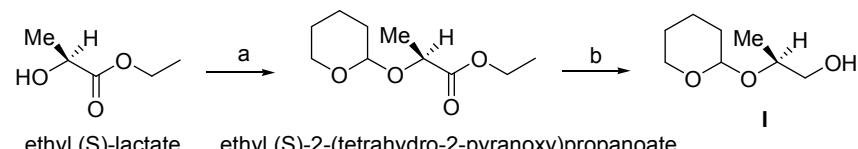


## Supplementary Information

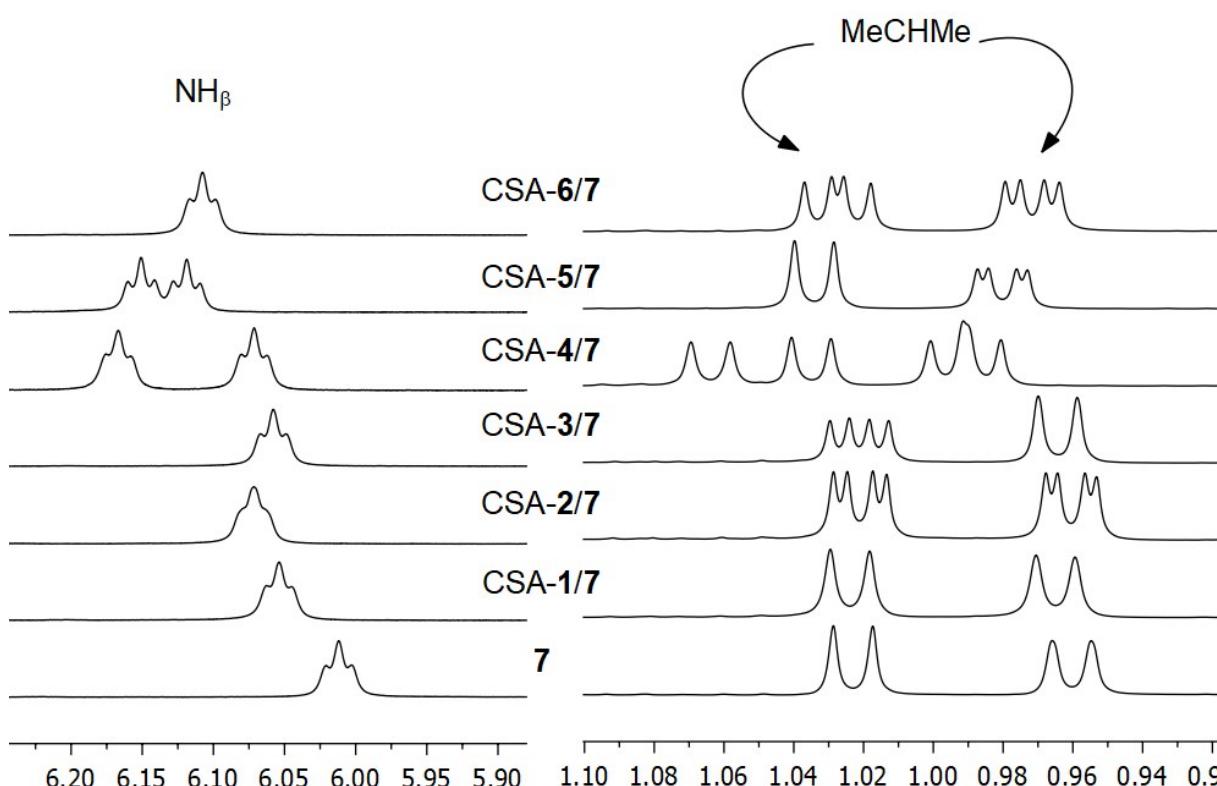
### Chiral Mono- and Dicarbamates Derived from (*S*)-Ethyl Lactate: Convenient Chiral Solvating Agents for the Direct and Efficient Enantiodiscrimination of Amino Acids Derivatives by $^1\text{H}$ NMR Spectroscopy

