



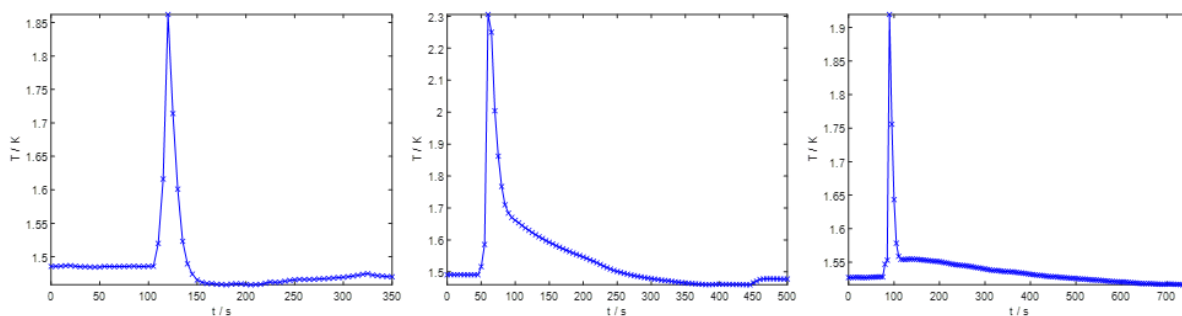
*Supplement of*

## **A novel sample handling system for dissolution dynamic nuclear polarization experiments**

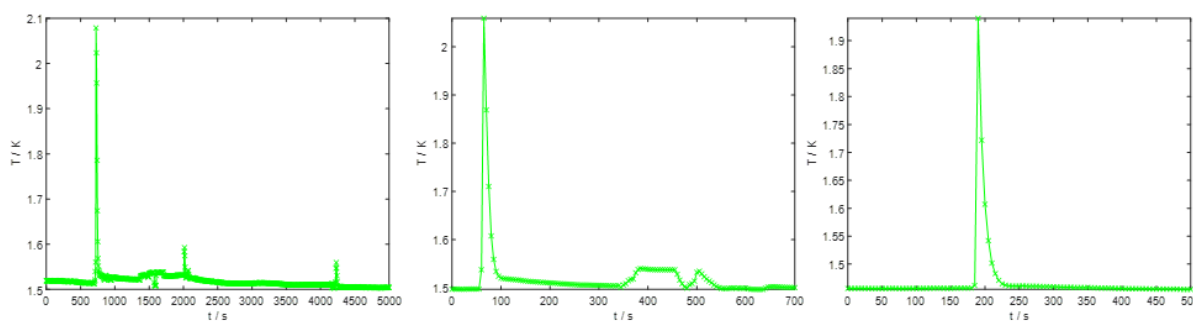
**Thomas Kress et al.**

*Correspondence to:* Dennis Kurzbach (dennis.kurzbach@univie.ac.at)

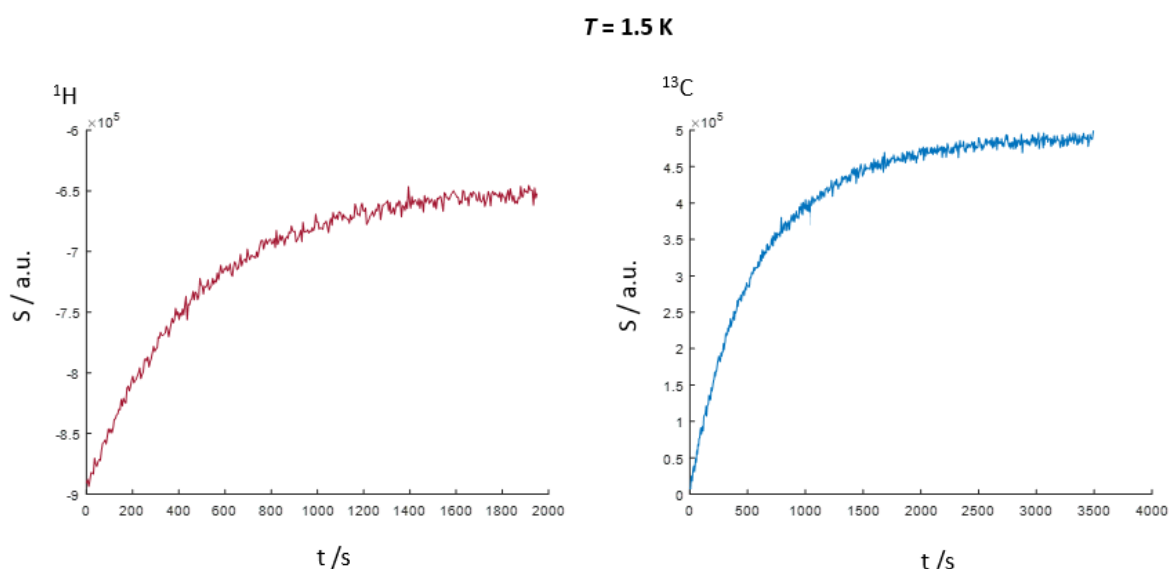
The copyright of individual parts of the supplement might differ from the article licence.



