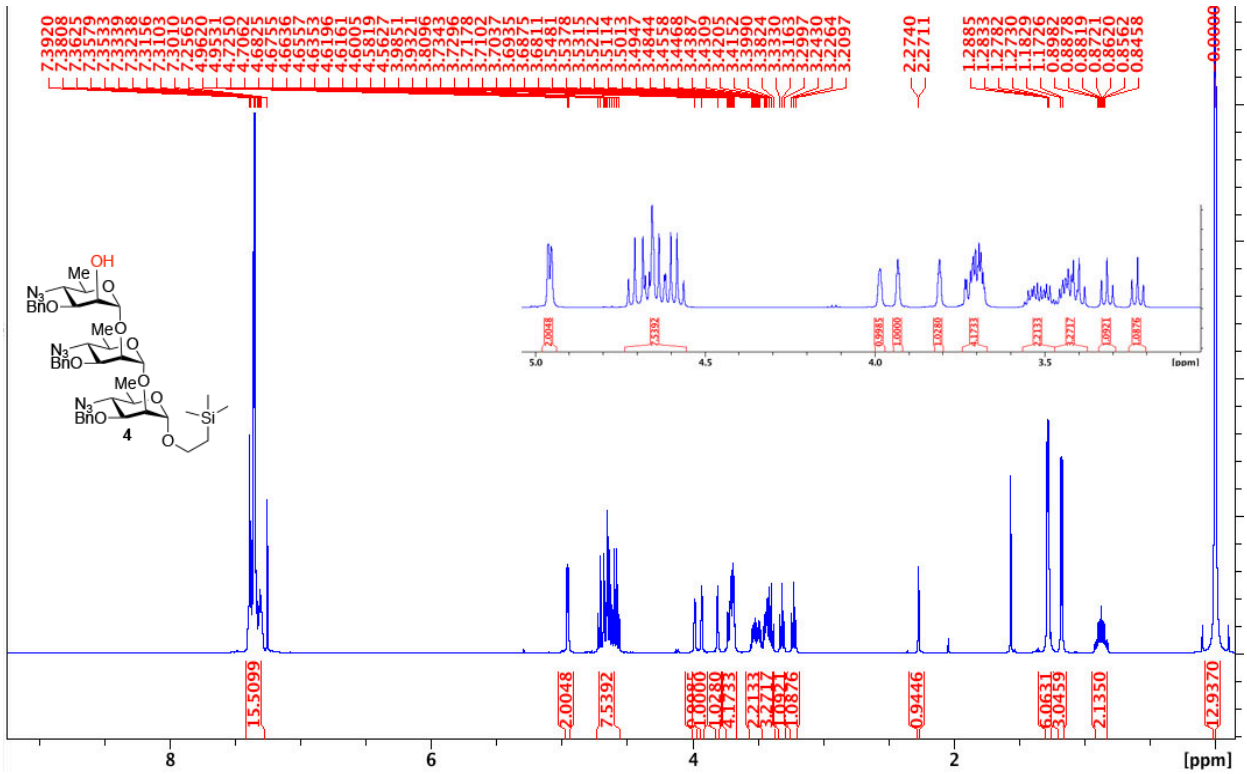

Fig. S30: ^{1}H NMR spectra of compound **4** ($CDCl_3$, 600 MHz).

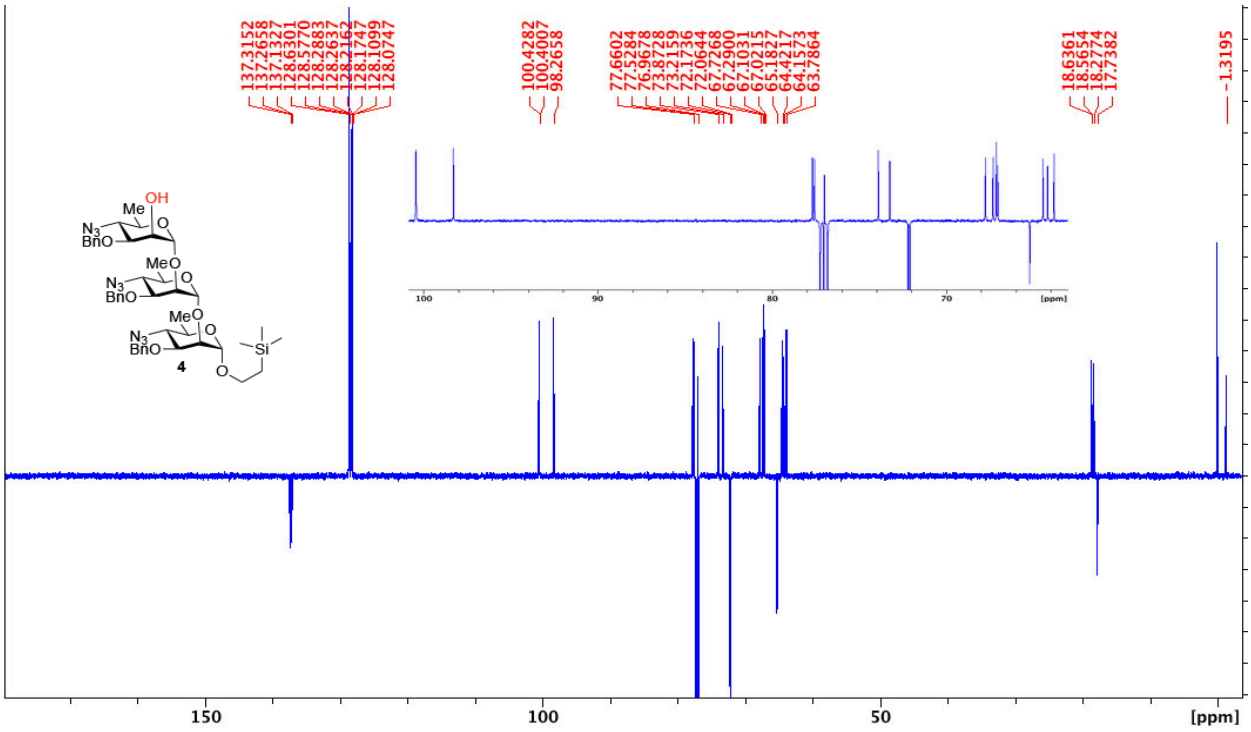

Fig. S31: $^{13}C\{1H\}$ NMR spectra of compound **4** ($CDCl_3$, 150 MHz).

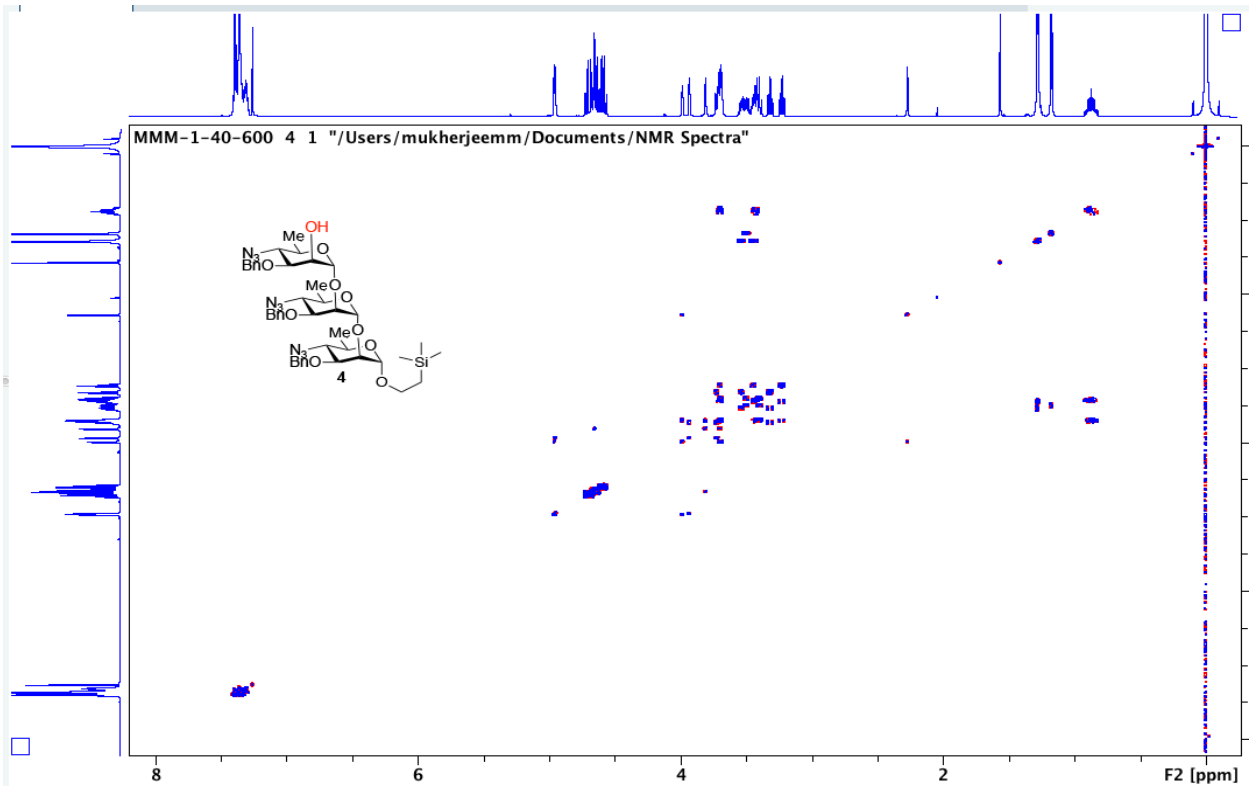

Fig. S32: COSY NMR spectra of compound **4** ($CDCl_3$, 600 MHz).

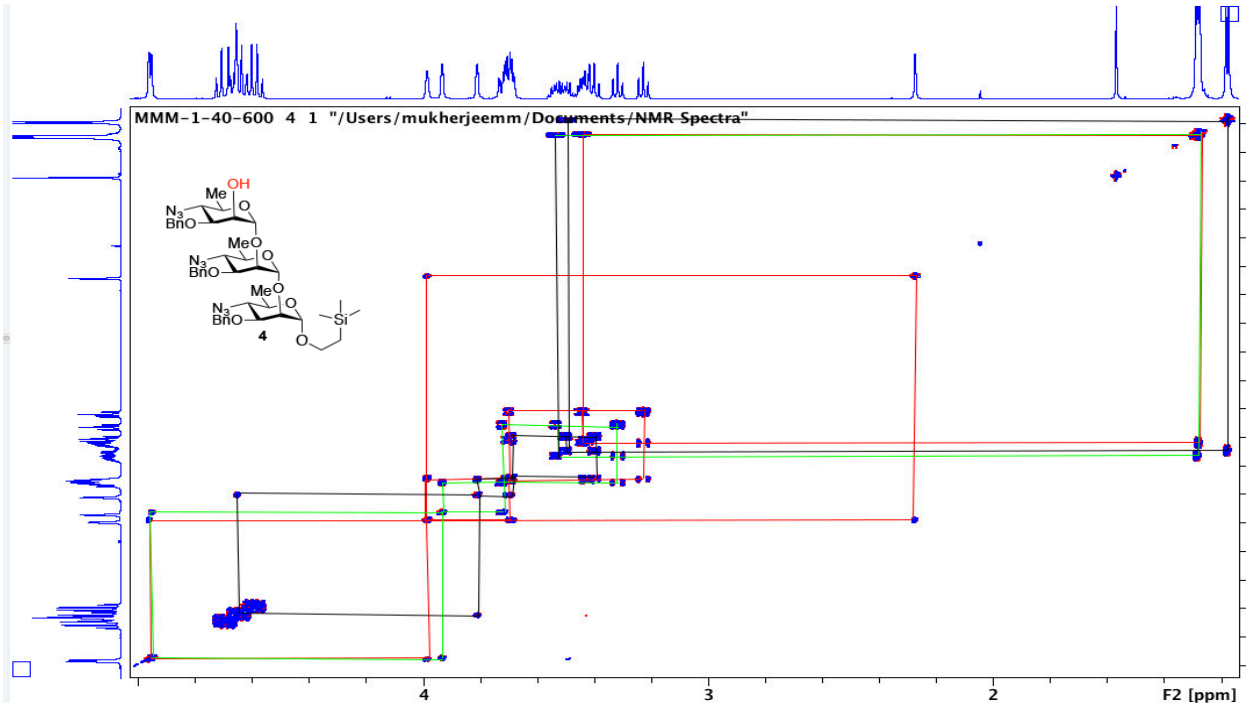

Fig. S33: COSY expansion (1.5 ppm to 5 ppm) NMR spectra of compound **4** ($CDCl_3$, 600 MHz).

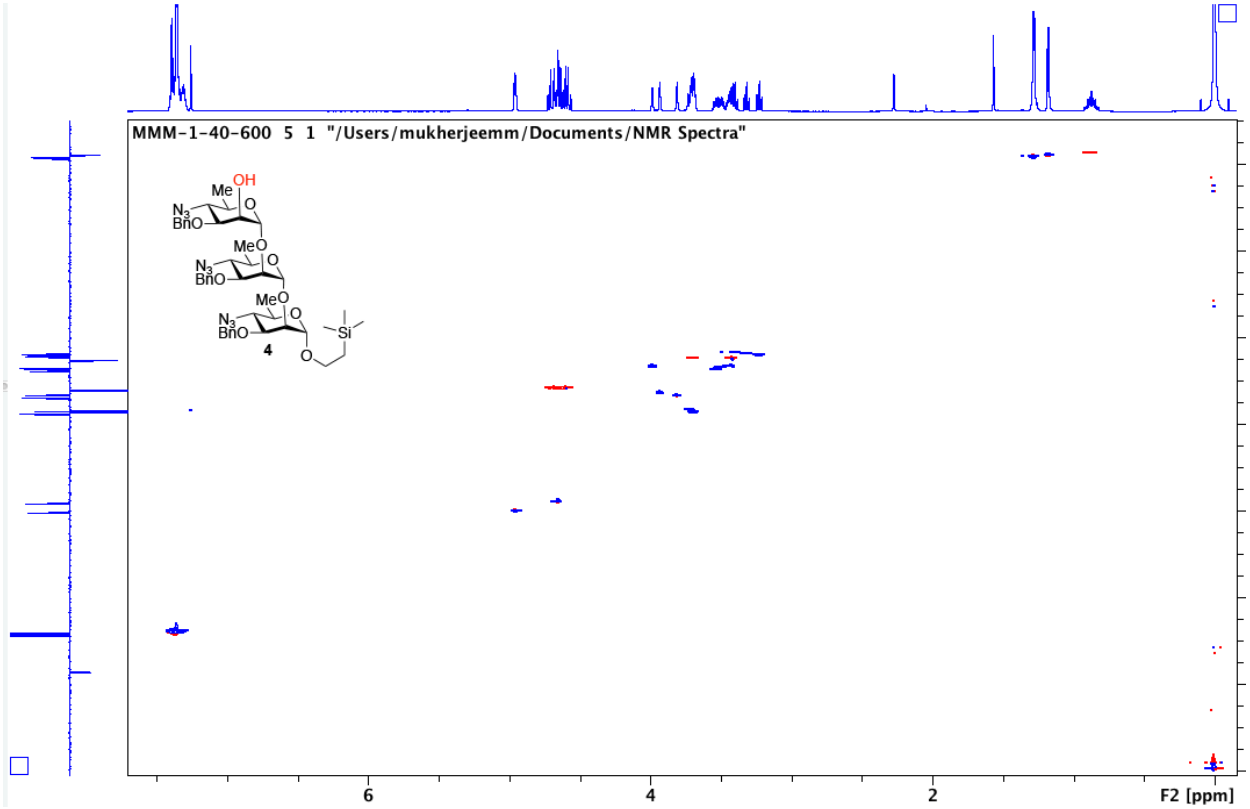

Fig. S34: HSQC NMR spectra of compound **4** ($CDCl_3$).

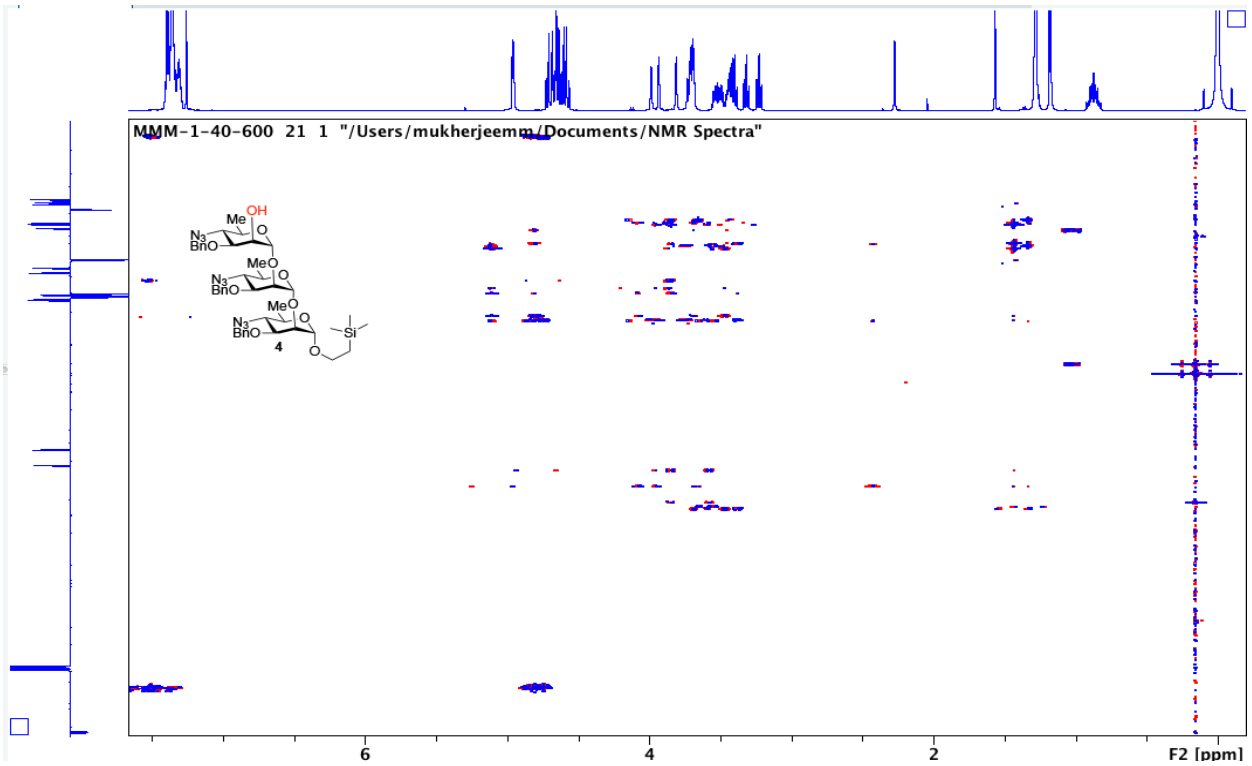

Fig. S35: HMBC NMR spectra of compound **4** ($CDCl_3$).

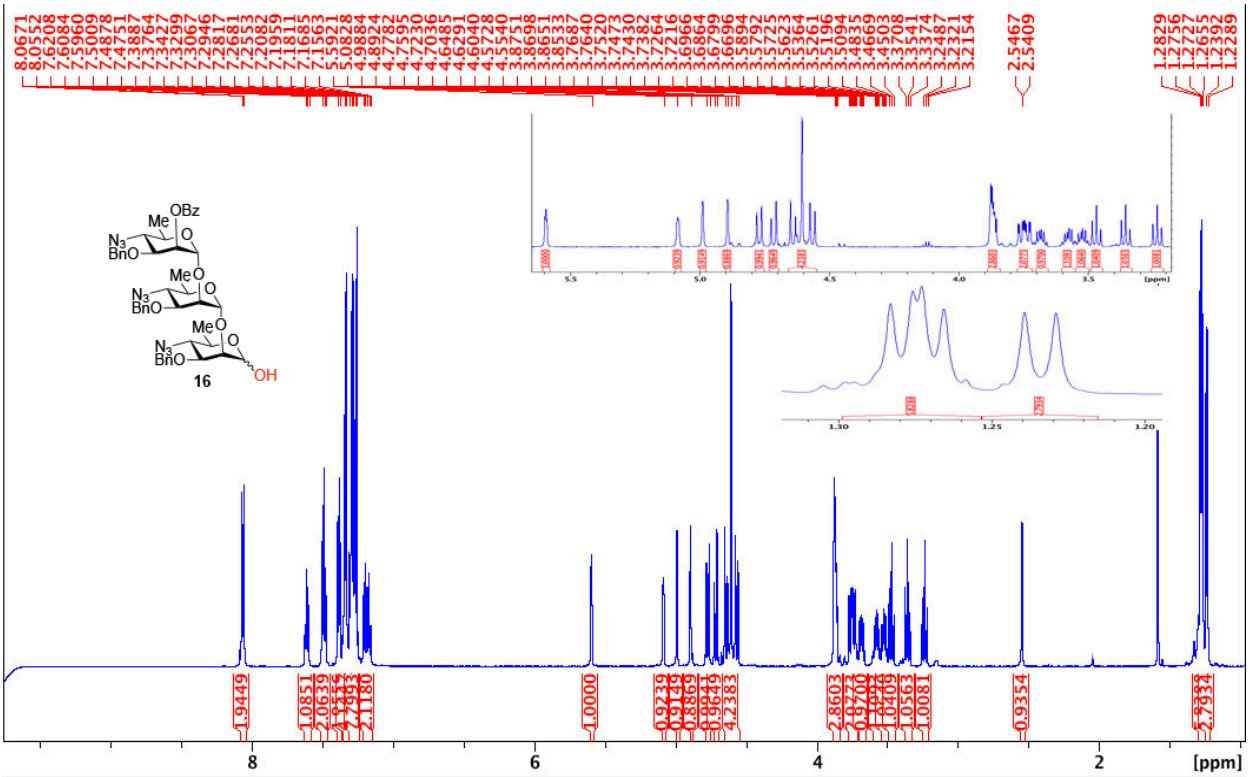

Fig. S36: ^1H NMR spectra of compound **16** ($CDCl_3$, 600 MHz).

