

Supporting Information
for
Lyotropic liquid crystalline phases from helical β -peptides as alignment media

by

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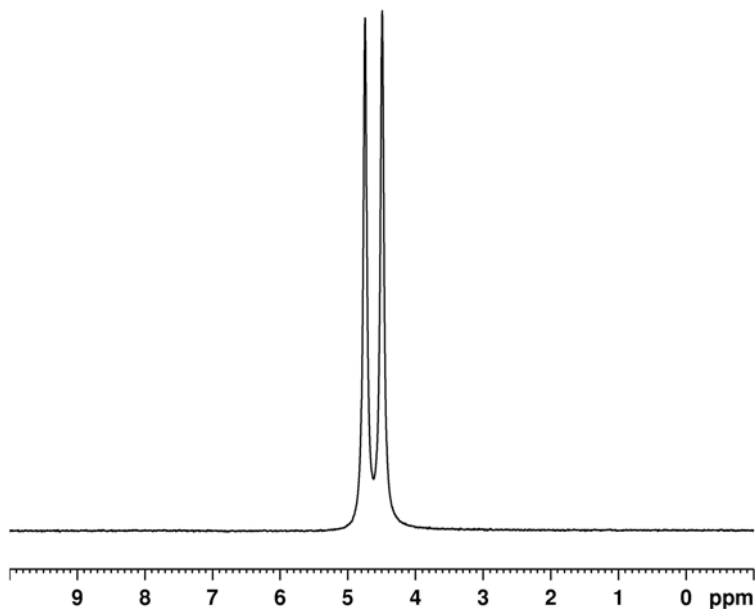
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Figure SI1: ^2H NMR spectrum (76.8 MHz, proton NMR frequency: 500 MHz) of the anisotropic sample containing L-proline

exact composition: 6.9 mg β -peptide **1**^[1], 320.2 mg D_2O , 7.0 mg L-proline (2.06 % w/w LLC phase) with resulting $\Delta_\nu\text{Q} = 19.3$ Hz



For the resulting CLAP-HSQC^[2] see Figure 1b of the main text.

Figure SI2: ^2H NMR spectrum (76.8 MHz) of the anisotropic sample containing D-proline

exact composition:

6.1 mg β -peptide **1**, 340.5 mg D_2O , 7.0 mg D-proline (1.73 % w/w LLC phase) with resulting $\Delta_\nu\text{Q} = 15.1$ Hz

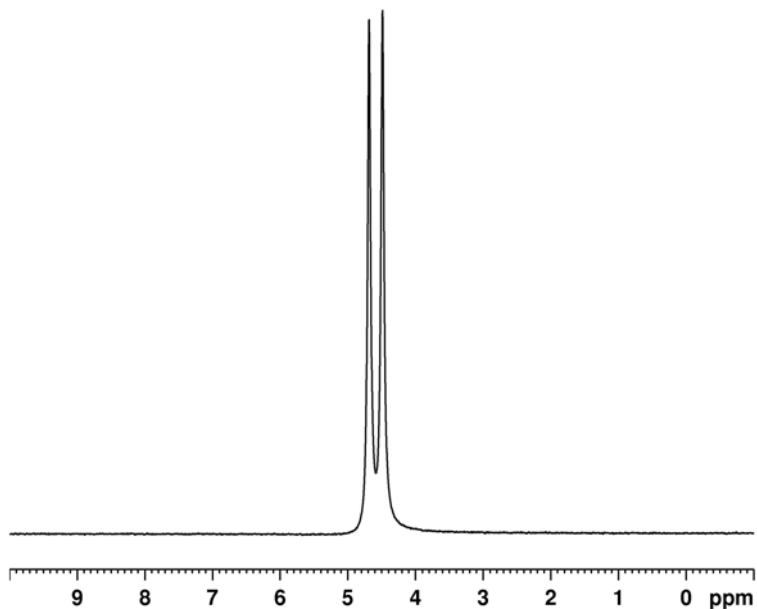
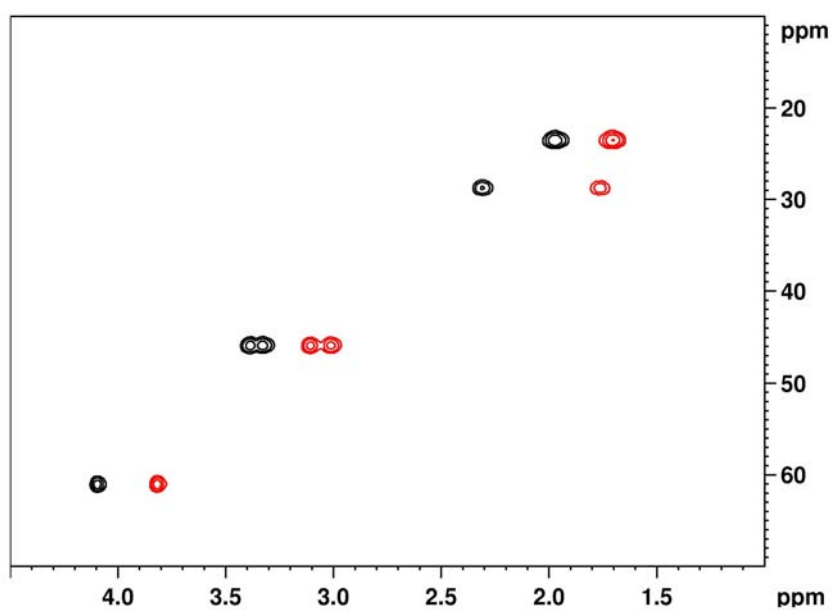


