Electronic Supplementary Material (ESI) for RSC Advances.

Supporting information

Synthesis, Supramolecular Organization and Thermotropic Phase Behaviour of N acyltris(hydroxymethyl)aminomethane

Sengan Megarajan[a], Siva Bala Subramaniyan[a], Sureshan Muthuswamy[a], Savarimuthu Philip Anthony[a], Dohyun Moon[b]*, Joyti Arunachalam[a], Veerappan Anbazhagan[a]*

[a]School of Chemical and Biotechnology, SASTRA Deemed University, Thanjavur - 613401, Tamil Nadu, India.

[b]Beamline Department, Pohang Accelerator Laboratory, 80 Jigokro-127 beongil, Nam-gu, Pohang, Korea

Table S1: ^{1}H NMR spectral data of N-acyl tromethamine in CD_3OD. Chemical shift values are given in ppm (δ scale).

Acyl chain length	CH_3-$(CH_2)_n$-	CH_3-$(CH_2)_n$-	-CH_2-CH_2-C=O	-CH_2- (C=O)	-HN-C-$(CH_2)_3$-
10	0.81, t	1.22, bs (12H)	1.52, m	2.16, t	3.63, s
12	0.80, t	1.20, bs (16H)	1.48, m	2.15, t	3.62, s
14	0.80, t	1.19, bs (20H)	1.50, m	2.15, t	3.62, s
16	0.81, t	1.20, bs (24H)	1.52, m	2.16, t	3.63, s
18	0.92, t	1.31, bs (28H)	1.62, m	2.26, t	3.73, s

Table S2: ^{13}C NMR spectral data of N-acyl tromethamine. Chemical shift values are given in ppm (δ scale).

Acyl chain length	CH_3-CH_2-$(CH_2)_n$-	CH_3-CH_2-$(CH_2)_n$-	CH_3-CH_2-$(CH_2)_n$-	-$(CH_2)_n$-CH_2-CH_2-C=O	-CH_2-C=O	-CH_2-C=O	-HN-C-	-C-CH_2-	solvent	solvent peak
10	14.10	22.66	29.12-29.69	25.79	37.05	175.25	62.25	61.64	$CDCl_3$	76.72-77.35
12	13.05	22.33	28.86-29.34	25.59	36.17	176.06	62.13	61.25	CD_3OD	46.75-48.46
14	13.58	22.29	28.78-29.26	25.47	36.49	175.61	61.43	61.55	$CDCl_3$ + CD_3OD	47.55-49.26, 76.57-77.43
16	13.52	22.26	28.75-29.27	25.45	36.46	175.63	61.42	61.55	$CDCl_3$ + CD_3OD	47.46-49.16, 76.57-77.43
18	13.30	22.12	28.63-29.14	25.33	36.28	175.61	61.32	61.51	$CDCl_3$ + CD_3OD	47.18-48.88, 76.57-77.43

Table S3: Single crystal parameters

Compound	**N10TM** (1831968)	**N12TM** (1831969)	**N14TM** (1831970)
chemical formula	$C_{14}H_{29}NO_4$	$C_{16}H_{33}NO_4$	$C_{18}H_{37}NO_4$
formula mass	275.38	303.43	331.48
crystal system	Monoclinic	Monoclinic	Monoclinic
space group	$P2_1/c$	$P2_1/c$	$P2_1/c$
a (Å)	20.667(4)	23.076(5)	a = 25.529(5)
b (Å)	8.6810(17)	8.6760(17)	b = 8.6680(17)
c (Å)	9.0510(18)	9.0510(18)	c = 9.0460(18)
β (deg)	91.06(3)°	91.41(3)°	93.26(3)°
unit cell volume (Å^3)	1623.6(6)	989.2(4)	1998.5(7)
temp (K)	290(2) K	284(2) K	288(2) K
Z	4	4	4
Wavelength (Å)	0.610	0.610	0.610
Absorption coefficient	0.059 mm^{-1}	0.057 mm^{-1}	0.056 mm^{-1}
F(000)	608	672	736
Theta range for data collection	2.184 to 24.998°	2.792 to 25.000°.	2.744 to 24.998°
Reflections collected	20622	16669	17599
Independent reflections	4509 [R(int) = 0.0463]	4799 [R(int) = 0.0453]	5336 [R(int) = 0.0311]
Completeness to theta = 21.469°	99.7%	96.3 %	96.6 %
Max. and min. transmission	0.994 and 0.990	1.000 and 0.948	1.000 and 0.937
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data / restraints / parameters	4509 / 0 / 177	4799 / 0 / 195	5336 / 0 / 213
Goodness-of-fit on F^2	1.005	1.078	1.080
Final R indices [I>2sigma(I)]	R1 = 0.0547, wR2 = 0.1561	R1 = 0.0688, wR2 = 0.2175	R1 = 0.0492, wR2 = 0.1508
R indices (all data)	R1 = 0.0919, wR2 = 0.1734	R1 = 0.0907, wR2 = 0.2292	R1 = 0.0594, wR2 = 0.1604

