Supplementary Material (ESI) for Soft Matter

Solvent-free Construction of Self-Assembled 1D Nanostructures from Low-Molecular-Weight Organogelators: Sublimation vs. Gelation

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Electronic Supporting Information

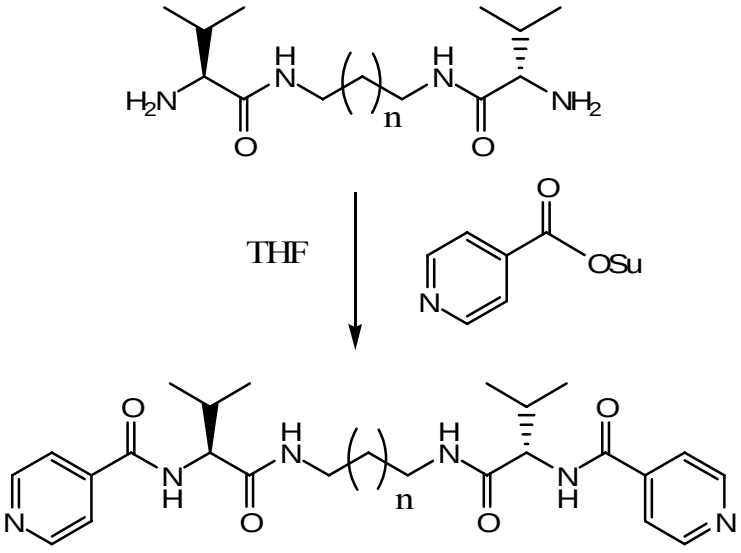
-Synthetic details
-Characterization of new compounds.
-Experimental methodology.
-Additional WAXD graphs.
-Additional DSC graphs.
-IR spectra.

Synthesis of activated (iso)nicotinic acids.

10 g (0.081 mol) of (iso)nicotinic acid together with 9.34 g (0.081 mol) of N-hydroxysuccinimide were suspended in 50 mL dry THF under nitrogen at 0 ºC. 16.43 g (0.1624 mol) of Et_3N were added and, finally, 16.75 g (0.081 mol) of dicyclohexyl carbodiimide (DCC) in 40 mL of dry THF were added dropwise slowly. The mixture was stirred overnight at room temperature. Afterwards, the reaction was left for 2 hours at -20 ºC, filtered off and washed with cool THF to remove the dicyclohexylurea. The solvent was vacuum evaporated and the solid crude was recrystallized from 2-propanol. The product X was obtained after filtration as white crystals.

Activated Nicotinic Acid: White solid (84% yield). ^{1}HNMR (300 MHz, [D_6]DMSO, 30 ºC): δ = 9.20 (d, 1H, J=1.95Hz), 8.96 (dd, 1H, J1=1.78 Hz, J2=4.80Hz), 8.44 (dt, 1H, J1=.178 Hz, J2=7.99 Hz), 7.68 (dd, 1H, J1=3.02 Hz, J2=4.44 Hz), 2.92 (s, 4H) ppm. ^{13}CNMR (75 MHz, [D_6]DMSO, 30 ºC): δ = 170.79, 161.65, 156.26, 151.11, 138.38, 125.13, 121.76, 26.26 ppm.
Activated Isonicotinic Acid: White solid (76% yield). ^{1}HNMR (300 MHz, [D_6]DMSO, 30 ºC): δ = 8.93 (dd, 2H, J1=5.96 Hz, J2=2.13 Hz), 7.97 (dd, 2H, J1=5.96 Hz, J2=2.13 Hz), 2.91 (s, 4H) ppm. ^{13}CNMR (75 MHz, [D_6]DMSO, 30 ºC): δ = 170.64, 161.75, 152.11, 132.53, 123.44, 26.28 ppm.

Synthesis of 1a-h and 2a-h.



1 eq of the diamine[1] was dissolved in 100 mL of dry THF under nitrogen at 50 ºC and 2.2 eq of the corresponding activated (iso)nicotinic acid were added dropwise in 100 mL of dry THF. The reaction was left at 50 ºC for 16 h. Then, the solvent was vacuum evaporated to obtain a white-yellowish solid which was purified either washing it thoroughly with cool acetonitrile or dissolving it with 20 mL of 0.3 M HCl(aq), filtering it and adding NaOH(s) until pH > 10 to the filtrate in an ice bath and filtering the precipitated. The product was obtained as a white to an off-white solid after column chromatography purification (Silica gel, CH_2Cl_2:MeOH from 1% to 5%).

[1] Miravet, J. F. et al. *J. Am. Chem. Soc.* **2003**, *125*, 6677-6686.

1a: White solid (58% yield). M. p. 302 - decomp. ºC. ^{1}HNMR (300 MHz, [D_6]DMSO, 30 ºC): δ = 9.03 (s, 2H), 8.69 (d, 2H, J=4.60 Hz), 8.51 (d, 2H, J=8.48 Hz), 8.21 (d, 2H, J=7.97 Hz), 8.08 (s, 2H), 7.48 (m, 2H), 4,24 (t, 2H, J=8.04 Hz), 3.17 (m, 4H), 2.10 (m, 2H), 0.89 (t, 12H, J=6.24 Hz) ppm. ^{13}CNMR (75 MHz, [D_6]DMSO, 30 ºC): δ = 171.68, 165.84, 152.51, 136.02, 130.43, 123.95, 59.87, 38.92, 30.58, 20.00, 19.48 ppm. IR: ν = 3273, 3082, 2957, 1628, 1530 cm^{-1}. HRMS (ESI-TOF+): Calcd. for $C_{24}H_{32}N_6NaO_4^+$ [M + Na^+] 491.2383; found 491,2378 (Δ = 0,9 ppm).

2a: White solid (54% yield). M. p. 296 - decomp. ºC. ^{1}HNMR (300 MHz, [D_6]DMSO, 30 ºC): δ = 8.71 (d, 4H, J=5.07 Hz), 8.60 (t, 2H, J=8.11 Hz), 8.13 (d, 2H, J=7.66 Hz), 7.80 (d, 4H, J=5.07 Hz), 4.27 (t, 2H, J=8.04 Hz), 3.20 (m, 4H), 2.10 (m, 2H), 0.90 (m, 12H) ppm. ^{13}CNMR (75 MHz, [D_6]DMSO, 30 ºC): δ = 171.52, 165.72, 150.73, 141.82, 122.28, 59.95, 38.89, 30.57, 19.97, 19.47 ppm. IR: ν = 3273, 3078, 2955, 1634, 1528 cm^{-1}. HRMS (ESI-TOF+): Calcd. for $C_{24}H_{32}N_6NaO_4^+$ [M + Na^+] 491.2383; found 491,2383 (Δ = 0,1 ppm).

1b, 2b: (*Chem. Commun*. **2005**, 5796)

1c: White solid (71% yield). M. p. 270 - 272 ºC. ^{1}HNMR (300 MHz, [D_6]DMSO, 30 ºC): δ = 9.02 (s, 2H), 8.70 (d, 2H, J=4.60 Hz), 8.50 (d, 2H, J=8.48 Hz), 8.22 (d, 2H, J=7.97 Hz), 8.04 (s, 2H), 7.50 (m, 2H), 4,26 (t, 2H, J=8.04 Hz), 3.12 (m, 4H), 2.10 (m, 2H), 1.43 (m, 4H), 0.90 (m, 12H) ppm. ^{13}CNMR (75 MHz, [D_6]DMSO, 30 ºC): δ = 171.31, 165.71, 152.50, 138.98, 130.49, 123.96, 59.83, 38.90, 30.70, 27.20, 20.00, 19.56 ppm. IR: ν = 3279, 3084, 2959, 1630, 1532 cm^{-1}. HRMS (ESI-TOF+): Calcd. for $C_{26}H_{36}N_6NaO_4^+$ [M + Na^+] 519.2696; found 519,2687 (Δ = 1,8 ppm).

2c: White solid (69% yield). M. p. 285 - 290 ºC. ^{1}HNMR (300 MHz, [D_6]DMSO, 30 ºC): δ = 8.70 (d, 4H, J=5.07 Hz), 8.56 (t, 2H, J=8.11 Hz), 8.05 (d, 2H, J=7.66 Hz), 7.78 (d, 4H, J=5.07 Hz), 4.23 (t, 2H, J=8.11 Hz), 3.11 (m, 4H), 2.09 (m, 2H), 1.41 (m, 4H), 0.90 (m, 12H) ppm. ^{13}CNMR (75 MHz, [D_6]DMSO, 30 ºC): δ = 171.17, 165.62, 150.73, 141.89, 122.25, 59.93, 38.89, 30.67, 27.17, 19.96, 19.55 ppm. IR: ν = 3288, 3080, 2958, 1633, 1551 cm^{-1}. HRMS (ESI-TOF+): Calcd. for $C_{26}H_{36}N_6NaO_4^+$ [M + Na^+] 519.2696; found 519,2682 (Δ = 2,6 ppm).

1d: White solid (65% yield). M. p. 256,1 - 258,3 ºC. ^{1}HNMR (300 MHz, [D_6]DMSO, 30 ºC): δ = 9.02 (s, 2H), 8.69 (d, 2H, J=4.60 Hz), 8.51 (d, 2H, J=6.98 Hz), 8.20 (d, 2H, J=7.88 Hz), 8.02 (s, 2H), 7.48 (m, 2H), 4,26 (t, 2H, J=7.88 Hz), 3.05 (m, 4H), 2.11 (m, 2H), 1.41 (m, 4H), 1.27 (m, 2H), 0.91 (m, 12H) ppm. ^{13}CNMR (75 MHz, [D_6]DMSO, 30 ºC): δ = 171.29, 165.73, 152.48, 149.36, 135.98, 130.49, 123.95, 59.98, 39.08, 30.69, 29.29, 24.41, 19.98, 19.55 ppm. IR: ν = 3265, 3086, 2958, 1624, 1529 cm^{-1}. HRMS (ESI-TOF+): Calcd. for $C_{27}H_{38}N_6NaO_4^+$ [M + Na^+] 533.2855; found 533,2860 (Δ = 0,9 ppm).