Figure SI3: CLAP-HSQC^[2] of D-proline in 1.7 % w/w LLC phase of β -peptide **1** in D_2O .



Scalemic sample:

To investigate whether the chemical shift anisotropy (CSA) difference of the two enantiomers is large enough to observe enantiodiscrimination by monitoring the chemical shifts a scalemic sample was prepared (L:D-proline=8:5). The sample was in principle prepared as described in the main text. As no difference in CSAs was observed from the initial composition, the concentration of **1** was increased. The whole sample preparation involved a series of manipulations (removing from NMR tube, adding **1** in D₂O, vortexing, etc.). Thus no exact concentration can be given (see below for approximate composition).

As measurements at 500 MHz did not show split signals (data not shown), a second series of measurements was conducted at the BMRZ in Frankfurt at a spectrometer with proton frequency of 800 MHz (see Figures SI4-6). Nevertheless no difference in CSA of the two enantiomers was observed.

This is in accordance with our previous experience. When investigating the orientational properties of high MW PBLG we found out, that reducing the order (as is achieved by the lower c_{crit} of high MW PBLG) sometimes leads to the situation, that signals of enantiomers are not split anymore.^[3] This does not mean that enantiomers are not discriminated, as RDCs of the two enantiomers remain clearly different. The CSA difference of this very weakly ordered systems is just too small to exhibit a detectable influence on the line shapes and/or number of lines.

Thus it seems as if being able to discriminate signals at first glance from split signals strong orientation needs to be induced, which on the other hand prohibits accurate and precise extraction of RDCs.

approximate sample composition of scalemic sample:

ca. 13 mg β -peptide **1**^[1]

ca. 500 mg D₂O,

ca. 52 mg of a parent solution of D- and L-proline (containing 8,2 mg L-proline, 5,2 mg D-proline per 100 mg)

resulting $\Delta_{\nu}Q = 18.2$ Hz at 122.8 MHz (800 MHz ¹H NMR frequency)

Figure SI4: ^1H NMR spectrum (800 MHz) of the anisotropic sample containing a scalemic mixture of D- and L-proline (full range and expansion is shown)

