nature portfolio

https://doi.org/10.1038/s41596-022-00693-8

Supplementary information

Hyperpolarized water as universal sensitivity booster in biomolecular NMR

In the format provided by the authors and unedited

Hyperpolarized water as universal sensitivity booster in biomolecular NMR

Christian Hilty[1], Dennis Kurzbach[2], Lucio Frydman[3]

[1] Chemistry Department, Texas A&M University, 3255 TAMU, College Station, TX 77843, USA.
[2] Faculty of Chemistry, Institute for Biological Chemistry, University of Vienna, Währinger Straße 38, 1090 Vienna, Austria.
[3] Department of Chemical and Biological Physics, Weizmann Institute of Science, 76100 Rehovot, Israel.

Supplementary Material

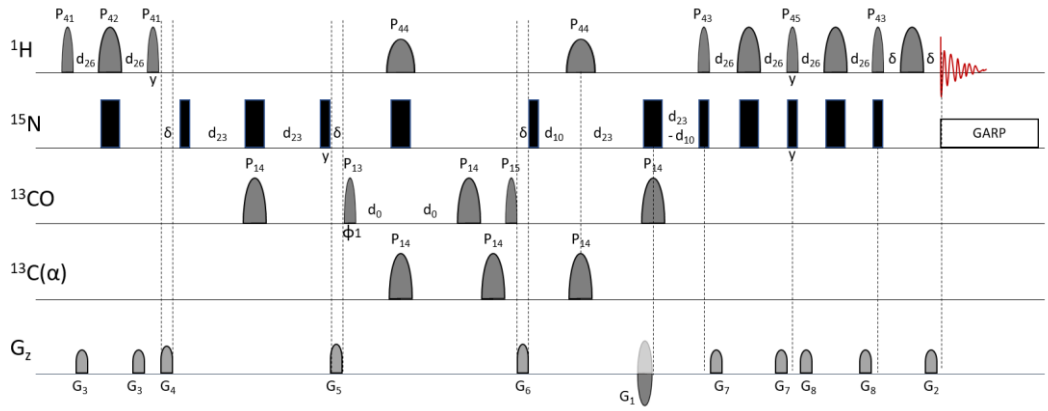

Figure S1. BEST-HNCO pulse sequence.[1] Shaped and hard pulses are shown as oval or square shapes, respectively. The thin shapes indicate 90° pulses and the broad shapes indicate 180° pulses. Delays were D23 = 14.5 ms, D26 = 2.4 ms, and δ = 1100 µs. ^{1}H selective pulses were PC9 (P41, 2251 µs) 180° pulses Reburp (P42, 1498 µs), Eburp2 and Eburp2tr (P43 and P45, each 1439 µs), and BIP720,50,20 (P44, 150 µs). ^{13}C selective pulses were G4 and G4tr (P13 and P15, each 308 µs), and Q3 (P14, 210 µs). Non-selective ^{15}N 90° pulse durations were 39.75 µs. GARP decoupling of ^{15}N was applied during acquisition. The phase cycle was Φ_1 = [x, -x], and Φ_{rec} = [x, -x]. All other pulses were applied along the x axis (Φ = 0), except when indicated otherwise. Gradients were G_1 (42.8 G/cm, 1 ms), G_2 (4.3 G/cm, 1 ms), G_3 (3.75 G/cm, 300 µs), G_4 (-21.4 G/cm, 1 ms), G_5 (-26.75 G/cm, 1 ms), G_6 (32.1 G/cm, 1 ms), G_7 (-2.68 G/cm, 500 µs), and G_8 (2.68 G/cm, 300 µs), all generated using the Bruker shape library file 'SMSQ10'.