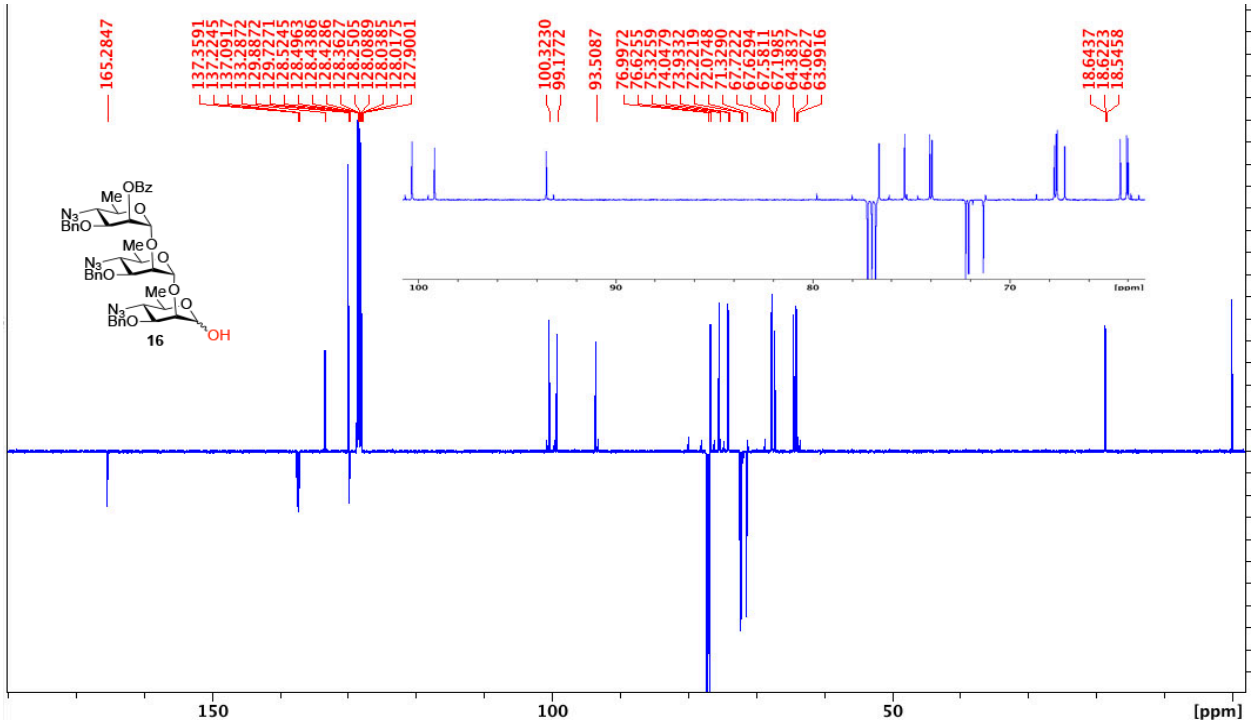

Fig. S37: $^{13}C\{1H\}$ NMR spectra of compound **16** ($CDCl_3$, 150 MHz).

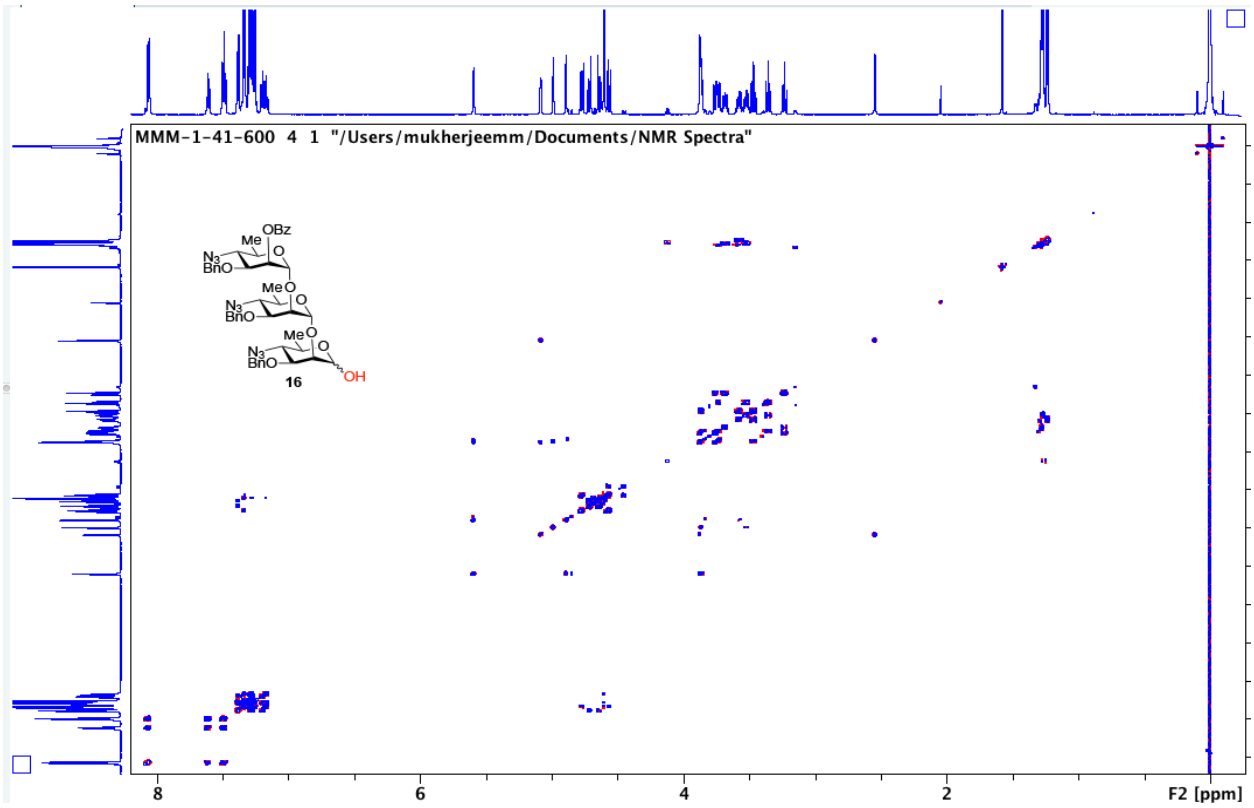

Fig. S38: COSY NMR spectra of compound **16** ($CDCl_3$, 600 MHz).

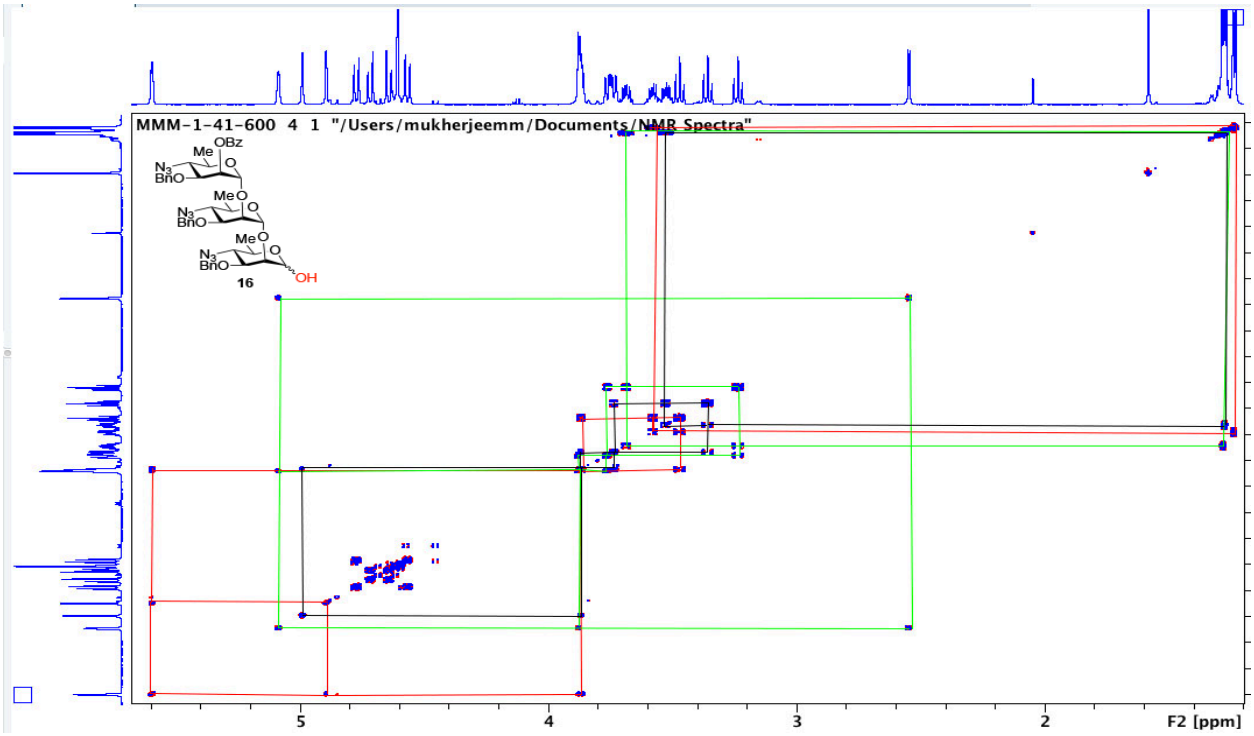

Fig. S39: COSY expansion (1.5 ppm to 5 ppm) NMR spectra of compound **16** ($CDCl_3$, 600 MHz).

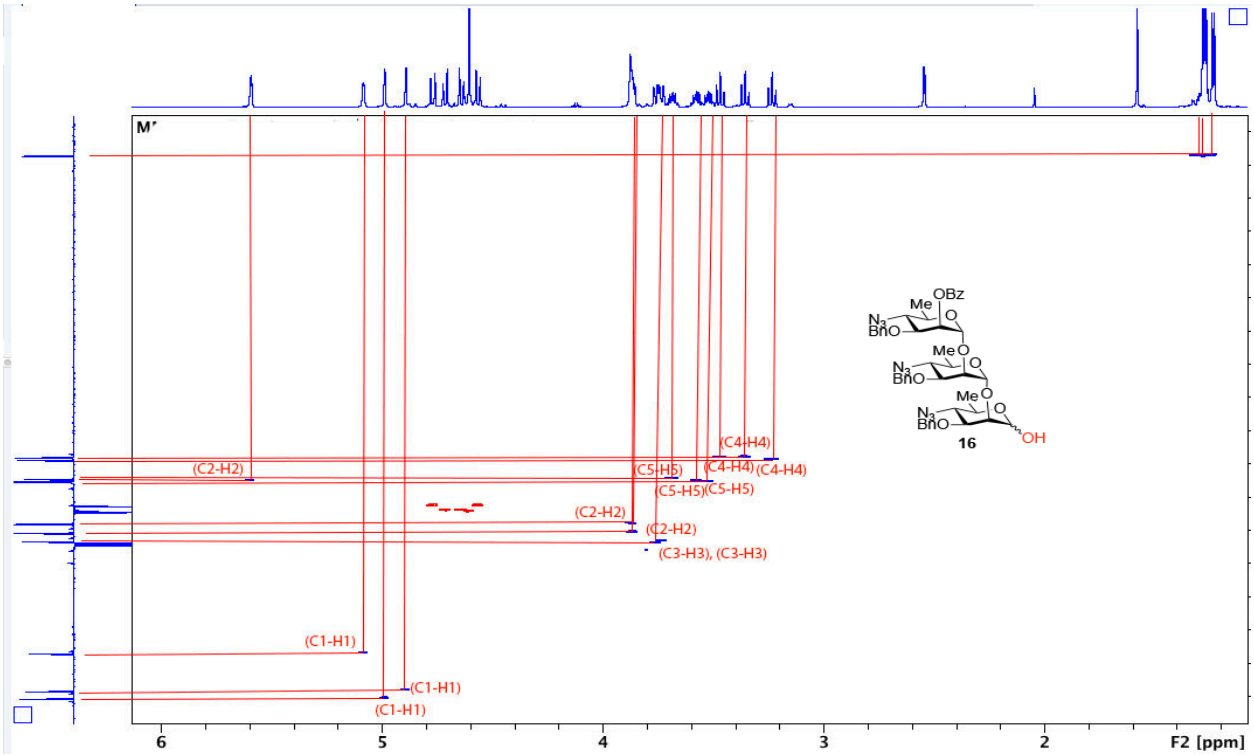

Fig. S40: HSQC NMR spectra of compound **16** ($CDCl_3$).

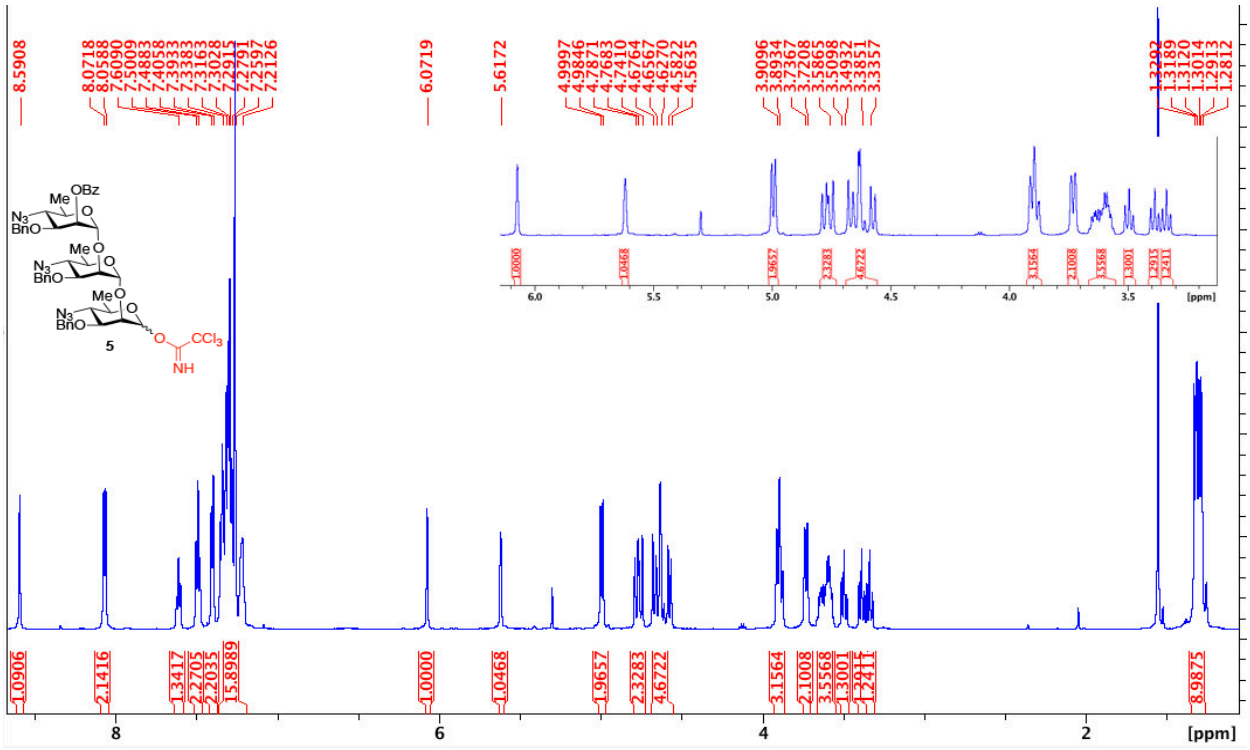

Fig. S41: ^{1}H NMR spectra of compound **5** ($CDCl_3$, 600 MHz).

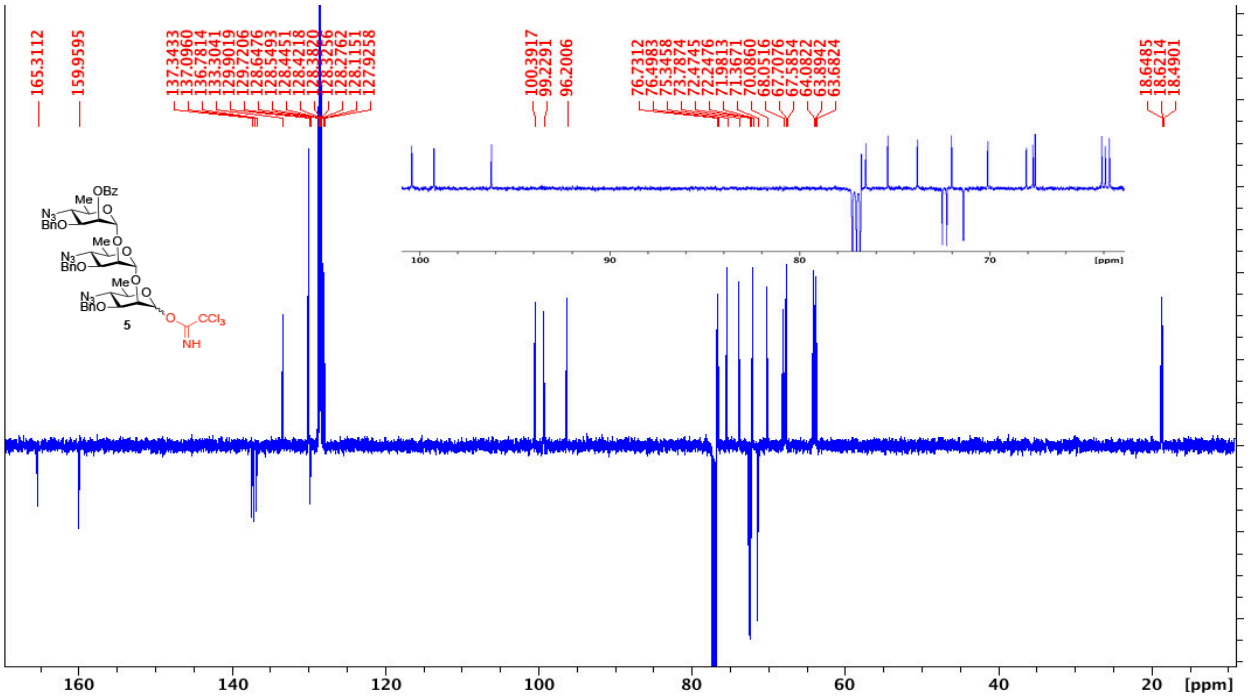

Fig. S42: ^{13}C{1H} NMR spectra of compound **5** ($CDCl_3$, 150 MHz).

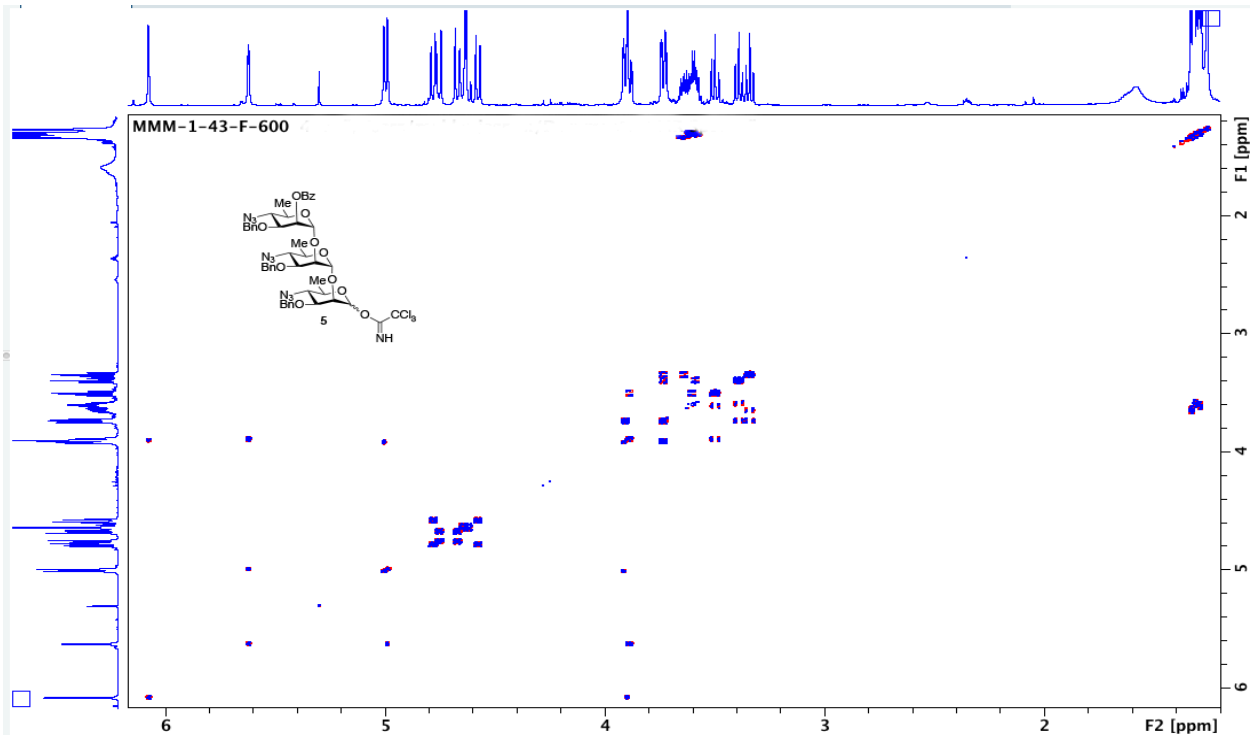

Fig. S43: COSY NMR spectra of compound **5** ($CDCl_3$, 600 MHz).

