

# IMSERC User Manual for Solids

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# IMSERC User Manual for Hg400-Solids (V2.0)

## INTRODUCTION

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Solid state NMR is useful to those samples not soluble in any solvent or structural changed in solution. CPMAS is a common solid NMR Experiment for 1D X-nucleus. It has two advantages: increase the peak intensity for dilute spins, e.g.  $^{13}\text{C}$ , and shorten delay time  $d_1$ .

## SAFETY

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All users of IMSERC must review the general safety policies at <http://imserc.northwestern.edu/about-policies.html>. Plus, users should know the points below:

- Users with pacemaker are not allowed
- Emergency Exits
- Location of shower and eyewash
- No food and drink
- Dressing code
- 5-Gauss line

## DATA MANAGEMENT

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Your personal data folder is created during training. Please save data under your personal folder, which must be located under your supervisor's group folder. See a staff member if you do not have a personal folder on this instrument yet.

NMR data on this instrument are copied on 'imsercdata.northwestern.edu' under 'PI/AREA/INSTRUMENTNAME' within a few seconds. Please follow instructions at <http://imserc.northwestern.edu/about-general-faq.html#data> for details about data access.

## SOFTWARE

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Offline data processing and analysis can be performed with MNOVA and Topspin. Northwestern has campus wide license for MestraNova. Please refer to Mnova installation instructions at <http://imserc.northwestern.edu/about-general-faq.html#software> to install the program on your computer. Make sure to connect to Northwestern VPN when you work off campus. You are also encouraged to download the Topspin software from Bruker at

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<https://www.bruker.com/service/support-upgrades/software-downloads/nmr.html> and claim your free academic license.

## PREPARATION

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Spectrometer: NMR-Hg400-Solid only

Probe: Bruker 4mm HX probe

Prerequisite: users have done the basic NMR training; also users should understand the differences between solution and solid NMR, e.g. hardware, software, T1 and T2, etc.

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

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

Sample tools: from Left to right: Sample filling funnel, sample packing/removing rod/drill, inserting cap gauge, removing cap blade, spatula, black Sharpie



## DEFAULT INSTRUMENT STATUS

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The default measurement mode of Hg400-Solids is 4mm HX probe with 1H in H channel and 13C in X channel. If you would like to run an experiment in a different mode than H/C, e.g. 31P, you need to tune back to the default setting when you finish the experiment.

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The default working condition of INSTRUMENT-NAME is as follows:

1. Computer screen is by default deactivated. You must start your reservation through NUCore in order to be able to turn on the computer screen. If screen is already on, start your reservation through NUCore
2. Acquisition software tospin should be kept on screen. Leave the acquisition software open when you are done with the measurement.

If there is an error or problem with the instrument, please report the issue by following at least one of the steps below:

1. If you have already started your reservation using NUCore, please logoff by selecting the error reporting option and a brief description about the issue
2. If you have not started your reservation using NUCore, please report problems with the instrument at <http://imserc.northwestern.edu/contact-issue.html> add place the 'Stop' sign near the instrument computer. 'Stop' signs are located at XXXX and online at the link above
3. Email or talk to a staff member

## EXPERIMENT SETUP

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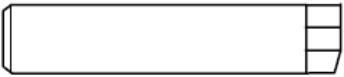


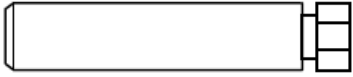

### STEP 1. LOAD SOLID POWDER INTO A 4MM ROTOR

Before you start, you need to watch this Bruker video <https://www.youtube.com/watch?v=bNFJj2g0UjI>