2d: White solid (62% yield). M. p. 257,5 - 258,6 ºC. ^{1}HNMR (300 MHz, [D_6]DMSO, 30 ºC): δ = 8.71 (d, 4H, J=5.07 Hz), 8.57 (t, 2H, J=8.11 Hz), 8.03 (d, 2H, J=7.66 Hz), 7.78 (d, 4H, J=5.07 Hz), 4.23 (t, 2H, J=8.18 Hz), 3.05 (m, 4H), 2.10 (m, 2H), 1.40 (m, 4H), 1.28 (m, 2H), 0.91 (m, 12H) ppm. ^{13}CNMR (75 MHz, [D_6]DMSO, 30 ºC): δ = 171.12, 165.63, 150.74, 141.89, 122.26, 59.94, 39.08, 30.66, 29.27, 24.40, 19.95, 19.56 ppm. IR: ν = 3266, 3077, 2958, 1631, 1528 cm^{-1}. HRMS (ESI-TOF+): Calcd. for $C_{27}H_{38}N_6NaO_4^+$ [M + Na^+] 533.2855; found 533,2853 (Δ = 0,4 ppm).

2e. White solid (45% yield). M. p. 257,5 - 258,6 ºC. ^{1}HNMR (300 MHz, [D$_6$]DMSO, 30 ºC): δ = 8.71 (d, 4H, J=4.70Hz), 8.58 (t, 2H, J=9.52 Hz), 8.05 (d, 2H, J=7.74 Hz), 7.76 (d, 4H, J=4.37 Hz), 4.23 (t, 2H, J=7.74), 3.03 (m, 4H), 2.10 (m, 2H), 1.38 (m, 4H), 1.26 (m, 4H), 0.90 (m, 12H) ppm. ^{13}CNMR (75 MHz, [D$_6$]DMSO, 30 ºC): δ = 171.10, 165.65, 150.75, 141.95, 122.26, 59.96, 39.07, 30.70, 29.65, 26.69, 19.96, 19.57 ppm. IR: ν = 3291, 3077, 2955, 1630, 1534cm^{-1}

1f: White solid (91% yield). M. p. 240,2 - 241,9 ºC. ^{1}HNMR (300 MHz, [D$_6$]DMSO, 30 ºC): δ = 9.00 (s, 2H), 8.68 (d, 2H, J=5.31 Hz), 8.51 (d, 2H, J=8.53 Hz), 8.20 (d, 2H, J=8.18 Hz), 8.03 (t, 2H, J=4.09 Hz), 7.48 (m, 2H), 4,26 (t, 2H, J=8.18 Hz), 3.03 (m, 4H), 2.07 (m, 2H), 1.36 (m, 4H), 1.21 (m, 4H), 0.89 (t, 12H, J=5.26 Hz), 0.81 (m, 2H) ppm. ^{13}CNMR (75 MHz, [D$_6$]DMSO, 30 ºC): δ = 171.26, 165.70, 152.50, 149.36, 135.98, 130.49, 123.97, 59.85, 39.08, 30.69, 29.60, 29.06, 27.00, 19.98, 19.56 ppm. IR: ν = 3281, 3081, 2928, 1628, 1534 cm^{-1}. HRMS (ESI-TOF+): Calcd. for $C_{28}H_{40}N_6NaO_4^+$ [M + Na$^+$] 561.3165; found 561,3159 (Δ = 1,1 ppm).

2f: White solid (60% yield). M. p. 247,5 - 249,2 ºC. ^{1}HNMR (300 MHz, [D$_6$]DMSO, 30 ºC): δ = 8.72 (d, 4H, J=4.75 Hz), 8.56 (d, 2H, J=8.60 Hz), 8.03 (t, 2H, J=4.07 Hz), 7.79 (d, 4H, J=5.43 Hz), 4.25 (t, 2H, J=8.71 Hz), 3.06 (m, 4H), 2.09 (m, 2H), 1.40 (m, 4H), 1.24 (m, 4H), 0.91 (m, 12H), 0.83 (m, 2H) ppm. ^{13}CNMR (75 MHz, [D$_6$]DMSO, 30 ºC): δ = 171.10, 165.60, 150.75, 141.89, 122.25, 59.94, 39.08, 30.66, 29.58, 29.04, 26.99, 19.94, 19.56 ppm. IR: ν = 3275, 3071, 2926, 1629, 1535 cm^{-1}. HRMS (ESI-TOF+): Calcd. for $C_{28}H_{40}N_6NaO_4^+$ [M + Na$^+$] 561.3165; found 561,3166 (Δ = 0,1ppm).

1g: White solid (75% yield). M. p. 199,3 - 202,4 ºC. ^{1}HNMR (300 MHz, [D$_6$]DMSO, 30 ºC): δ = 9.00 (s, 2H), 8.68 (d, 2H, J=5.29 Hz), 8.52 (d, 2H, J=9.00), 8.20 (d, 2H, J=7.90 Hz), 8.03 (t, 2H, J=4.06 Hz), 7.47 (m, 2H), 4.23 (t, 2H, J=7.97 Hz), 3.03 (m, 4H), 2.07 (m, 2H), 1.36 (m, 4H), 1.19 (m, 6H), 0.89 (m, 12H), 0.81 (m, 2H) ppm. ^{13}CNMR (75 MHz, [D$_6$]DMSO, 30 ºC): δ = 171.27, 165.70, 152.48, 149.36, 135.96, 130.48, 123.95, 59.87, 39.09, 30.68, 29.63, 29.33, 26.95, 19.97, 19.56 ppm. IR: ν = 3266, 3079, 2922, 1624, 1529 cm^{-1}. HRMS (ESI-TOF+): Calcd. for $C_{29}H_{42}N_6NaO_4^+$ [M + Na$^+$] 575.3322; found 575,3322 (Δ = 0,1 ppm).

2g: White solid (64% yield). M. p. 242,9 - 246,0 ºC. ^{1}HNMR (300 MHz, [D$_6$]DMSO, 30 ºC): δ = 8.69 (d, 4H, J=5.72 Hz), 8.59 (d, 2H, J=8.71 Hz), 8.05 (t, 2H, J=4.63 Hz), 7.77 (d, 4H, J=5.45 Hz), 4.22 (t, 2H, J=7.44 Hz), 3.03 (m, 4H), 2.06 (m, 2H), 1.36 (m, 4H), 1.20 (m, 6H), 0.90 (m, 12H), 0.81 (m, 2H) ppm. ^{13}CNMR (75 MHz, [D$_6$]DMSO, 30 ºC): δ = 171.11, 165.60, 150.75, 141.89, 122.25, 59.96, 39.09, 30.65, 29.61, 29.32, 26.95, 19.95, 19.57 ppm. IR: ν = 3274, 3077, 2923, 1630, 1529 cm^{-1}. HRMS (ESI-TOF+): Calcd. for $C_{29}H_{42}N_6NaO_4^+$ [M + Na$^+$] 575.3322; found 575,3327 (Δ = 0,9 ppm).

1h: White solid (76% yield). M. p. 205,9 - 208,7 ºC. ^{1}HNMR (300 MHz, [D$_6$]DMSO, 30 ºC): δ = 9.01 (s, 2H), 8.69 (d, 2H, J=4.83 Hz), 8.51 (d, 2H, J=8.58), 8.20 (d, 2H, J=7.51 Hz), 8.00 (t, 2H, J=5.63 Hz), 7.47 (m, 2H), 4.24 (t, 2H, J=8.31 Hz), 3.03 (m, 4H), 2.06 (m, 2H), 1.37 (m, 4H), 1.20 (m, 8H), 0.90 (m, 12H), 0.82 (m, 2H) ppm. ^{13}CNMR (75 MHz, [D$_6$]DMSO, 30 ºC): δ = 171.26, 165.71, 152.48, 149.36, 135.94, 130.52, 123.94, 59.87, 39.11, 30.71, 29.64, 29.28, 26.99, 19.97, 19.52 ppm. IR: ν =

3280, 3082, 2923, 1627, 1532 cm^{-1}. HRMS (ESI-TOF+): Calcd. for $C_{30}H_{44}N_6NaO_4^+$ [M + Na^+] 589.3478; found 589,3484 (Δ = 1,1 ppm).

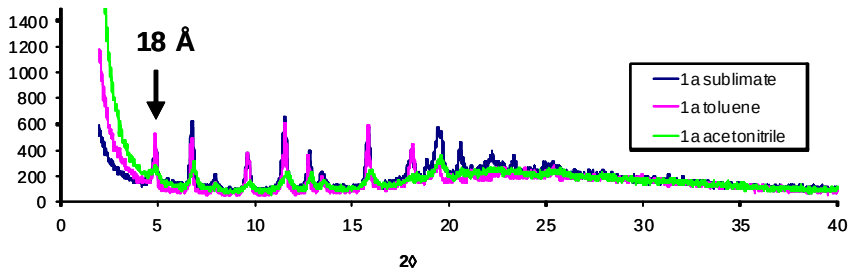
2h: White solid (86% yield). M. p. 222,6 - 225,4 ºC. ^{1}HNMR (300 MHz, [D_6]DMSO, 30 ºC): δ = 8.69 (d, 4H, J=4.23 Hz), 8.54 (d, 2H, J=8.47 Hz), 8.00 (t, 2H, J=5.44 Hz), 7.77 (d, 4H, J=4.23 Hz), 4.23 (t, 2H, J=8.06 Hz), 3.03 (m, 4H), 2.07 (m, 2H), 1.37 (m, 4H), 1.20 (m, 8H), 0.90 (m, 12H), 0.83 (m, 2H) ppm. ^{13}CNMR (75 MHz, [D_6]DMSO, 30 ºC): δ = 171.08, 165.60, 150.75, 141.93, 122.23, 59.95, 39.11, 30.68, 29.63, 29.28, 26.99, 19.94, 19.52 ppm. IR: ν = 3284, 3083, 2925, 1627, 1532 cm^{-1}. HRMS (ESI-TOF+): Calcd. for $C_{30}H_{44}N_6NaO_4^+$ [M + Na^+] 589.3478; found 589,3474 (Δ = 0,7 ppm).

