Supplementary Information

for

Paramagnet Induced Signal Quenching in MAS-DNP Experiments on Homogeneous Solutions

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Data acquisition and processing details of bleaching factor determination

Synopsis

For determination of the bleaching factor the assessment of the spectrometer stability and sensitivity over an extended period of time is of crucial importance. Table S1 lists the experimental date and the MAS stator temperature for each sample.

| Polarizing agent | conc. (mM) | date | temp. (K) |
|-------------------------|------------|------------|-----------|
| Undoped 1 | _ | 03/21/2011 | 78 |
| TOTAPOL | 5 | 03/22/2011 | 80 |
| TOTAPOL | 10 | 03/23/2011 | 79 |
| TOTAPOL | 20 | 03/23/2011 | 79 |
| TOTAPOL | 40 | 03/23/2011 | 79 |
| TOTAPOL | 2 | 03/24/2011 | 79 |
| trityl OX063 | 40 | 03/24/2011 | 81 |
| trityl OX063 | 10 | 03/24/2011 | 81 |
| trityl OX063 | 20 | 03/25/2011 | 81 |
| trityl OX063 | 60 | 03/26/2011 | 81 |
| trityl OX063 | 5 | 03/27/2011 | 81 |
| Gd-DOTA | 10 | 03/27/2011 | 81 |
| Gd-DOTA | 2 | 03/28/2011 | 81 |
| Gd-DOTA | 5 | 03/28/2011 | 81 |
| Gd-DOTA | 20 | 03/28/2011 | 82 |
| Gd-DOTA | 1 | 03/29/2011 | 81 |
| Undoped 2 ^a | _ | 03/31/2011 | 78 |
| 4-amino-TEMPO | 10 | 06/07/2011 | 83 |
| 4-amino-TEMPO | 5 | 06/08/2011 | 84 |
| 4-amino-TEMPO | 40 | 06/08/2011 | 85 |
| 4-amino-TEMPO | 60 | 06/08/2011 | 85 |
| 4-amino-TEMPO | 20 | 06/09/2011 | 84 |

Table S1. Overview of date and temperature of experiments.

^a Sample "Undoped 2" was measured because "Trityl 5 mM" and all TEMPAMINE samples were prepared from different stem solution than all other samples. While we measured $T_1^o = 54.6$ s for the first stem solution ("Undoped 1") we measured $T_1^o = 61.6$ s for the second stem solution ("Undoped 2"). All build-up time constants are scaled to $T_1^o = 54.6$ s using the relation $1/T_{B,a} - 1/T_{B,b} = 1/T_{1,a}^o - 1/T_{1,b}^o$.

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Many parameters were kept consistent during the experimental period, for example, retuning of the probe's rf channels was not necessary due to practically identical dielectric properties of all samples and therefore identical Q of the resonant circuit. Nevertheless, inherent drifts or fluctuations in sensitivity might still be present. To account for these influences, we correct the collected data (NMR signal amplitude of ¹³C-urea) by calibration to an internal standard.

Choice of internal standard

We used the NMR signal of the Vespel spacers as internal standard for NMR signal amplitude measurements. The spacer signal is well separated from the urea and glycerol signals, so it is well suited to serve as internal amplitude standard. Before amplitude determination the frequency domain spectrum was broadened by application of sinebell apodization function with a time constant of 2 ms in order to minimize noise.



Figure S1. ¹³C CP-MAS spectrum of the sample containing 10 mM trityl. The signal arising from the spacer material is used for internal standard correction.

Determination of internal standard signal amplitude of undoped samples

The internal standard amplitudes of samples "Undoped 1" and "Undoped 2" were determined by fitting the polarization buildup curve with a monoexponential function and dividing the preexponential factor by a factor of 10 (see Fig. S2). This division was necessary because "Undoped 1" and "Undoped 2" sample buildup curves were measured using a receiver gain 20 dB higher compared to the DNP enhanced signal measurements. The linearity of the receiver gain on the signal amplitude was confirmed by experiment (not shown).



