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Supporting information

Assembly of encapsulated water in hybrid bisamides: Helical and zigzag water chains

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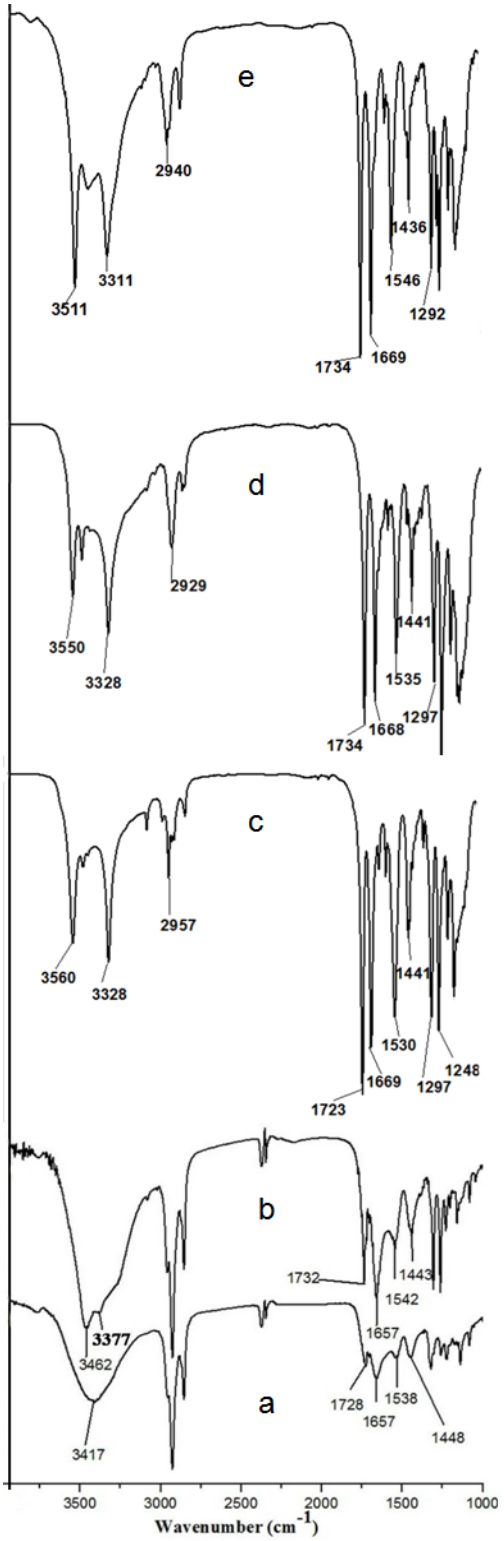


Figure S1. FT-IR spectra of (a) as synthesized bisamide **1.** (b), (c), (d) and (e) FT-IR spectra of solid obtained from methanol-water solution of bisamides **1-4** respectively.

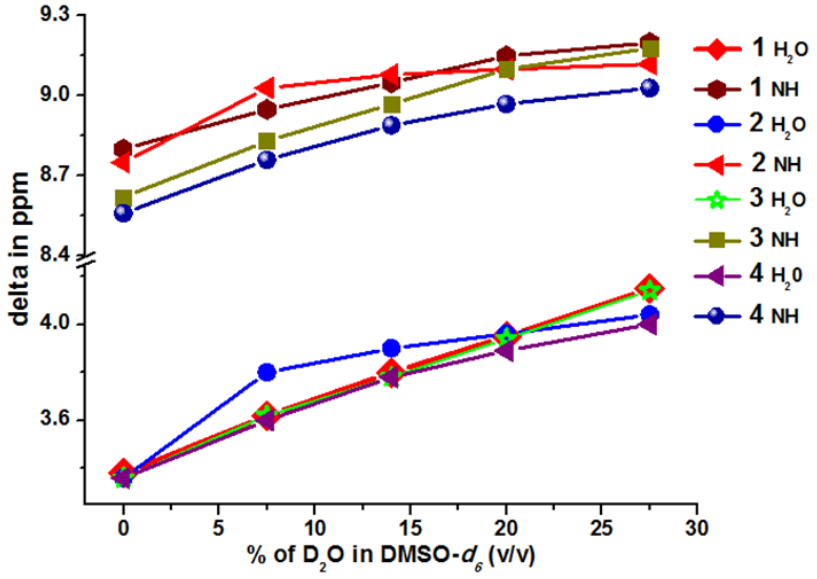


Figure S2. Plot of solvent dependence of NH chemical shifts for bisamides **1** - **4** and OH of water at varying concentrations of D_2O in $(CD_3)_2SO$ solutions.

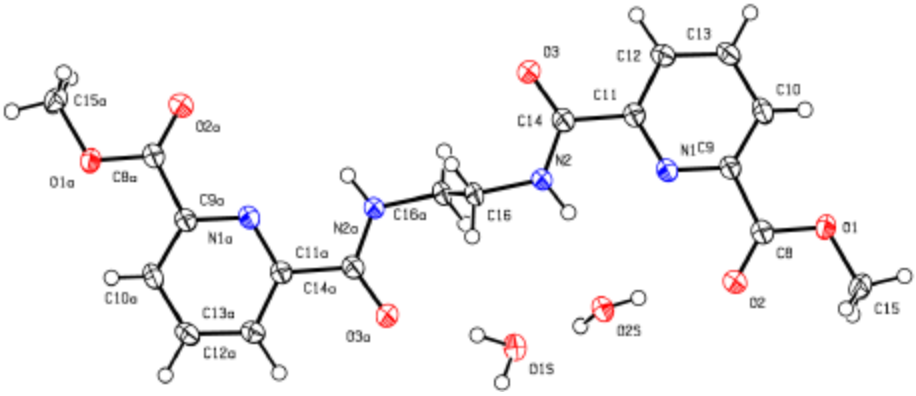


Figure S3: ORTEP diagram of bisamide **1** showing the atomic numbering scheme. Ellipsoids are drawn at the 50 % probability level. Equivalent position is invoked by the additional "a" letters in the atom labels is (-x,-y,-z).

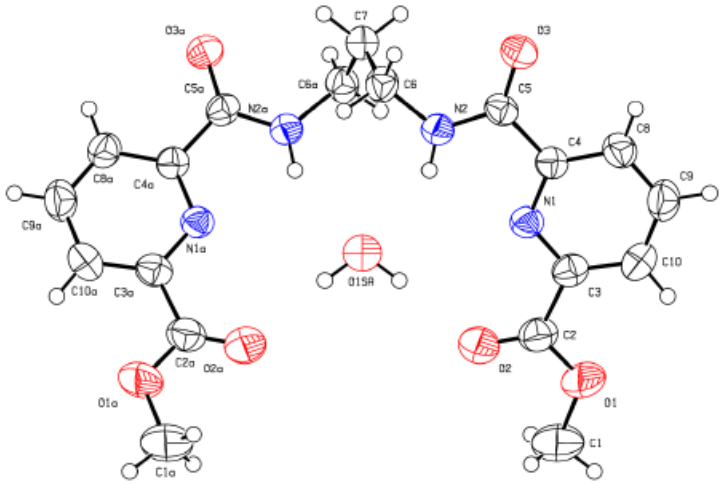


Figure S4: ORTEP diagram of bisamide **2** showing the atomic numbering scheme. Ellipsoids are drawn at the 50 % probability level. Equivalent position is invoked by the additional "a" letters in the atom labels is (2-x, 2-y, z).

