IMSERC User Manual for Hg400-Solids

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INTRODUCTION

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. 13C, and shorten delay time d1.

SAFETY

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

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

On Hg400-solids spectrometer, **topspin 3.6.2** is used for data collecting. For offline data processing and analysis, software **MNova** is recommended. The software can be downloaded from Mestrelab website https://mestrelab.com/download/mnova/ as a trial version for 45 days, then install our site license from \\imsercdata.northwestern.edu\public\MNova\License\. Please note that:

- Software MNova is available for both Windows and Mac of your personal computer
- Software is also installed on the communal computers located in the area outside room BG51
- If a user prefers using topspin software to process data, he/she can download a free academic version from Bruker website https://www.bruker.com/service/support-upgrades/software-downloads/nmr.html

PREPARATION

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-labscape.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

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:

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 topspin should be kept on screen. Leave the acquisition software open when you are done with the measurement.

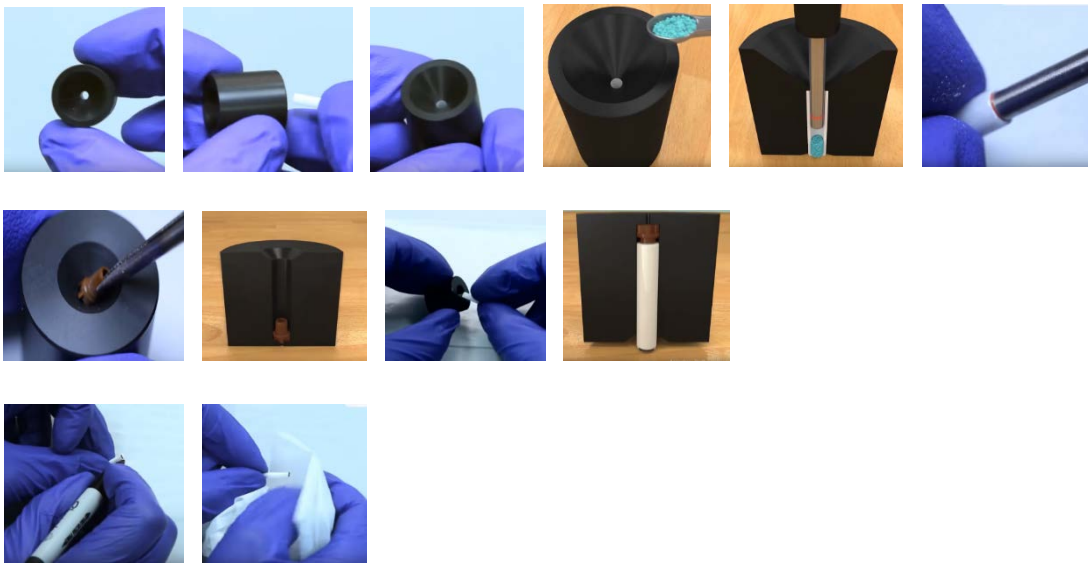
If there is an error or problem with the instrument, please report the issue by following at least one of the steps below:

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
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

STEP 1. LOAD SOLID POWDER INTO A 4MM ROTOR

By using special tools to pack sample and finally cap it (follow steps below)



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, then increase to any spin-rate as you want

Click Insert button, then GO

STEP 4. EDIT A NEW DATA SET

Start from Data Browser panel

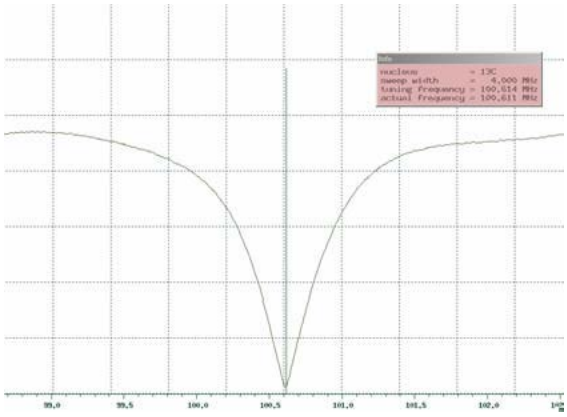
Make sure correct data saving path, i.e. /PI/netid/

You can select current parameter set or SELECT Expt

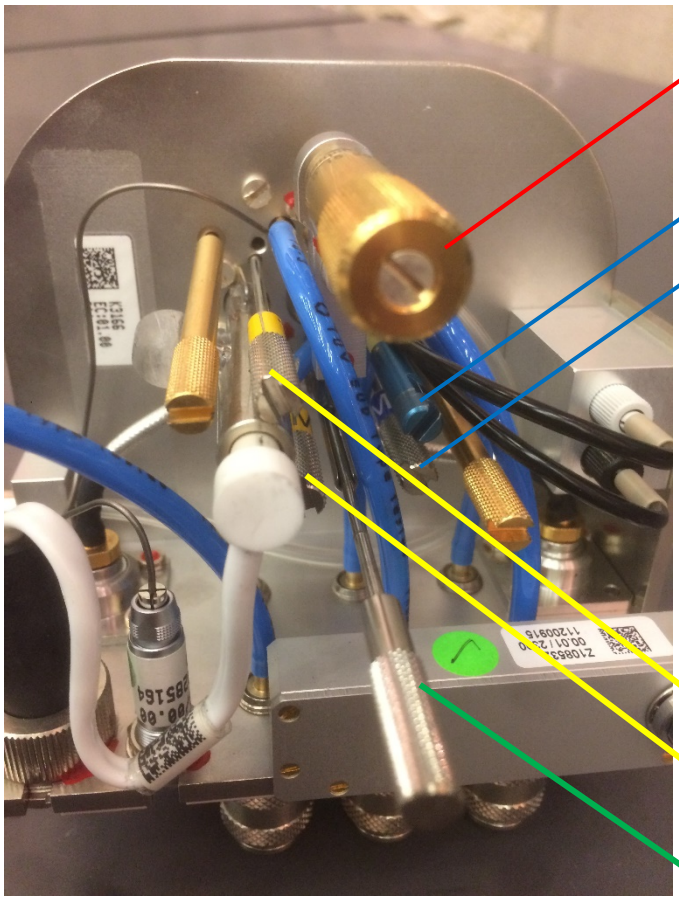
STEP 5. TUNING

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
  4. For channel switch rod (see below, indicated by green line), only up or down, never rotate



STEP 6. ACQUIRE DATA

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

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
   https://www.youtube.com/watch?v=bNFJj2g0UjI

- Note:
  1. Follow the instructions above to remove the cap
  2. Use drill tool to take powder out of rotor
  3. Use iso-propanol to remove powder if necessary, and dry rotor inside after

4) Keep desk top clean
5) Logout from NUcore

DATA ANALYSIS

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

PUBLICATION

EXPERIMENTAL SECTION

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

ACKNOWLEDGEMENT

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

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TROUBLESHOOTING

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

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.

APPENDICES

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

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

APPENDIX B: BRUKER SOLID STATE NMR MANUAL

https://www.nmr.ucdavis.edu/sites/g/files/dgvnsk4156/files/inline-files/solids.pdf

REVISIONS

V1.1 2020/02/12	• By yuyang
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