^{13}C-^{13}C NOESY: an Attractive Alternative for Studying Large Macromolecules

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Supporting Information

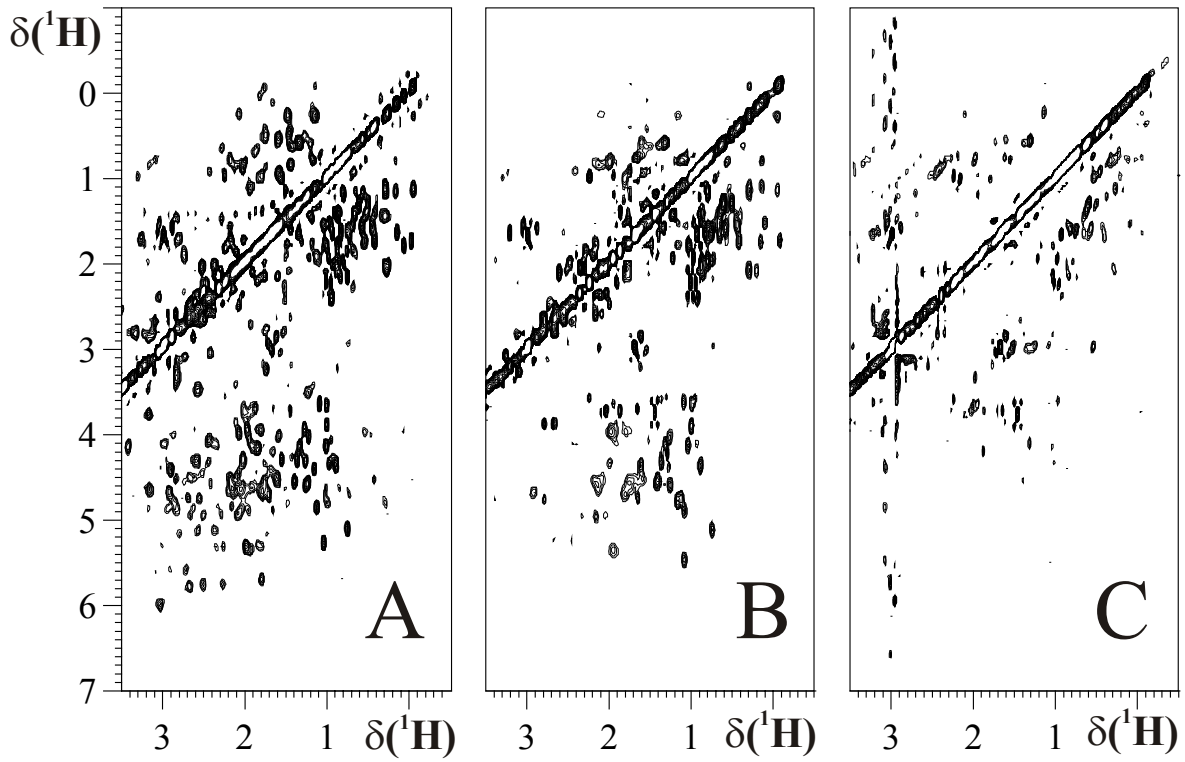

Figure S1. The figure reports spectra acquired with the experiment proposed by Zwuiderweg and coworkers (Fisher, M.W.F.; Zeng, L.; Zuiderweg, E.R.P. *J. Am. Chem. Soc.* **1996**, *118*, 12457-12458.) on three different samples: A) ^{13}C,^{15}N labeled monomeric SOD, B) ^{13}C,^{15}N labeled dimeric SOD, C) ^{13}C,^{15}N, 70% ^{2}H labeled dimeric SOD. The experiments were acquired in the 2D (^{1}H-^{1}H) version. The region containing Hβ-Hα correlations, as well as correlations between aliphatic protons is shown. Experiments were all acquired with the same parameters at 500 MHz, using cryoprobes oprimized for ^{1}H sensitivity, the only difference being inclusion of ^{2}H decoupling in case of the experiment shown in panel C. In this case a TCI ^{1}H cryoprobe was used.

Parameters were: spectral widths of 14 ppm in the two dimensions, 1024 x 256 datapoints were acquired in the two dimensions, 128 scans, the mixing time was 320 ms (with carbonyl pulses every 8 ms to suppress spin-diffusion) and the recycle delay was 1.0 s. All the other parameters were the same as those reported by Zwuiderweg and coworkers except that couplings with carbonyls were removed by $^{13}C'$ selective pulses and that the Watergate scheme was included to achieve water suppression, since our samples were in H_2O.

The behavior of the experiment with increasing molecular mass is evident. The experiment acquired on the monomeric form of SOD clearly shows the expected correlations. When the experiment is acquired on the dimeric form of SOD, only a fraction of the expected correlations can be detected, mainly those involving methyl groups. Finally, when the experiment is acquired on the dimeric form of SOD, fractionally ^{2}H labeled (70%), the number of cross peaks is very small due to low abundance of ^{1}H spins.