Figure S2. Determination of signal amplitude I_0 and relaxation time T_1 of the standard signal of the undoped samples.

Determination of urea signal amplitude of doped samples

Urea signal amplitudes of all paramagnetically doped samples were determined by first fitting the DNP enhanced buildup curves with monoexponential functions. The preexponential factor was then divided by the enhancement factor which was determined by on-/off-signal amplitude comparison of spectra measured with a polarization period of $1.26 \times T_{\rm B}$. Amplitudes are then normalized to the amplitude of the respective undoped sample (compare Fig. S2 and Table S1). The obtained normalized signal amplitudes are shown in Fig. S3. This procedure saves a significant amount of experimental time since no off-signal buildup-curve has to be measured for each doped sample. However, it introduces a small error in the case of polarizing agents allowing for SE DNP due to a minor shortening of $T_{\rm B}$ with respect to T_1 and therefore leads to an overestimation of the respective enhancement factor. Another study has shown a shortening ratio of ~0.87 under similar conditions for 40 mM trityl [1], leading to an error in the enhancement factor of less than 7 %. Since this sample yielded the most efficient SE and therefore is supposed to induce the strongest buildup time shortening, 7% marks the upper limit of the error introduced due to this analysis.



Figure S3. Uncorrected signal amplitude of all paramagnetically doped samples. Amplitudes are normalized to those of the respective undoped samples.

Correction for varying polarization times used in measurement of internal standard signal amplitude of undoped samples

For all paramagnetically doped samples the recorded off-signal was used for internal standard amplitude determination (see Fig. S4 left). Because the off-signals were measured using various polarization periods $t = 1.26 \times T_B$ (see Fig. S4 right), which were partly shorter than $5 \times T_1$ of the standard signal, the standard signal amplitude had to be corrected for this.



Figure S4. Signal amplitude of the internal standard signal (left). Data was obtained by ¹³C CP-MAS without microwave (off-spectrum). The polarization time used is shown in the right graph.

Since it is assumed that the standard signal's T_1 does not change between different samples, the T_1 of the internal standard signal was determined by monoexponential fitting of the respective buildup curve (Fig. S2). The time constant was then used to calculate the internal standard signal amplitude at infinite polarization time for each doped sample using

$$I_0 = \frac{I}{1 - \exp\left(-\frac{t}{T_1}\right)}.$$
(S1)

The resulting corrected internal standard amplitude I_0 is depicted in Fig. S5.



Figure S5. Internal standard amplitude corrected for variation in polarization time.

Final correction of NMR signal bleaching data using internal standard signal amplitude

The ¹³C-urea amplitudes were corrected for spectrometer sensitivity variation by division by the respective internal standard signal amplitude obtained for each sample following

$$1 - \xi = \frac{I}{I_0} \frac{C_0}{C},$$
 (S2)

where I and C are the uncorrected urea signal amplitude and the correction factor of a doped sample, and I_0 and C_0 describe the respective parameters for the undoped reference sample. The final corrected and normalized signal amplitudes are shown in Fig. S6.



Figure S6. Corrected and normalized signal amplitude.

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Exponential fitting of the signal quenching

The overall signal quenching we observe in Fig. 7 fits a simple model, which is qualitatively very similar to a treatment introduced by Lange *et al.* [2]. We assume a certain volume around the polarizing agent inside of which all NMR signals are quenched, whereas outside this volume there is no signal reduction. This approximation allows us to semi-quantitatively compare the quenching effects of different polarizing agents. The model is simplified in that quenching is expected to be a continuous process with larger effects closer to the electron spin. Since the paramagnetic interactions are highly non-linear in nature (couplings scale with r^{-3} and relaxation scales with r^{-6}), it is valid to assume a steep increase in signal quenching as the distance between the nuclear and the electron spin is reduced, justifying the validity of the simplicity of the model. The model does not take into account depolarization due to CR and electronic level crossings during sample rotation [3, 4]. Following a simple mathematical model [5], we introduce a void volume *V* of arbitrary shape around a PA inside of which no signal is observed. If we assume a random uncorrelated distribution of polarizing agent, and allow void volumes to overlap, the signal quenching can be described as

