Supplementary information for

Naphthalene Bis-carboxamide Amino Acid Bioconjugates as Super Organogelators

Zoran Kokan, Tamara Miljuš, Janja Makarević,†[1] Goran Štefanić, Tihomir Pospišil, Leo Frkanec, Berislav Perić,* and Srećko I. Kirin*

Correspondence: bperic@irb.hr, skirin@irb.hr

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†In memoriam, 02.12.2020.

1. FTIR

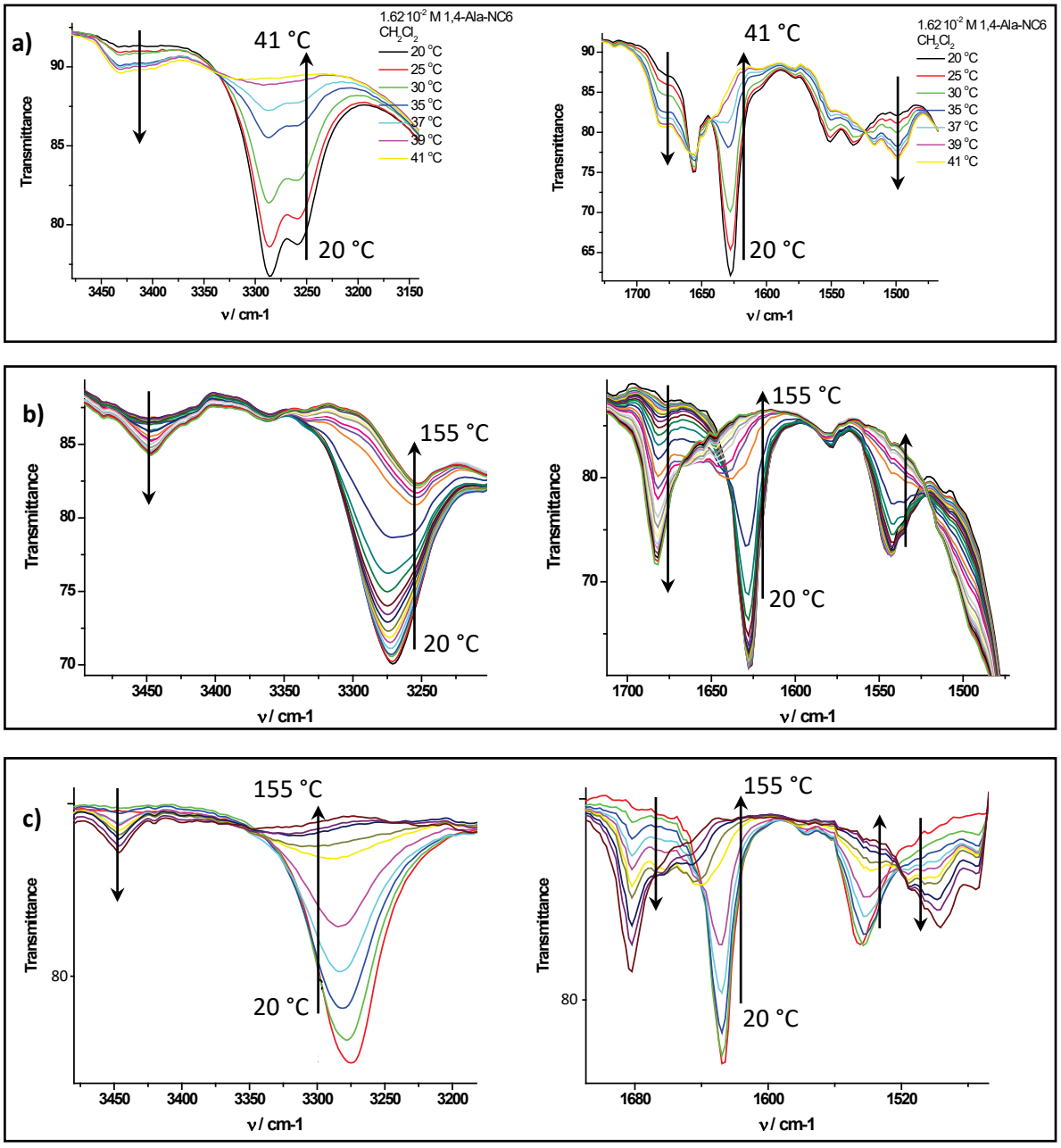


Figure S 1. Variable temperature FTIR spectra of a) **4b** CH_2Cl_2 gel; b) **6** decalin gel; c) **4b** decalin gel. Arrows indicate changes with increasing temperature.

2. UV-Vis

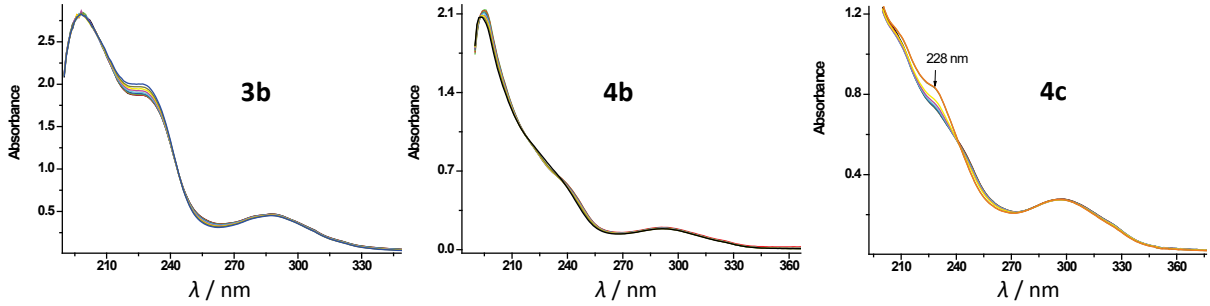


Figure S 2. Variable temperature UV-Vis spectra of decalin gels (20–100 °C).

3. CD/LD

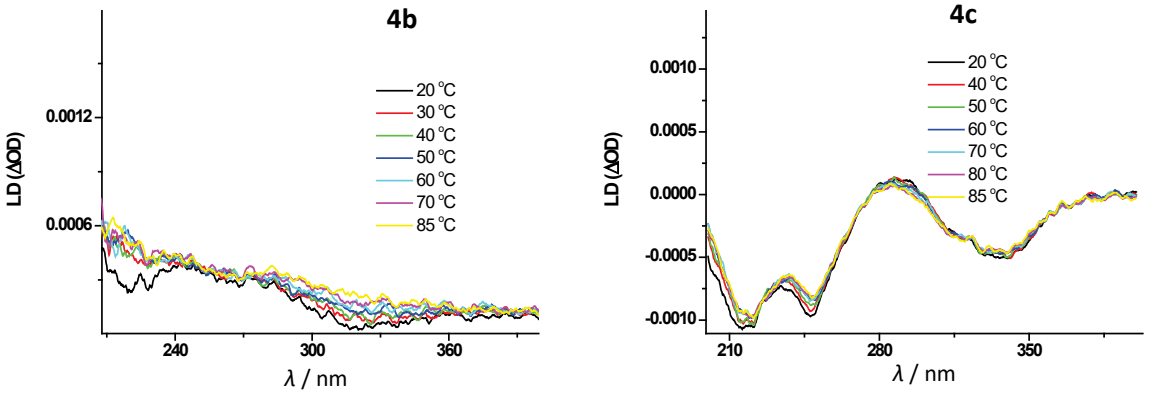


Figure S 3. Variable temperature LD spectra of **4b** and **4c** decalin gels (20–85 °C).

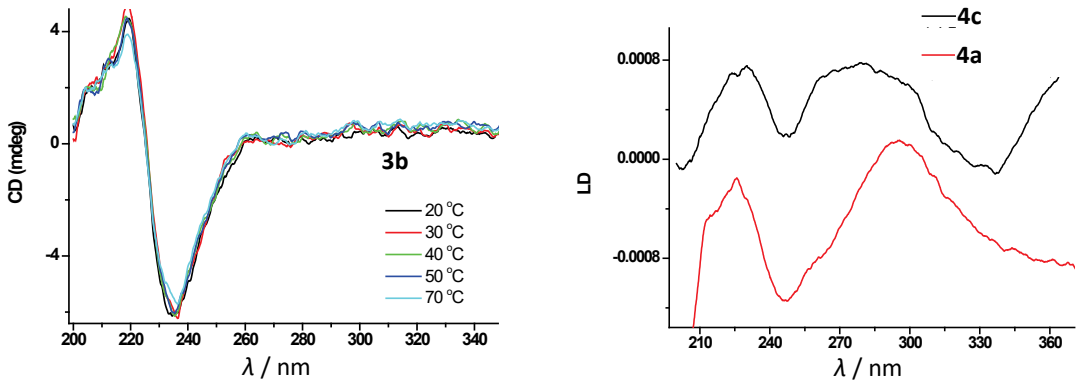


Figure S 4. Temperature dependent CD of **3b** decalin gel and LD spectra of **4b** and **4c** decalin gels.

4. ^{1}H NMR

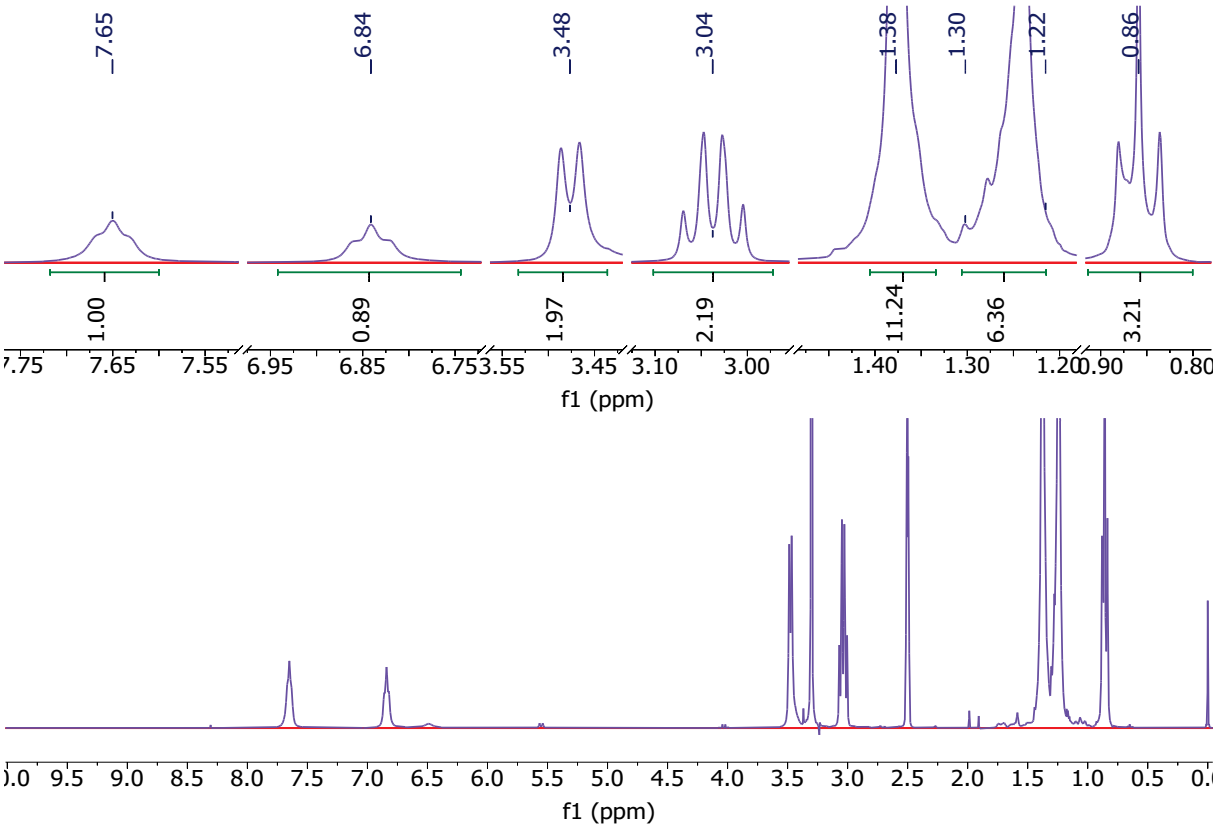


Figure S 5. ^{1}H NMR (300 MHz, DMSO-d_6) spectrum (full and intercepts) of Boc-Gly-nC$_6$H$_{13}$.

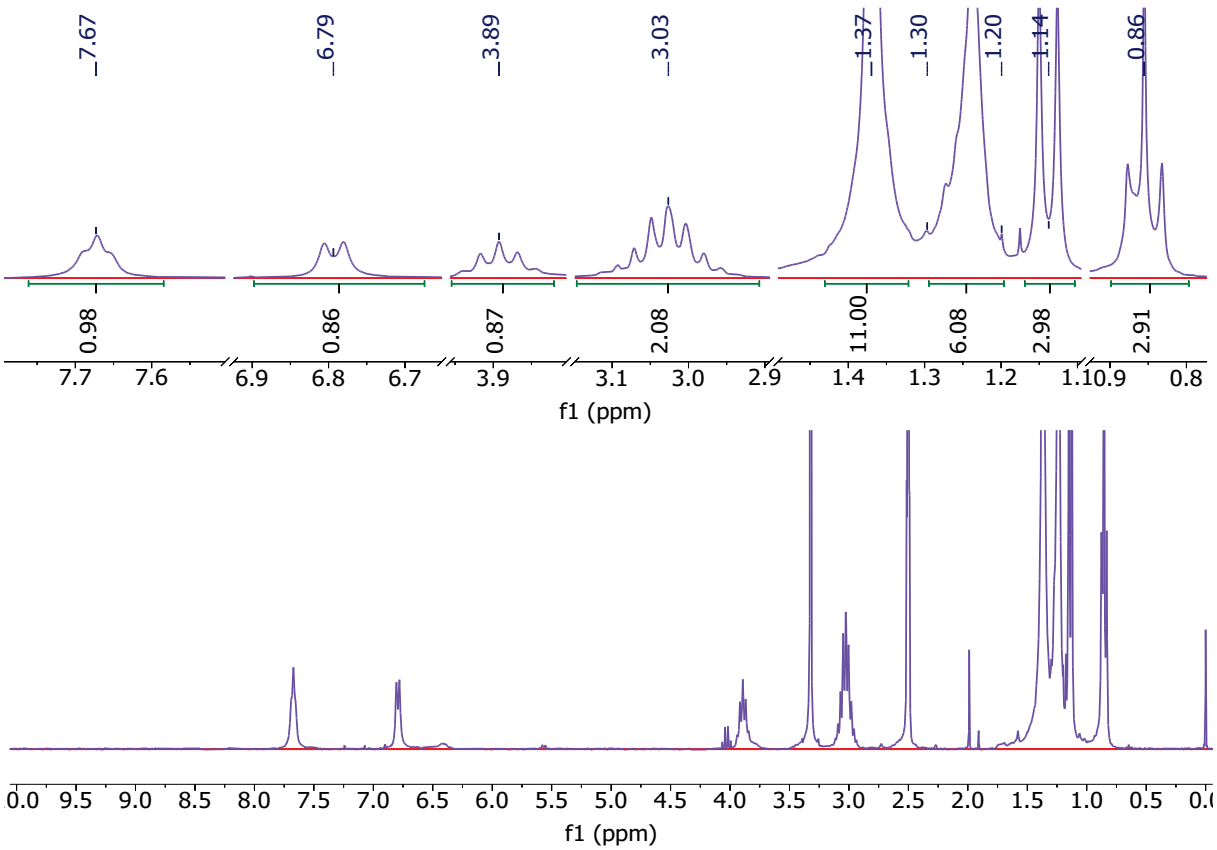


Figure S 6. ^{1}H NMR (300 MHz, DMSO-d_6) spectrum (full and intercepts) of Boc-Ala-nC$_6$H$_{13}$.