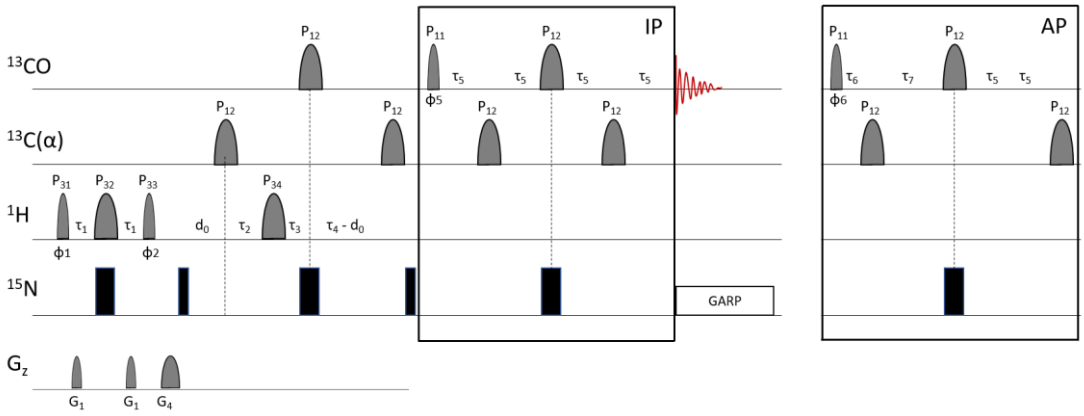

Figure S2. BEST-^{1}H^N-CON pulse sequence[2]. Delays were τ_1 = 137 µs, τ_2 = 1893 µs, τ_3 = 12994 µs, τ_4 = 16501 µs, τ_5 = 8201 µs, τ_6 = 4500 µs, and τ_7 = 11901 µs. Selective ^{1}H 90° and 180° pulses were PC9 (2001.4 µs) and Reburp (1600.2 µs), respectively. Selective ^{13}C 90° and 180° pulses were Q5_sebop (300.2 µs) and Q3_surbop (198.8 µs), respectively. Non-selective ^{15}N 90° pulse durations were 31.5 µs (ubiquitin) or 32.9 µs (OPN). GARP decoupling was applied to ^{15}N during acquisition. The phase cycle was Φ_1 = [x, -x], Φ_2 = [y], Φ_5 = [x], Φ_6 = [-y], and Φ_{rec} = [x, -x]. All other pulses were applied along the x axis (Φ = 0), IPAP was used to suppress CO-Cα splittings during acquisition. Gradients were G_1 (4.6 G/cm, 500 µs) and G_4 (32.85 G/cm, 1 ms), both generated using the Bruker shape library file 'SINE.100'.

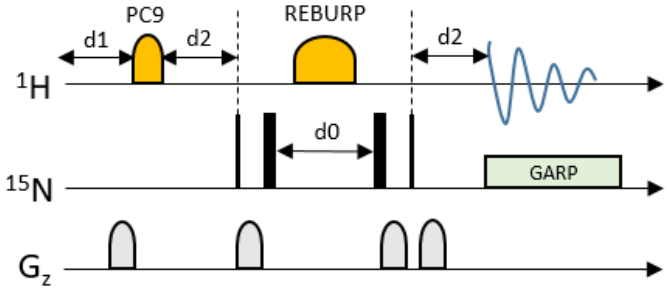

Figure S3. Pulse sequence used for detecting the ^{15}N-edited ^{1}H-1D time-series of Ubq. The rectangular shapes indicated 90˚ pulses. For the ^{1}H channel a selective 1000 μs long PC90 pulse was used for 90° excitation and a 2000 μs long REBURP pulse was used for inversion. The carrier frequency was set to 10 ppm to avoid pulsing on the water resonance. Hence, only signals with chemical shifts >8 ppm were detected. d2 was set to 0.00345 s, d1 to 0.5 s and d0 to 0.00002780 s. d0 was not incremented. The FID was detected for 0.1 s during GARP decoupling.