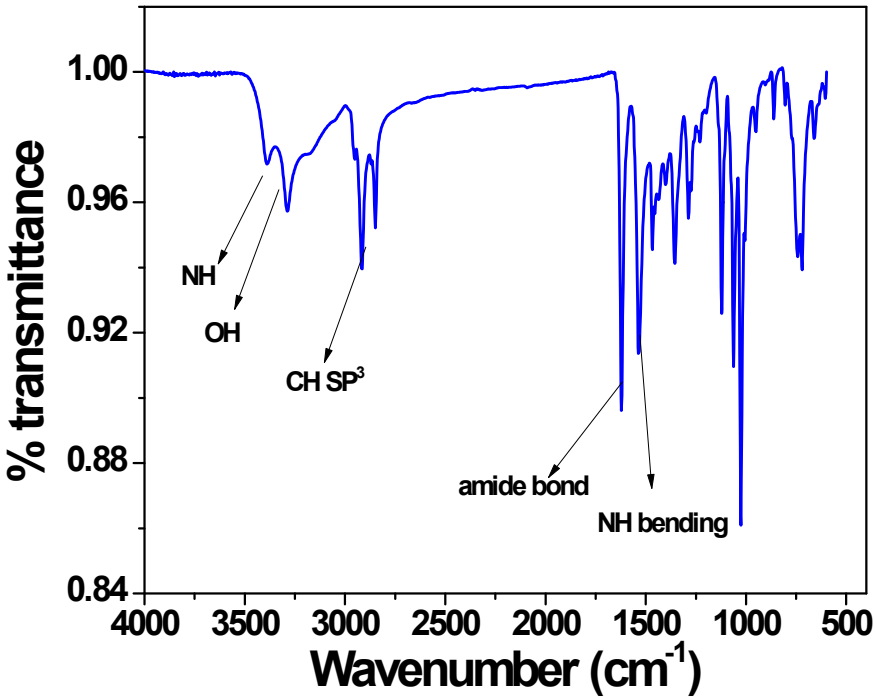

Fig. S1: FTIR spectra of N10TM

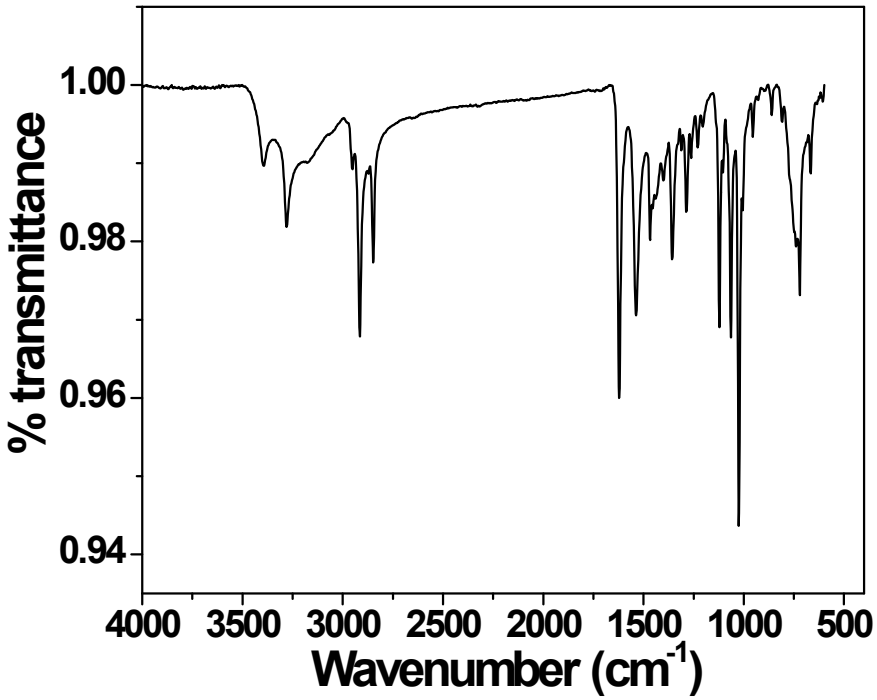

Fig. S2: FTIR spectra of N12TM

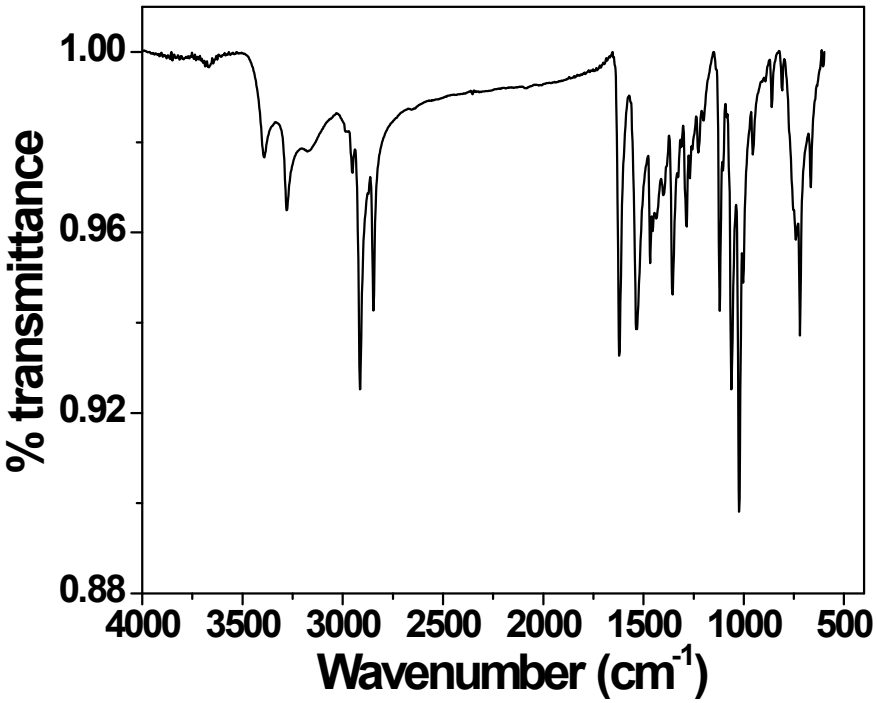

Fig. S3: FTIR spectra of N14TM

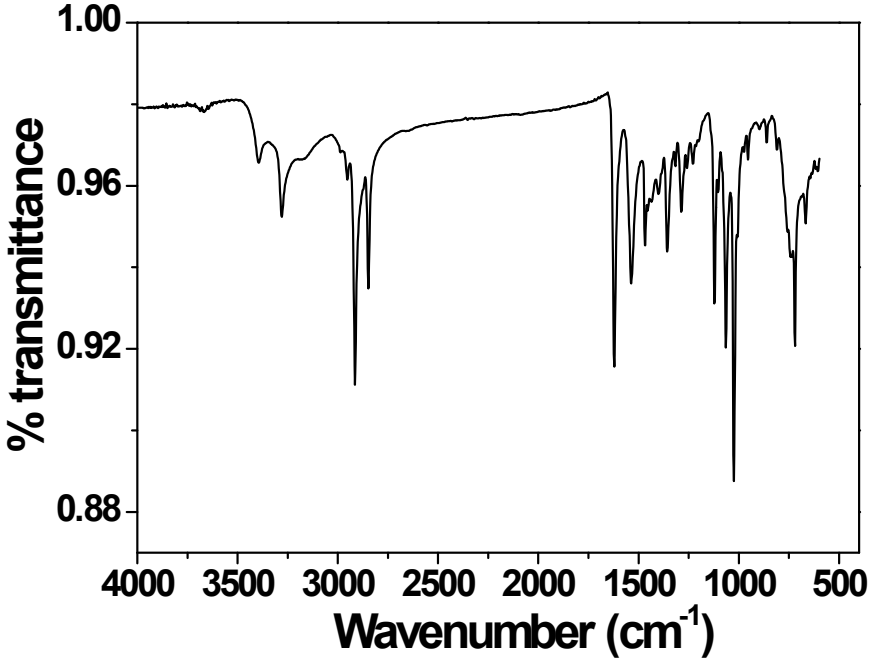

