

**[HL<sub>2</sub>][P(1,2-O<sub>2</sub>C<sub>6</sub>Cl<sub>4</sub>)<sub>3</sub>] (L = THF, DMF): Brønsted Acid Initiators for the Polymerization of *n*-Butyl Vinyl Ether and *p*-Methoxystyrene**

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Supporting Information

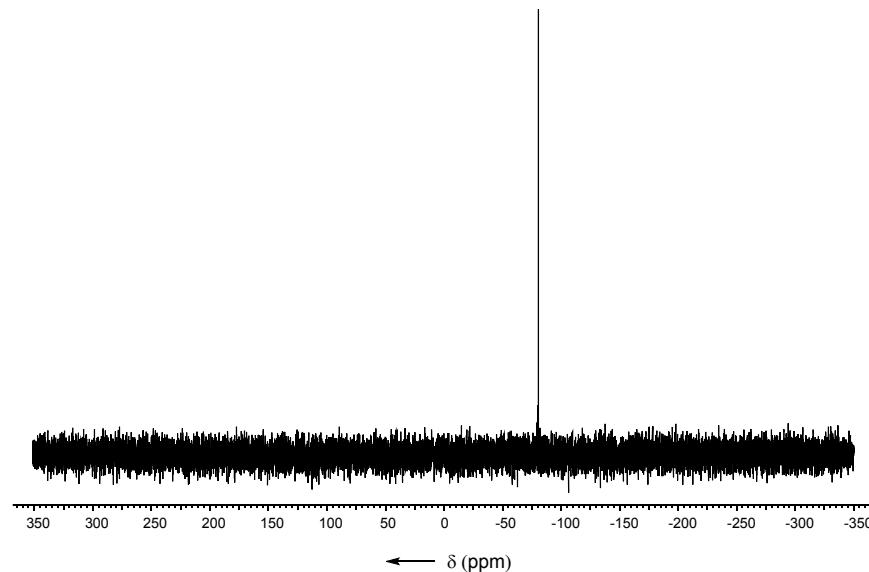


Fig. S1. <sup>31</sup>P{<sup>1</sup>H}-NMR (121 MHz, CD<sub>3</sub>CN, 25 °C) spectrum of H(DMF)<sub>2</sub>[1].

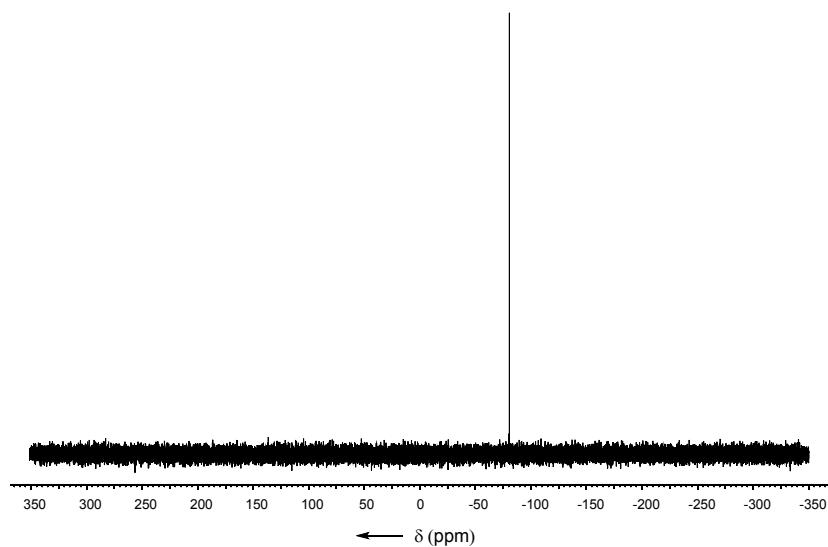


Fig. S2.  $^{31}\text{P}\{\text{H}\}$ -NMR (121 MHz,  $\text{CD}_3\text{CN}$ , 25 °C) spectrum of  $\text{H}(\text{THF})_2[\mathbf{1}]$ .