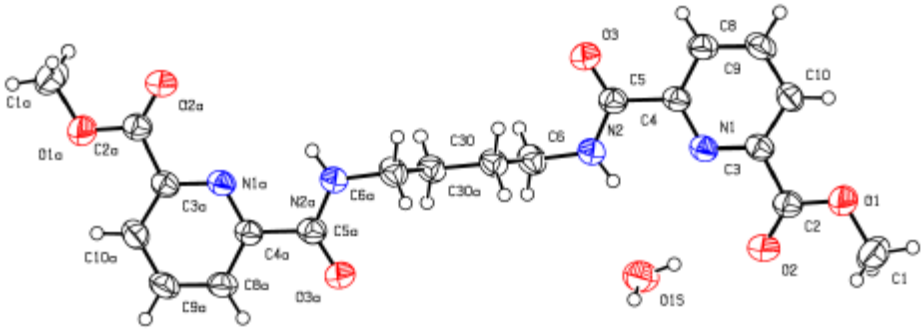


Figure S5: ORTEP diagram of bisamide **3** showing the atomic numbering scheme. Ellipsoids are drawn at the 50 % probability level. Equivalent position is invoked by the additional "a" letters in the atom labels is (-x, -1-y, -z).

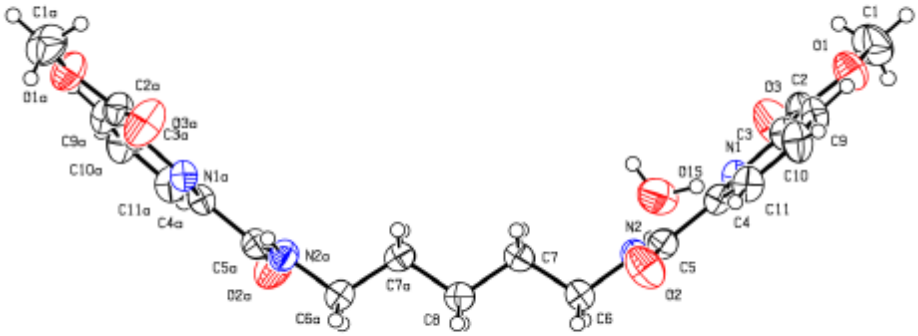


Figure S6: ORTEP diagram of bisamide **4** showing the atomic numbering scheme. Ellipsoids are drawn at the 50 % probability level. Equivalent position is invoked by the additional "a" letters in the atom labels is (1-x, y, -z).

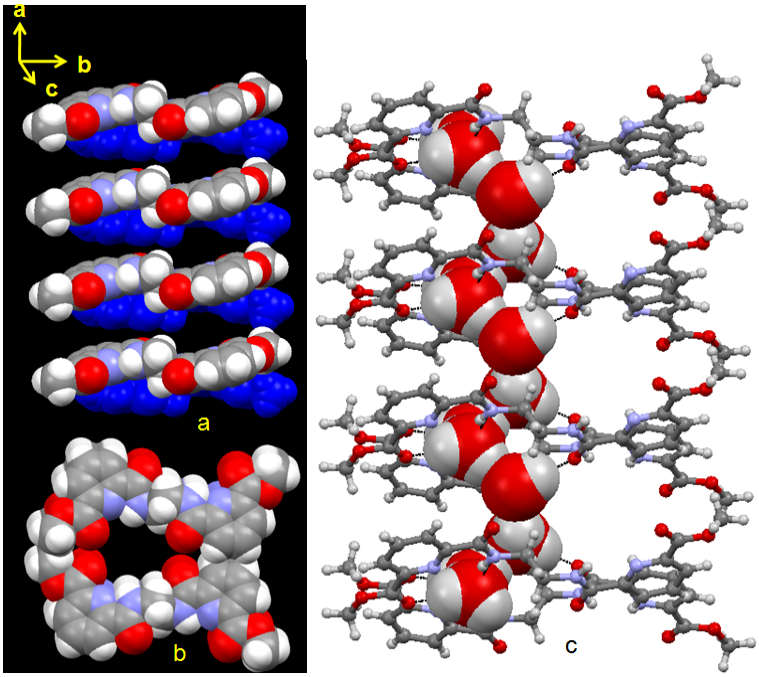


Figure S7: (a) and (b) The side and top view of superstructure of bisamide **1**. The water molecules have been omitted for clarity. (c) The side view of the packing presentation of water channel of bisamide **1**. Hydrogen bonds are shown as black dotted lines.

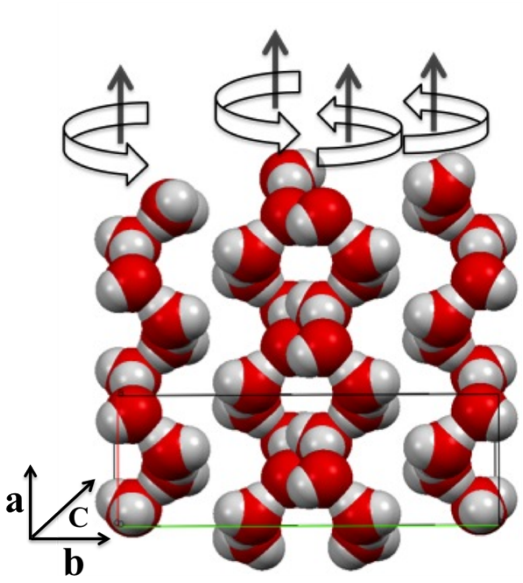


Figure S8: Packing of four helical water chains in the unit cell showing both left and right handedness. The molecules of bisamide **1** have been omitted for clarity.

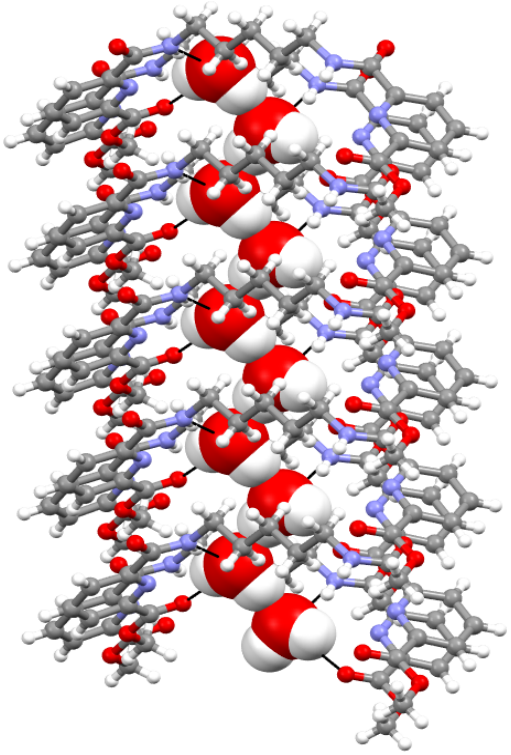


Figure S9: The side view of the packing presentation of water channel of bisamide **4**. Hydrogen bonds are shown as black dotted lines.