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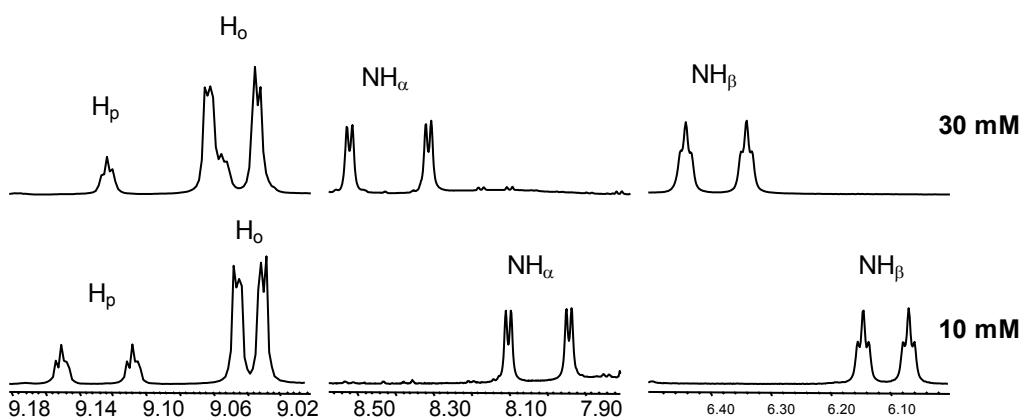
**Scheme S1.** Synthesis of I:<sup>21</sup> a) dihydropyran, HCl (12 N), RT, 16 h; b) LiAlH<sub>4</sub>, Et<sub>2</sub>O, 0 °C.



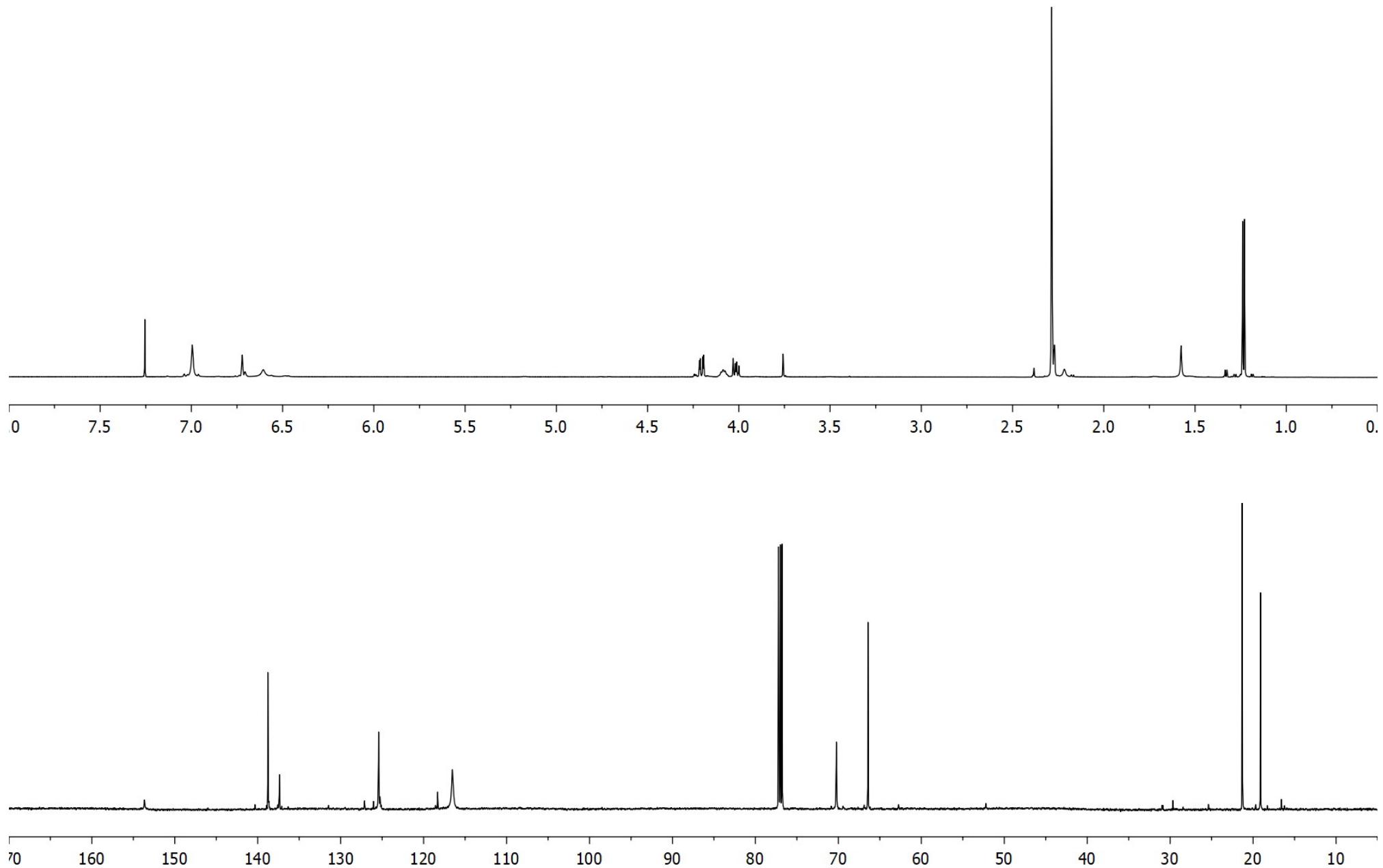
**Figure S1.**  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ , 25 °C) spectral regions corresponding