Academic Affairs Laboratory S.O.P.
Title: Basic Operation for Bruker 500 MHz NMR

Section One:

☐ Material Hazard

☐ Process/Task/Equipment

☒ Instrumentation/Equipment

PURPOSE AND SCOPE SECTION

The written SOP entitled Basic Operations for the Bruker 500 MHz NMR is designed to provide a standard operation procedure for approved faculty, staff, student, and student employees to operate the NMR in the Core Laboratory. This SOP addresses only the basic operations of the NMR and does not address any NMR maintenance including the use of cryogens, spectrometer modifications repair or emergency procedures.

HAZARDS AND CONTROL SECTION

1. Strong Magnetic Fields may interfere with a heart pacemaker or any metallic medical implants. Individuals with metallic medical implants shall not use the NMR and stay outside the 5 gauss boundary of the instrument indicated by warning signs, chains and floor tape. Individuals with any type of ferromagnetic materials within their body shall consult with the core lab director and a staff who is in charge of NMR spectrometer.

2. Strong Magnetic Fields may create an attraction of ferromagnetic materials or magnetic materials. No metallic objects shall be taken inside the 5 gauss boundary. Leave all objects such as mobile phone, watches, pens, keys, ID card, credit card, other electronic device, jewelry at the designated location outside the 5 gauss boundary.

3. Strong Magnetic Fields will damage magnetic tape and disks or magnetic strip such as credit cards and building access cards.

4. Cryogenic hazards: liquid helium and liquid nitrogen may cause burns/irritation if liquid comes into contact with skin.

5. Asphyxiation hazards: If large volume of helium escapes into a space with poor ventilation may result in asphyxiation. The liquid helium asphyxiation hazard would most likely occur during maintenance or filling operation of helium not the normal operations.

6. Magnet quenching: The rapid release of gaseous cryogens from the cryostat into the room which causes a rapid and sudden magnetic burst with subsequent degradation of the magnetic field. Evacuation of the Core Laboratory shall occur immediately.

7. Loading and removing NMR sample tubes into the magnet: Potential exposure to static magnetic fields. Operator shall not place hands on the magnet canister while inserting or removing samples from the magnets.

8. Spills or breakage of the tubes: Report any hazardous or dangerous spills and any spills in the in the instrument, to the Shimadzu Core Lab director or Core Lab personnel in charge of the NMR spectrometer. The broken tubes should be disposed of in broken glass waste disposal and any spill should be cleaned using appropriate chemical safety procedures. In the event of a tube breakage in the NMR instrument, all operation work shall stop until the users are notified to resume operation of the instrument.

9. The oxygen level should not be below 20%. Oxygen monitor is located in the Core Laboratory and checked according to manufacturing specifications.

PERSONAL PROTECTIVE EQUPIMENT SECTION

Safety glasses/goggles and other PPE are not required for operation of instrument.

PROCEDURE SECTION

A. Sample Preparation shall not occur in the Core Laboratory

1. Inspect the NMR tube prior to use. Do not use tubes that are in substandard condition. The NMR tubes of good quality designed for 500 MHz NMR spectrometers are recommended. Tubes manufactured from glass of Type 1 Class B standard can be used for room temperature NMR experiments. For variable temperature (at high and low temperatures) NMR experiments, Type 1 Class A glass are recommended.

2. Prepare the sample in the appropriate deuterated solvent. Ideal sample solution is clear without solid particles.

3. The recommended sample volume is 0.6 ml or 5 cm in a tube of 5mm outer diameter.

4. Transport the NMR sample tubes to the Core Laboratory in a secondary container.

B. NMR Operation

1. Before operating the NMR, users will receive instruction on instrument use by the Shimadzu Core Lab personnel in charge of the NMR.

2. Prior to crossing the designated gauss boundaries, remove all credit cards, ID card, cell phones, other electronic device, all ferromagnetic material including objects in the pockets, jewelry, and belts. Place these objects in the designated location (on white table).

3. Individuals with any type of ferromagnetic materials within their body (e.g., pacemakers, plates and screws) shall consult with the Core Laboratory director and/or Lab personnel in charge of the NMR spectrometer before crossing the designated gauss boundaries.

4. Operate the instrument using the experimental setup in the computer designated for the NMR.

5. Place the NMR sample tube in a spinner located on a Bruker NMR autosampler. The sample tube should sit tightly inside the spinner.