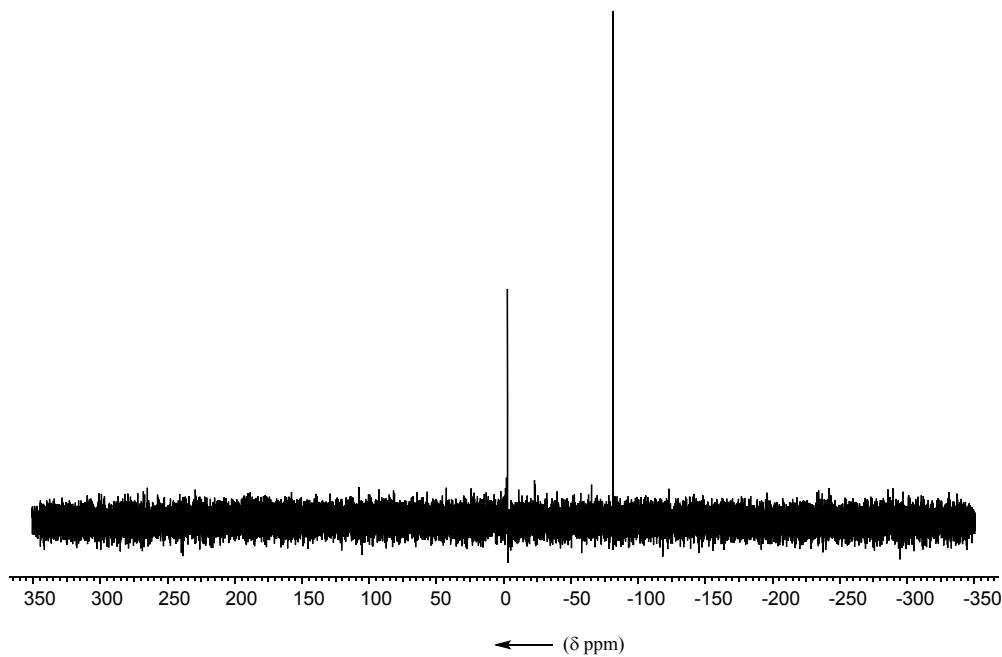


Fig. S3.  $^{31}\text{P}\{\text{H}\}$ -NMR (121 MHz,  $\text{CD}_3\text{CN}$ , 25 °C) spectrum of  $\text{H}(\text{THF})(\text{CH}_3\text{CN})[\mathbf{1}]$ .

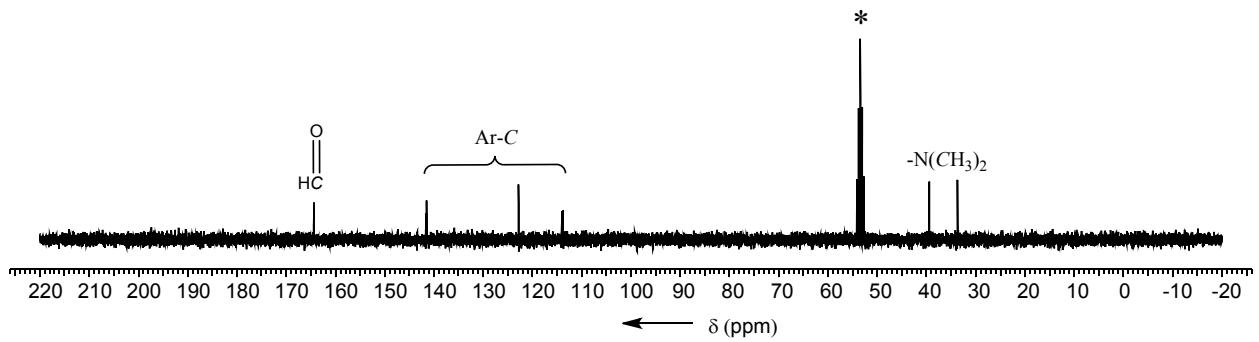


Fig. S4.  $^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CD}_2\text{Cl}_2$ , 25 °C) spectrum of  $\text{H}(\text{DMF})_2[\mathbf{1}]$ . (\*) indicates residual  $\text{CHDCl}_2$ .