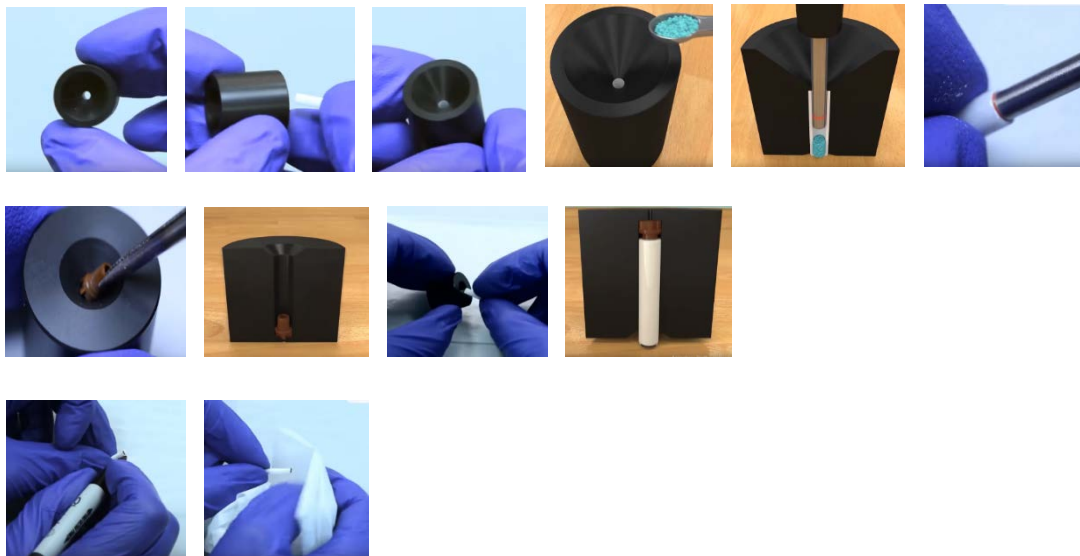
By using special tools to pack sample and finally cap it (more details are shown below)

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## Rotor and Kel-F Cap Trouble Shooting Guide

| Rotor and Cap Condition<br>(Exaggerated)  | Problem  | Cause/Prevention   |
|---|--|--|
|    | Dented cap will not spin properly  | <p>Kel-F caps can be dented with excessive force. Never use a hard surface to press the cap into the rotor</p> <p>Change another cap</p> |
|    | Cut cap will not spin properly   | <p>Caps are cut by improper use of metal cap puller.</p> <p>Consult puller instructions or use liquid nitrogen to remove caps</p>        |
|  | Tilt cap will not spin properly  | <p>Rotor was packed too full or not symmetrically-packed</p> <p>Remove excess sample or repack sample then reinsert cap</p>              |
|  | <p>Cap not inserted completely due to too much sample,</p> <p>or cap pops off during spin up (that could stop spinning abruptly)</p> | <p>Remove excess sample then re-cap it</p> <p>The cap is loose. Try another cap</p>  |
|  | Dark smudges or marks on end of rotor indicate contact with bearing and/or drive plates during spin up                               | <p>Non-symmetrically-packed rotors can 'wobble' during spin up</p> <p>Replace another rotor or use low spin-rate</p>                     |

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Note:

- Make the rotor and cap are in good condition (see the Trouble Shoot Guide above)
- Pack sample fairly tight inside of rotor
- Use packing rod to measure height, make sure enough space for a cap inserted
- Make sure the cap is not damaged and **tightly inserted**
- Re-label the black mark at bottom of rotor if necessary

## STEP 2. INSERT ROTOR INTO THE PROBE

From top of magnet, remove a special cage, black mark side down, then put cage back immediately

## STEP 3. SPIN SAMPLE

Open MAS display by double click icon MAS from screen bottom, then click Insert button, then GO

Note:

- Set starting spin rate 5000, then increase to another spin-rate as you want
- Avoid using the up-limit of spin rate 15 KHz, instead, use only the spin rate you needed
- Don't keep rotor spinning when data acquiring completed, e.g. spin overnight but experiment is finished at 11 PM

## STEP 4. EDIT A NEW DATA SET

Start from Data Browser panel

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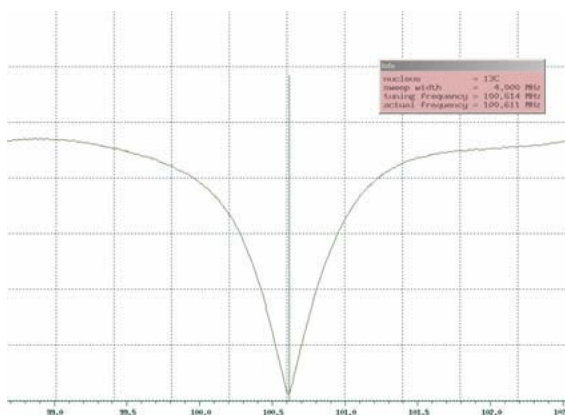
Make sure correct data saving path, i.e. /PI/netid/

