# <sup>13</sup>C-<sup>1</sup>H Dipolar couplings for probing rod-like hydrogen bonded mesogens

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#### **Electronic Supplementary Information (ESI)**

Tables pertaining to solution 2D NMR and DSC of mesogens are included. Figure 1

describes the SAMPI-4 pulse sequence while Figure 2 shows the FT-IR spectra of mesogens.

# Table 1 2D <sup>1</sup>H-<sup>1</sup>H DQF-COSY and 2D <sup>13</sup>C-<sup>1</sup>H HSQC data of PMAPHMB



Carbon	$^{1}$ H	<sup>13</sup> C
Number		
1	-	
2	6.89	114.4
3	8.06	132.3
4	-	-
5	-	-
6	-	-
7	7.18	122.0
8	7.23	122.7
9	-	-
10	8.39	157.7
11	-	-
12	7.69	122.2
13	8.69	150.6
14	-	-
15	-	-
16	7.95	132.2
17	6.86	113.7
18	-	-

### Table 2 Transition temperatures and their enthalpy values of PMAPH and PMAPHMB

Code	Transitions	Temperature Heating(°C)	ΔH (k.cal/mole)
РМАРН	Cr <sub>1</sub> -Cr <sub>2</sub>	92.38	1.90
	Cr <sub>2</sub> -N	105.06	3.56
	N-I	152.14	0.08
РМАРНМВ	Cr- S <sub>A</sub>	101.23	9.77
	S <sub>A</sub> -N	121.04	0.009
	N-I	200.75	0.63

## **Figure Captions**

Figure 1: Measurement of <sup>13</sup>C-<sup>1</sup>H Dipolar Couplings using Separated Local Field (SLF) NMR Spectroscopy Figure 2: FT-IR spectra of (A) 4-methoxy benzoic acid (B) PMAPH and (C) PMAPHMB. For measuring the <sup>13</sup>C-<sup>1</sup>H dipolar couplings of the molecules in their mesophase, the SAMPI4 pulse sequence <sup>1</sup> (Figure 1) was applied on the oriented sample under static conditions. The method yields a 2D spectrum with carbon chemical shifts along the  $F_2$  dimension and the proton-carbon dipolar oscillation frequencies along the  $F_1$  dimension.



Figure 1

The spectra were recorded by using 62.5 kHz of r.f. for both the proton and carbon channels during the  $t_1$  period. During the  $t_2$  period, a broadband hetero-nuclear decoupling pulse scheme SPINAL-64 <sup>2</sup> with 30 kHz decoupling strength was used.  $\tau_1$  and  $\tau_2$  were adjusted to be equal to  $7\pi/4\omega_1$  and  $6\pi/4\omega_1$ , where  $\omega_1$  is the r.f. field strength and were respectively 14 µs and 12 µs. In the pulse sequence, for polarization inversion a contact time  $\tau$  of 2 ms was used. The scale factor of the sequence was estimated experimentally by using the proton coupled <sup>13</sup>C spectrum of chloroform oriented in the liquid crystal N-(4-ethoxybenzylidine)-4-n-butylaniline (EBBA) as reference. Typically 16 transients were used for each  $t_1$  period with a recycle delay of 15 s between scans to avoid sample heating and 128  $t_1$  increments were employed. A shifted sine bell window function was applied to the time domain data and the spectra were processed in the phase sensitive mode.



Figure 2

#### References

- 1 A. A. Nevzorov and S. J. Opella, J. Magn. Reson., 2007, 185, 59.
- 2 B. M. Fung, A. K. Khitrin and K. Ermolaev, J. Magn. Reson., 2000, 142, 7.