Electronic Supplementary Information (ESI)

Side Chain-specific 11/9-Helix Propensity of α/β-Peptides with Alternating Residue Type

Jaeyeon Lee, Jihyun Shim, Philjae Kang, Moon-Gun Choi and Soo Hyuk Choi*

Department of Chemistry, Yonsei University, Seoul, 03722, Republic of Korea

Table of Contents

Synthesis and Characterization Data

General

α-Amino acids and 1-ethyl-3-(3-dimethylaminopropyl)-carbodiimide (EDCI) were purchased from Chem-Impex International. Other reagents were purchased from Sigma-Aldrich, Alfa Aesar, Samchun Chemical, and TCI. *cis*-2-Aminocyclophexanecarboxylic acid (*cis*-ACHC) and related analogs were prepared by the method reported previously.[S1] Analytical thin-layer chromatography (TLC) was carried out on Pre-coated silica gel glass plate (Merck silica gel 60, F254, 0.25 mm). Silica gel 60 (230~240 mesh, Merck) was used for flash column chromatography. Mass spectra (MS) were acquired using an LTQ Orbitrap Spectrometer (ThermoFisher scientific Inc.). Yields for general procedures were not optimized..

General procedure for peptide coupling I

To a 0.1 M solution of an amine (1 equiv) and an acid (1 equiv) in DCM(or DMF), EDCI (1.5 equiv), HOBt(1.3equiv) and TEA (1.1 equiv) are added, and the reaction mixture is stirred at rt for 2-3 days. The reaction mixture is diluted with EtOAc and then washed with 10% aqueous citric acid, aqueous saturated $NaHCO_3$, and brine. The organic layer is then dried over $MgSO_4$, filtered and concentrated to give a crude product, which is purified by flash column chromatography.

General procedure for peptide coupling II

To a 0.1 M solution of an amine (1 equiv) and an acid (1 equiv) in DCM(or DMF), EDCI (1.5 equiv), DMAP(0.3equiv) are added, and the reaction mixture is stirred at rt for 1-2 days. The reaction mixture is diluted with EtOAc and then washed with 10% aqueous citric acid, aqueous saturated $NaHCO_3$, and brine. The organic layer is then dried over $MgSO_4$, filtered and concentrated to give a crude product, which is purified by flash column chromatography.

General procedure for deprotection of the Boc group

An *N*-Boc protected oligomer is treated with TFA in DCM (1:1) for 30 min with stirring, and the mixture is then concentrated under a nitrogen gas stream. The concentrated mixture is used without purification to next step.

General procedure for saponification.

To a solution of an amino ester in MeOH/H_2O(2:1) or THF:MeOH:H_2O (3:1:1) at 0℃ was added LiOH·H_2O (5.0eq). The resulting mixture was stirred at 0℃ for 12h. The pH of the mixture is adjusted to 1.0 by addition of aqueous 1M HCl solution. The resulting mixture is then extracted with EtOAc four times. The combined organic fractions are dried over magnesium sulfate, filtered, and concentrated by rotary evaporation under reduced pressure to give a white solid.

Synthesis of peptide oligomers

α/β-peptides **2-10** were synthesized by the general procedures for peptide coupling analogous to the method reported previously.[S1]

Boc-Ala-*cis*-ACHC-Val-*cis*-ACHC-Ala-OMe (2): R_f : 0.4 (CH_2Cl_2 : EtOAc = 1:1): ^1H ($CDCl_3$, 400 MHz) δ 7.99 (d, 1H, *J* = 7.6 Hz), 7.68 (dd, 2H, *J* = 11.8, 11.2 Hz), 7.31 (d, 1H, *J* = 5.9 Hz), 5.10 (d, 1H, *J* = 6.0 Hz), 4.63 (quin, 1H) 4.20-4.09 (m, 3 H,) 3.77 (s, 3H), 3.63 (q, 1H), 2.61 (s, 1H), 2.55 (s

1H), 2.21 (t, 1H), 2.11-2.00 (m, 3H), 1.82 (q, 3H), 1.72-1.52 (m, 7H), 1.46 (d, 5H, *J* = 7.6 Hz), 1.42 (s, 9H), 1.36 (d, 5H, *J* = 6.9 Hz), 1.02 (dd, 6H, *J* = 6.5, 6.8 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 176.1, 175.6, 174.3, 172.9, 172.8, 155.9, 79.8, 63.3, 52.6, 51.1, 50.0, 48.7, 48.5, 44.0, 43.7, 29.7, 28.5, 28.3 (3C), 28.0, 27.4, 27.2, 25.4, 25.2, 20.8 (2C), 20.7, 19.7, 17.8, 16.0; HRMS calculated for $[M+Na]^+$ 646.3786, found 646.3787.

Boc-Ala-*cis*-ACHC-Aib-*cis*-ACHC-Ala-OMe (3): R_f : 0.18 (CH_2Cl_2 : EtOAc = 2:1): ^{1}H ($CDCl_3$, 400 MHz) δ 7.99 (d, 1H, *J* = 6.1 Hz), 7.62 (d, 1H, *J* = 9.7 Hz), 7.38 (s, 1H), 7.20 (d, 1H, *J* = 9.4 Hz), 5.19 (d, 1H, *J* = 5.5 Hz), 4.52 (quin, 1H), 4.17 (t, 1H), 4.11 (q, 2H), 3.74 (s, 3H), 2.68 (s, 1H), 2.55 (s, 1H), 2.29-1.54 (m, 16 H), 1.43 (t, 12H), 1.38 (d, 9H, *J* = 9.6 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 175.5, 175.1, 174.8, 174.2, 172.8, 155.7, 80.0, 57.2, 52.4, 51.2, 50.3, 48.8, 48.6, 43.7, 43.1, 28.4 (3C), 27.9, 27.5, 27.4, 27.3, 27.0, 25.6, 25.0, 23.3, 20.9 (2C), 17.9, 15.9; HRMS calculated for $[M+Na]^+$ 632.3630, found 632.3629.

Boc-Ala-*cis*-ACHC-Gly-*cis*-ACHC-Ala-OMe (4): R_f : 0.2 (CH_2Cl_2 : EtOAc = 1:1): ^{1}H ($CDCl_3$, 400 MHz) δ 8.00 (d, 1 H, *J* = 9.7 Hz), 7.82 (d, 1 H, *J* = 7.2 Hz), 7.69 (s, 1 H), 7.45 (d, 1 H, *J* = 9.9 Hz), 5.07 (d, 1 H, *J* = 6.0 Hz), 4.60 (p, 1 H), 4.16-4.08 (m, 2 H), 4.04 (quin, 1 H), 3.95 (dd, 1 H, *J* = 20.8, 9.5 Hz), 3.76 (s, 3 H), 3.67 (dd, 1 H, *J* = 21.6, 8.8 Hz), 2.66 (s, 2 H), 2.23-1.53 (m, 16 H), 1.44 (d, 3 H, *J* = 7.5 Hz), 1.41 (s, 9 H), 1.35 (d, 3 H, *J* = 7.0 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 176.1, 175.5, 174.4, 172.9, 170.8, 156.3, 80.1, 52.6, 51.2, 49.5, 48.6 (2C), 44.8, 43.6, 43.4, 28.3 (3C), 27.9, 27.5, 27.2, 26.8, 25.2 (2 C), 20.9, 20.8, 17.8, 16.0; HRMS calculated for $[M+Na]^+$ 604.3317, found 604.3316.

Boc-Ala-*cis*-ACHC-D-Ala-*cis*-ACHC-Ala-OMe (5): R_f : 0.5 (CH_2Cl_2 : EtOAc = 1:1): ^{1}H ($CDCl_3$, 400 MHz) δ 7.04 (d, 1H, *J* = 8.5 Hz), 6.58 (s, 1H), 5.23 (s, 1H), 4.52 (t, 1H), 4.13 (s, 2H), 3.74 (s, 3H), 2.64 (q, 1H), 1.93-1.39 (m, 8H), 1.43 (s, 9H), 1.40 (d, 3H, *J* = 7.1 Hz), 1.32 (d, 3H, *J* = 6.9 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 173.8, 173.5, 172.2, 155.3, 79.7, 52.5, 50.1, 48.0 (2C), 44.4, 29.1, 28.3 (3C), 27.1, 23.4, 22.2, 18.6, 17.8; HRMS calculated for $[M+Na]^+$ 618.3473, found 618.3473.

Boc-Ala-*cis*-ACHC-Aib-*cis*-ACHC-Aib-OMe (6): R_f : 0.1 (CH_2Cl_2 : EtOAc = 2:3): ^{1}H ($CDCl_3$, 400 MHz) δ 7.62 (s, 2H), 7.32 (s, 1H), 6.92 (d, 1H, *J* = 9.4 Hz), 5.26 (s, 1H), 4.16 (d, 1H, *J* = 9.9 Hz), 4.09 (quin, 2H), 3.74 (s, 3H), 2.59 (d, 1H, *J* = 2.6 Hz), 2.55 (d, 1H, *J* = 3.3 Hz), 2.15-1.37 (m, 40H, broad); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 175.9, 175.2, 174.4, 173.9, 172.8, 155.7, 80.0, 57.1, 56.6, 52.5, 51.1, 48.9, 48.5, 43.9, 43.5, 28.4 (3C), 28.2, 27.7 (2C), 27.1, 26.9, 25.8, 25.1, 24.7 (2C), 23.5, 21.2, 21.0, 18.0; HRMS calculated for $[M+Na]^+$ 646.3786, found 646.3785.

Boc-Aib-*cis*-ACHC-Aib-*cis*-ACHC-Ala-OMe (7): R_f : 0.1 (CH_2Cl_2 : EtOAc = 2:3): ^{1}H ($CDCl_3$, 400 MHz) δ 7.80 (s, 1H), 7.26 (d, 1H, *J* = 7.6 Hz), 7.15 (d, 2H, *J* = 9.2 Hz), 5.17 (s, 1H), 4.51 (p, 1H), 4.20 (s, 1H), 4.15-4.05 (m, 1H), 3.74 (s, 3H), 2.68 (d, 1H, *J* = 3.3 Hz), 2.51 (d, 1H, *J* = 4.4 Hz), 2.18-1.49 (m, 19H, broad), 1.44-1.41 (m, 21H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 174.8, 174.3, 174.2, 174.1, 173.9, 154.8, 79.8, 60.0, 56.4, 52.4, 49.8, 48.7 (2C), 44.4, 43.3, 28.6, 28.4 (3C), 27.9, 27.3, 26.8 (2C), 26.6, 25.2, 24.4, 24.1, 23.7, 21.8, 21.2, 16.2; HRMS calculated for $[M+Na]^+$ 646.3786, found 646.3786.