$$\xi = 1 - e^{-nV} = 1 - e^{-N_A c_{PA} V}, \tag{S3}$$

where *n* is the number density of polarizing agent molecules, N_A is Avogrado's constant and c_{PA} is the polarizing agent concentration. Due to the binary nature of this model (spins are either outside of any void volume or inside a volume created by one or many voids), it does not account for enhanced effects due to couplings of one nuclear spin to two or more electron spins or effects relayed by another nuclear spin; the orientation dependence of the secular dipole coupling is neglected, too. Also effective concentration changes due to polarizing agent molecules are neglected. This is a valid simplification because the maximum effective volume any polarizing agent occupies in the sample is expected to be less than 5 % even for the samples containing 60 mM of polarizing agent. Signal quenching by all polarizing agents

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except TOTAPOL follow an exponential reduction within margin of error (see Fig. S7). The effective void volumes are 29 and 41 nm³ for trityl and 4-amino-TEMPO, respectively, which correspond to similar quenching radii of 1.9 and 2.1 nm, respectively for a spherical volume. For the S = 7/2 Gd-DOTA, the void volume is 133 nm³, corresponding to 3.2 nm quenching radius. Signal quenching induced by TOTAPOL shows an initially steep concentration dependence at low concentration after which there is a leveling effect at higher concentrations. Even though the exponential fit is of poor quality, we can extract a void volume of 105 nm³ around each TOTAPOL molecule, yielding to a quenching radius of 2.9 nm. This behavior might be caused if TOTAPOL molecules have a propensity to self-associate at higher concentrations, thus increasing overlap of void volume. More likely it is caused by the fact that TOTAPOL is a biradical in which the strong electron-electron dipole couplings mediate efficient CR even at low concentrations. 4-amino-TEMPO at concentrations of 40 mM and higher seem to show a similar effect, where the signal quenching is slightly more pronounced compared to trityl at the same concentration. Although the significance of that difference in quenching factor is too small to certainly determine CR as the clear cause of the effect, it is in agreement with depolarization due to CR and electronic level crossings during MAS.



Figure S7. Exponential fit of the bleaching effect 1–ξ using equation (S3), following the void volume model described in the text. Void volume around each polarizing agent molecule inside which NMR signals are quenched are given, together with the radius of a spherically shaped volume. The signal quenching induced by TOTAPOL cannot be perfectly described with this model; the rather unsatisfactory least-square fit is shown in the bottom graph.

By including the electron spin quantum number of the polarizing agent we show that in the case of all monomeric radicals the void volume around each radical molecule is proportional to the absolute magnetic moment of the electron spin, which is proportional to $\sqrt{S(S+1)}$, as can be seen in Fig. S8. Here, we fitted a single exponential function following

$$\xi = 1 - e^{-N_{\rm A}c_S \sqrt{S(S+1)}V}$$
(S4)

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to the experimental data of all samples plotted against $c_s\sqrt{S(S+1)}$, where c_s is the concentration of electron spins. Except for the two highest concentration samples of 4-amino-TEMPO and all concentrations of TOTAPOL (which were excluded from the fit) there is semi-quantitative agreement between all data points and the fitting function. Therefore we can conclude that signal bleaching can be described by a single parameter for trityl, Gd-DOTA, and for concentrations of <40 mM for 4-amino-TEMPO. This parameter is $32 \text{ nm}^3 \times \sqrt{S(S+1)}$, which corresponds to 28 nm^3 void volume for S = 1/2and 127 nm^3 for S = 7/2. These values closely match the values obtained by separate fits of each polarizing agent of 29 and 133 nm³ for trityl and Gd-DOTA, respectively. The slight discrepancy of the separate-fit value of 4-amino-TEMPO might be caused by an onset of efficient CR as has already been discussed above. As can be clearly seen in Fig. 10, this model does not apply to 4-amino TEMPO at concentrations of 40 mM and above as well as for TOTAPOL in general, most probably because in these cases the efficient electron-nuclear CR allows for a different mechanism of signal quenching [3, 4].