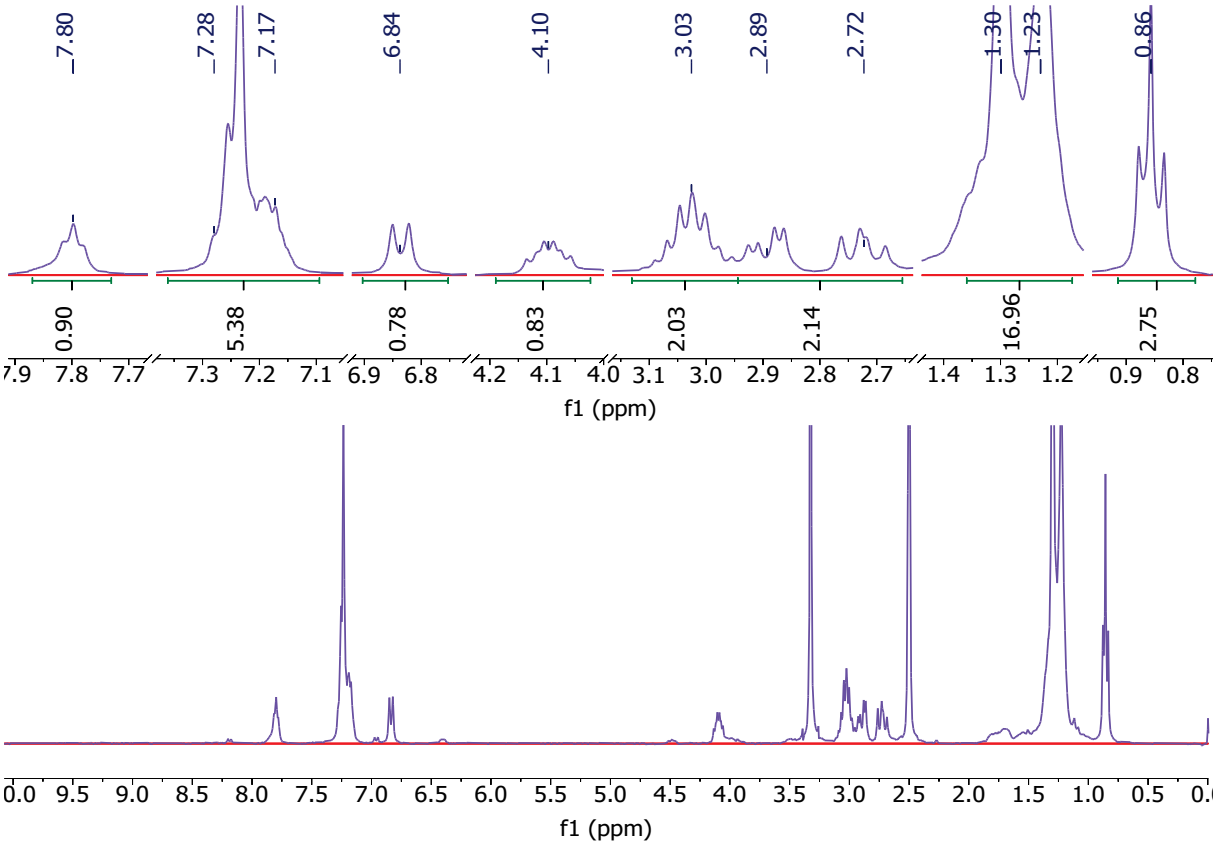


Figure S 7. ^{1}H NMR (300 MHz, DMSO-d_6) spectrum (full and intercepts) of Boc-Phe-*n*C_6H_{13}.

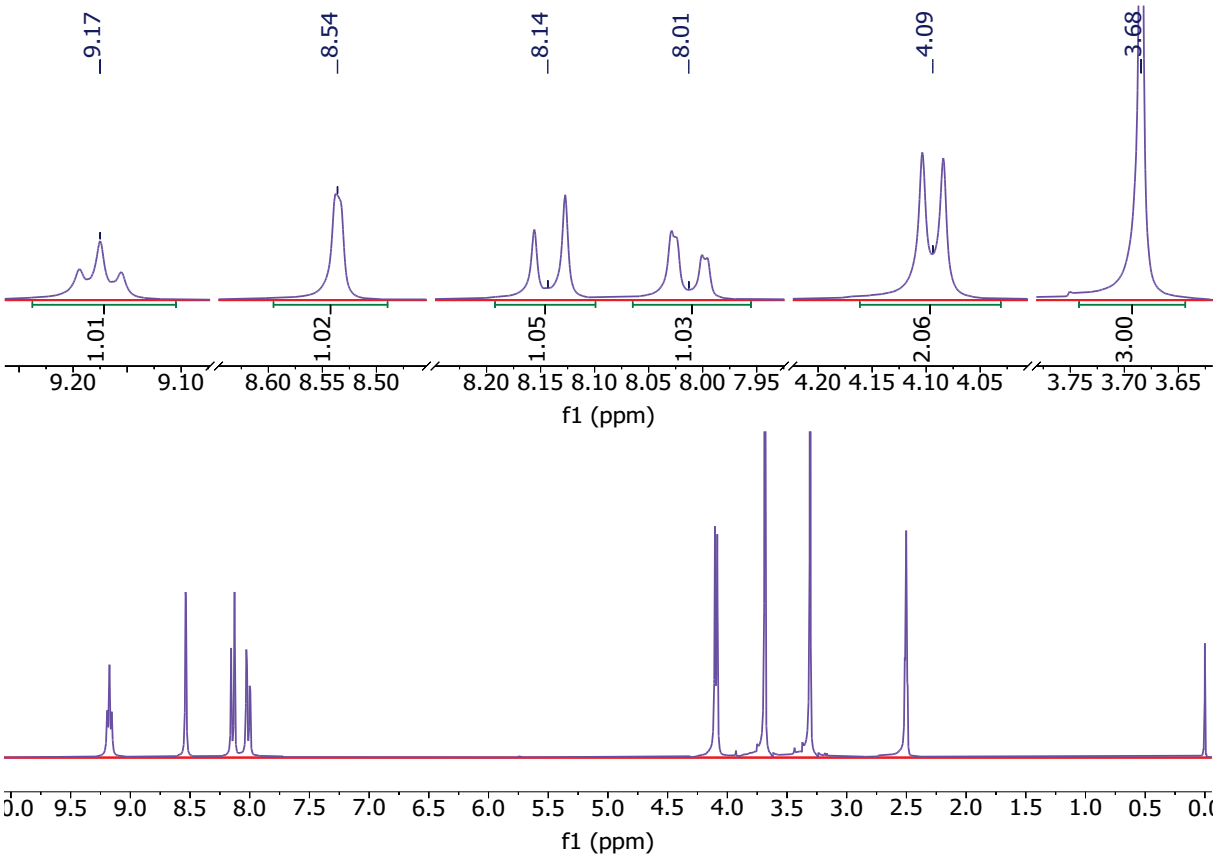


Figure S 8. ^{1}H NMR (DMSO-d_6) spectrum (full and intercepts) of **1**.

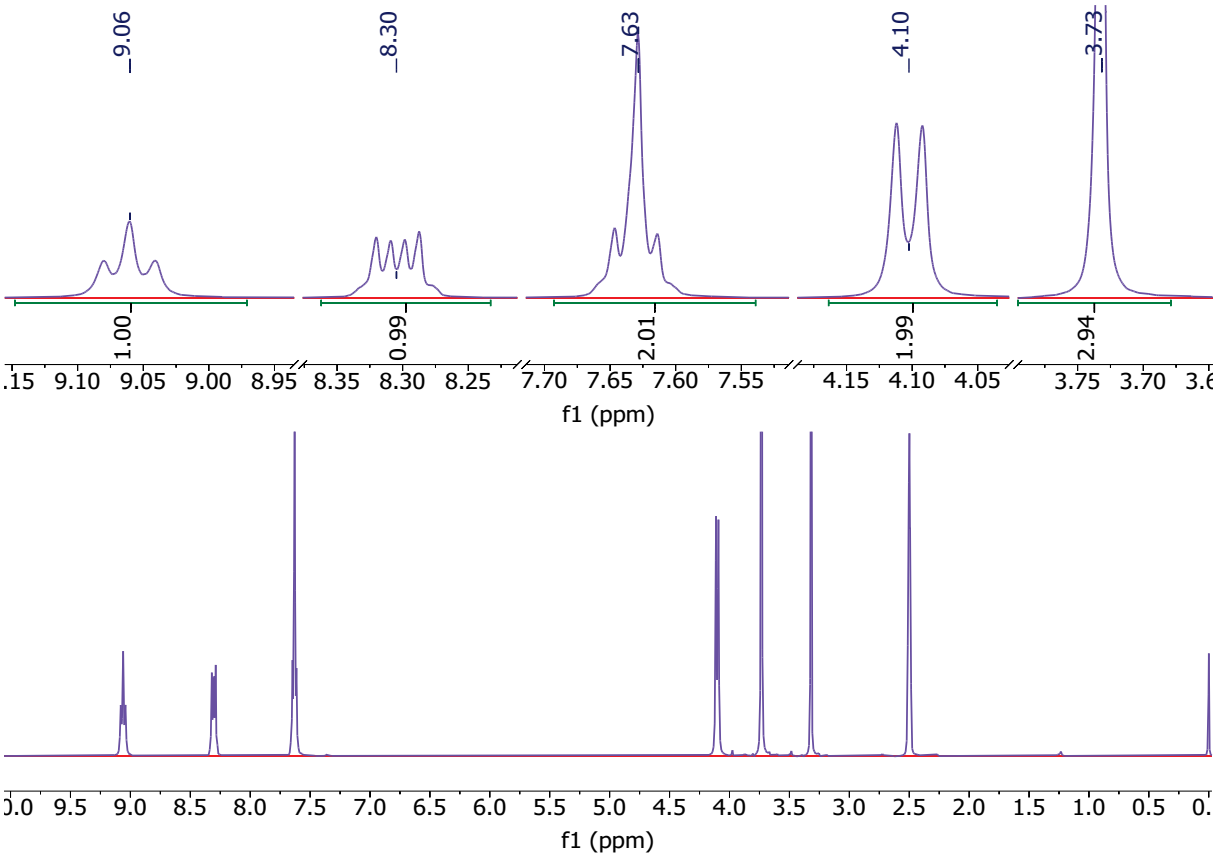


Figure S 9. ^{1}H NMR (DMSO-d_6) spectrum (full and intercepts) of **2**.

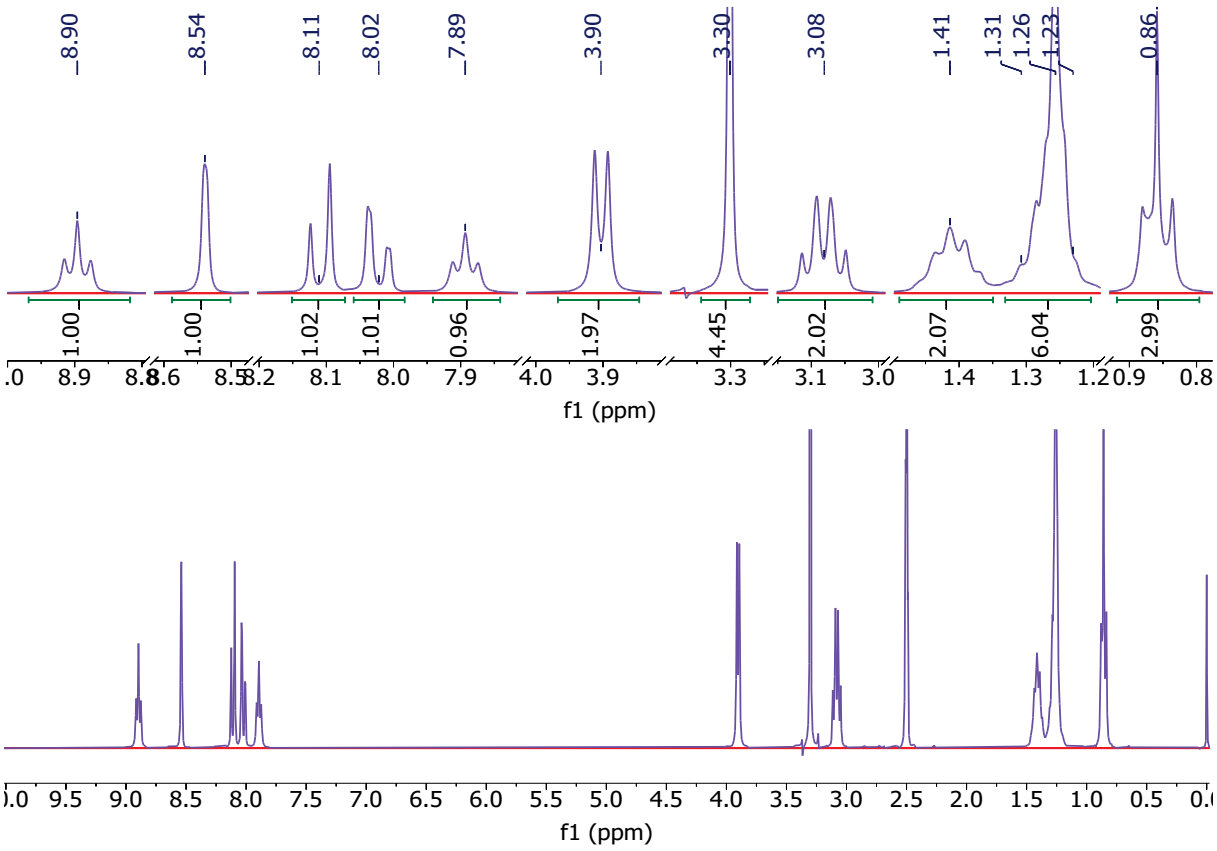


Figure S 10. ^{1}H NMR (DMSO-d_6) spectrum (full and intercepts) of **3a**.

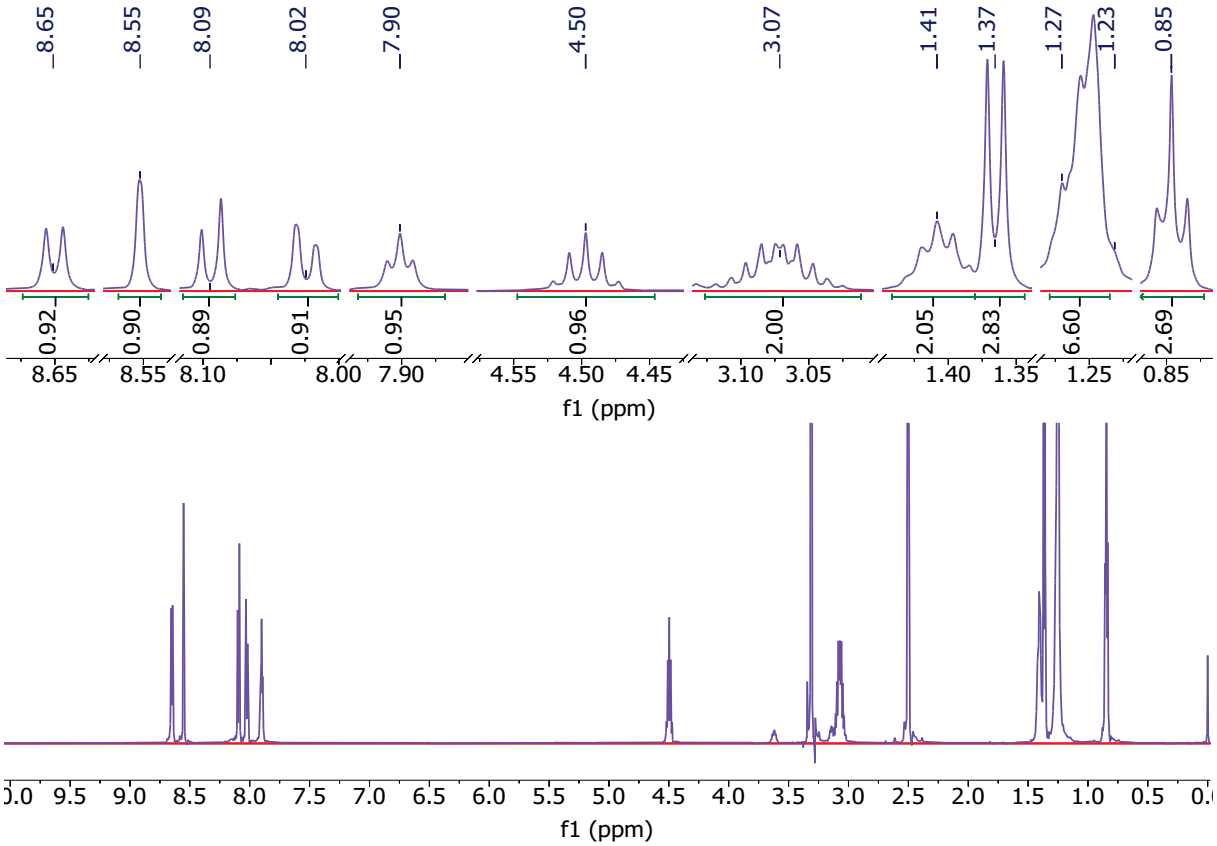


Figure S 11. ^{1}H NMR (DMSO-d_6) spectrum (full and intercepts) of **3b**.