Boc-Aib-*cis*-ACHC-Aib-*cis*-ACHC-Aib-OMe (8): R_f : 0.1 (CH_2Cl_2 : EtOAc = 2:3): ^{1}H ($CDCl_3$, 400 MHz) δ 7.29 (d, 1H, *J* = 11.9 Hz), 7.22 (s, 1H), 6.97 (d, 2H, *J* = 8.8 Hz), 5.18 (s, 1H), 4.21 (d, 1H, *J* = 3.7 Hz), 4.11 (q, 1H), 3.73 (s, 3H), 2.59 (t, 1H), 2.51 (q, 1H), 1.96-1.43 (m, 43H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 175.6, 174.3, 174.2, 173.8, 173.6, 154.8, 79.8, 56.9, 56.5, 56.4, 52.5, 49.1, 48.3, 44.9, 43.9, 29.0, 28.4 (3C), 28.2, 27.4, 26.7, 26.5, 26.0, 25.5, 24.7, 24.5, 24.4, 24.1, 23.4, 22.4, 21.6; HRMS calculated for $[M+Na]^+$ 660.3943, found 660.3942.

Boc-Ala-β³-(R)-hAla-Ala-*cis*-ACHC-Ala-OMe (9): R_f : 0.12 (EtOAc 100%): ^{1}H ($CDCl_3$, 400 MHz) δ 7.78 (d, 1H, *J* = 5.6 Hz), 7.72 (d, 2H, *J* = 6.7 Hz), 7.58 (d, 1H, *J* = 10 Hz), 5.05 (d, 1H, *J* = 5.8 Hz), 4.58 (quin, 1H), 4.46 (s, 1H), 4.21-4.08 (m, 2H), 3.98 (quin, 1H), 3.76 (s, 3H), 2.59 (s ,1H), 2.54 (dd, 1H, *J* = 17, 7.5 Hz), 2.25-1.51 (m, 8H), 2.11 (dd, 1H, *J* = 15, 9.0 Hz), 1.44 (d, 3H, *J* = 7.6 Hz), 1.42 (s, 9H), 1.40 (d, 3H, *J* = 7.4 Hz), 1.36 (d, 3H, *J* = 6.9 Hz), 1.20 (d, 3H, *J* = 7.0 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 176.3, 174.5, 174.3, 172.9, 171.8, 155.9, 80.2, 52.7, 51.6, 51.4, 49.3, 48.7, 44.0, 42.3, 42.0, 28.2 (3C), 27.6, 27.2, 25.2, 20.8, 18.9, 17.5, 16.7, 16.1; MALDI-TOF MS calculated for $[M+Na]^+$ 578.3160, found 578.90.

Boc-Ala-*cis*-ACHC-Ala-β³-(R)-hAla-Ala-*cis*-ACHC-Ala-OMe (10): ^{1}H NMR ($CDCl_3$. 400 MHz) δ 8.44 (s, 1H), 8.03 (d, *J*=8, 1H), 7.87 (d, *J* = 4Hz, 1H), 7.73 (d, *J* = 8Hz, 1H), 7.65 (d, *J* = 8Hz, 1H), 7.56 (d, *J* = 4Hz, 1H), 5.21 (d, *J* = 4Hz, 1H), 4.58 (s, 1H), 4.43 (s, 1H), 4.20-4.08 (m, 5H), 3.76 (s, 3H), 2.64 (m, 1H), 2.60-2.53 (m, 1H), 2.26-2.21 (m, 1H), 2.15-2.12 (m, H), 2.08-1.50 (m, 16H), 1.45-1.35 (m, 15H), 1.23–1.21 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 176.4, 175.2, 174.9, 174.3, 172.9, 171.6, 155.8, 79.98, 77.24, 52.7, 51.8, 51.7, 51.1, 49.3, 48.7, 48.4, 44.0, 43.6, 42.6, 41.9, 29.7, 28.2(3C), 27.5, 27.2, 26.7, 25.3, 24.8, 20.9, 20.7, 18.6, 17.7, 16.6, 16.0; MALDI-TOF MS calculated for $[M+Na]^+$ 774.4372, found 774.46.

Backbone Proton Chemical Shifts (ppm, in $CDCl_3$)

α/β-Peptides **2** in $CDCl_3$:

	Boc	Hα	Hβ	HN	OMe
N-term	1.42	-	-	-	-
Ala1	-	4.11	-	5.09	
ACHC2	-	2.60	4.16	7.71	-
Val3	-	3.62	-	7.33	-
ACHC4	-	2.55	4.16	7.68	-
Ala5	-	4.63	-	8.00	-
C-term	-	-	-	-	3.72

α/β-Peptides **2** in CD_3OH :

	Boc	Hα	Hβ	HN	OMe
N-term	1.44				
Ala1		4.44		6.80	
ACHC2		2.73	4.08	7.74	
Val3		4.00		7.83	
ACHC4		2.68	4.14	7.67	
Ala5		4.44		8.28	
C-term					3.72

α/β-Peptides **3** in $CDCl_3$:

	Boc	Hα	Hβ	HN	OMe
N-term	1.44				
Ala1		4.11		5.16	
ACHC2		2.54	3.17	7.64	
Aib3				7.38	
ACHC4		2.68	4.08	7.22	
Ala5		4.52		7.99	
C-term					3.75

α/β-Peptides **3** in CD_3OH:

	Boc	Hα	Hβ	HN	OMe
N-term	1.44				
Ala1		4.04		6.87	
ACHC2		2.61	4.14	7.60	
Aib3				7.96	
ACHC4		2.68	4.01	7.43	
Ala5		4.38		8.25	
C-term					3.71

α/β-Peptides **4** in $CDCl_3$:

	Boc	Hα	Hβ	HN	OMe
N-term	1.40				
Ala1		4.02		5.00	
ACHC2		2.65	4.11	8.02	
Gly3		3.57			
		3.93			
ACHC4		2.65		7.70	
Ala5		4.60	4.11	7.45	
C-term					3.76

α/β-Peptides **4** in CD_3OH:

	Boc	Hα	Hβ	HN	OMe
N-term	1.43				
Ala1		4.02		5.00	
ACHC2		2.65	4.11	8.02	
Gly3		3.57		7.70	
		3.93			
		2.65	4.11	7.45	
ACHC4		4.60		7.84	
Ala5		4.38		8.25	
C-term					3.72

α/β-Peptides **5** in $CDCl_3$:

	Boc	Hα	Hβ	HN	OMe
N-term	1.43				
Ala1		4.12		5.12	
ACHC2		2.58	4.12	7.37	
D-Ala3		4.12		7.01	
ACHC4		2.64	4.12	7.13	
Ala5		4.50		7.01	
C-term					3.75

α/β-Peptides **9** in $CDCl_3$:

	Boc	Hα	Hβ	HN	OMe
N-term	1.41				
Ala1		3.97		5.01	
β^3-hAla2		2.11	4.47	7.73	
		2.54			
Ala3		4.16		7.81	
ACHC4		2.58	4.12	7.60	
Ala5		4.58		7.75	
C-term					3.75

α/β-Peptides **10** in $CDCl_3$:

	Boc	Hα	Hβ	HN	OMe
N-term	1.42				
Ala1		4.11		5.21	
ACHC2		2.64	4.16	7.73	
Ala3		4.20		7.56	
β^3-hAla4		2.14 2.55	4.43	8.03	
Ala5		4.16		8.44	
ACHC6		2.60	4.12	7.65	
Ala7		4.58		7.87	
C-term					3.76

Copies of Two-dimensional NMR spectra

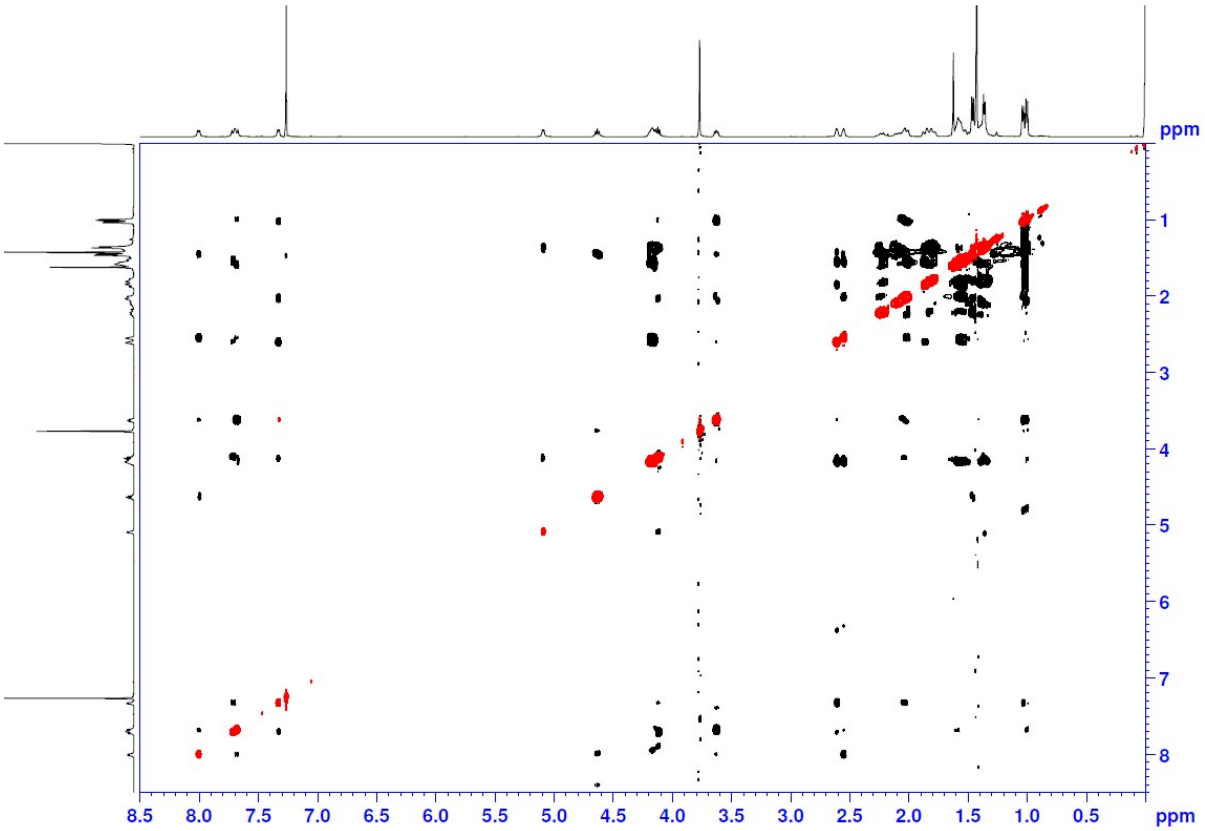

Figure S1. ROESY spectra of **2** in $CDCl_3$.

