Supplementary Material (ESI) for Journal of Materials Chemistry

Supramolecular chirality of columnar mesophases consisting of H-bonded complexes of melamine and polycatenar benzoic acids

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Supporting information

Analytical data of T2*:

^{1}H-NMR (400 MHz, $CDCl_3$): δ 0.86 (t, ^{3}J = 6.8Hz, 3H, CH_3), 1.12 (d, ^{3}J = 6.4Hz, 3H, CH_3), 1.2-1.38 (m, 6H, CH_2), 1.40-1.54 (m, 2H, CH_2), 3.99 (m, 1H, CHNH), 4.81 (d, ^{3}J = 8.4Hz, 1H, NH), 4.95 (s, broad, 4H, NH_2). ^{13}C-NMR (75 MHz, $CDCl_3$): δ 14.0, 21.1, 22.5, 25.6, 31.7, 37.1, 46.0, 166.1, 167.7. IR (nujol, NaCl): 3486, 3327, 3171 (N-H), 814 (C_{Ar}-H) cm^{-1}. ME (FAB+) m/z: 225 (100%, M^{+}+1). EA: Calc. for $C_{10}H_{20}N_6$ (%): C 53.57 H 8.93 N 37.50. Found: C 53.35 H 9.05 N 37.60. $[\alpha]_D^{22}$ (c=0.01g/ml, $CHCl_3$) = +35.5

Preparation of hydrogen-bonded complexes.

Mixtures of the triazine and the chiral benzoic acid derivatives in a 1:3 proportion were obtained by mixing a THF solution of the appropriate amount of each component and evaporating the solvent by stirring at room temperature. The mixtures, once heated to their isotropic states, were used for further experiments.

Characterization.

Thermal properties were examined by polarizing optical microscopy (Olympus BH-2 polarizing microscope equipped with a Linkam THMS600 hot stage and a Linkam TMS91 controller) and differential scanning calorimetry (TA Instruments 2910 and Perkin-Elmer DSC-7). Both calorimeters were calibrated with indium (156.6 °C, 28.44 J/g). Powder X-ray diffraction patterns were obtained using a Pinhole (Anton–Paar) diffractometer and Ni-filtered Cu-*Kα* radiation. The samples were held in Lindemann glass capillaries (Φ = 1 mm) and heated when it is necessary with a variable temperature attachment. The diffraction patterns were collected on photographic films. NMR experiments were performed on a Bruker Avance spectrometer at 400 MHz. IR spectra

were obtained with a Nicolet Avatar 380 spectrophotometer. Optical absorption measurements were taken using an ATI Unicam UV4 spectrophotometer. CD spectra were recorded in a Jasco J-710 at room temperature and a speed scan of 200 nm/min. High-temperature CD spectra were carried out by placing a Mettler FP82 hotstage, conveniently adapted within the CD sample chamber, controlled by a Mettler FP80 temperature controller.

Infrared spectroscopy

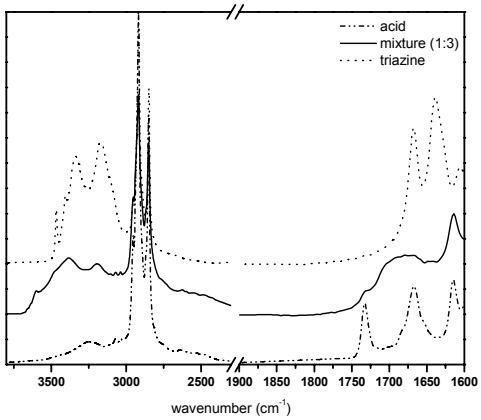
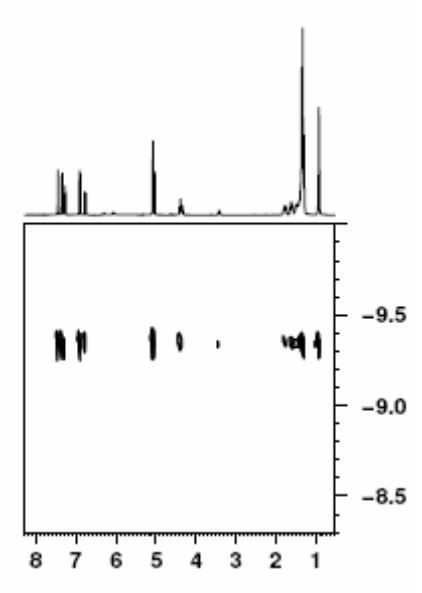


Fig. S1. FTIR spectra of the pure triazine (T1), complex T1-A2 and pure acid (A2).