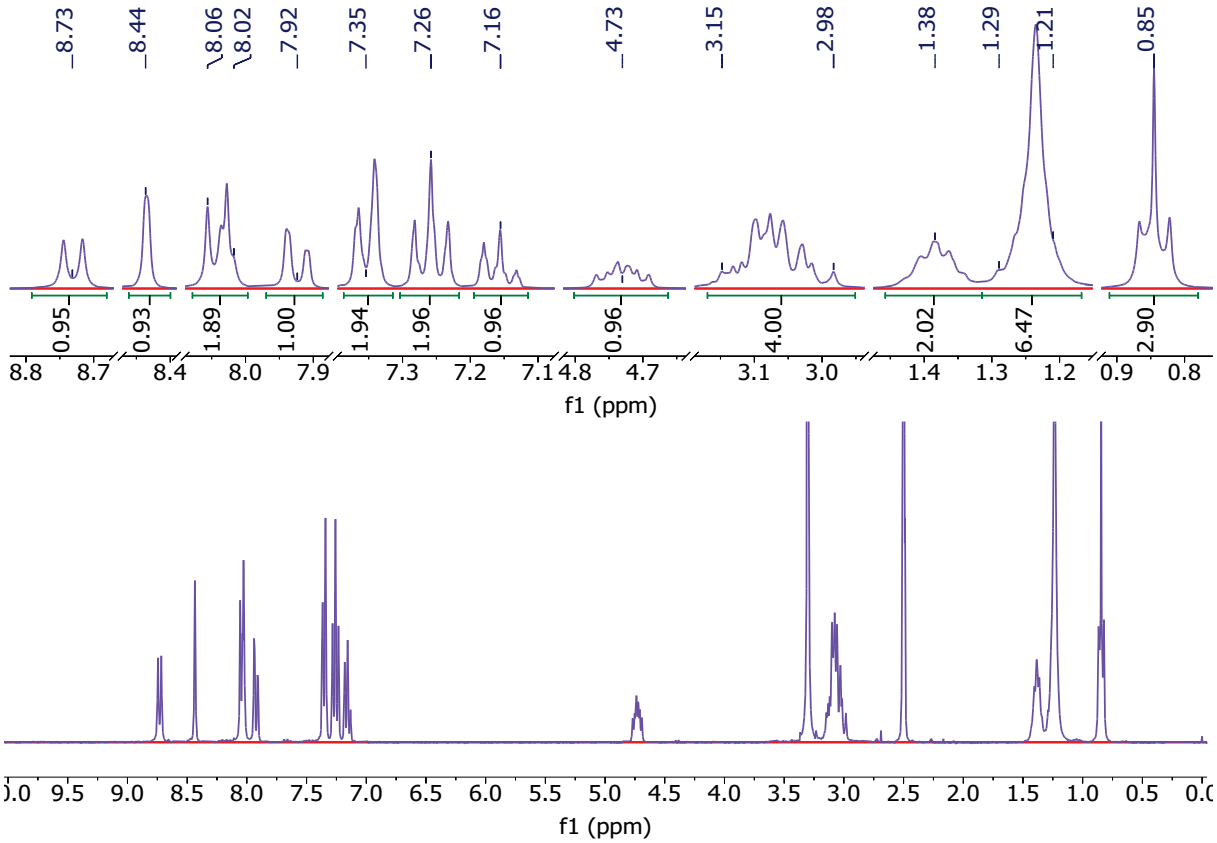


Figure S 12. ^{1}H NMR (DMSO-d_6) spectrum (full and intercepts) of **3c**.

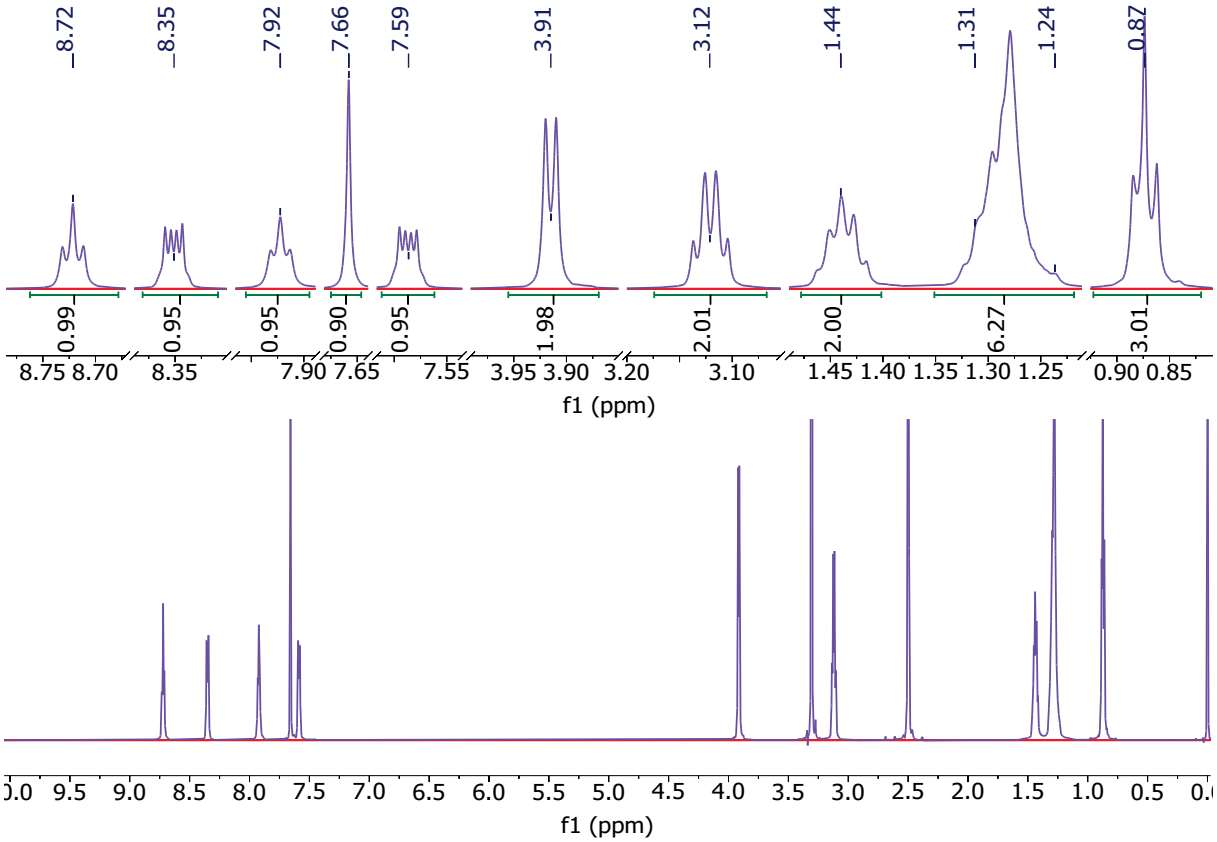


Figure S 13. ^{1}H NMR (DMSO-d_6) spectrum (full and intercepts) of **4a**.

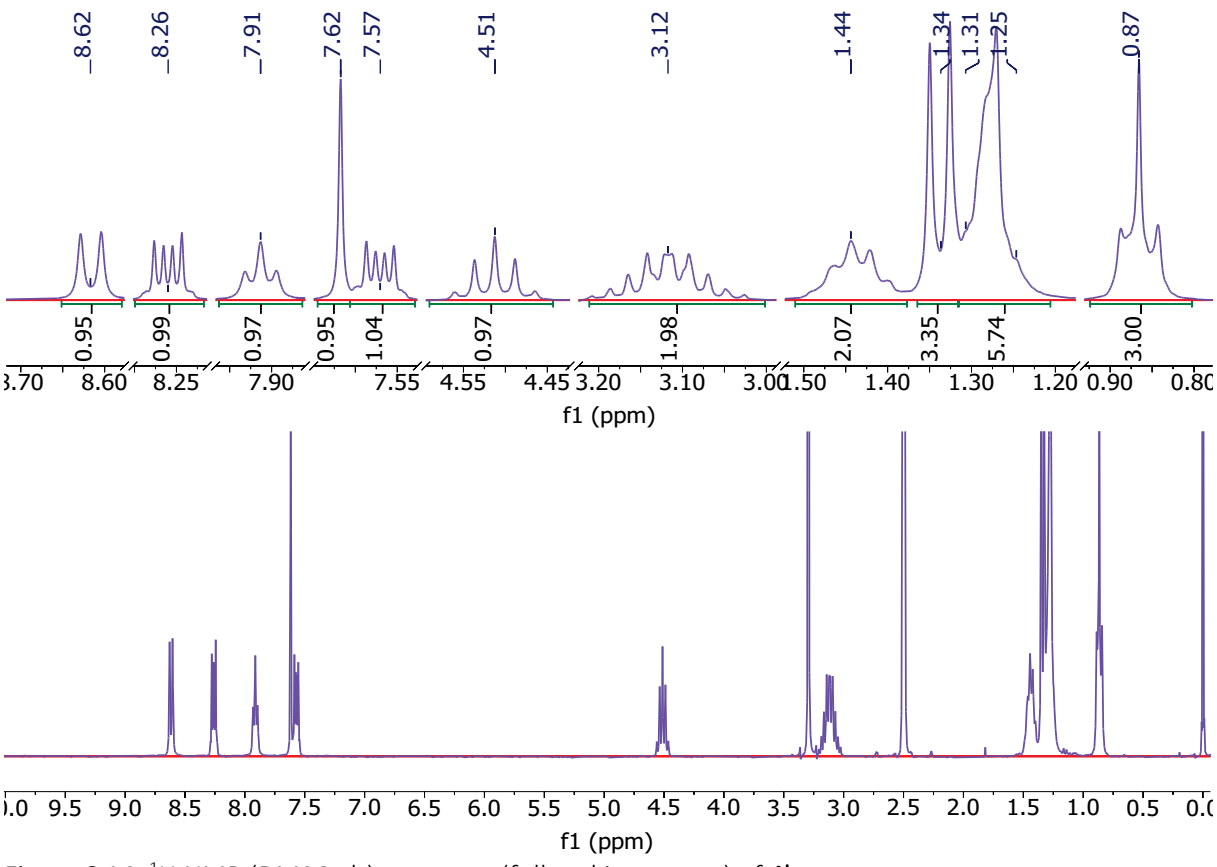


Figure S 14. ^{1}H NMR (DMSO-d_6) spectrum (full and intercepts) of **4b**.

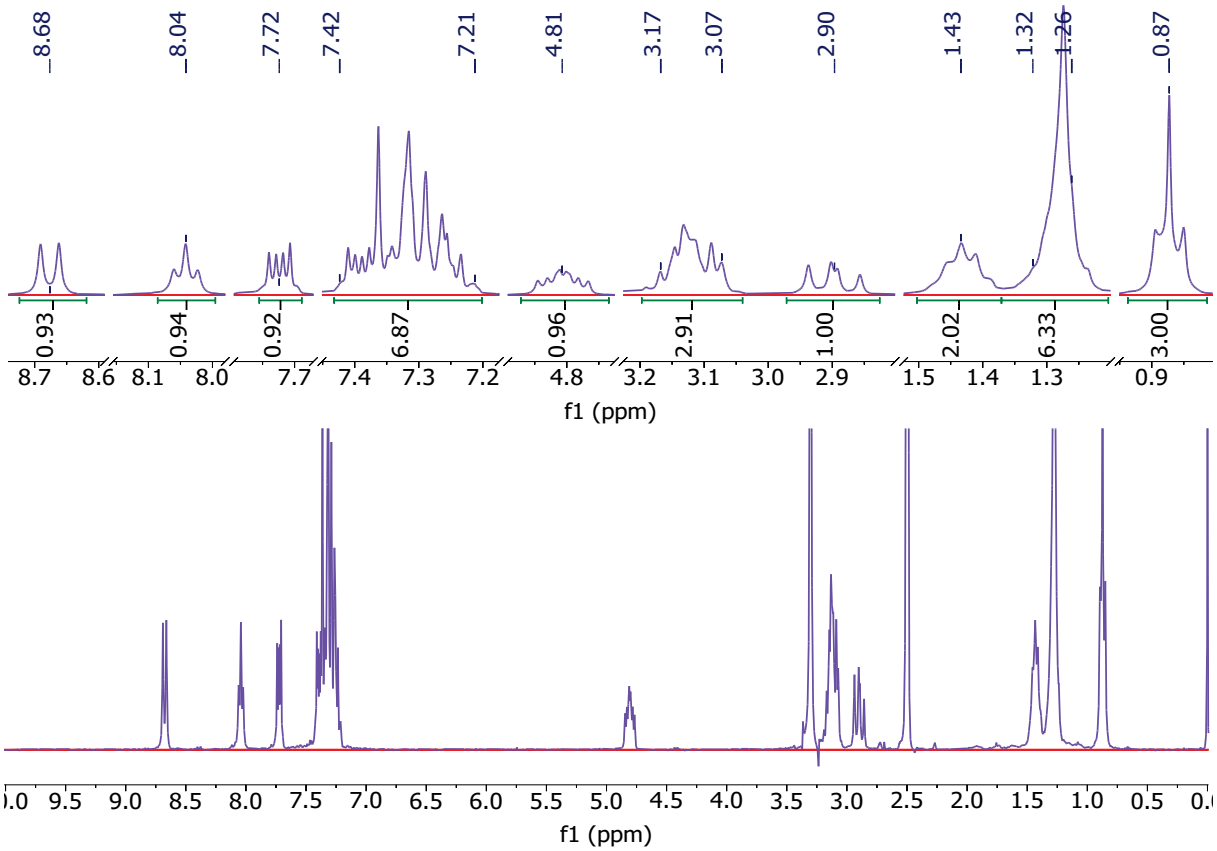


Figure S 15. ^{1}H NMR (DMSO-d_6) spectrum (full and intercepts) of **4c**.