to  $\text{NH}_\beta$  and methyl protons of **7** (10 mM) in the presence of one equivalent of CSA

**Table S1.**  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ , 25 °C) non-equivalences ( $\Delta\Delta\delta = |\Delta\delta_{\text{S}} - \Delta\delta_{\text{R}}|$ , ppm) data of **7** (30 mM) in the presence of one equivalent of CSA (30 mM)

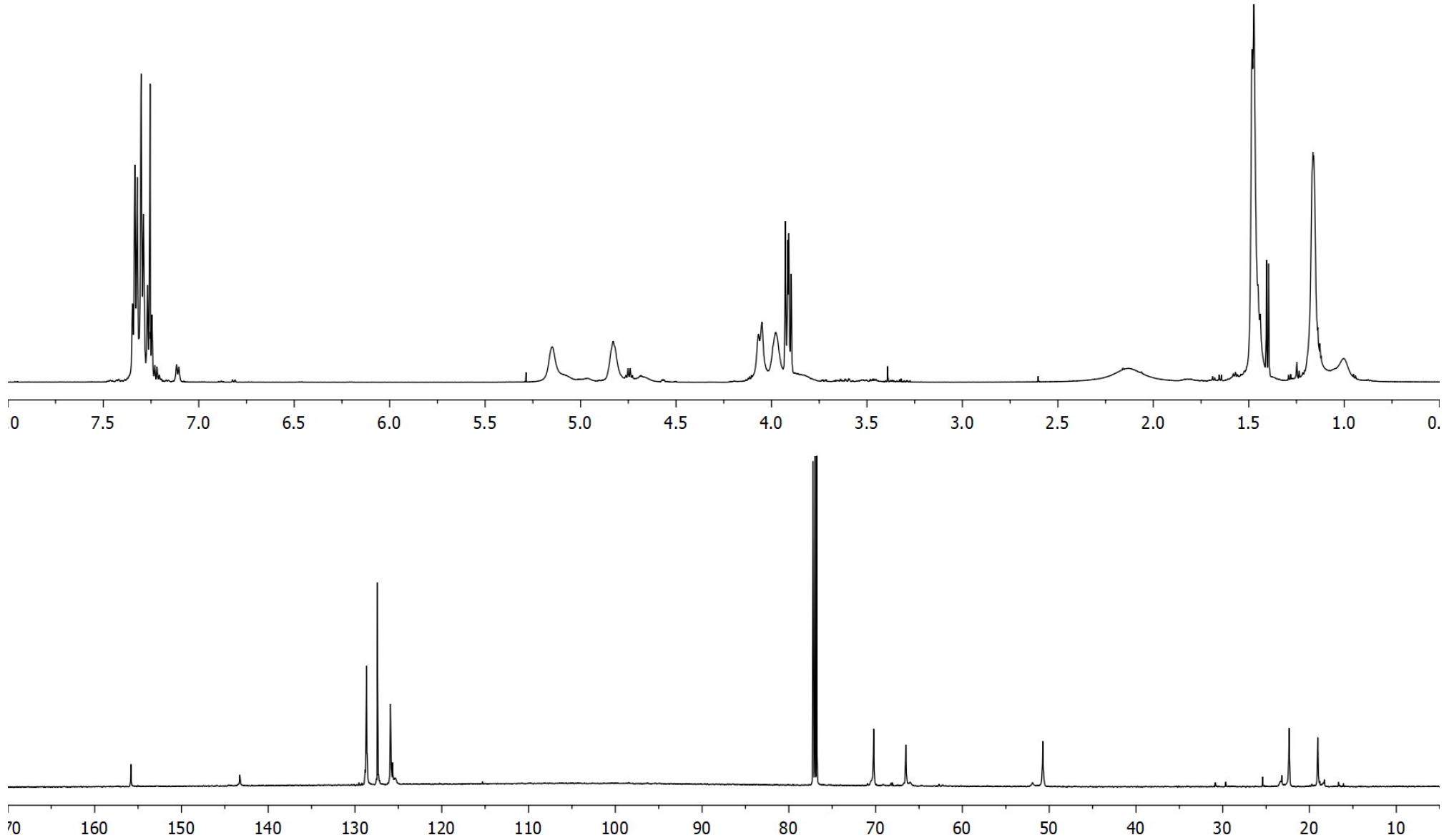