Diffusion Ordered Spectroscopy, DOSY.



As an example, Figure 2 represents the DOSY spectrum of the complex T1-A3* in which it is graphically clear that signals corresponding all the protons, those of the triazine as well as those of the acid moiety, exhibit the same diffusion coefficient.

Fig. S2. 2D spectrum, in $CDCl_3$, representing chemical shifts versus diffusion coefficients -logD(m^2s^{-1})- for the complex T1-A3*.

Polarized Optical Microscopy

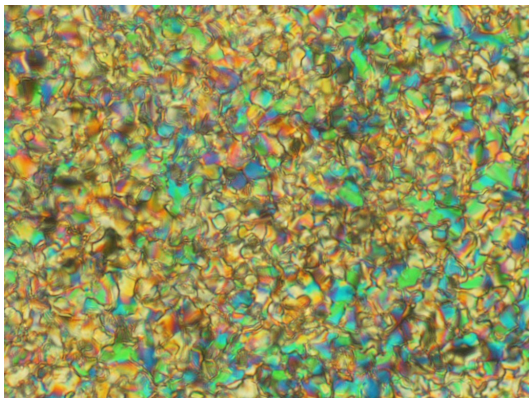


Fig. S3. POM photograph taken for complex T2*-A2 at room temperature (Col_h).

a) **b)**

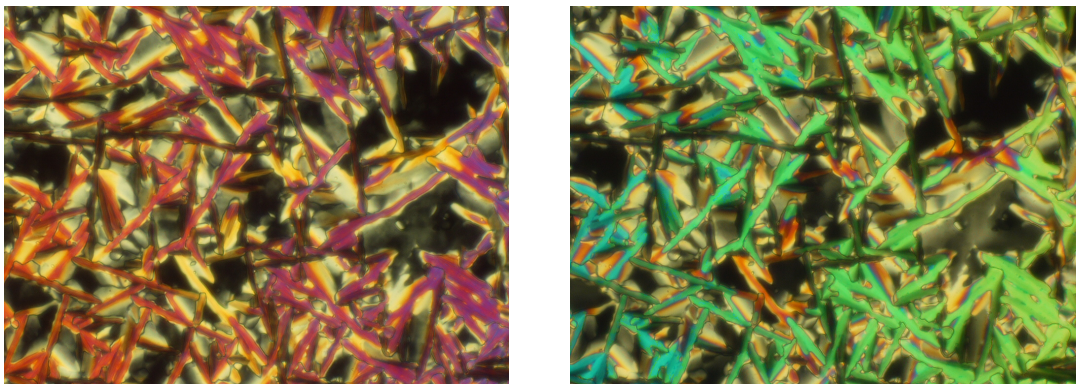


Fig. S4. POM photographs taken for complex T1-A4* at (a) room temperature (Col_r) and (b) 51 ºC (Col_h).