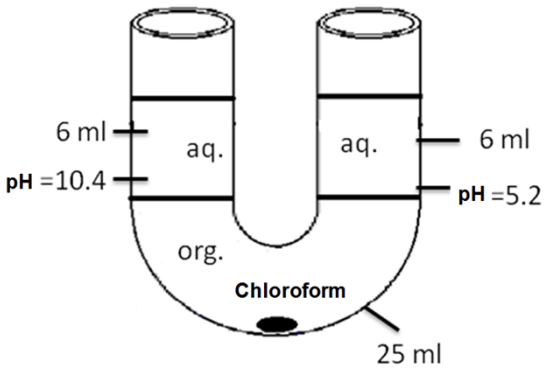


Figure S10: U-tube experiment setup for the determination of proton transport rates under pH gradients.

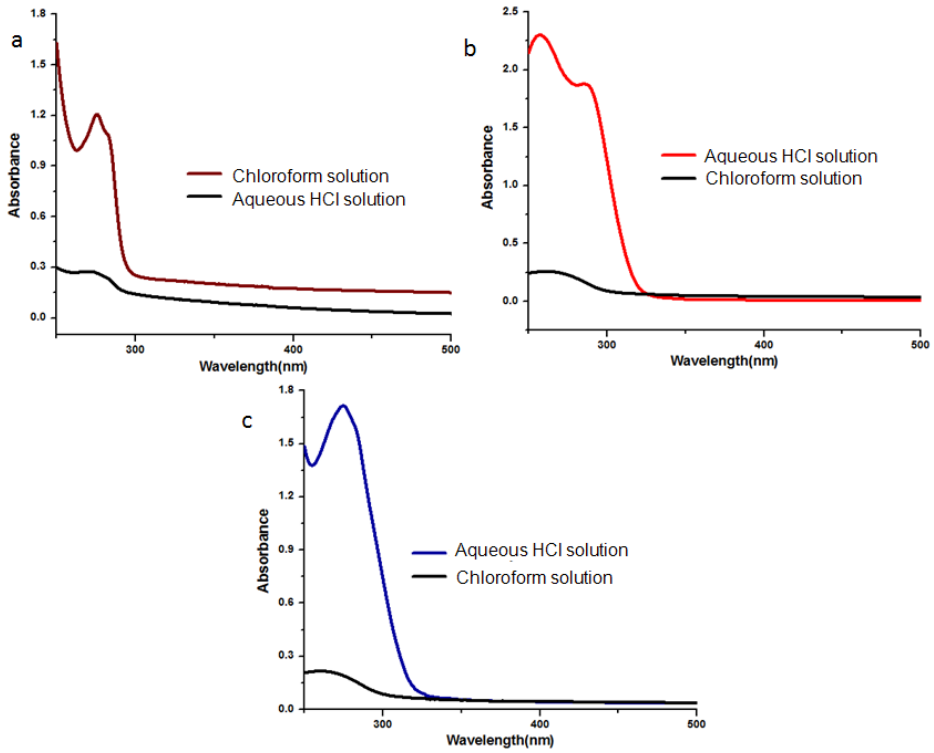


Figure S11: UV-Vis spectra of aqueous HCl part and chloroform part after the U-tube experiments of (a) bisamide **1**, (b) bisamide **2** and (c) bisamide **4** showing that the solubility of bisamide.Hcl in aqueous HCl part is not significant with respect to the solubility of bisamides in chloroform part. Also the solubility in aqueous HCl part is very close for bisamides **1**-**4**.

Table 1: Result of transport experiment of bisamides.

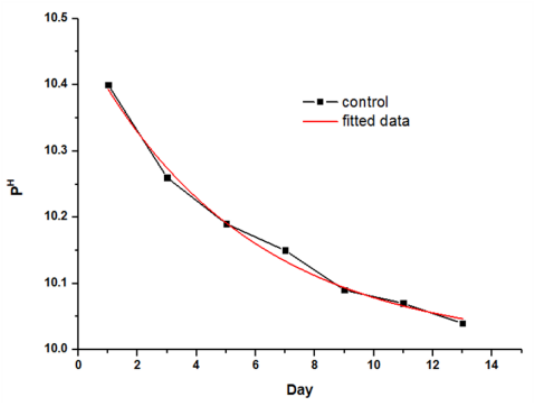
SL. no.	bisamide	Conc.(mmol)	K_1[c] (day^{-1})
1	1	$6.0x10^{-3}$	$6.410x10^{-1}$
2	2	$6.0x10^{-3}$	$1.836x10^{-1}$
3	3	$6.0x10^{-3}$	$2.452x10^{-1}$
4	4	$6.0x10^{-3}$	$26.060x10^{-1}$

[c] Data obtained by fitting the curve to eqn (1) with Origin 8. For control, $K_2 = 1.823x10^{-1}$ day^{-1}.

Rate constant calculations:

Control experiment was carried out without bisamide. The transport rate of solvent (k_{CHCl3}= $1.823x10^{-1}$ day^{-1}) was determined by fitting the curve obtained (t = 1 day to t = 13 day) to Equation (1) using Origin 8 software, where $(1/t1) = k_{CHCl3}$.

$$y = y_0 + Ae^{-x/t} \quad \text{.....(1)}$$

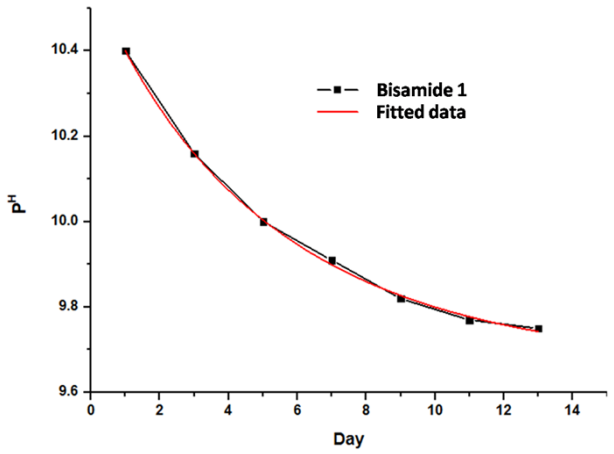


Model	ExpDec1		
Equation	Y= A1*exp(-x/t1) + y0		
Reduced chi-sqr	1.46094E-4		
Adj. R-Squ	0.99078		
		Value	Standard E
$CDCl_3$	y0	10.00296	0.02343
$CDCl_3$	A1	0.46898	0.0197
$CDCl_3$	t1	5.48398	0.79876

Figure S12: Fitting of pH vs time plot for $CHCl_3$ using Origin 8 and the table shows fitting results.