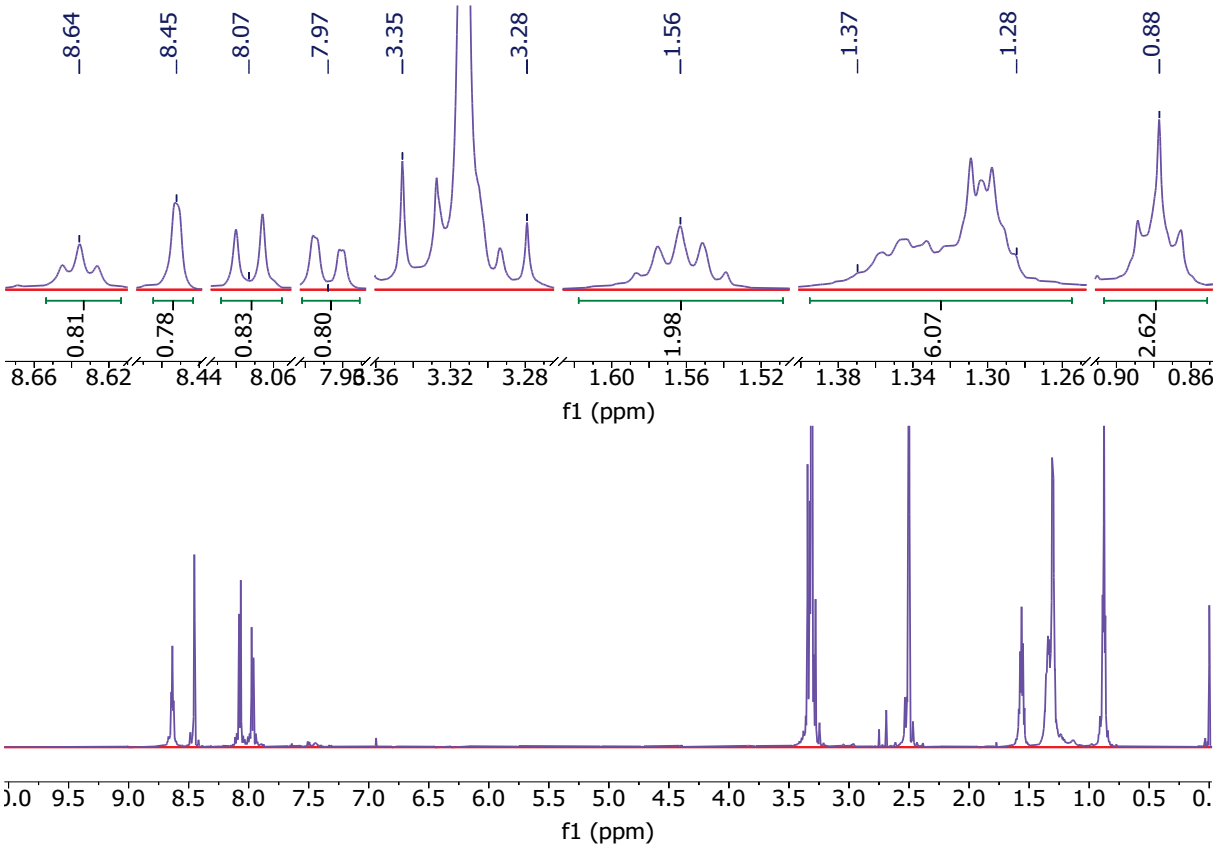


Figure S 16. ^{1}H NMR (DMSO-d_6) spectrum (full and intercepts) of **5**. Multiplet at 3.35–3.28 ppm is overlapped with the water peak.

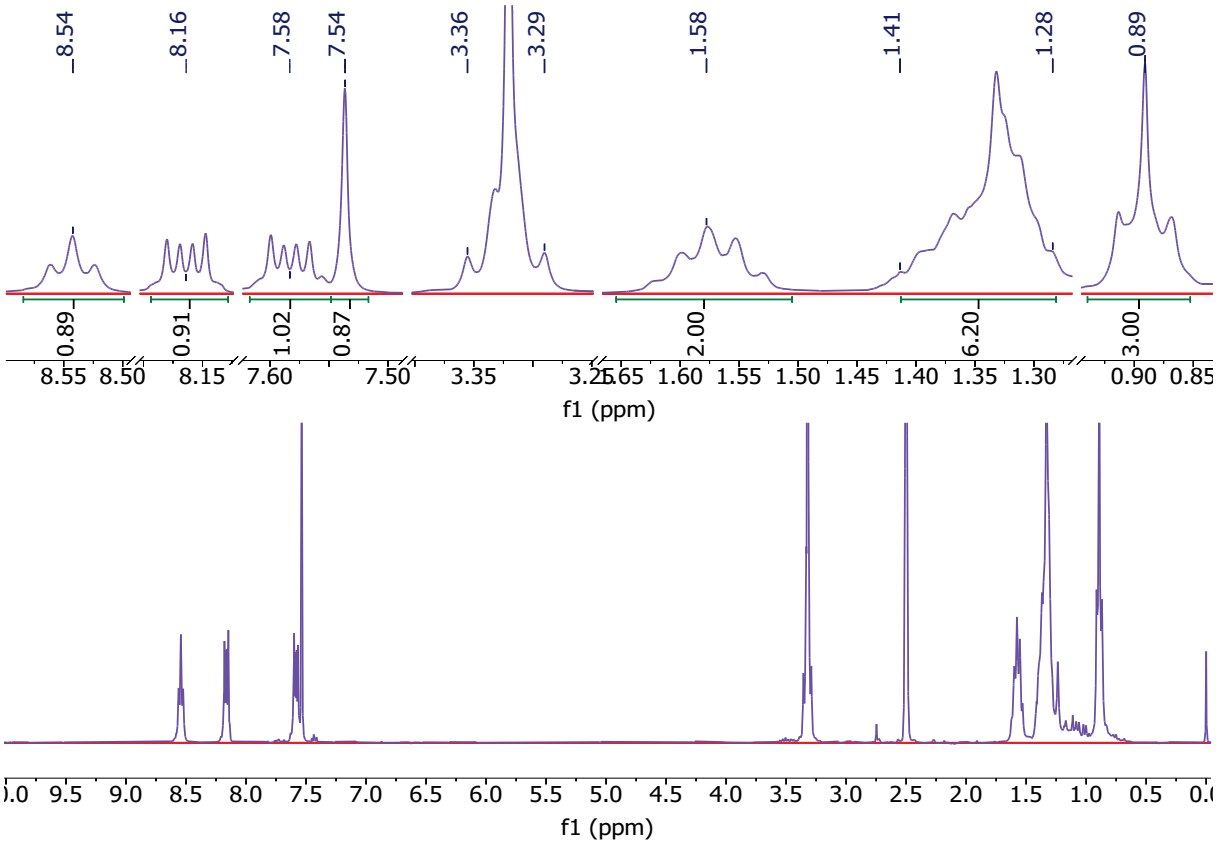


Figure S 17. ^{1}H NMR (DMSO-d_6) spectrum (full and intercepts) of **6**. Multiplet at 3.36–3.29 ppm is overlapped with the water peak.

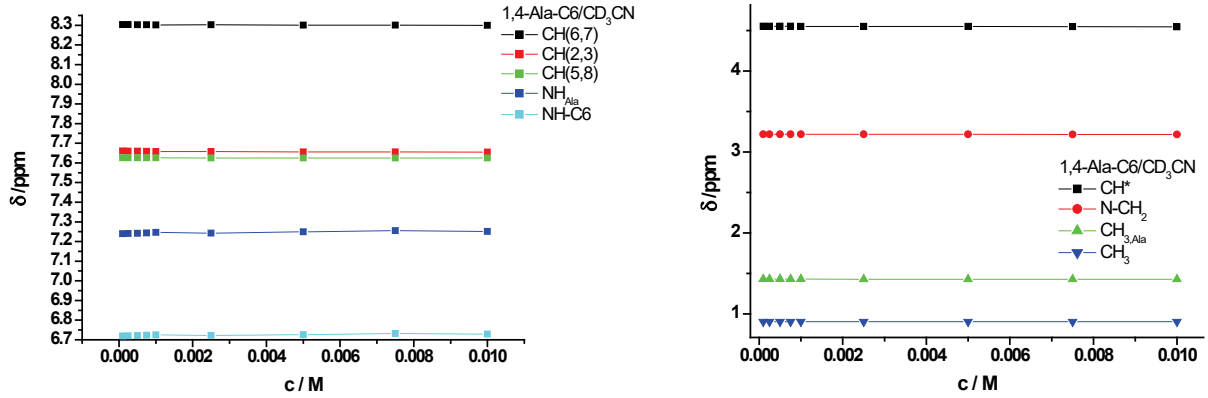


Figure S 18. Chemical shift data from variable concentration ^{1}H NMR spectra of **4b** in CD_3CN.

5. ^{13}C NMR

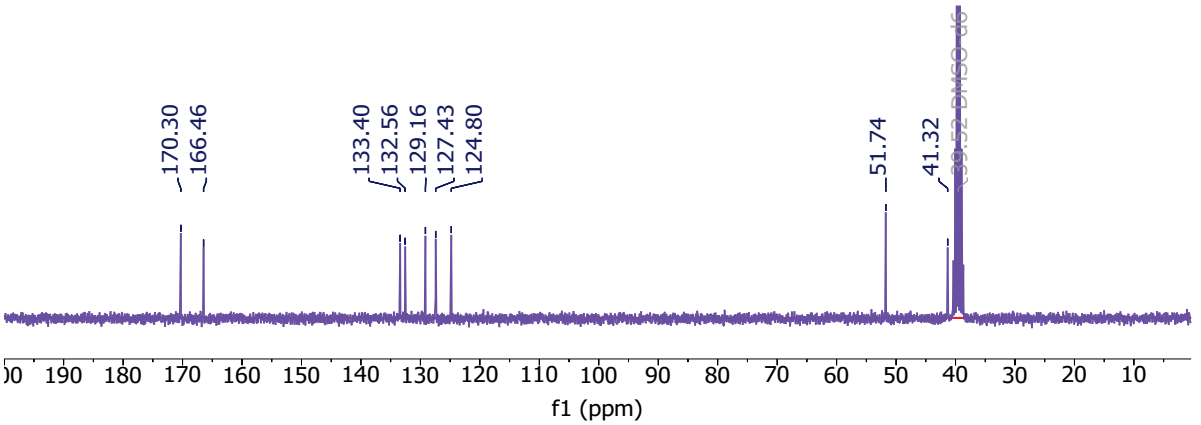


Figure S 19. ^{13}C NMR (DMSO-d_6) spectrum of **1**.

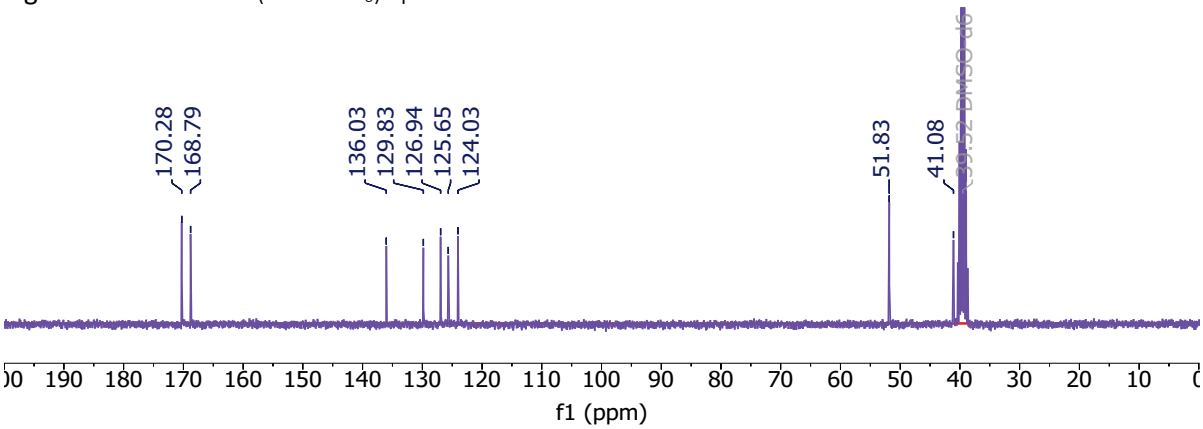


Figure S 20. ^{13}C NMR (DMSO-d_6) spectrum of **2**.

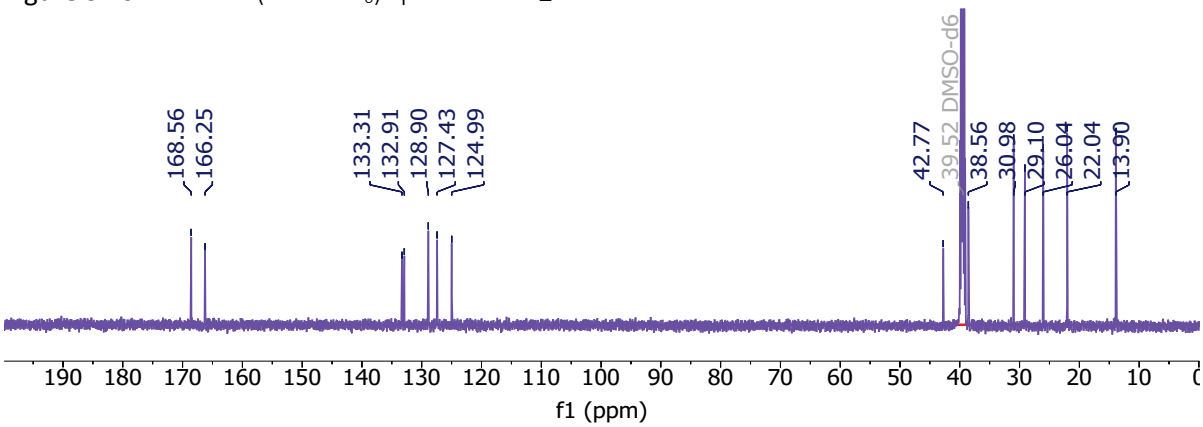


Figure S 21. ^{13}CH NMR (DMSO-d_6) spectrum of **3a**.

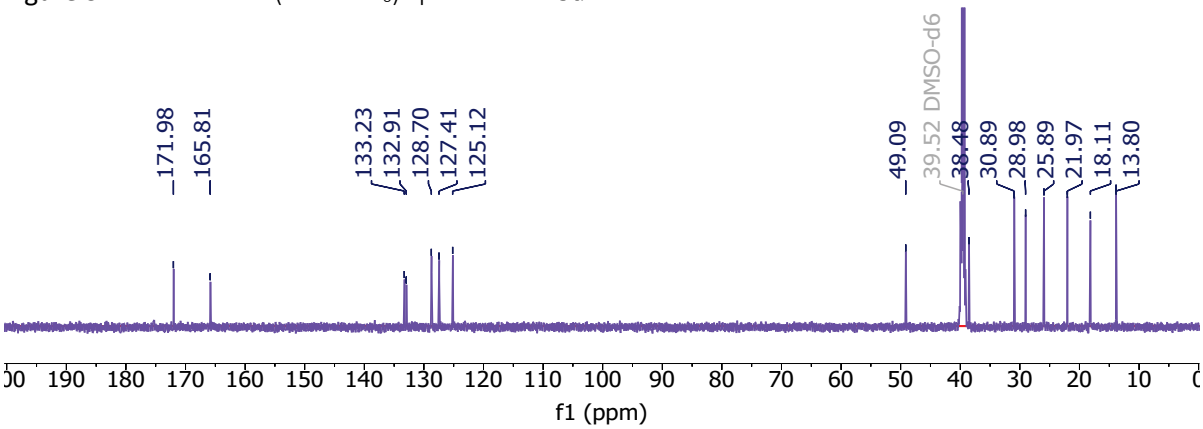


Figure S 22. ^{13}C NMR (DMSO-d_6) spectrum of **3b**.

