Supporting Information for:

Dynamic Nuclear Polarization of ^{17}O: Direct Polarization

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Table S1: GIPAW calculated ^{17}O CS and EFG parameters for ice.[1]

Site	C_Q (MHz)	η	$\sigma_{cal.}$ (ppm)	δ_{iso} (ppm)	Ω (ppm)	κ
O1	-6.632	0.91	324.57	-69.57	34.42	-0.53
O2	-7.034	0.88	319.25	-66.91	36.76	-0.27
Average	-6.833	0.90	321.91	-68.24	35.59	-0.40

Table S2: GIPAW calculated ^{17}O CS and EFG parameters for urea.[2]

Site	C_Q (MHz)	η	$\sigma_{cal.}$ (ppm)	δ_{iso} (ppm)	Ω (ppm)	κ
O1	7.576	0.96	82.63	172.37	262.98	-0.82

Table S3: GIPAW calculated ^{17}O CS and EFG parameters for phenol.[3]

Site	C_Q (MHz)	η	$\sigma_{cal.}$ (ppm)	δ_{iso} (ppm)	Ω (ppm)	κ
O1	-8.691	0.85	172.84	82.16	78.73	0.44
O2	-8.661	0.83	173.39	81.61	70.73	0.48
O3	-8.706	0.83	173.93	81.07	63.91	0.68
Average	-8.686	0.84	173.39	81.61	71.12	0.53

NB: Calculated chemical shieldings ($\sigma_{cal.}$) were corrected using a shielding reference (σ_{ref}) of 255.0 ppm to determine the calculated chemical shift (δ_{iso}); $\delta_{iso} = -(\sigma_{cal.} - \sigma_{ref})$; $\delta_{iso} = -(\sigma_{cal.} - 255.0)$.[5]

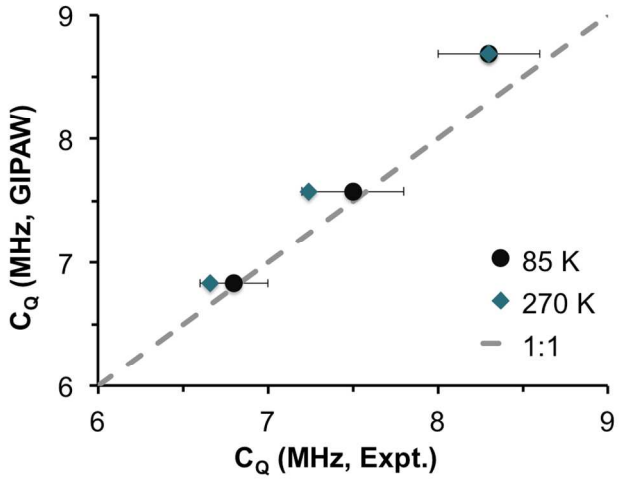

Figure S1: Experimental (DNP (circle) and MAS NMR (diamond)) and GIPAW calculated quadrupolar coupling constants (C_Q, MHz). NB: Experimental MAS NMR data are recorded for the crystalline solid without modification (errors are within the diamond), while the DNP samples were dissolved in an aqueous media (i.e., cryprotected), followed by rapid cooling to an amorphous solid.

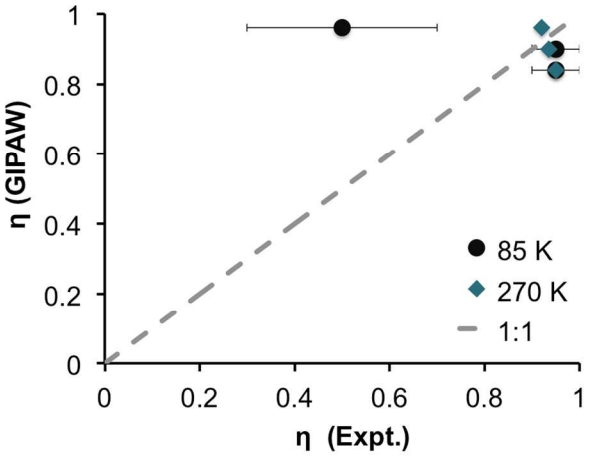

Figure S2: Experimental (DNP (circle) and MAS NMR (diamond)) and GIPAW calculated asymmetry parameter (η). NB: Experimental MAS NMR data are recorded for the crystalline solid without modification (errors are within the diamond), while the DNP samples were dissolved in an aqueous media (i.e., cryoprotected), followed by rapid cooling to an amorphous solid.