Figure S8. Relative off-signal intensity not affected by paramagnetic quenching $(1-\zeta)$ as a function of the electron spin concentration c_S normalized to the absolute magnetization of each electron spin. The red line indicates an exponential fit according to eq. (S4).

Supplementary Figures

DNP build-up time constants $T_{\rm B}$ and transverse relaxation times T_2



Figure S9. ¹H DNP build-up time constants $T_{\rm B}$ and ¹³C transverse relaxation time constants T_2 as function of electron spin concentration of various polarizing agents.



Comparison between 50 and 100 kHz spin-locking fields on ${}^{1}H T_{1\rho}$

Figure S10. ¹H $P_{1\rho}^{\prime 0}$ at a spin-lock fields of $\omega_{SL}/2\pi = 50$ kHz (top) and 100 kHz (bottom) as function of electron spin concentration of various polarizing agents.



Field positions for EPR measurements of nitroxides

Figure S11. EPR spectrum of TOTAPOL with field positions used for T_{1S} and T_{2S} measurements in the case of TOTAPOL and 4-amino-TEMPO.



Determination of T_{2S}

Figure S12. Determination of electron spin transverse relaxation time constant T_{2S} by extrapolation of excitation bandwidth dependent phase memory time T_m to infinite flip pulse length.



Comparison of polarization build-up for static and MAS DNP

Figure S13. Build-up of polarization under static and MAS conditions for an undoped sample, 10 mM TOTAPOL, and 20 mM trityl recorded at 380 MHz and 80 K. The sample was a 30/30/30/10 mixture (vol.) of 2-¹³C,d₈-glycerol/d₈-glycerol/D₂O/H₂O. The MAS spinning frequency was 4975 Hz.