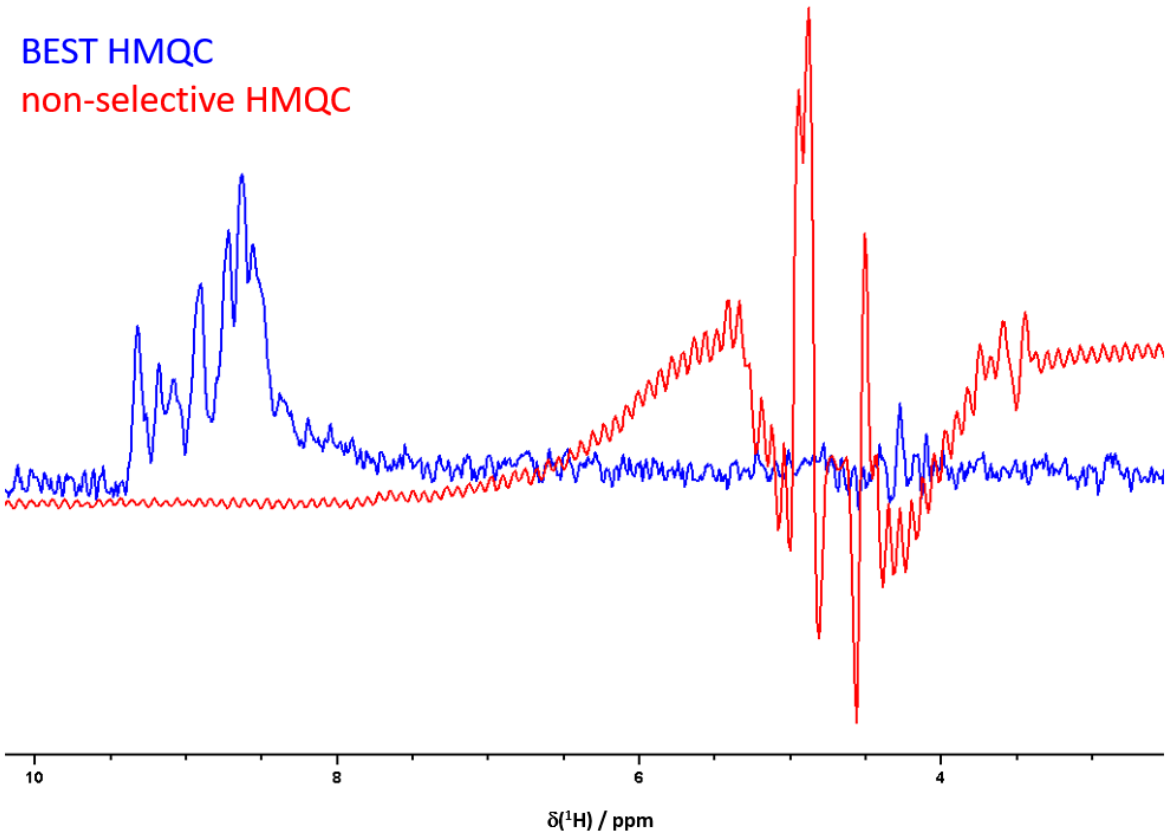

Figure S4. Comparison of HyperW spectra of Ubiquitin. The blue spectrum represents the first transient of a spectrum recorded with a well calibrated selective (BEST) HMQC. The water line is not excited. In contrast, the red spectrum results from a miscalibrated spectrum, in which the water line is excited. The strong water polarization leads to radiation damping prohibitively distorting of the spectrum.

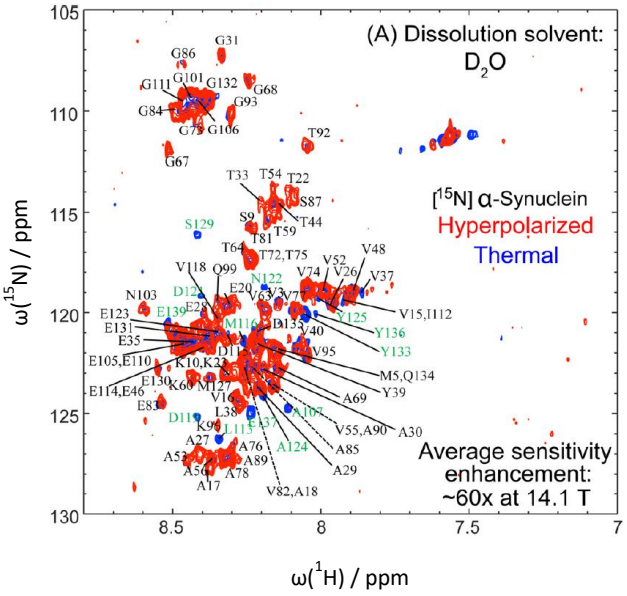

Figure S5. Superposition of the spectra of Figure 7c in the main text.