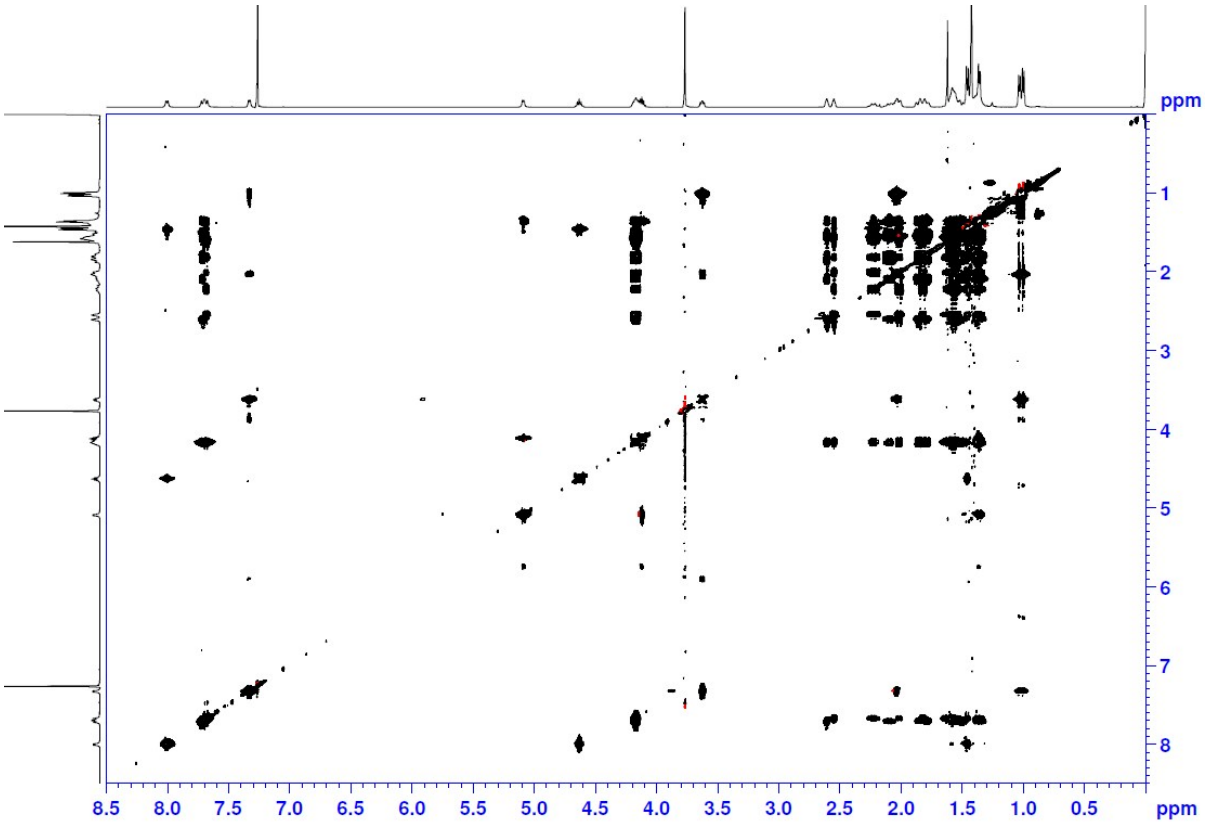

Figure S2. TOCSY spectra of **2** in $CDCl_3$.

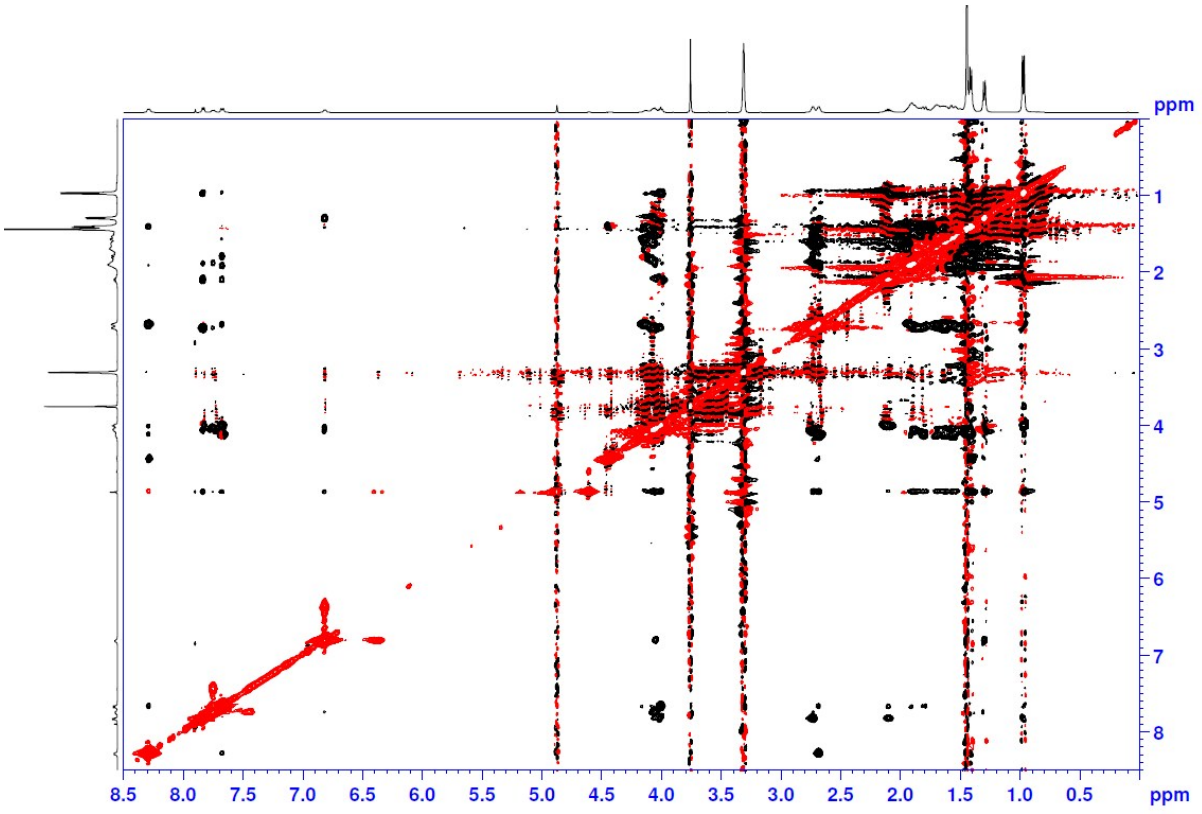

Figure S3. ROESY spectra of **2** in CD_3OH.

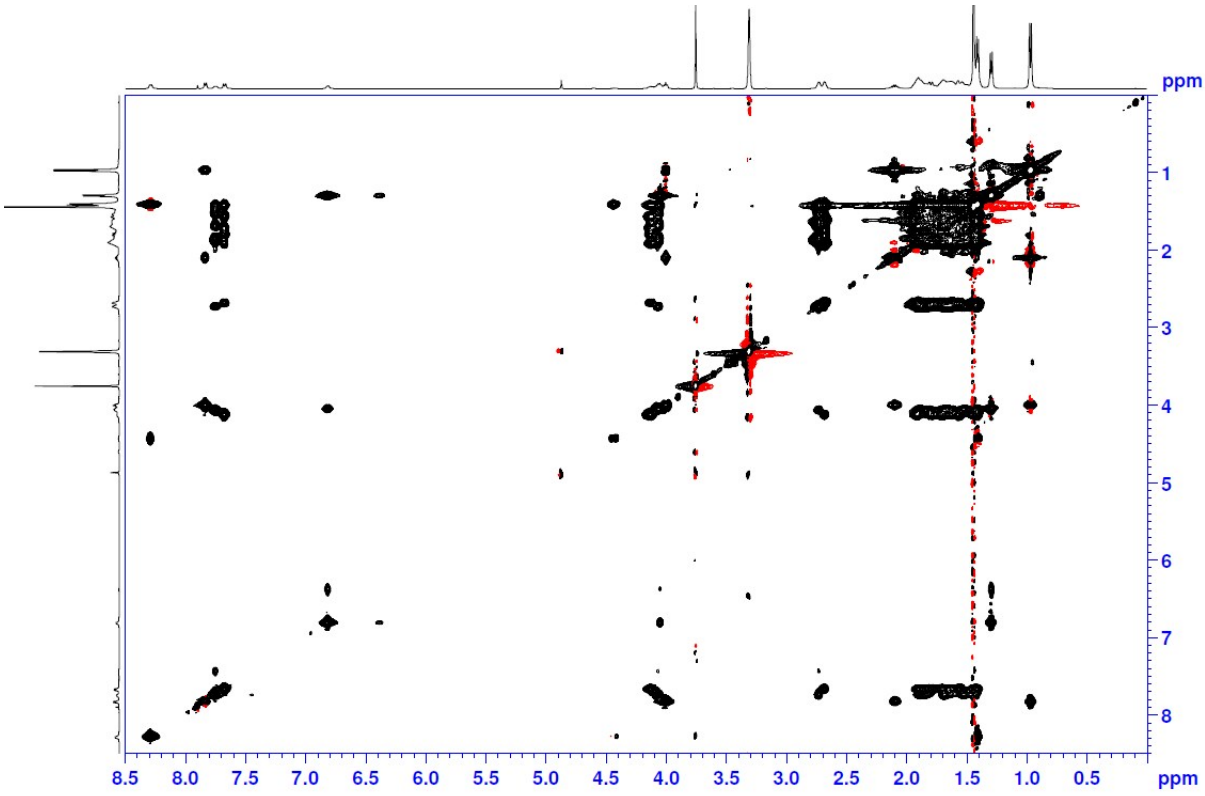

Figure S4. TOCSY spectra of **2** in CD_3OH.