6. Adjust the sample depth in a sample depth gauge. The top of the slider (white part) is fixed at 2 cm from 0 cm on top. Do not change the slider level from this fixed level. The base of the sample tube should be positioned as shown in **5**, **Fig. 1**.

7. Wipe the sample tube with Kimwipe and place it on the sample changer.

8. Log into the computer with username and password given at training.

9. Click topspin on desktop followed by **Acquire/ More/ Icon NMR Automation (icona)**.

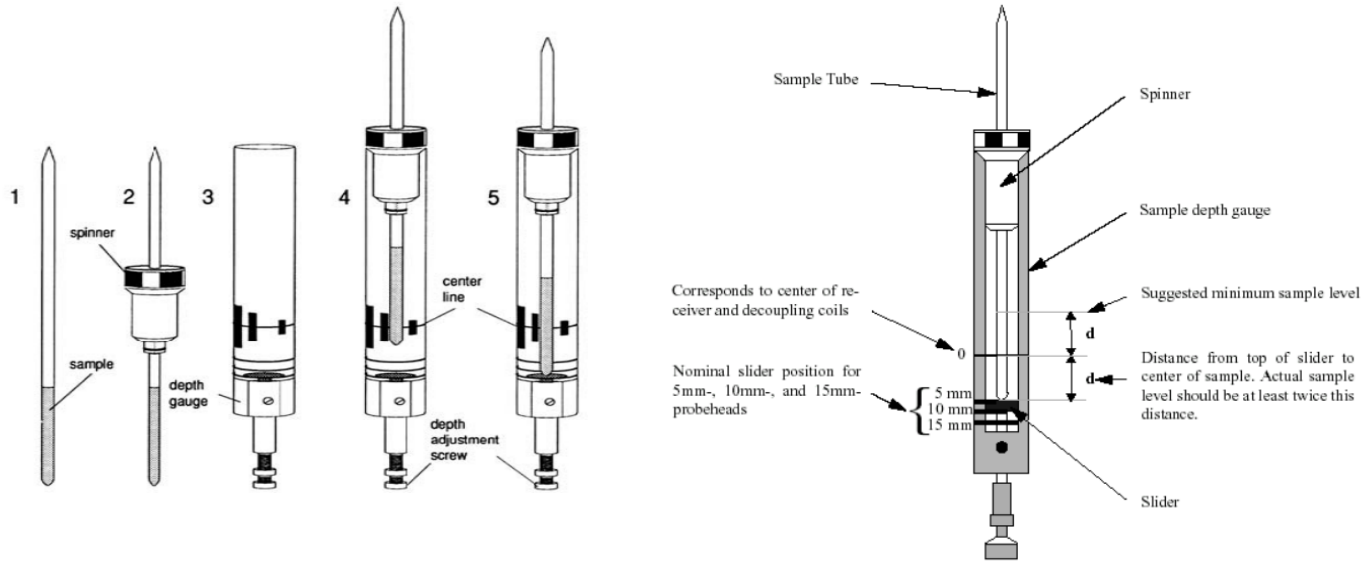


Fig. 1: Tube position and leveling of sample depth

10. Select the user ID on Icon NMR: Identify user and enter password. The “Icon NMR Automation” window will appear (Fig. 2). The students will use their research group faculty’s/PI’s ID.