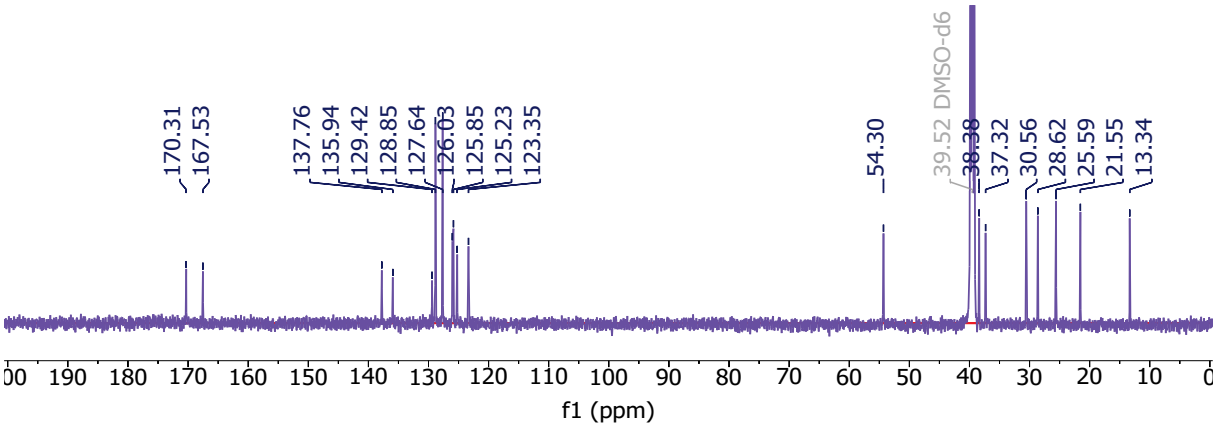


Figure S 23. ^{13}C NMR (DMSO-d_6, 70 °C) spectrum of **3c**.

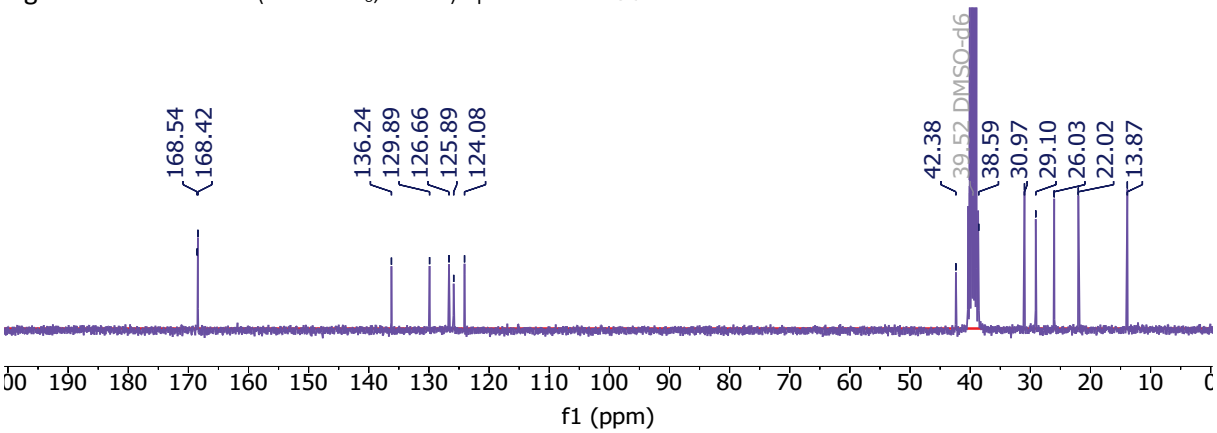


Figure S 24. ^{13}C NMR (DMSO-d_6) spectrum of **4a**.

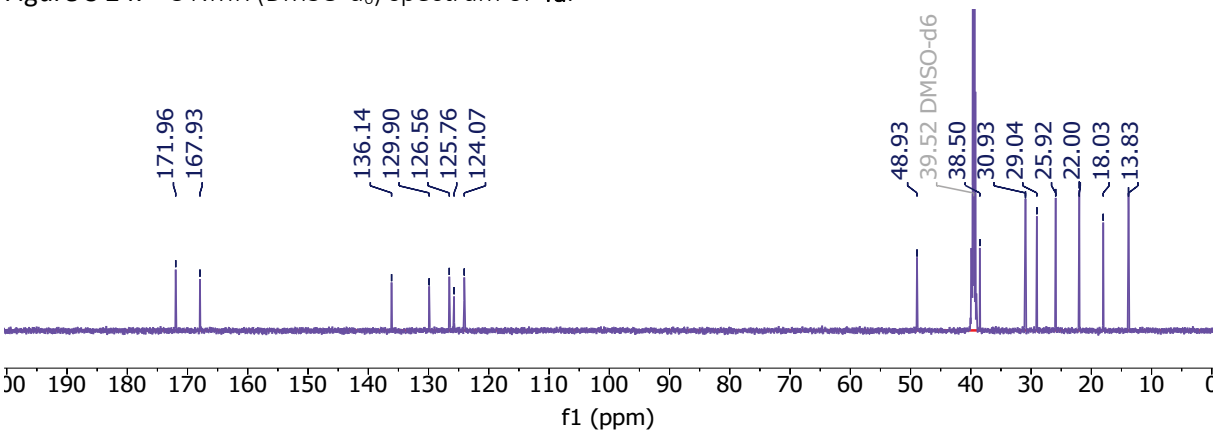


Figure S 25. ^{13}C NMR (DMSO-d_6) spectrum of **4b**

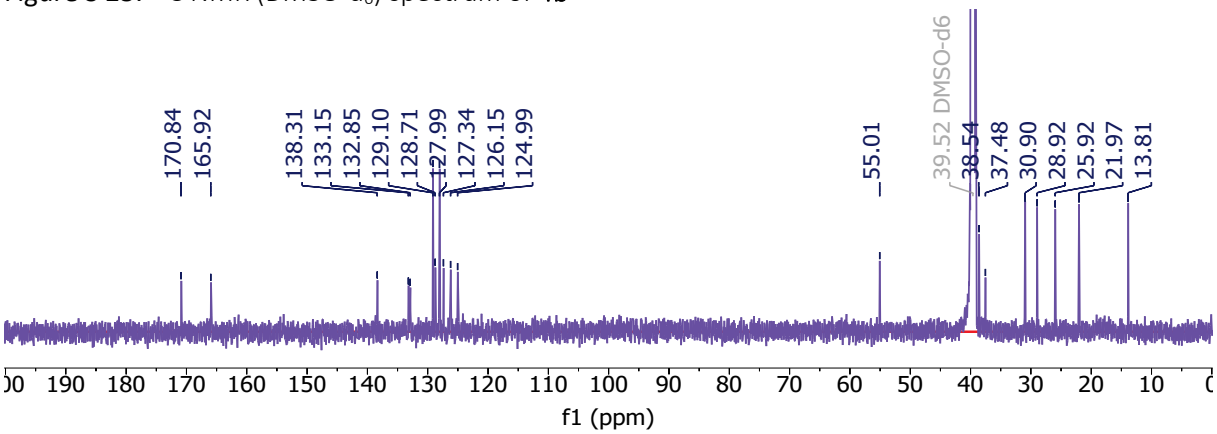


Figure S 26. ^{13}C NMR (DMSO-d_6) spectrum of **4c**.

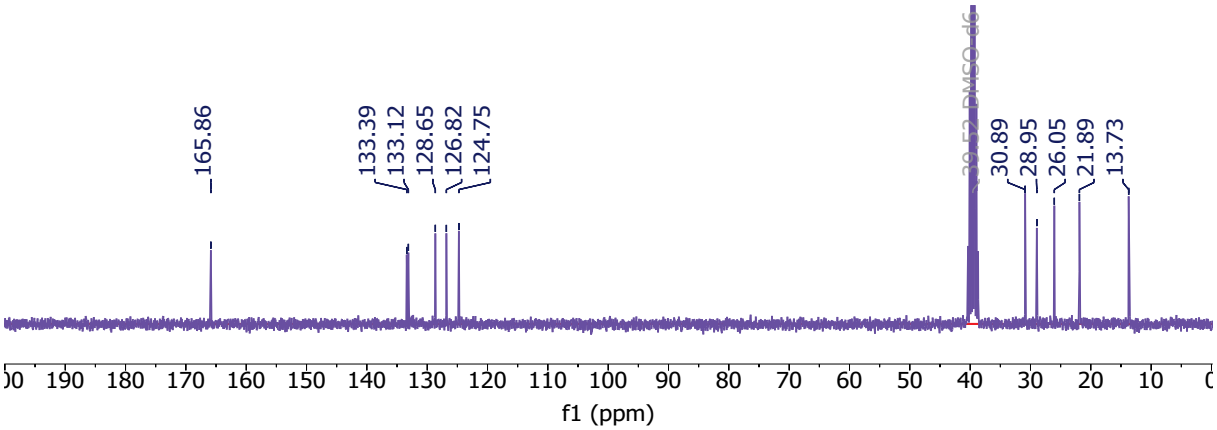


Figure S 27. ^{13}C NMR (DMSO-d_6) spectrum of **5**.

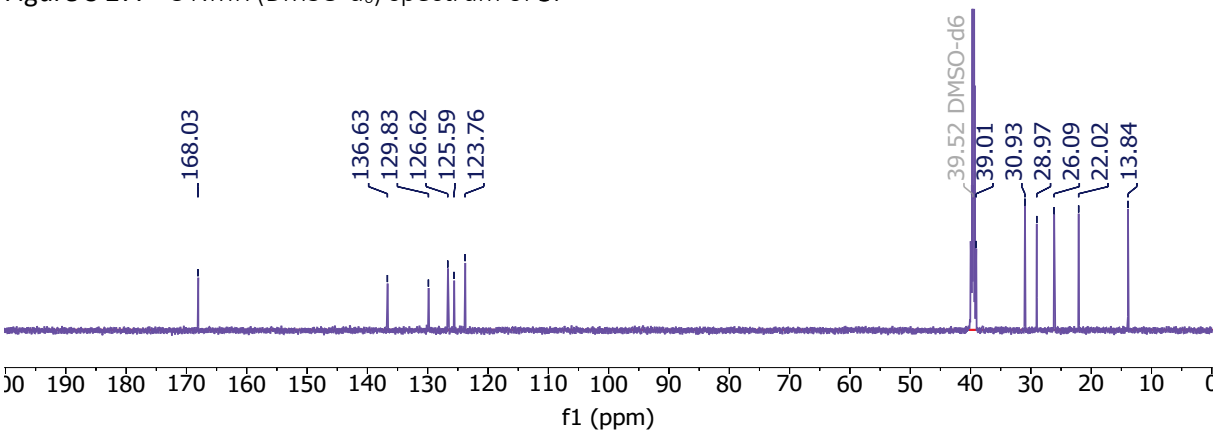


Figure S 28. ^{13}C NMR (DMSO-d_6) spectrum of **6**.

6. ESI-MS

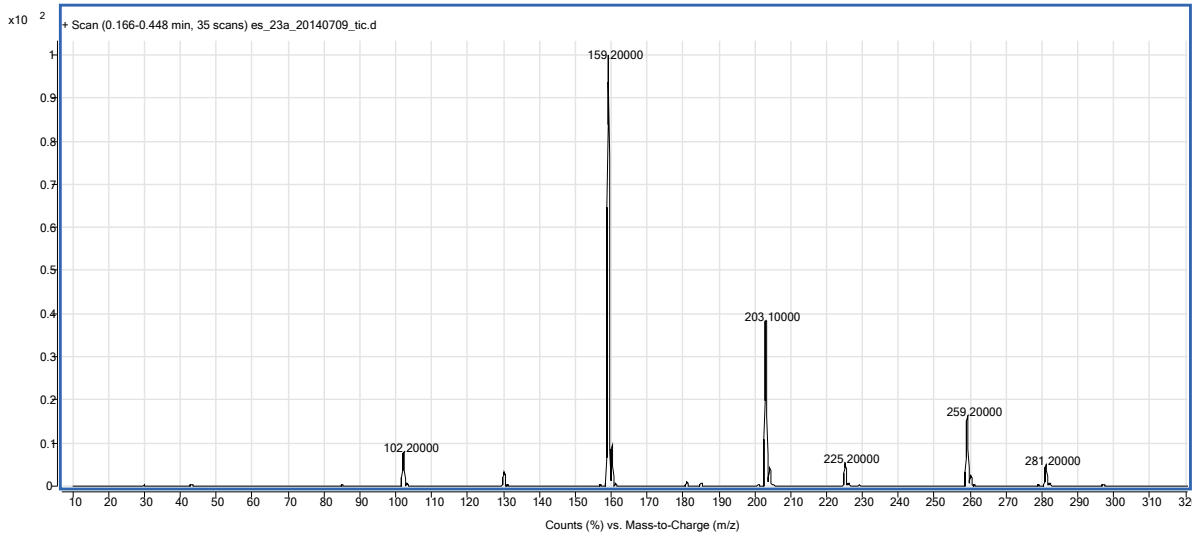


Figure S 29. +ESI-MS spectrum of Boc-Gly-nC$_6$H$_{13}$.

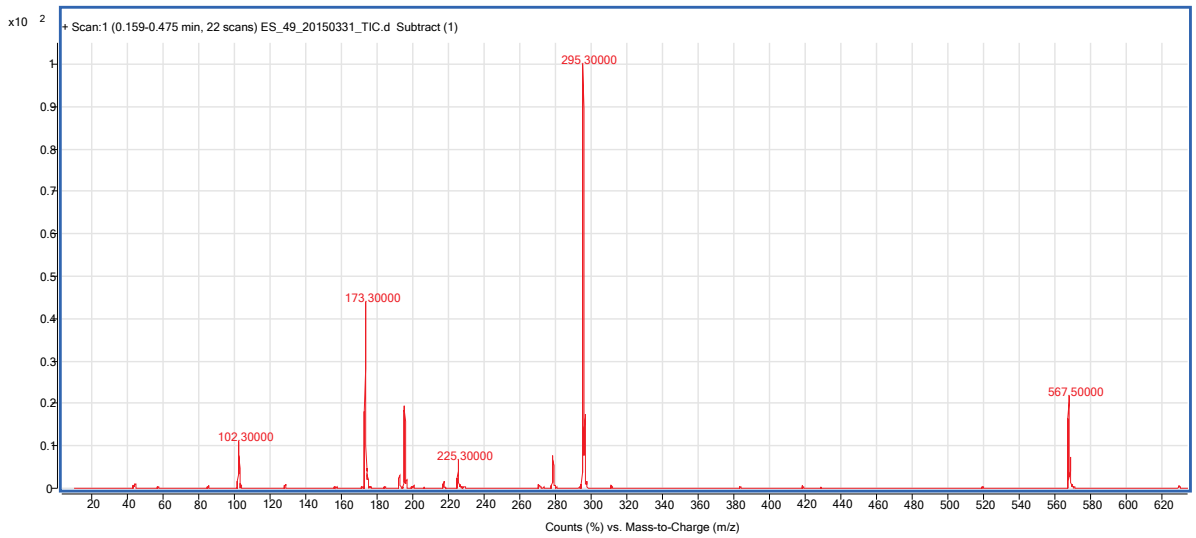


Figure S 30. +ESI-MS spectrum of Boc-Ala-nC$_6$H$_{13}$.