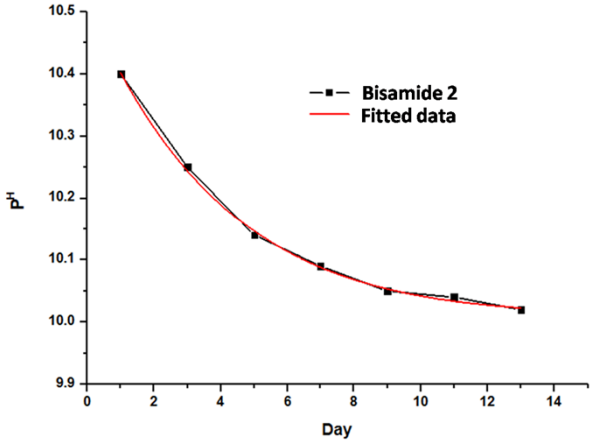
Proton transport rates through the water channel of bisamides were determined by fitting the curves obtained to Eqn. (2), using Origin 8 software, where $(1/t_1) = k_{CHCl3} = 1.823x10^{-1}$; $(1/t_2) = k_{bisamide}$. Only the value for t_1 was kept fixed at 5.484 for the fits.

$$y = y_0 + A_1e^{-x/t_1} + A_2e^{-x/t_2} \quad \text{.....(2)}$$



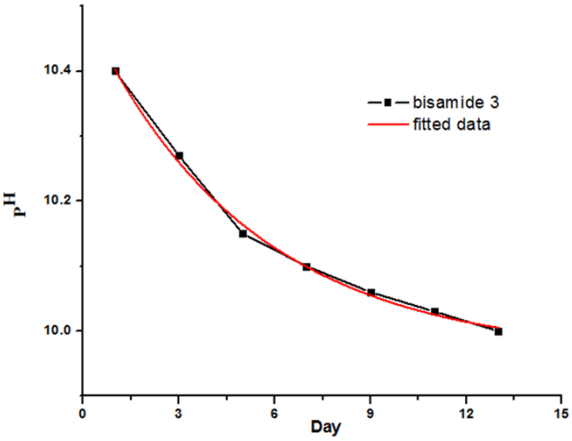
Model	ExpDec2		
Equation	Y= A1*exp(-x/t1) + A2*exp(-x/t2) + y0		
Reduced chi-sqr	9.68421E-5		
Adj. R-Squ	0.99827		
		Value	Standard E
Bisamide 1	y0	9.66467	0.01526
Bisamide 1	A1	0.83723	0.09603
Bisamide 1	t1	5.48398	0
Bisamide 1	A2	0.07157	0.05196
Bisamide 1	t2	1.55741	3.35797

Figure S13: Fitting of pH vs time plot for bisamide **1** and the fitting results table.



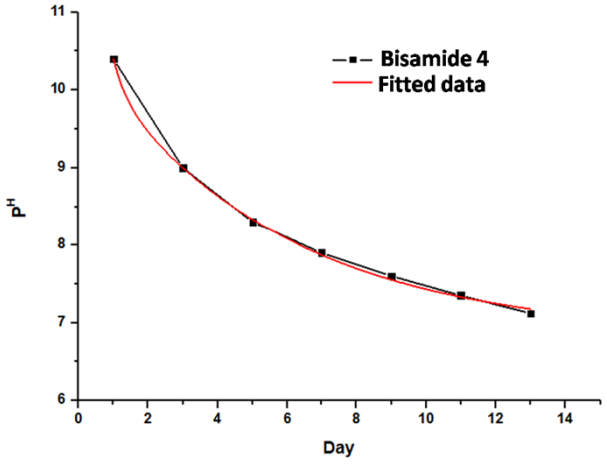
Model	ExpDec2		
Equation	Y= A1*exp(-x/t1) + A2*exp(-x/t2) + y0		
Reduced chi-sqr	5.03214E-5		
Adj. R-Squ	0.99737		
		Value	Standard E
Bisamide 2	y0	10.01975	0.06629
Bisamide 2	A1	-27.53099	4421.0971
Bisamide 2	t1	5.48398	0
Bisamide 2	A2	28.02517	4421.01856
Bisamide 2	t2	5.44584	5.97705

Figure S14: Fitting of pH vs time plot for bisamide **2** using Origin 8 and the table showing fitting results.



Model	ExpDec2		
Equation	Y= A1*exp(-x/t1) + A2*exp(-x/t2) + y0		
Reduced chi-sqr	1.24238E-4		
Adj. R-Squ	0.994		
		Value	Standard E
Bisamide 3	y0	9.96134	0.06217
Bisamide 3	A1	0.41416	3.06281
Bisamide 3	t1	5.48398	0
Bisamide 3	A2	0.12244	2.97863
Bisamide 3	t2	4.07852	27.50548

Figure S15: Fitting of pH vs time plot for bisamide **3** using Origin 8 and the table showing fitting results.



Model	ExpDec2		
Equation	Y= A1*exp(-x/t1) + A2*exp(-x/t2) + y0		
Reduced chi-sqr	0.00255		
Adj. R-Squ	0.99805		
		Value	Standard E
Bisamide 4	y0	6.82707	0.05082
Bisamide 4	A1	3.75027	0.20751
Bisamide 4	t1	5.48398	0
Bisamide 4	A2	6.06274	113.49905
Bisamide 4	t2	0.38379	2.79269

Figure S16: Fitting of pH vs time plot for bisamide **4** using Origin 8 and the table showing fitting results.

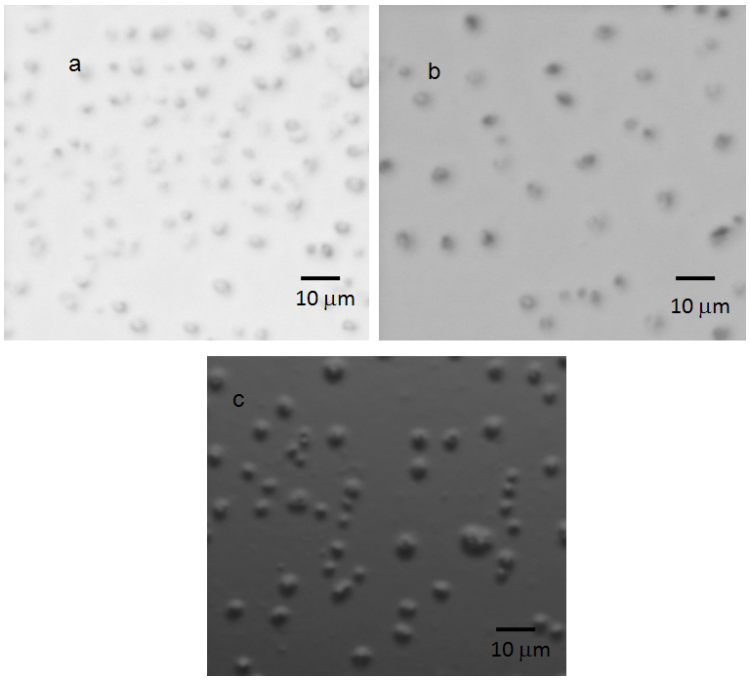


Figure S17: The microscopic image of (a) bisamide 2, (b) bidamide 3 and (c) bisamide 4 from dry methanol solution showing polydisperse microspheres.