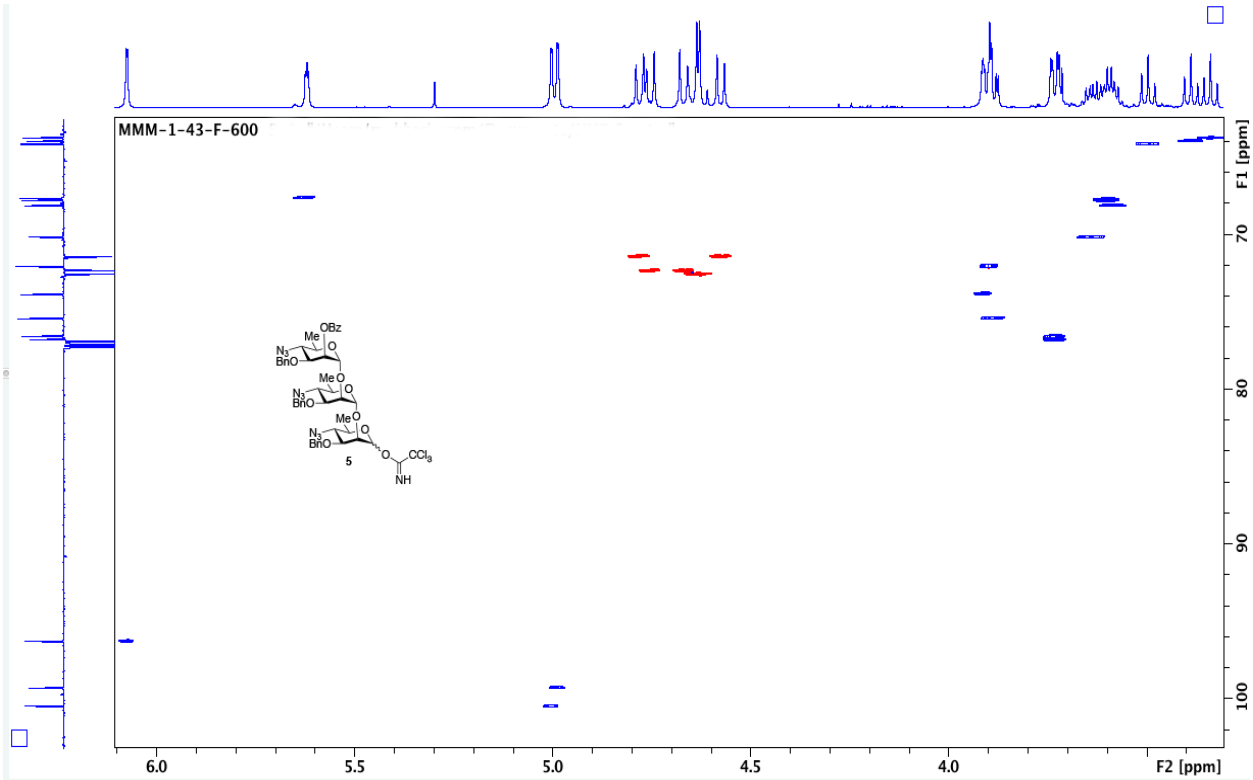

Fig. S44: HSQC NMR spectra of compound **5** ($CDCl_3$).

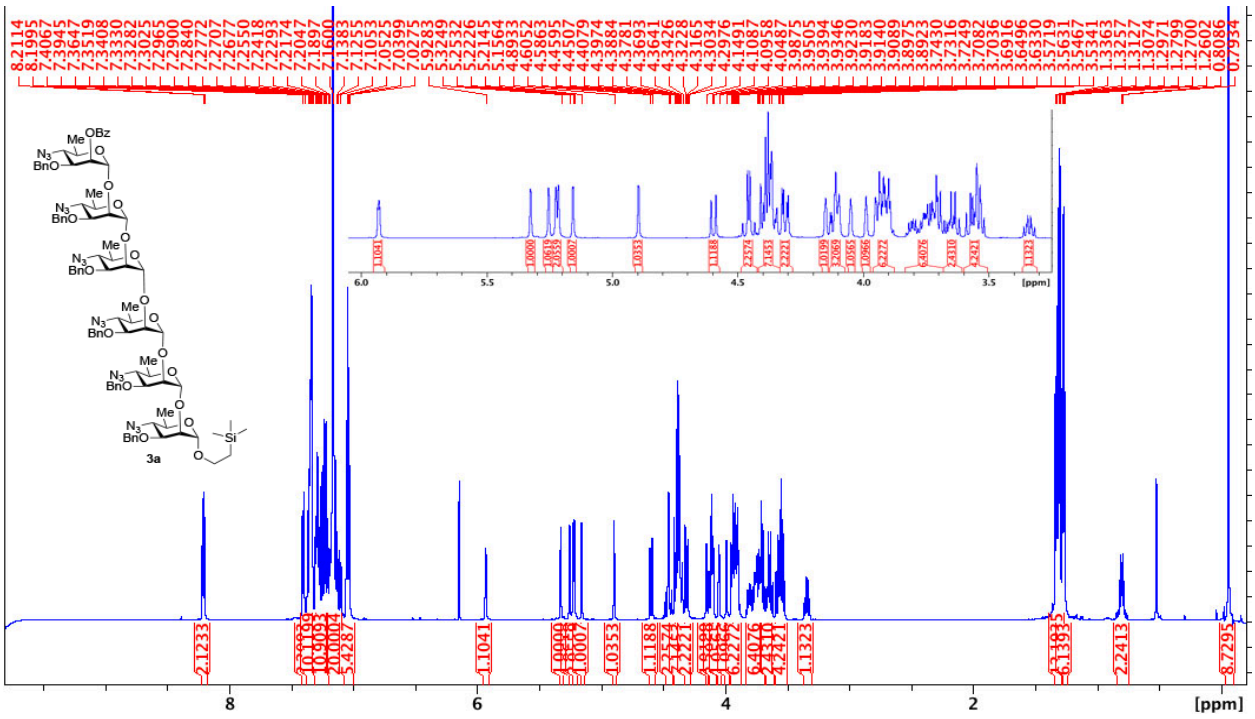

Fig. S45: ^{1}H NMR spectra of compound **3a** (C_6D_6, 600 MHz).

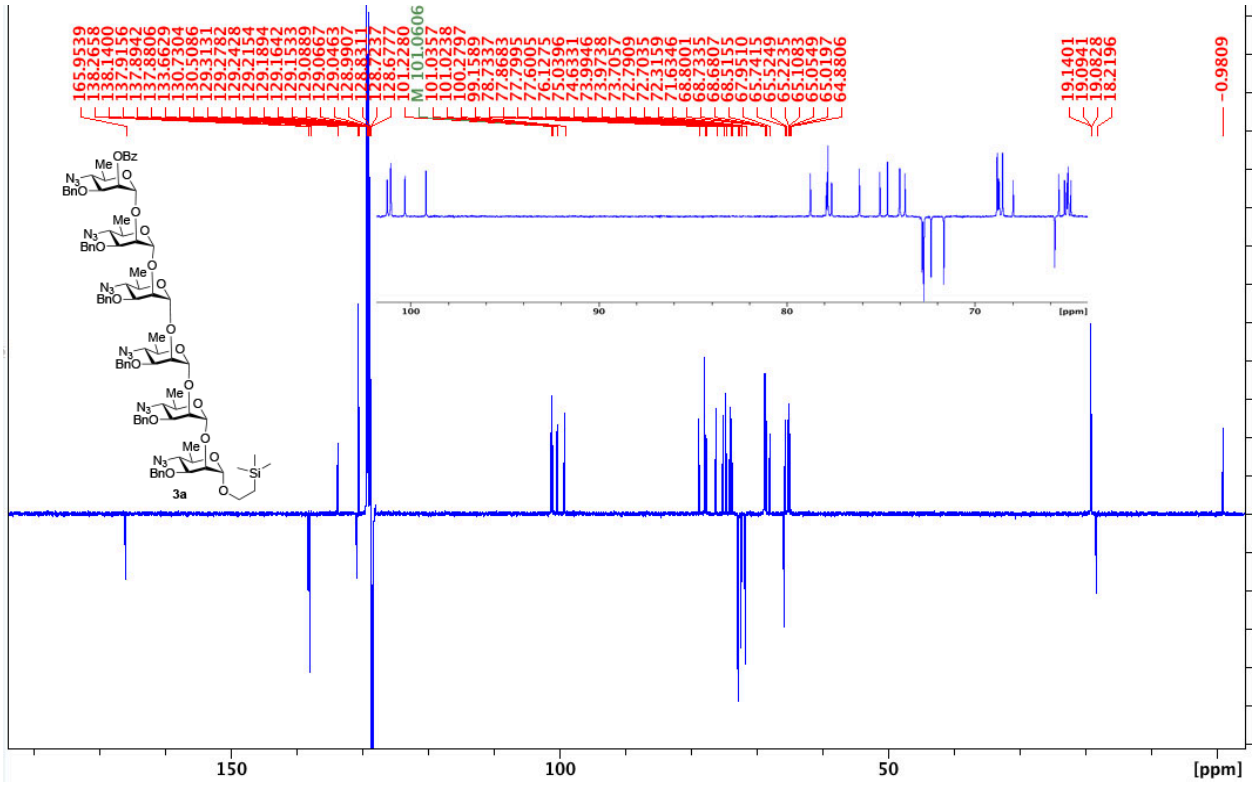

Fig. S46: ^{13}C{1H} NMR spectra of compound **3a** (C_6D_6, 150 MHz).

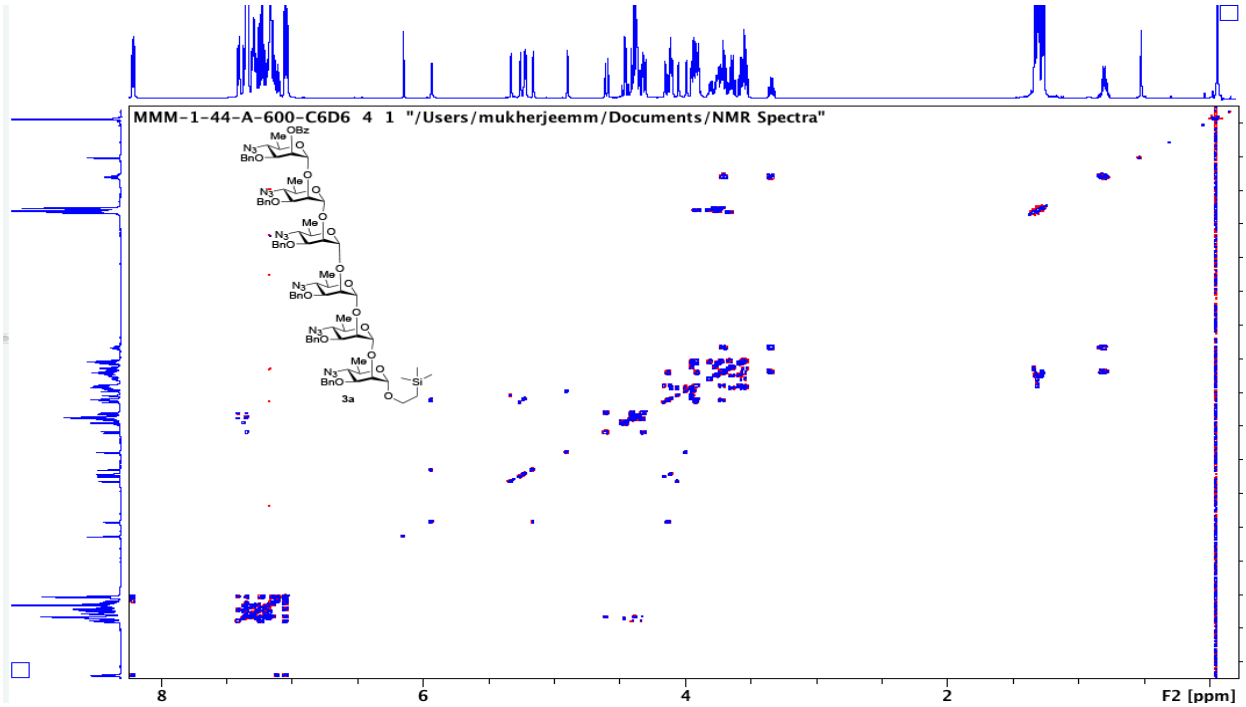

Fig. S47: COSY NMR spectra of compound **3a** (C_6D_6, 600 MHz).

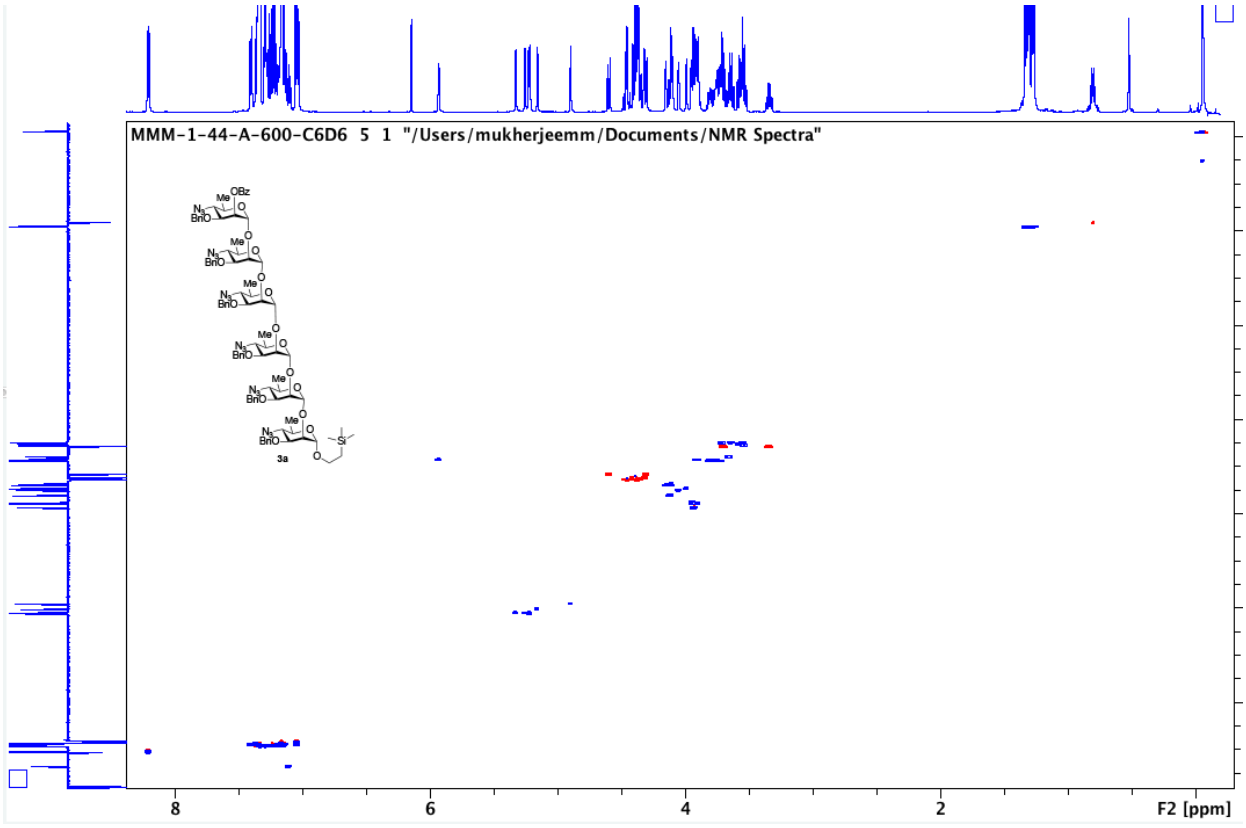

Fig. S48: HSQC NMR spectra of compound **3a** (C_6D_6).

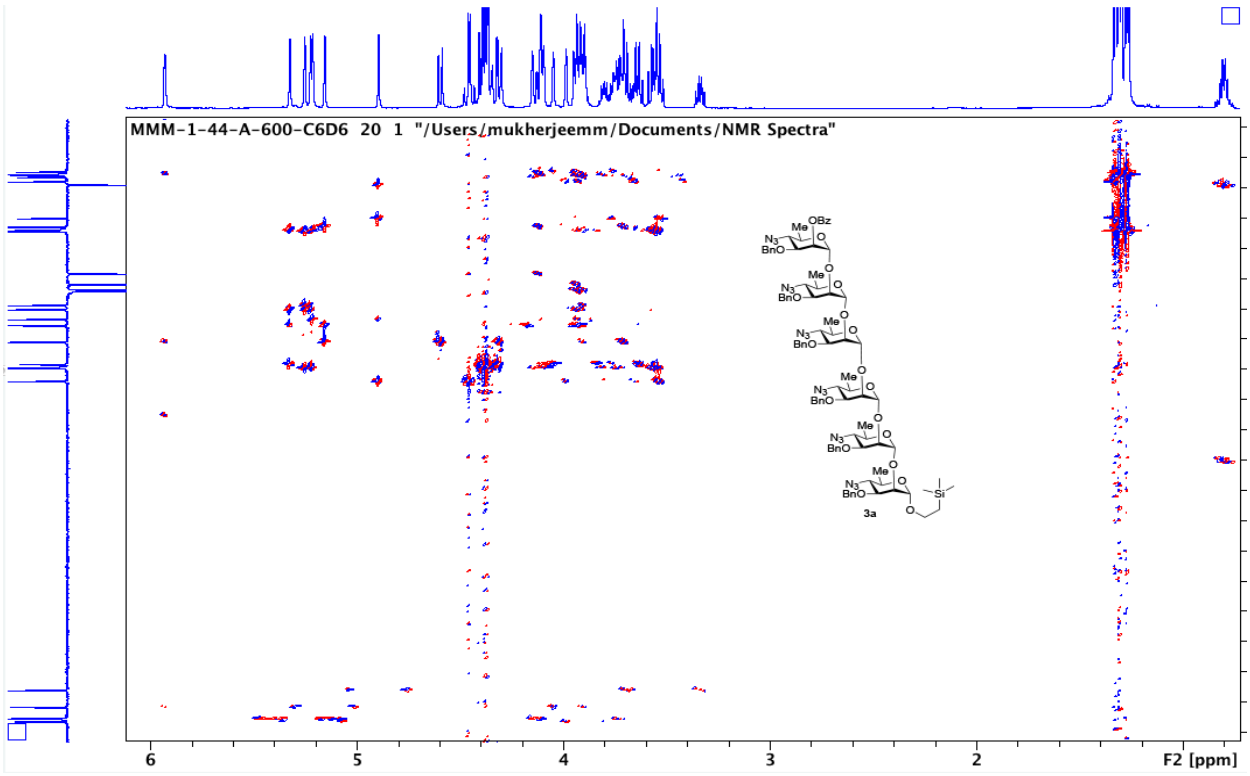

Fig. S49: HMBC NMR spectra of compound **3a** (C_6D_6).

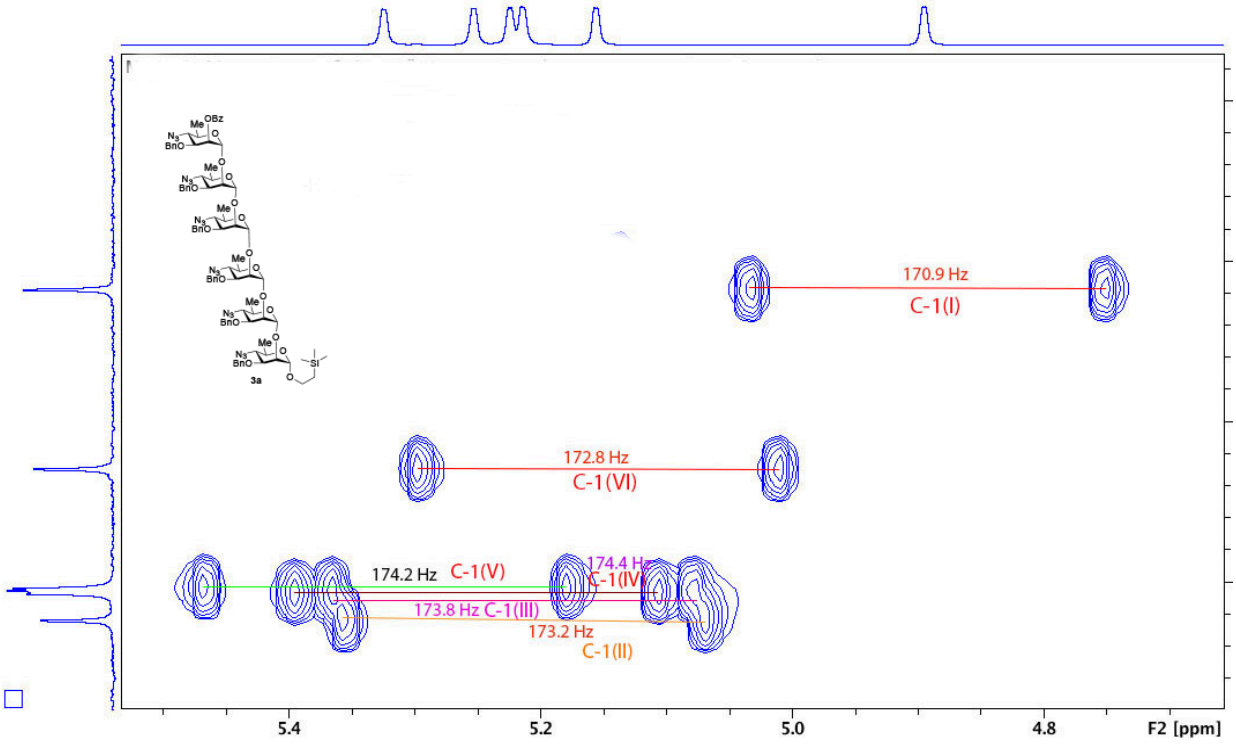

Fig. S50: ^{1}H-^{13}C Coupled NMR spectra of compound **3a** (C_6D_6).

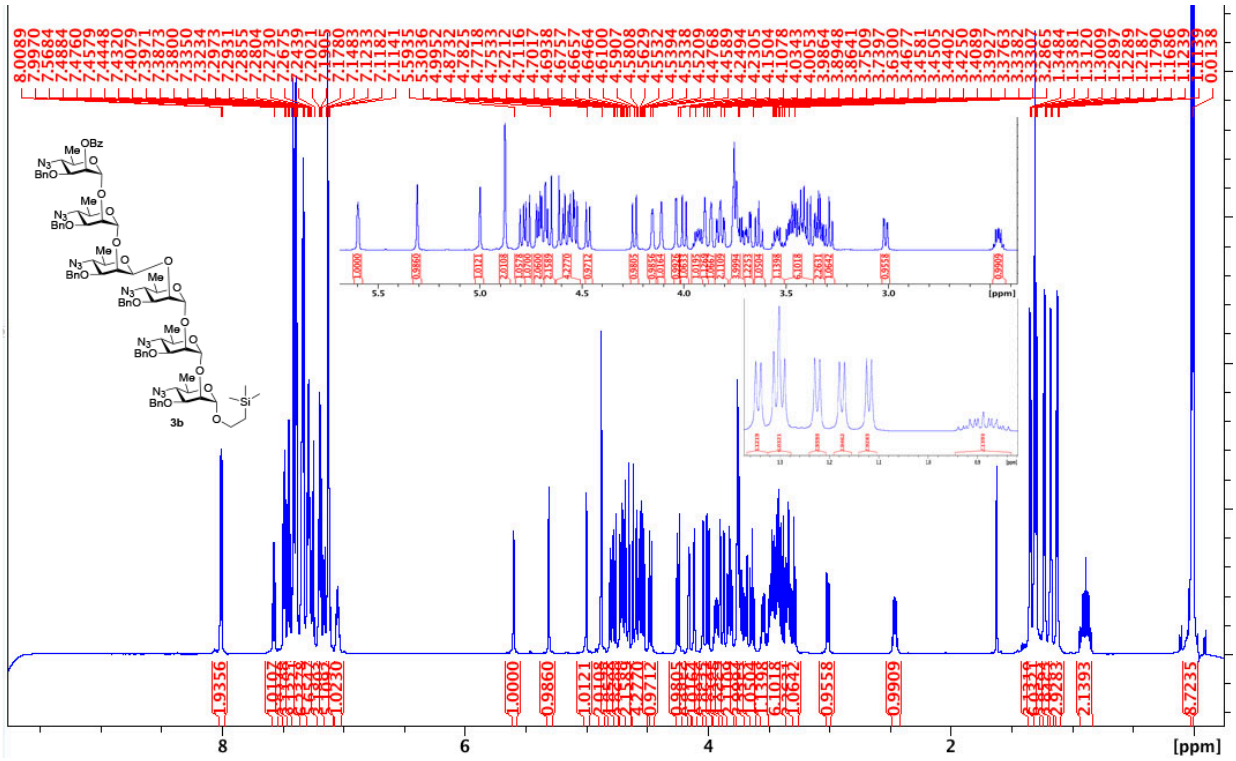

Fig. S51: ^{1}H NMR spectra of compound **3b** ($CDCl_3$, 600 MHz).