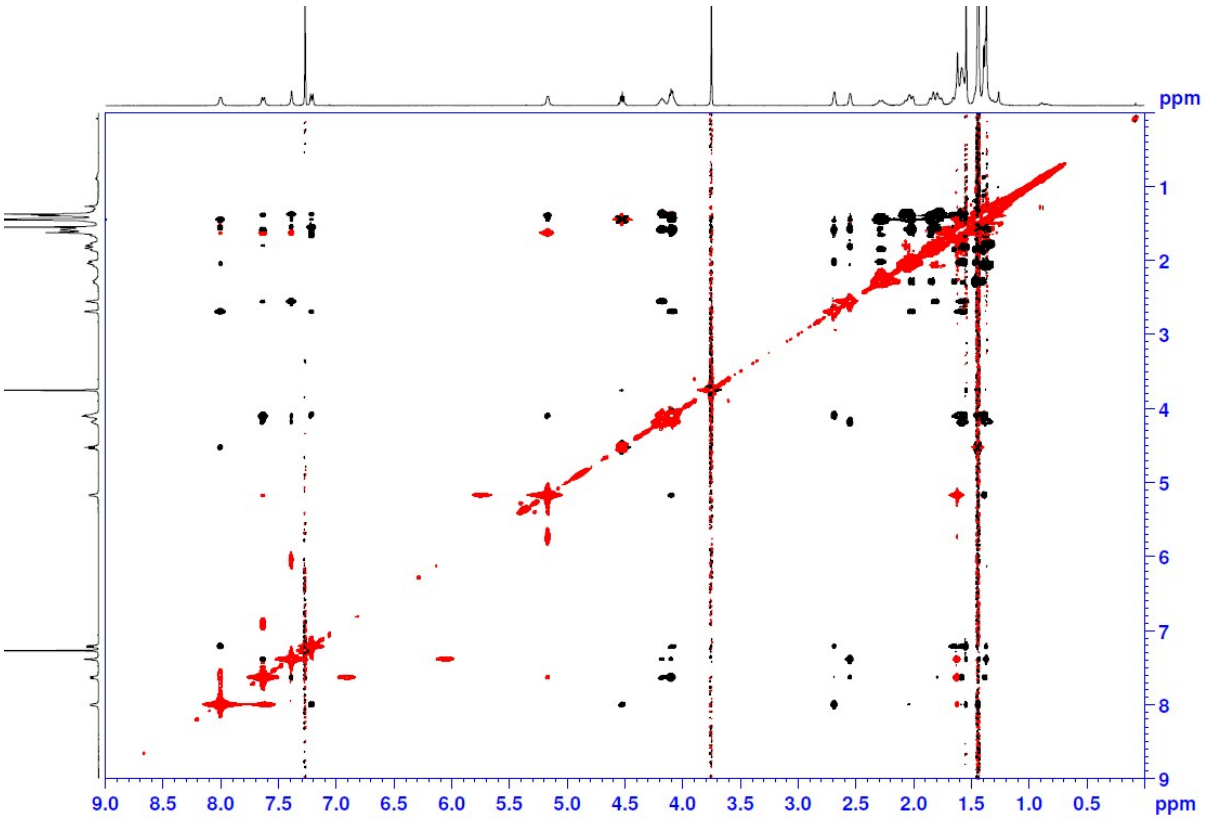

Figure S5. NOESY spectra of **3** in $CDCl_3$.

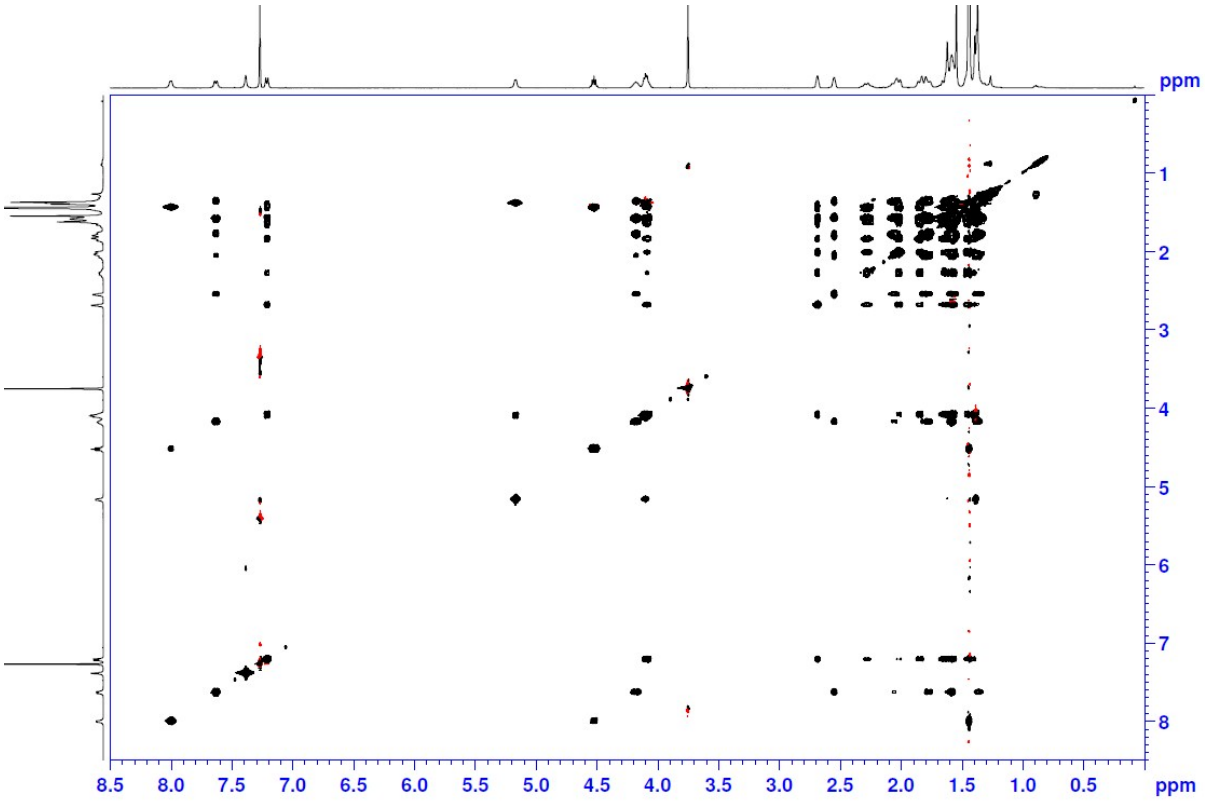

Figure S6. TOCSY spectra of **3** in $CDCl_3$.

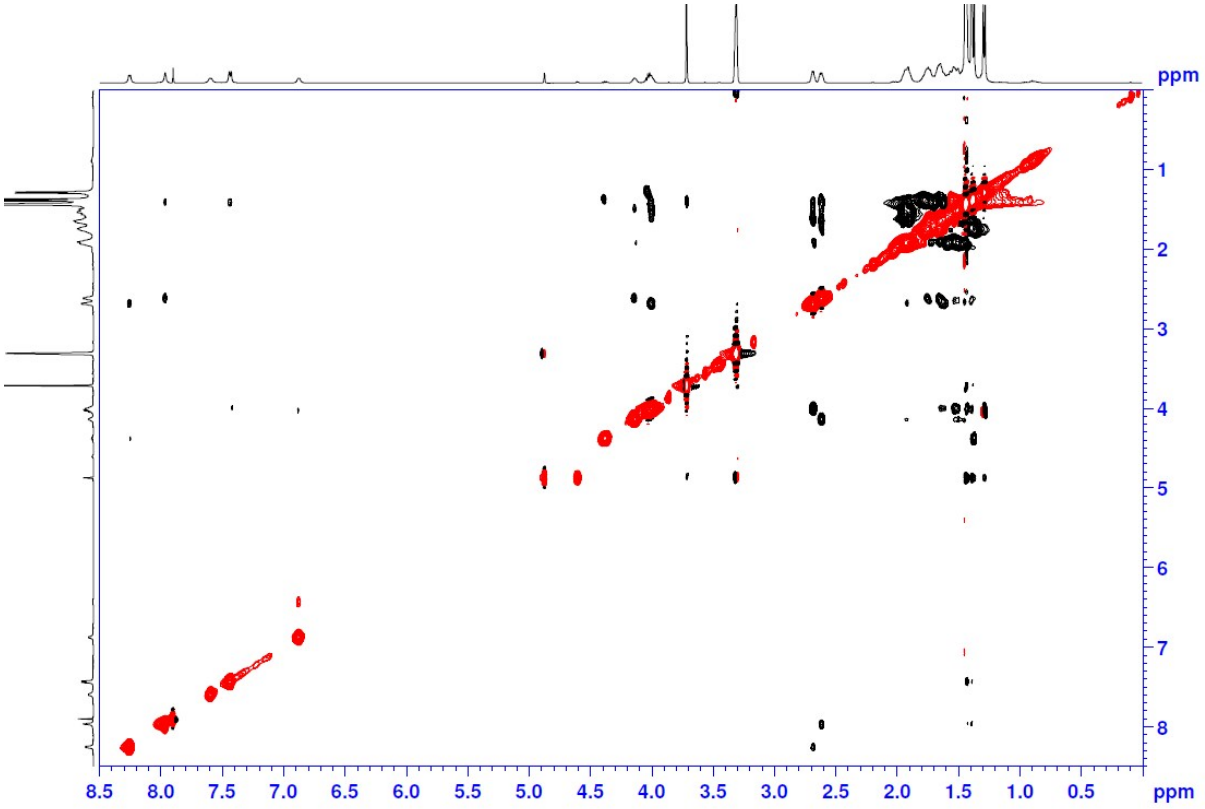

Figure S7. ROESY spectra of **3** in CD_3OH.

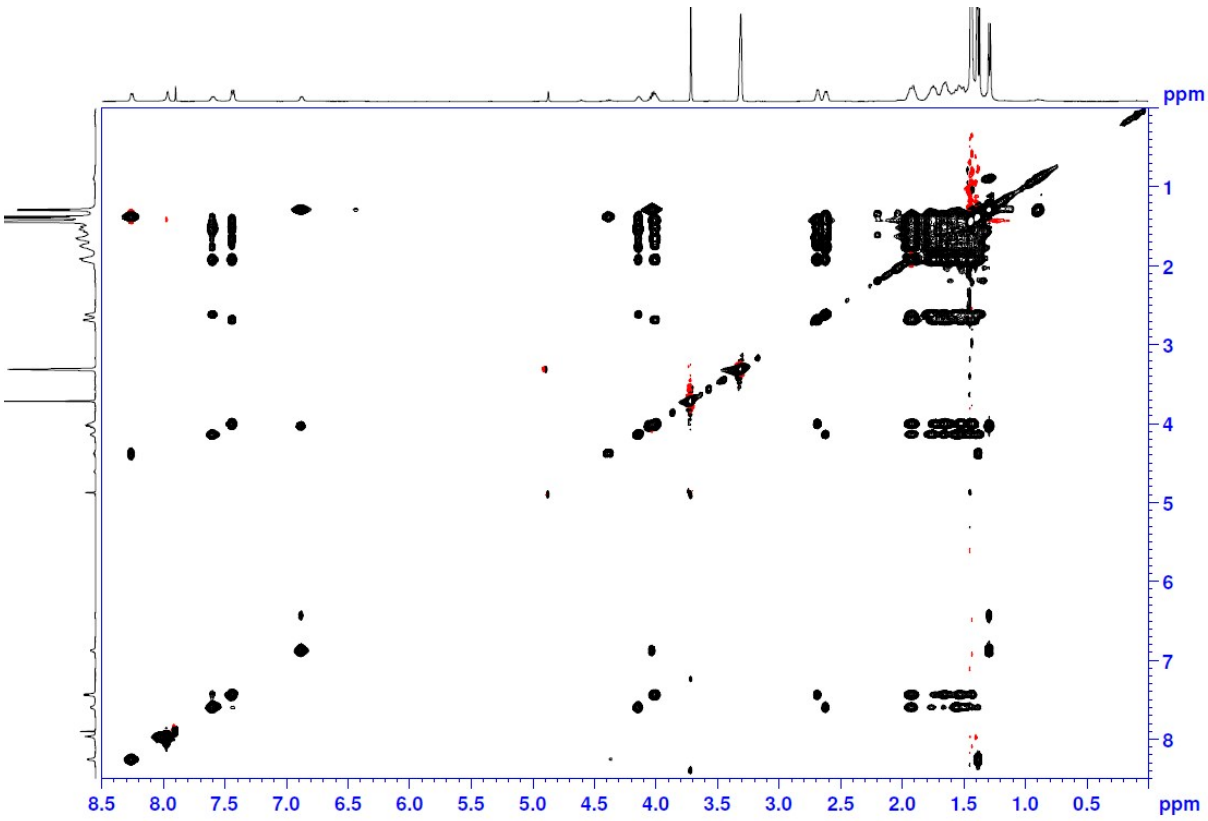

Figure S8. TOCSY spectra of **3** in CD_3OH.