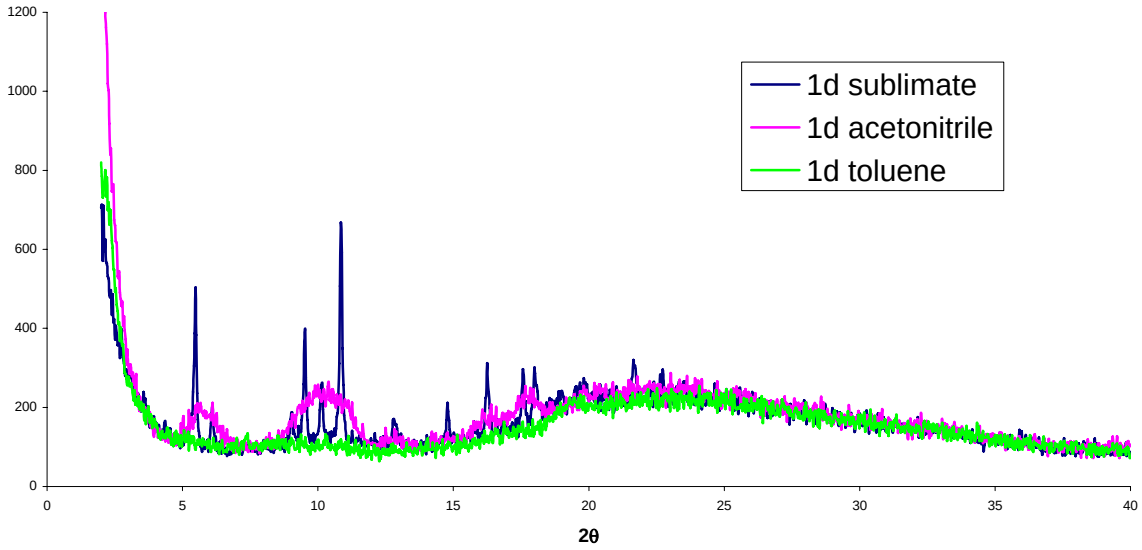
Electrospray ionization mass spectrometry.

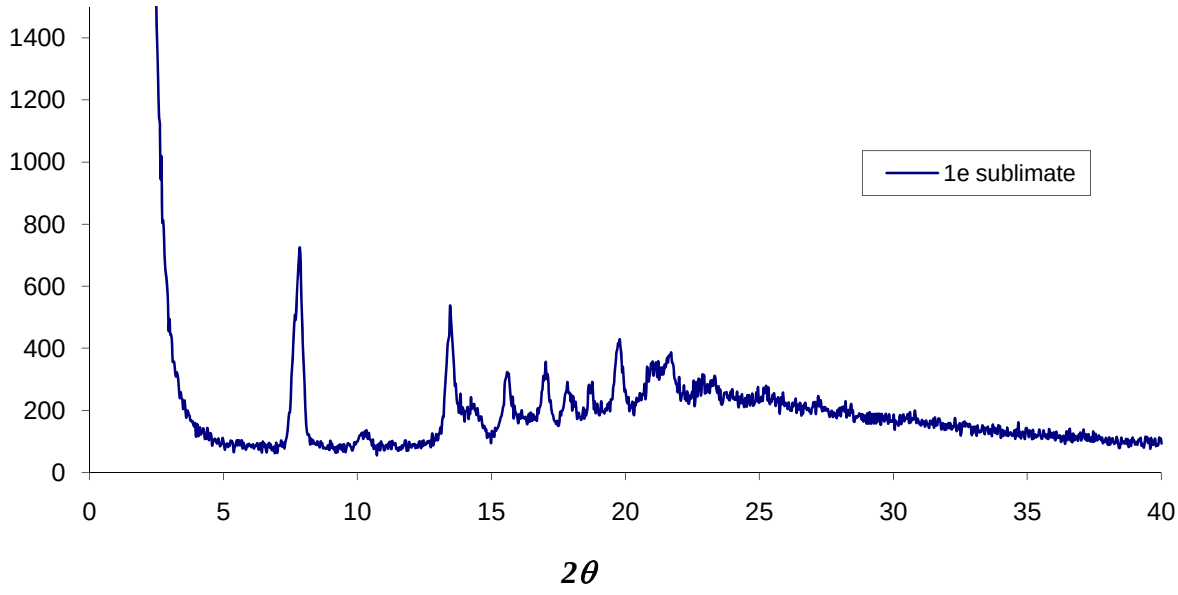
A Q-TOF premier mass spectrometer with an electrospray source (Waters, Manchester, UK) was used. The drying gas as well as nebulizing gas was nitrogen at a flow of 400L/h and 80 L/h respectively. The temperature of the source block was set to 120 ºC and the desolvation temperature to 150 ºC. A capillary voltage of 3.3 kV was used in the positive scan mode and the cone voltage was set to 25 V. Methanol sample solutions were infused via syringe pump directly connected to the ESI source at a flow rate of 10 μL/min. Mass calibration was performed using a mixture of NaOH 0.05 M: formic acid 10 % (50:50) from m/z 100 to 1150. For the accurate mass measurements, a solution of leucine enkephalin (m/z = 556.2771) was introduced via the lock spray needle at a flow rate of 30 μL/min.

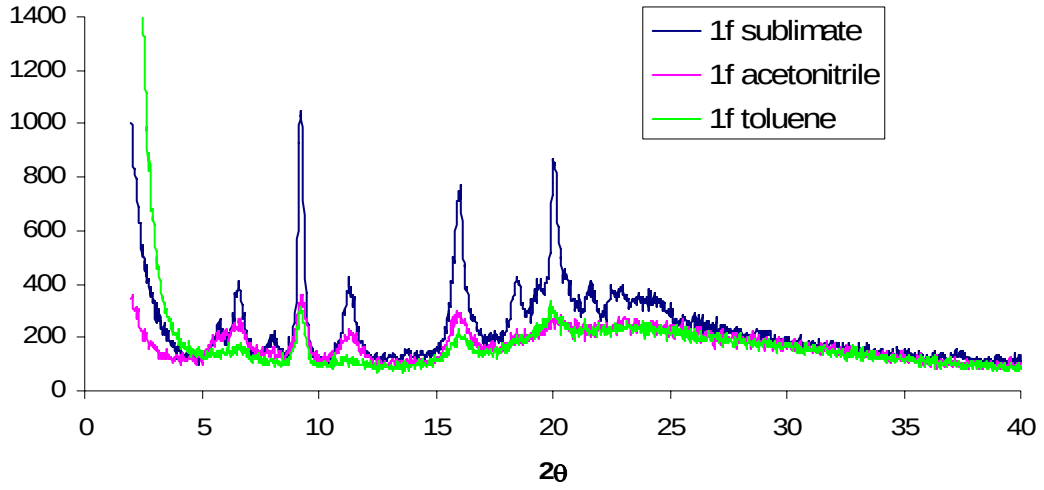
Figure ESI1. Microsublimation apparatus with glass slide.

Figure ESI2. Additional Wide-Angle X-ray Diffraction patterns of sublimates and xerogels of compounds **1** and **2**.