Fig. S4: FTIR spectra of N16TM

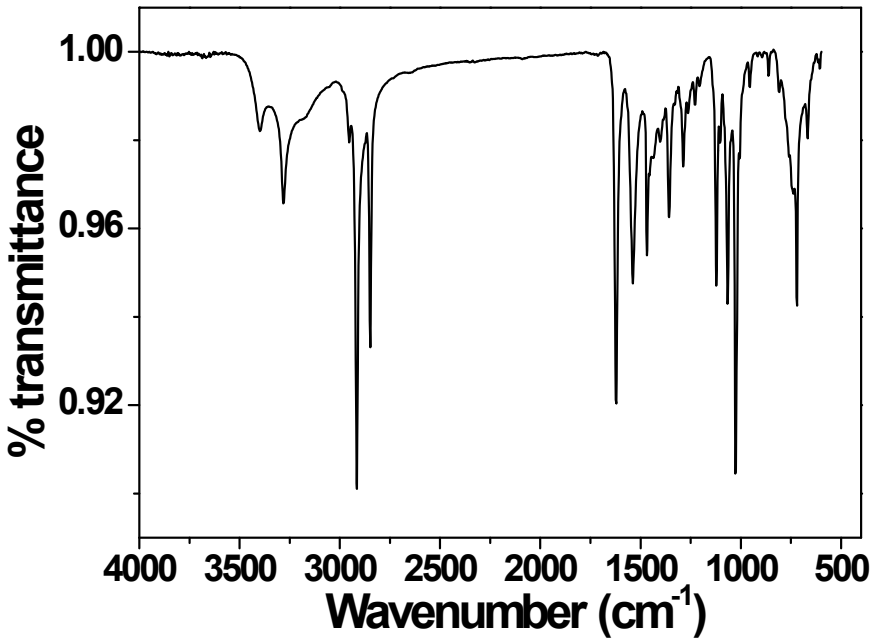

Fig. S5: FTIR spectra of N18TM

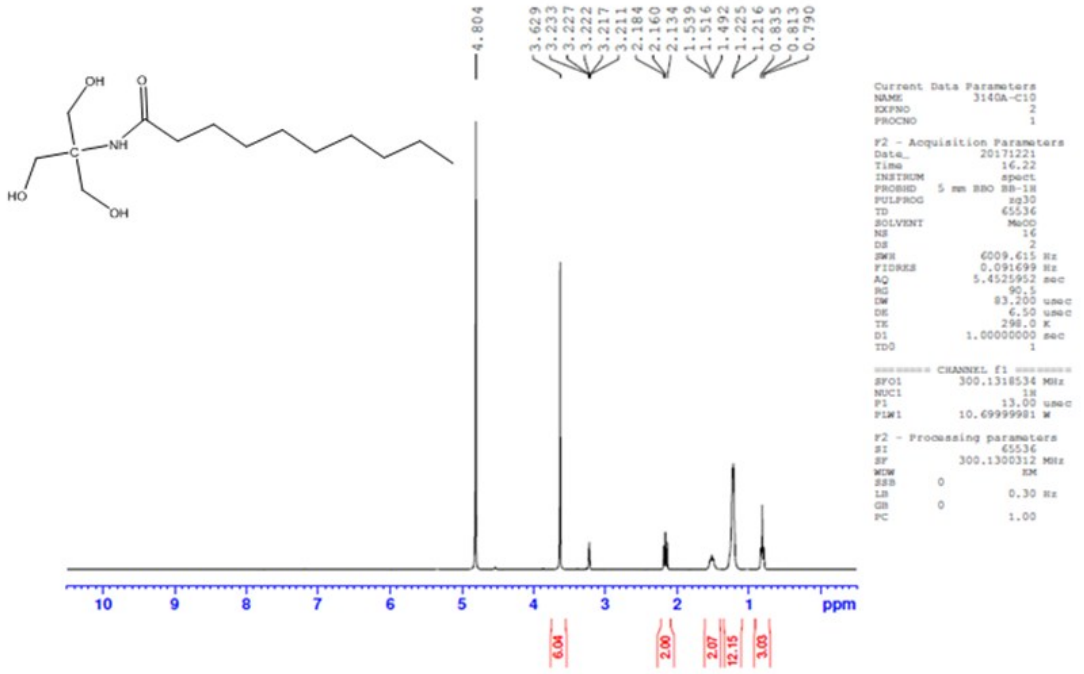

Fig. S6: ^{1}H-NMR spectra of N10TM

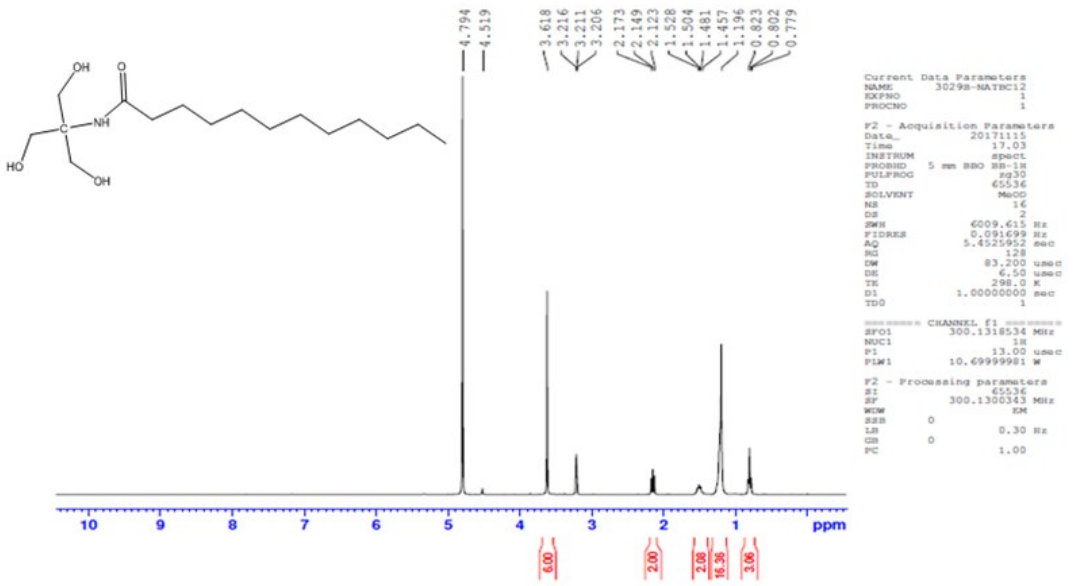

Fig. S7: ^{1}H-NMR spectra of N12TM