You can select current parameter set or SELECT Expt

## STEP 5. TUNING

Type command **wobb** or **wobb thick** to see a tuning dip (below)

Go to the probe bottom to rotate tune/match rods for X then H (below)



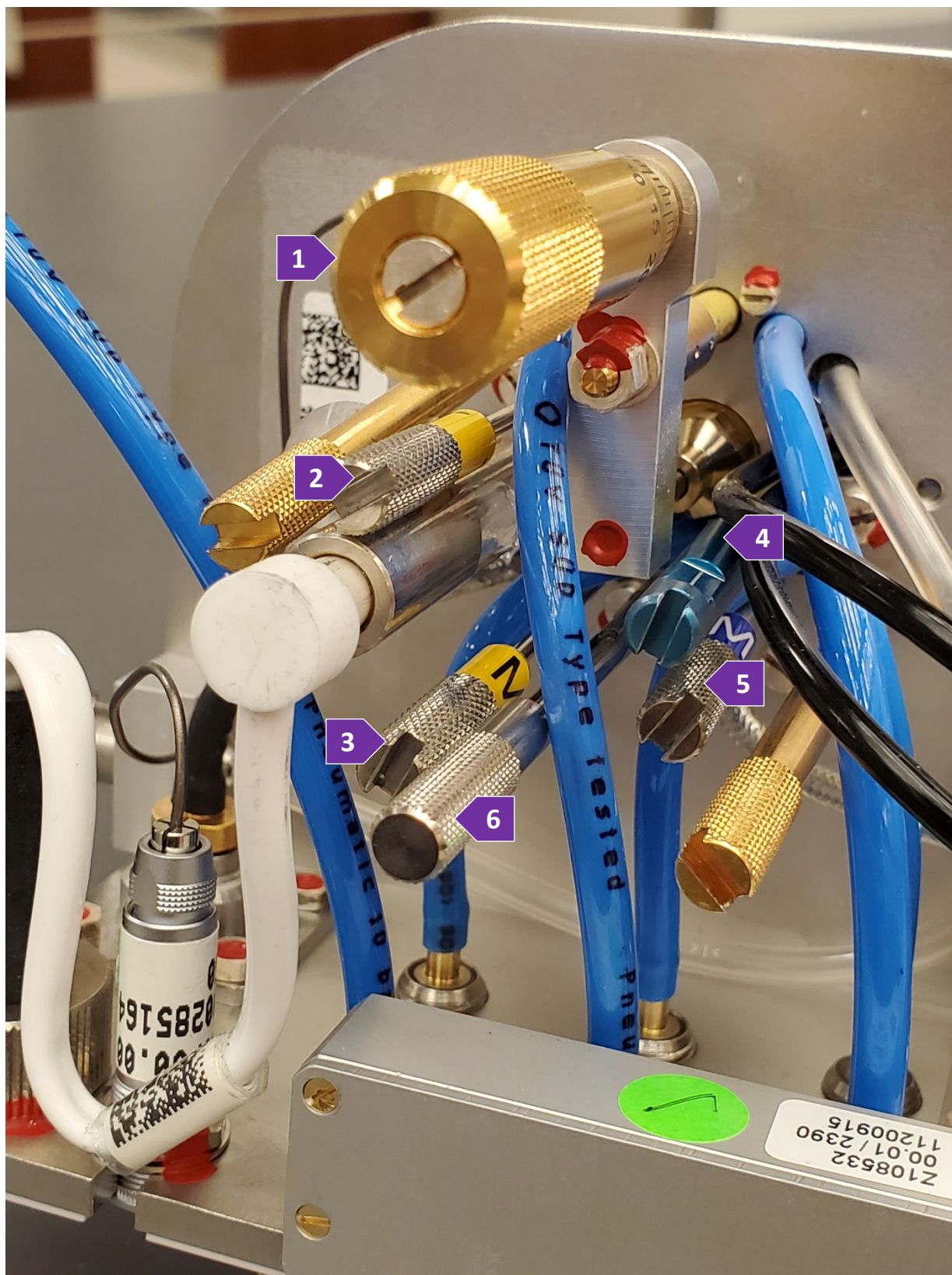
- Note:
  1. It takes about 20 seconds to show the wobb signal
  2. You may change tuning scale to a large one if you don't see a tune dip
  3. Never overturned tune rods, all the moves, either rotate or up/down, should be gently
  4. For X-band switch rod (see picture below), **you may gently move it up or down**. There are three vertical positions (see chart below)