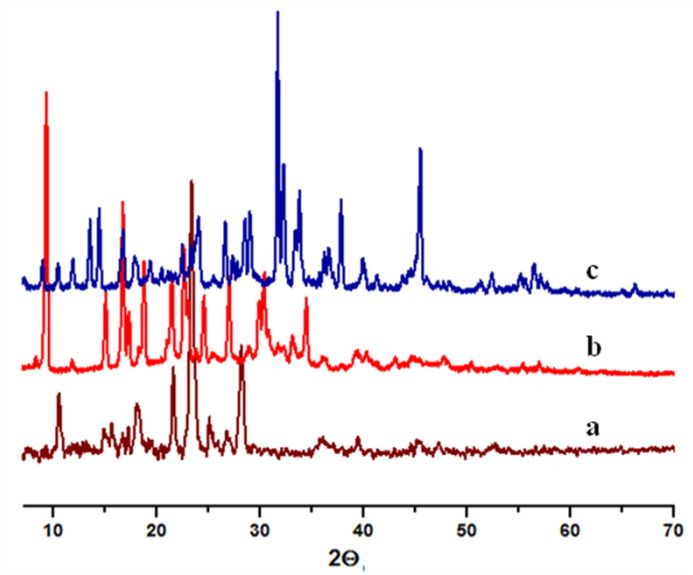
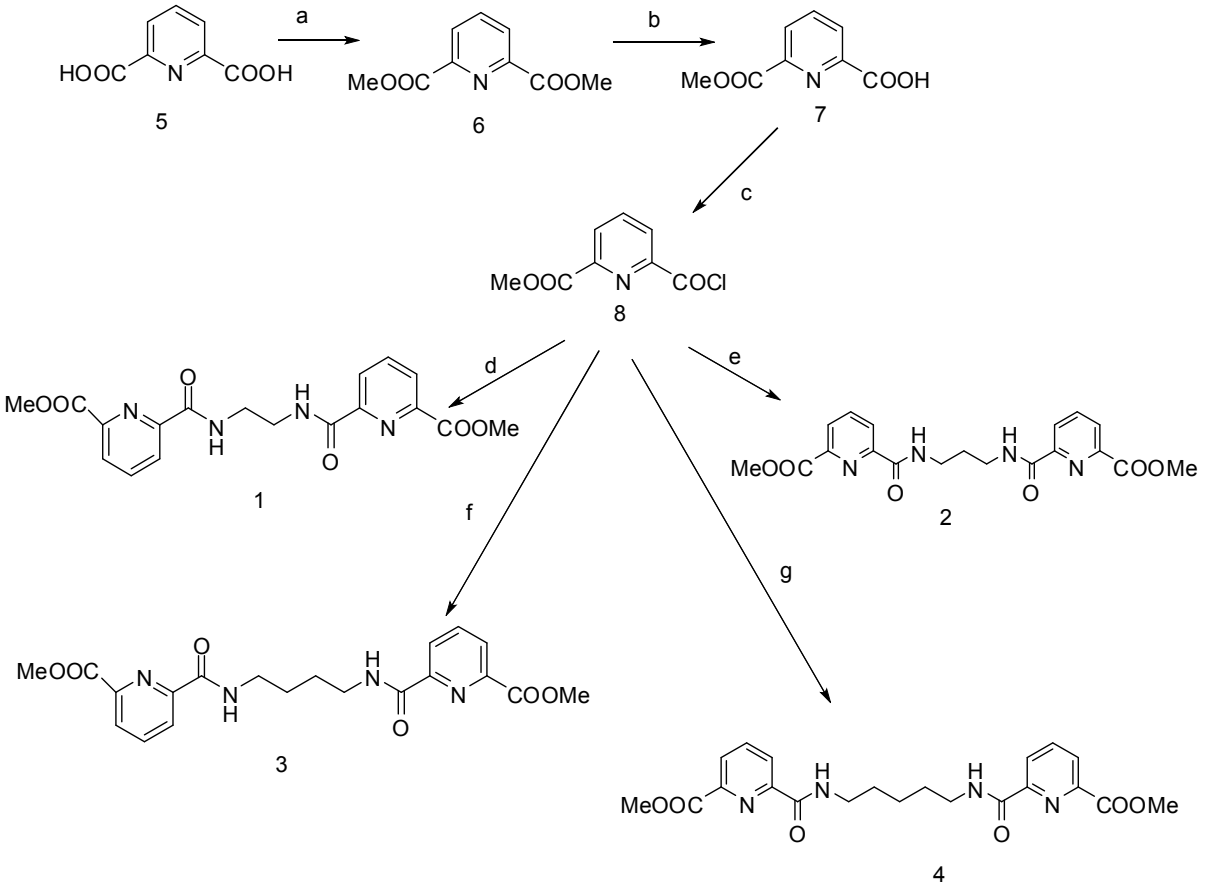


Figure S18: PXRD pattern of (a) bisamide 1 (b) bisamide 2 (c) bisamide 3.



Scheme 1. The synthesis of bisamides **1-4**. *Reagents and conditions*: (a) MeOH, $SOCl_2$, 0°C; (b) NaOH (1 eqv.), MeOH: H_2O (9:1); (c) $SOCl_2$, reflux; (d) $H_2NCH_2CH_2NH_2$, Et_3N, DCM; (e) $H_2NCH_2CH_2CH_2NH_2$, Et_3N, DCM; (f) $H_2NCH_2CH_2CH_2CH_2NH_2$, Et_3N, DCM; (g) $H_2NCH_2CH_2CH_2CH_2CH_2NH_2$, Et_3N, DCM.