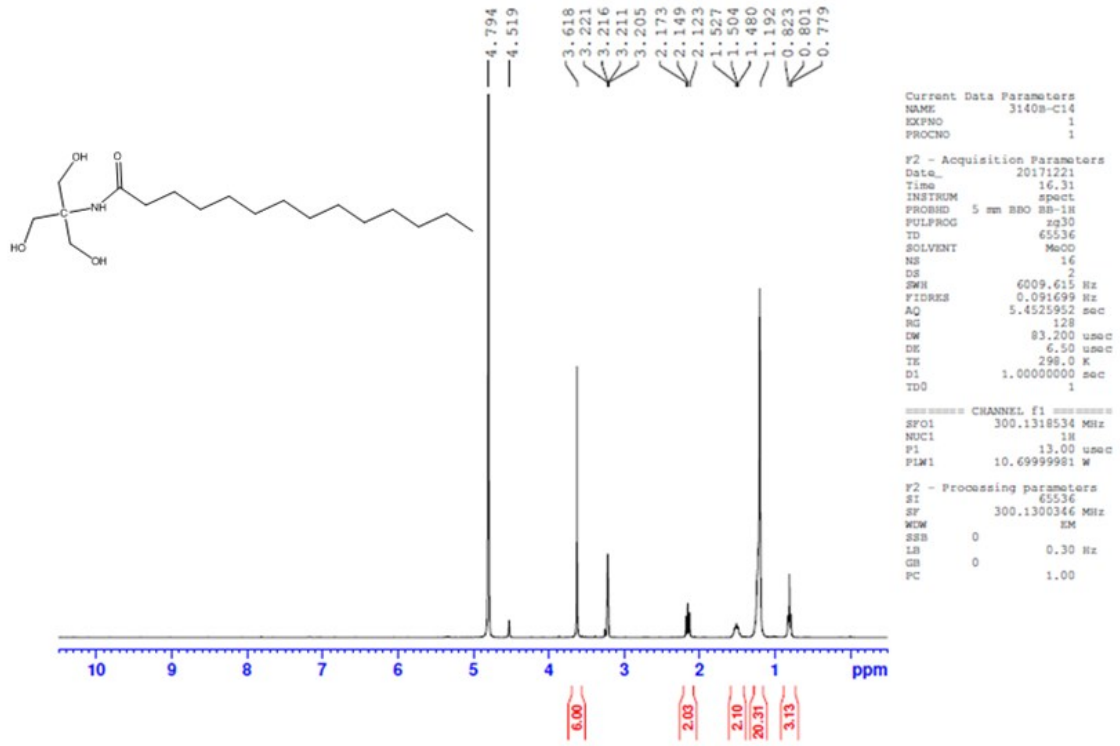

Fig. S8: ^{1}H-NMR spectra of N14TM

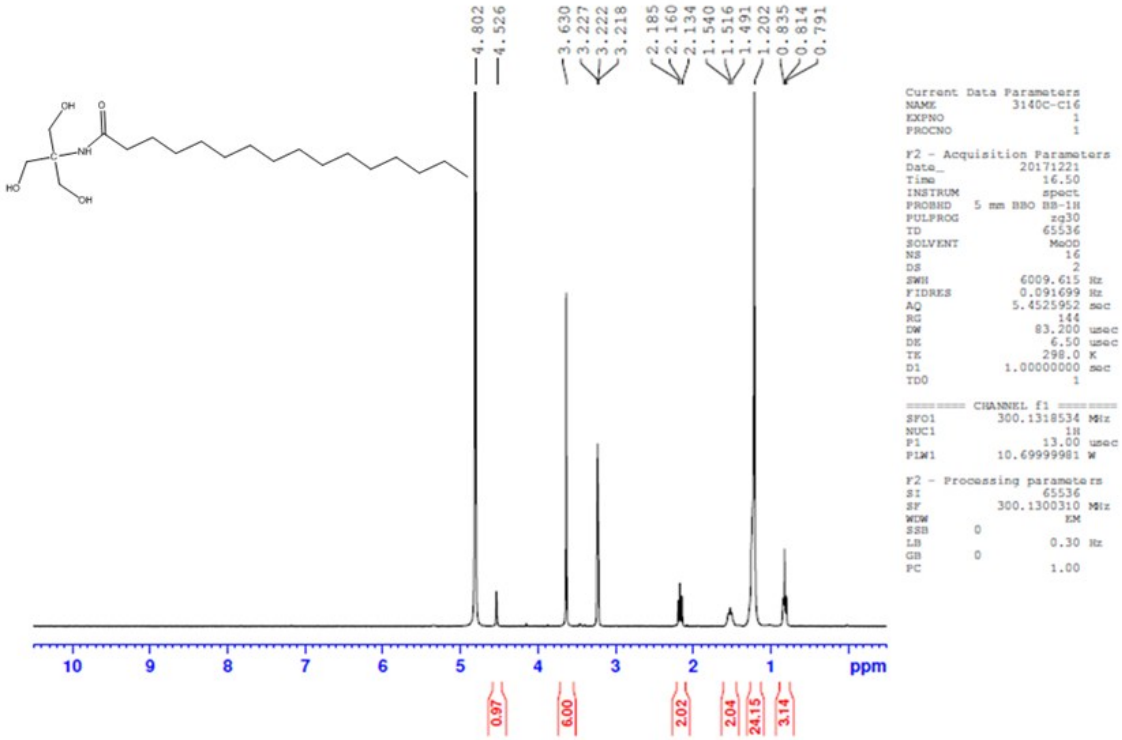

Fig. S9: ^{1}H-NMR spectra of N16TM

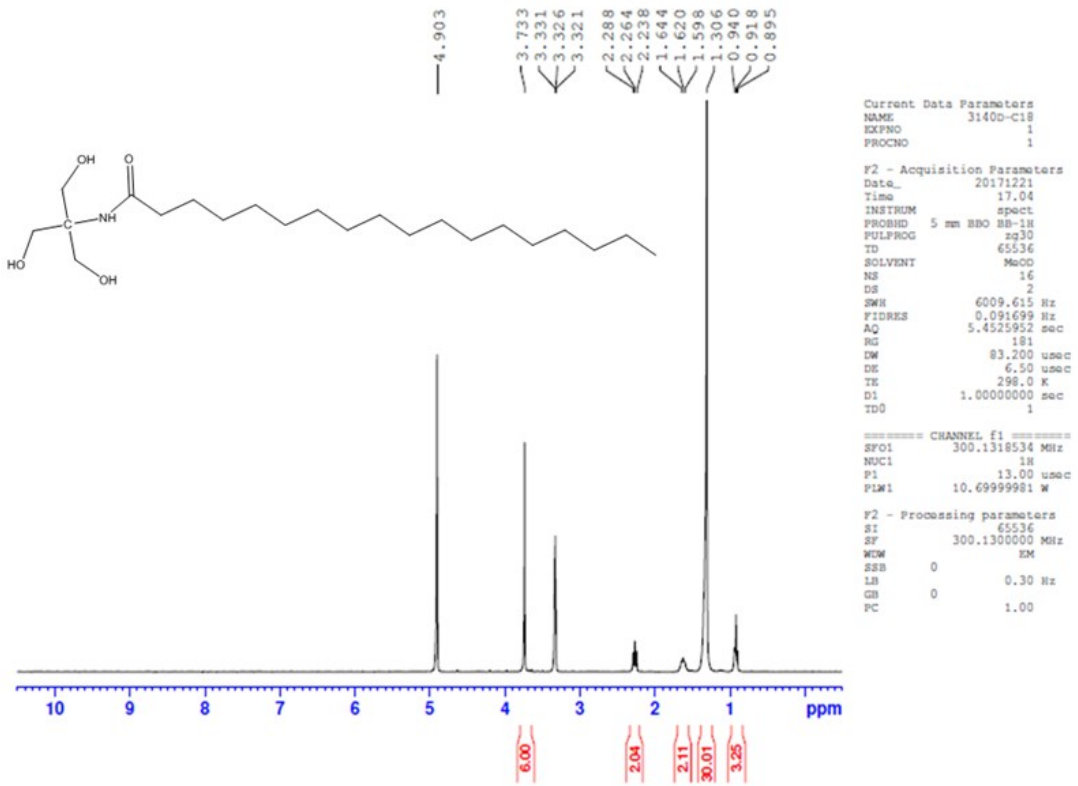

Fig. S10: ^{1}H-NMR spectra of N18TM