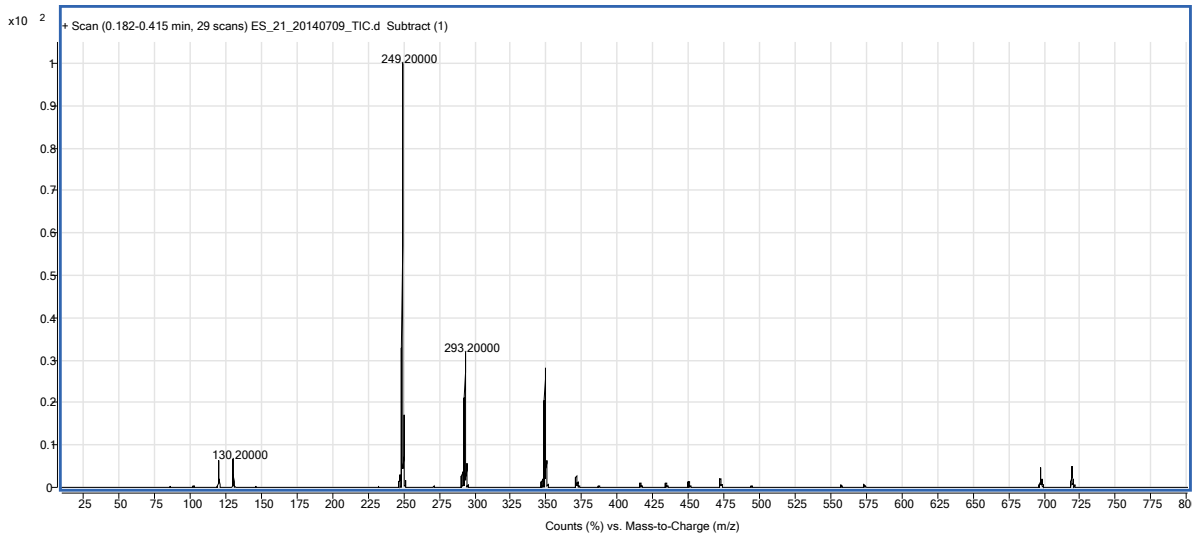


Figure S 31. +ESI-MS spectrum of Boc-Phe-nC$_6$H$_{13}$.

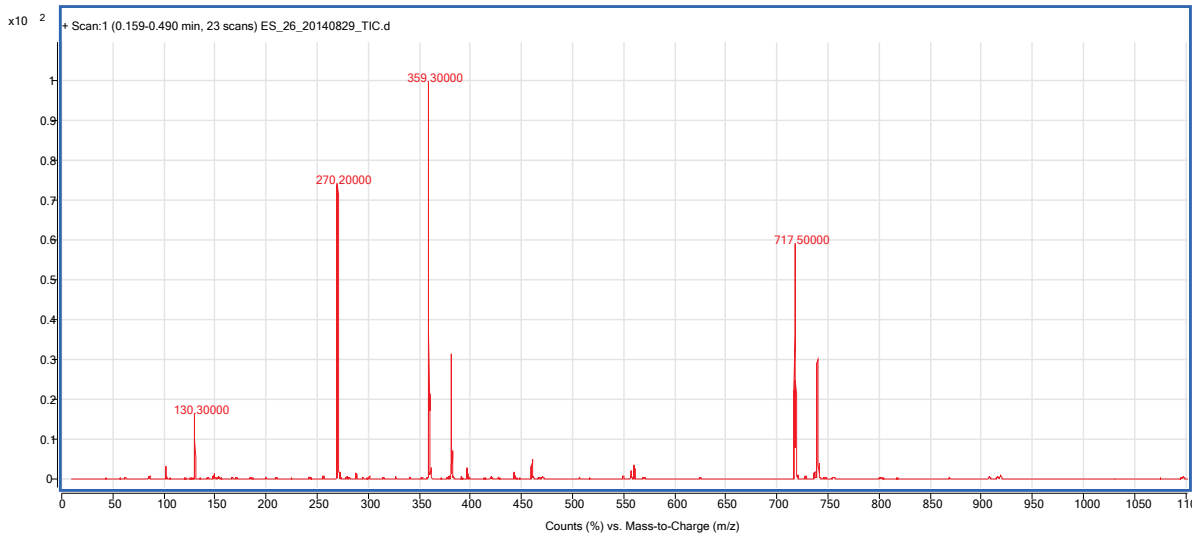


Figure S 32. +ESI-MS spectrum of **1**.

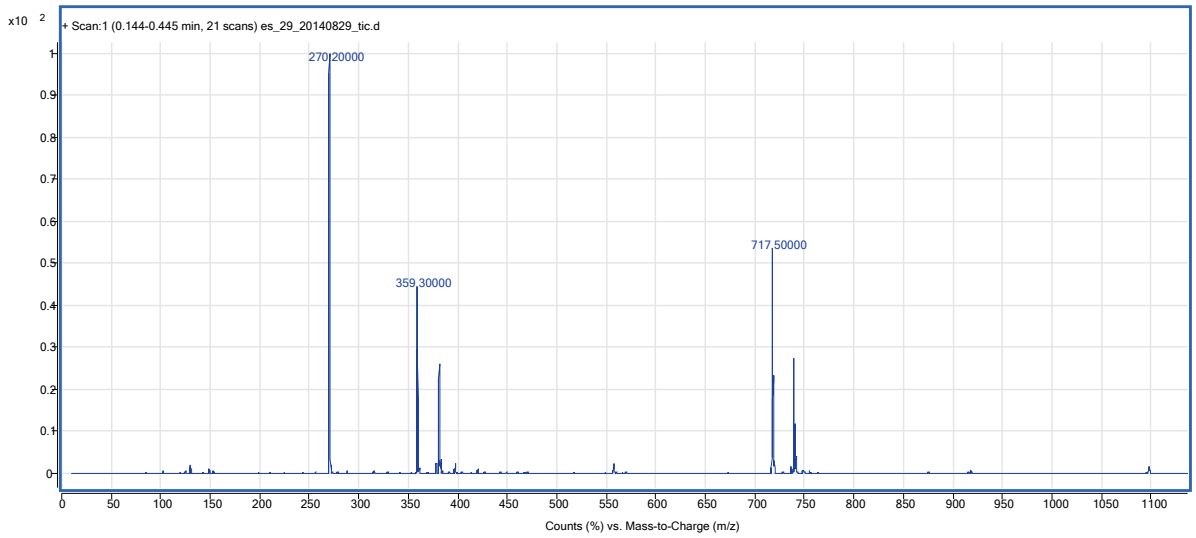


Figure S 33. +ESI-MS spectrum of **2**.

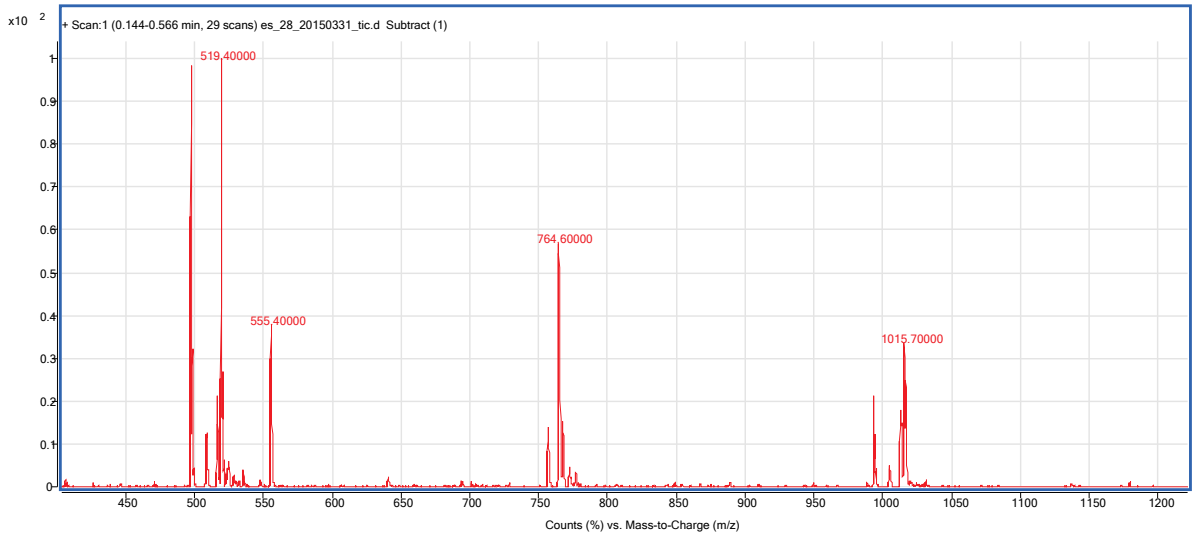


Figure S 34. +ESI-MS spectrum of **3a**.

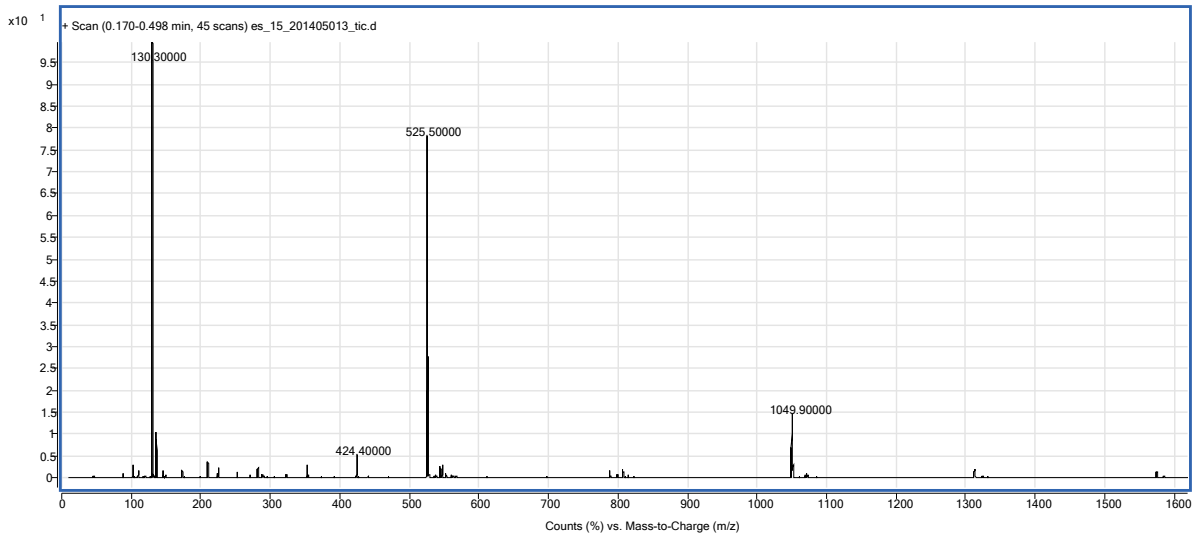


Figure S 35. +ESI-MS spectrum of **3b**.

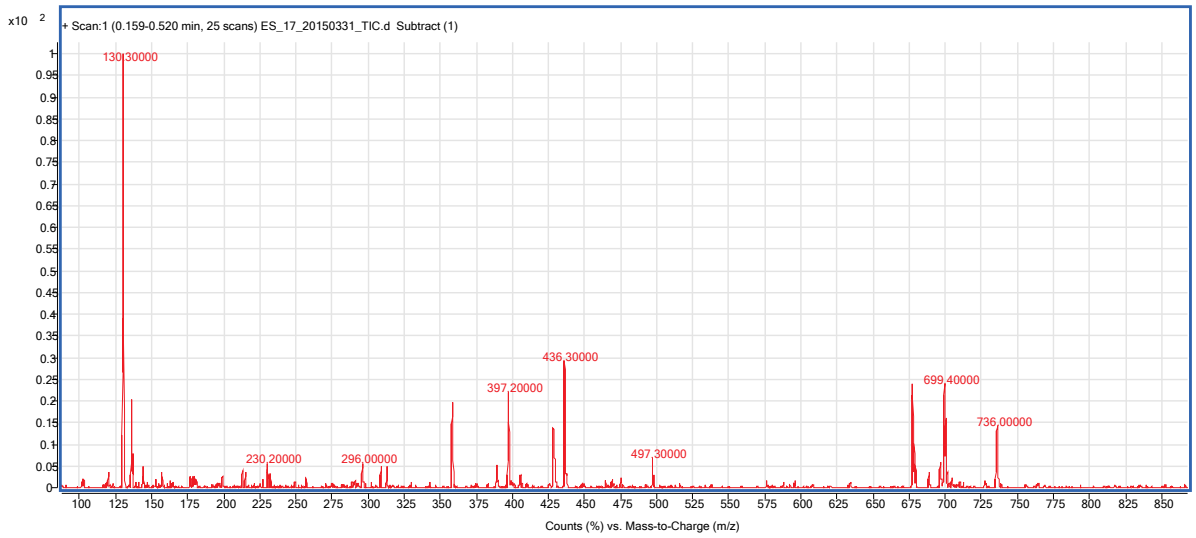


Figure S 36. +ESI-MS spectrum of **3c**.

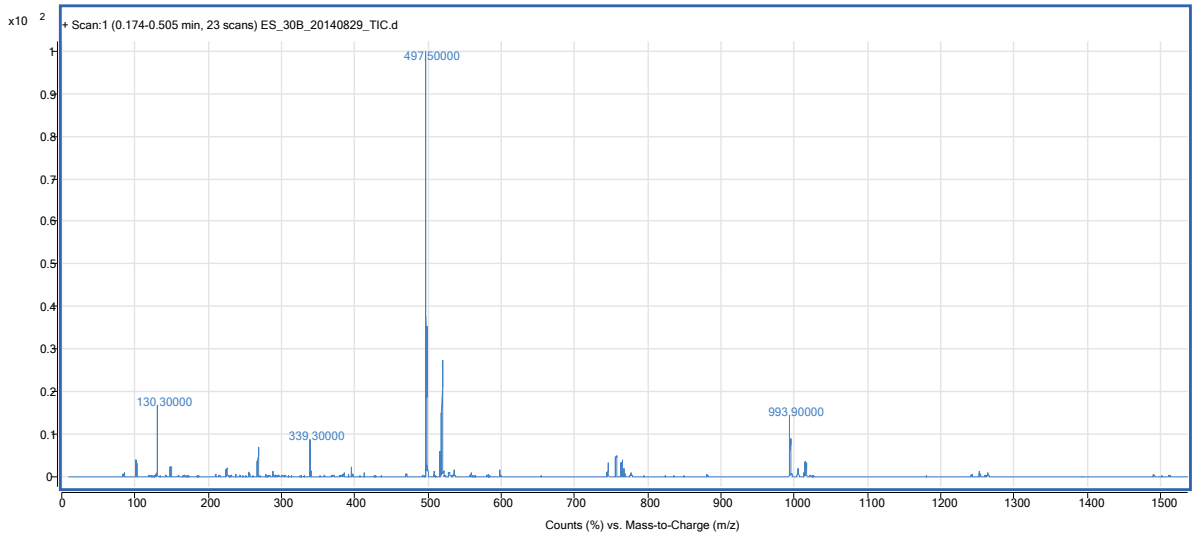


Figure S 37. +ESI-MS spectrum of **4a**.

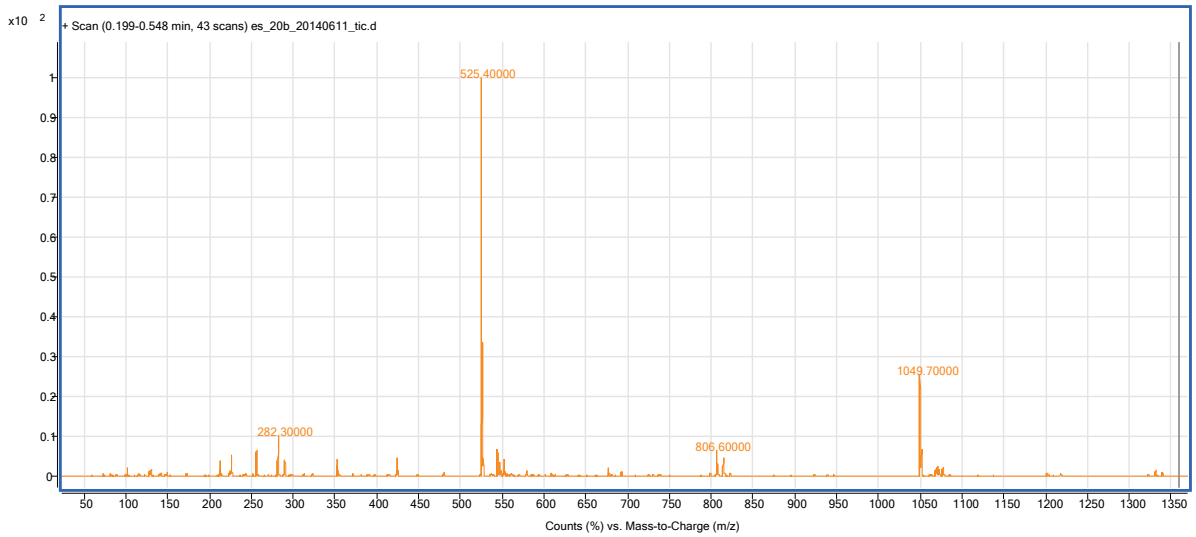


Figure S 38. +ESI-MS spectrum of **4b**.