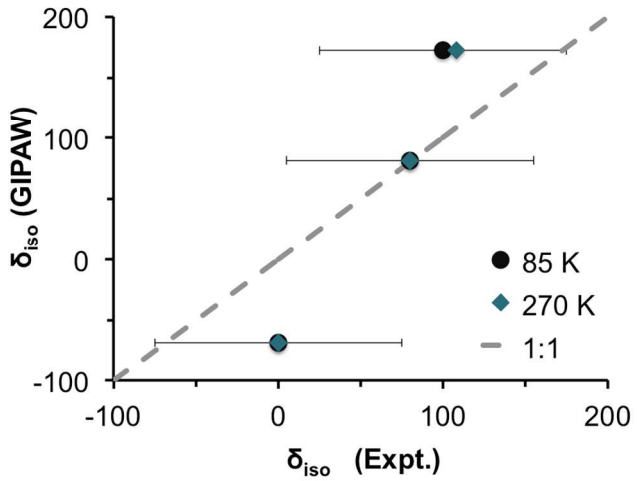

Figure S3: Experimental (DNP (circle) and MAS NMR (diamond)) and GIPAW calculated isotropic chemical shift (δ_{iso}, ppm). NB: Experimental MAS NMR data are recorded for the crystalline solid without modification (errors are within the diamond), while the DNP samples were dissolved in an aqueous media (i.e., cryoprotected), followed by rapid cooling to an amorphous solid.

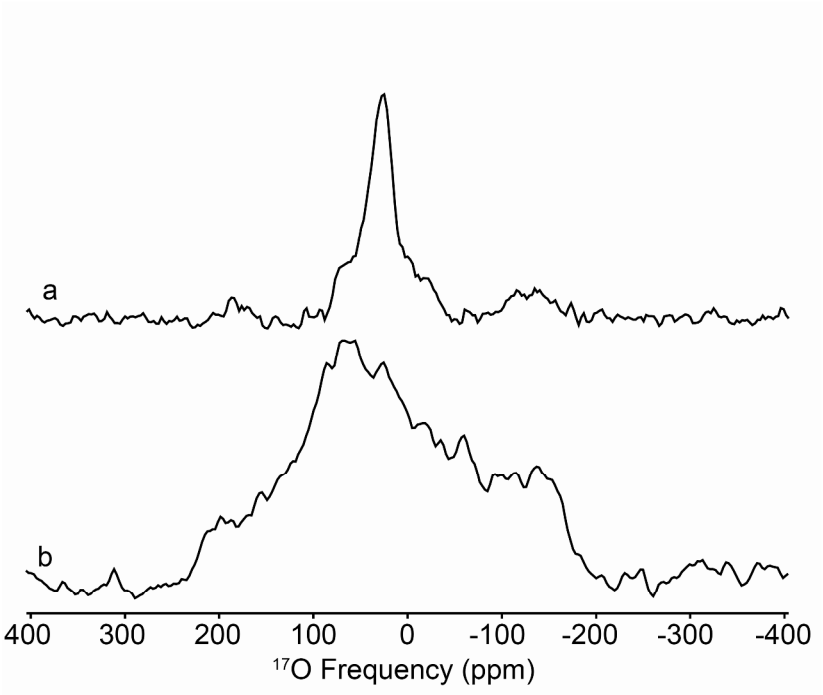

Figure S4: ^{17}O solid-state NMR of ^{17}O-Phenol (35 mgs of sample, 3.2 mm ZrO_2 rotor) acquired at 17.4 T (748 MHz, ^{1}H). a) MAS, ω_r = 16 kHz and b) non-spinning. Data were acquired using a

Hahn-echo with CW ^{1}H decoupling ($\gamma\beta_1/2\pi$ of 120 kHz (^{17}O, solid) and 83 kHz (^{1}H)), with 4,096 and 16,384 co-added transients and recycle delays of 10 seconds.