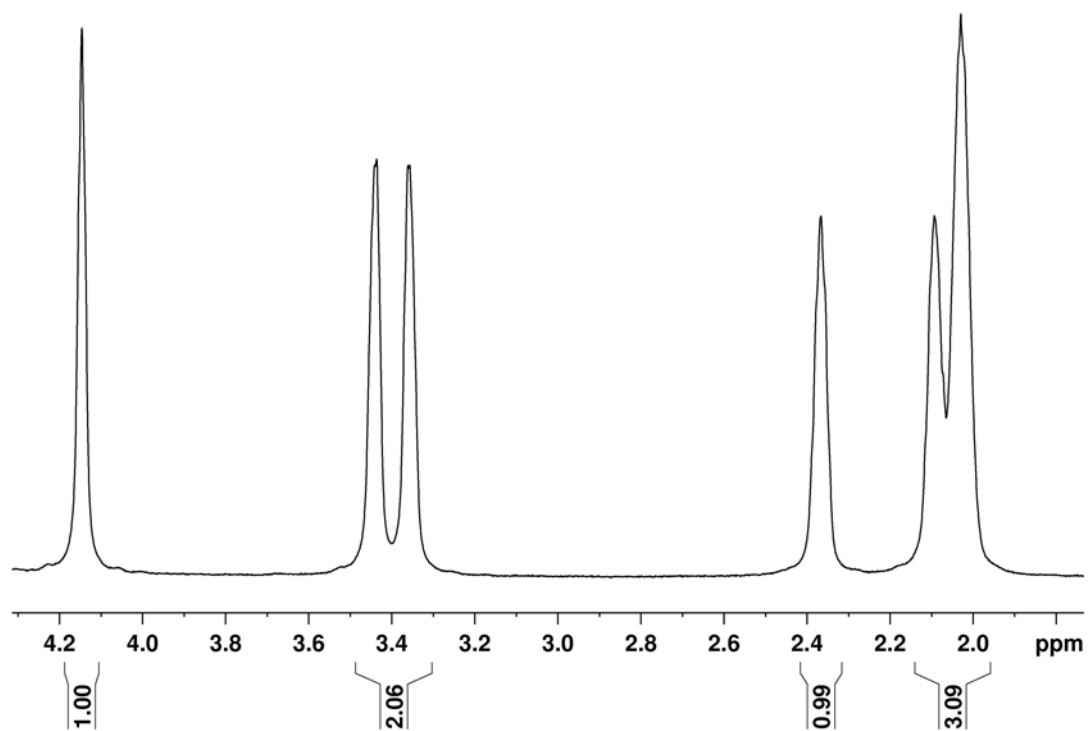
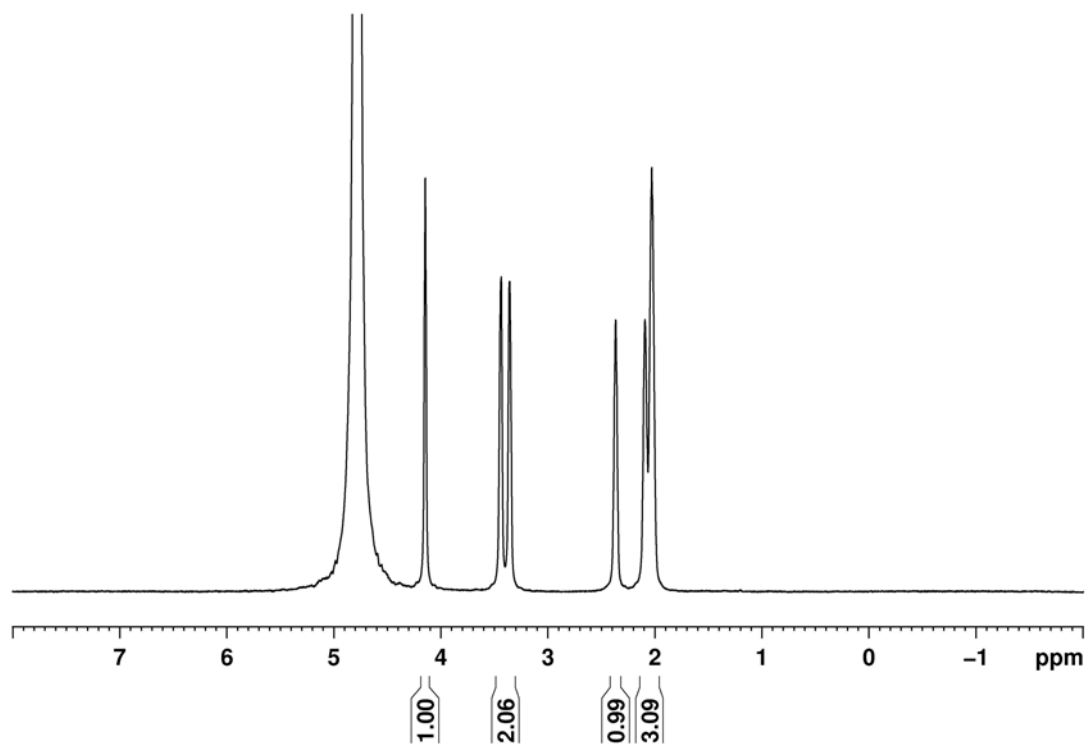


Figure SI5: ^2H NMR spectrum (122.8 MHz, ^1H NMR frequency: 800 MHz) of the anisotropic sample containing a scalemic mixture of D- and L-proline

