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MQMAS Using Split-t_1 Acquisition V.2

In 1 collection

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Alexander L. Paterson[1]

[1]National Magnetic Resonance Facility at Madison (NMRFAM), University of Wisconsin-Madison, Madison, WI, United States

NMRFAM Facility
UW-Madison

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We use this protocol and it's working

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Abstract

Purpose
Acquisition of a multiple quantum MAS experiment with a 3Q coherence pathway and split-t_1 acquisition.

Scope
This protocol is intended for use on a sample of interest after completing SOP Materials Central-Transition-Selective Pulse Width Optimization and SOP Materials Spin Echo. MQMAS should only be performed when a) the 1D spectrum has insufficient resolution for adequate deconvolution, and b) all peaks are in the fast-spinning limit.

Materials

Definitions:
1. MQMAS: Multiple-quantum magic angle spinning
2. 3QMAS: Triple-quantum magic angle spinning
3. CT: Central transition
4. rf: Radiofrequency
5. υ_{rf}: Radiofrequency amplitude
6. C_Q: Quadrupolar coupling constant

Appendix:
If the phasing in F2 requires adjustment, transform F2 using the command **xf2** and correct the phasing on the first row.

Before start

User should be familiar with the power limitations and duty cycle of the probe being used.

Expected amount of time: 24 hours.

Procedure

1 Begin with an optimized 90° pulse using the highest safe rf power allowed by probe. Note the duration and power level.

Safety information

If you are not sure what the highest safe rf power of the probe is, check with your local facility manager. Using a lower power will primarily affect the sensitivity of the experiment, so when in doubt use a power that is known to be safe.

2 Optimize a weak CT-selective 180° pulse. Note the duration and power level.

Note

This pulse will usually be on the order of 10-20 μs long.

3 Using a Hahn echo with whole-echo acquisition, test to see if full echo acquisition is possible.

3.1 Set the parameter **l20** to ensure that there is no residual FID and a full echo is acquired.

4 Load the 3QMAS pulse sequence **mp3q**.

5 Set initial values for later optimization:

5.1 The triple-quantum excitation pulse **p1** and power level **pl11** should be set to the 90° pulse optimized in Step 1.

5.2 The conversion pulse **p2** should be set to **p1**/3.

5.3 The selective pulse **p4** and power level **pl12** should be set to the CT-selective 180° pulse values optimized in Step 2.

5.4 The relaxation delay **d1** should be set to 1.3 $\times T_1$, where T_1 is the largest value in the sample.

5.5 **l20** should be set to the value optimized in Step 3.1.

5.6 **cnst31** should be set to the MAS frequency.

5.7 **ns** should be set to 96.

1. If the experimental time with **ns**=96 is too long to allow for optimization, using **ns**=48 should allow for adequate optimization of **p1** and **p2**.

6 Using **popt**, optimize **p2**, followed by **p1**.

6.1 Both will have maxima at somewhat greater than 90°; optimizing over a range from 70° to 140° should find an appropriate maximum.

6.2 Consider all resonances when optimizing these pulses. The optimal length depends on the C_Q of the resonance, so in systems with a wide range of C_Q values there may not be an universally optimal pulse length. Choose a value which provides adequate sensitivity for all resonances.

7 Once **p1** and **p2** are optimized, create a new experiment and convert it to 2D acquisition.

8 Set the following parameters for 2D acquisition:

8.1 **FnMode** QF

8.2 **MC2** QF

8.3 **F1 NUC1** to match the nucleus being acquired. Note that this must be set in the **eda** panel and cannot be set from the command line.

Note

The default value is often ^{1}H, which interferes with the ability to set the F1 sweep width.

8.4 **1 td** 64

Note

The optimal value of **1 TD** depends on the desired resolution in F1, the T_2 of the environments in the sample, and the available spectrometer time. Too few **1 TD** points could lead to truncation artifacts, while too many wastes time.

Note

1 td 64 will likely be acceptable or even excessive for amorphous samples, but too short for crystalline samples with long T_2; even if too short, 64 can be an adequate starting point.

8.5 **1 swh** to the MAS rate in Hz.

Note

Be sure **1 NUC1** is set appropriately before setting **1 swh**.

Note

If this is too small to observe all of the resonances in the sample without folding, half-rotor synchronization (where **1 swh** is twice the MAS rate) is permissible but will lead to spinning sidebands being observed in the spectrum.

8.6 **in0** to one rotor period

8.7 **in10** to the following value depending on nuclear spin:

1. For I = 3/2, **in0 × 7/9**

2. For I > 3/2, **0**

8.8 **in11** to the following value depending on nuclear spin:

1. For I = 3/2, **0**
2. For I = 5/2, **in0 × 19/12**
3. For I = 7/2, **in0 × 101/45**

9 Adjust both **ns** (in multiples of 96) and **1 td** to make best use of available experimental time.

10 Acquire the spectrum.

11 As this is a split-t_1 acquisition, no special post-processing is required.

Protocol references

Amoureux, J.-P. and Pruski, M. (2008). MQMAS NMR: Experimental Strategies and Applications. In eMagRes (eds R.K. Harris and R.L. Wasylishen).
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Yannick Millot, Pascal P Man, Procedures for Labeling the High-Resolution Axis of Two-Dimensional MQ-MAS NMR Spectra of Half-Integer Quadrupole Spins, Solid State Nuclear Magnetic Resonance, Volume 21, Issues 1–2, 2002, Pages 21-43,
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Example Data at 1.1 GHz: **usnan.org/ark:/83454/e1db493711-9f53-4866-b79a-696c38f7f053**
Example Data at 900 MHz: **usnan.org/ark:/83454/e12d1b859c-4650-4011-a42c-0f1d8aa37d27**
Example Data at 600 MHz: **usnan.org/ark:/83454/e1e19e4de0-8930-45c7-9e6c-465cd281b194**

Protocol

NAME

Saturation Recovery for Quadrupolar Nuclei

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Spin Echo with Whole-Echo Acquisition

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Quadrupole Non-Selective Pulse Width Optimization

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Quadrupole CT-Selective Pulse Width Optimization

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