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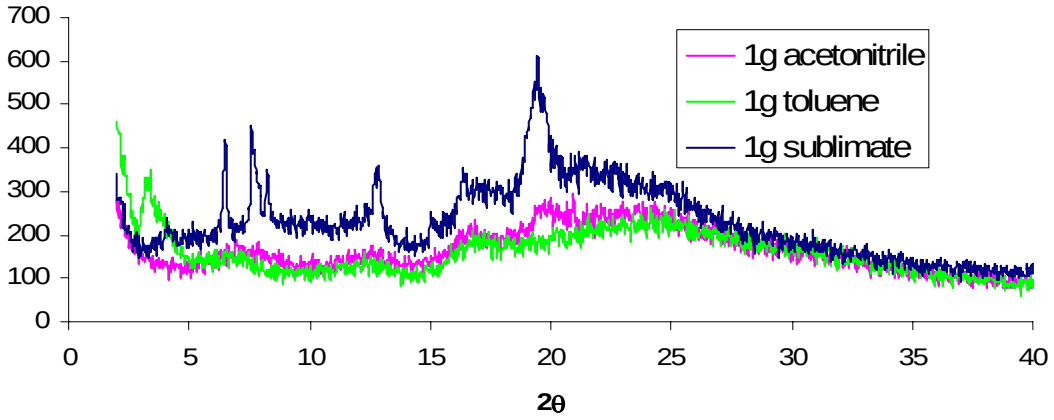
1f toluene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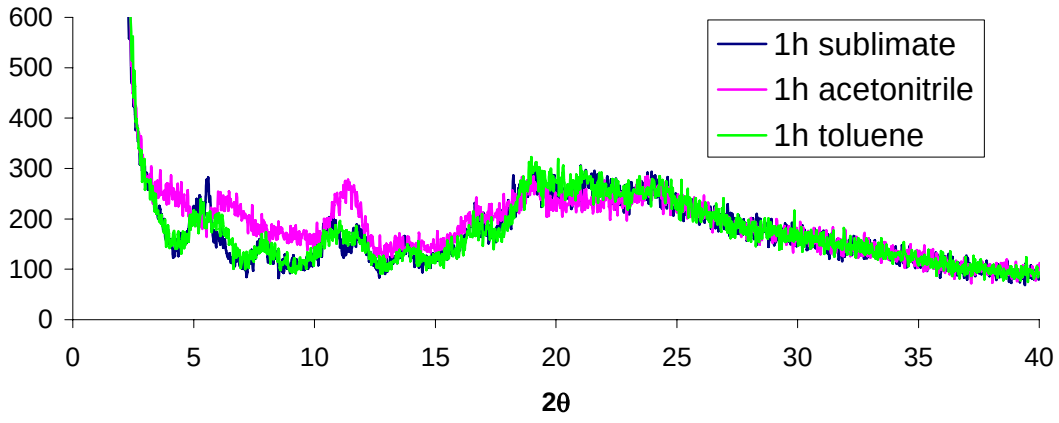
1g sublimate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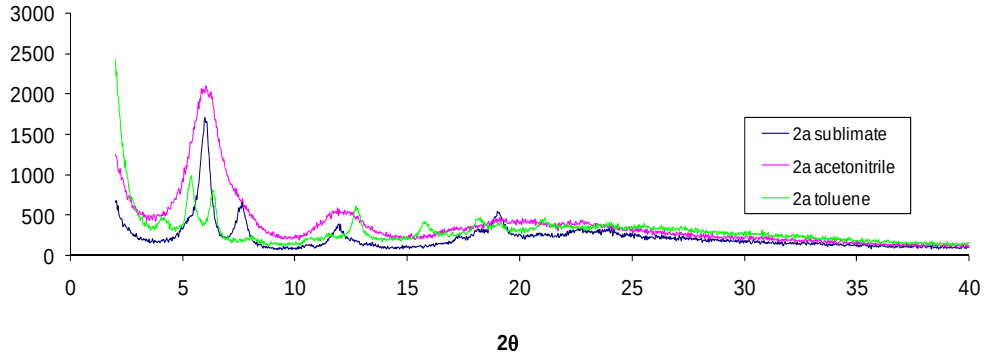
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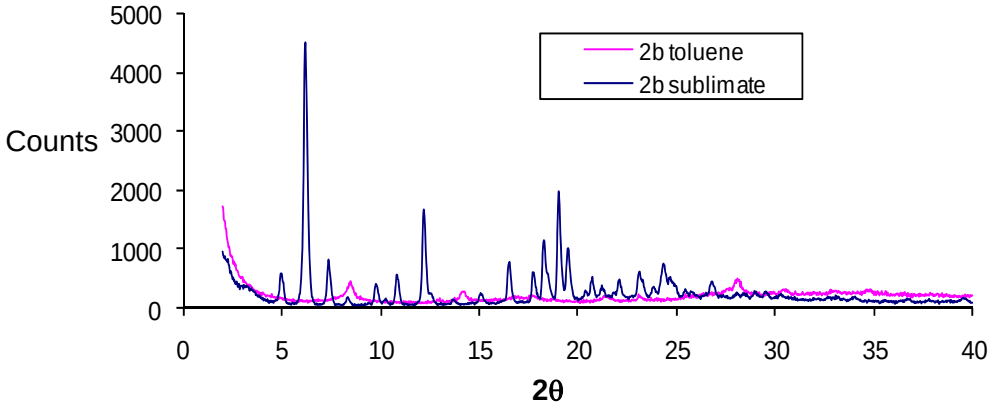
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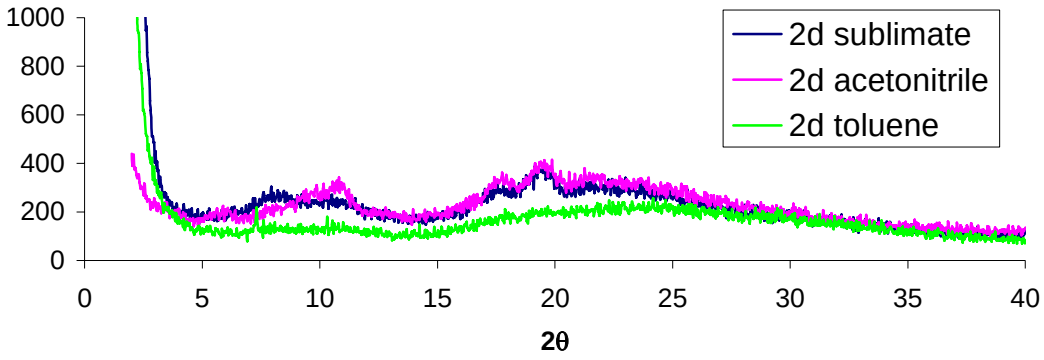
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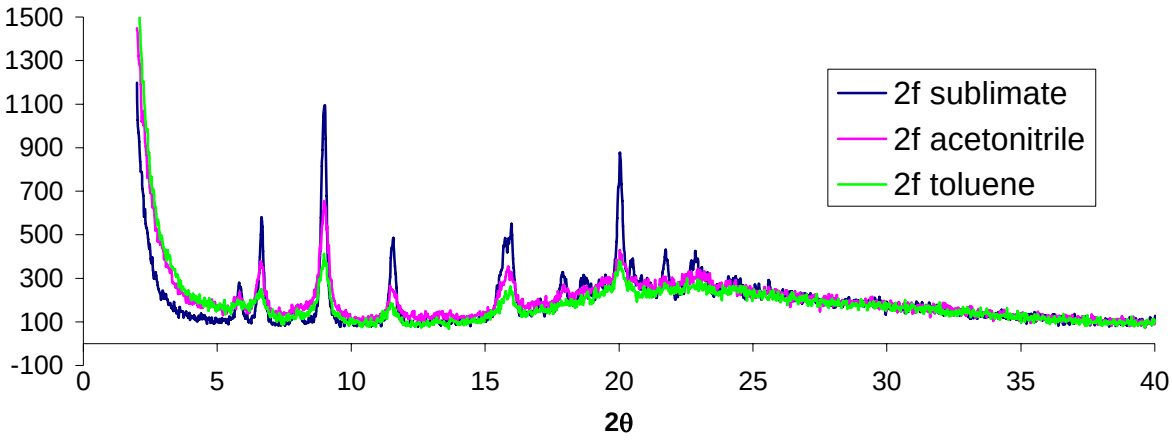
2d toluene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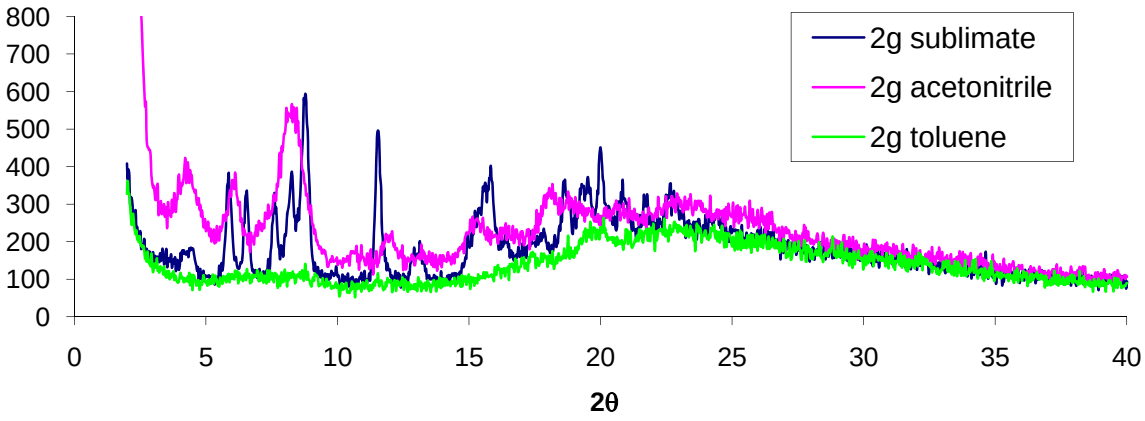
2f acetonitrile
2f toluene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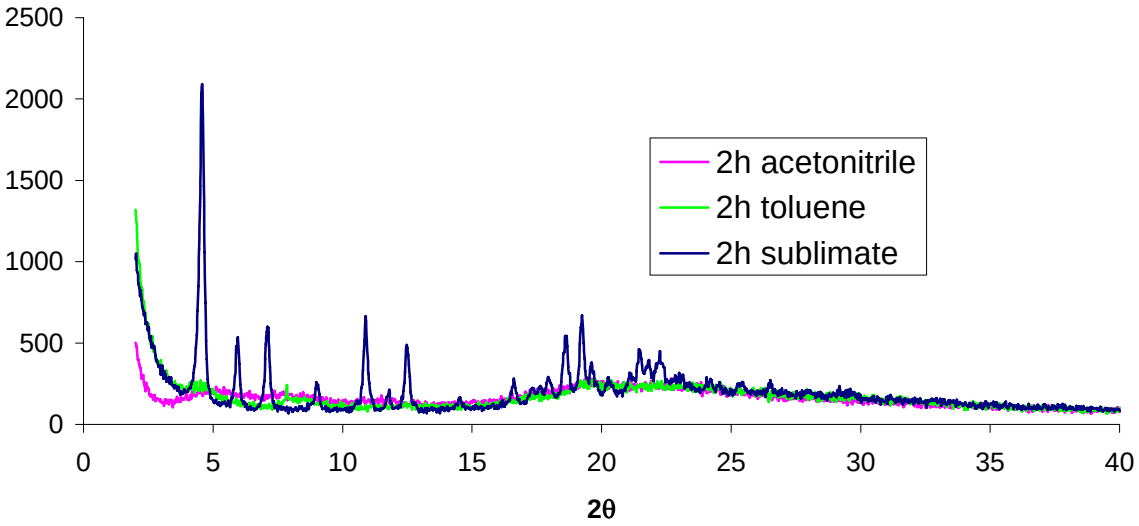
2g acetonitrile
2g toluene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2θ
2h acetonitrile
2h toluene
2h sublimate

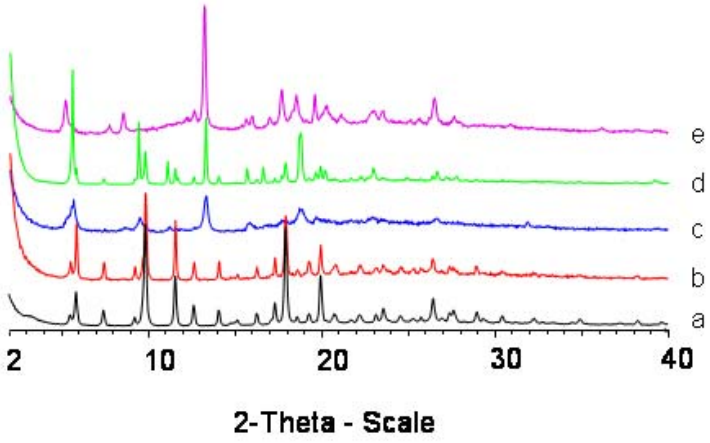


Figure ESI3. WAXD of compound **2e** from different solvents and after thermal tratment: a) obtained from chemical reaction; b) xerogel from acetonitrile ; c) xerogel from toluene; d) solid from methanol; e) sublimate.