| Switch Position   | All the way Down | Middle  | All the way Up |
|-------------------|------------------|---------|----------------|
| Freq. Range (MHz) | 44 - 111         | 32 - 44 | 63 - 171       |
| Typical Nucleus   | 13C              | 15N     | 31P            |
|                   |                  |         |                |

## Diagram of 4mm Probe Tuning Rods



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Note for probe tuning rods:

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1. Magic Angle Adjust Rod, Golden (never touch it)
2. H-Tune Rod, yellow with a label T
3. H-Match Rod, yellow with a label M
4. X-Tune Rod, blue with a label T
5. X-Match Rod, blue with a label M
6. X-Band Switch Rod, silver color with a label X-band (move it Up or Down only)

|        |        |
|--------|--------|
| 2. H-T | 4. X-T |
| 3. H-M | 5. X-M |

## STEP 6. ACQUIRE DATA

Click AcquPar Tab to change **ns** as you want

## ENDING WORK

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- 1) Stop spinning
- 2) Eject rotor out of probe
- 3) Remove sample out of rotor by special tools and clean up the rotor

<https://www.youtube.com/watch?v=bNFJj2gOUjI>



- 
- Note:
  - a. Follow the instructions above to remove the cap

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- b. Use drill tool to take powder out of rotor
  - c. Use iso-propanol to remove powder if necessary, and dry rotor inside after
- 
- 4) Keep desk top clean
  - 5) Logout from NUcore

## DATA ANALYSIS

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MNova is recommended (see SOFTWARE Part). More details can be seen from the link of MNova Tutorial [http://imserc.northwestern.edu/downloads/nmr-mnova\\_chemists8.pdf](http://imserc.northwestern.edu/downloads/nmr-mnova_chemists8.pdf)

## PUBLICATION

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### EXPERIMENTAL SECTION

“Solid state NMR data were collected at room temperature on a Bruker Avance III 400 MHz spectrometer equipped with a 4mm HX probe.”

### ACKNOWLEDGEMENT

Add Acknowledgement info as listed under <http://imserc.northwestern.edu/about-acknowledgements.html>

Use was made of NMR-Hg400-solids at Northwestern University, which has received support from National Science Foundation (CHE-9871268) and International Institute for Nanotechnology (IIN).

## TROUBLESHOOTING

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Provide information about common mistakes users do that prevents them from starting a measurement

1. If auto-spinning fails, double check the following:
  - a. Cap loose or tight
  - b. Cap all the way inserted
  - c. Any scratching strip on the rotor
  - d. Rotor bottom black mark worn
  - e. Re-pack sample

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2. If auto-spinning fails and device MAS III has red lid, you can do rebooting as following:
  - f. Go to the back of MAS device
  - g. Switch power off and wait for 10 seconds
  - h. Switch power on then wait for all lights solid green. This takes about 3 minutes.

