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Four Pulse Recoupling

Navin Khaneja ^{*†} Ashutosh Kumar [‡]

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Abstract

The paper describes a family of novel recoupling pulse sequences in magic angle spinning (MAS) solid state NMR, called four pulse recoupling. These pulse sequences can be employed for both homonuclear and heteronuclear recoupling experiments and are robust to dispersion in chemical shifts and rf-inhomogeneity. The homonuclear pulse sequence consists of a building block $(\frac{\pi}{2})_{0^\circ}(\frac{3\pi}{2})_{\phi^\circ}(\frac{\pi}{2})_{180^\circ+\phi^\circ}(\frac{3\pi}{2})_{180^\circ}$ where $\phi = \frac{\pi}{n}$ ($\phi^\circ = \frac{180^\circ}{n}$), and n is number of blocks in a two rotor period. The heteronuclear recoupling pulse sequence consists of a building block $(\frac{\pi}{2})_{0^\circ}(\frac{3\pi}{2})_{\phi_1^\circ}(\frac{\pi}{2})_{180^\circ+\phi_1^\circ}(\frac{3\pi}{2})_{180^\circ}$ and $(\frac{\pi}{2})_{0^\circ}(\frac{3\pi}{2})_{\phi_2^\circ}(\frac{\pi}{2})_{180^\circ+\phi_2^\circ}(\frac{3\pi}{2})_{180^\circ}$ on channel I and S , where $\phi_1 = \frac{3\pi}{2n}$, $\phi_2 = \frac{\pi}{2n}$ and n is number of blocks in a two rotor period. The recoupling pulse sequences mix the y magnetization. We show that four pulse recoupling is more broadband compared to three pulse recoupling [1]. Experimental quantification of this method is shown for $^{13}\text{C}_\alpha\text{-}^{13}\text{CO}$, homonuclear recoupling in a sample of Glycine and $^{15}\text{N}\text{-}^{13}\text{C}_\alpha$, heteronuclear recoupling in Alanine. Application of this method is demonstrated on a sample of tripeptide N-formyl-[U- ^{13}C , ^{15}N]-Met-Leu-Phe-OH (MLF).

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