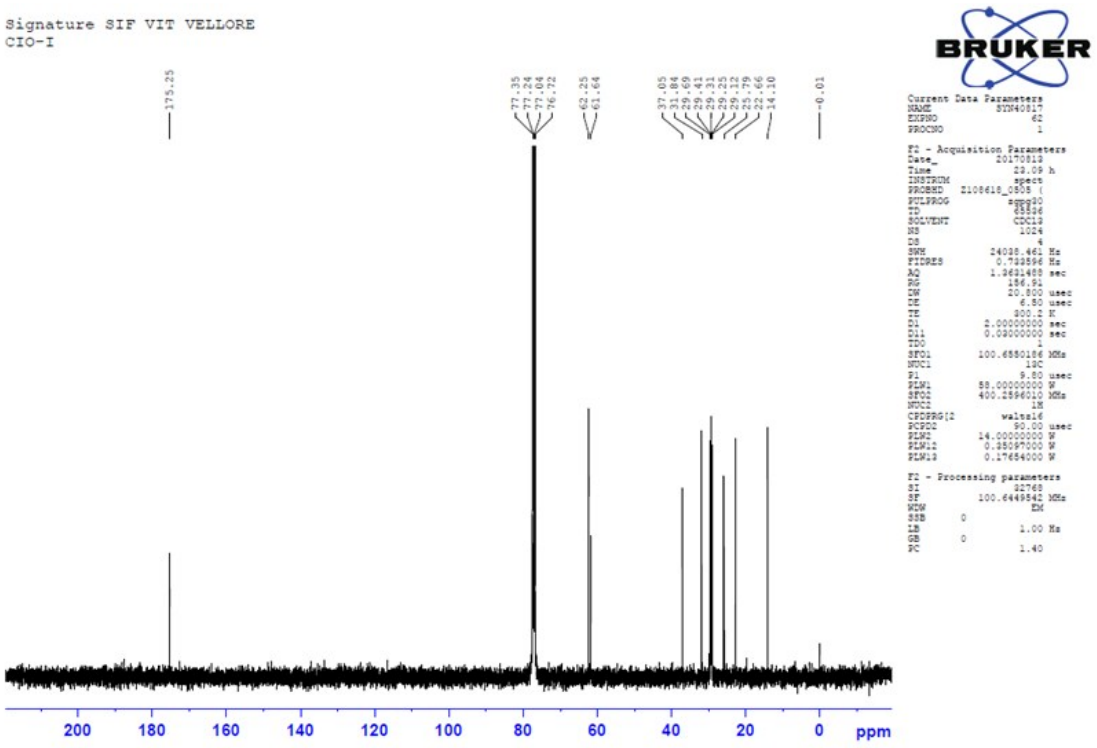

Fig. S11: ^{13}C-NMR spectra of N10TM

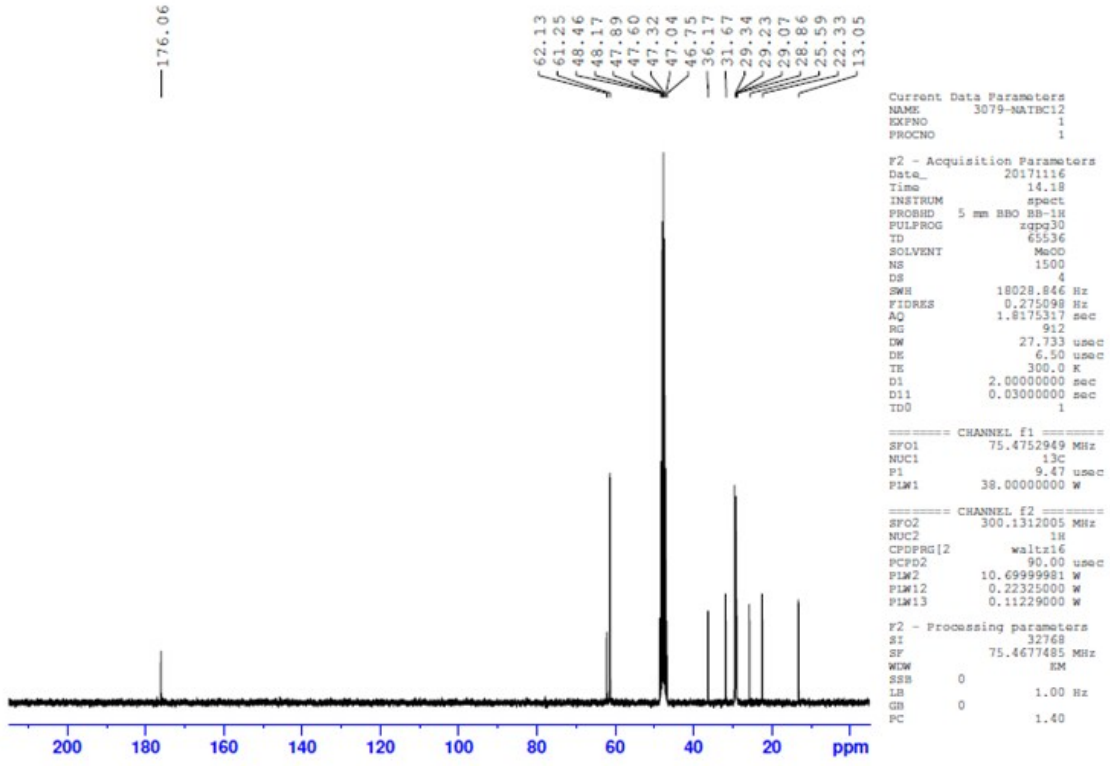

Fig. S12: ^{13}C-NMR spectra of N12TM

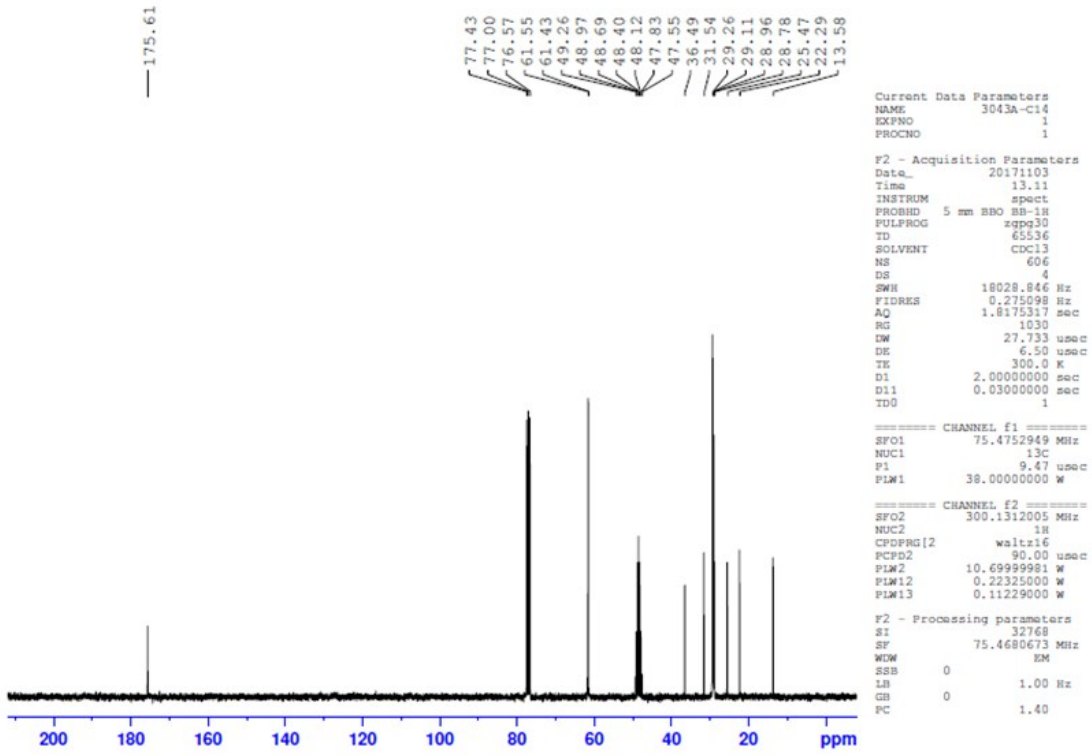

Fig. S13: ^{13}C-NMR spectra of N14TM

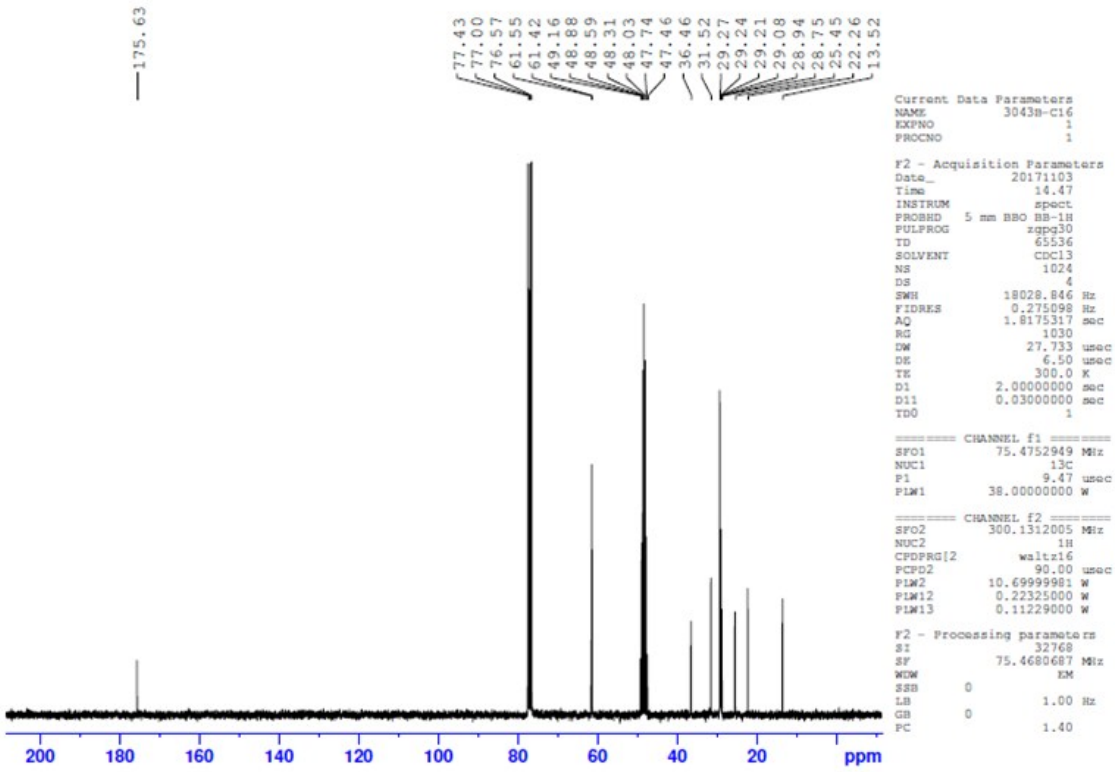