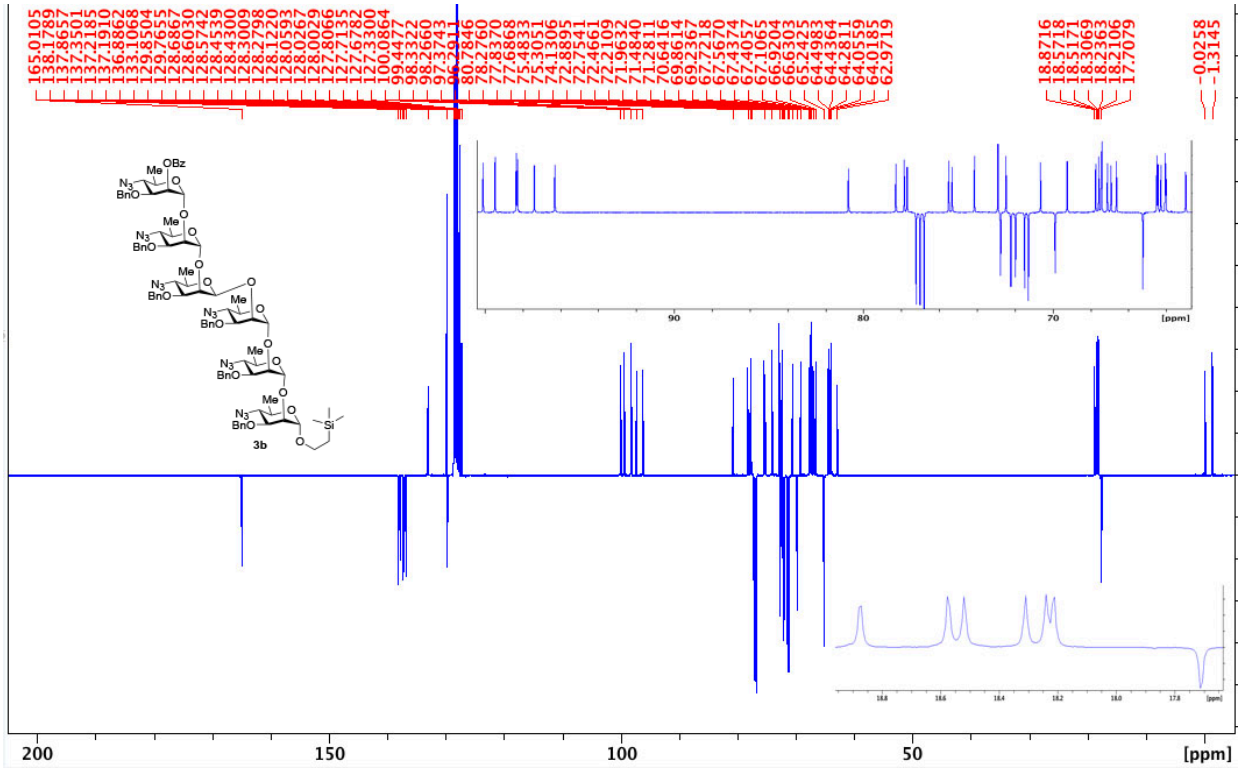

Fig. S52: ^{13}C{1H} NMR spectra of compound **3b** ($CDCl_3$, 150 MHz).

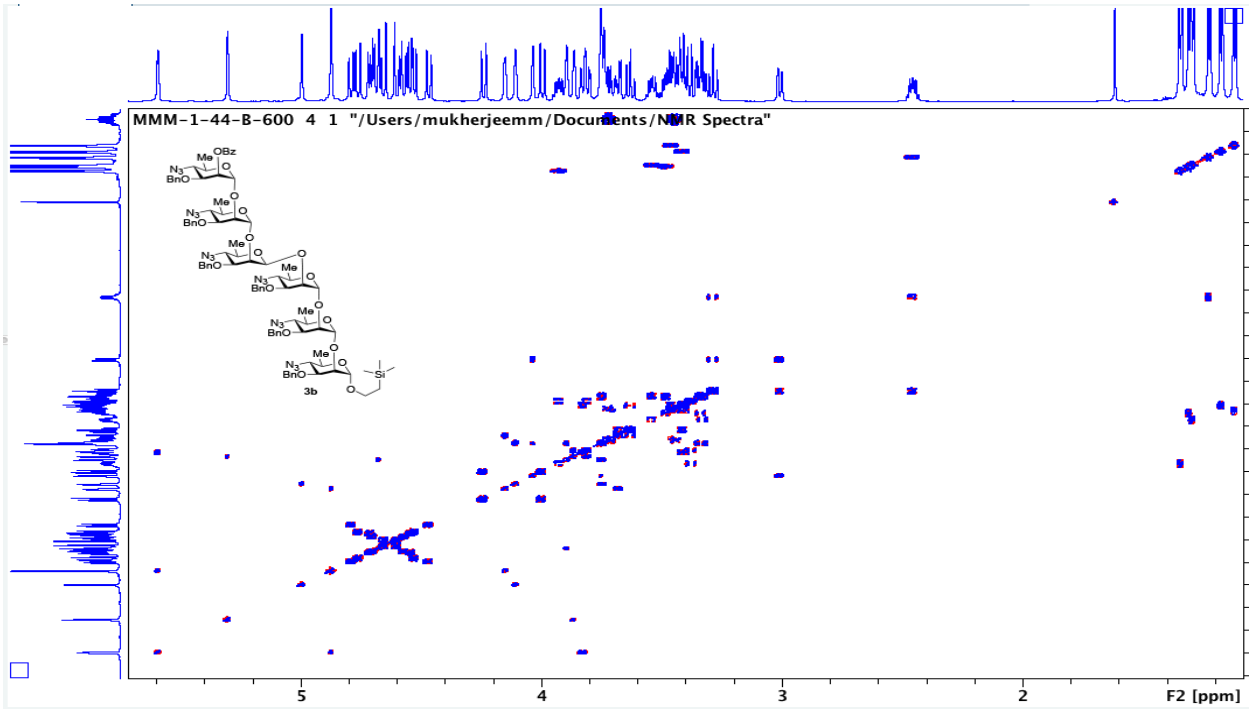

Fig. S53: COSY NMR spectra of compound **3b** ($CDCl_3$, 600 MHz).

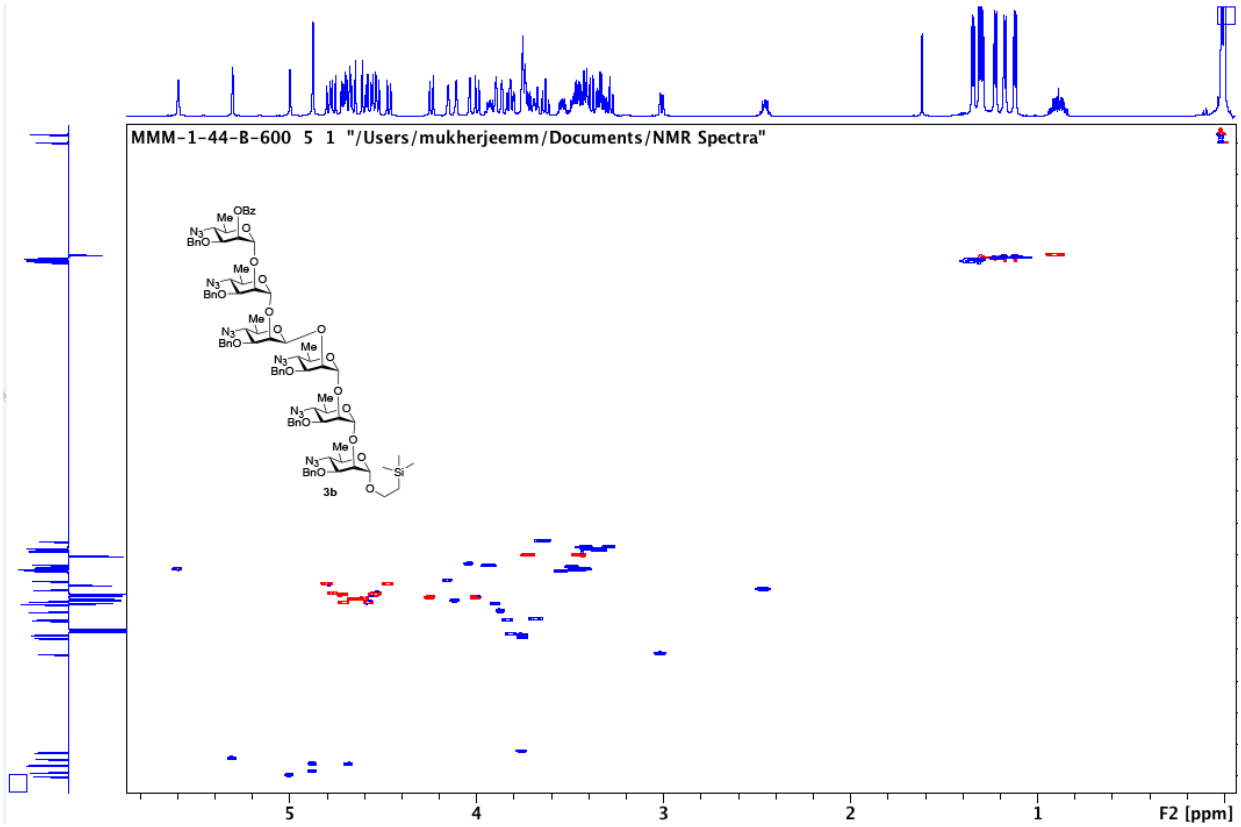

Fig. S54: HSQC NMR spectra of compound **3b** ($CDCl_3$).

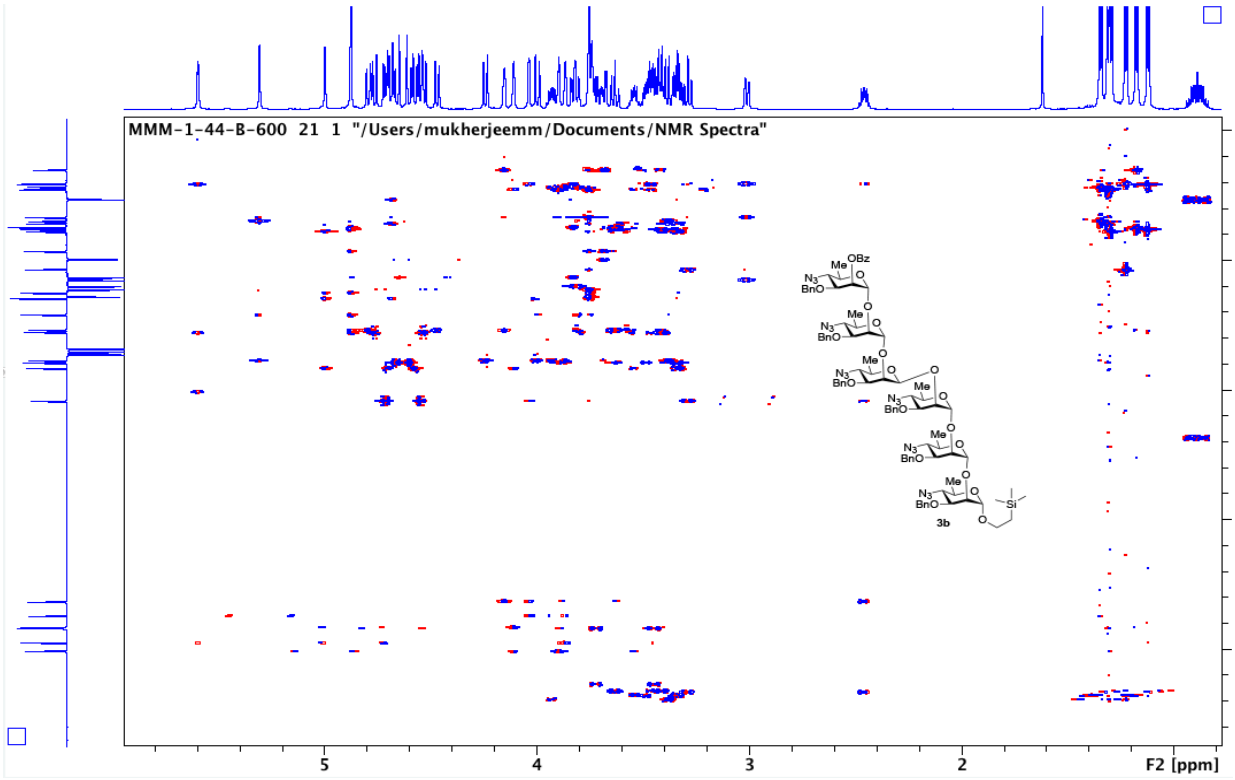

Fig. S55: HMBC NMR spectra of compound **3b** ($CDCl_3$).

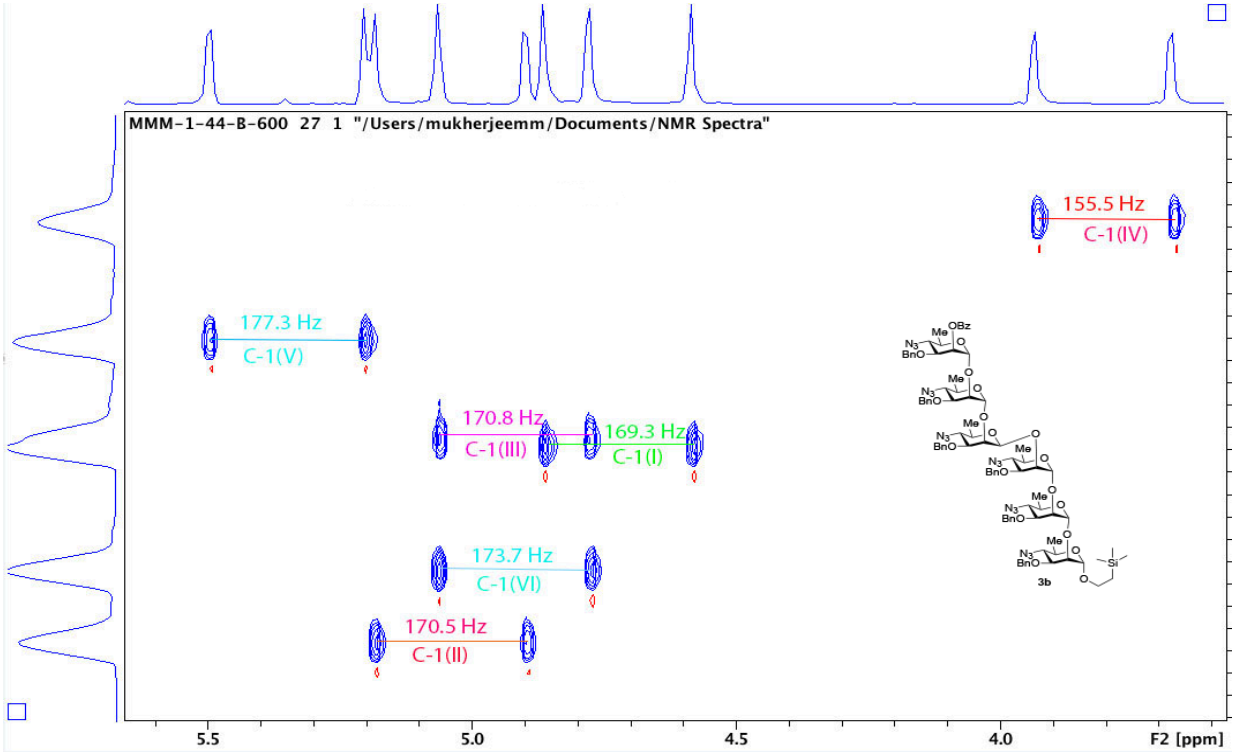

Fig. S56: ^{1}H-^{13}C Coupled NMR spectra of compound **3b** ($CDCl_3$).

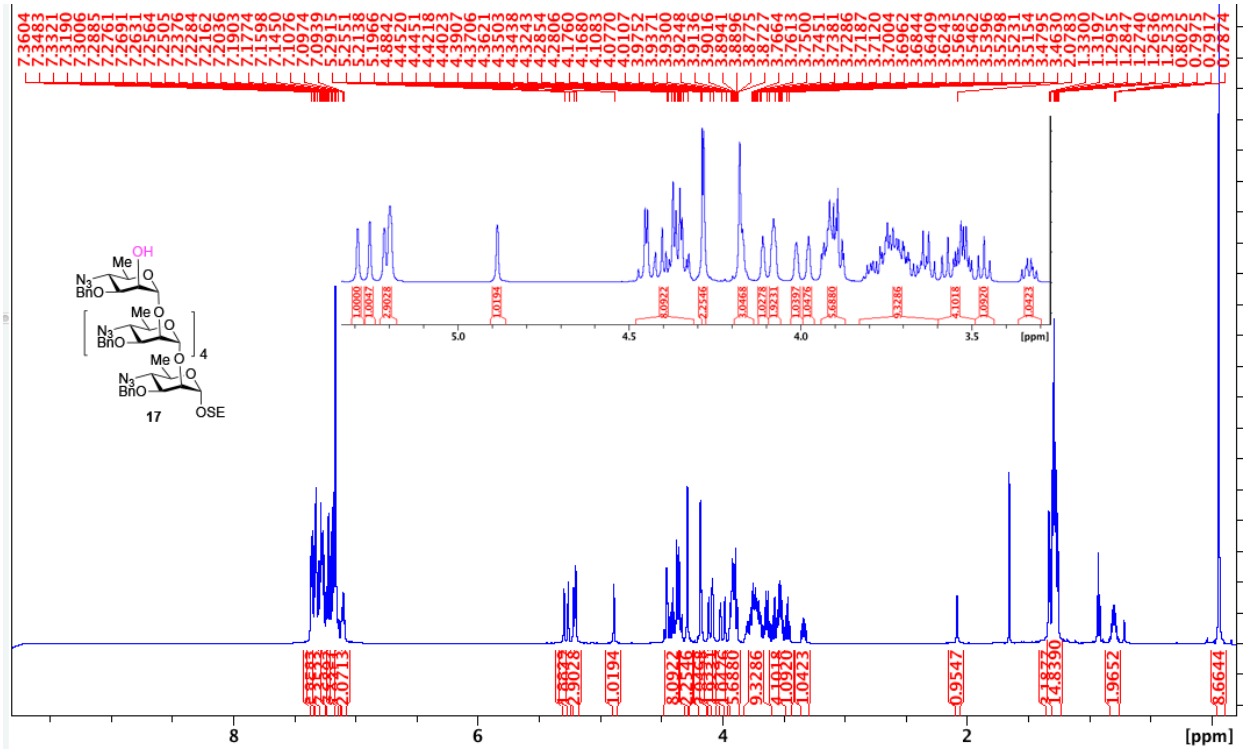

Fig. S57: ^{1}H NMR spectra of compound **17** (C_6D_6, 600 MHz).

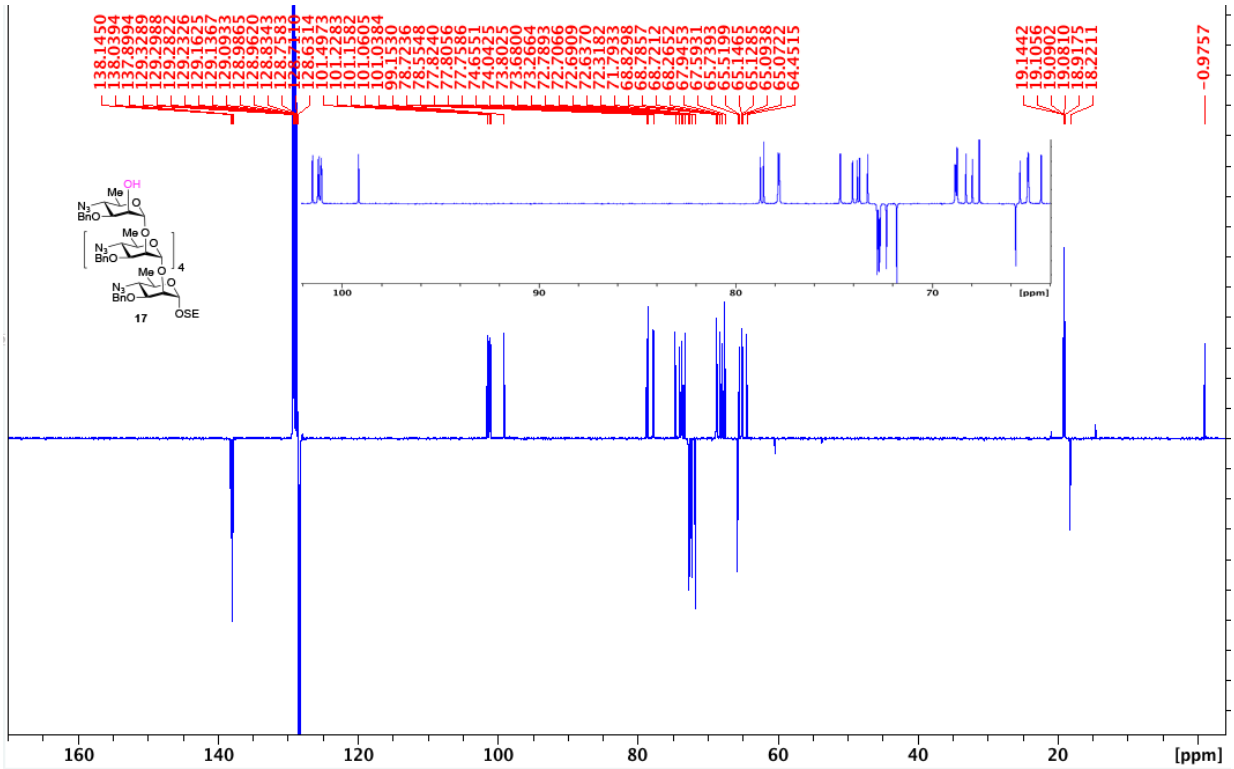

Fig. S58: ^{13}C{1H} NMR spectra of compound **17** (C_6D_6, 150 MHz).