Differential Scanning Calorimetry

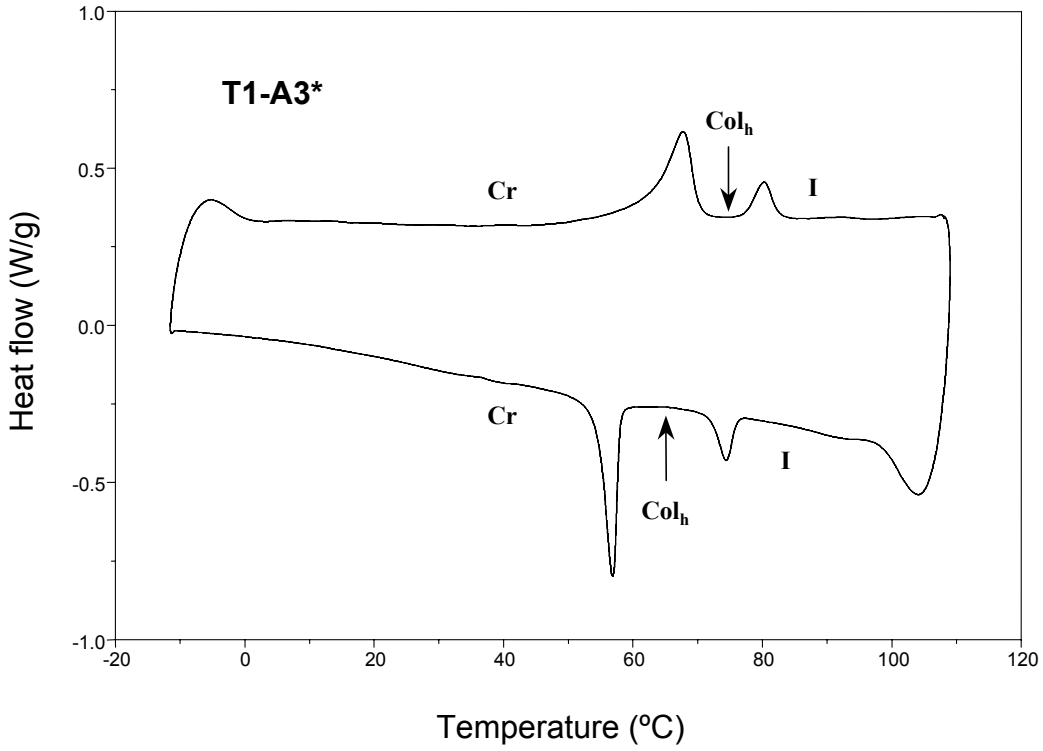


Fig. S5. DSC thermogram of the complex T1-A3* corresponding to the second heating-cooling cycle

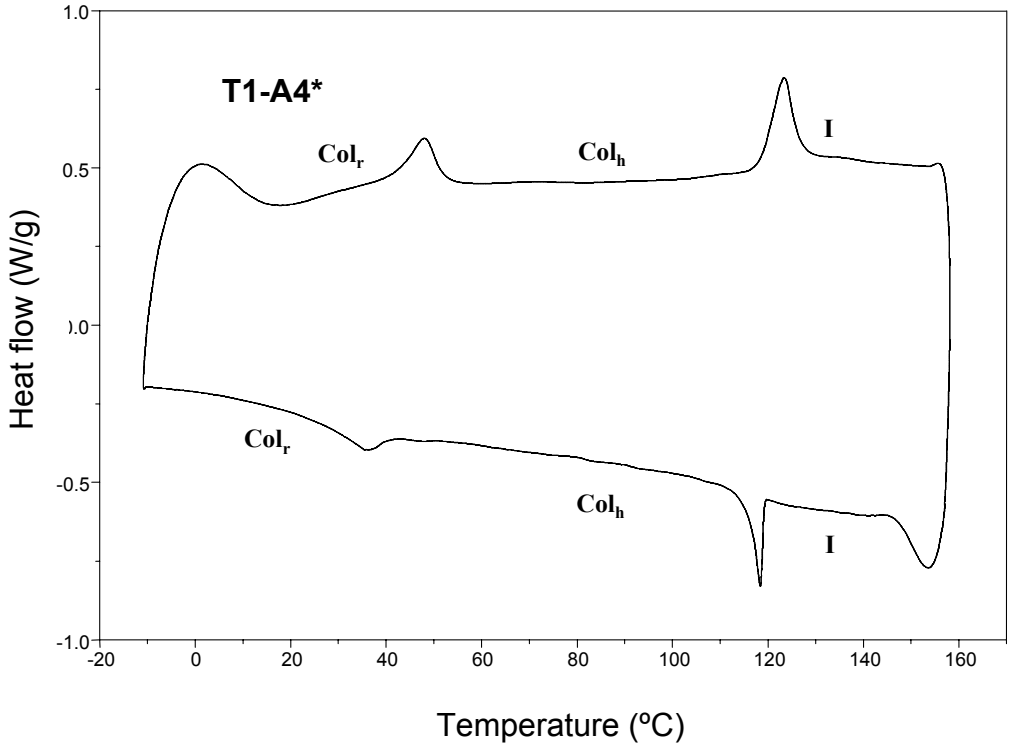


Fig. S6. DSC thermogram of the complex T1-A4* corresponding to the second heating-cooling cycle

X-ray Diffraction. Density Calculation procedure.

Density can be calculated using the following equation:

$$\rho = (M/N)/(V/Z)$$

where M is the molar mass (in g) of the complex, N is Avogadro's number, V is the volume of the unit cell (in cm^3) and Z is the number of complexes per unit cell. The volume is $V = (\sqrt{3}/2)a^2c \cdot 10^{24}$ for a hexagonal unit cell, and $V = abc \cdot 10^{24}$ for an orthorhombic unit cell, where a, b are the lattice constants and c is the stacking distance.