Supplement of Magn. Reson., 2, 643–652, 2021
https://doi.org/10.5194/mr-2-643-2021-supplement


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MAGNETIC
RESONANCE

*Supplement of*

# Solid-state $^{1}$H spin polarimetry by $^{13}CH_3$ nuclear magnetic resonance

**Stuart J. Elliott et al.**

*Correspondence to:* Stuart J. Elliott (stuart.elliott@liverpool.ac.uk)

Contents

## 1. $^{13}C$ NMR Spectra

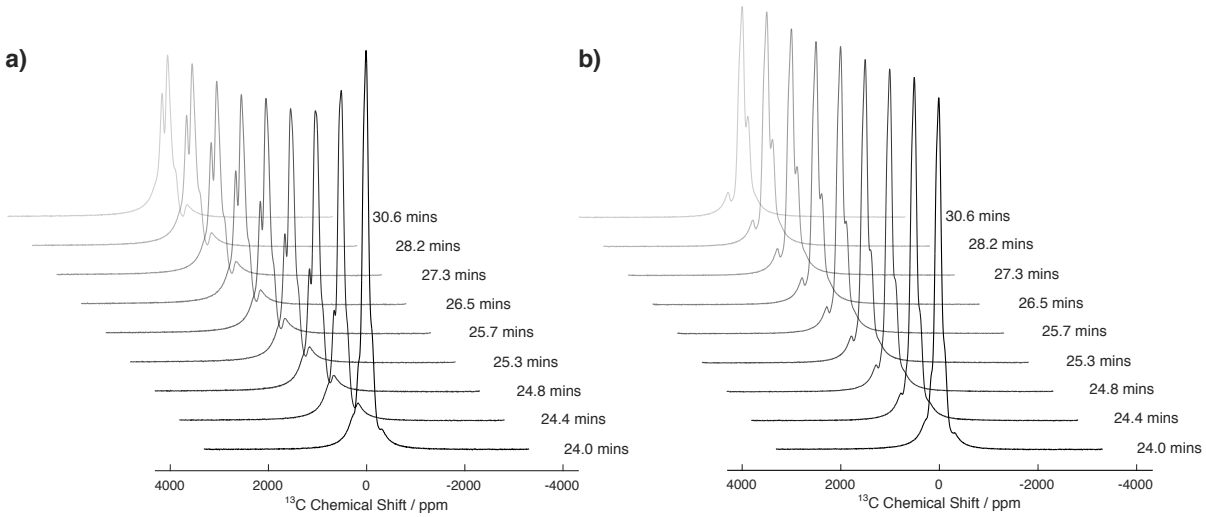


Figure S1: Relevant portions of the experimental $^{13}C$ NMR spectra belonging to the $^{13}C$-labelled methyl group ($^{13}CH_3$) of [2-$^{13}C$]sodium acetate acquired at 7.05 T ($^{1}H$ nuclear Larmor frequency = 300.13 MHz, $^{13}C$ nuclear Larmor frequency = 75.47 MHz) and 1.2 K with a single transient (*rf*-pulse flip angle = 3.5°) as a function of $^{1}H$ DNP time. (a) Positive microwave irradiation; and (b) Negative microwave irradiation. The labels indicate the $^{1}H$ DNP time at which the spectra were recorded.

Figure S1 shows the relevant part of the experimental $^{13}C$ NMR spectra acquired with a small flip angle *rf*-pulse ($\beta$ = 3.5°) as a function of $^{1}H$ DNP time. The $^{13}C$ NMR spectra in Figure S1 were acquired by using the *rf*-pulse sequence shown in Figure 1 of the main text. The timings coincide with those shown in Figure 2 of the main text.

## 2. *$^{13}$C NMR Peak Normalized CoG Deviation vs. $^{1}$H Polarization*

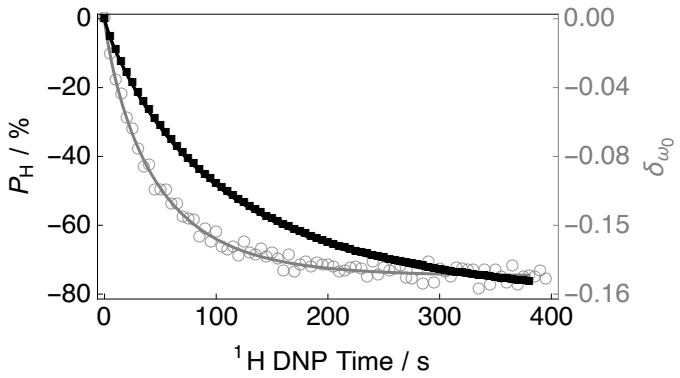


Figure S2: Experimental $^{1}$H polarization $P_H$ DNP build-up curve (black filled squares) and $^{13}$C NMR peak normalized CoG deviation $\delta_{\omega_0}$ (grey empty circles) as a function of $^{1}$H DNP time acquired at 7.05 T ($^{1}$H nuclear Larmor frequency = 300.13 MHz, $^{13}$C nuclear Larmor frequency = 75.47 MHz) and 1.2 K with a single transient per data point for the case of negative microwave irradiation. The timings coincide with those shown in Figure 2 of the main text. The black solid line indicates the best fit of the experimental data points for the $^{1}$H polarization $P_H$ DNP build-up curve, and has the corresponding fitting function: $A(1-\exp\{-(t/\tau_{DNP}^{-})^{\beta}\})$. Mean $^{1}$H DNP build-up time constant: $\langle\tau_{DNP}^{-}\rangle = 122.0 \pm 0.4$ s.

Figure S2 shows the DNP build-up curve for the $^{1}$H polarization $P_H$ as a function of $^{1}$H DNP time for negative microwave irradiation. Figure S2 also displays the $^{13}$C NMR peak normalized CoG deviation $\delta_{\omega_0}$ as a function of $^{1}$H DNP time.

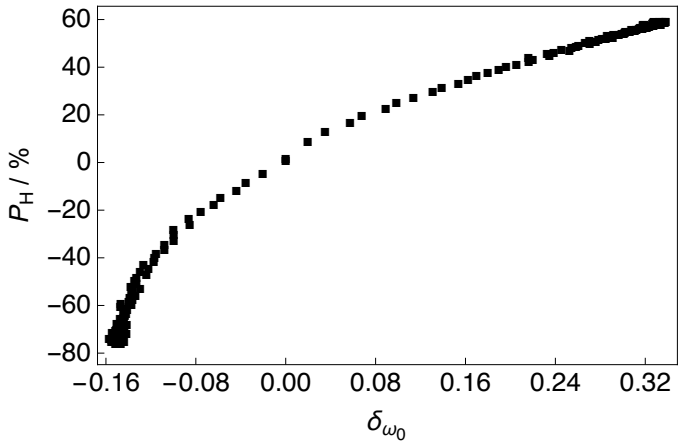


Figure S3: Experimental $^{1}$H polarizations $P_H$ as a function of the $^{13}$C NMR peak CoG normalized deviation $\delta_{\omega_0}$ acquired at 7.05 T ($^{1}$H nuclear Larmor frequency = 300.13 MHz, $^{13}$C nuclear Larmor frequency = 75.47 MHz) and 1.2 K with a single transient per data point. The absolute $^{1}$H polarizations $P_H$ were measured by comparison with a thermal equilibrium $^{1}$H NMR signal.

Figure S3 shows the $^{13}$C NMR peak CoG normalized deviation $\delta_{\omega_0}$ for sample **I** as a function of the $^{1}$H polarization $P_H$. An overall trend could be gleamed from this data set by fitting the experimental curve with an $n^{th}$ order spline function.