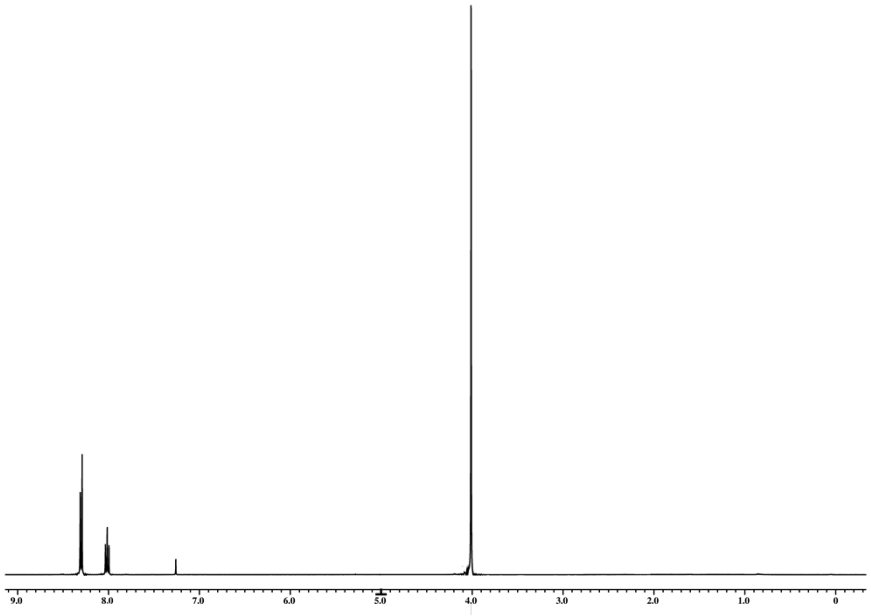


Figure S19: ^{1}H NMR ($CDCl_3$, 500MHz, δ_{ppm}) spectra of compound **6.**

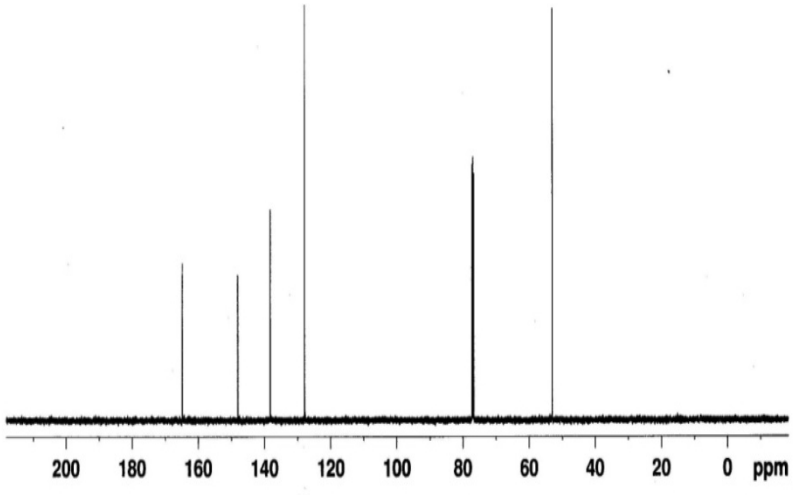


Figure S20: ^{13}C NMR ($CDCl_3$, 125MHz, δ_{ppm}) spectra of compound **6.**

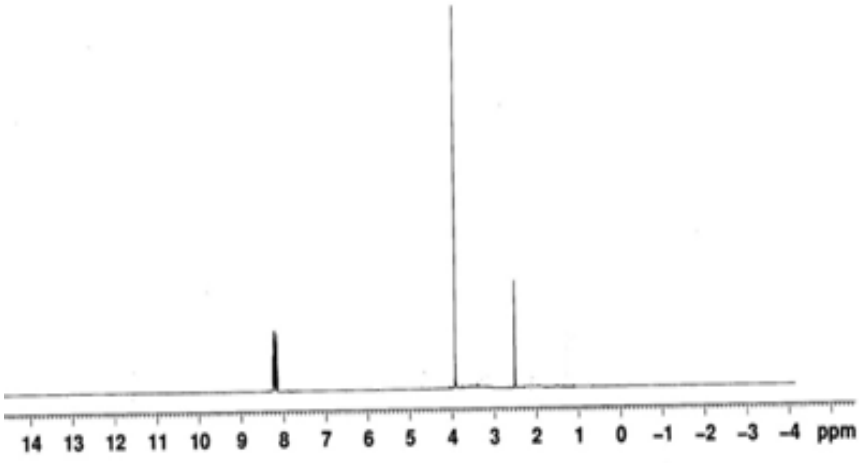


Figure S21: ^{1}H NMR (DMSO-*d6*, 500 MHz, δ_{ppm}) spectra of compound **7**.