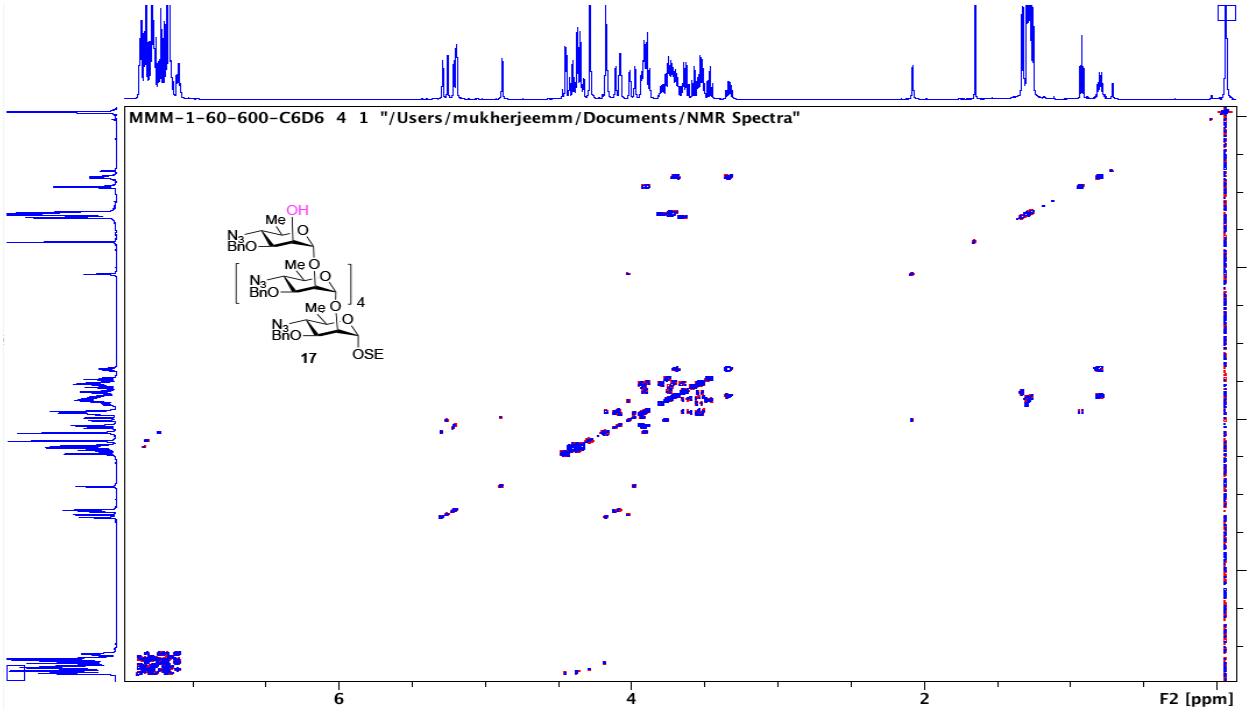

Fig. S59: COSY NMR spectra of compound **17** (C_6D_6, 600 MHz).

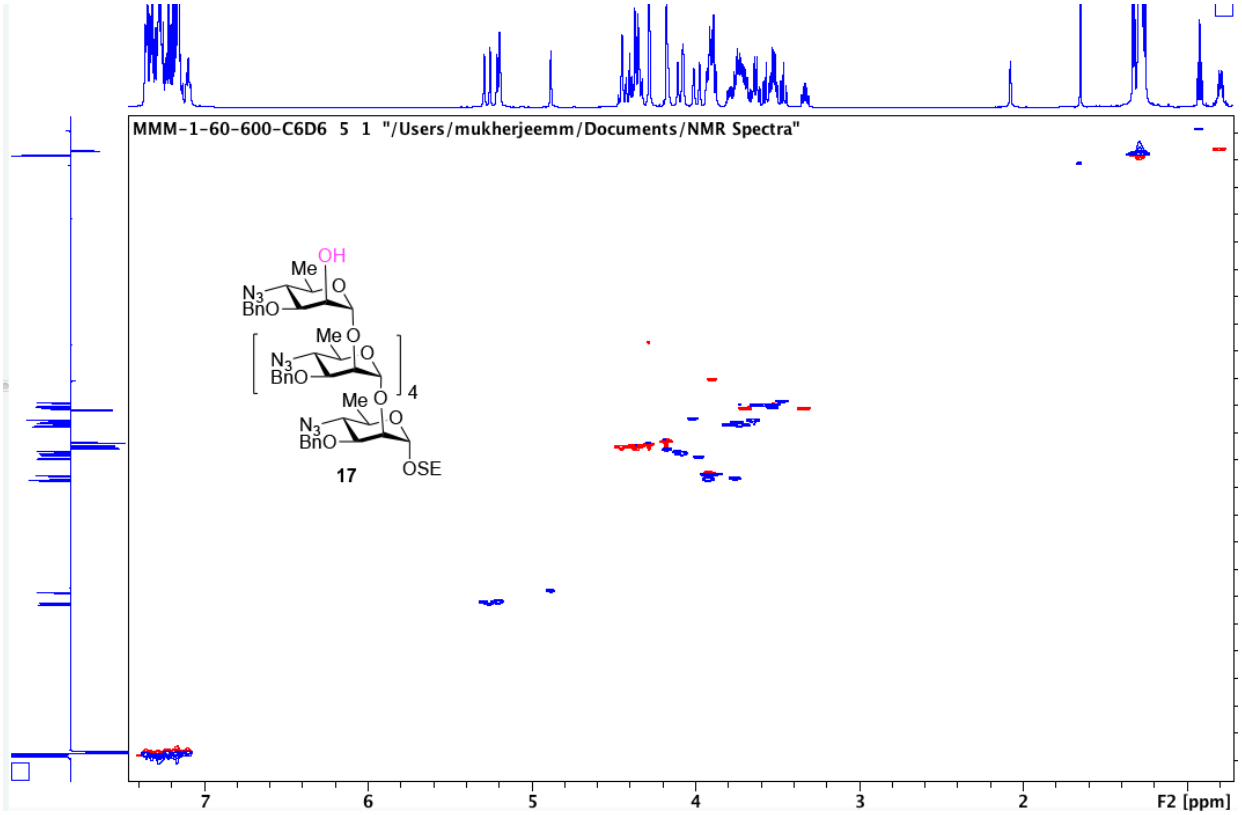

Fig. S60: HSQC NMR spectra of compound **17** (C_6D_6).

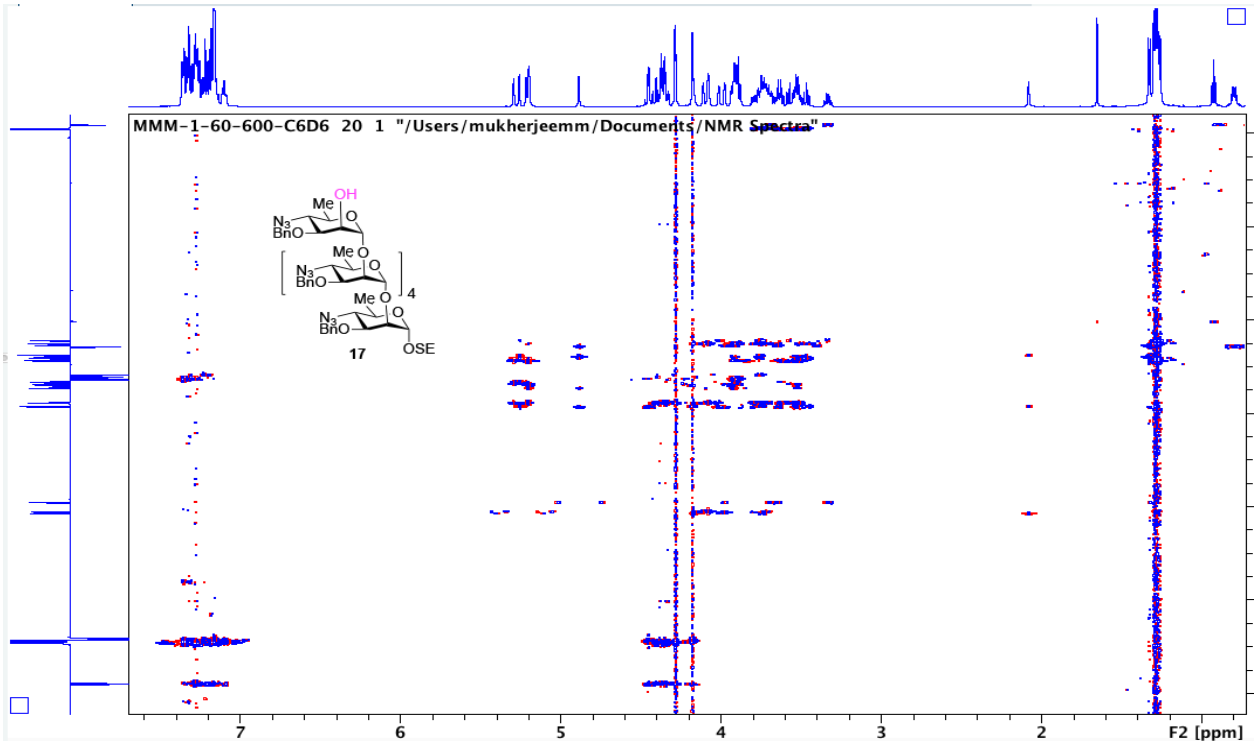

Fig. S61: HMBC NMR spectra of compound **17** (C_6D_6).

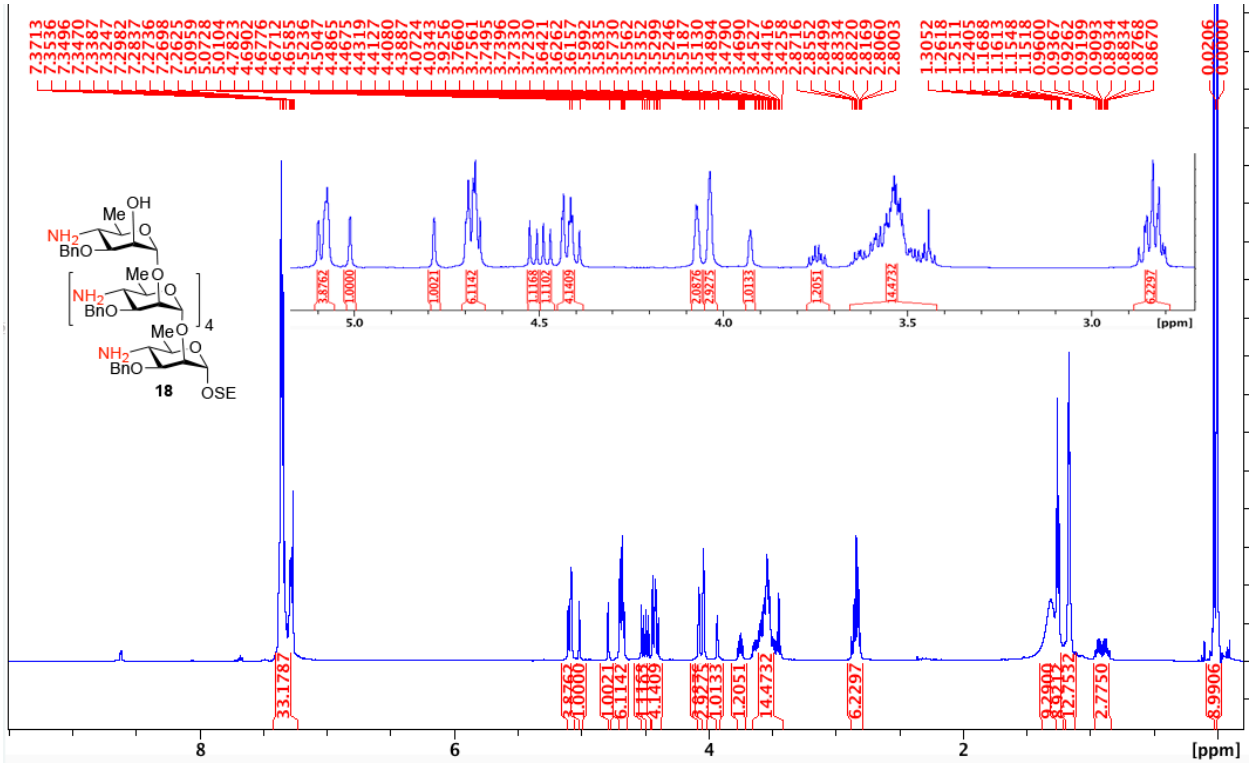

Fig. S62: ^{1}H NMR spectra of compound **18** ($CDCl_3$, 600 MHz).

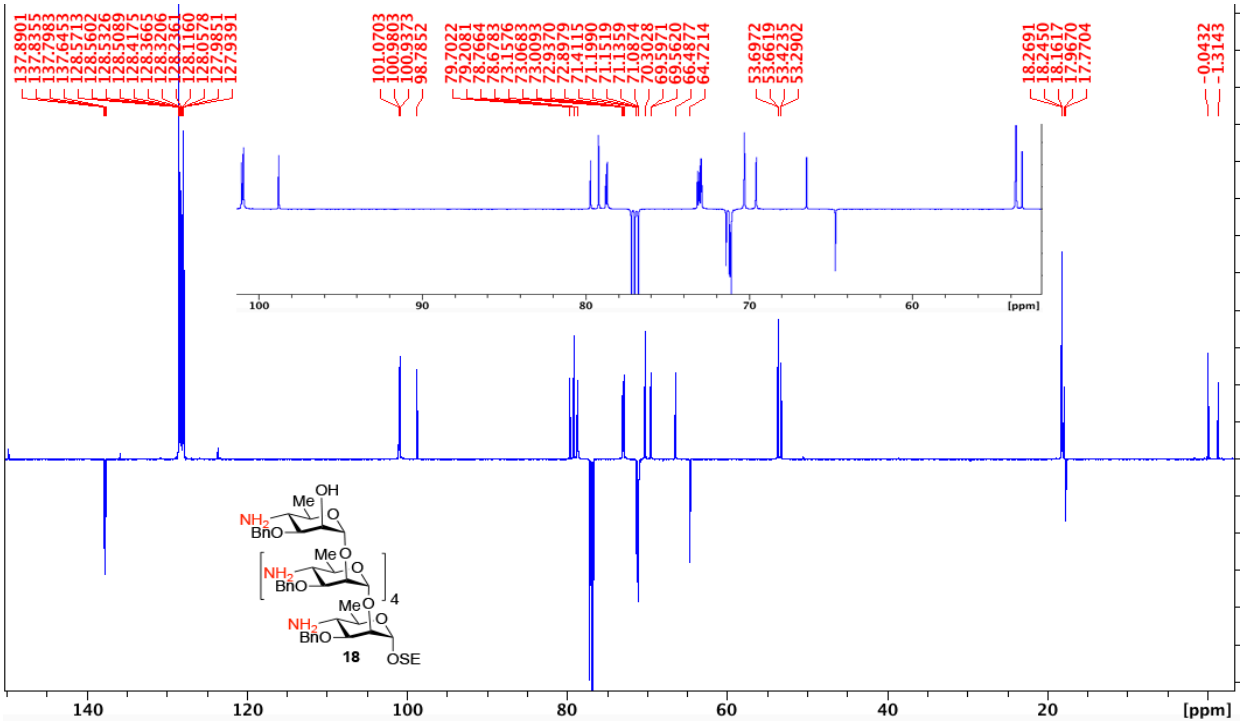

Fig. S63: $^{13}C\{1H\}$ NMR spectra of compound **18** ($CDCl_3$, 150 MHz).

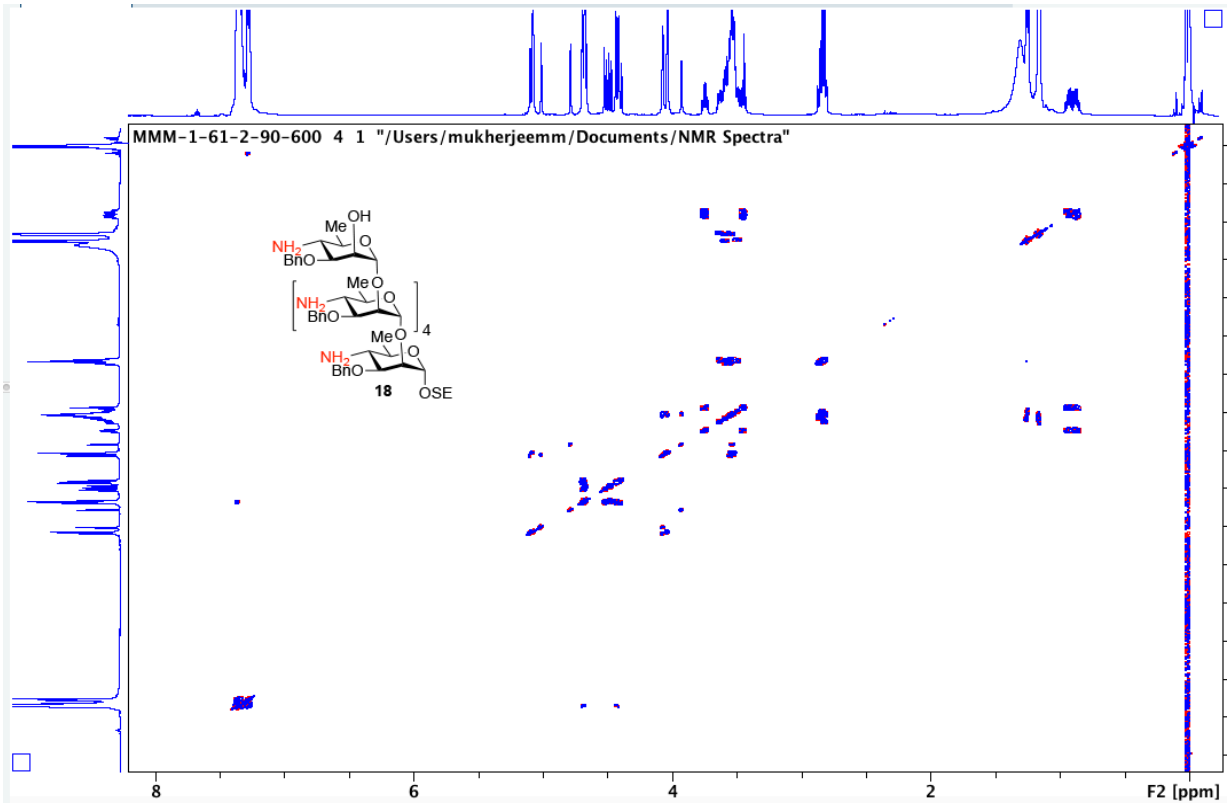

Fig. S64: COSY NMR spectra of compound **18** ($CDCl_3$, 600 MHz).

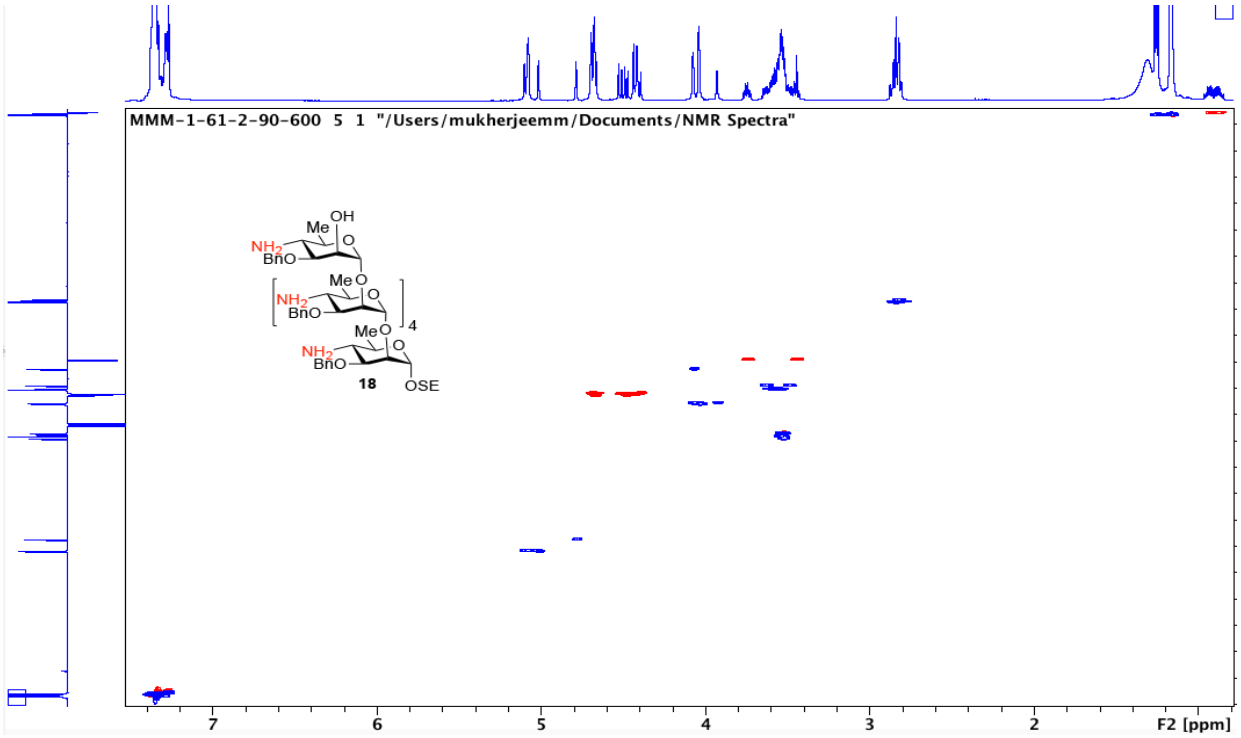

Fig. S65: HSQC NMR spectra of compound **18** ($CDCl_3$).