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## APPENDIX A: HOW TO PACK/REMOVE SAMPLE INTO/OUT OF A ROTOR

Watch a video: <https://www.youtube.com/watch?v=bNFJj2g0UjI>

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## APPENDIX B: MULTI QUANTUM MAGIC ANGLE SPINNING (MQMAS)

### INTRODUCTION

Solid state NMR is useful to those samples not soluble in any solvent or structural changed in solution. 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

1. Spectrometer: NMR-Hg400-Solid only
2. Probe: Bruker 4mm HX probe
3. Prerequisite: users have done the basic NMR training and solid NMR training.
4. Sample: must be in powder form; and about 80 mg
5. Rotor: Bruker <https://bruker-labscape.store/products/4mm-mas-rotor-kit> Part# H14355

### DEFAULT INSTRUMENT STATUS

The default measurement mode of Hg400-Solids is 4mm HX probe with 1H in H channel and 13C in X channel. If you would like to run an experiment in a different mode than H/C, e.g. 31P, you need to tune back to the default setting when you finish the experiment.

The default working condition of INSTRUMENT-NAME is as follows:

3. Computer screen is by default deactivated. You must start your reservation through NUCore in order to be able to turn on the computer screen. If screen is already on, start your reservation through NUCore
4. Acquisition software topspin should be kept on screen. Leave the acquisition software open when you are done with the measurement.

If there is an error or problem with the instrument, please report the issue by following at least one of the steps below:

1. If you have already started your reservation using NUCore, please logoff by selecting the error reporting option and a brief description about the issue

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2. If you have not started your reservation using NUCore, please report problems with the instrument at <http://imserc.northwestern.edu/contact-issue.html> add place the 'STOP' sign near the instrument computer. 'Stop' signs are located at XXXX and online at the link above
3. Email or talk to a staff member

## 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**
9. When finish
  - a. Stop spinning and eject rotor out of probe
  - b. Remove sample out of rotor by special tools and clean up the rotor
  - c. Keep desk top and prep bench clean
  - d. Logout from NUcore


## EXPERIMENT SETUP (DETAILED PROCEDURES)

### STEP 1. LOAD SOLID POWDER INTO A 4MM ROTOR

Run a regular 1D first. You can use pulse program "onpulse" 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.

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## STEP 2. INSERT ROTOR INTO THE PROBE

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

## STEP 3. SPIN SAMPLE

Using  $ns=12*n$  to run 1D. Zoom in the peak region, then type **dpl**

## STEP 4. EDIT A NEW DATA SET

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.

Note:  $d0=1\mu s$  and  $d4=20\mu s$

| 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 |

## STEP 5. TUNING

Edit another new data set for 2D. Underneath the Tab ACQUPAR, click an icon 



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## STEP 6. ACQUIRE DATA

Set up all parameters with details below

|            | F2                    | F1  |
|------------|-----------------------|-----|
| Experiment |                       |     |
| PULPROG    | mp3qzqf               | ... |
| AQ_mod     | DQD                   |     |
| FnTYPE     | traditional(planes) v |     |
| FnMODE     | States v              |     |
| TD         | 512                   | 64  |
| DS         | 0                     |     |
| NS         | 24                    |     |
| TD0        | 1                     |     |
| TDav       | 0                     |     |

Note: NS must be 12\*n. TD F1 could be 64 or 128

| Width       |             |            |
|-------------|-------------|------------|
| SW [ppm]    | 152.9185    | 38.2296    |
| SWH [Hz]    | 20000.000   | 5000.000   |
| IN_F [µsec] |             | 200.00     |
| AQ [sec]    | 0.0128000   | 0.0064000  |
| FIDRES [Hz] | 78.125000   | 156.250000 |
| FW [Hz]     | 4032000.000 |            |

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

| Receiver          |                                |
|-------------------|--------------------------------|
| RG                | 203                            |
| DW [ $\mu$ sec]   | 25.000                         |
| DWOV [ $\mu$ sec] | 0.025                          |
| DECIM             | 1000                           |
| DSPFIRM           | rectangle                      |
| DIGTYP            | #RU (digitizer not installed!) |
| DIGMOD            | baseopt                        |
| DR                | 22                             |
| DDR               | 10                             |
| DE [ $\mu$ sec]   | 6.50                           |
| NBL               | 1                              |
| HPPRGN            | normal                         |
| PRGAIN            | high                           |
| DQDMODE           | add                            |
| PH_ref [degree]   | 46.000                         |
| OVERFLW           | check                          |