Supplementary Tables

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|--|---------------------------------------|------------------------|--|--|---|---------------------------------|--|---|--|---|---------------|--------------|--------------|----------------|
| Table S1 140 GHz | • PRE an microwa | d DNP da we irradia | ta for vari tion. | ous polariz | ing agents | at differen | t concentra | tions. Data | recorded a | tt 5 T and ~ | -84 K ur | nder~8 | W of | |
| c_{PA} | $T_{ m B}$ | $^{13}C T_2$ | ¹ H $T_{1\rho}$ (50 kHz) | ¹ H $T_{1\rho}$ (100 kHz) | ¹³ C T _{1p} (50 kHz) | ¹ H 1% | ¹³ C P ₂ | ¹ H <i>P</i> ₁₀ (50 kHz) | ¹ H P % (100 kHz) | ¹³ C P ₁₀ (50 kHz) | G | K ° | 1-5r | E |
| (mM) | (s) | (ms) | (ms) | (ms) | (ms) | (s ⁻¹) | (s ⁻¹) | (s ⁻¹) | (s ⁻¹) | (s ⁻¹) | | | | |
| undoped 0 | 54.59 ^a | 14.69 | - _в | - _в | ۹ ا | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 |
| TOTAPO | | | | | | | | | | | | | | |
| 2^{c} | 17.50 | 13.15 | 103.9 | 194.1 | 492.8 | 0.039 | 8.0 | 9.6 | 5.2 | 2.0 | 96.3 | 1.77 | 0.73 | 124.7 |
| 2° | 9.74 | 11.11 | 61.0 | 113.5 | 325.9 | 0.084 | 22.0 | 16.4 | 8.8 | 3.1 | 119.6 | 2.37 | 0.61 | 174.0 |
| 10° | 4.48 | 7.22 | 25.1 | 50.4 | 153.3 | 0.205 | 70.5 | 39.9 | 19.8 | 6.5 | 139.0 | 3.49 | 0.47 | 225.9 |
| 20° | 1.84 | 3.84 | 7.6 | 17.0 | 63.6 | 0.525 | 192.1 | 131.0 | 58.7 | 15.7 | 138.3 | 5.45 | 0.29 | 217.3 |
| | | | | | | | | | | | | | | |
| 5 | 26.88 ^d | 11.98 | ۱ _° | ۱ [₽] | ۱ [₽] | 0.019 | 15.4 | ۱ _۴ | ٩ | ٩ | 14.9 | 1.43 | 0.96 | 20.4 |
| 10 | 16.37 ^d | 8.72 | 39.59 | 83.53 | 445.7 | 0.043 | 46.7 | 25.3 | 12.0 | 2.24 | 26.8 | 1.83 | 0.87 | 42.8 |
| 20 | 8.66 ^d | 5.09 | 16.67 | 33.48 | 147.5 | 0.097 | 128.3 | 60.0 | 29.9 | 67.8 | 48.7 | 2.51 | 0.68 | 82.6 |
| 60 60 | 2.82" 1.64 ^d | 2.62 1.74 | 4.527 2.093 | 11.21 5.414 | 51.0 27.3 | 0.336 0.592 | 313.0 508.3 | 220.9 477.8 | 89.2 184.7 | 19.6 36.7 | 78.5 106.9 | 4.40 5.77 | 0.35 0.22 | 122.0 133.2 |
| trityl OX0 | 63 | | | | | | | | | | | | | |
| S | 49.09 ^d | 11.74 | 122.2 | 287.9 | 709.6 | 0.002 | 17.1 | 8.2 | 3.5 | 1.4 | 19.1 | 1.05 | 0.93 | 18.7 |
| 10 | 44.52 | 8.07 | 40.3 | 81.1 | 261.7 | 0.004 | 1 22.9 | 24.8 | 12.3 | 0.00 0.00 | 29.7 | 1.11 | 0.93 | 30.6 |
| 40 | 24.02 | 4.97 3.01 | 12.0 3.2 | 29.2 8.2 | 108.4 35.9 | 0.007 | 133.1 264.7 | 78.1 311.8 | 34.2 121.7 | 9.2 27.8 | 62.0 | 1.51 | 0.48 | 42.7 45.1 |
| 60 | 13.20 | 2.20 | 1.4 | 3.5 | 18.0 | 0.057 | 387.5 | 729.4 | 283.0 | 55.7 | 37.7 | 2.03 | 0.37 | 28.4 |
| Gd-DOT A | - | | | | | | | | | | | | | |
| <u> </u> | 39.55 | 13.39 | 43.4 | 67.0 | 272.6 | 0.007 | 6.6 | 23.0 | 14.9 | 3.7 | 5.9 | 1.17 | 1.00 | 6.9 |
| 2 | 29.88 | 11.78 | 25.6 | 40.2 | 185.9 | 0.015 | 16.9 | 39.0 | 24.9 | 5.4 | 8.2 | 1.35 | 0.89 | 9.8 |
| ς γ | 17.47 | 8.90 | 8.4 | 15.8 | 70.5 | 0.039 | 44.3 | 118.6 | 63.2 | 14.2 | 11.0 | 1.77 | 0.79 | 15.2 |
| 10 | 5.60 | 5.42 | 1.5 | 3.2 | 20.7 | 0.160 | 116.5 | 658.7 | 309.6 | 48.2 | 10.5 | 3.12 | 0.43 | 14.1 |
| 20 | 2.55 | 3.54 | 0.6 | 1.4 | 9.2 | 0.374 | 214.4 | 682.1 | 725.0 | 109.1 | 6.3 | 4.63 | 0.19 | 5.4 |
| ^a This value | actually re | presents T_1° | . ^b Data coul | d not be dete | mined becau | se fitting die | d not yield ph | ysically mea | ningful result | s due to limit | ed spin-lo | ck time. | | |
| ^c Polarizing $T_1 = 61.57$ | agent conc s. T _B was n | ormalized t | f the biradica o the same | al; electron co T_1° given in th | ncentration in table by the | s twice the g e relation $1/$ | given value. ^a $T_{\rm B,a} - 1/T_{\rm B,b} =$ | Data was acq $1/T_{1,a}^{o} - 1/T_{1,b}^{o}$. | uired using a | different buf | fer solutic | on with | | |
| 1 – UL.J | s. IB was i | | | и Виснии и | ic lable by m | | $I_{B,a} - I I_{B,b} -$ | $1/1_{l,a} - 1/1_{l,b}$ | | | | | | |