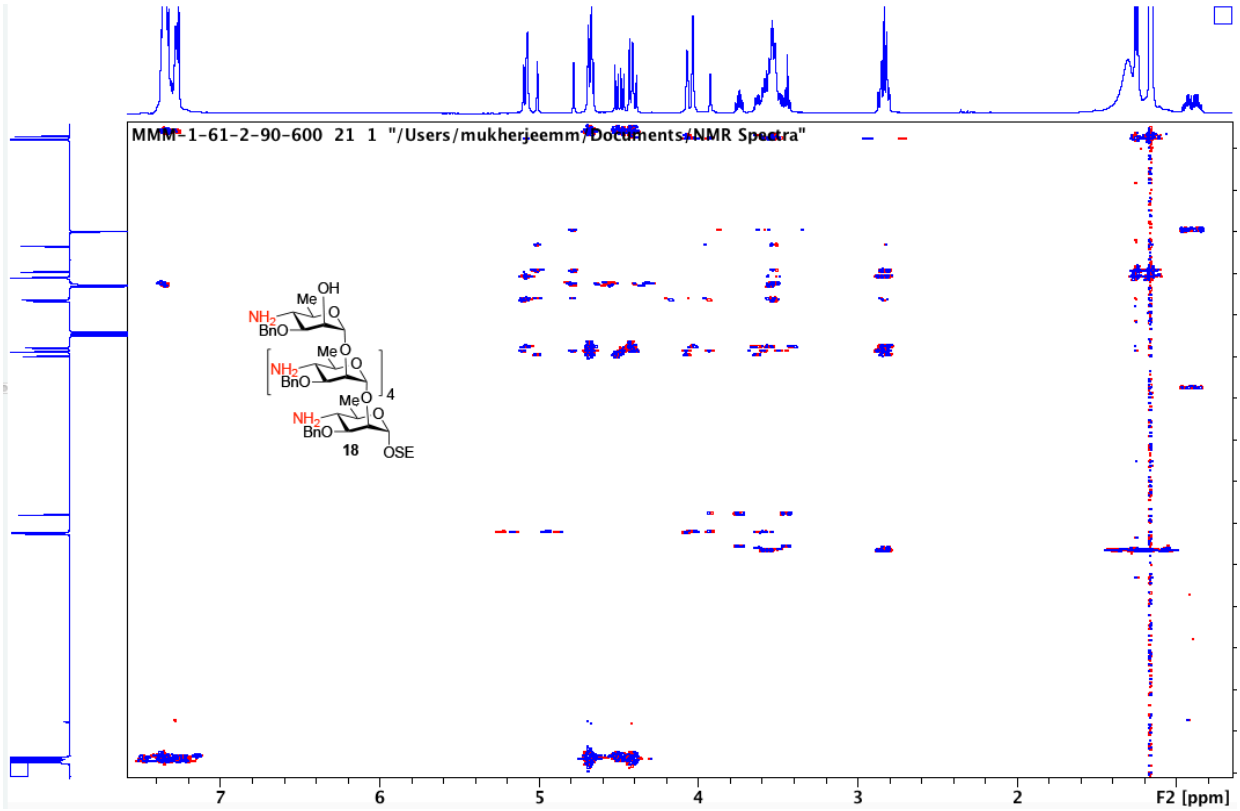

Fig. S66: HMBC NMR spectra of compound **18** ($CDCl_3$).

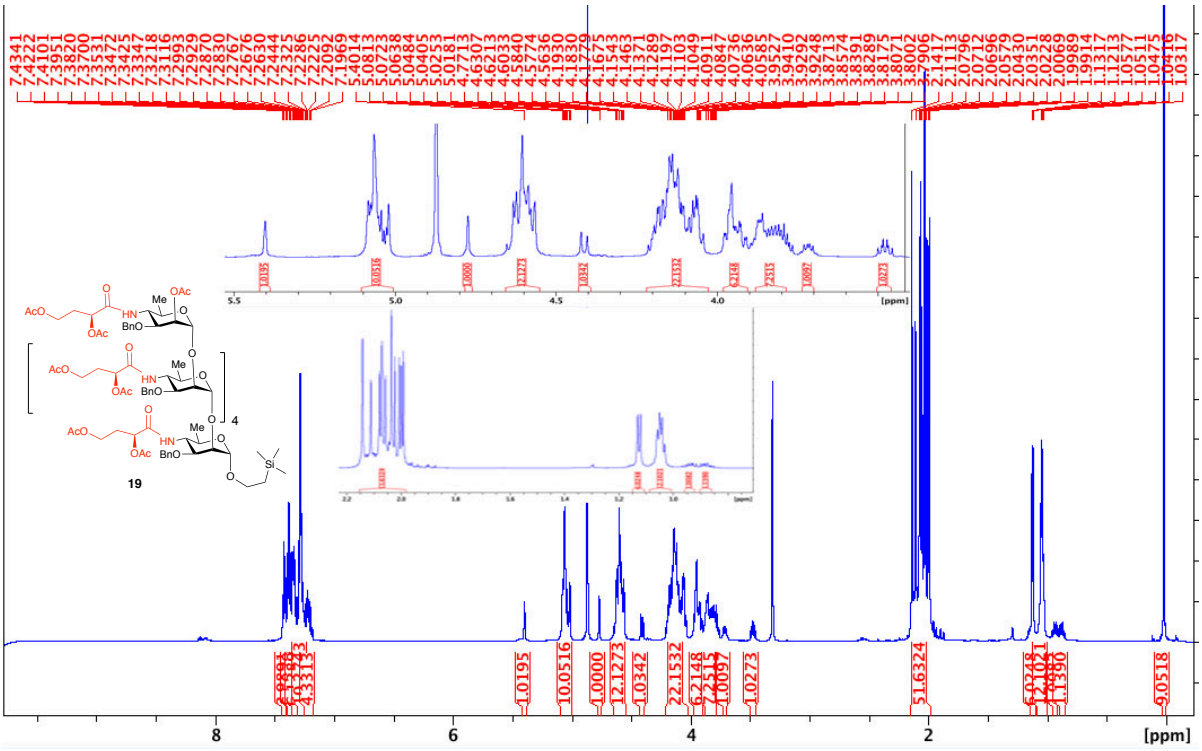

Fig. S67: ^{1}H NMR spectra of compound **19** (CD_3OD, 600 MHz).

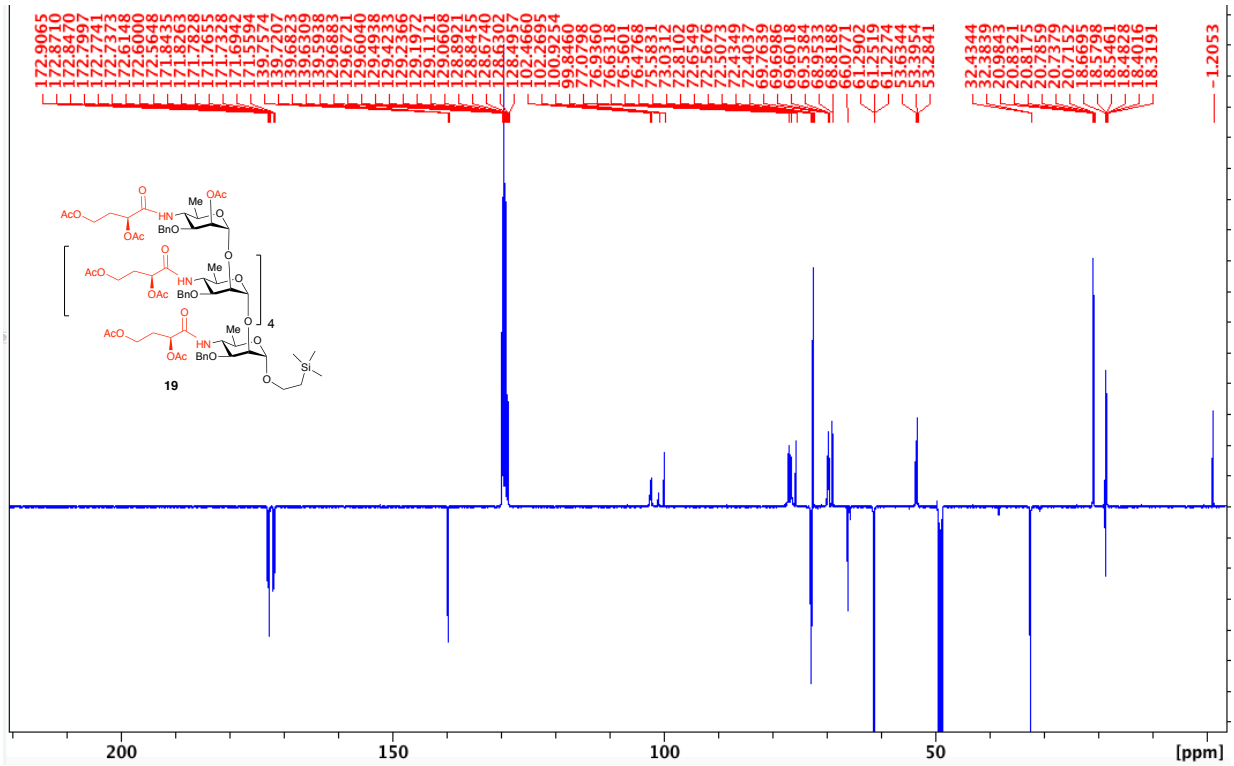

Fig. S68: ^{13}C{1H} NMR spectra of compound **19** (CD_3OD, 150 MHz).

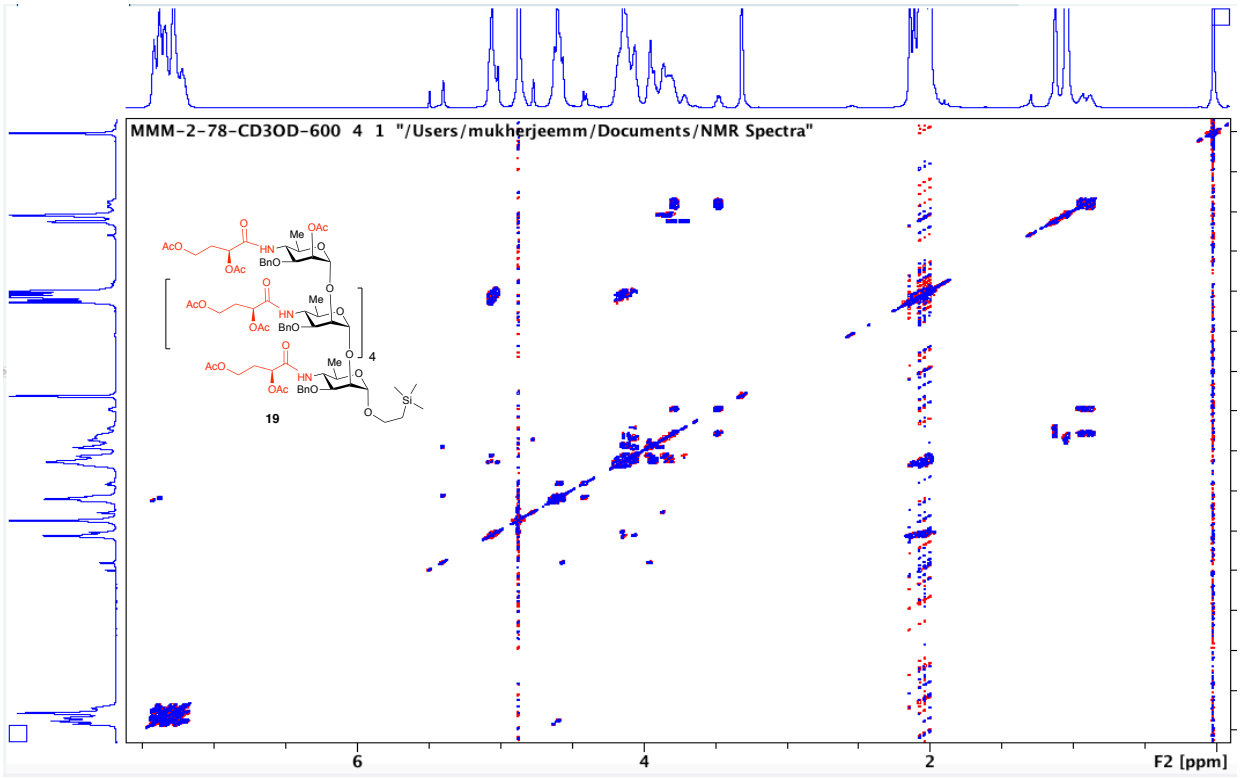

Fig. S69: COSY NMR spectra of compound **19** (CD_3OD, 600 MHz).

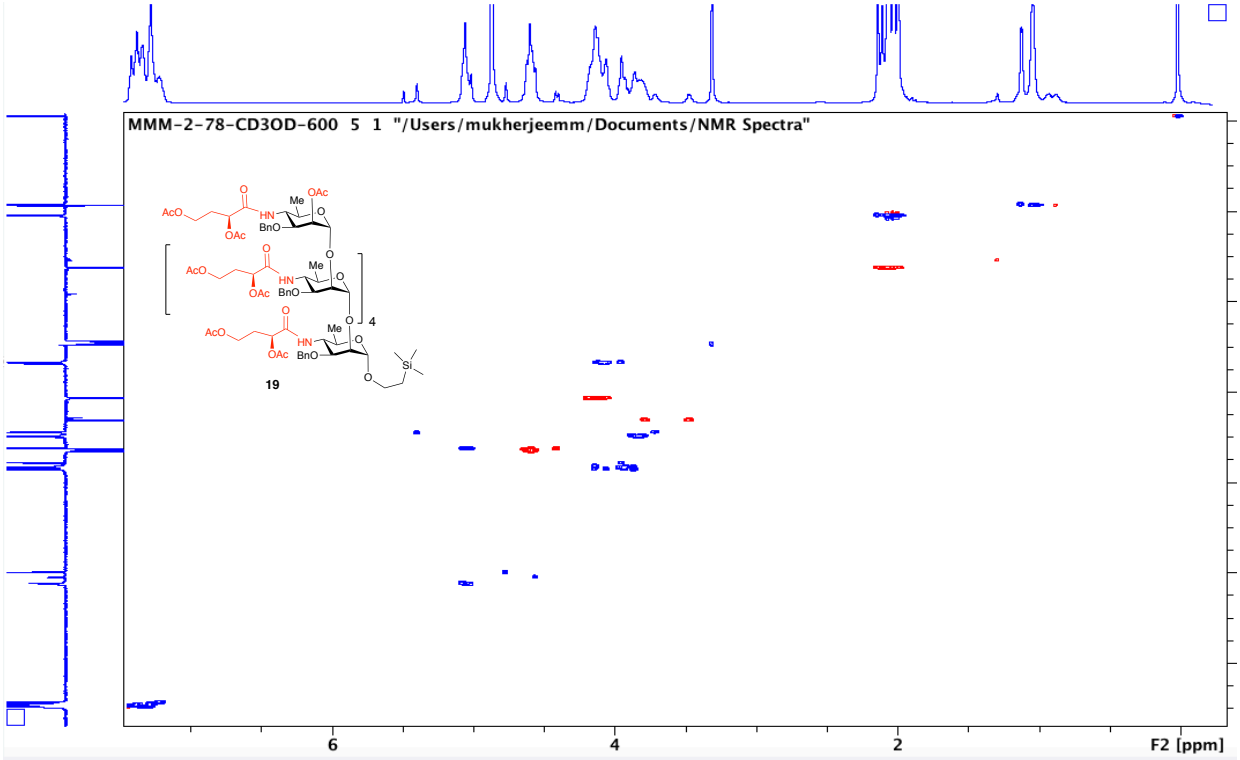

Fig. S70: HSQC NMR spectra of compound **19** (CD_3OD).

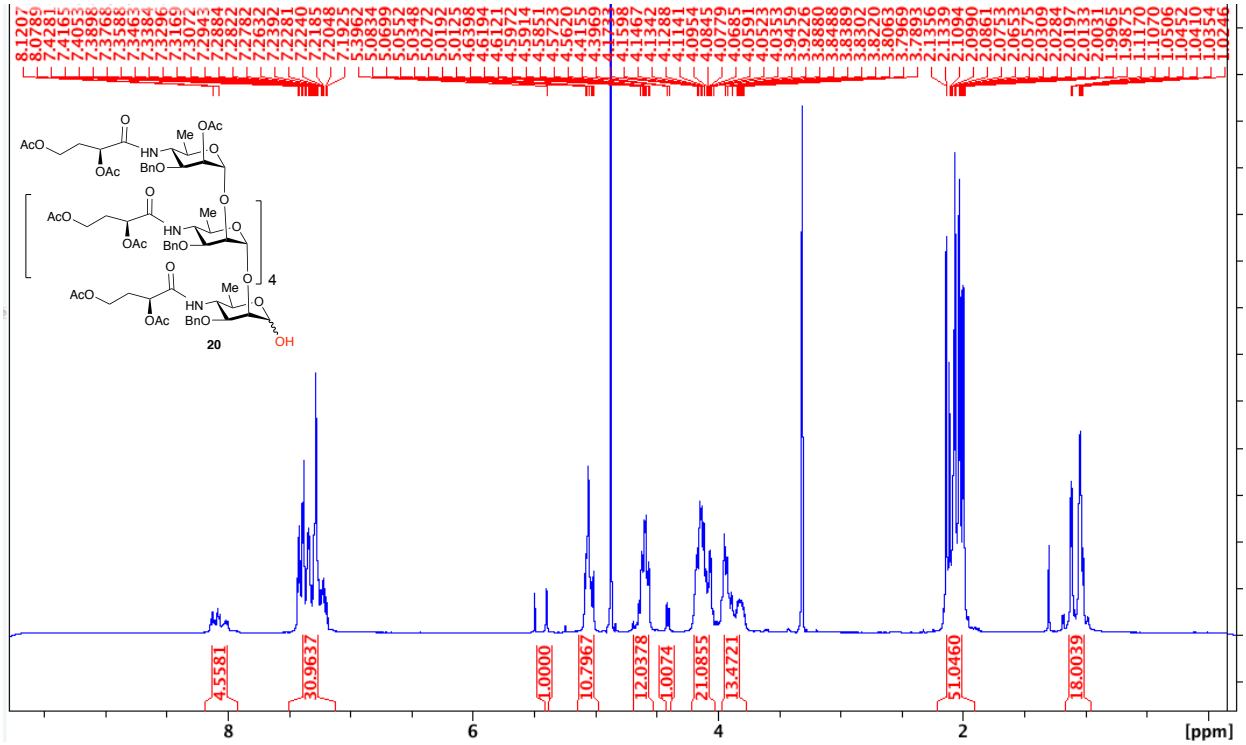

Fig. S71: ^{1}H NMR spectra of compound **20** (CD_3OD, 600 MHz).

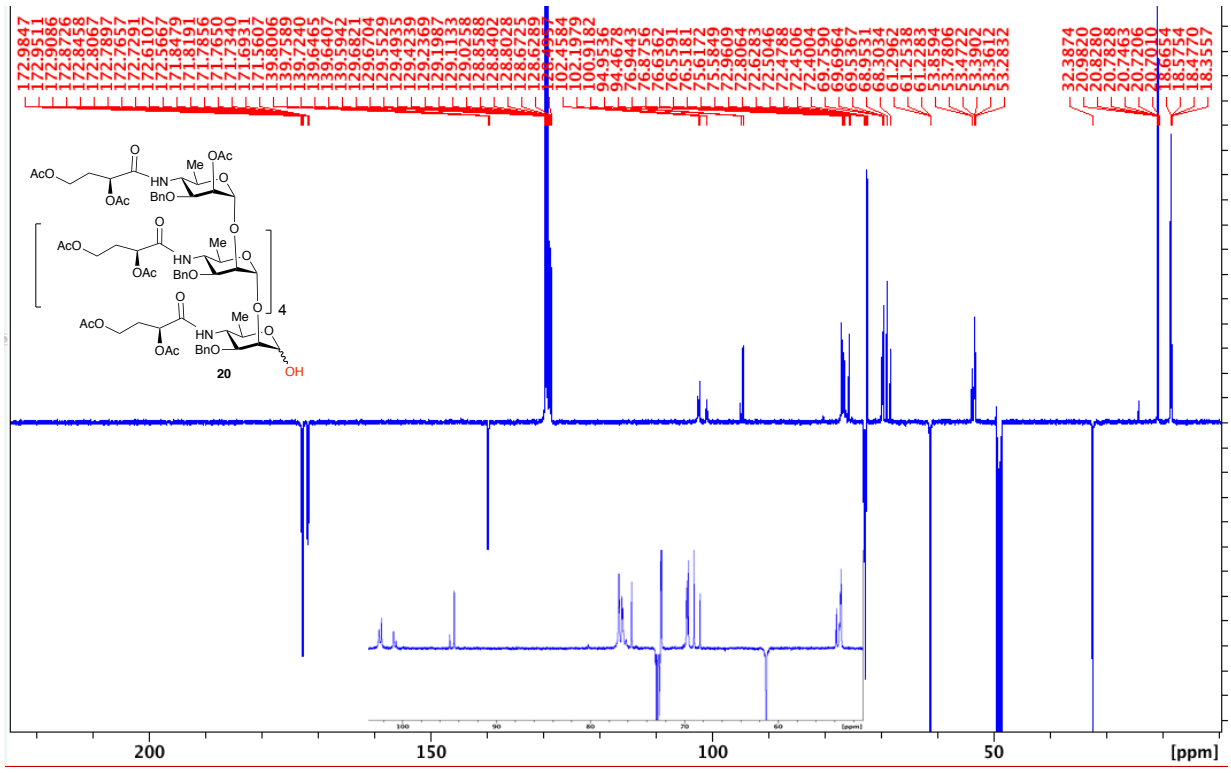

Fig. S72: ^{13}C{1H} NMR spectra of compound **20** (CD_3OD, 150 MHz).