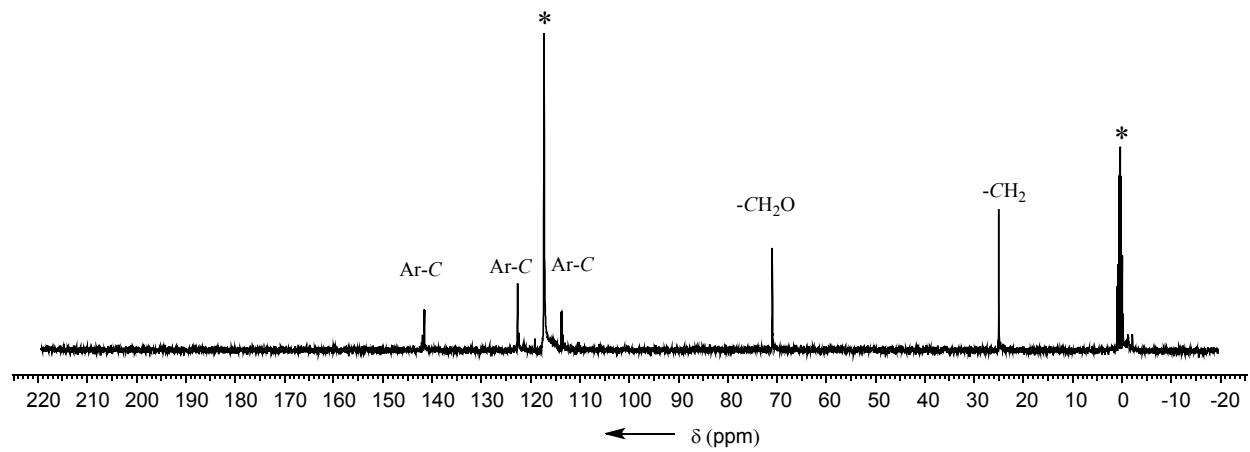


Fig. S5.  $^{13}\text{C}\{^1\text{H}\}$  NMR (75 MHz,  $\text{CD}_3\text{CN}$ , 25 °C) spectrum of  $\text{H}(\text{THF})_2[\mathbf{1}]$ . (\*) indicates residual  $\text{CHD}_2\text{CN}$ .

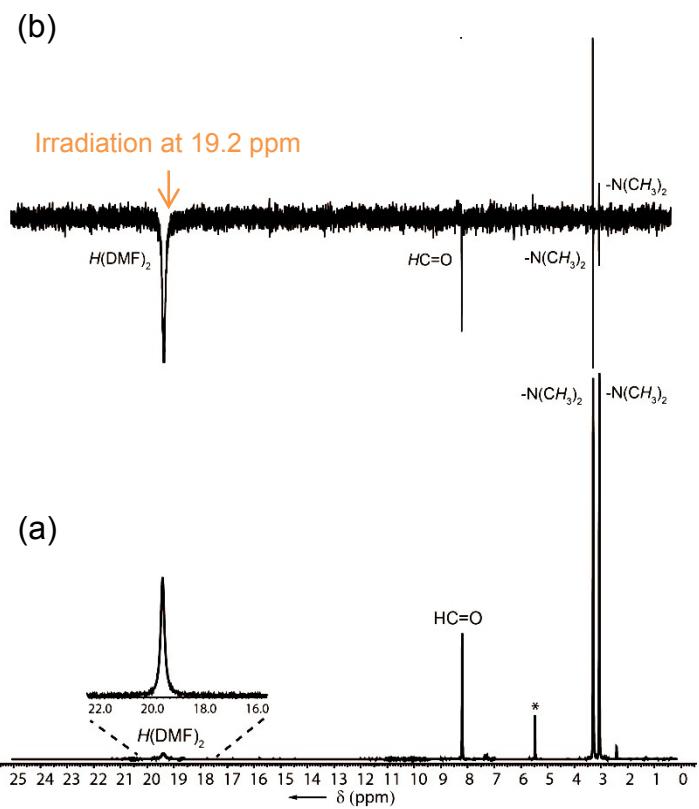


Fig. S6. (a)  $^1\text{H}$ -NMR Spectrum of  $\text{H}(\text{DMF})_2[\mathbf{1}]$  at  $-85^\circ\text{C}$ . (b) 1D  $^1\text{H}$ -NOE spectrum of  $\text{H}(\text{DMF})_2[\mathbf{1}]$  at  $-85^\circ\text{C}$ . (\*) indicates residual  $\text{CHDCl}_2$ .