Table ESI1. Powder diffraction data summary for sublimates and xerogels of compounds **1a-h**.

Compound	treatment	(n00)	(0n0)	(001)
1a	S=A=T	18.3, 9.18,	13.1, 6.55, 4.3	4.6
1b	S=A=T	21.85	12, 6.1, 4	4.4
1c	S=A=T	24.9, - , 8.2	10.8, 5.4, 3.6	4.6
1d	S	[27.6][a], 13.8, 9.2	16.0, 8.2, 5.4	4.5
	A	amorphous		
	T	amorphous		
1e	[b]	[b]	[b]	[b]
1f	S=A=T	[28.5] [a], 15.4, 9.6	13.4	4.4
1g	S	[27.3] [a], 13.6, 6.9	(21.5), 10.75, 5.4	4.55
	A	amorphous		
	T	amorphous		
1h	S=A=T	Broad		

[a] Possible low angle peak (absent or under background). [b] D. S. Tsekova, B. Escuder, J. F. Miravet, *Cryst. Grow. Des*., 2008, **8**, 11..

S: sublimate, A: acetonitrile xerogel, T: toluene xerogel.

Table ESI2. Powder diffraction data summary for sublimates and xerogels of compounds **2a-h**.

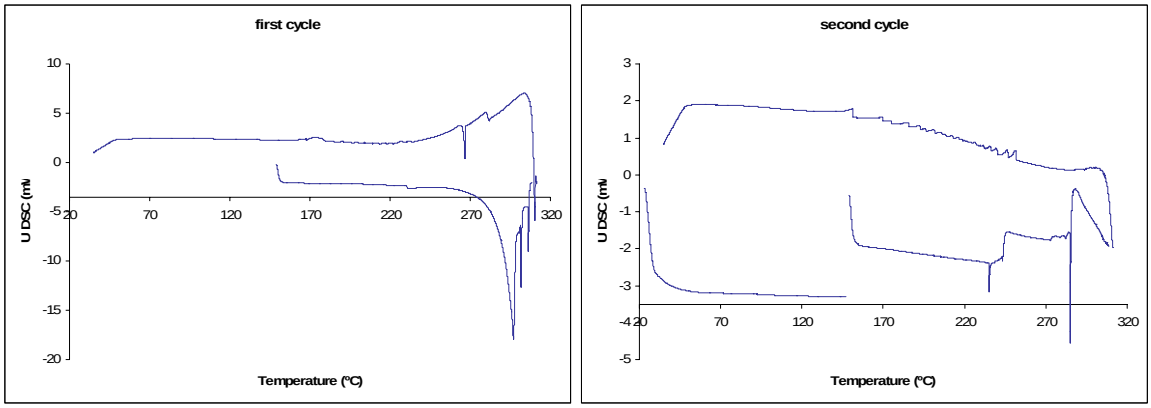
Compound	treatment	(n00)	(0n0)	(001)
2a	S=A	[22][a], 14.7, 7.4	11.6	4.6
	T	21.4, 10.9, 5.6	[c]	[c]
2b	S	[21][a],14.2, 7.2	complex	4.6
	A[e]	[22][a], 14.8, 7.3	8.8	4.5
	T	[c]	[c]	[c]
2c	S=A[d]	12.6, 6.3	10.7	4.6
	T	18.2, 9.1	d	d
2d	S=A=T	amorphous		
2e	S	[33][a], 16.6, 11.1	6.7	4.7
	A	[c]	[c]	[c]
	T	[c]	[c]	[c]
2f	S=A=T	[30] [a], 15.2, 9.8, 7.65	13.3	4.4
2g	S[e]	[30.1] [a], 15.05, 10.06	13.5, 6.72, 4.9 [b]	4.4
	A	20.8, 10.4	14.6, 7.3	4.5-4.9 broad
	T	amorphous		
2h	S	[38.6] [a], 19.3, 12.8, 9.65	14.8, 7.1	complex
	A	amorphous		
	T	amorphous		

[a] Possible low angle peak (absent or under background). [b] Additional peaks from acetonitrile polymorph. [c]: mixture of polymorphs. [d]: minor amount of additional polymorph. [e]: *Org. Biomol. Chem*., 2008, **6**, 4378.

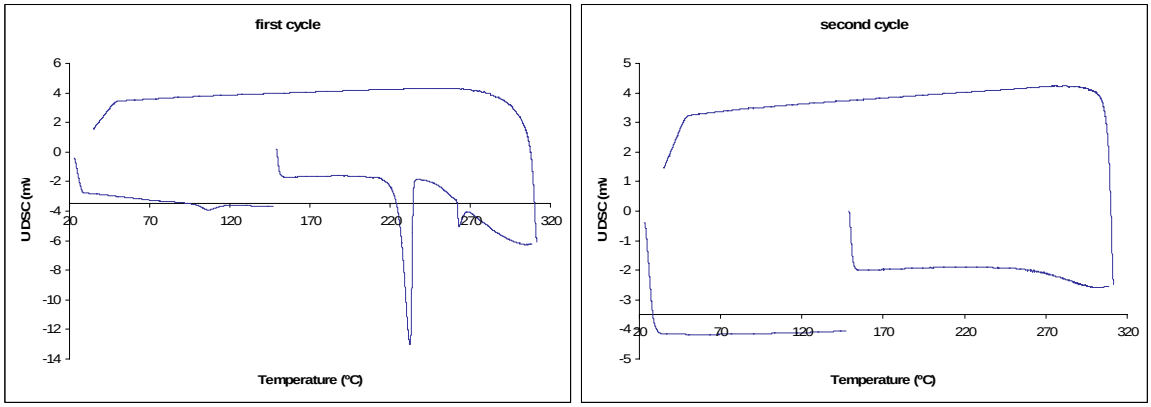
S: sublimate, A: acetonitrile xerogel, T: toluene xerogel.

Figure ESI4. DSC thermograms of compounds **1a-h** and **2a-h** before thermal treatment. (left: first cycle, right: second cycle; step 1: 25 ºC to 150 ºC, 10 ºC/min; step 2: 150 ºC to 300 ºC, 5 ºC/min; step 3: 300 ºC to 25 ºC, 10 ºC/min).