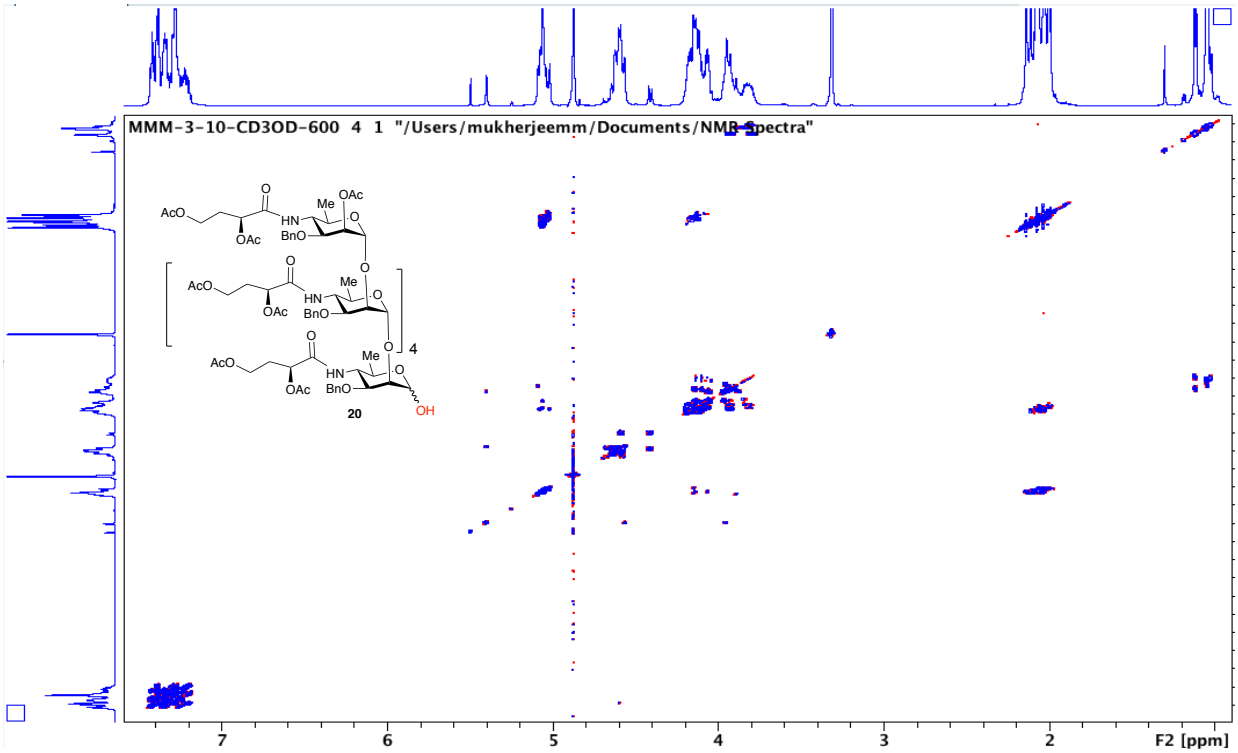

Fig. S73: COSY NMR spectra of compound **20** (CD_3OD, 600 MHz).

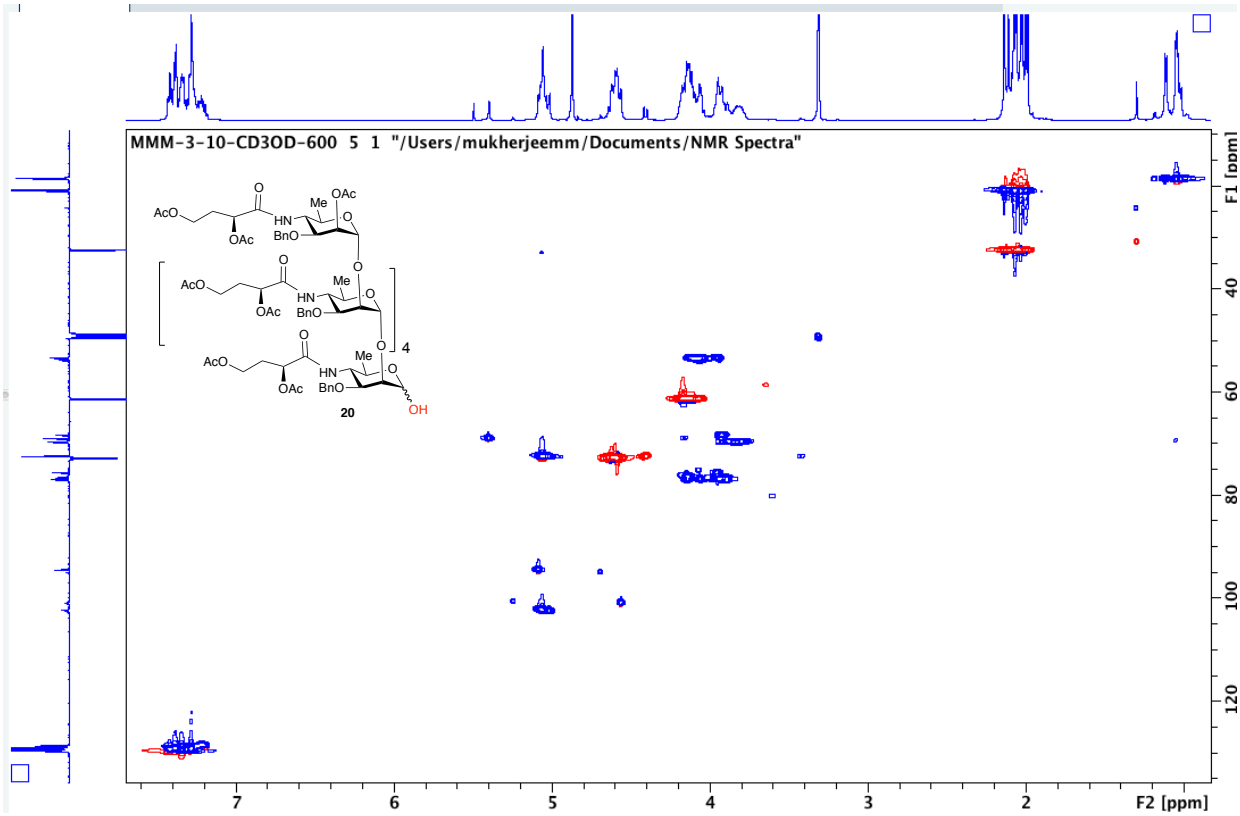

Fig. S74: HSQC NMR spectra of compound **20** (CD_3OD).

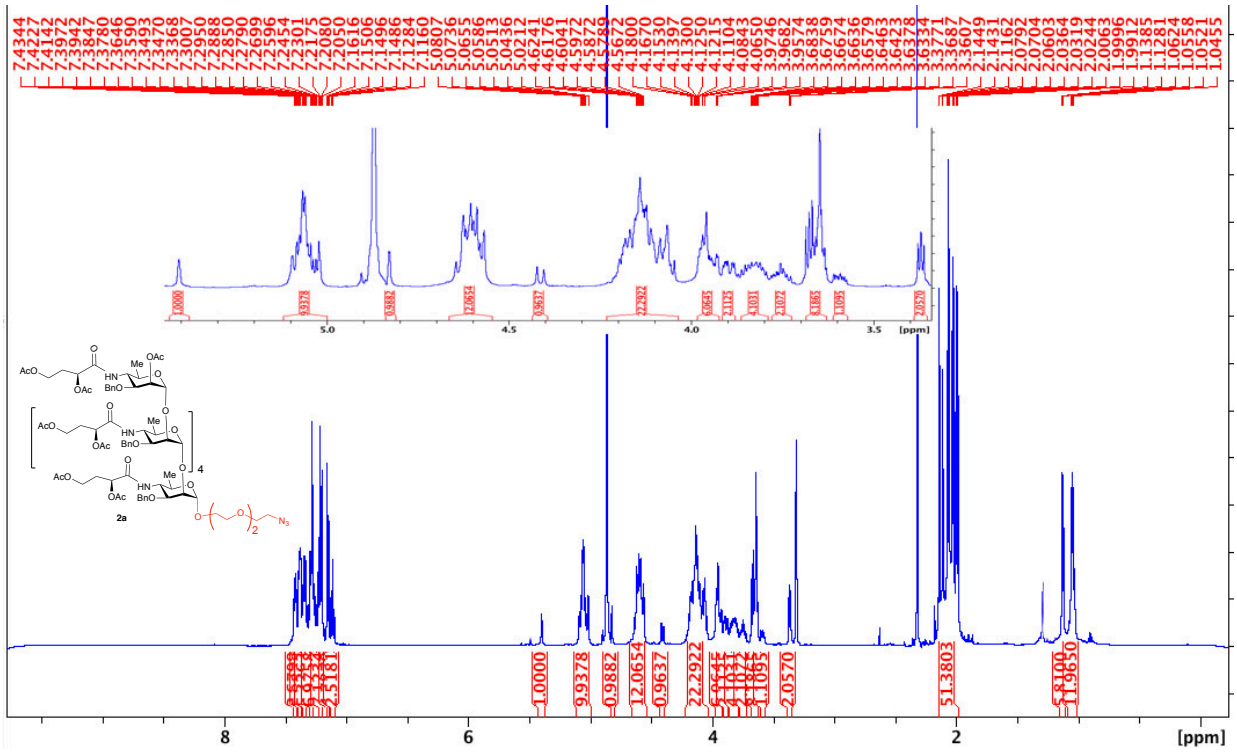

Fig. S75: ^{1}H NMR spectra of compound **2a** (CD_3OD, 600 MHz).

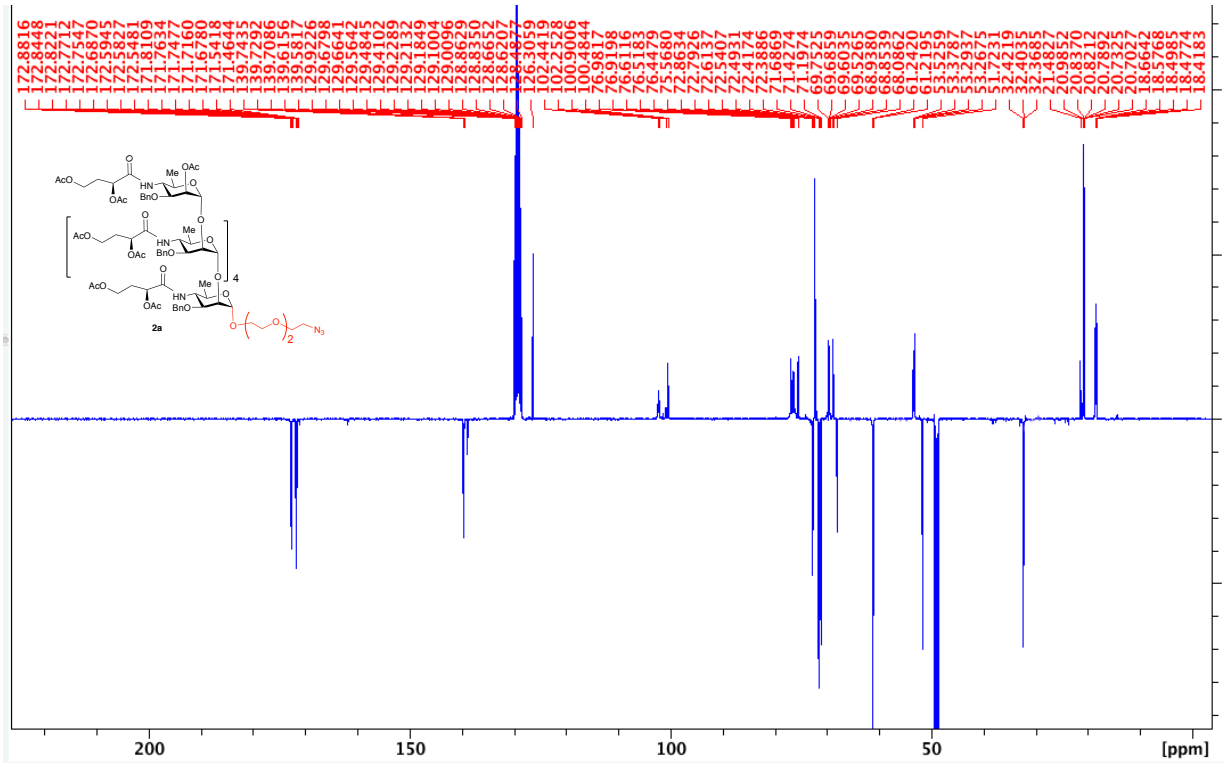

Fig. S76: ^{13}C{1H} NMR spectra of compound **2a** (CD_3OD, 150 MHz).

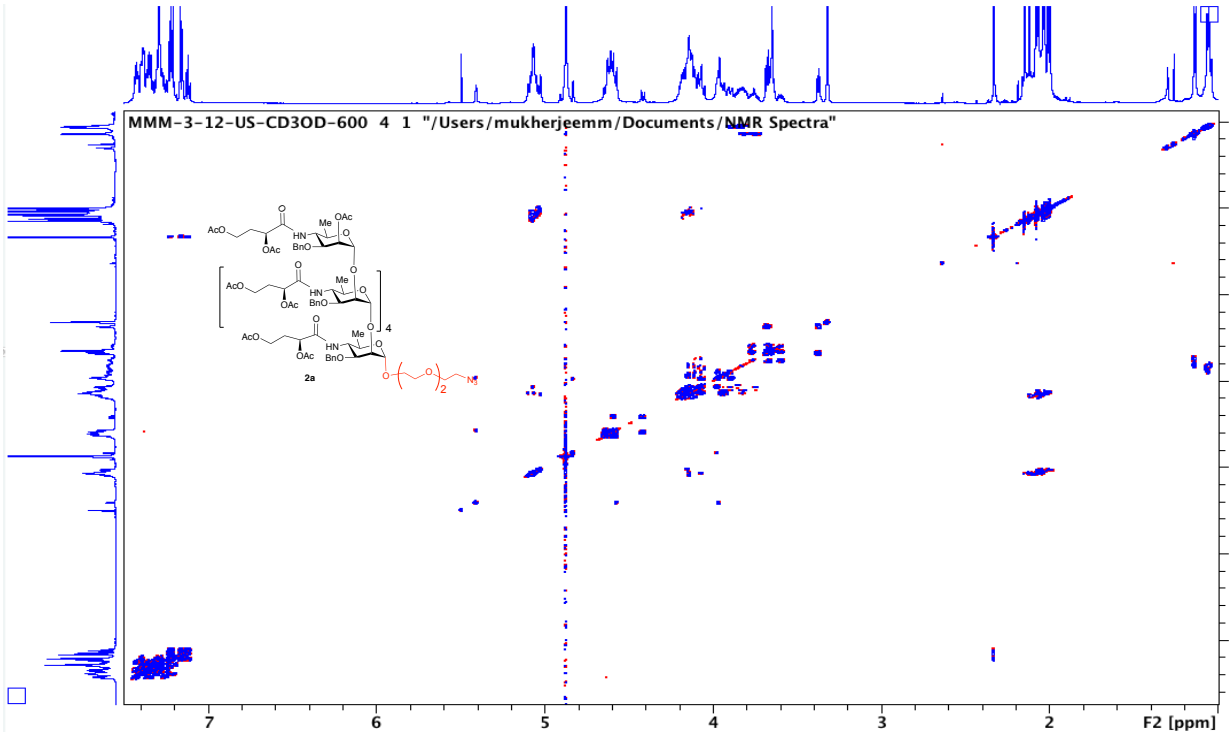

Fig. S77: COSY NMR spectra of compound **2a** (CD_3OD, 600 MHz).

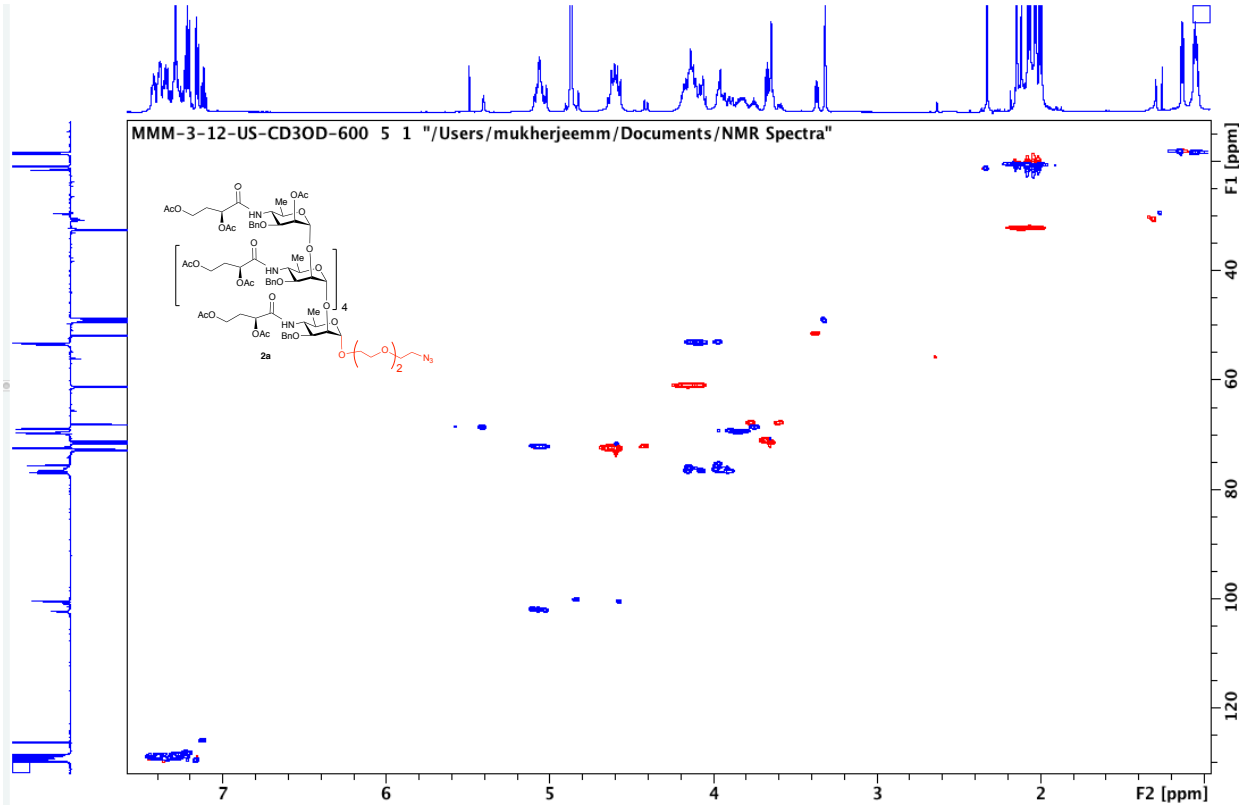

Fig. S78: HSQC NMR spectra of compound **2a** (CD_3OD).

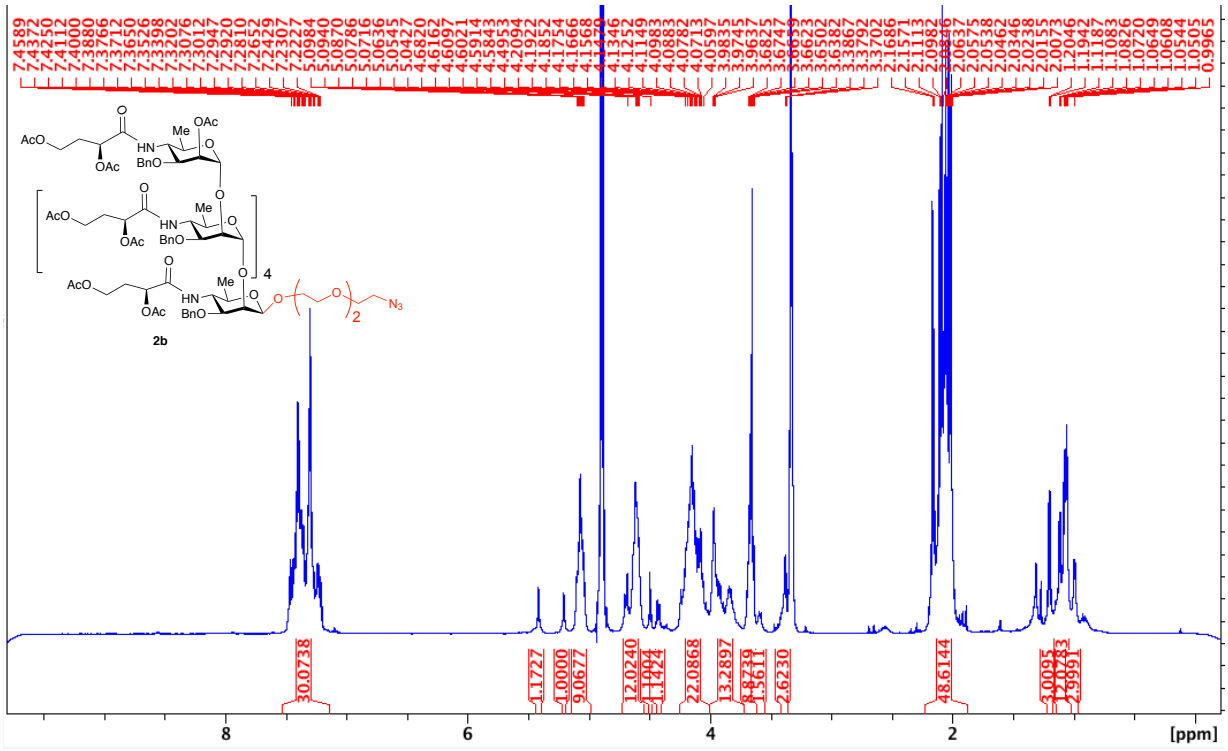

Fig. S79: ^{1}H NMR spectra of compound **2b** (CD_3OD, 600 MHz).