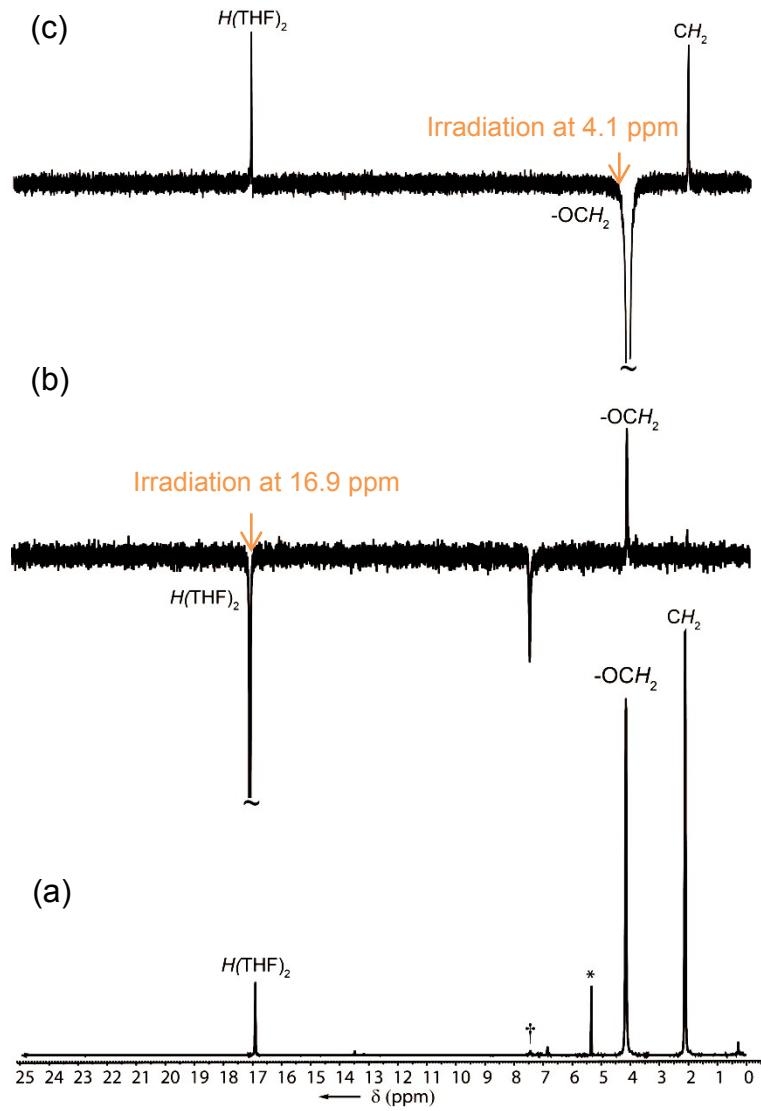


Fig. S7. (a)  $^1\text{H}$ -NMR Spectrum of  $\text{H}(\text{THF})_2[\mathbf{1}]$  at  $-85^\circ\text{C}$ . (b) and (c) 1D  $^1\text{H}$ -NOE spectrum of  $\text{H}(\text{THF})_2[\mathbf{1}]$  at  $-85^\circ\text{C}$ . (\*) indicates residual  $\text{CHDCl}_2$ .