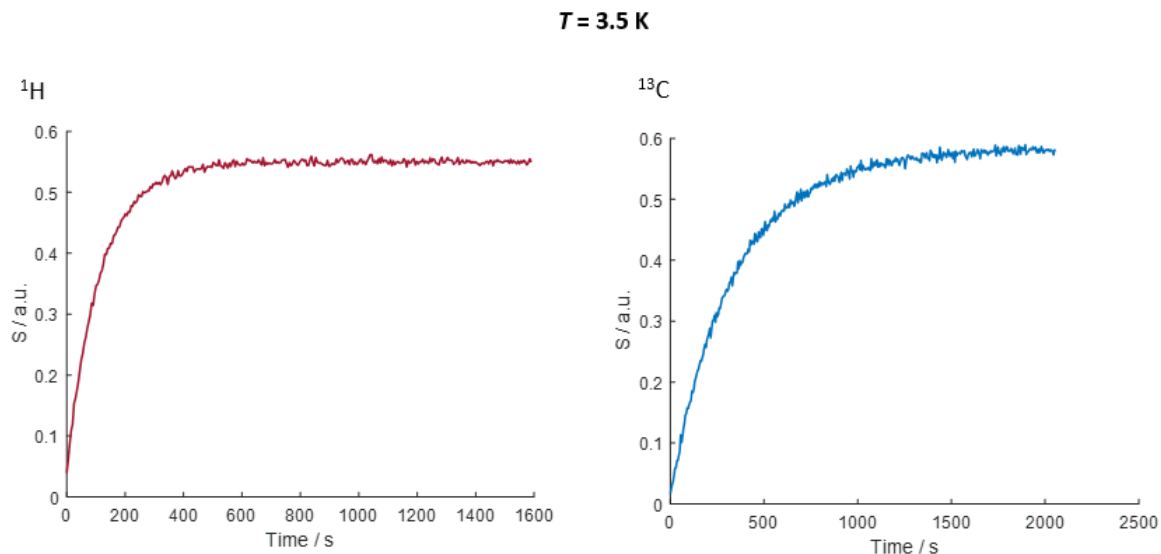
**Figure S1.** Temperature profiles for three different dissolutions. Note how the major temperature shock decays within a minute. Depending on the liquid helium level within the VTI, the system needs between one and 10 minutes to fully equilibrate after the dissolution experiment.