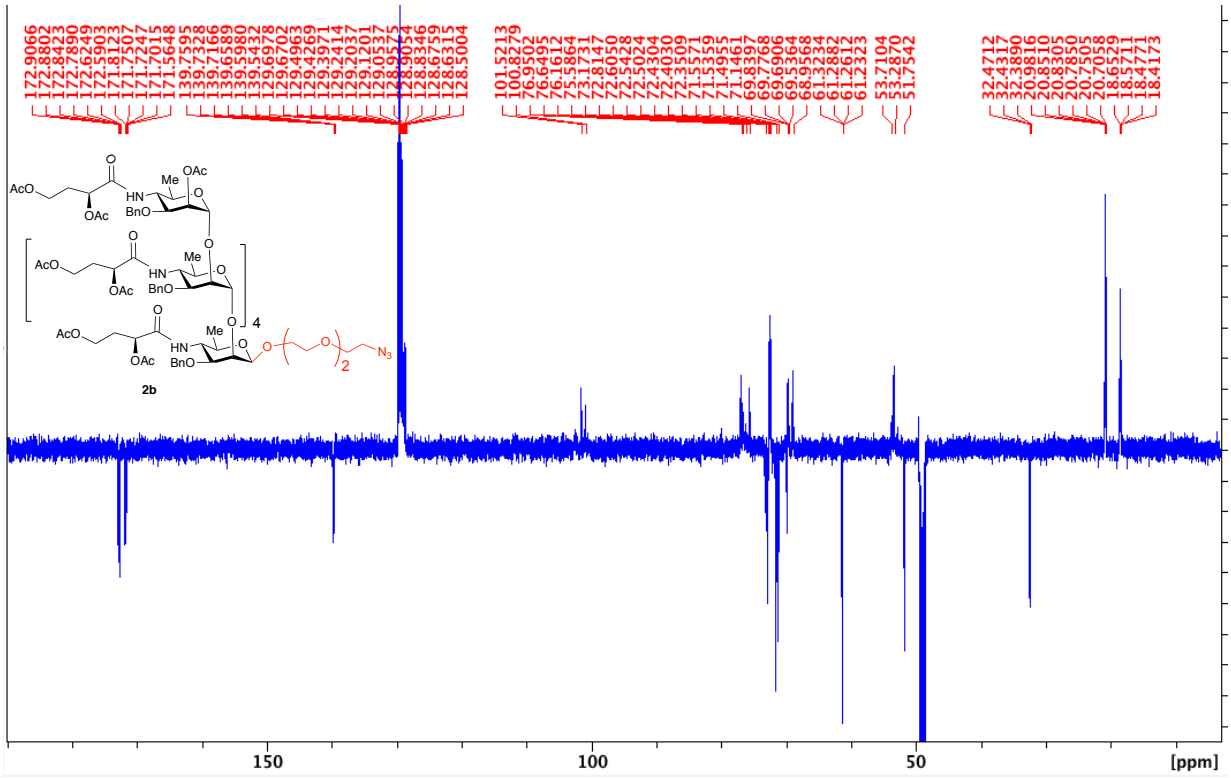

Fig. S80: ^{13}C{1H} NMR spectra of compound **2b** (CD_3OD, 150 MHz).

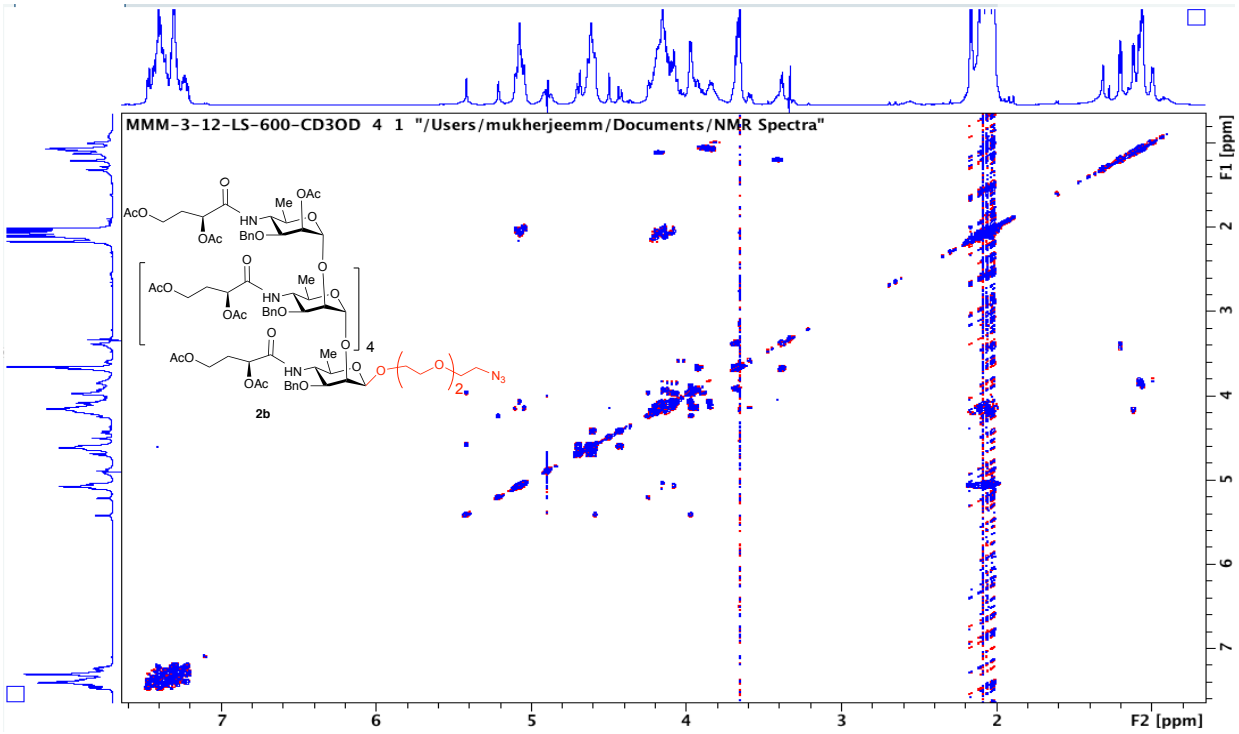

Fig. S81: COSY NMR spectra of compound **2b** (CD_3OD, 600 MHz).

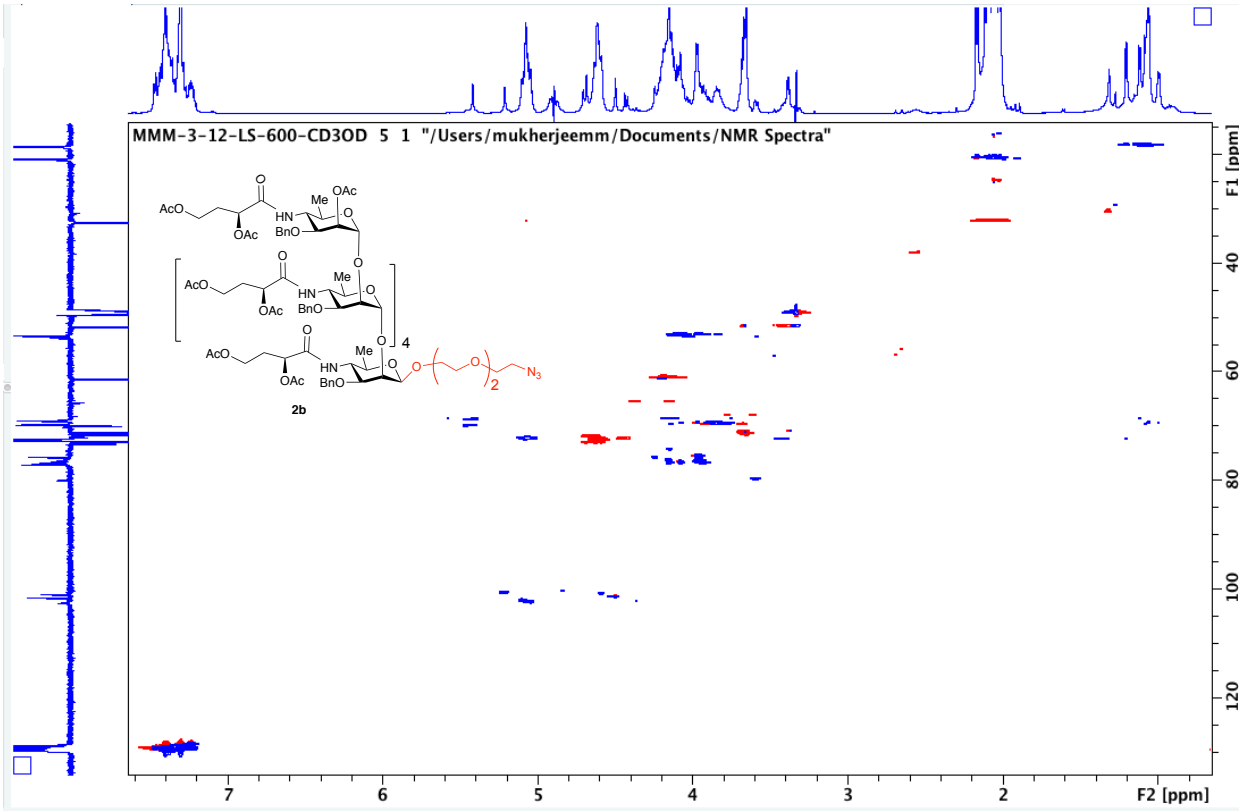

Fig. S82: HSQC NMR spectra of compound **2b** (CD_3OD).

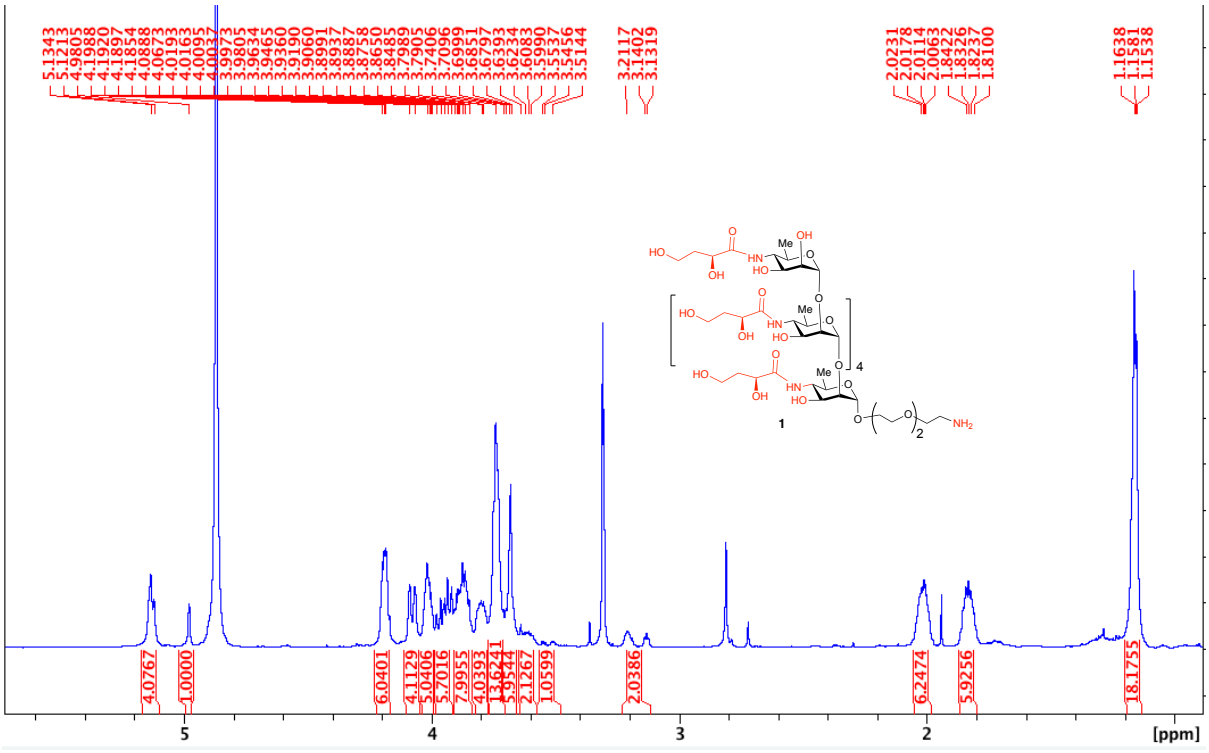

Fig. S83: ^{1}H NMR spectra of compound **1** (CD_3OD, 600 MHz).

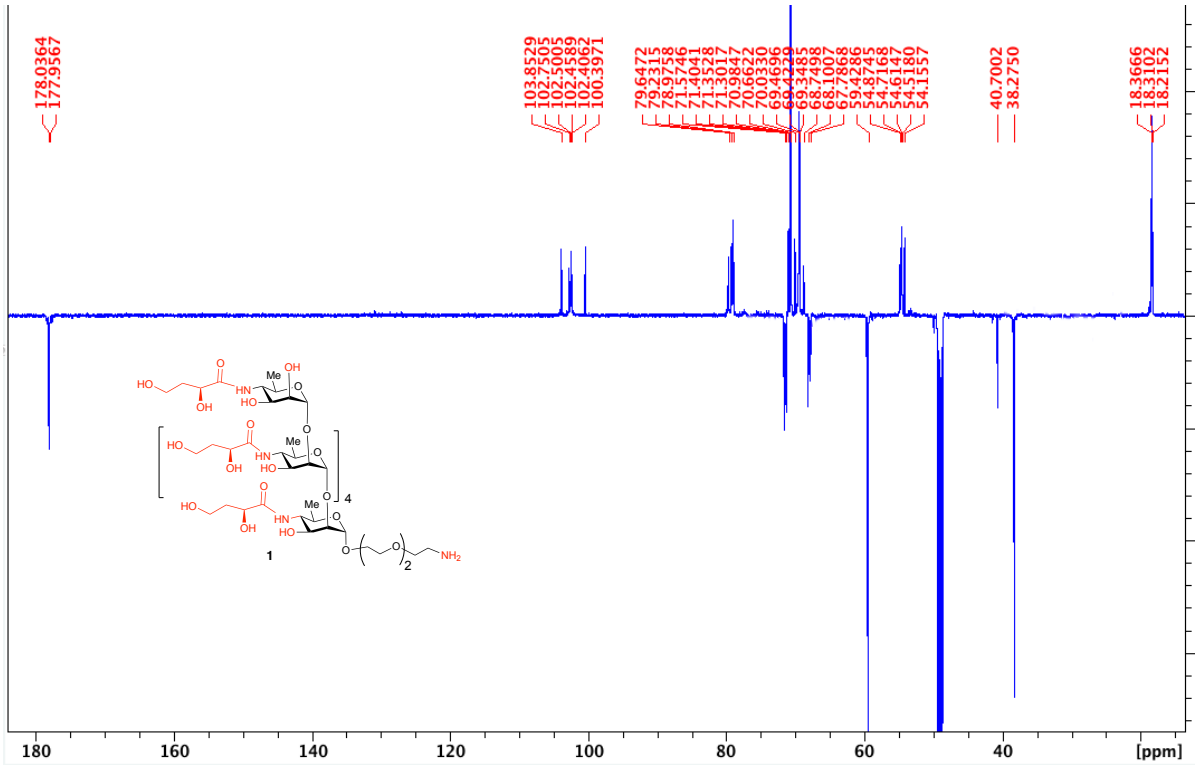

Fig. S84: ^{13}C{1H} NMR spectra of compound **1** (CD_3OD, 150 MHz).

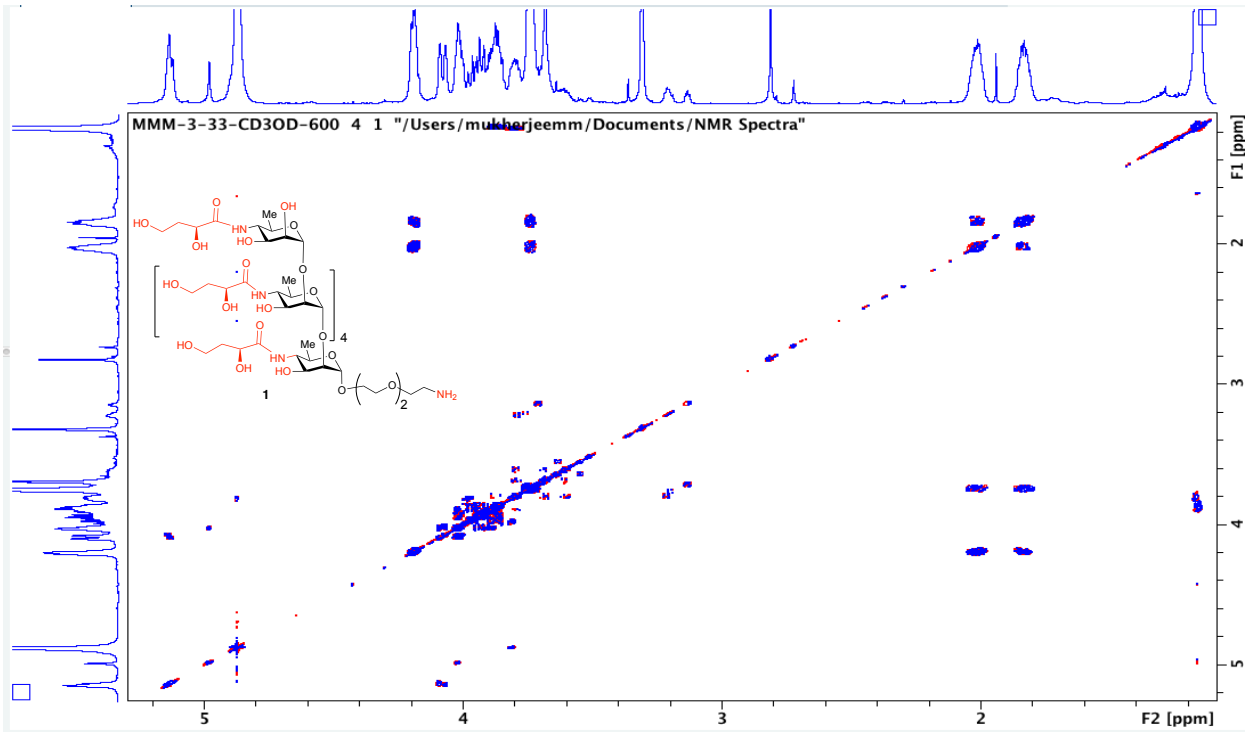

Fig. S85: COSY NMR spectra of compound **1** (CD_3OD, 600 MHz).

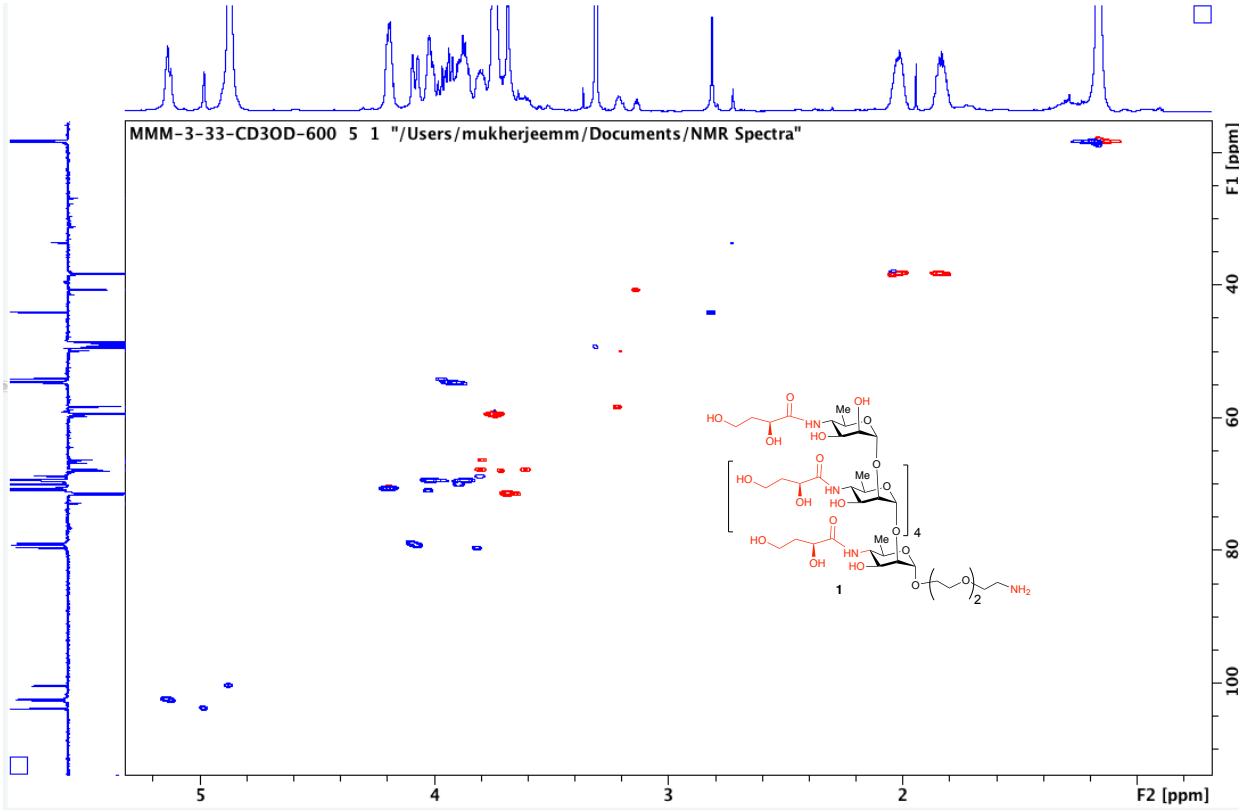

Fig. S86: HSQC NMR spectra of compound **1** (CD_3OD).

Table for Crystal data and structure refinement for **4**.

CCDC deposition number	**1939745**	
Empirical formula	$C_{44}H_{59}N_9O_{10}Si$	
Formula weight	902.09	
Temperature	120(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1$	
Unit cell dimensions	a = 11.530(3) Å	α = 90°
	b = 16.862(4) Å	β = 98.067(4)°
	c = 25.697(6) Å	γ = 90°
Volume	4947(2) $Å^3$	
Z	4	
Density (calculated)	1.211 Mg/m^3	
Absorption coefficient	0.105 mm^{-1}	
F(000)	424	
Crystal size	0.40 × 0.30 × 0.30 mm^3	
Theta range for data collection	1.45 to 47.95°.	
Index ranges	$-24 \le h \le 24$, $-35 \le k \le 23$, $-52 \le l \le 52$	
Reflections collected	268004	
Independent reflections	72975 (R(int) = 0.0362)	
Completeness to theta = 47.95°	96.9%	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7038 and 0.7470	
Refinement method	Full-matrix least-squares on F^2	
Data/restraints/parameters	56235/1/1167	
Goodness-of-fit on F^2	1.038	
Final R indices (I > 2sigma(I))	R1 = 0.0445, wR2 = 0.1034	
R indices (all data)	R1 = 0.0691, wR2 = 0.1171	
Absolute structure parameter	-0.013(14)	
Largest diff. peak and hole	1.376 and −0.410 $e \cdot Å^{-3}$	