Fig. S14: ^{13}C-NMR spectra of N16TM

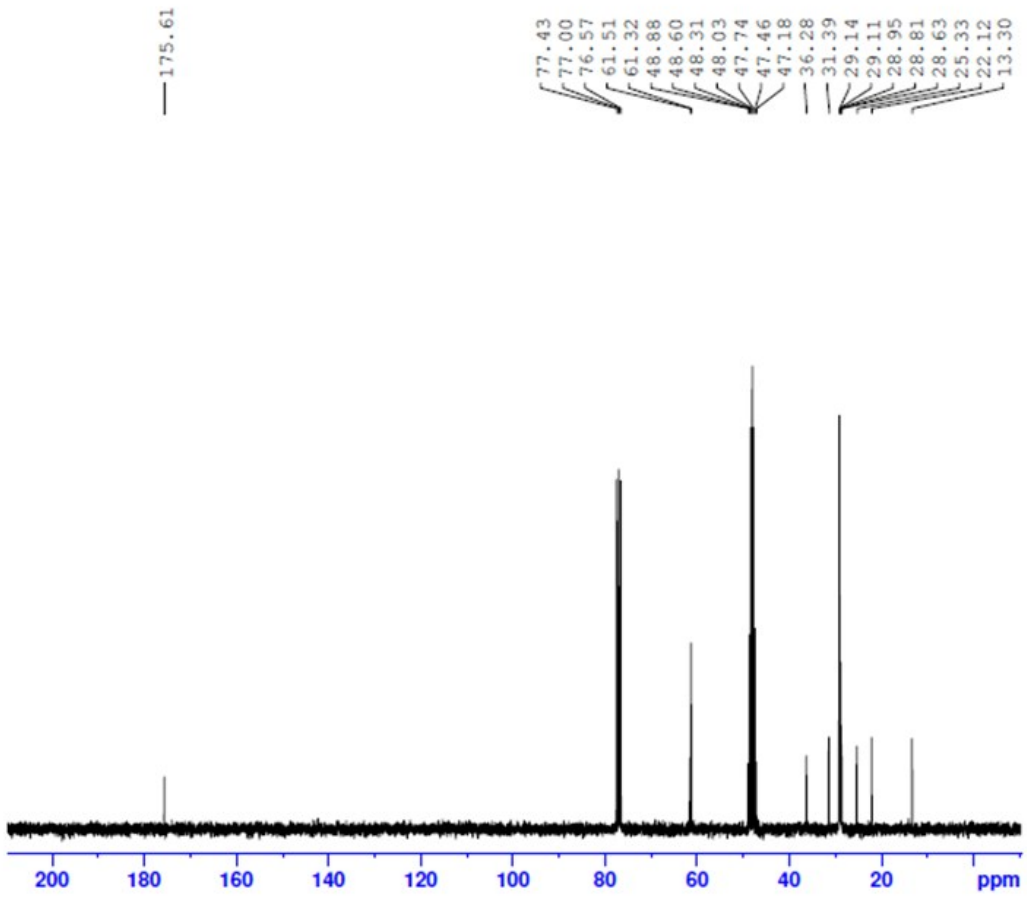

Fig. S15: ^{13}C-NMR spectra of N18TM

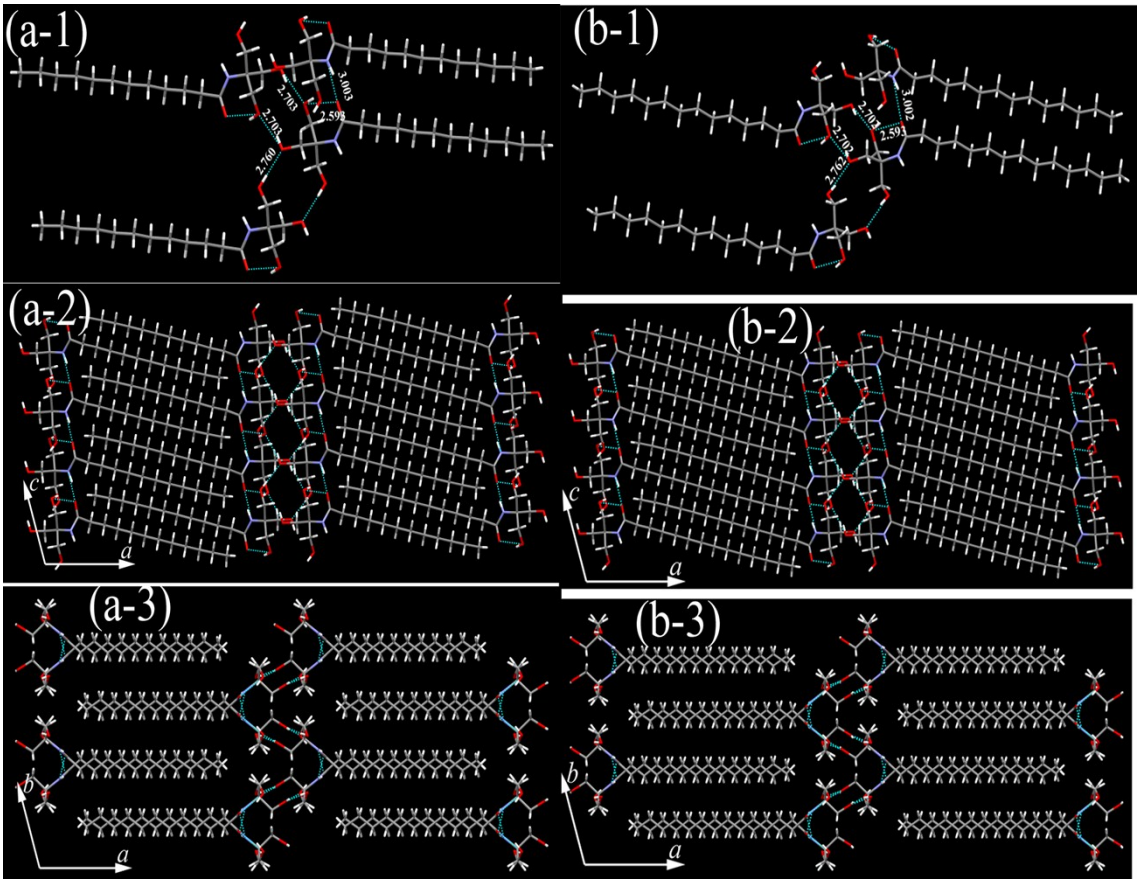

Fig. S16: Hydrogen bonding interaction of head group of (a-1) NATM-12 and (b-1) NATM-14 and fully interdigitized molecular packing along *ac*-plane of (a-2) NATM-12 and (b-2) NATM-14 and along *ab*-plane of (a-3) NATM-12 and (b-3) NATM-14. C (grey), N (blue), O (red), H (white). H-bonding interactions (broken line) distances are marked in Å.