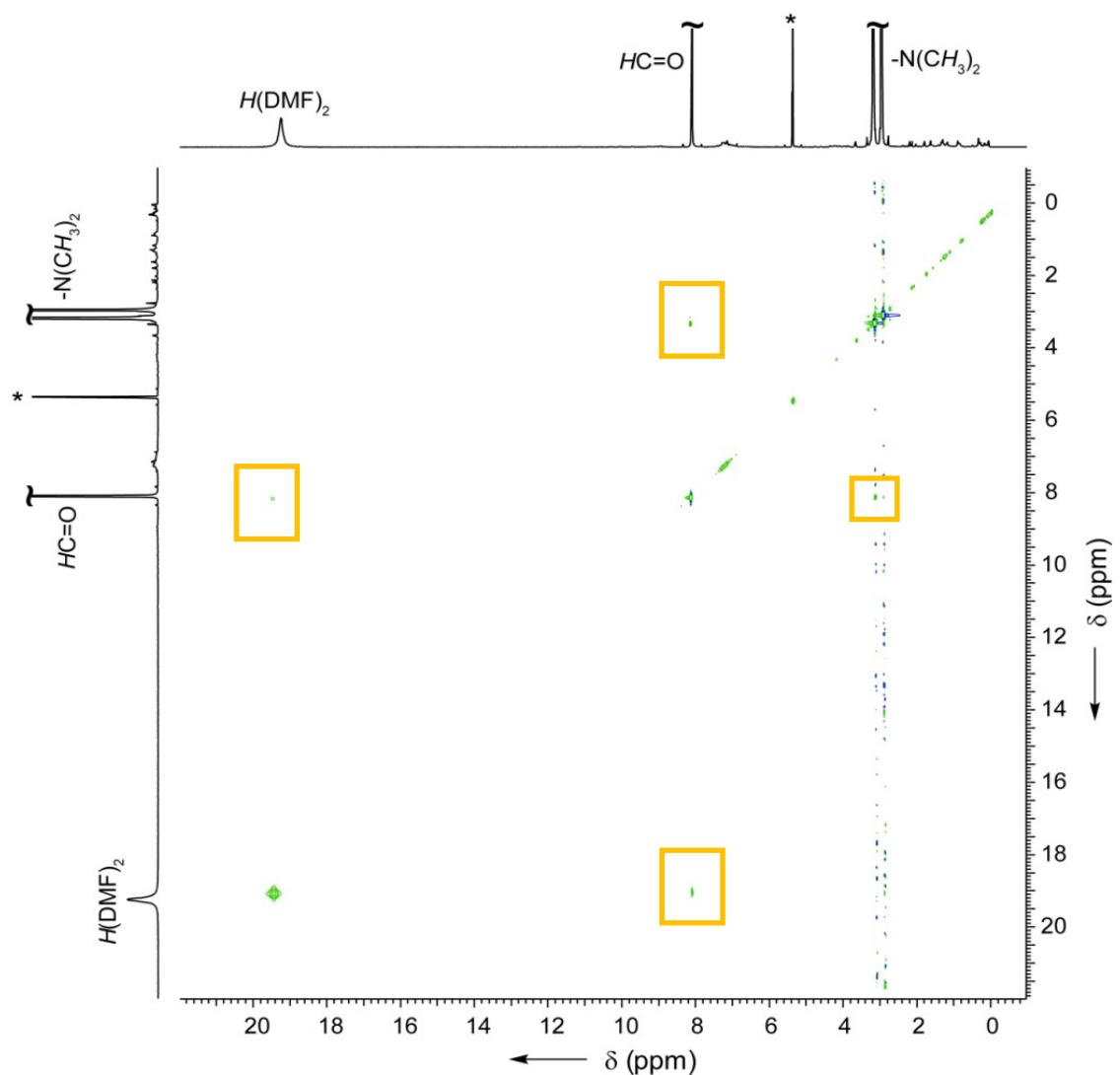


Fig. S8.  $^1\text{H}$ - $^1\text{H}$  NOESY experiment of  $\text{H}(\text{DMF})_2[\mathbf{1}]$  at  $-85^\circ\text{C}$ . (\*) indicates residual  $\text{CHDCl}_2$ .