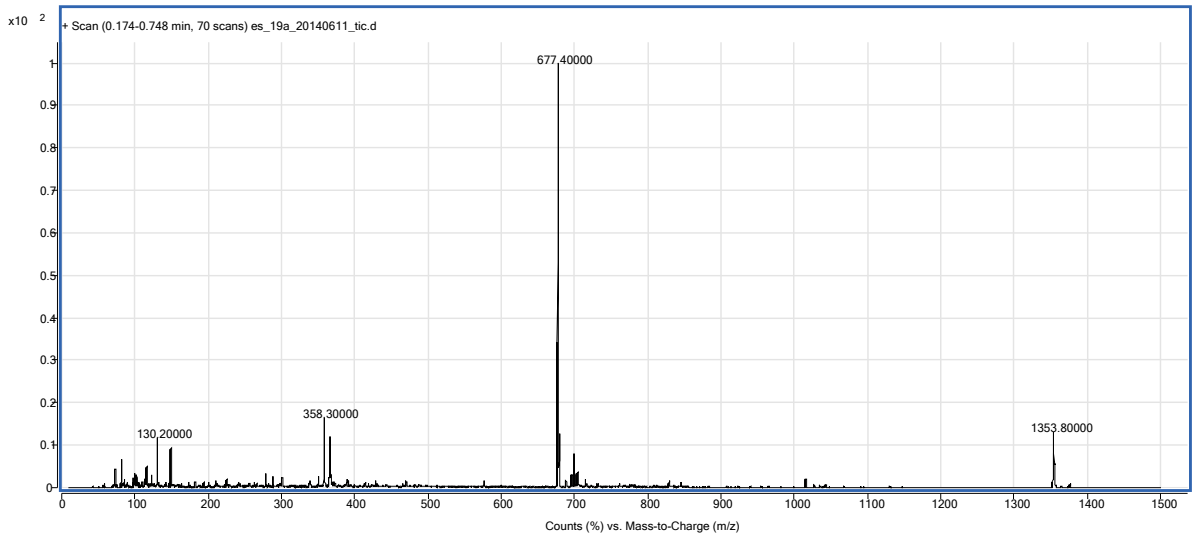


Figure S 39. +ESI-MS spectrum of **4c**.

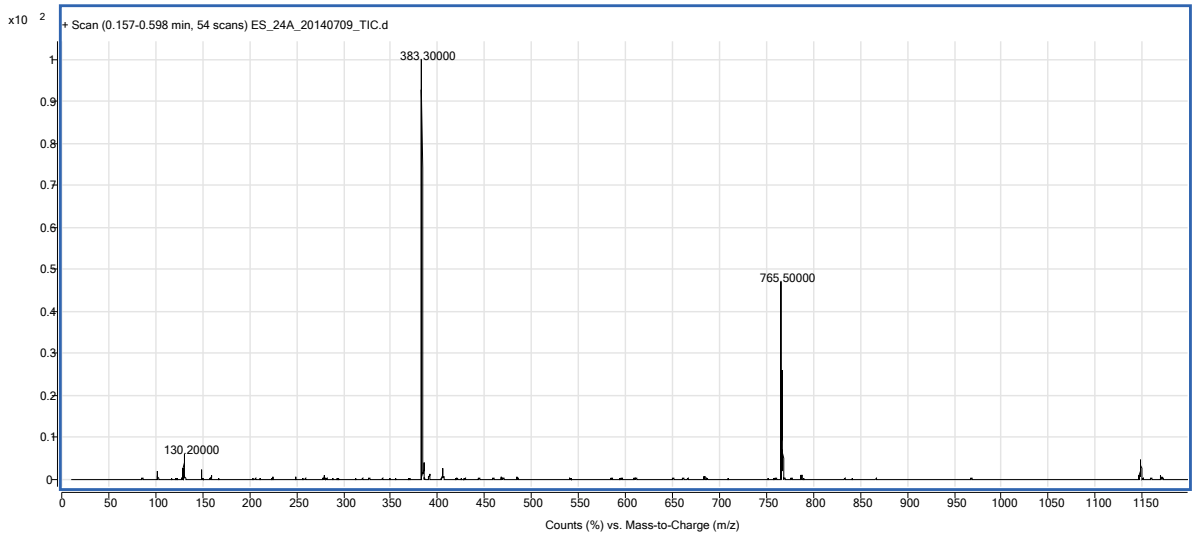


Figure S 40. +ESI-MS spectrum of **5**.

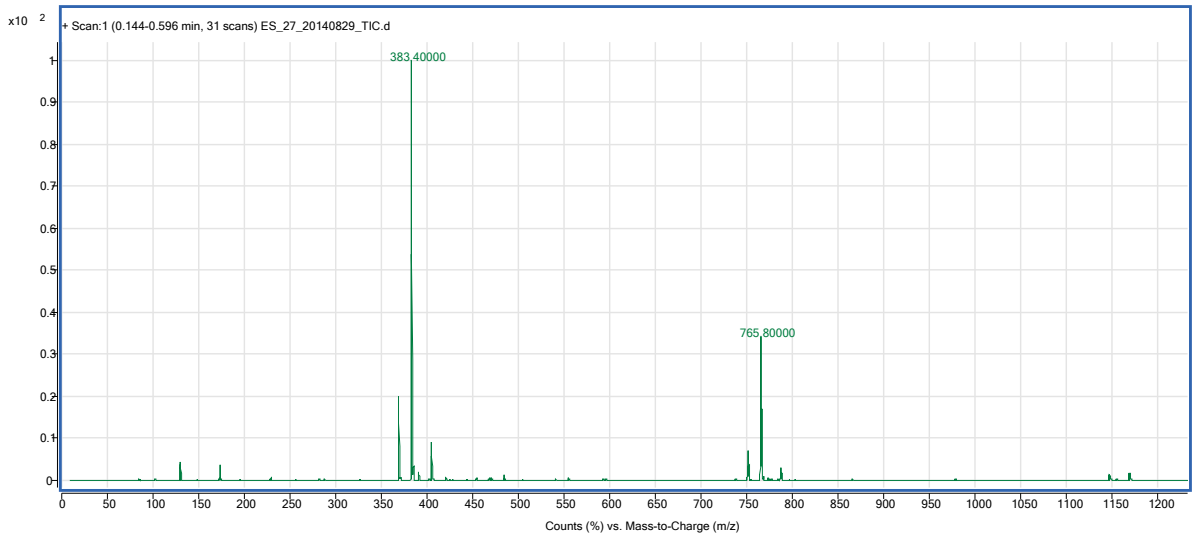


Figure S 41. +ESI-MS spectrum of **6**.

7. X-ray

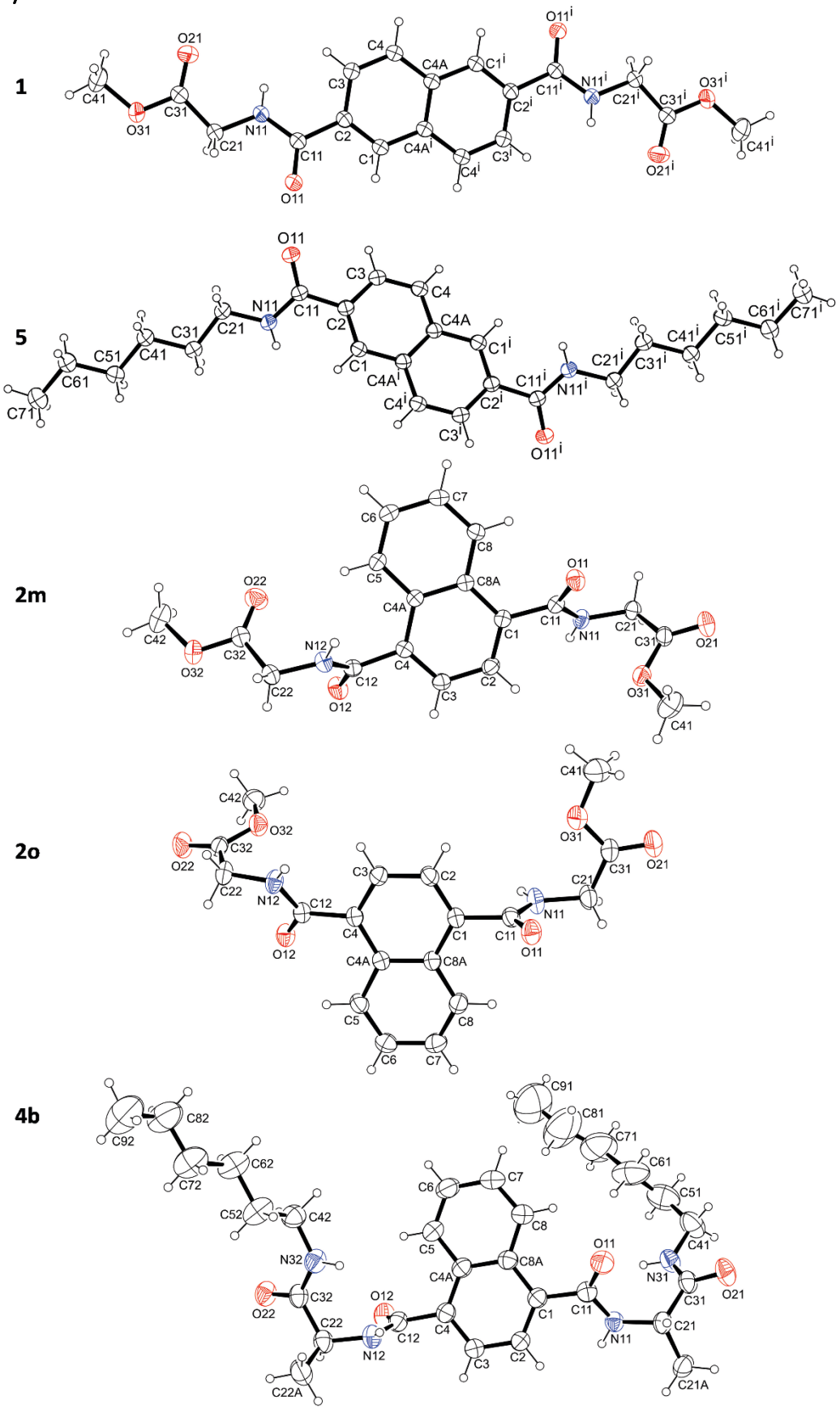


Figure S 42. Atom labeling in the SCXRD structures herein.

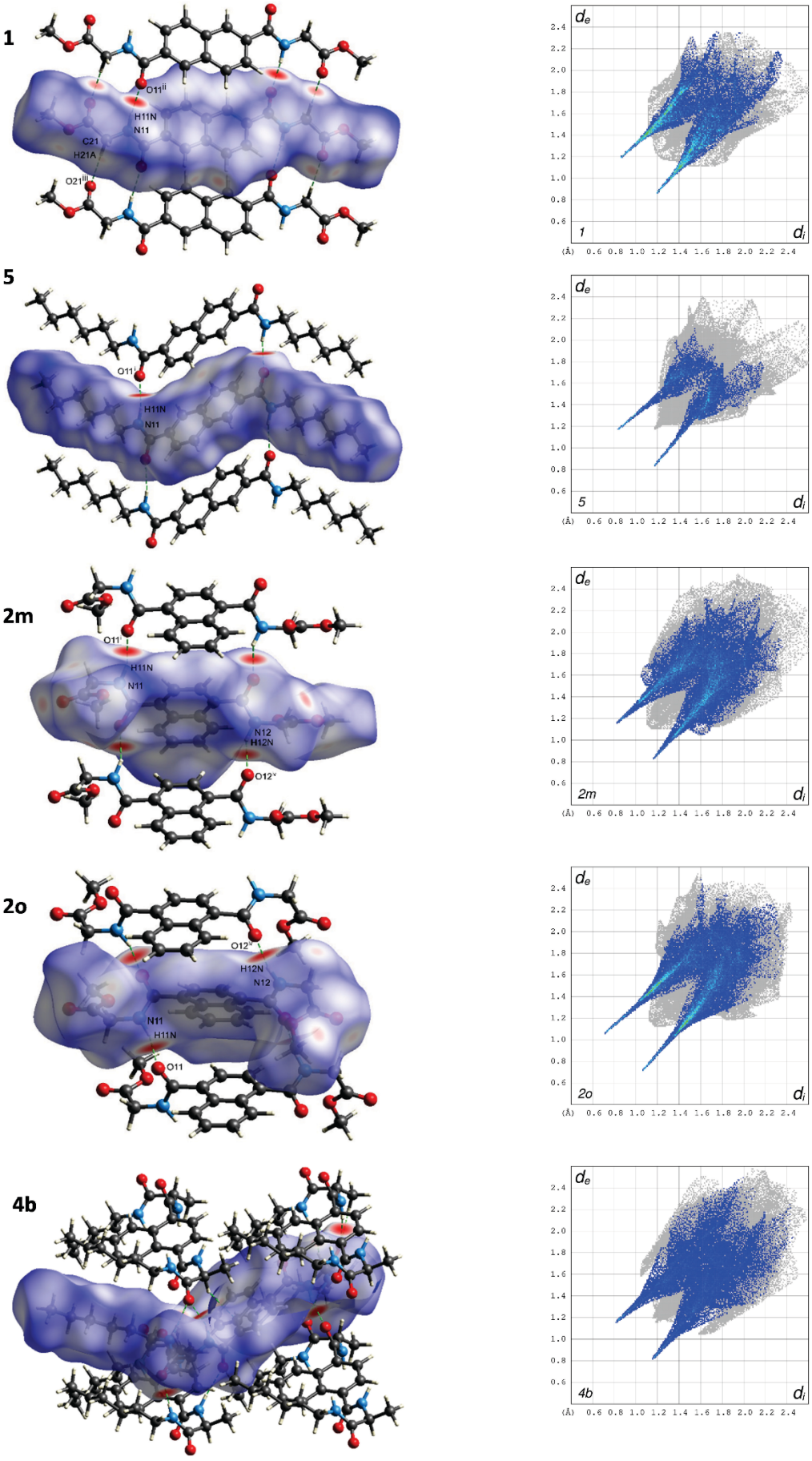


Figure S 43. Hirshfield surfaces and fingerprint plots (N–H···O in blue, other atom contacts in gray).