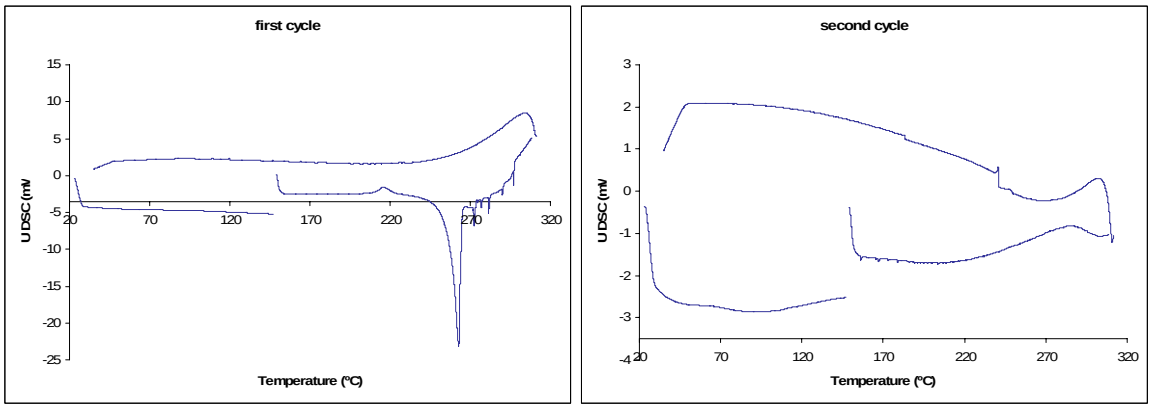
1a



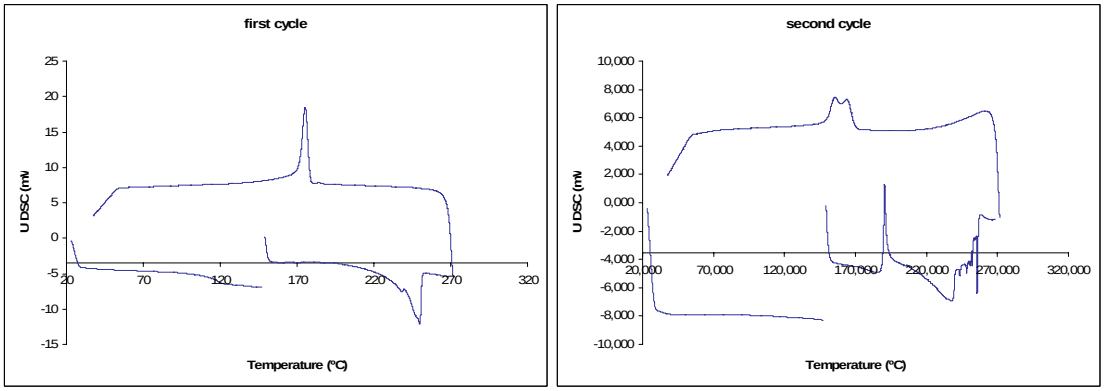
1b



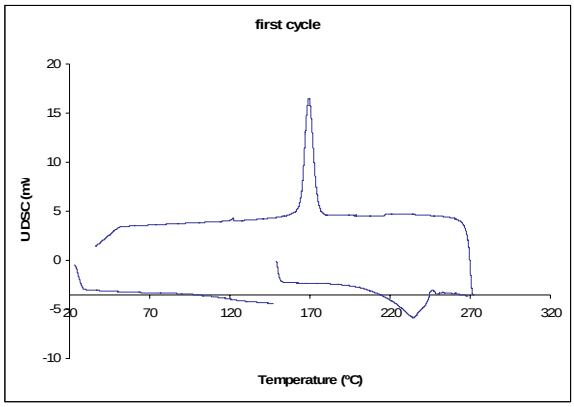
1c

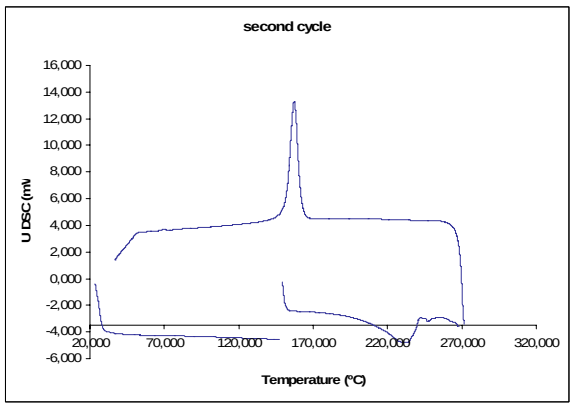


1d

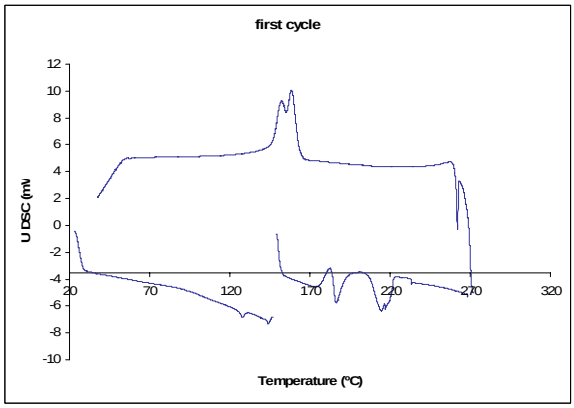


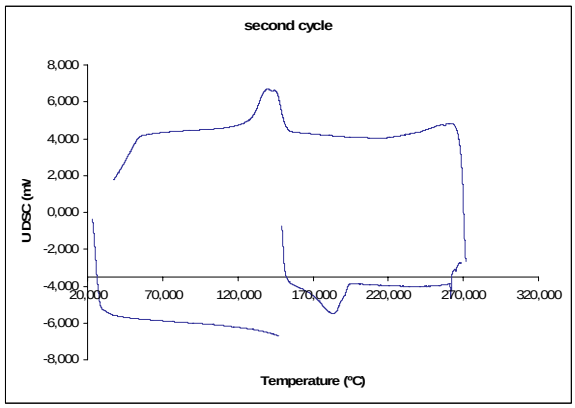
1f


first cycle
U DSC (mW
Temperature (°C)

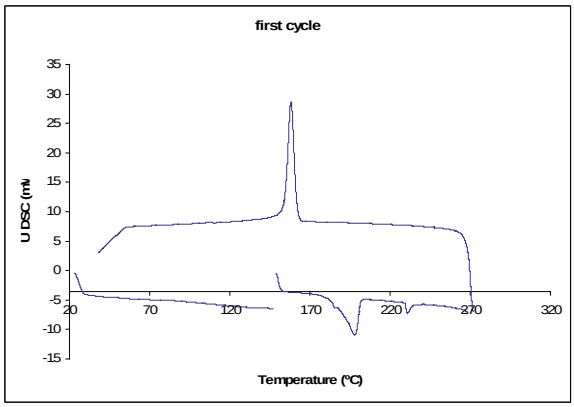

second cycle
U DSC (mW
Temperature (°C)

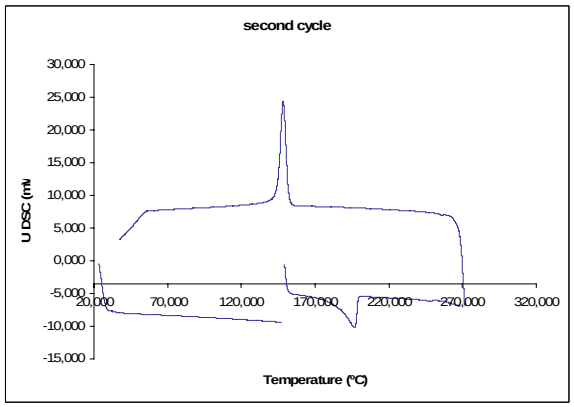
1g


first cycle
U DSC (mW
Temperature (°C)

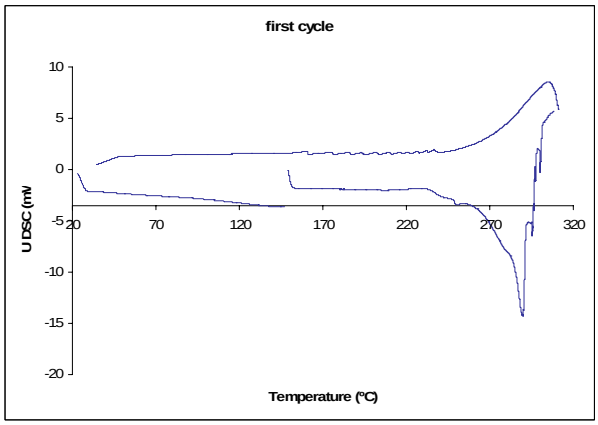

second cycle
U DSC (mW
Temperature (°C)

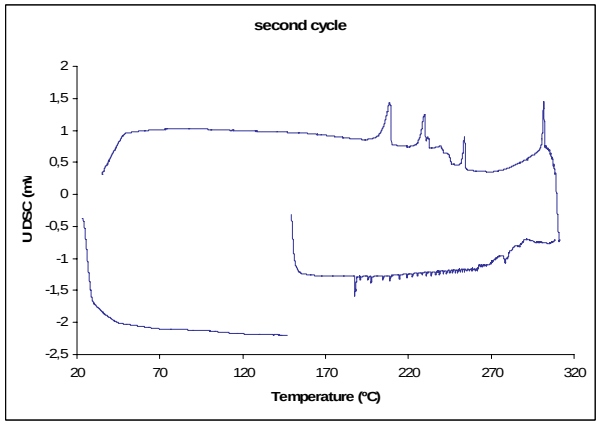
1h


first cycle
U DSC (mW
Temperature (°C)

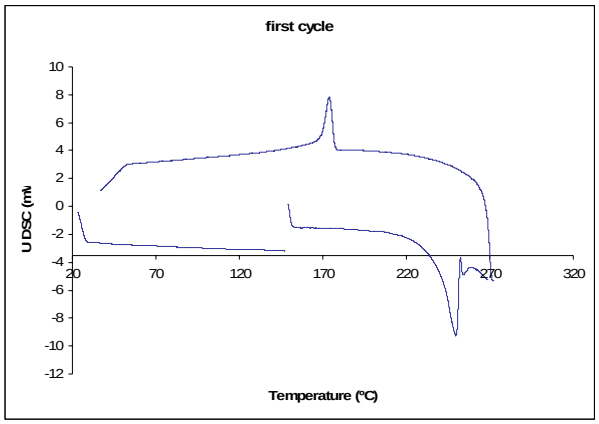

second cycle
U DSC (mW
Temperature (°C)

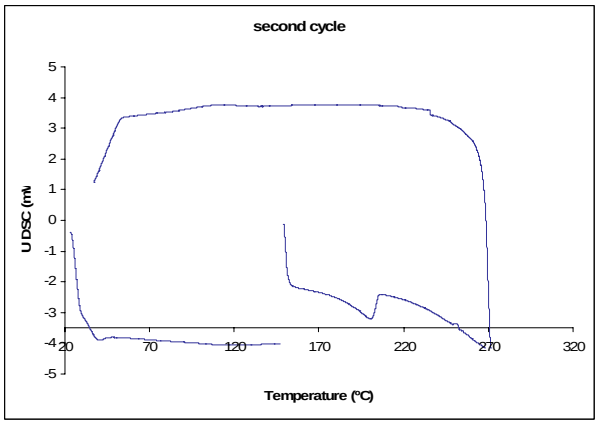
2a


first cycle
U DSC (mW
Temperature (°C)

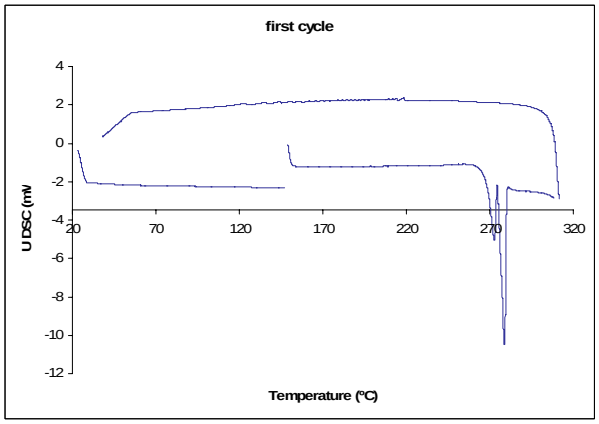

second cycle
U DSC (mW
Temperature (°C)