Table S2. T_{1S} of TOTAPOL and 4-amino-TEMPO in 60/30/10 (v/v) d₈-glycerol/D₂O/H₂O (incl. 1 M ¹³C-urea) at various concentrations and magnetic fields positions at 80 K.

| <i>c</i> (mM) | | | T_{1S} (ms) | | |
|---------------|---------|---------|---------------|---------|-------|
| | 4980 mT | 4986 mT | 4994 mT | 4998 mT | mean |
| TOTAPO | Ĺ | | | | |
| 2 | 0.248 | 0.250 | 0.463 | 0.686 | 0.412 |
| 10 | 0.212 | 0.209 | 0.399 | 0.649 | 0.367 |
| 40 | 0.125 | 0.160 | 0.243 | 0.334 | 0.216 |
| 4-amino-T | EMPO | | | | |
| 5 | 0.319 | 0.314 | 0.593 | 0.892 | 0.530 |
| 20 | 0.240 | 0.207 | 0.381 | 0.741 | 0.392 |
| 60 | 0.180 | 0.165 | 0.297 | 0.444 | 0.272 |
| | | | | | |

Table S3. T_{2S} of TOTAPOL and 4-amino-TEMPO in 60/30/10 (v/v) d₈-glycerol/D₂O/H₂O (incl. 1 M ¹³C-urea) at various concentrations and magnetic fields positions at 80 K.

| <i>c</i> (mM) | | | T_{2S} (µs) | | |
|---------------|---------|---------|---------------|---------|------|
| | 4980 mT | 4986 mT | 4994 mT | 4998 mT | mean |
| TOTAPOI | | | | | |
| 2 | 3.13 | 2.73 | 2.82 | 4.39 | 3.27 |
| 10 | 1.58 | 1.33 | 1.80 | 2.04 | 1.68 |
| 40 | 0.38 | 0.35 | 0.40 | 0.42 | 0.39 |
| 4-amino-T | EMPO | | | | |
| 5 | 4.02 | 3.69 | 4.35 | 4.29 | 4.09 |
| 20 | 1.97 | 1.57 | 1.91 | 2.76 | 2.05 |
| 60 | 0.60 | 0.53 | 0.73 | 0.85 | 0.68 |

Table S4. T_{1S} and T_{2S} of trityl OX063 in 60/30/10 (v/v) d₈-glycerol/D₂O/H₂O (incl. 1 M ¹³C-urea) at various concentrations at 80 K.

| <i>c</i> (mM) | <i>T</i> _{1S} (ms) | T_{2S} (µs) |
|---------------|-----------------------------|---------------|
| trityl OX0 | 63 | |
| 5 | 1.88 | 5.52 |
| 20 | 1.50 | 1.82 |
| 60 | 1.47 | 0.38 |

Table S5. T_{1S} and T_{2S} of Gd-DOTA in 60/30/10 (v/v) d₈-glycerol/D₂O/H₂O (incl. 1 M ¹³C-urea) at various concentrations at 80 K.

| <i>c</i> (mM) | <i>T</i> _{1S} (ms) fast comp. | T _{1S} (ms) slow comp. | T _{2S} (μs) |
|---------------|--|---------------------------------|----------------------|
| Gd-DOTA | | | |
| 1 | 2.65 | 6.55 | 0.66 |
| 5 | 1.48 | 7.94 | 0.36 |
| 20 | 4.51 | 4.84 | 0.11 |

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