substrate	proton	CSA-1	CSA-2	CSA-3	CSA-4	CSA-5	CSA-6
<b>7</b>	$\text{NH}_{\alpha}$	0.006	0.034	0.021	0.200	0.073	0.016
	$\text{NH}_{\beta}$	-	0.005	-	0.101	0.039	-
	$\text{CH}^*$	0.002	0.006	0.004	0.036	0.015	0.004
	$\text{H}_{\text{ortho}}$	-	0.010	0.004	0.029	-	-
	$\text{H}_{\text{para}}$	0.004	0.004	0.003	0.067	0.021	0.003



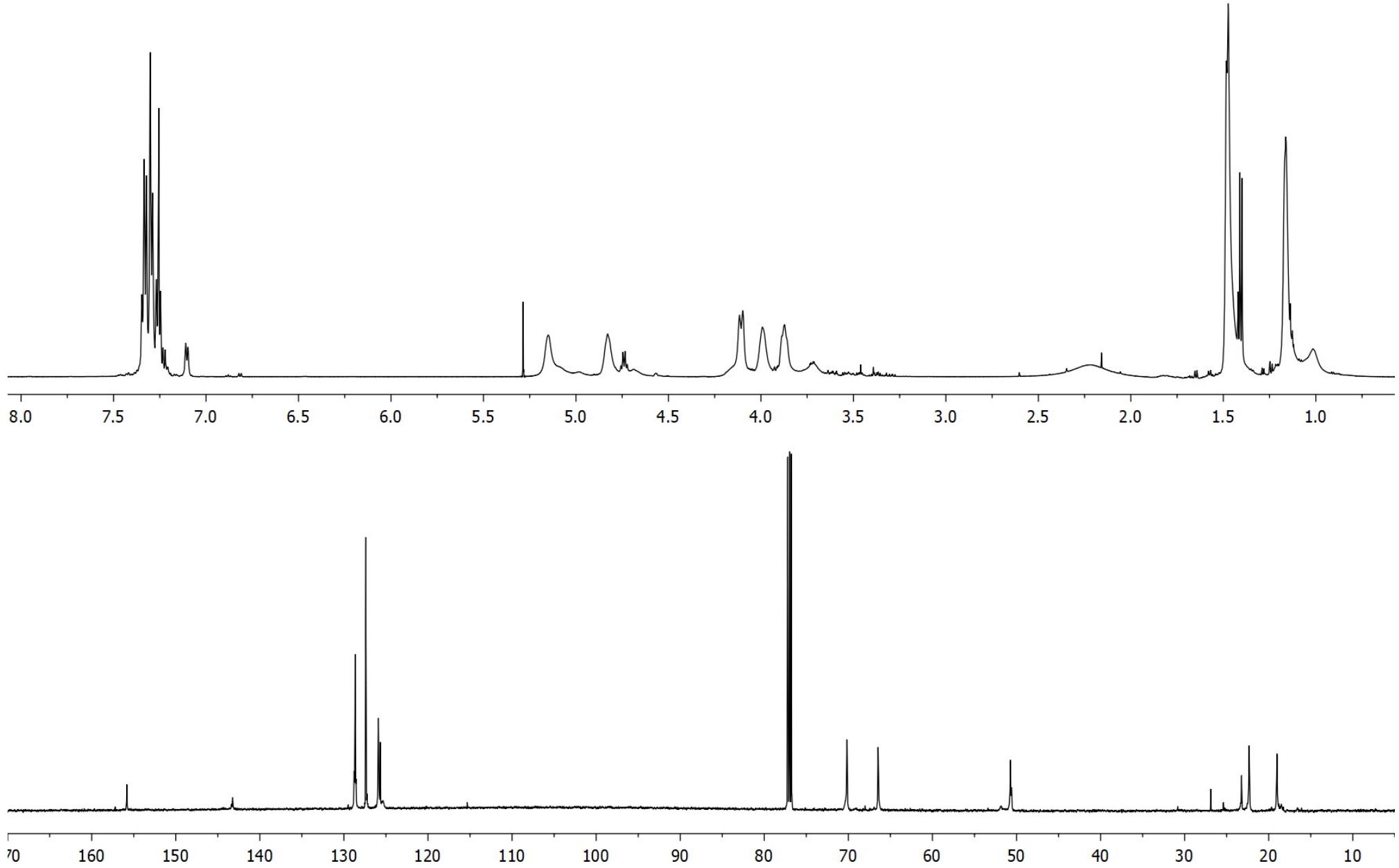
**Figure S2.**  $^1\text{H}$  NMR (600 MHz,  $\text{CDCl}_3$ , 25 °C) spectral regions corresponding to DNB protons and amide ( $\text{NH}_{\alpha}$  and  $\text{NH}_{\beta}$ ) protons of **7** (10 mM and 30 mM) in the presence of one equivalent of CSA



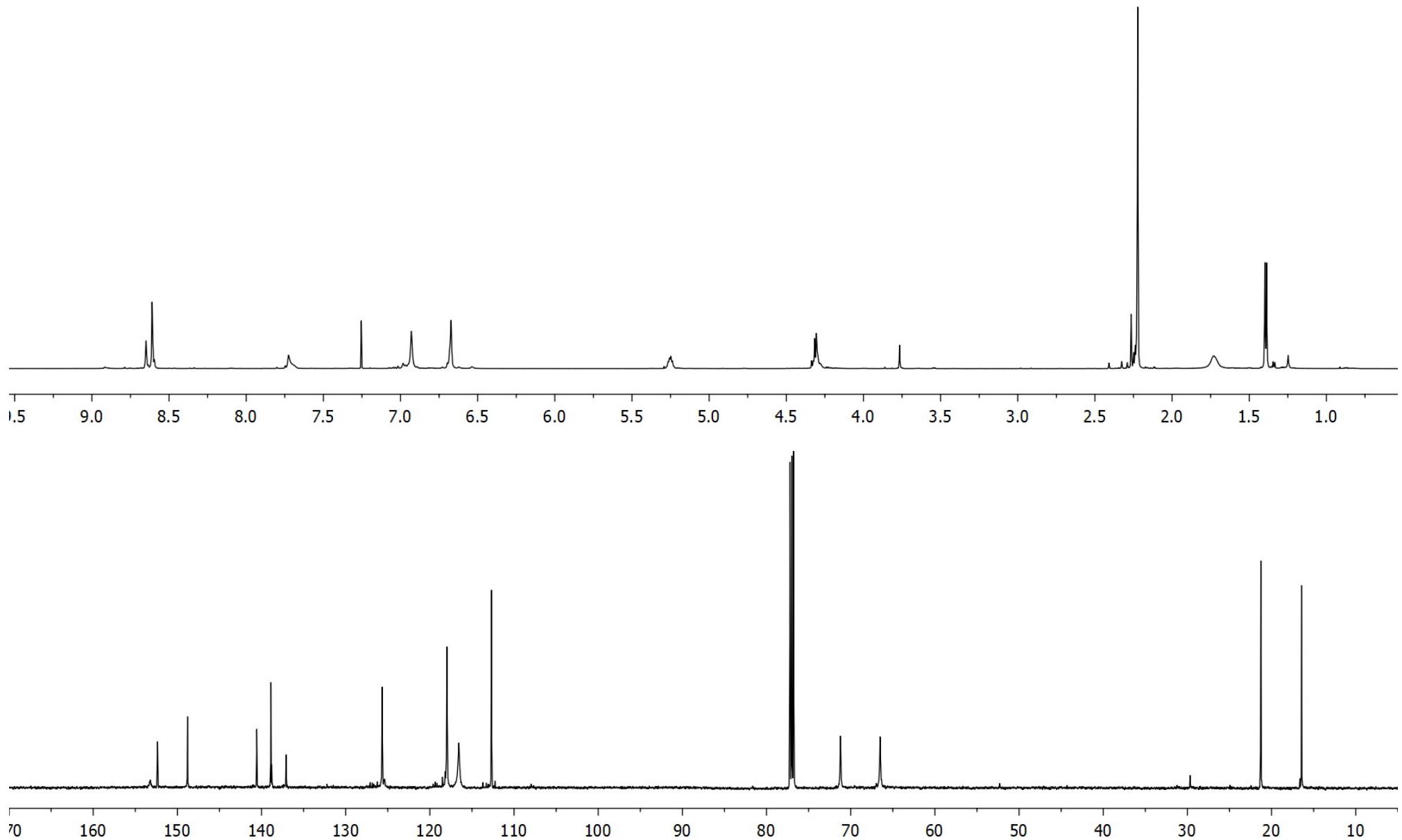
