

A new field of solid-state NMR by very fast MAS: ^1H DQMAS NMR for volume limited samples

^1H DQMAS NMR at a very fast MAS frequency of 80 kHz is presented. ^1H DQMAS spectra give information on proximity between protons. ^1H - ^1H homonuclear dipolar decoupling has been applied to ^1H DQMAS to enhance resolution, i.e. ^1H DQ-CRAMPS (CPL 394 (2004) 423, JACS 126 (2004) 13230, JEOL application note: NM090008E). Here we show that highly resolved ^1H DQMAS spectra can be obtained at a very fast MAS rate of 80 kHz without ^1H - ^1H decoupling. This can be done by 1 mm MAS probe which we recently developed. ^1H DQMAS by 1 mm MAS probe realizes high sensitivity collection of volume limited samples of 0.8 μL due to ^1H observation without rf irradiation and high sensitivity per unit volume.

DQ excitation and reconversion are done by post C7 under moderate MAS rate. Here we applied high efficiency and recoupling scheme $\text{R}12_2^5$, which is developed by Wang et al. This realizes efficient observation with low rf field strength requirements. The sequence is shown below.



POST C7 /
 $\text{R}12_2^5$

DQ evolution

POST C7 /
 $\text{R}12_2^5$

SQ acq.

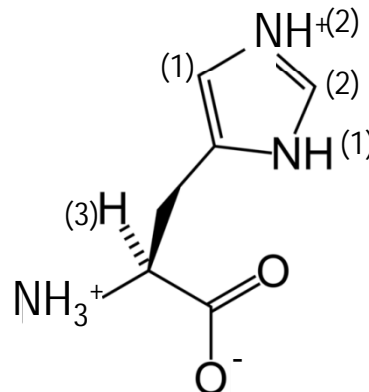
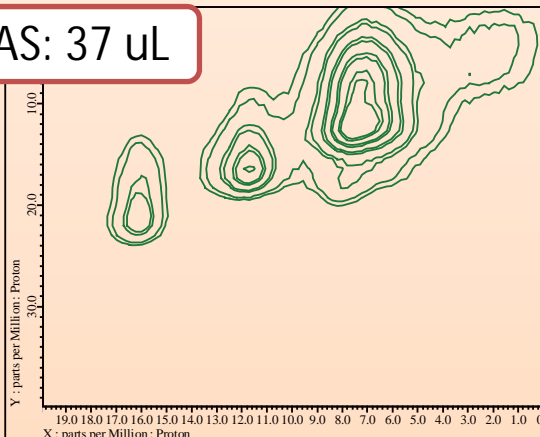
$\text{R}12_2^5$: Q. Wang, B. Hu, O. Lafon, J. Trebosc, F. Deng, J.-P. Amoureux, J. Magn. Reson. 203 (2010) 113-128.



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^1H DQ-MAS NMR of L-histidine•HCl

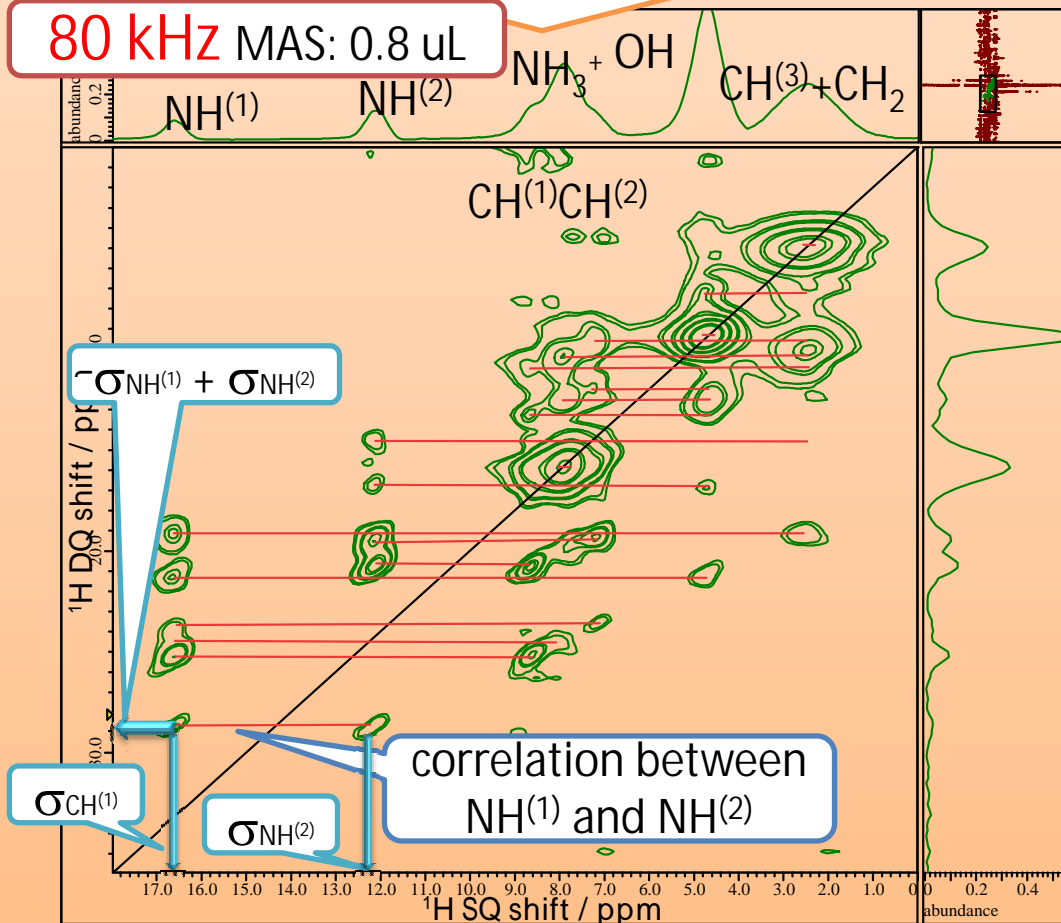
15 kHz MAS: 37 μL



4.0mm CPMAS probe@ECA500, experimental time: 0.6 hours

sensitivity enhancement by very fast MAS!

80 kHz MAS: 0.8 μL



1.0mm CPMAS probe@ECA600, experimental time: 10 hours