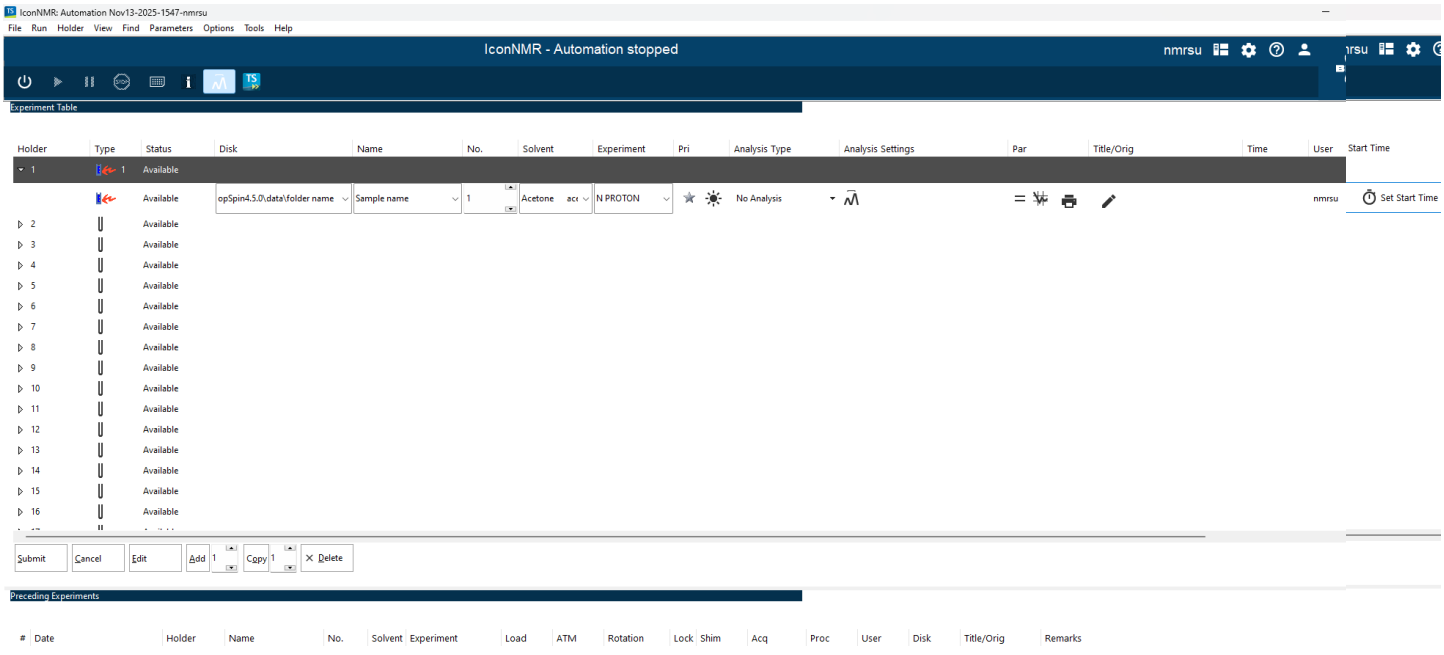


Fig 2: Icon NMR Automation window

11. On the “Icon NMR Automation” window, select the available holder and double click to fill in the details of sample.

a. Enter the information for data to be saved below disk in format **C:\Bruker\Topspin 4.5.0\data\folder name**. The folder name is the name of faculty/PI. After completion of acquisition, the data including FID file will be automatically saved in respective folder. The students have to use the folder assigned to their research group Faculty/PI.

b. Enter the name of sample, experiment number, solvent used for preparing sample, and experiment type.

c. Chose star icon to run experiment ASAP or sun icon to run it overnight.

d. It is not required to enter or edit anything in analysis type, analysis settings, par, and title/origin for basic NMR experiment used to characterize molecules.

e. The time column automatically displays the required time of experiment when it starts.

f. The user column displays the user ID.

g. The start time column allows you to schedule the start time.

h. For running multiple samples, select the separate available holder for each sample and enter the information as in (i) to (iii).

i. After all sample details are entered, click submit followed by the power sign on the top to start automation. The instrument will eject sample/spinner from the magnet and insert the new sample queued on “Icon NMR” window into the magnet.

12. After the acquisition is complete, the instrument will eject the sample and place it back to the original sample holder position.

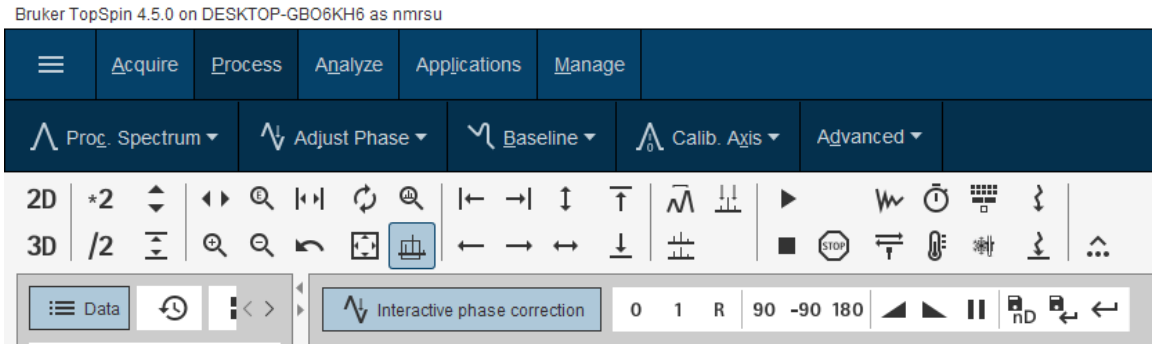
13. Remove your sample from the holder and insert blank sample from topspin window when additional sample(s) is/are not in queue.