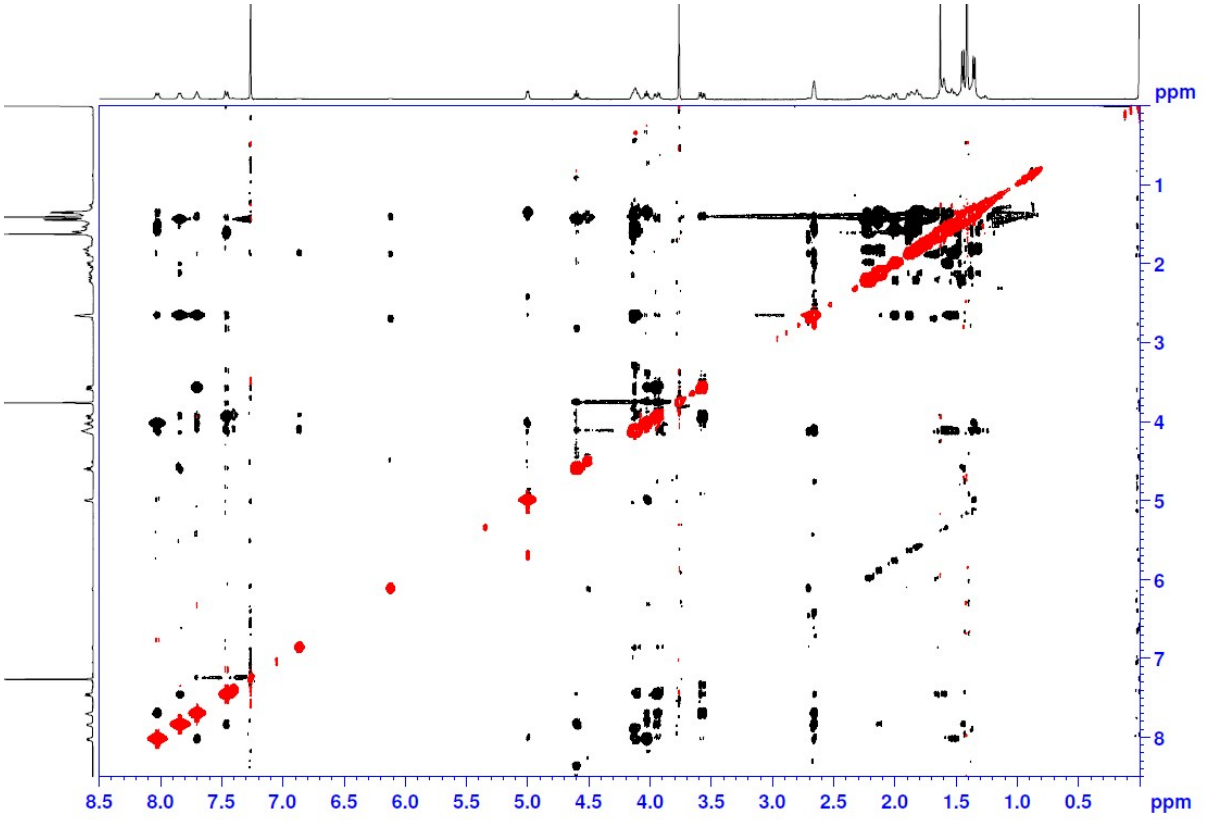

Figure S9. NOESY spectra of **4** in $CDCl_3$.

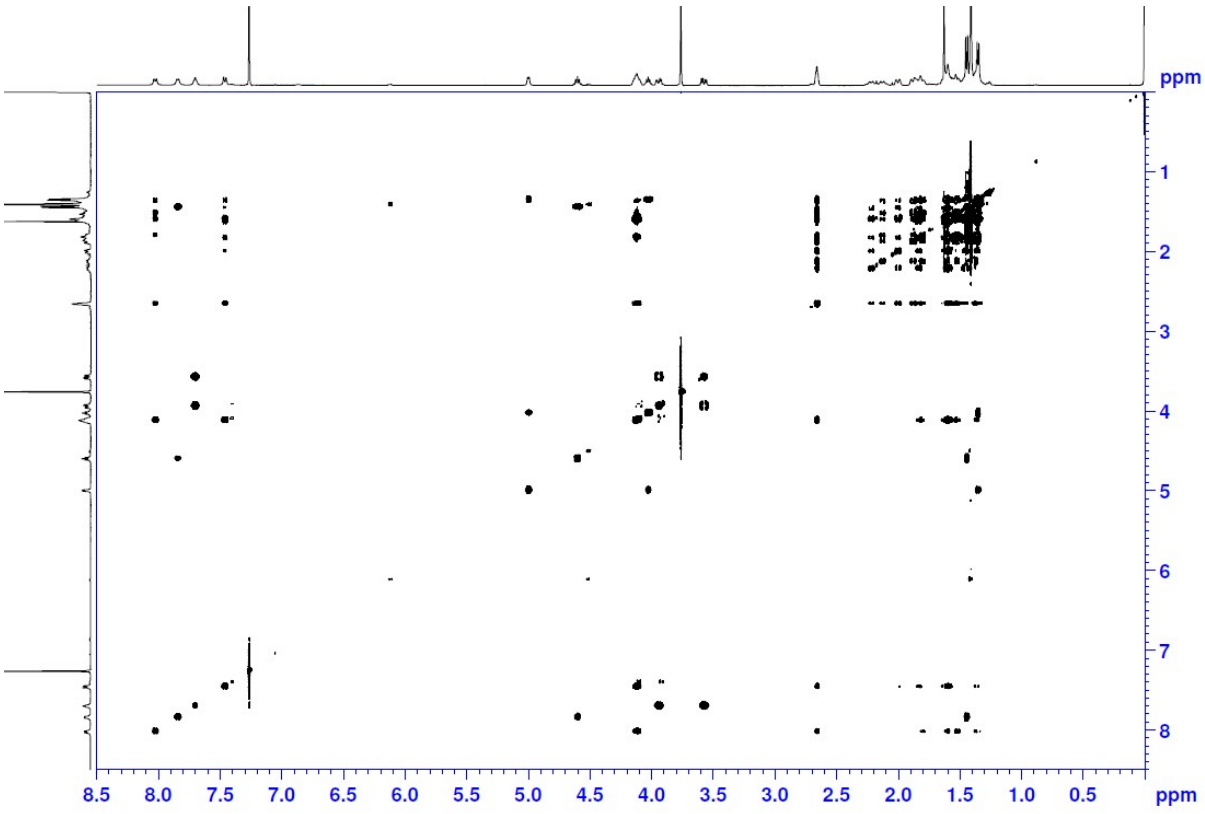

Figure S10. TOCSY spectra of **4** in $CDCl_3$.

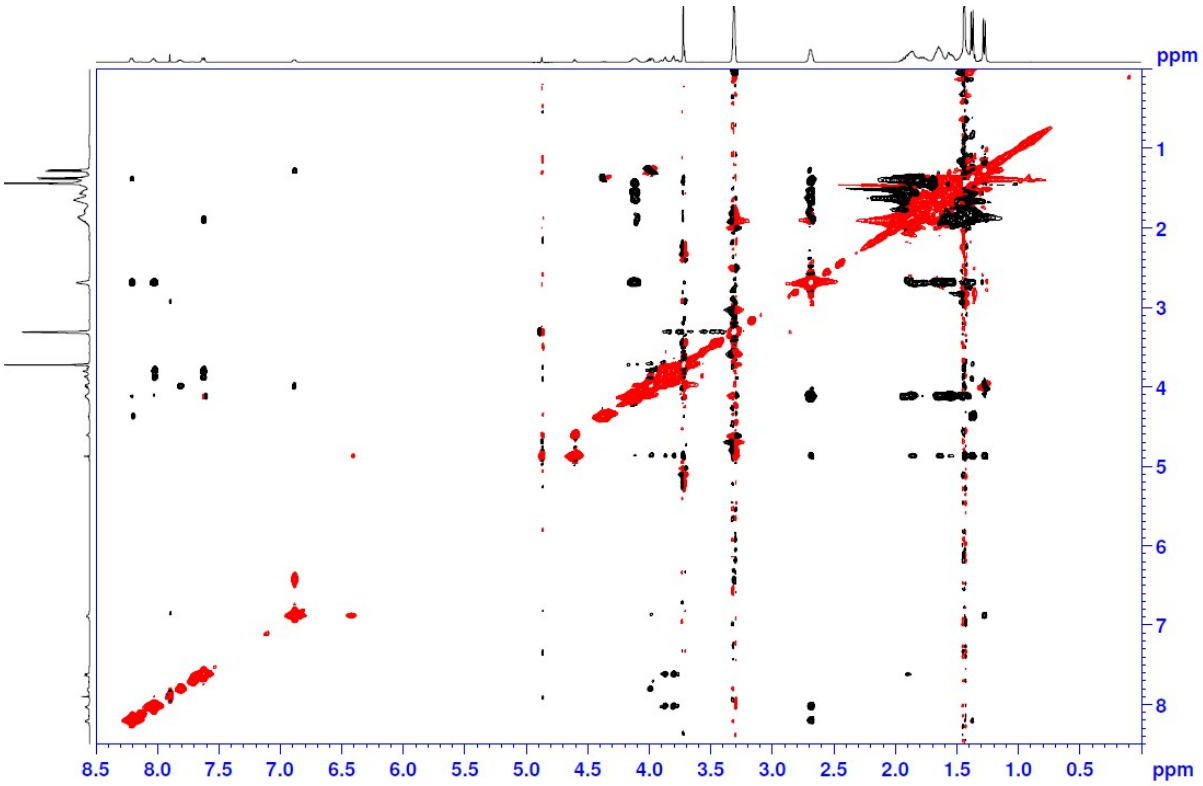

Figure S11. ROESY spectra of **4** in CD_3OH.

