Recycling up to 10x faster with 3x SNR: Flip-back CP in MAS DNP

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DNP methods can provide significant sensitivity enhancements in magic angle spinning solid-state NMR, but in systems with long polarization build up times long recycling periods are required to optimize sensitivity.

We show how the sensitivity of such experiments can be improved by the classic flip-back method to recover bulk proton magnetization following continuous wave proton heteronuclear decoupling. Experiments were performed on formulations with characteristic build up times spanning two orders of magnitude: a BDPA radical doped *o*-terphenyl glass and microcrystalline samples of theophylline, L-histidine monohydrochloride monohydrate, and salicylic acid impregnated by incipient wetness. For these systems, addition of flip-back is simple, improves the sensitivity beyond that provided by modern heteronuclear decoupling methods such as SPINAL64, and provides optimal sensitivity at shorter recycle delays.

The shortening of the recycle delay is predicted by numerical simulations using a diffusion model of polarization transport. **The model predicts that 90% of the signal is recovered after the CP spin-lock, which explains such dramatic reduction in the acquisition time.**

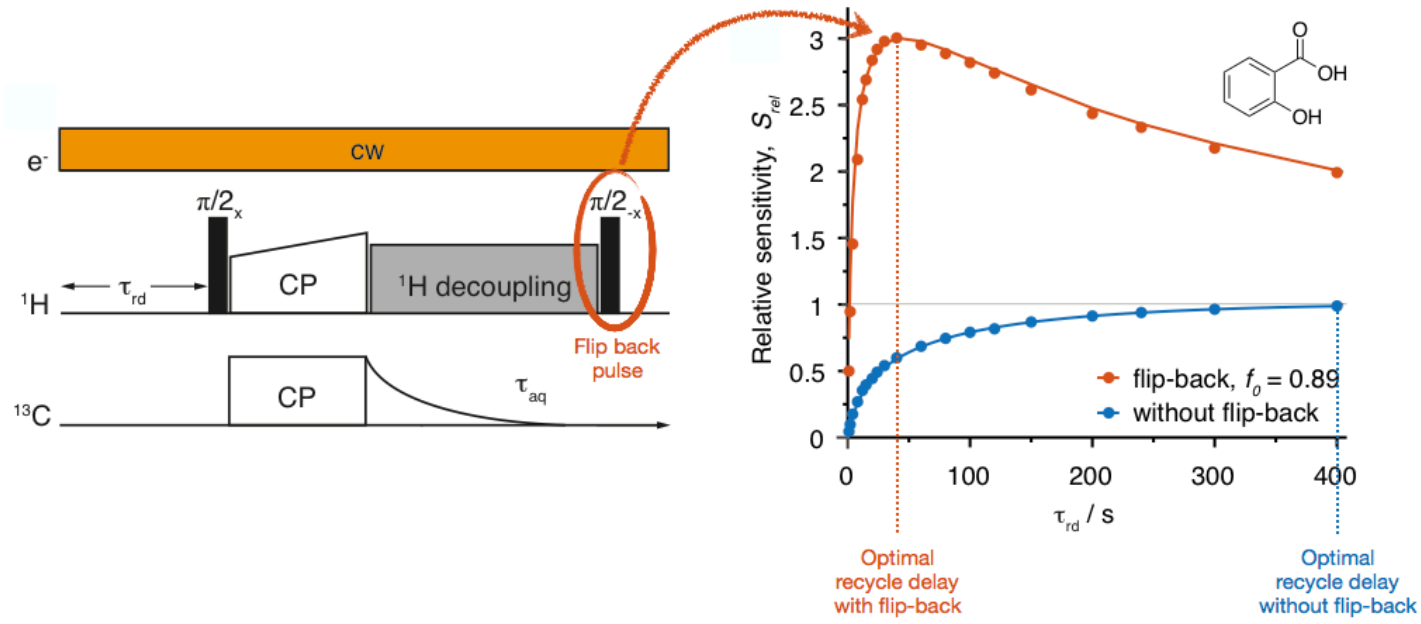

Figure : (Left) Flip-back Cross Polarization sequence. **(Right)** Measured sensitivity gains (filled circles) using DNP enhanced flip-back CP MAS (red) on salicylic acid. Data for decoupled CP MAS without flip-back recovery (blue) was acquired for each sample as a sensitivity benchmark. The solid lines are predictions from numerical simulations for in the microcrystals according to a diffusion model of polarization transport. The optimal recycle delay is 10x samller with flip-back, with a sensitivity enhancement of 3 in this system.