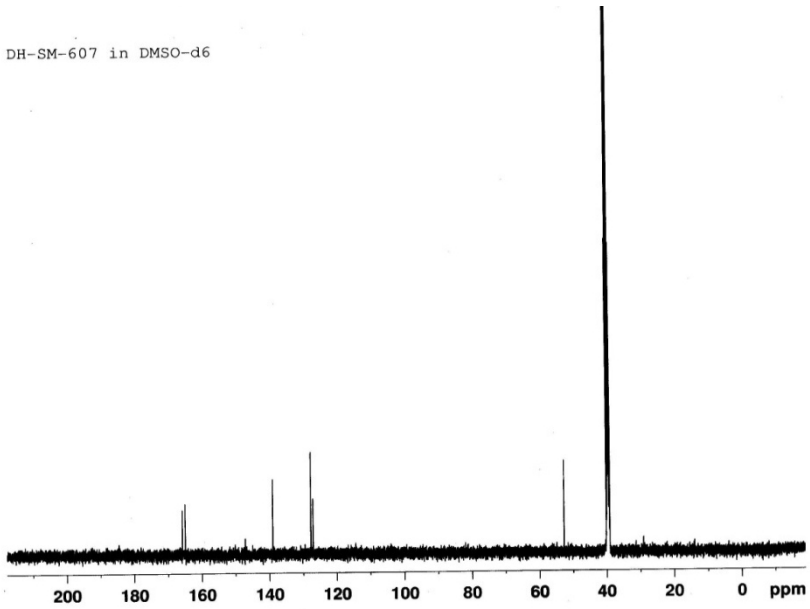


Figure S22: ^{13}C NMR (DMSO-*d6*, 125MHz, δ_{ppm}) spectra of compound **7.**

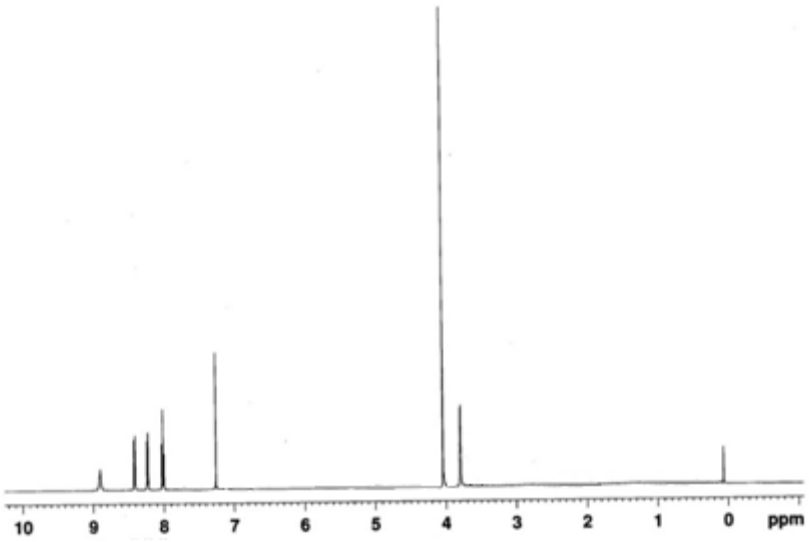


Figure S23: ^{1}H NMR ($CDCl_3$, 500MHz, δ_{ppm}) spectra of bisamide **1.**

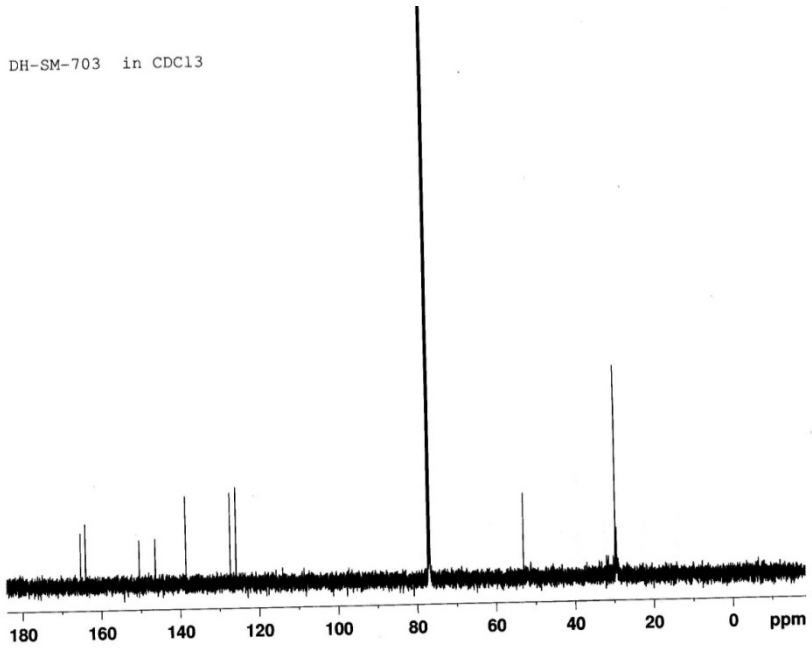


Figure S24: ^{13}C NMR ($CDCl_3$, 125MHz, δ_{ppm}) spectra of bisamide **1.**

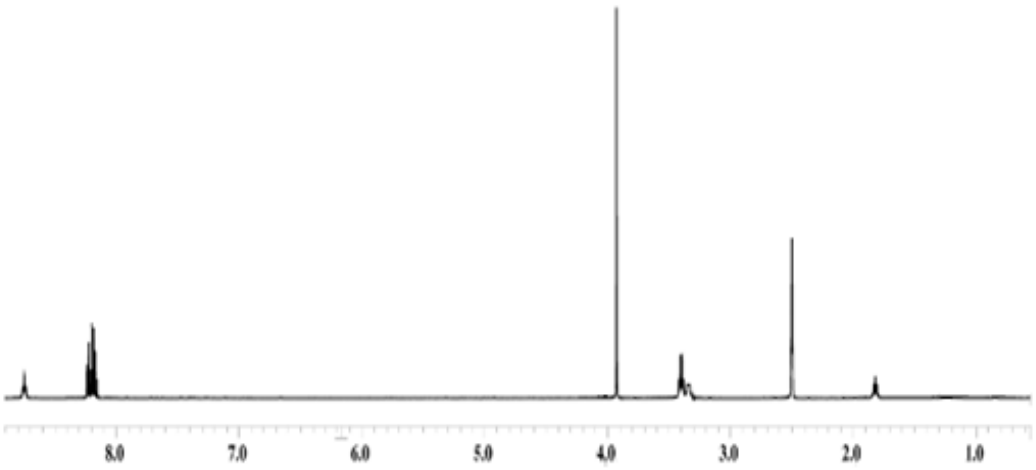


Figure S25: ^{1}H NMR (DMSO-d_6, 500MHz, δ_{ppm}) spectra of bisamide **2.**

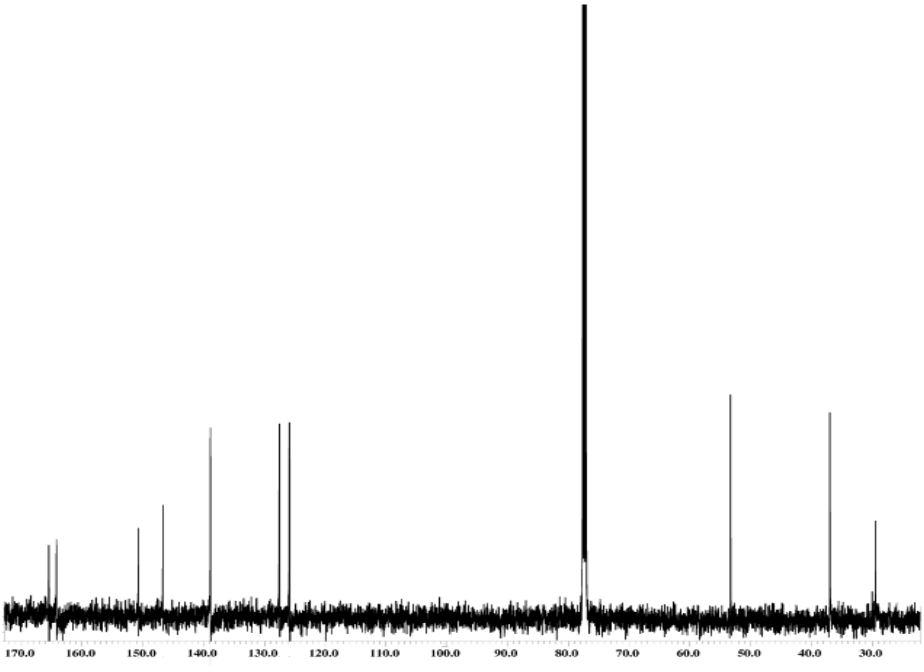