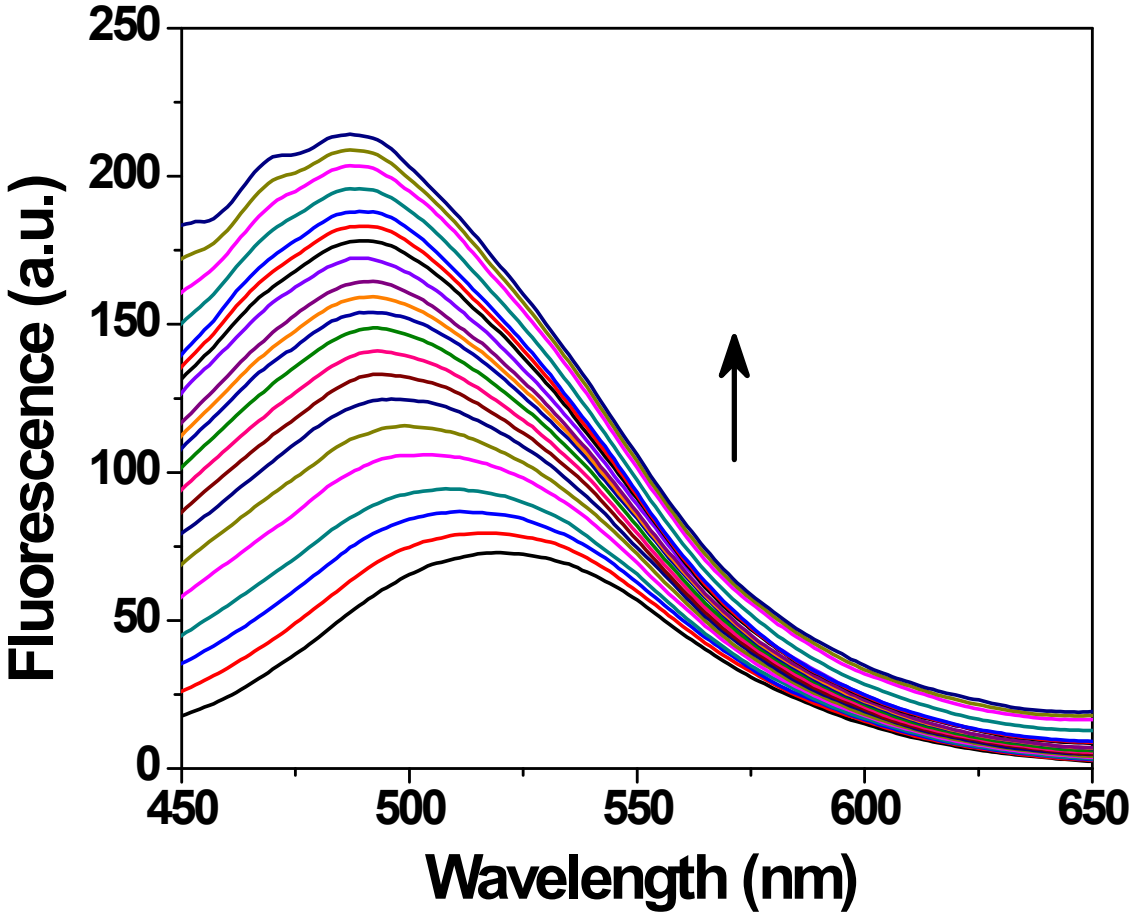

Fig. S17: Titration to determine critical aggregation concentration. ANS fluorescence spectra were measured in the presence of increasing concentration of N10TM. It is clear that ANS emission maximum undergoes blue shift with concomitant increase in intensity. Arrow indicates the addition of N10TM. All the solution are prepared in 10 mM phosphate buffer, pH-7.4. Excitation wavelength - 370 nm.

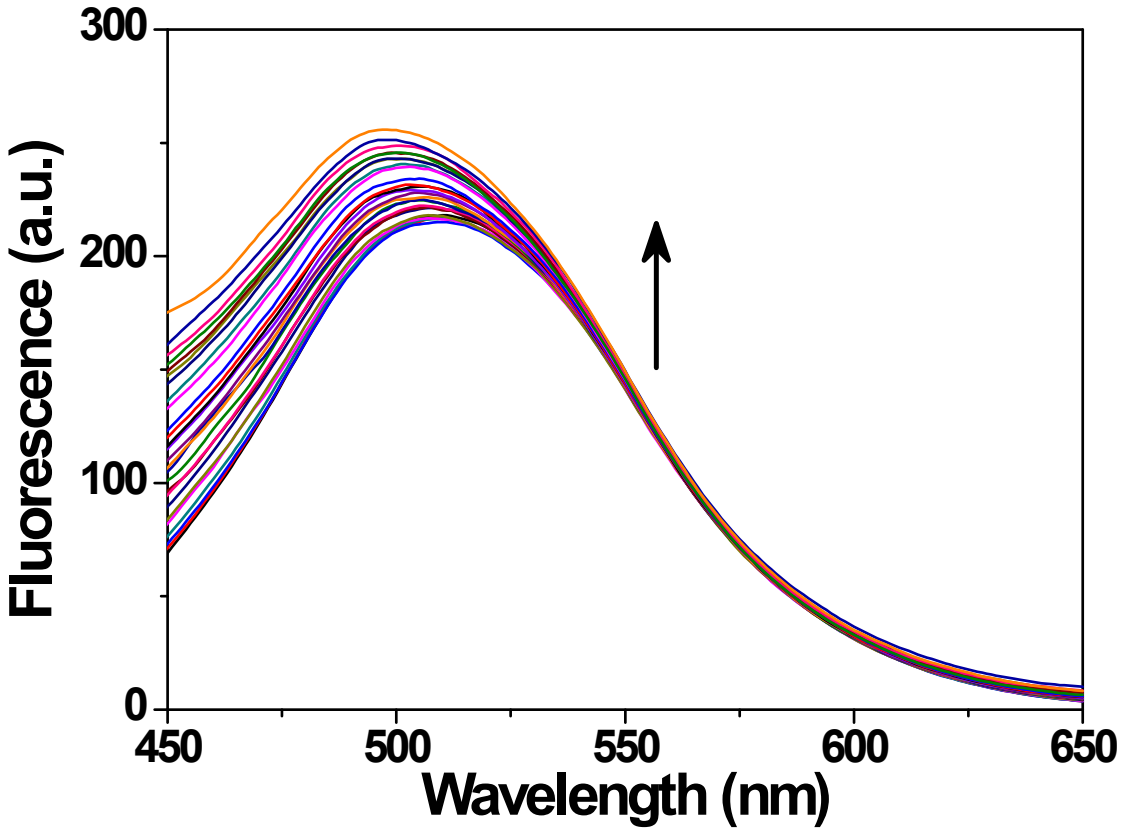

Fig. S18: Effect of isoproponol on CAC determination. ANS solutions prepared with 25 % isoproponol were titrated with N10TM. Arrow indicates the addition of N10TM. As noted the fluorescence of ANS increases slightly with small blue shift, suggesting the absence of self-assembly. The small changes in intensity and wavelength can be attributed to change in solvent polarity.

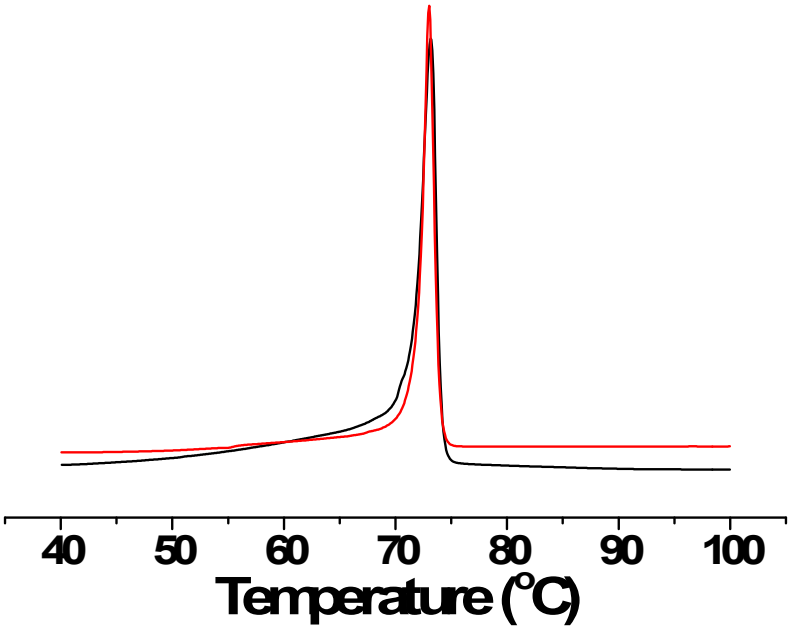

Fig. S19: Effect of sodium chloride on the phase transition of N10TM. Black-no sodium chloride, Red line – 1 M NaCl. As noted from this calorigram, NaCl has no significant influence of the phase behavior of N10TM.