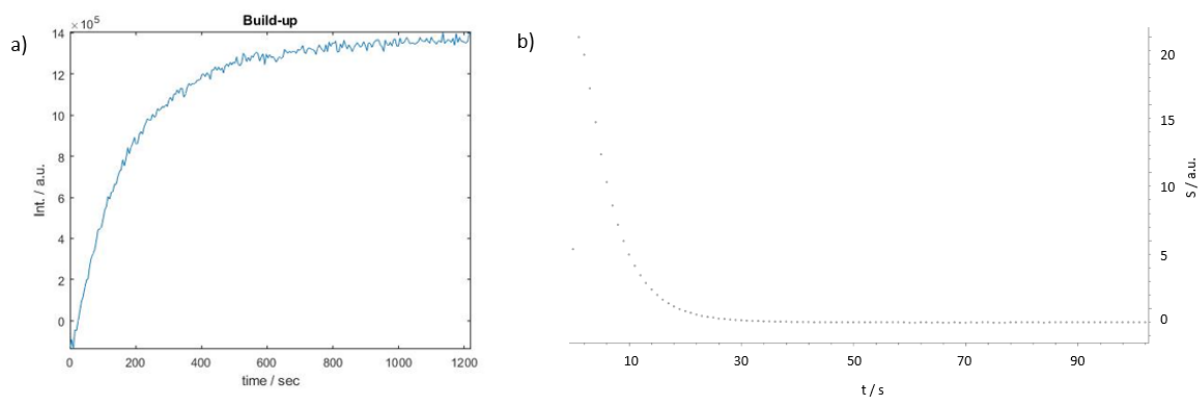
**Figure S2.** Temperature profiles for three different sample insertions. Note how the major temperature shock decays within a minute. As for dissolutions, depending on the liquid helium level within the VTI, the system needs between one and 10 minutes to fully equilibrate after the insertion.



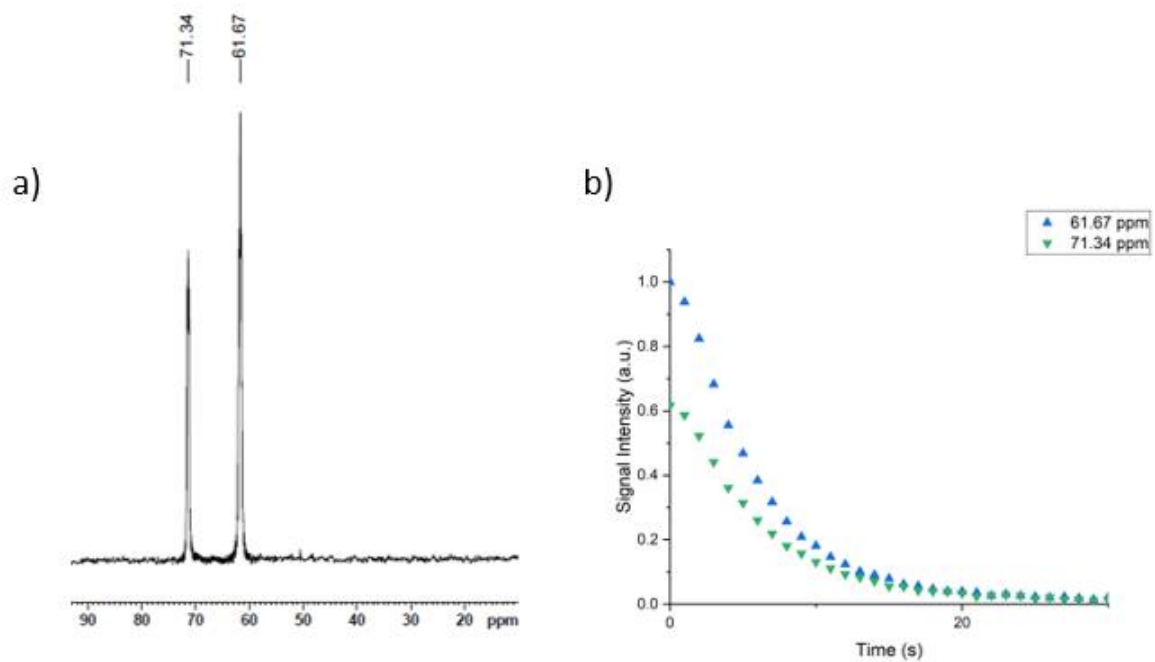
**Figure S3.** DNP build-up curves for  $^1\text{H}$  and  $^{13}\text{C}$  nuclei recorded at 1.5 K, at a TEMPOL concentration of 40 mM and a 50  $\mu\text{L}$  sample (10%  $\text{H}_2\text{O}$ , 40%  $\text{D}_2\text{O}$ , 50% glycerol- $\text{d}_8$ , 3 M sodium acetate).