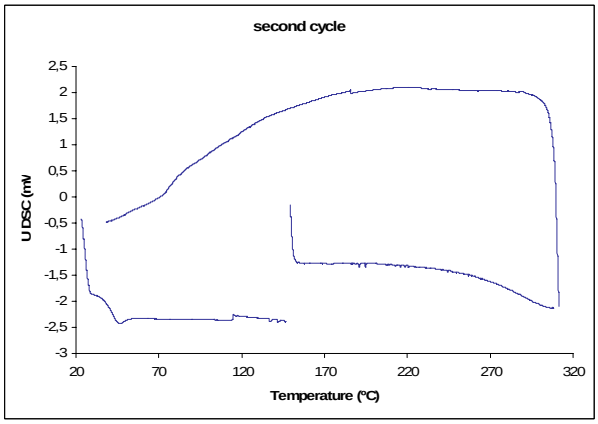
2b


first cycle
U DSC (mW
Temperature (°C)

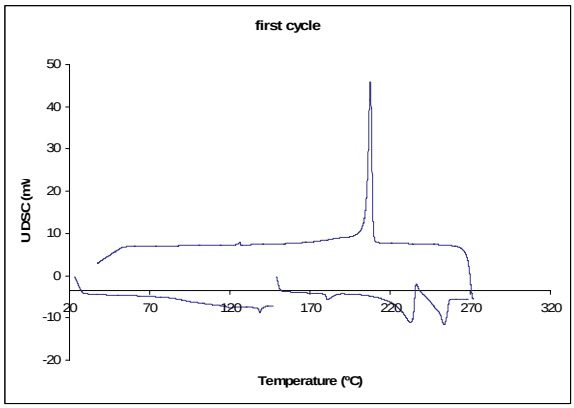

second cycle
U DSC (mW
Temperature (°C)

2c


first cycle
U DSC (mW
Temperature (°C)


second cycle
U DSC (mW
Temperature (°C)

2d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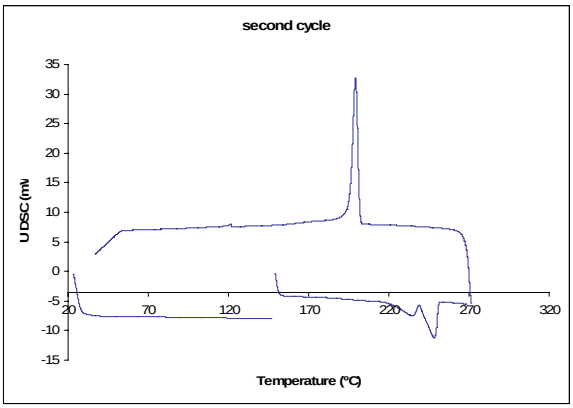
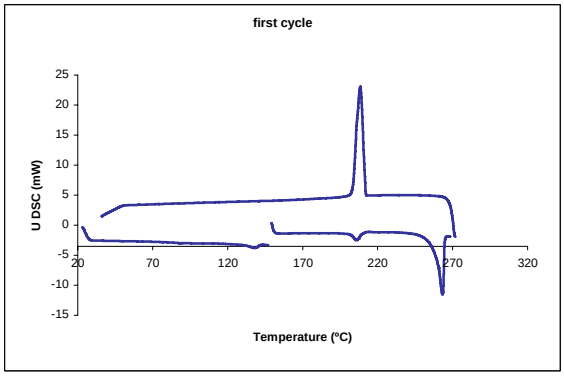
Temperature (°C)


second cycle
U DSC (mW
Temperature (°C)

2e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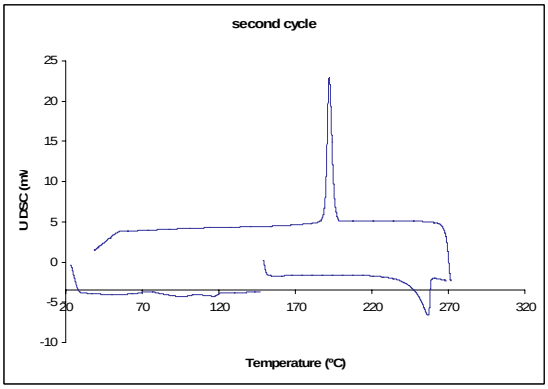
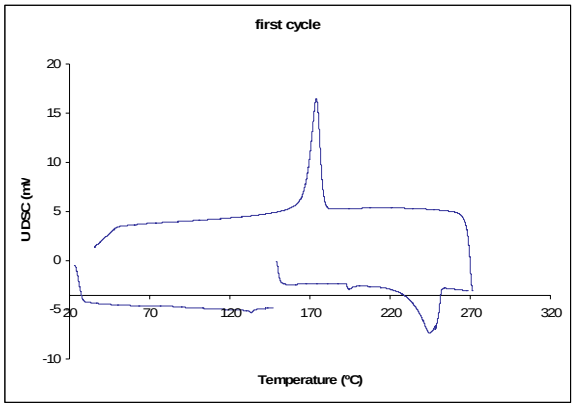
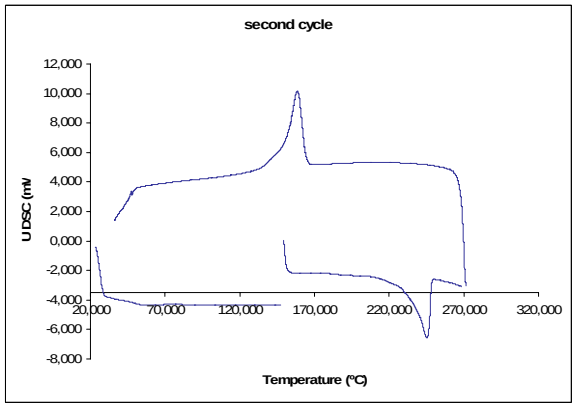
Temperature (°C)

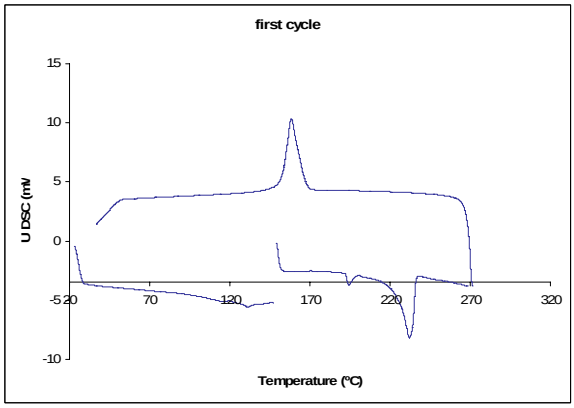

second cycle
U DSC (mW
Temperature (°C)

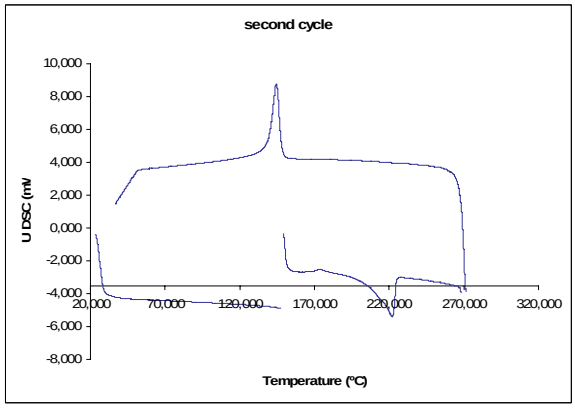
2f


first cycle
U DSC (mW
Temperature (ºC)

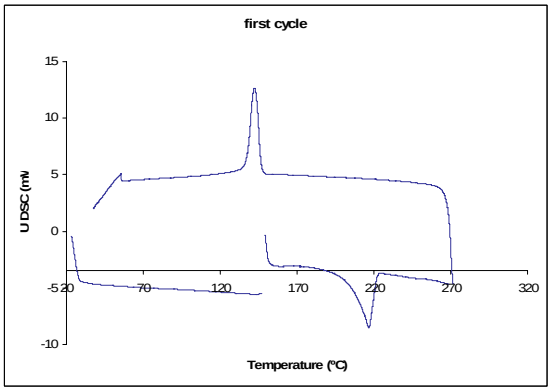

second cycle
U DSC (mW
Temperature (ºC)

2g


first cycle
U DSC (mW
Temperature (ºC)


second cycle
U DSC (mW
Temperature (ºC)

2h


first cycle
U DSC (mW
Temperature (ºC)

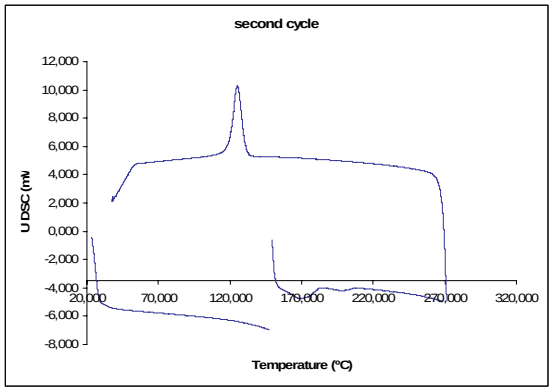

second cycle
U DSC (mW
Temperature (ºC)

Figure ESI5. DSC thermogram of compounds **1e** (top) and **2e** (bottom) before thermal treatment. (1st cycle, 25 ºC to 290 ºC, 5 ºC/min).