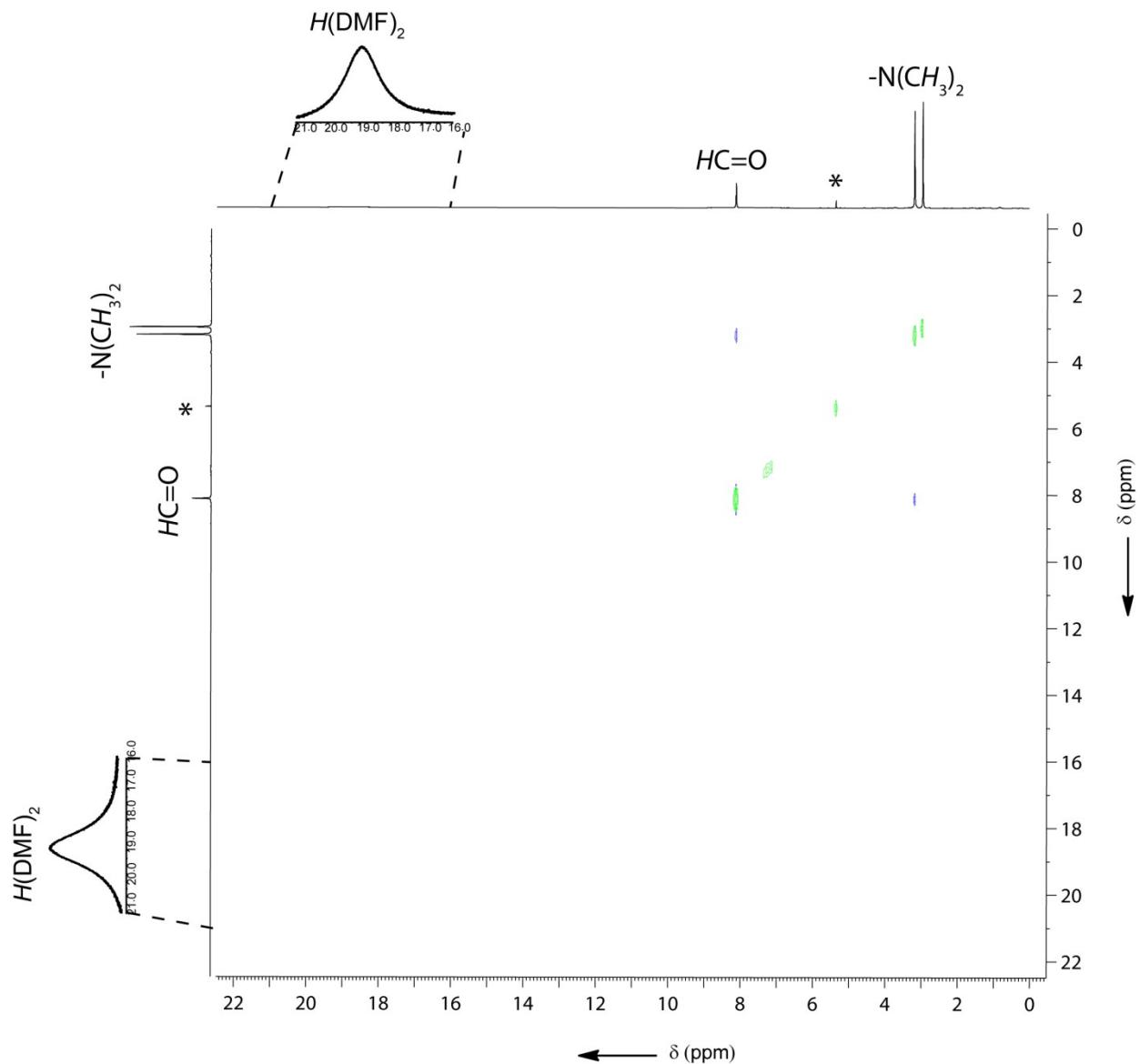


Fig. S9.  $^1\text{H}$ - $^1\text{H}$  ROESY experiment of  $\text{H}(\text{DMF})_2[\mathbf{1}]$  at  $-85^\circ\text{C}$ . (\*) indicates residual  $\text{CHDCl}_2$ .