**Figure S4.** DNP build-up curves for  $^1\text{H}$  and  $^{13}\text{C}$  nuclei recorded at 3.5 K, at a TEMPOL concentration of 40 mM and a 50  $\mu\text{L}$  sample (10%  $\text{H}_2\text{O}$ , 40%  $\text{D}_2\text{O}$ , 50% glycerol- $\text{d}_8$ , 3 M sodium acetate).



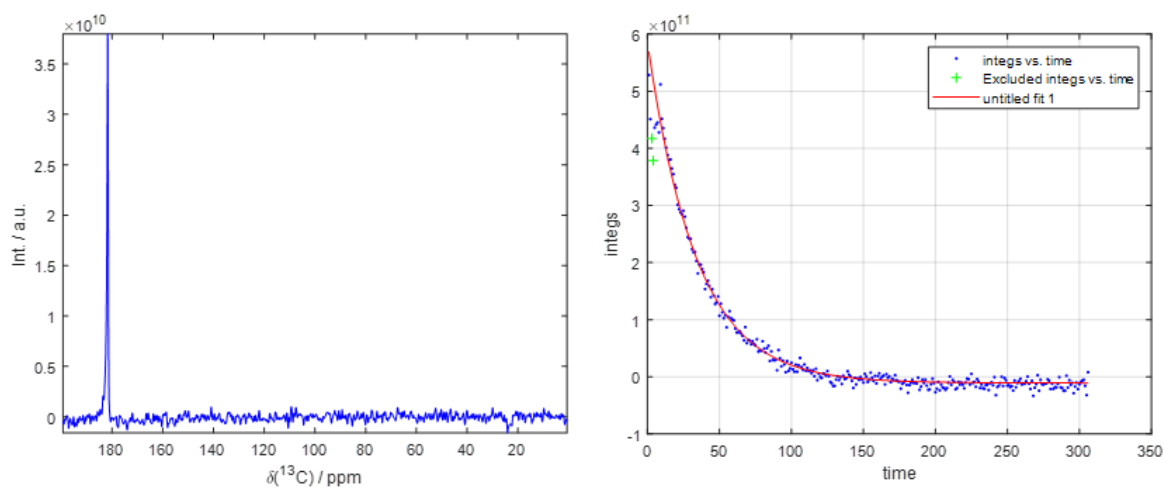
**Figure S5.** a) DNP build-up curves for  $^1\text{H}$  nuclei recorded at 1.5 K, at a TEMPOL concentration of 40 mM and a 150  $\mu\text{L}$  sample (10%  $\text{H}_2\text{O}$ , 40%  $\text{D}_2\text{O}$ , 50% glycerol- $\text{d}_8$ ). b) Signal decay of the hyperpolarized water line upon injection into a 500 MHz Bruker NMR spectrometer. The signal enhancement was >800-fold in comparison to thermal equilibrium.



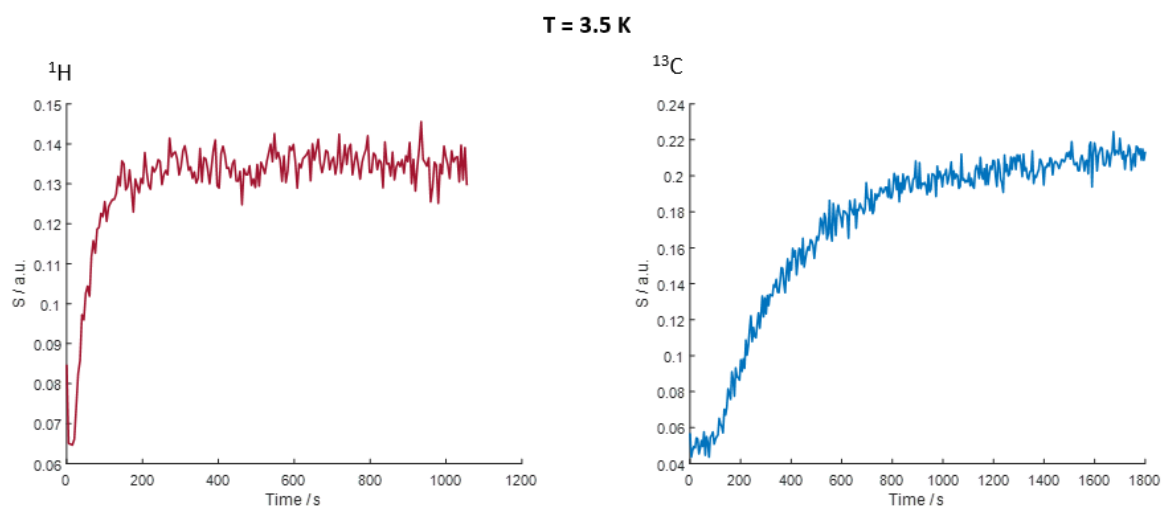
**Figure S6.** a)  $^{13}\text{C}$ -hyperpolarized NMR signal of glycerol- $\text{d}_8$  upon injection into a 500 MHz Bruker NMR spectrometer. b) Signal decay of the two glycerol signals. The signal enhancement was  $>3000$  in comparison to the signal in thermal equilibrium.