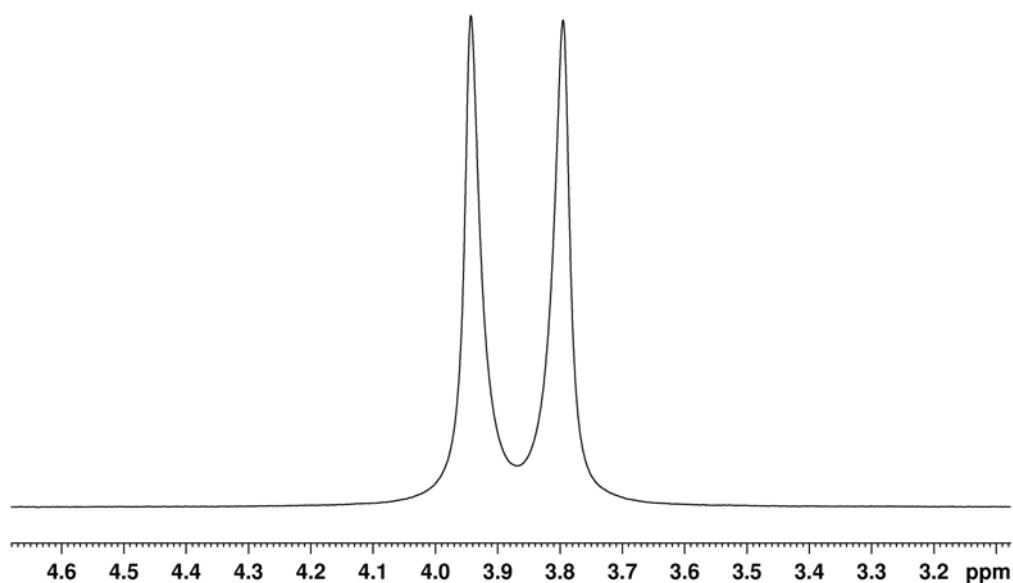
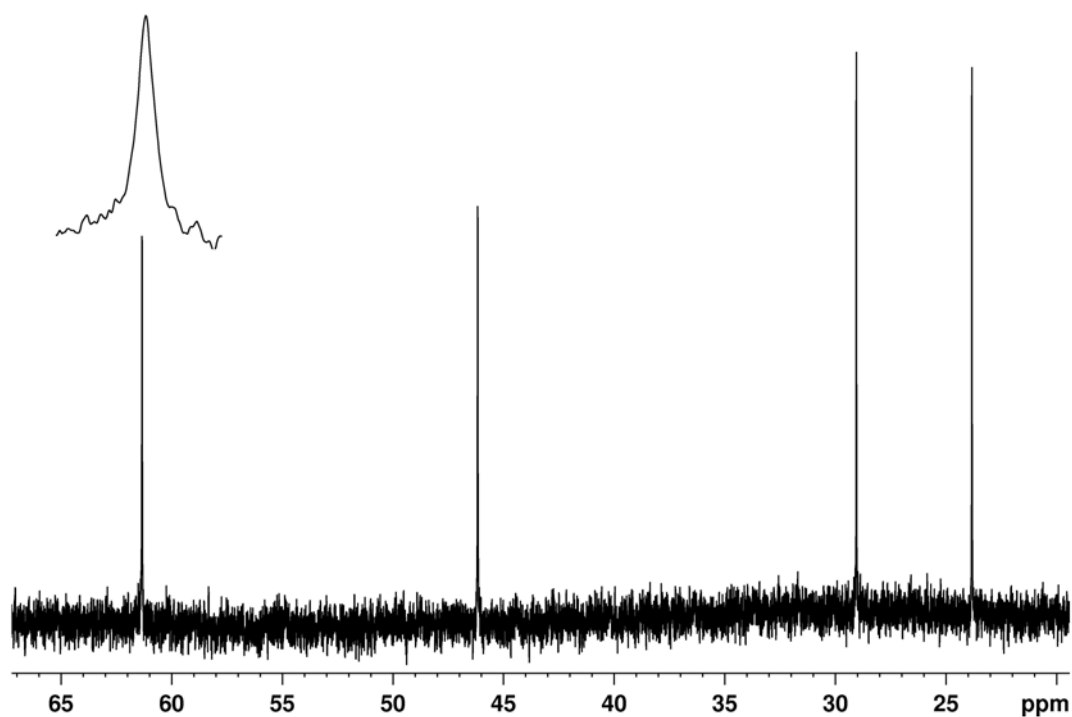


Figure SI6: ^{13}C NMR spectrum (200 MHz, proton NMR frequency: 800 MHz) of the anisotropic sample containing a scalemic mixture of D- and L-proline. Expansion of C_α -peak is shown above the signal.



References:

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- ² A. Enthart, J. C. Freudenberger, J. Furrer, H. Kessler, B. Luy, *J. Magn. Reson.* **2008**, *192*, 314.
- ³ A. Marx, C. M. Thiele, *Chem. Eur. J.* **2009**, *15*, 254.