Supplementary Material (ESI) for *PCCP*

High-resolution solid-state ^{13}C μMAS NMR with long coherence life times

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Electronic Supplementary Information:

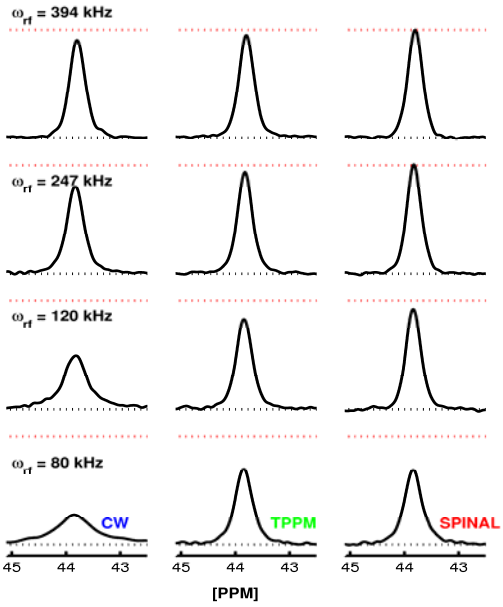

Figure S1: The effect of different heteronuclear decoupling sequences at different rf field strengths on the linewidth and intensity of ^{13}C CPMAS spectrum of 2-^{13}C-^{15}N Glycine powder is shown. The magic angle was set using $NaNO_3$ sample in the macro coil of the regular 2.5mm stator.

Comparison of Single crystal spectrum with powder:

A Narrow line was observed for single crystal than powder under similar experimental conditions. The powder spectrum shows an absorptive Lorentzian shape whereas the single crystal does not. This is because of shimming limitation. We tried to improve the shimming inside the μcoil using an additional Helmholtz shim coil near to the μcoil. The field gradients produced by the Helmholtz shim coil along the magic angle alone were not sufficient enough to shim the probe. Further improvements to the probe design are in progress.

Table S1 **.** Optimized decoupling parameters for TPPM and SPINAL sequences at a spinning speed of 10 kHz, the offset was fixed for all the sequences at 917.3 Hz.

	TPPM		SPINAL	
	Pulse width (μs)	Phase (degrees)	Pulse width (μs)	Phase (degrees)
ω_{rf} = 80 kHz	6.45	7	6.9	5.7
ω_{rf} = 120 kHz	4.25	10	4.25	8
ω_{rf} = 247 kHz	2.14	3.5	2.2	11
ω_{rf} = 394 kHz	1.36	2.5	1.4	11

Experimental details for 2D refocused INADEQUATE:

[98% U-^{13}C, 98% ^{15}N] L-isoleucine sample was purchased from Sigma-Aldrich. The sample was used without further crystallization. For 2D experiment, a total of 256 t_1 acquisitions (STATES procedure) were acquired with 64 scans and a recycle delay of 4 seconds. The spectral width was 25 kHz in both dimensions and the double quantum excitation and conversion time was 5.6 ms. The decoupling parameters were optimized by looking at the echo signal intensity at $2\tau_{echo}$ = 20 ms. A 32 step phase cycle was used for the pulse sequence.