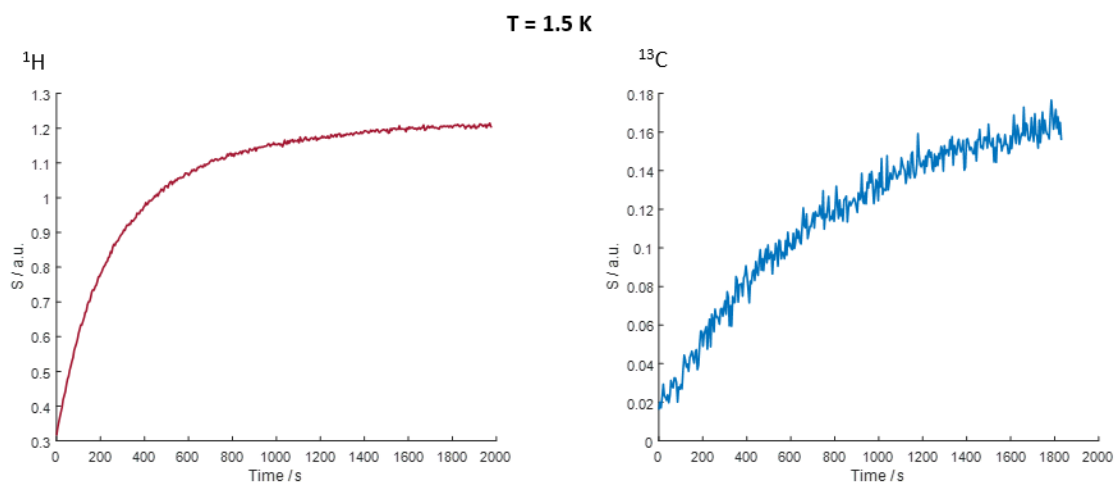
**Figure S7.** Picture of the magnetic tunnel used to transfer the sample to the 500 MHz NMR spectrometer. (Images by M. Negroni.)



**Figure S8.** a)  $^{13}\text{C}$ -hyperpolarized NMR signal of sodium acetate upon injection into a 500 MHz Bruker NMR spectrometer. b) Signal decay of the two acetate signal.



**Figure S9.** DNP build-up curves for  $^1\text{H}$  and  $^{13}\text{C}$  nuclei recorded at 3.5 K, at a TEMPOL concentration of 70 mM and a 50  $\mu\text{L}$  sample (10%  $\text{H}_2\text{O}$ , 40%  $\text{D}_2\text{O}$ , 50% glycerol- $\text{d}_8$ , 3 M sodium acetate).



**Figure S10.** DNP build-up curves for  $^1\text{H}$  and  $^{13}\text{C}$  nuclei recorded at 1.5 K, at a TEMPOL concentration of 70 mM and a 50  $\mu\text{L}$  sample (10%  $\text{H}_2\text{O}$ , 40%  $\text{D}_2\text{O}$ , 50% glycerol-d8, 3 M sodium acetate).