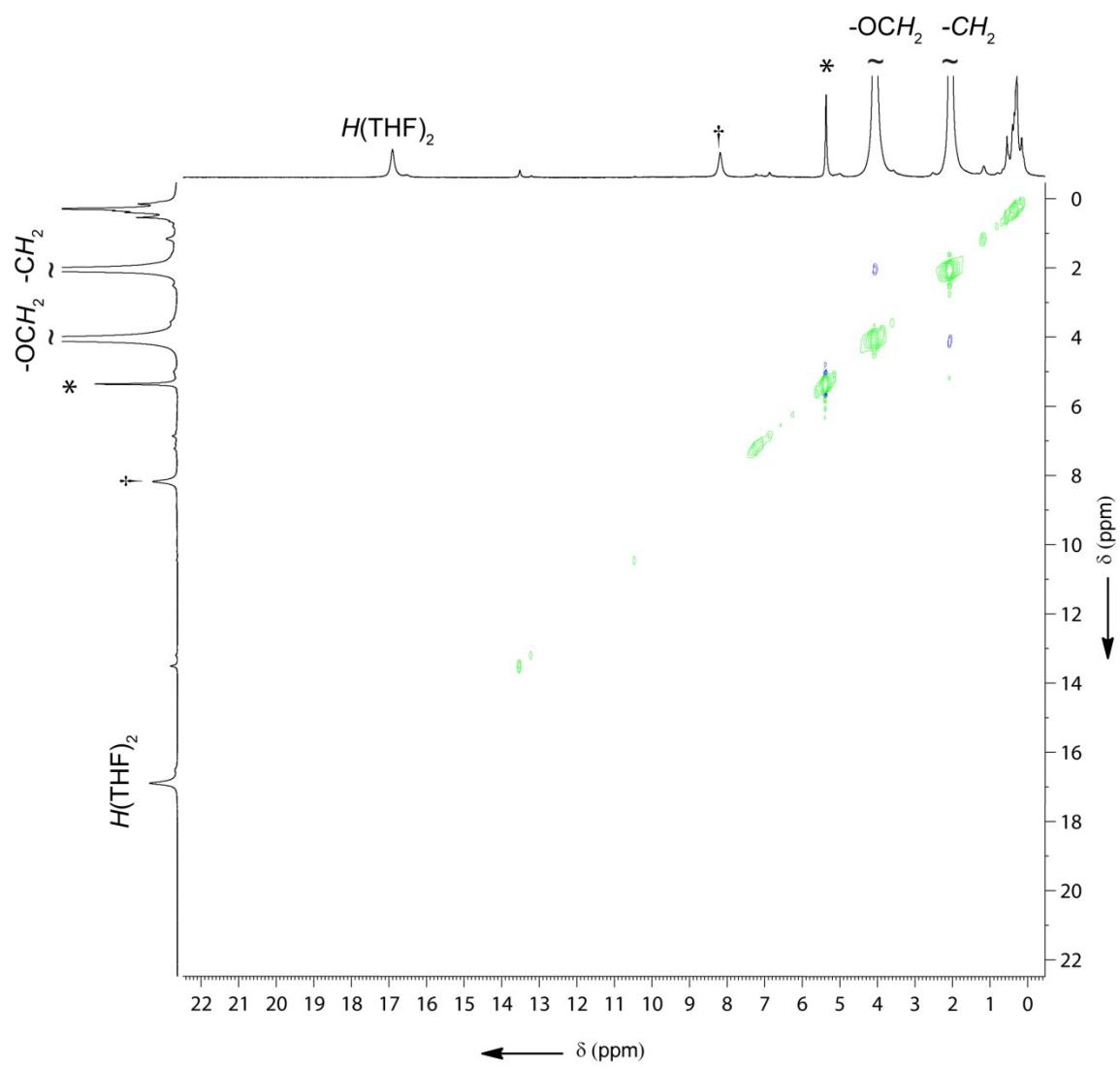


Fig. S10.  $^1\text{H}$ - $^1\text{H}$  ROESY experiment of  $\text{H}(\text{THF})_2[\mathbf{1}]$  at  $-85^\circ\text{C}$ . (\*) indicates residual  $\text{CHDCl}_2$ , (†) indicates residual toluene solvent.