Figure S26: ^{13}C NMR ($CDCl_3$, 125MHz, δ_{ppm}) spectra of bisamide **2.**

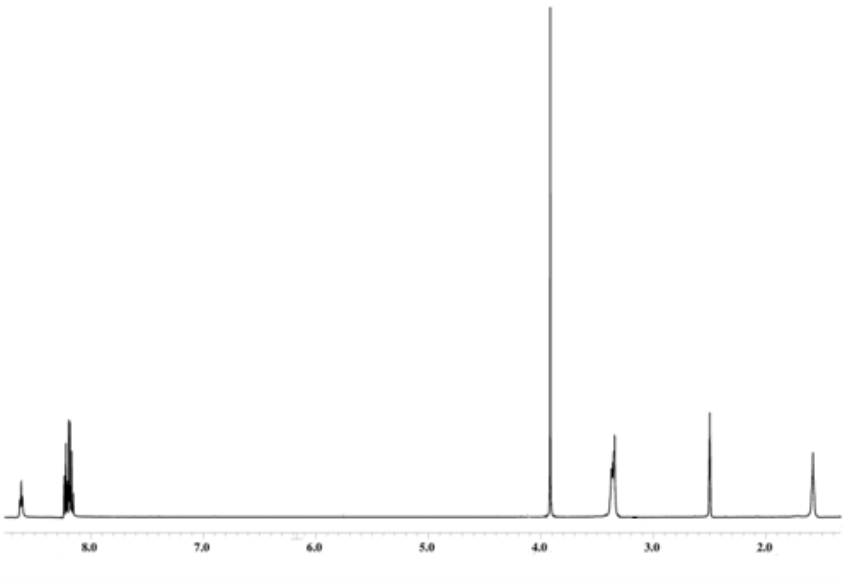


Figure S27: ^{1}H NMR (DMSO-d_6, 500MHz, δ_{ppm}) spectra of bisamide **3.**

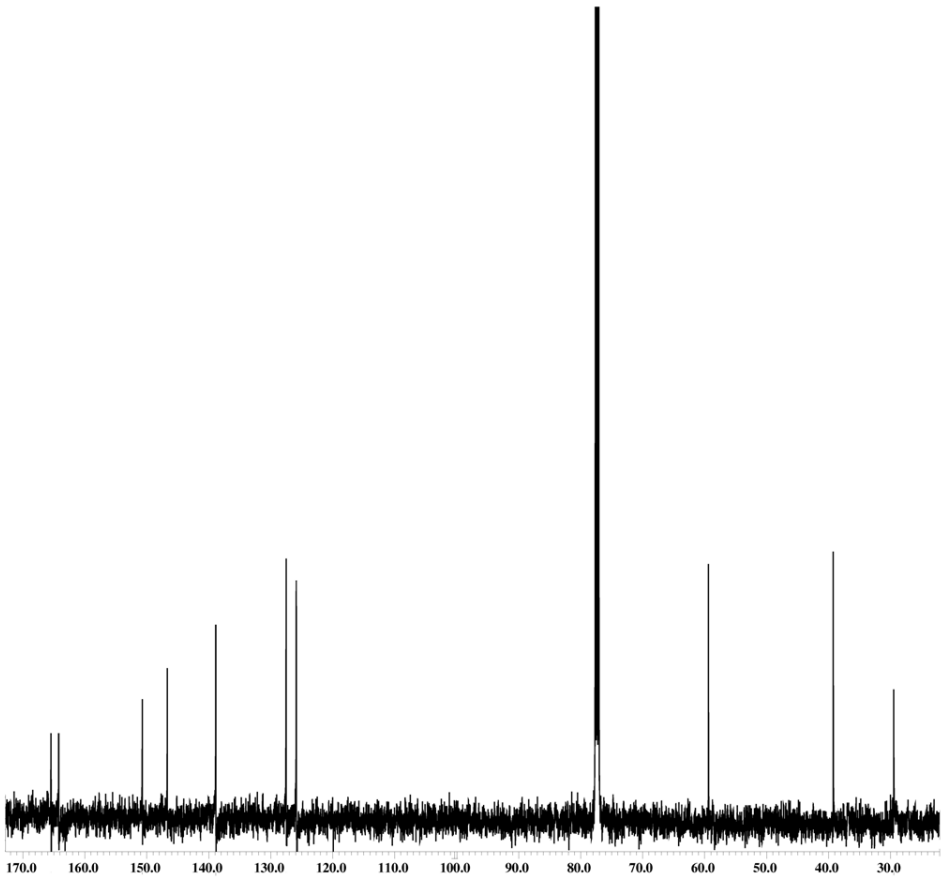


Figure S28: ^{13}C NMR ($CDCl_3$, 125MHz, δ_{ppm}) spectra of bisamide **3.**

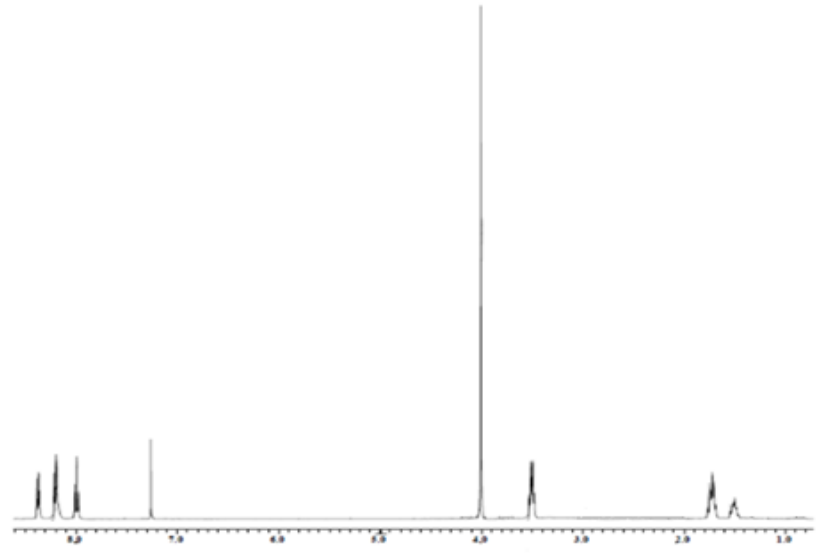


Figure S29: ^{1}H NMR ($CDCl_3$, 500MHz, δ_{ppm}) spectra of bisamide **4.**

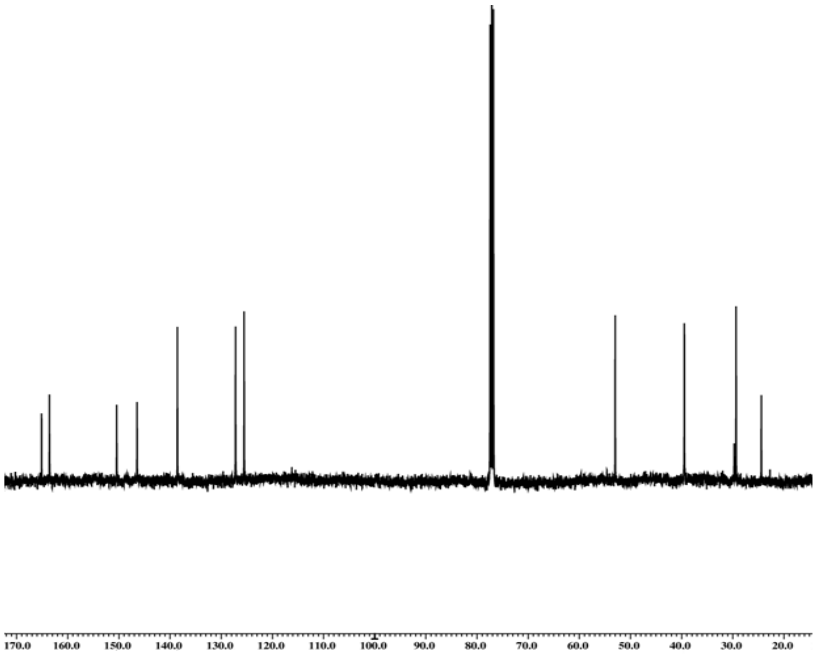


Figure S30: ^{13}C NMR ($CDCl_3$, 125MHz, δ_{ppm}) spectra of bisamide **4.**