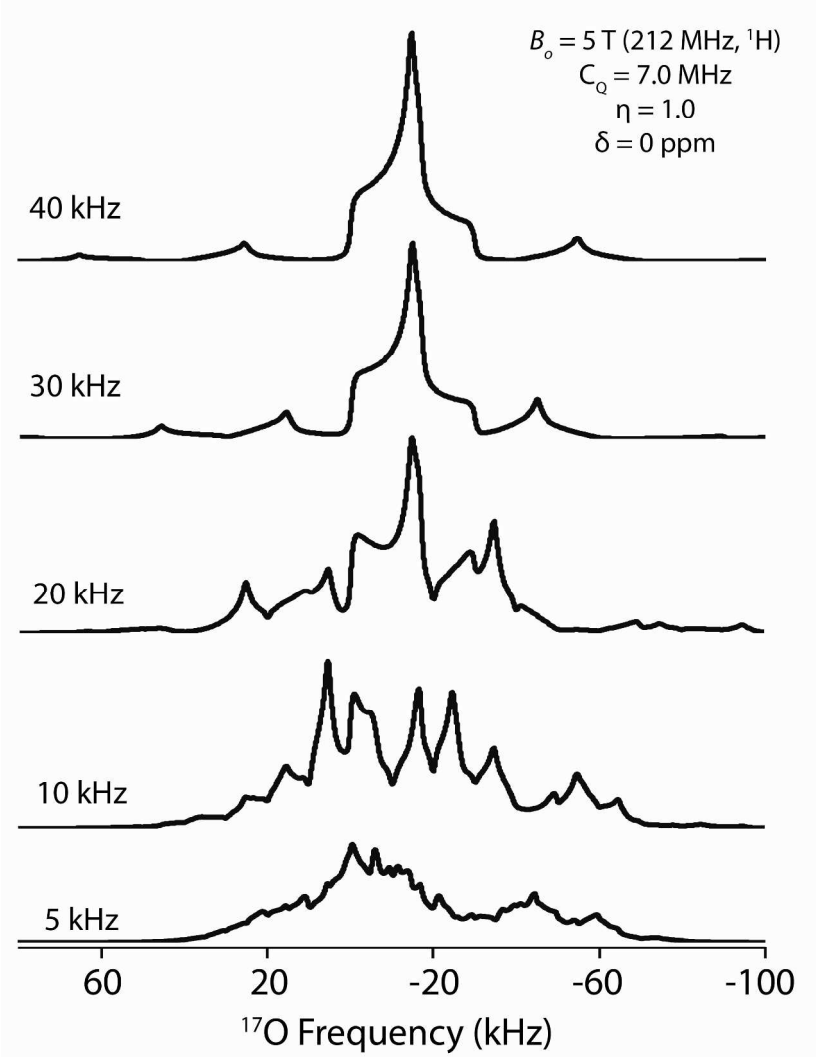

Figure S5: ^{17}O NMR simulations of magic-angle spinning spectra at 5 T (212 MHz, ^{1}H) with a spinning frequency ranging between 5 and 40 kHz. Parameters for simulations (set within SPINEVOLUTION[6]) were: C_Q=7.0 MHz, η=1.0 and δ_{iso} = 0.0 ppm with a magnetic field strength of 212 MHz (^{1}H nuclear Larmor frequency).

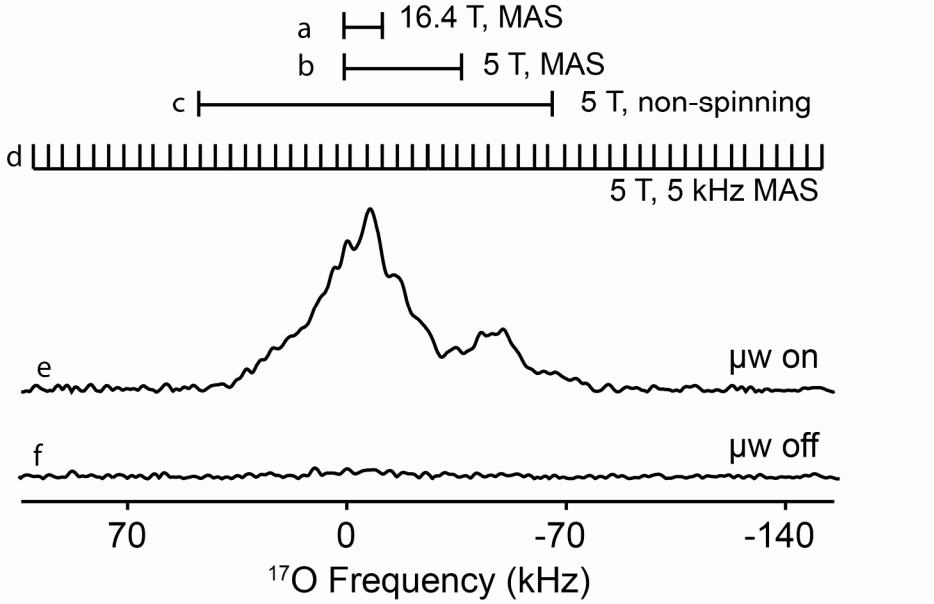

Figure S5: ^{17}O DNP MAS NMR of a 60/30/10 (v/v) d_8-glycerol/D_2O/$H_2{}^{17}O$ glass (10 microliter volume, 2 microliters of $H_2{}^{17}O$-35%) using 40 mM trityl acquired under MAS (ω_r = 5 kHz) at 5 T. a) breadth of central transition at 16.4 T (700 MHz, ^1H) under MAS, b) breadth of central transition at 5 T (212 MHz, ^1H) under MAS, c) breadth of central transition at 5 T (212 MHz, ^1H) non-spinning, d) 5 kHz spacing at 5 T representing the spinning sideband manifold spacing, e) microwave on (256 scans, ~30 minutes), ^{17}O DNP spectrum using direct polarization ($\varepsilon > 130$), T_B = 5.3 s and f) microwave off spectrum (16,000 scans, ~ 32 hours).

References for Supporting Information-

1- Bernal, J.D. and Fowler, R.H. (1933), Journal of Chemical Physics, 1, 515.

2- Sklar, N., Senko, M.E. and Post, B. (1961), Acta Crystallographica, 14, 716.

3- Scheringer, C. (1963), Zeitschrift für Kristallographie, 119, 273.

4- Herzfeld, J. and Berger, A.E. (1980), Journal of Chemical Physics, 73, 6021.

5- Yates, J.R., Pickard, C.J., Payne, M.C., Dupree, R., Profeta, M. and Mauri, F. (2004), Journal of Physical Chemistry A, 108, 6032.

6- Veshtort, M. and Griffin, R.G. (2006), Journal of Magnetic Resonance, 178, 248.