Note: DSPFIRM must be rectangle

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| Nucleus 1  |             |   |
|------------|-------------|---|
| NUC1       | 87Rb        | <input type="button" value="Edit..."/> 87Rb |
| O1 [Hz]    | -5800.00    | -5800.00                                    |
| O1P [ppm]  | -44.344     | -44.344                                     |
| SFO1 [MHz] | 130.7886520 | 130.7886520                                 |
| BF1 [MHz]  | 130.7944520 | 130.7944520                                 |

| Nucleus 2  |             |  |
|------------|-------------|--|
| NUC2       | off         | <input type="button" value="Edit..."/> |
| O2 [Hz]    | 0           |  |
| O2P [ppm]  | 0           |  |
| SFO2 [MHz] | 399.7330000 |  |
| BF2 [MHz]  | 399.7330000 |  |

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

| Durations   |  |
|-------------|--|
| P [µsec]    | <input type="button" value="Edit..."/> |
| D [sec]     | <input type="button" value="Edit..."/> |
| IN [sec]    | <input type="button" value="Edit..."/> |
| INP [µsec]  | <input type="button" value="Edit..."/> |
| HDDUTY [%]  | 20.0                                   |
| HDRATE      | 1                                      |
| PCPD [µsec] | <input type="button" value="Edit..."/> |
| V9 [%]      | 5.00                                   |

Note: must use optimized values for p1, p2 and p3. Check d0=0.000001, d4=0.000020

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Power

|             |         |
|-------------|---------|
| PLW [W]     | Edit... |
| PLdB        | Edit... |
| PLSTRT [dB] | -6      |
| PLSTEP      | 0.1     |
| SHAPE       | Edit... |
| GRADIENT    | Edit... |
| CAGPARS     | Edit... |
| AMP [%]     | Edit... |

Note: use calibrated power level value for plw11. Check plw21 no more than 0.5 W.

Also check a parameter MASR is the same value of your spinning rate

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## APPENDIX C: HETERONUCLEAR CORRELATION (HETCOR)

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### INTRODUCTION

Based on CPMAS and dipolar coupling, HETCOR is a most common 2D solid NMR Experiment for H-X correlation, especially for H-C. This proton-hetero nucleus chemical shift correlation provides better resolution on 1H dimension and therefore more structural information, which is helpful to the data analysis and elucidation.

### PREPARATION

Spectrometer: NMR-Hg400-Solid only

Probe: Bruker 4mm HX probe

**Prerequisite:** users have done the basic NMR training and **solid state 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

#### STEP 1. LOAD SOLID POWDER INTO A 4MM ROTOR

By using special tools to pack sample and finally cap it

#### NOTE:

- Pack sample fairly tight inside of rotor
- Use packing rod to measure height, make sure enough space for a cap inserted
- Make sure the cap is not damaged and tightly inserted
- Re-label the black mark at bottom of rotor if necessary

#### STEP 2. INSERT ROTOR INTO THE PROBE

From top of magnet, remove a special cage, black mar side down, then put cage back immediately

#### STEP 3. SPIN SAMPLE

Open MAS display by double click icon MAS Spin Rate from screen bottom

Set starting spin rate 5000, click Insert button, then GO

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Increase spin rate to 10000 (recommended for hetcor)

