# High Resolution $^1$H NMR in Organic Solids at Natural Abundance

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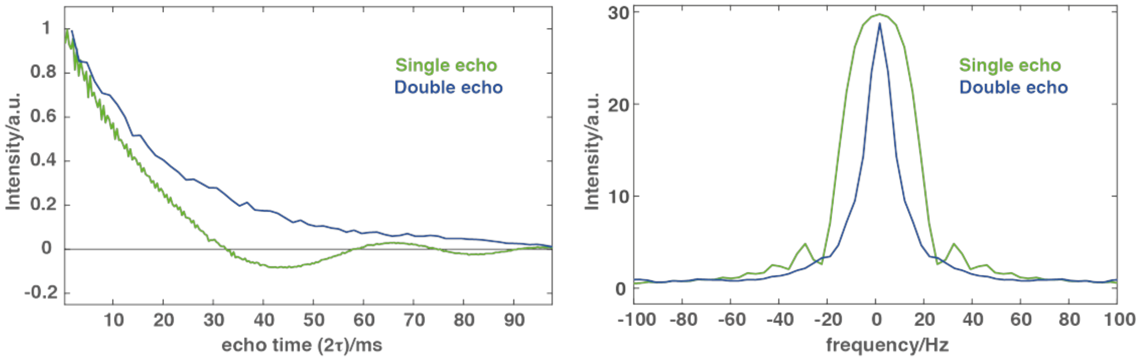
High resolution $^1$H NMR spectra have transformed molecular chemistry over the last 60 years. However, due to the absence of molecular tumbling, in solid samples proton spectra usually have linewidths of more than 50 ppm, obscuring all chemical information. To obtain high resolution $^1$H spectra in solids would provide a key tool for the atomic-level characterization of modern materials, especially since NMR does not require long range order.

$^1$H line broadening is primarily due to strong $^1$H homonuclear dipolar couplings. It is possible to improve the resolution with the application of techniques such as magic angle spinning (MAS)[1] and multi-pulse sequences specifically designed to lead to homonuclear dipolar decoupling. The best results achievable today are obtained using these two techniques simultaneously, in the so-called combined rotation and multi-pulse spectroscopy (CRAMPS) approaches,[2] but linewidths are still on the order of 150 Hz.[3,4] The underlying reasons for this apparent limit on $^1$H resolution are not understood.

Here we discover one of the limiting factors of resolution: an undecoupled residual anisotropic interaction that leads to a residual splitting. The mechanisms of line broadening were evaluated by measuring transverse dephasing times $T'_2$, which are indicators of the presence of non-refocusable interactions[5,6]. We have discovered an unexpected oscillating component, present in the spin-echo dephasing curves for BR-24, eDUMBO-$1_{22}$, LG4 and even WAHUHA. The Fourier transforms of these curves show that the oscillations correspond to an anisotropic splitting of up to 80 Hz. We also show that this residual interaction, which is one of the limiting factors on the lifetime of the decay, can be removed by the application of a double echo. As a result, the dephasing curves have slower decay, characterized by $T'_2$ of up to 22 ms, which corresponds to a refocused linewidth of 14 Hz.



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