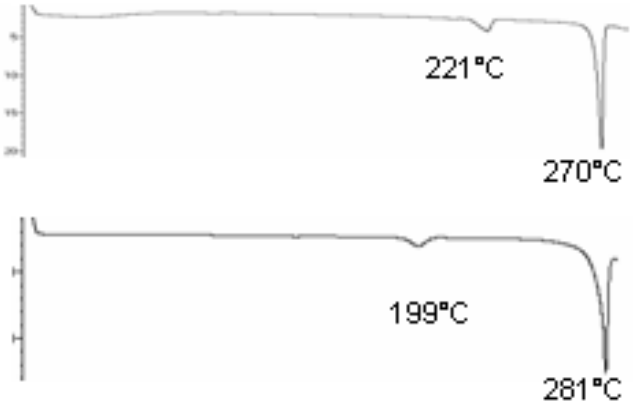
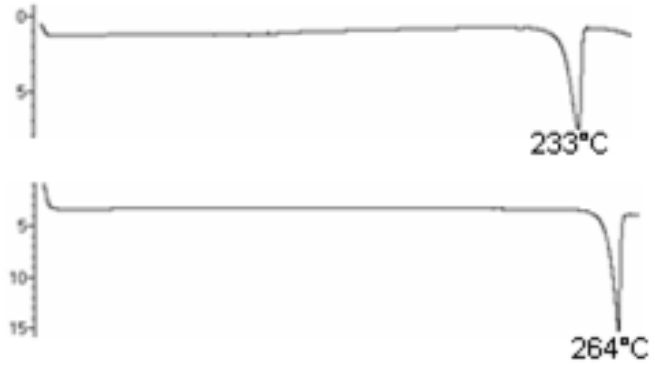


Figure ESI6. DSC thermogram of compounds **1b** (top) and **2b** (bottom) before thermal treatment. (1st cycle, 25 ºC to 290 ºC, 5 ºC/min).



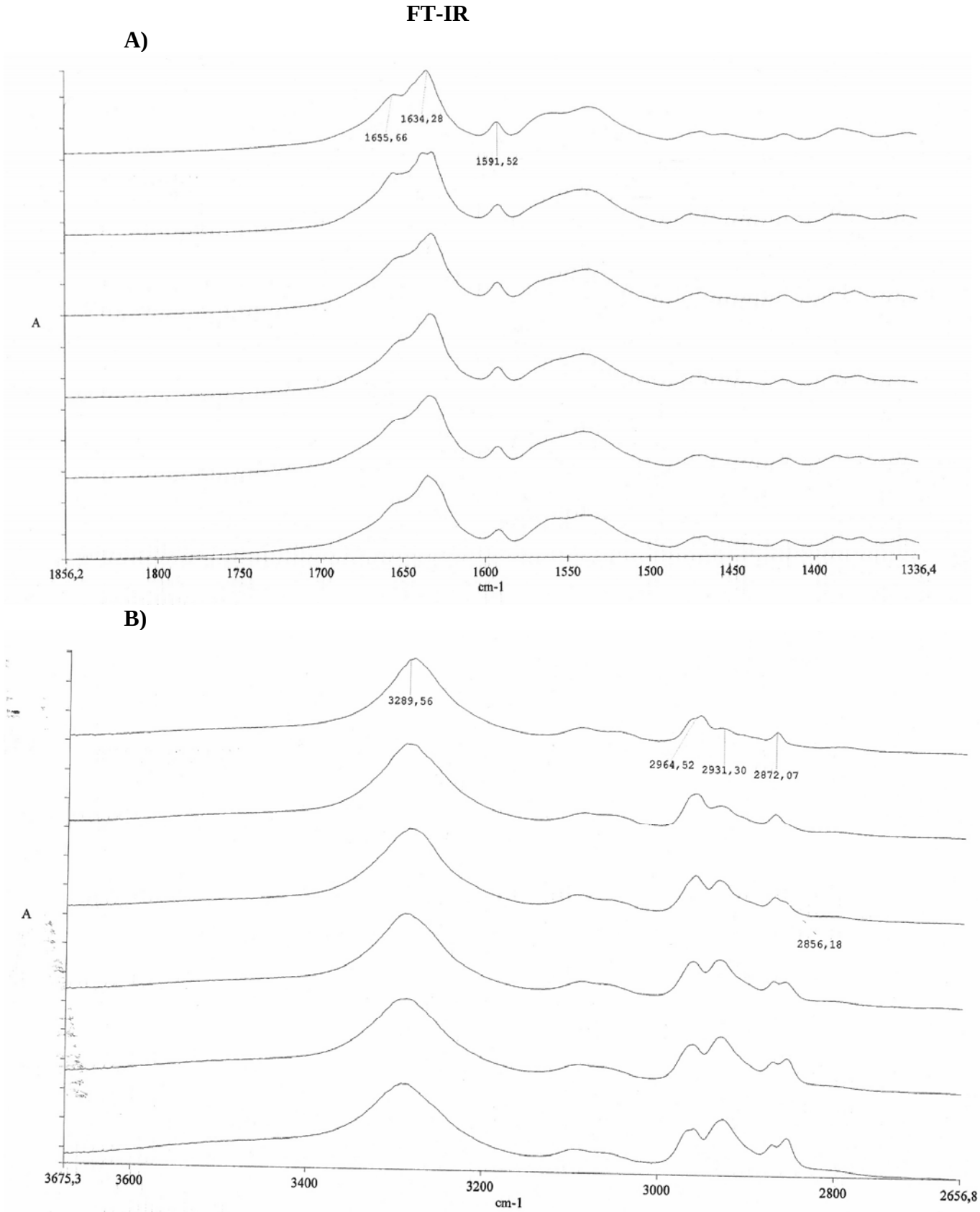


Figure ESI6 . FT-IR spectra of compounds **1a**, **1c**, **1d**, **1f**, **1g** and **1h** (top to bottom). A) C=O amide region; B) N-H and CH_2 stretching region.

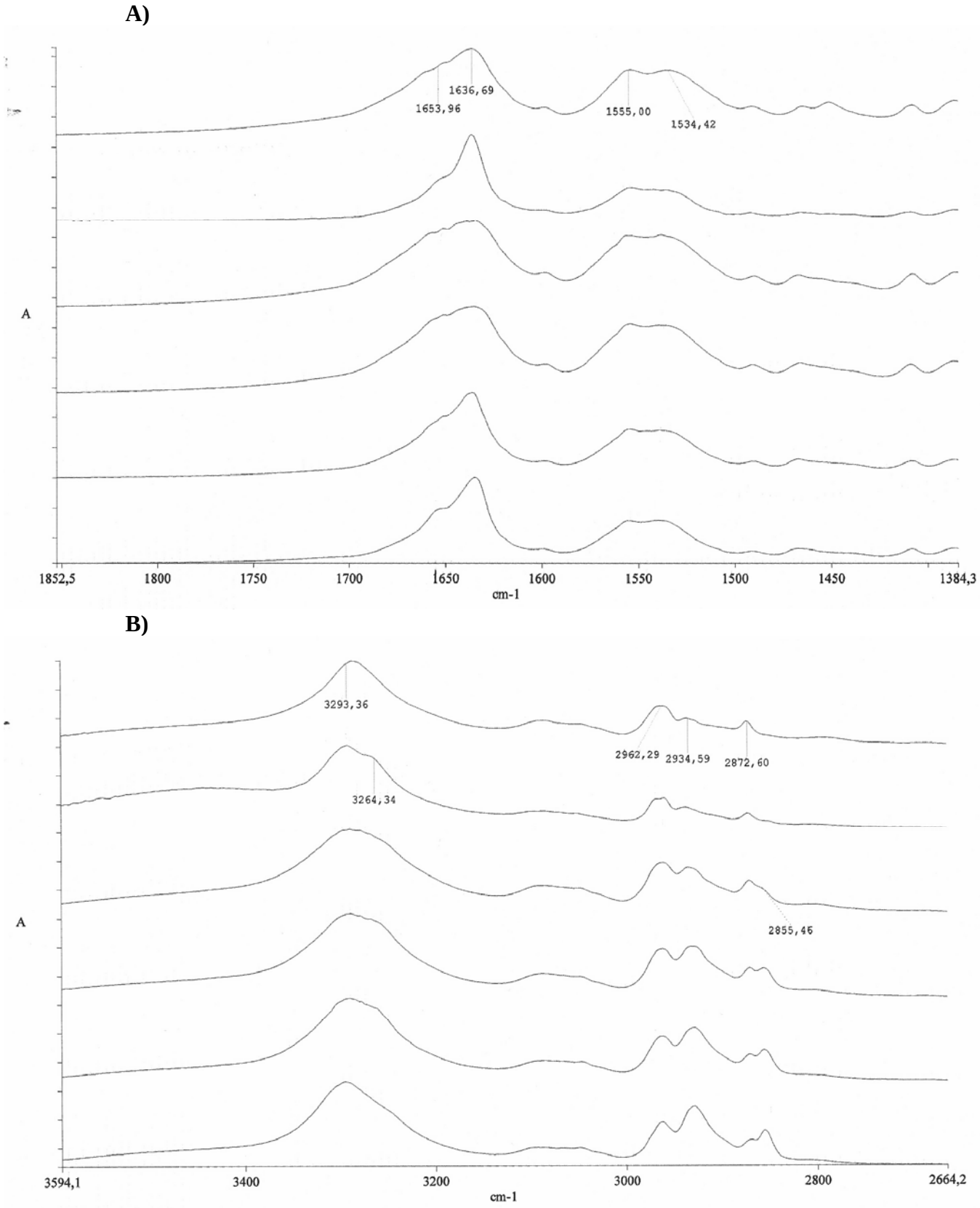


Figure ESI6 . FT-IR spectra of compounds **2a**, **2c**, **2d**, **2f**, **2g** and **2h** (top to bottom). A) C=O amide region; B) N-H and CH_2 stretching region.