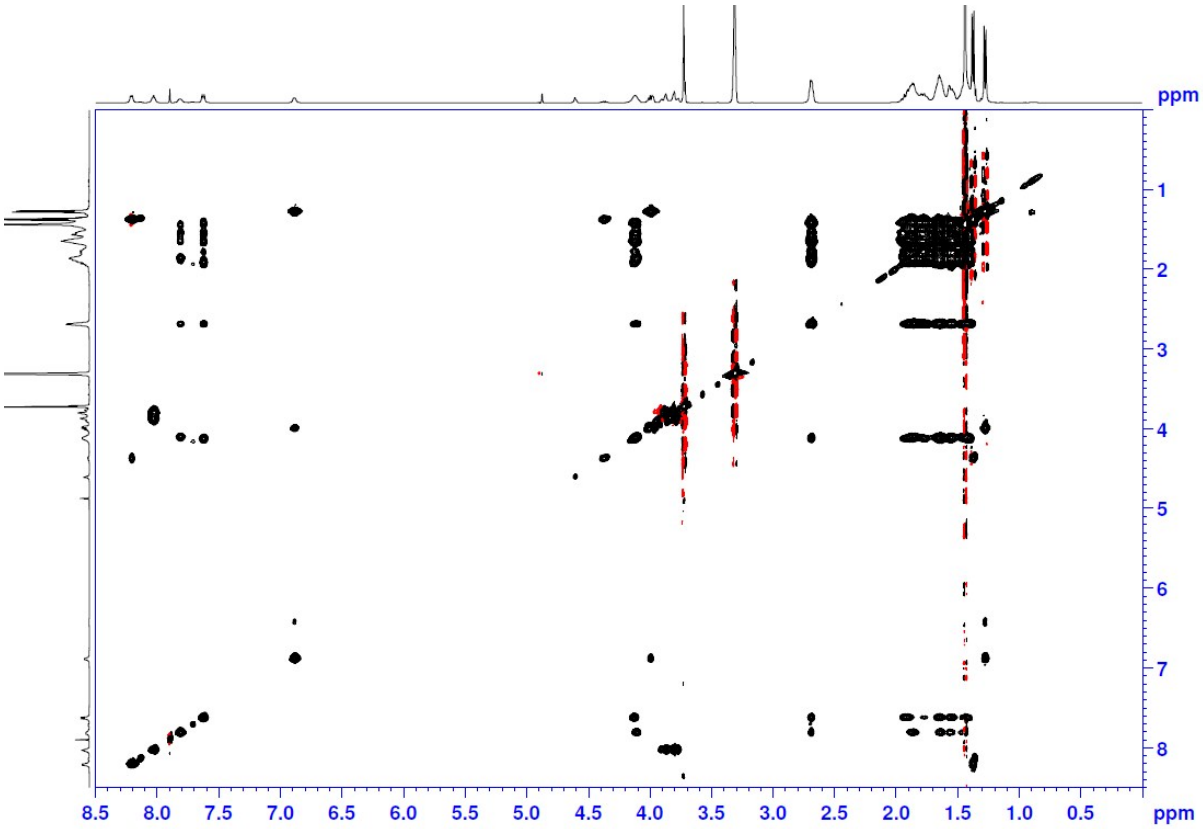

Figure S12. TOCSY spectra of **4** in CD_3OH.

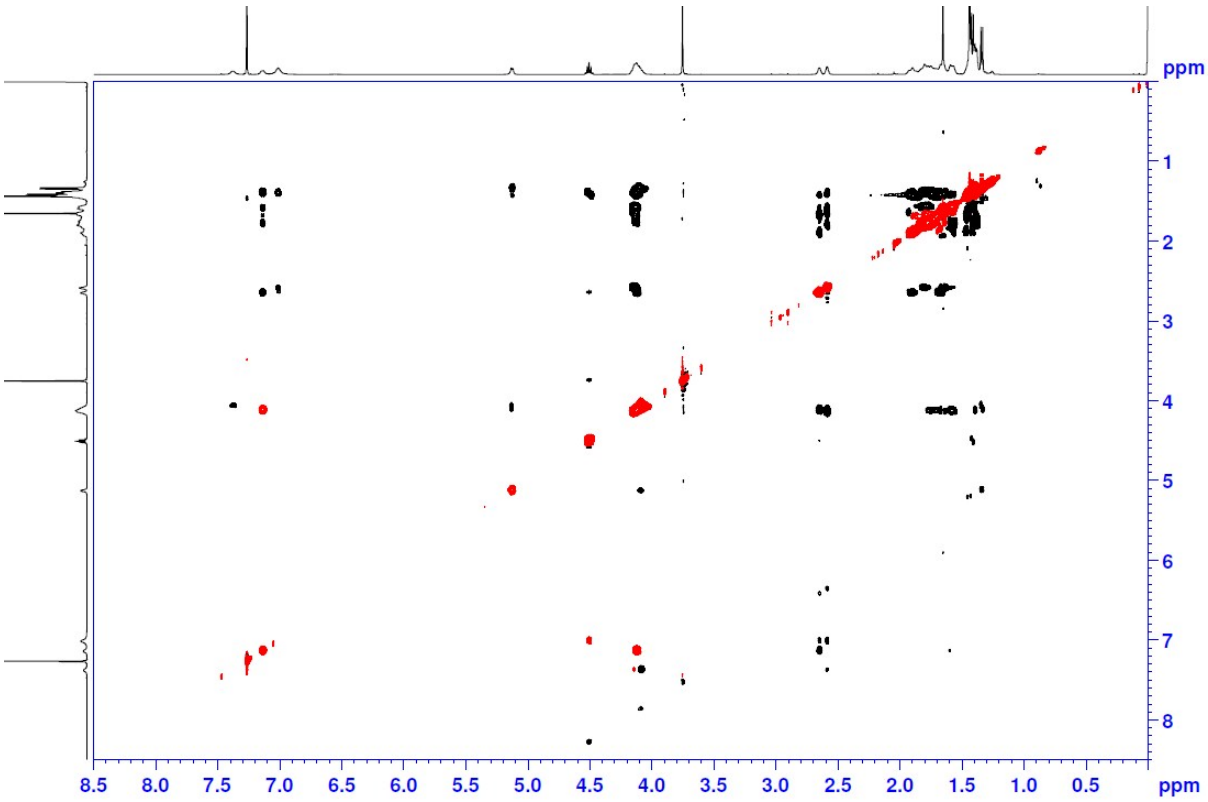

Figure S13. ROESY spectra of **5** in $CDCl_3$.

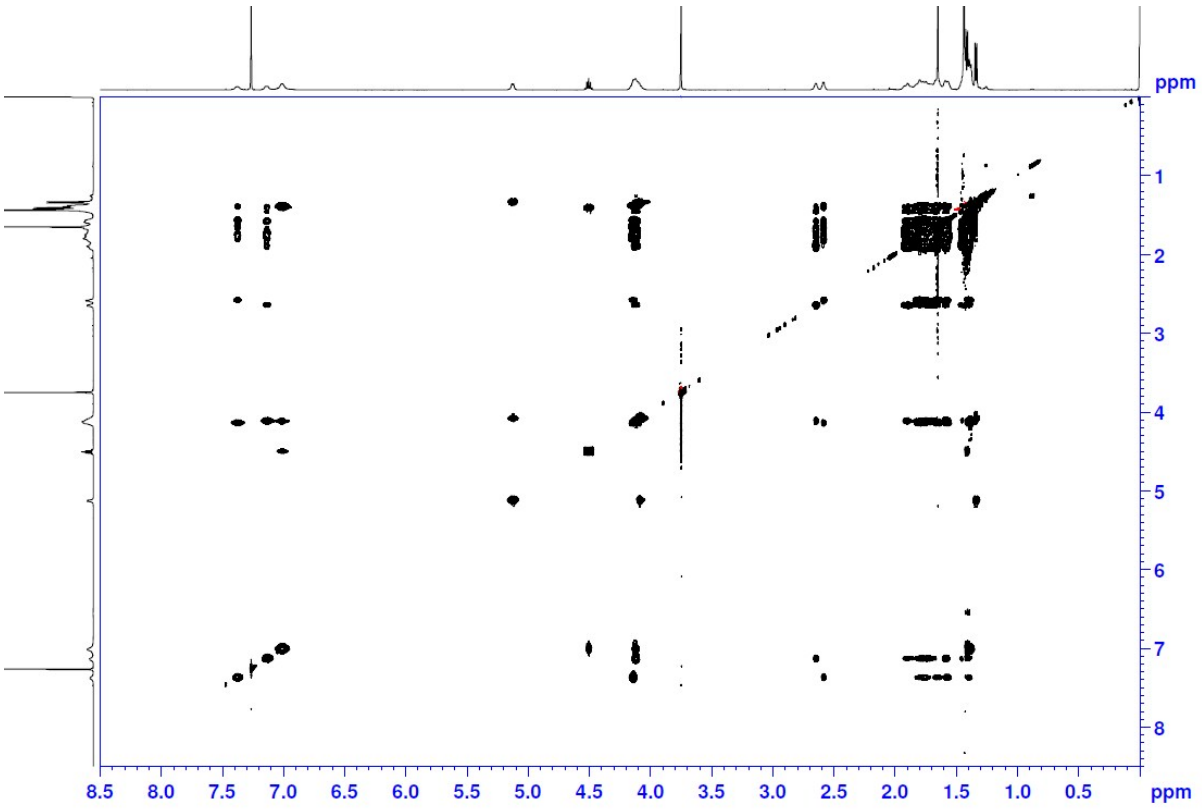

Figure S14. TOCSY spectra of **5** in $CDCl_3$.

Copies of mass spectra

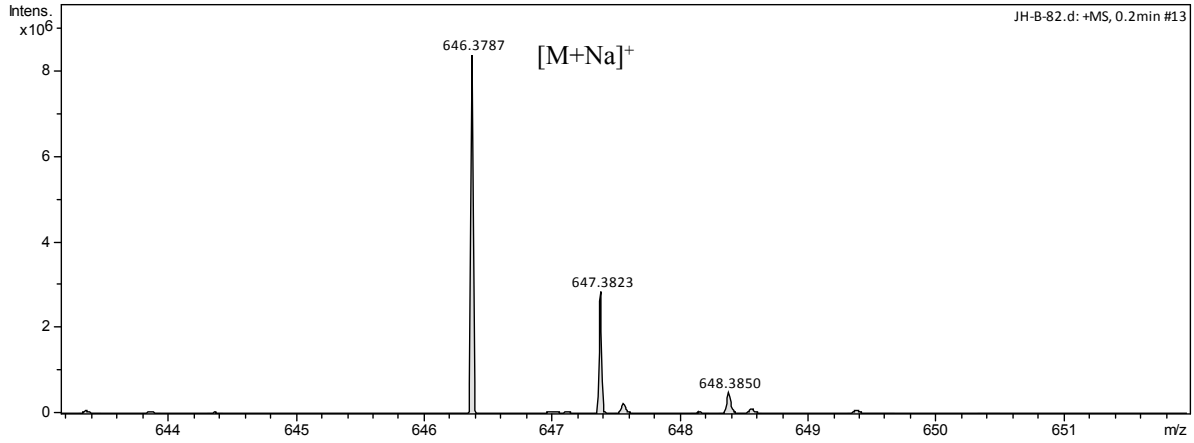

Figure S15. HRMS data for **2**.