**Figure S3.**  $^1\text{H}$  (600 MHz,  $\text{CDCl}_3$ , 25 °C) and  $^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ , 25 °C) NMR spectra of CSA-1



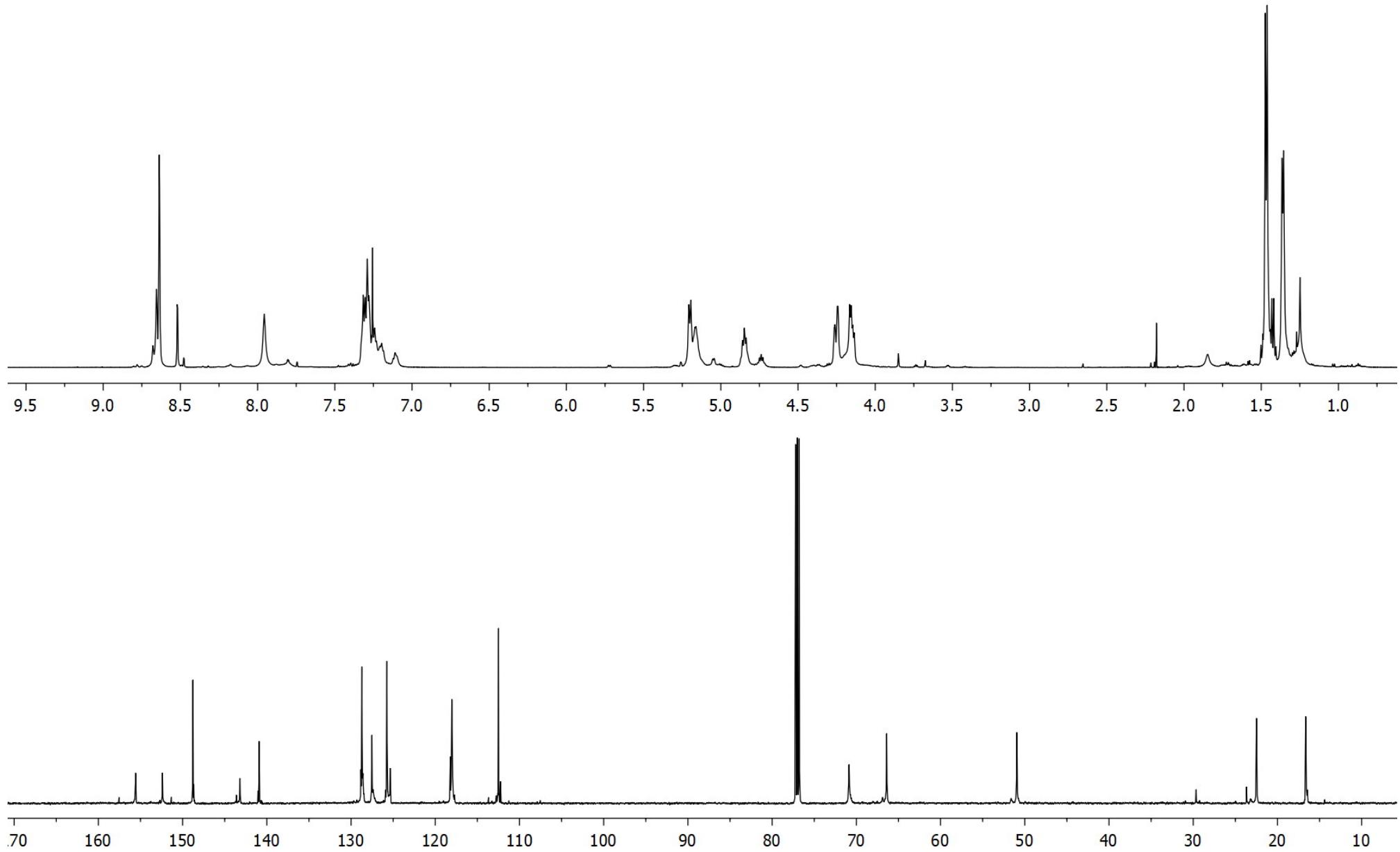
**Figure S4.**  $^1\text{H}$  (600 MHz,  $\text{CDCl}_3$ , 25 °C) and  $^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ , 25 °C) NMR spectra of CSA-2



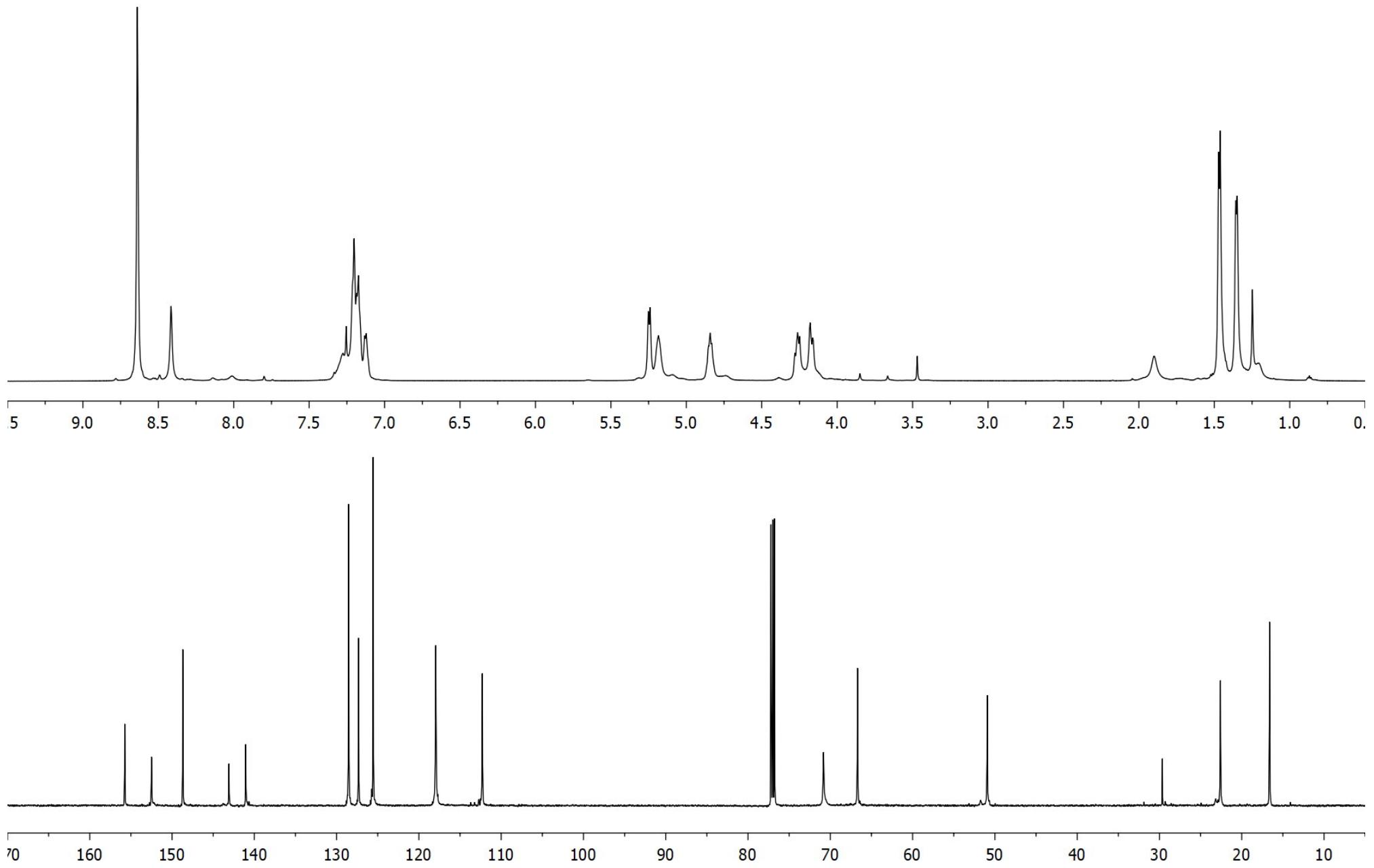
**Figure S5.**  $^1\text{H}$  (600 MHz,  $\text{CDCl}_3$ , 25 °C) and  $^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ , 25 °C) NMR spectra of CSA-3



**Figure S6.**  $^1\text{H}$  (600 MHz,  $\text{CDCl}_3$ , 25 °C) and  $^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ , 25 °C) NMR spectra of CSA-4



**Figure S7.**  $^1\text{H}$  (600 MHz,  $\text{CDCl}_3$ , 25 °C) and  $^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ , 25 °C) NMR spectra of CSA-5



**Figure S8.**  $^1\text{H}$  (600 MHz,  $\text{CDCl}_3$ , 25 °C) and  $^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ , 25 °C) NMR spectra of CSA-6