Title: Bruker NMR Manual for MSMAS Experiment

Introduction

MQMAS stands for Multi Quantum Magic Angle Spinning which is a 2D solid state NMR Experiment for quadruple hetero-nuclei, such as B11 (spin 3/2), Al27 (spin 5/2), Rb87 (spin 3/2), etc. Due to the quadrupole effect, the 1D spectrum peaks are usually very broad. MQMAS 2D spectrum helps to separate the overlapping peaks in the second dimension.

Preparation

Spectrometer: NMR-Hg400-Solid only

Probe: Bruker 4mm HX probe

Prerequisite: users have done the basic NMR training and solid NMR training.

Sample: must be in powder form; and about 80 mg

Rotor: Bruker https://bruker-labscape.store/products/4mm-mas-rotor-kit Part# H14355

Experiment Setup (quick procedures)

1) Use Rb87 as an example. Run a regular 1D MAS of Rb87 at a certain spin rate, e.g. 5000
2) Edit a new data set with a pulse program "mp3qzqf"
3) Keep 1d mode first and run a quick Rb87 with 12 scans
4) Optimize p1, p2, p3 back and forth
5) Edit another new data set and convert it to 2D
6) Check all parameters (see detailed procedures below)
7) Acquire data
8) Process data by **xfb** or **xfshear**

Ending Work

1) Stop spinning
2) eject rotor out of probe
3) Remove sample out of rotor by special tools and clean up the rotor
4) Keep desk top clean
5) Logout from NUcore

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Experimental Section

Solid state NMR data were collected at room temperature on a Bruker Avance III HD spectrometer equipped with a 4mm HX probe. Spinning rate 5000-15000 (4mm) or 5000-40000 (1.6mm). The other parameters can be seen from topspin Tab AcqPar.

Acknowledgement

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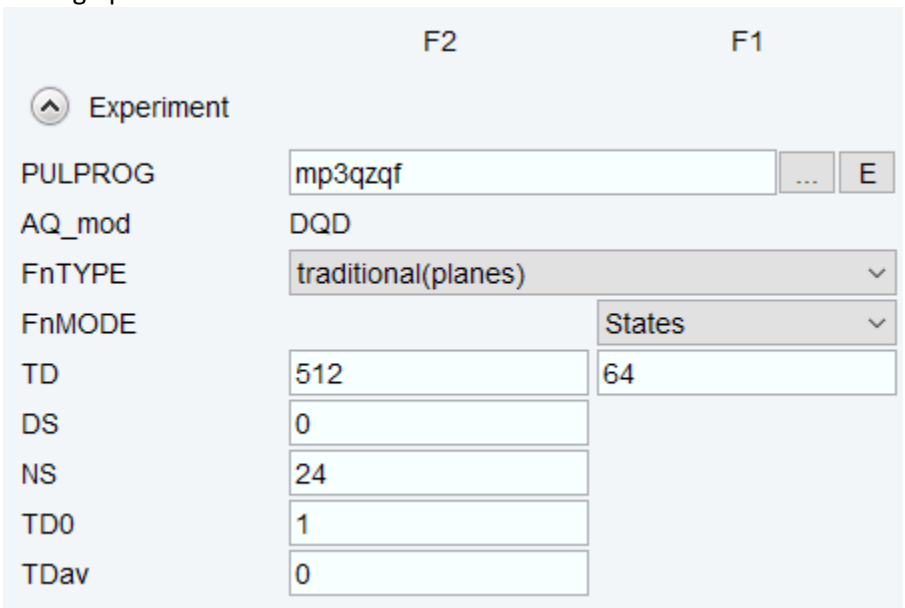
Experiment Setup (detailed procedures)

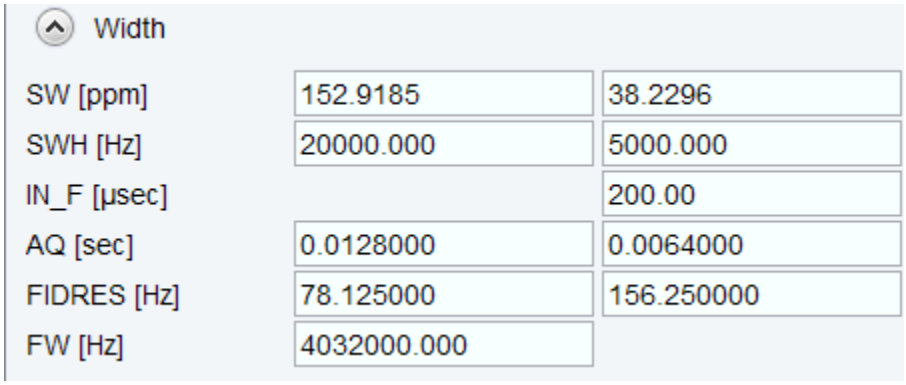
1) Run a regular 1D first. You can use pulse program “onepulse” without proton decoupling, or use “hpdec” with proton decoupling. Note there’s a tuning concern: you can tune both X and H, or just X, because when you use mqmas pulse program “mp3qzqf”, H1 channel is off for both 1D and 2D.
2) After regular 1D is done, edit a new data set for mqmas preparation. In the PULPROG input frame type “mp3qzqf” or click the icon  to select.
3) Using ns=12*n to run 1D. Zoom in the peak region, then type **dpl**.
4) Optimize parameters, p1, p2, p3. Type **popt**. It will pop up a new window (shown below). You will go back and forth several times, until the 3 parameters are optimized. Note: for the first round of optimization, you can use relatively large value range and step size, then, reduce the range and step size.