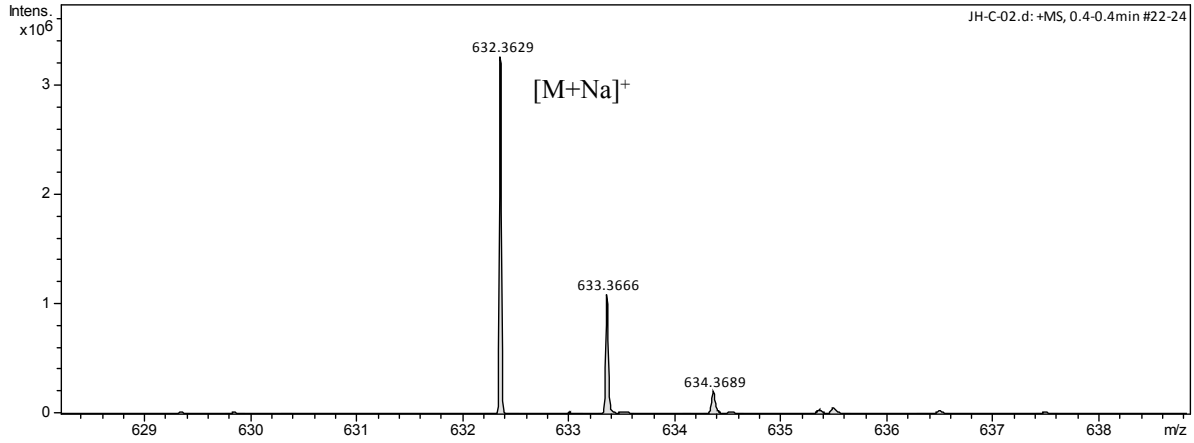

Figure S16. HRMS data for **3**.

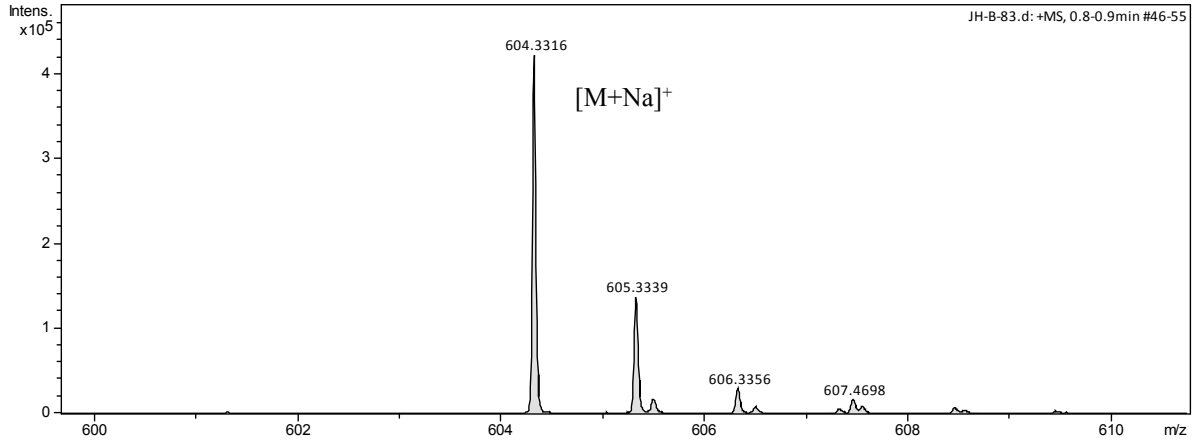

Figure S17. HRMS data for **4**

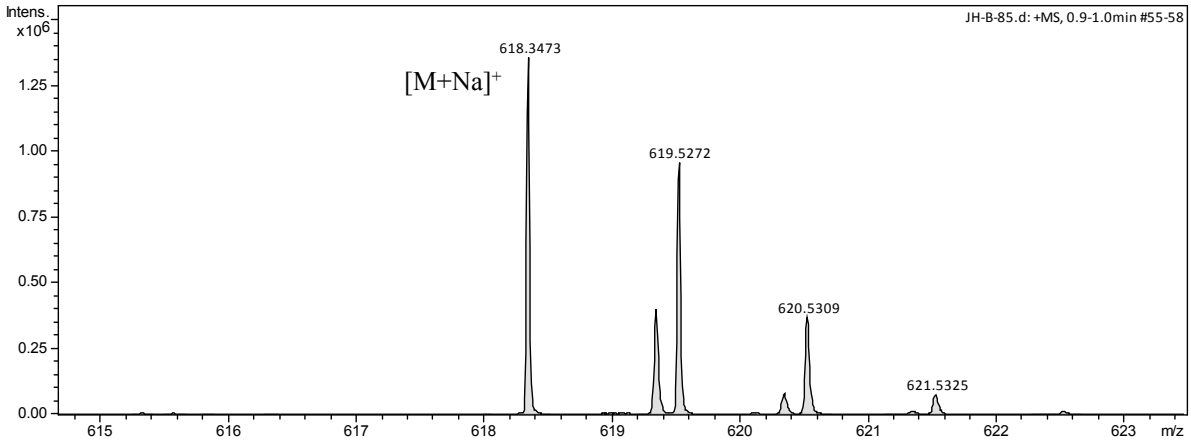

Figure S18. HRMS data for **5**.

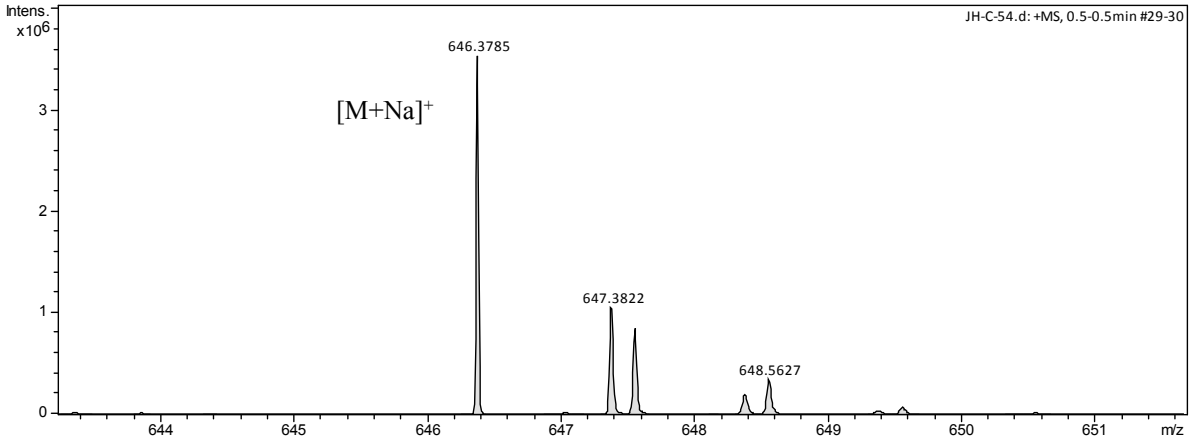

Figure S19. HRMS data for **6**.

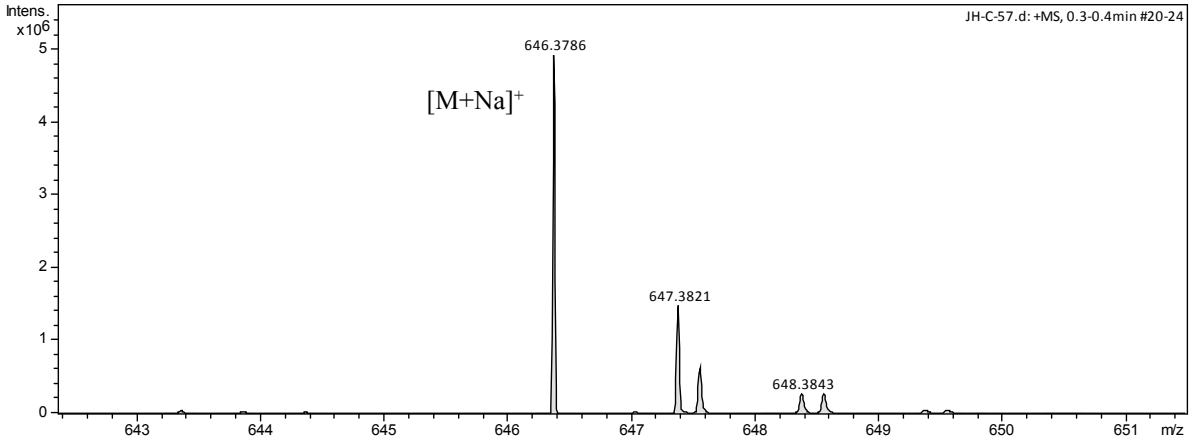

Figure S20. HRMS data for **7**.

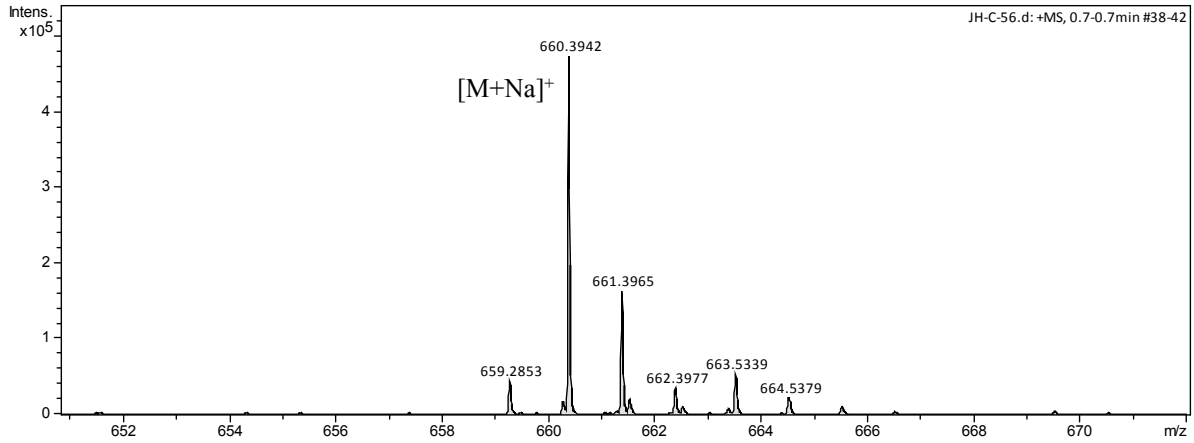

Figure S21. HRMS data for **8**.