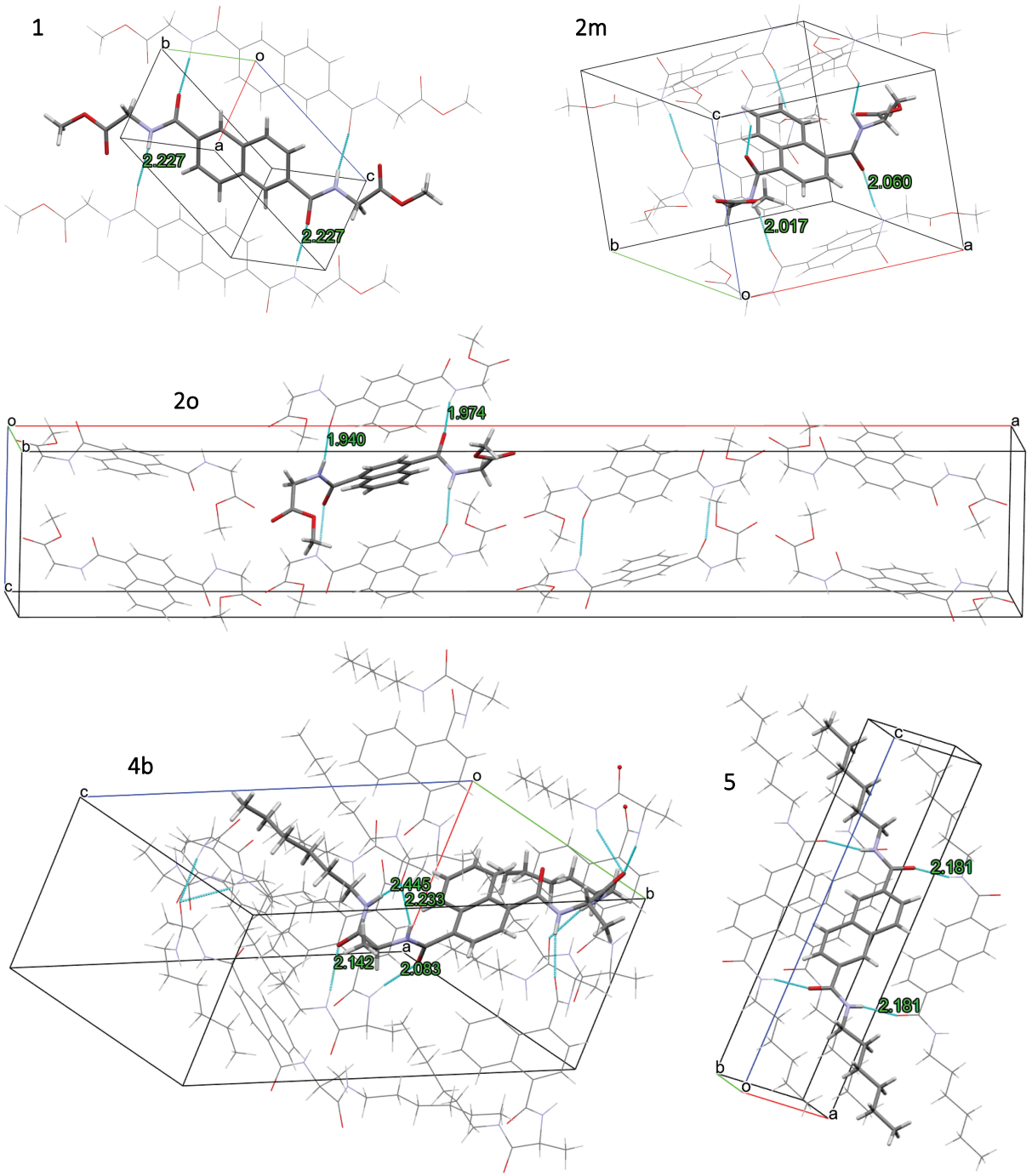


Figure S 44. Crystal packing of molecules in **1**, **2m**, **2o**, **4b**, and **5**. Central molecule is drawn in capped sticks, with characteristic amide hydrogen bonding accented as H···O intermolecular distances in Å.

Table S 1. Crystallographic data and details of the single crystal x-ray diffraction experiments

Compound	1	2o	2m	4b	5
Chemical formula	$C_{18}H_{18}N_2O_6$	$C_{18}H_{18}N_2O_6$	$C_{18}H_{18}N_2O_6$	$C_{30}H_{44}N_4O_4$	$C_{24}H_{34}N_2O_2$
Formula Mass	358.34	358.34	358.34	524.69	382.53
Crystal system	Triclinic	Orthorhombic	Monoclinic	Orthorhombic	Triclinic
a/Å	5.0243(2)	53.4067(15)	13.78785(18)	9.04914(2)	5.0984(3)
b/Å	7.1890(5)	7.3348(2)	12.58632(17)	11.5097(2)	5.2962(2)
c/Å	11.9124(9)	8.9040(2)	10.01527(12)	29.6646(7)	20.1929(11)
α/°	78.122(6)	90.00	90.00	90.00	87.277(4)
β/°	85.234(5)	90.00	96.9791(12)	90.00	84.783(4)
γ/°	89.266(5)	90.00	90.00	90.00	87.054(4)
Unit cell volume/Å^3	419.60(5)	3487.94(16)	1725.16(4)	3089.64(11)	541.77(5)
Space group	$P\bar{1}$(No. 2)	*Pbcn* (No. 60)	$P2_1/c$ (No. 14)	$P2_12_12_1$ (No. 19)	$P\bar{1}$ (No. 2)
No. of formula units per unit cell, *Z*	1	8	4	4	1
Absorption coefficient, μ/mm^{-1}	0.906	0.872	0.881	0.600	0.578
No. of reflections measured	3372	10813	17040	10365	4077
No. of independent reflections	1718	3612	3576	5819	2213
R_{int}	0.0171	0.0282	0.0221	0.0177	0.0261
Final R_1 values ($I > 2\sigma(I)$)	0.0491	0.0578	0.0855	0.0518	0.0535
Final w$R(F^2)$ values ($I > 2\sigma(I)$)	0.1442	0.1478	0.3033	0.1635	0.1536
Final R_1 values (all data)	0.0577	0.0623	0.0874	0.0576	0.0594
Final w$R(F^2)$ values (all data)	0.1559	0.1518	0.3044	0.1635	0.1663
Goodness-of-fit on F^2	1.045	1.065	1.126	1.037	1.062
Flack parameter	-	-	-	0.14(13)	-
CCDC number	CCDC 2580557	CCDC 2580559	CCDC 2580558	CCDC 2580560	CCDC 2580561

Table S 2. Parameters for intermolecular N–H···O and C–H···O interactions in **1**, **2o**, **2m**, **4b**, and **5**.

Interaction	D-H (Å)	H···A (Å)	D ···A (Å)	D-H···A (°)
1				
N11–H11N···O11^{ii}	0.85(3)	2.23(2)	3.0455(18)	162(2)
C21–H21A···O21^{iii}	0.97	2.45	3.416(3)	178
C21–H21B···O31^{iv}	0.97	2.59	3.355(2)	136
2o				
N11–H11N···O11^{i}	0.83(3)	1.98(3)	2.783(2)	164(2)
C21—H21A···O21^{ii}	0.97	2.63	3.449(4)	143
C41—H41C···O11^{iii}	0.96	2.62	3.392(4)	138
N12–H12N···O12^{iv}	0.85(3)	1.94(3)	2.778(2)	168(2)
C22—H22B···O22^{v}	0.97	2.60	3.565 (3)	176
C42—H42A···O22^{vi}	0.96	2.54	3.473 (3)	166
2m				
N11–H11N···O11^{i}	0.97(5)	2.02(5)	2.899(4)	150(4)
C41–H41A···O22^{ii}	0.96	2.49	3.355(8)	149
C41–H41B···O21^{iii}	0.96	2.52	3.383(7)	150
N12–H12N···O12^{iv}	1.00(5)	2.06(5)	2.914(4)	142(4)
C22–H22A···O21^{v}	0.97	2.45	3.347(6)	154
4b				
N11–H11N···O12^{i}	0.93(4)	2.08(4)	3.002(3)	168(3)
N31–H31N···O22^{i}	0.85(4)	2.14(4)	2.969(4)	165(3)
N12–H12N···O21^{ii}	0.71(4)	2.23(4)	2.893(4)	156(4)
C22A–H22A···O21^{ii}	0.96	2.60	3.296(5)	130
N32–H32N···O21^{ii}	0.96(5)	2.45(5)	3.300(4)	148(4)
5				
N11–H11N···O11^{ii}	0.83(2)	2.18(2)	2.9921(15)	166.8(18)

Symmetry codes: (ii) *x*+1, *y*, *z*; (iii) *x*−1, *y*, *z* and (iv) −*x*+1, −*y*+3, −*z* for **1**; (i) *x*, −*y*, *z*+1/2; (ii) −*x*, −*y*, −*z*; (iii) *x*, *y*+1, *z*; (iv) *x*, −*y*, *z*−1/2; (v) −*x*+1/2, −*y*+1/2, *z*−1/2 and (vi) −*x*+1/2, *y*+1/2, *z* for **2o**; (i) *x*, −*y*+1/2, *z*−1/2; (ii) −*x*+1, *y*+1/2, −*z*+1/2; (iii) −*x*, −*y*+1, −*z*; (iv) *x*, −*y*+1/2, *z*+1/2 and (v) *x*+1, *y*, *z* for **2m**; (i) −*x*+1, *y*+1/2, −*z*+1/2 and (ii) −*x*, *y*−1/2, −*z*+1/2 for **4b** and (ii) *x*+1, *y*, *z*. for **5**.

Table S 3. Selected torsional angles (°) for **1**, **2o**, **2m**, **4b** and **5**

Angle	1	2o	2m	4b	5
C1-C2-C11-O11	23.35(2)				144.59(1)
C2-C1-C11-O11		94.9(3)	–128.31(1)	–139.0(3)	
C3-C4 C12-O12		144.8(2)	–57.03(1)	–106.9(3)	
C11-N11-C21-C31	160.26(1)	–87.1(3)	64.15(1)	–71.6(3)	-173.85(1)
C12-N12-C22-C32		–70.3(3)	–76.70(1)	–72.1(3)	
N11-C21-C31-O21(N31)[a]	–5.29(1)	176.7(3)	–160.0(4)	–24.8(4)	
N12-C22-C32-O22(N32)[b]		166.1(2)	–4.67(1)	–30.0(4)	
C31-N41-C41-C51				–158.9(4)	
C32-N42-C42-C52				88.5(4)	

[a] N11-C21-C31-O21 for structures **1**, **2o** and **2m** and N11-C21-C31-N31 for structure **4b**

[b] N12-C22-C32-O22 for structures **1**, **2o** and **2m** and N12-C22-C32-N32 for structure **4b**

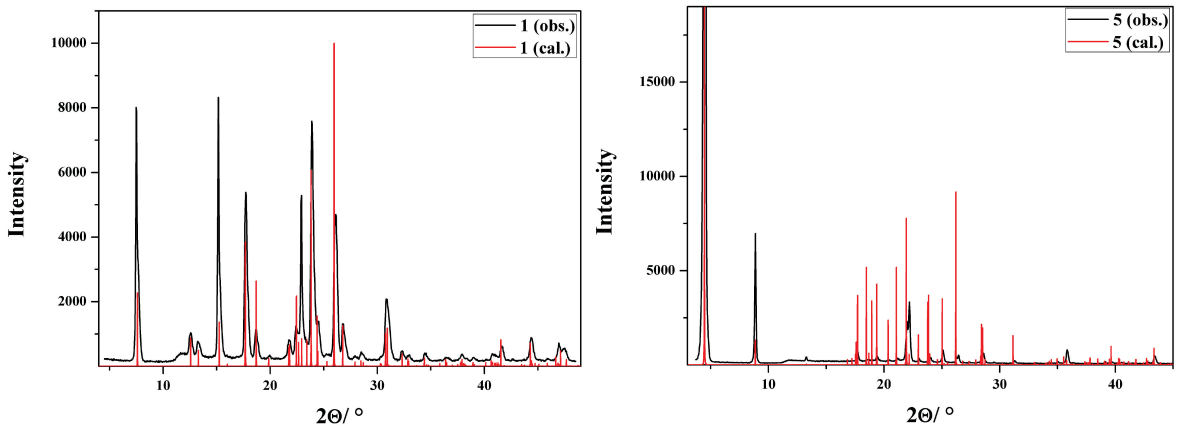


Figure S 45. PXRD pattern of bioconjugate **1** and **5** (black lines) and the corresponding position of the diffraction lines calculated from the SCXRD structure (red lines).

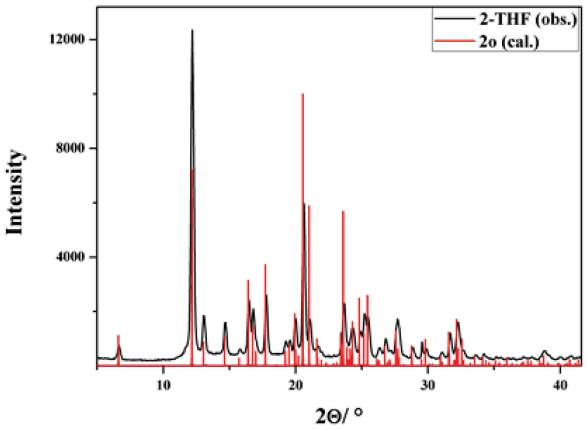


Figure S 46. PXRD pattern of batch **2** sample from tetrahydrofuran solvent (black line) and the corresponding position of the diffraction lines from the SCXRD structure of **2o** (red line).

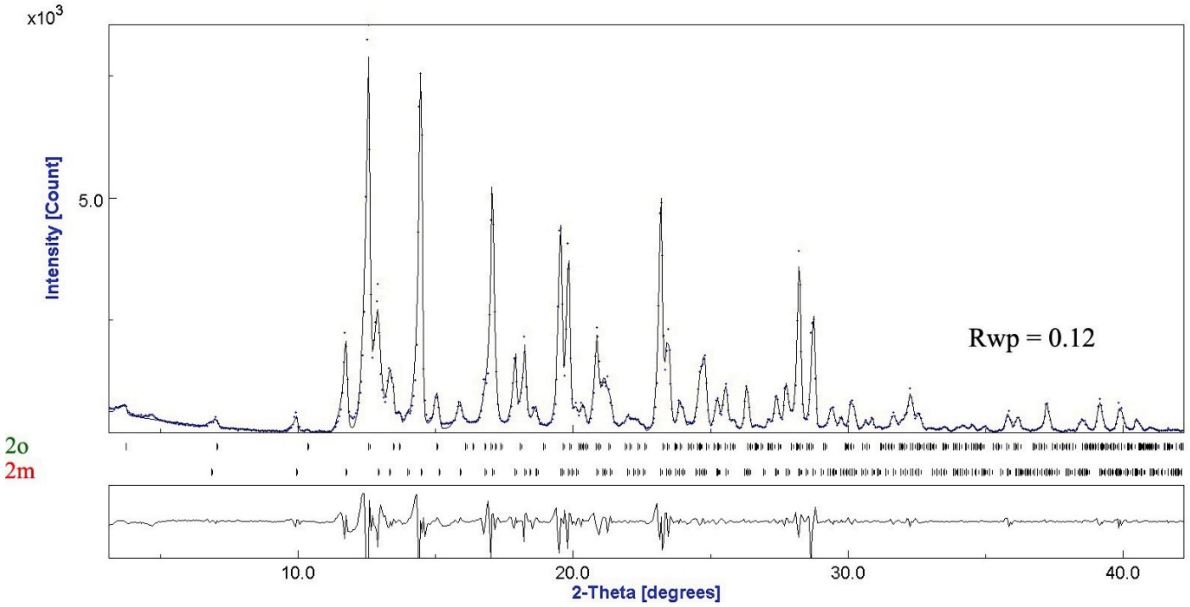


Figure S 47. The results of Rietveld quantitative crystal phase analysis (program MAUD) of the product 2-EtOH. Vertical bars below the diffraction pattern mark the positions of diffraction lines calculated from structure of orthorhombic (2o) or monoclinic (2m) polymorphs determined by single crystal analysis, respectively. The differences between the observed and refined patterns are shown in the box below.