14. Close the Icon NMR and topspin, and log out.

15. Users are responsible for taking their samples back to their labs or designated space in the Shimadzu Core Lab. Do not leave NMR sample tubes in the core lab after experiment is complete.

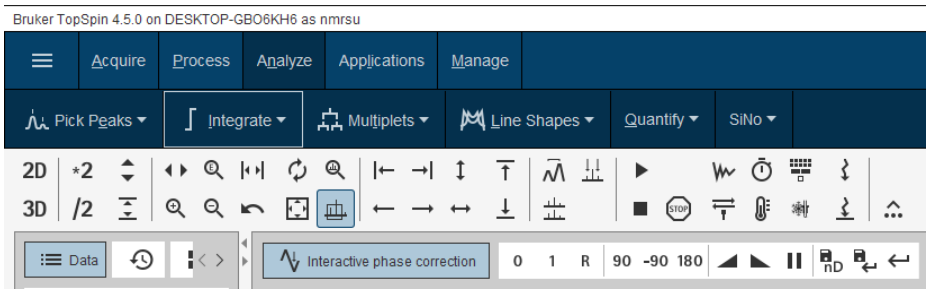
C. DATA PROCESSING

1. Click ≡ in topspin and open the file saved in user’s folder.



2. Go to process ⟶ calib axis (highlighted below) to perform calibration of solvent peak on spectrum. The phase adjustment and baseline correction can be performed from topspin when not corrected by default settings.

3. Click on “Analyze” menu button to form pick labeling, integration, and other analysis shown below.



WASTE DISPOSAL SECTION

Dispose the Kimwipe in trash.

In case of NMR tube breakage follow the Hazard and Controls Section 8 described above.

EMERGENCY RESPONSE SECTION

Magnetic quench: **immediately evacuate the area**.

1. Pull the fire alarm to evacuate the building

2. Call 911 and notify emergency responders that a superconducting magnet has been quenched and the Core Laboratory environment may contain asphyxiation hazards and high magnetic fields.

3. **Do not re-enter the Core Laboratory** until instructed to do so by emergency responders who have determined the oxygen content is greater than 19.5 % with portable handheld oxygen monitors.

Object becomes stuck in NMR:

1. If an object becomes stuck to a superconducting magnet, do not attempt to remove it.

2. Contact the Director of the Shimadzu Core Laboratory or the Core Laboratory in charge of

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TRAINING RECORD KEEPING & DEFINITIONS

1. The Director of the Shimadzu Core Laboratory and/or Core Lab personnel in charge of the NMR will verify all operators of the NMR have received the designated training and demonstrated a proficiency of the basic operations of the NMR prior to receiving approval to operate that NMR. The Director of the Shimadzu Core Laboratory and/or Core Lab personnel in charge of the NMR may request any operator to demonstrate proficiency if the operator:
    a. was involved in an incident or accident involving the NMR or
    b. has not used the NMR for over one year.

2. All operators receiving approval to operate the NMR shall not perform maintenance of any kind. They are required to report to the Director of the Shimadzu Core Laboratory and/or Core Lab personnel in charge of the NMR any incident or accident involving the NMR. The operator is required to stop all work until the incident or accident has been investigated.

3. The Director of the Shimadzu Core Laboratory personnel in charge of the NMR will maintain the training documentation for the operations of the NMR.

REFERENCES

Bruker 500'54 Ascend Evo Magnet System and topspin 4.5.0 user manual.

Approved by:

1. Name: Thomas Dowling Signature:

   Date Approved: 12/8/2025

2. Name: Mike McKay Signature:

   Date Approved: 12/16/2025

Review Date: _____ ☐ annual review ☐ changes made

Changes made on review date: _____