OPTI...	GROUP	PARA...	OPTI...	STAR...	ENDVAL	NEXP	VARM...	INC
No opti...	0	p1	POSMAX	4	8.0	9	LIN	0.5
No opti...	0	p2	POSMAX	0.5	4	8	LIN	0.5
No opti...	0	p3	POSMAX	40	80.0	11	LIN	4
No opti...	0	p3	POSMAX	30	54.0	13	LIN	2
No opti...	0	p2	POSMAX	2	5.0	7	LIN	0.5
No opti...	0	p1	POSMAX	6	10.5	10	LIN	0.5
No opti...	0	p1	POSMAX	8	20.0	61	LIN	0.2
Step by...	0	p2	POSMAX	2	5.0	16	LIN	0.2

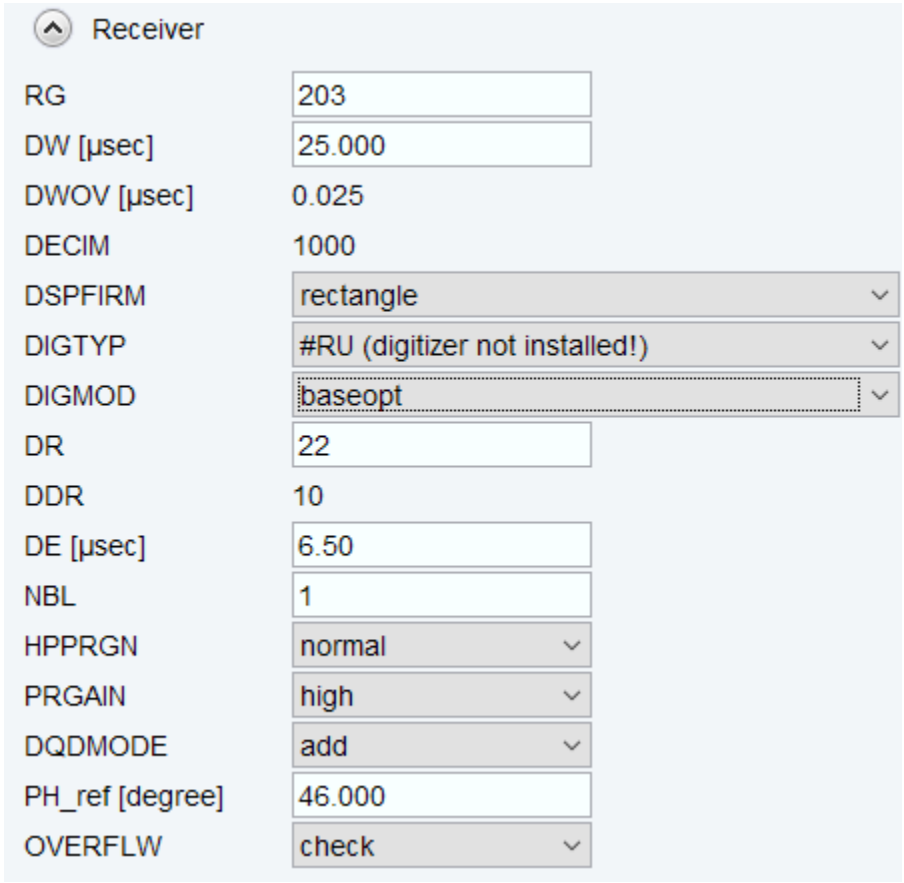
5) Edit another new data set for 2D. Underneath the Tab ACQUPAR, click an icon 
6) Setting up conditions can be referred as below:



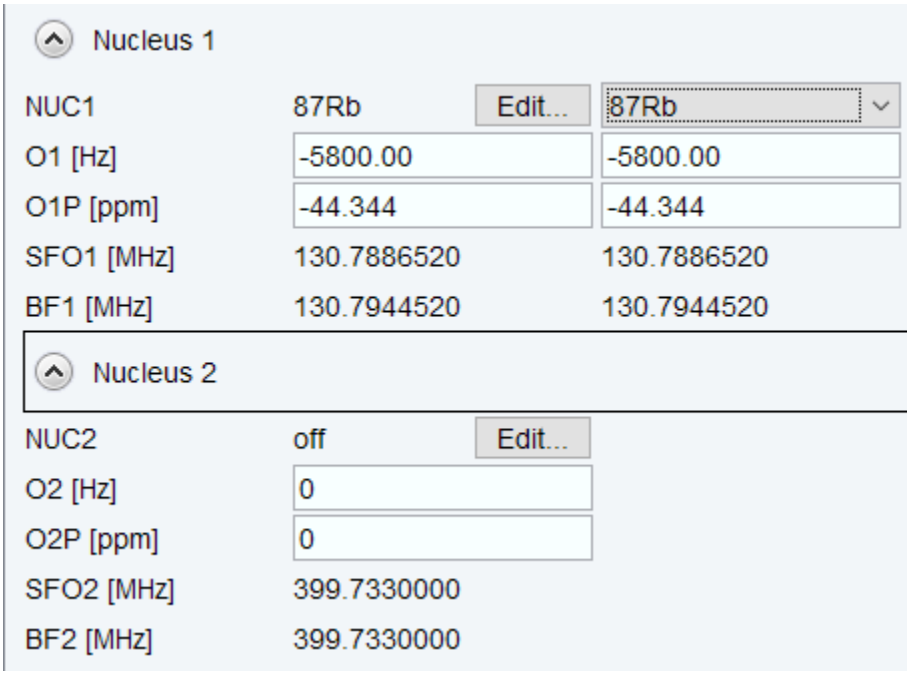
Note: NS must be 12*n. you can increase TD F1 to 128.



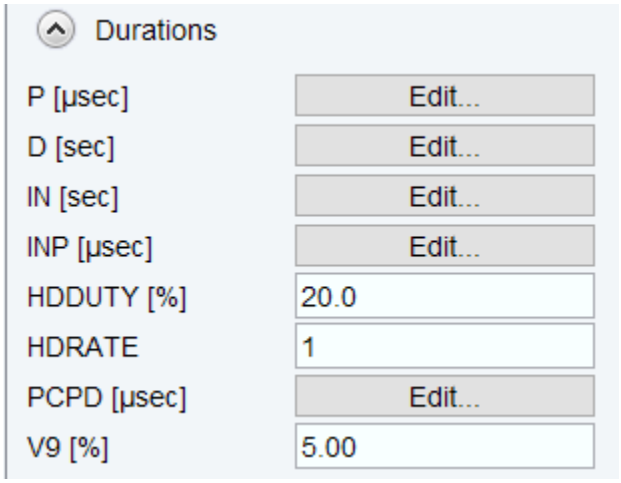
Note: SWH for F1 must be the same as spinning rate for the frequency synchronization. For instance, if you spin 5000, input 5000 here.



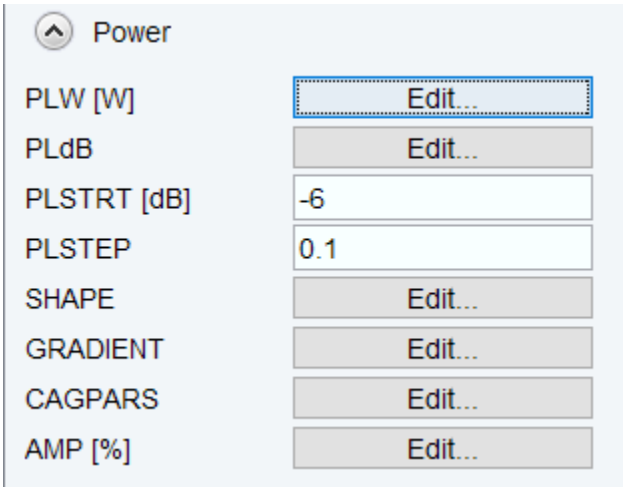
Note: DSPFIRM must be rectangle



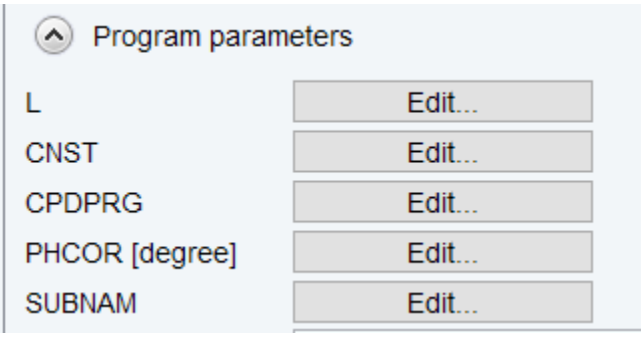
Note: NUC1 must be the same for F2 and F1, and NUC2 must be checked off.



Note: must use optimized values for p1, p2 and p3. Check p15=2000, d4=0.000020.



Note: use same power level for plw1, plw11 and plw12. Check plw21 about 0.2-0.3 W.



Note: all the others use default

7) Command **zg** to collect data. During data acquisition, you can check data quality by **xfb** or **xfshear**.
8) When experiment is finished, follow the steps from Ending Work Part (see above)