Supporting Information

Estimating Side-Chain Order in [U-^{2}H; $^{13}CH_3$]-labeled High Molecular Weight Proteins from Analysis of HMQC/HSQC Spectra

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Figure S1

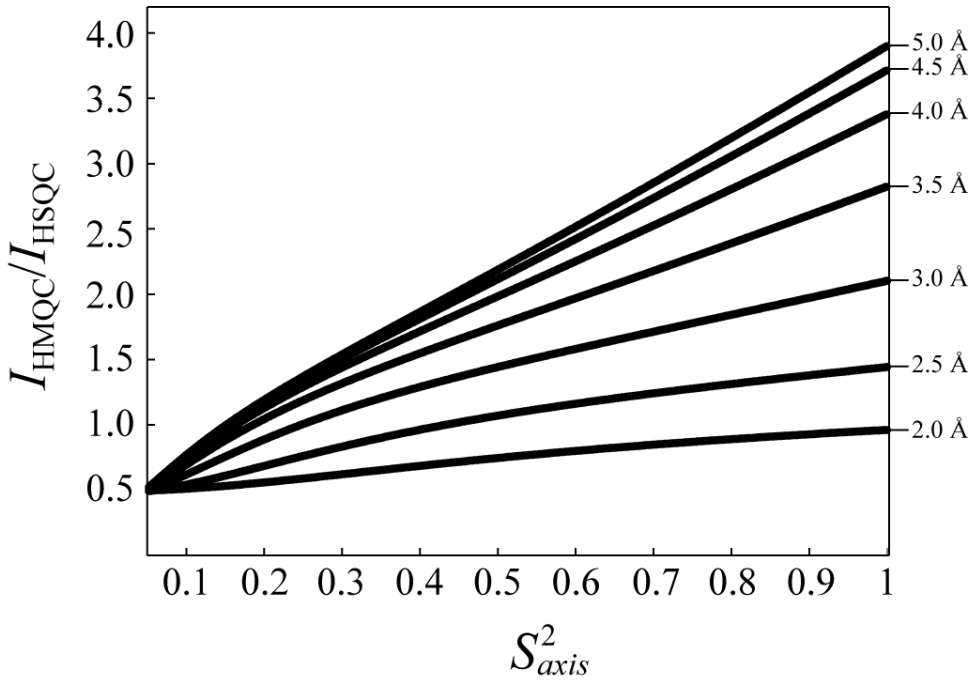

Figure S1. Plots of I_{HMQC}/I_{HSQC} ratios (y-axis) calculated as a function of S^2_{axis} (x-axis) for several values of r_{HHext} (Å), with τ_C fixed at 100 ns. The values of r_{HHext} corresponding to each of the curves are indicated on the right. Calculations have been performed using the same set of parameters as in Figure 1 of the main text.

Figure S2

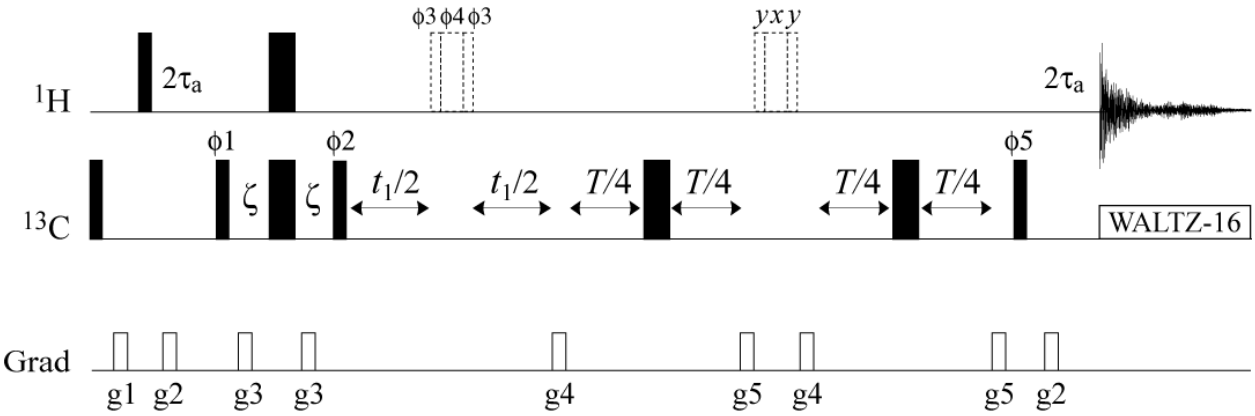

Figure S2. Pulse scheme for the measurement of R_{MQ}^{S} relaxation rates in $^{13}CH_3$-methyl labeled highly deuterated proteins. All narrow (wide) pulses are applied with flip angles of 90°(180°) along the *x*-axis unless indicated otherwise. The ^{1}H and ^{13}C carriers are positioned in the center of the methyl region: 0.7 ppm and ~20 ppm, respectively. All ^{1}H and ^{13}C pulses are applied with the highest available power, while ^{13}C WALTZ-16 decoupling[1] is achieved using a 2.5-kHz field. The pulses shown with dashed lines are implemented as composite ^{1}H pulses[2], with the phases indicated on top. Delays are: τ_a = 2.0 ms; $\zeta = (8^1J_{CH})^{-1}$ = 1.0 ms; *T* is a variable relaxation delay. The phase cycle is: ϕ1 = *x*,-*x*; ϕ2 = *y*,*y*,-*y*,-*y*; ϕ3 = 2(*y*),2(-*x*),2(-*y*),2(*x*); ϕ4 = 2(*x*),2(*y*),2(-*x*),2(-*y*); ϕ5 = *x*; rec. = *x*,-*x*,-*x*,*x*. Quadrature detection in t_1 is achieved by the STATES-TPPI[3] incrementation of ϕ5 and the phase of the receiver. The durations and strengths of the *z*-gradients in units of (ms; G/cm) are: g1 = (1; 25), g2 = (0.5; 15), g3 = (0.3; 10), g4 = (0.5; 12), g5 = (0.4; 15).

Supporting References

(1) Shaka, A. J.; Keeler, J.; Frenkiel, T.; Freeman, R. An Improved Sequence for Broadband Decoupling: Waltz-16. *J. Magn. Reson.* **1983**, *52*, 335-8.

(2) Levitt, M.; Freeman, R. NMR Population Inversion Using a Composite Pulse. *J. Magn. Reson.* **1978**, *33*, 473-6.

(3) Marion, D.; Ikura, M.; Tschudin, R.; Bax, A. Rapid Recording of 2D NMR Spectra without Phase Cycling. Application to the Study of Hydrogen Exchange in Proteins. *J. Magn. Reson.* **1989**, *85*, 393-9.