A three-dimensional NMR experiment with improved sensitivity for carbonyl-carbonyl J correlation in proteins

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Recently, a quantitative J correlation technique has been presented that permits measurement of (3)J(C'C') in proteins isotopically enriched with C-13 [HU, J.-S. and Bar, A. (1996) J. Am. Chem. Soc, 118, 8170-8171]. Here, we describe an analogous experiment that is less sensitive to transverse C-13' relaxation, which is the principal limiting factor in all C-13-C-13 long-range correlation experiments on macromolecules. The new scheme utilizes homonuclear Hartmann-Hahn cross polarization (TOCSY) instead of a COSY-type transfer to accomplish magnetization transfer; a description of the relevant relaxation terms is presented. The experiment is demonstrated for ubiquitin and HIV-I Nef The results show excellent agreement between (3)J(C'C') values measured for ubiquitin with the new scheme and those reported previously. The experiment is particularly useful for distinguishing backbone phi angles that are smaller than -120 degrees from those larger than -120 degrees.
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