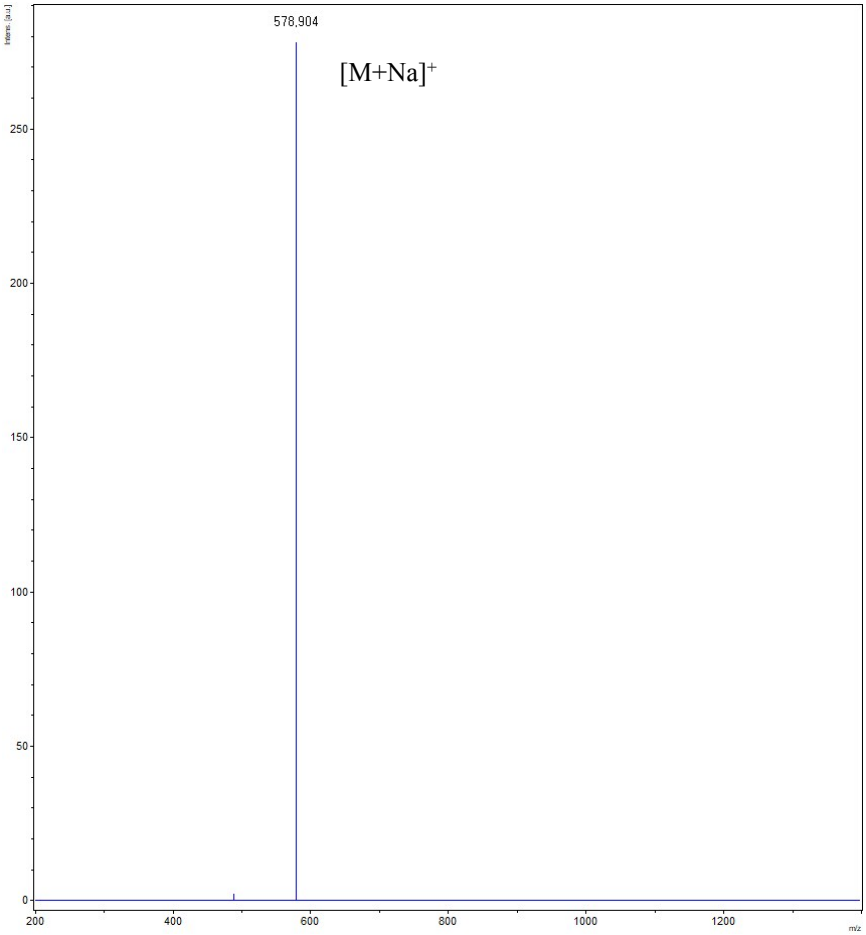

Figure S22. MALDI-TOF MS data for **9**.

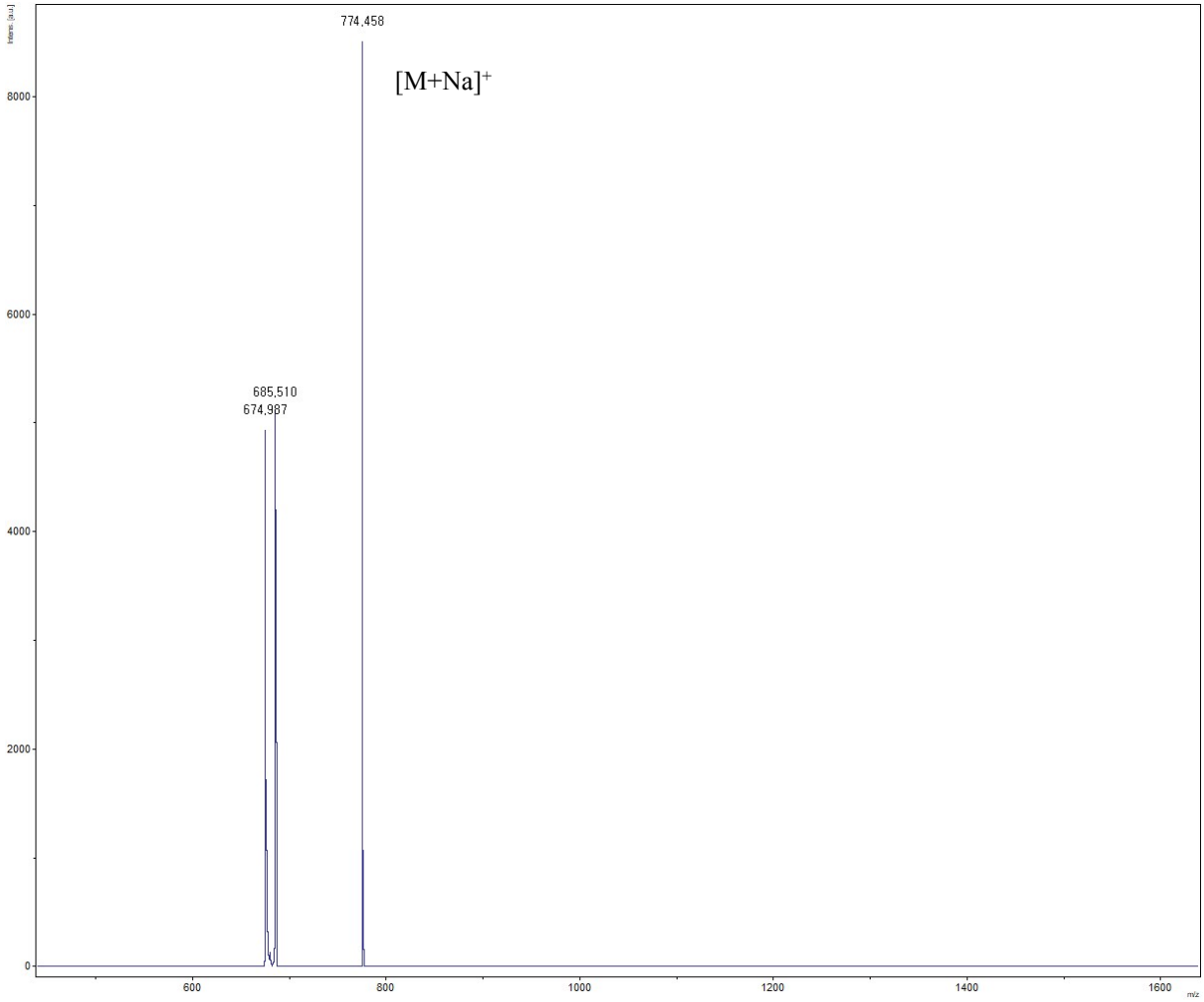

Figure S23. MALDI-TOF MS data for **10**.

Crystallographic structure report

• **Boc-Ala-cis-ACHC-Val-cis-ACHC-Ala-OMe (2)**

Data Collection

A colorless crystal with approximate dimensions 0.24 × 0.21 × 0.11 mm^3 was selected under oil under ambient conditions and attached to the tip of a MiTeGen MicroMount©. The crystal was mounted in a stream of cold nitrogen at 220 K and centered in the X-ray beam by using a video camera.

The crystal evaluation and data collection were performed on a Bruker D8 Venture diffractometer with Mo K_α (λ = 0.71073 Å) radiation and the diffractometer to crystal distance of 4.00 cm.

The initial cell constants were obtained from two series of ω scans at different starting angles. Each series consisted of 12 frames collected at intervals of 0.5º in a 6º range about ω with the exposure time of 20 seconds per frame. The reflections were successfully indexed by an automated indexing routine built in the APEXII program. The final cell constants were calculated from a set of 9936 strong reflections from the actual data collection.

The data were collected by using the half sphere data collection routine to survey the reciprocal space to the extent of a half sphere to a resolution of 0.81 Å. A total of 39981 data were harvested by collecting 6 sets of frames with 0.5º scans in ω and φ with an exposure time 20 sec per frame. These highly redundant datasets were corrected for Lorentz and polarization effects. The absorption correction was based on fitting a function to the empirical transmission surface as sampled by multiple equivalent measurements. [S2]

Structure Solution and Refinement

The systematic absences in the diffraction data were uniquely consistent for the space group $P2_12_12_1$ that yielded chemically reasonable and computationally stable results of refinement [S3-4].

A successful solution by the direct methods provided most non-hydrogen atoms from the *E*-map. The solvent molecules, chloroform, were found to be disordered, and were modeled in two different orientations. The remaining non-hydrogen atoms were located in an alternating series of least-squares cycles and difference Fourier maps. All non-hydrogen atoms were refined with anisotropic displacement coefficients. All hydrogen atoms were included in the structure factor calculation at idealized positions and were allowed to ride on the neighboring atoms with relative isotropic displacement coefficients.

The final least-squares refinement of 505 parameters against 8711 data resulted in residuals *R* (based on F^2 for $I \geq 2\sigma$) and *wR* (based on F^2 for all data) of 0.0772 and 0.2342, respectively. The final difference Fourier map was featureless.

Summary

Crystal Data for $C_{33}H_{55}Cl_6N_5O_8$ (M =862.52): orthorhombic, space group $P2_12_12_1$ (no. 19), a = 10.1637(4) Å, b = 17.2472(5) Å, c = 25.5023(9) Å, V = 4470.4(3) $Å^3$, Z = 4, T = 219.9 K, μ(MoKα) = 0.433 mm^{-1}, *Dcalc* = 1.282 g/mm^3, 39981 reflections measured ($4.65 \leq 2\Theta \leq 51.94$), 8711 unique ($R_{int}$ = 0.0389, R_{sigma} = 0.0327) which were used in all calculations. The final R_1 was 0.0772 ($I > 2\sigma(I)$) and wR_2 was 0.2342 (all data).

Table 1. Crystal data and structure refinement for **2.**

Empirical formula	$C_{33}H_{55}Cl_6N_5O_8$
Formula weight	862.52
Temperature/K	219.9
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	10.1637(4)
b/Å	17.2472(5)
c/Å	25.5023(9)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	4470.4(3)
Z	4
ρ_{calc}mg/mm^3	1.282
m/mm^{-1}	0.433
F(000)	1816.0
Crystal size/mm^3	0.24 × 0.21 × 0.11
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection	4.652 to 51.942°
Index ranges	$-12 \le h \le 12$, $-21 \le k \le 20$, $-31 \le l \le 31$
Reflections collected	39981
Independent reflections	8711 [R_{int} = 0.0389, R_{sigma} = 0.0327]
Data/restraints/parameters	8711/0/505
Goodness-of-fit on F^2	1.030
Final R indexes [I>=2σ (I)]	R_1 = 0.0772, wR_2 = 0.2108
Final R indexes [all data]	R_1 = 0.1037, wR_2 = 0.2342
Largest diff. peak/hole / e Å^{-3}	0.63/-0.53
Flack parameter	0.003(19)

References

[S1] M. Lee, J. Shim, P. Kang, M.-G. Choi and S. H. Choi, *Chem. Commun.*, 2016, **52**, 5950-5952

[S2] Bruker-AXS. (2007-2013) APEX2 (Ver. 2013.2-0), SADABS (2012-1), and SAINT+ (Ver. 8.30C) Software Reference Manuals. Bruker-AXS, Madison, Wisconsin, USA.

[S3] G. M. Sheldrick, (2008) SHELXL. *Acta Cryst.* **A64**, 112-122.

[S4] O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard, H. Puschmann, "OLEX2: a complete structure solution, refinement and analysis program". *J. Appl. Cryst.* (2009) **42**, *339-341*.