## STEP 4. EDIT A NEW DATA SET

Start from Data Browser panel

Make sure correct data saving path, i.e. /PI/netid/

You can select current parameter set or SELECT Expt, e.g. 4mm\_13C\_hetcor

## STEP 5. TUNING

Type command **wobb** or **wobb thick** to see a tuning dip

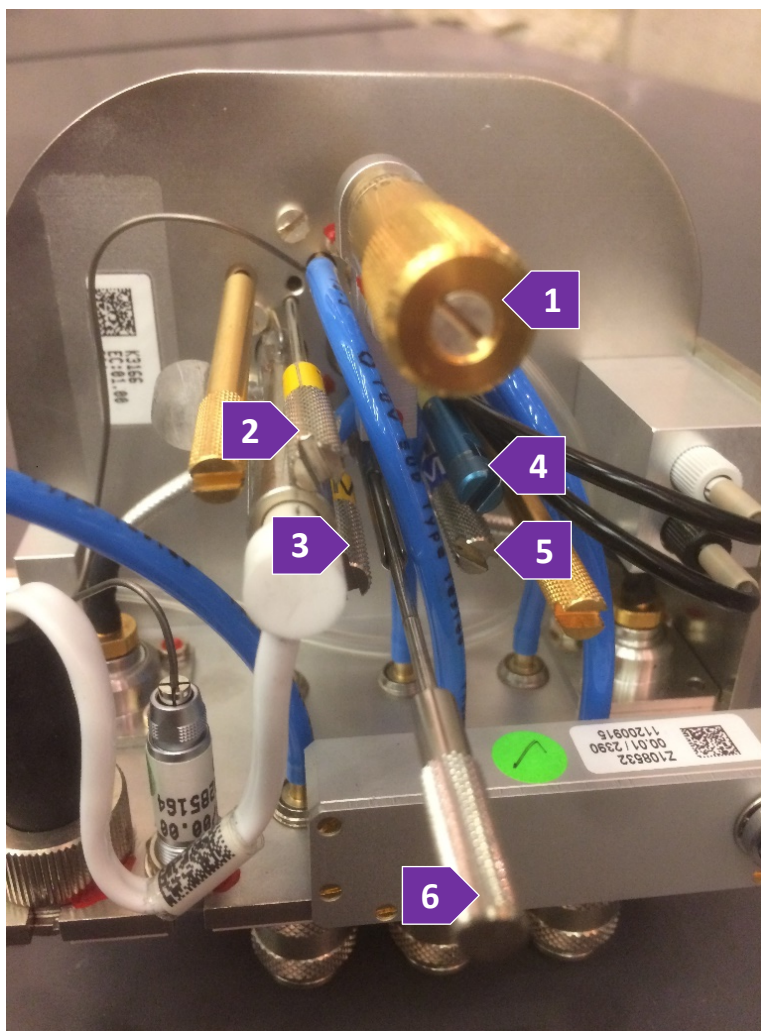
Go to the probe bottom to rotate tune/match rods for X then H (below)

Note:

5. It takes about 20 seconds to show the wobb signal
6. You may change tuning scale to a large one if you don't see a tune dip
7. **Never overturned tune rods**
8. For X-channel switch rod (see below), **never rotate, only up or down**. There are three vertical positions (see chart below)

| Switch Position   | All the way Down | Middle  | All the way Up |
|-------------------|------------------|---------|----------------|
| Freq. Range (MHz) | 44 - 111         | 32 - 44 | 63 - 171       |
| Typical Nucleus   | 13C              | 15N     | 31P            |
|                   |                  |         |                |

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- Magic Angle Adj – never touch it
- H Tune rod
- H Match rod
- X Tune rod
- X Match rod
- X-band select rod – gently touch it Up or Down,

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## STEP 6. OPTIMIZE ACQUISITION PARAMETERS

Click AcquPar Tab then switch icon to Short format, then double check parameters below. Here is an example for <sup>13</sup>C-1H HETCOR, but could be applied to other hetero-nuclei.

PULPROG lghetfq  
TD 1024 to 4096  
NS 4\*n  
TD1 64 to 128  
SWH 50000 (recommend using same scale as 1d <sup>13</sup>C CPMAS)  
AQ 0.01 to 0.05  
CNST20 100000 (in this case 1H 90 degree pulse p3=2.5)  
CNST24 -3000  
D1 5s (recommended)  
In0 0.000056  
L3 3  
P15 50 to 200  
PLW1 300 (same as 1d <sup>13</sup>C CPMAS)  
P1 4 (same as 1d <sup>13</sup>C CPMAS)  
SPNAME0 ramp50100.100  
CPDPRG2 spinal64  
P3 2.5  
PCPD2 4.8  
PLW12 130  
PLW13 130  
SPW0 80  
MASR 10000  
FnMODE STATES-TPPI

## STEP 7. ACQUIRE DATA

zg



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## REVISIONS

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|            |   |
|------------|---|
| V2.0       | Added MQMAS and HETCOR into the main manual |
| 2021-06-29 |   |

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