PINMRF

FACILITY INFORMATION
AND REFERENCE GUIDE

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PINMRF
Facility Overview

The Purdue Interdepartmental NMR Facility (PINMRF) is a university-wide resource dedicated to supporting NMR spectroscopy and to making this analytical technique available to researchers at Purdue and elsewhere in the scientific community. PINMRF currently has ten NMR spectrometers located in four laboratories in four buildings on the Purdue campus, with additional laboratory locations under consideration. PINMRF is set up to allow individual researchers direct access 24/7 to the spectrometers, after appropriate training and testing has been completed. However, we will gladly provide spectra of submitted samples, either on a service basis or as part of a collaborative research project. PINMRF maintains a website at www.pinmrf.purdue.edu.   The website has updated Facility information along with downloadable copies of our spectrometer training handouts, along with other information.

Spectrometers and Locations

PINMRF's current complement of spectrometers is shown in the table below.

Instrumental Overview – Spectrometers and Locations

Vendor	Type/Field/Name	Location	Software	Probe(s)
Bruker	ARX-300	G43 RHPH	XWinNMR	5mm QNP
Bruker	AV-III-HD-400	369 WTHR	TopSpin 3.2	5mm BBFO
Bruker	AV-III-HD-500	B055 DRUG	TopSpin 3.2	5mm BBFO cryoprobe
Bruker	AV-III-800	LB124 BRWN	TopSpin 3.2	5mm QCI cryoprobe
Bruker	Avance DRX-500-1	367 WTHR	TopSpin 1.3	5mm BBFO
Bruker	Avance DRX-500-2	G43 RHPH	TopSpin 1.3	5mm BBFO
Chemagnetics	Infinity-400WB	LB124 BRWN	Spinsight	5mm H-X-Y MAS
Varian	Inova-300-1	365 WTHR	VNMR	5mm 4-nucleus
Varian	Inova-300-2	369 WTHR	VNMR	5mm 4-nucleus

All of our liquids-state spectrometers have standard-bore magnets and sample-temperature controllers.

PINMRF has three workstations available for various tasks such as iLab kiosk (see pg. 3) and data transfer and processing. These are located in G43 RHPH (Linux), and 365 WTHR (Linux and Sun). We ask that you move your NMR data off the spectrometer host computers as soon as

possible, either to your own computer or to the off-line workstations. **PLEASE NOTE THAT PINMRF DOES NOT ARCHIVE USERS' NMR DATA**, **NOR CAN PINMRF GUARANTEE THE SECURITY OF ANY NMR DATA.**

The sensitivity and resolution of each spectrometer depends upon its field strength and other facets of its configuration. The table below shows the approximate sensitivity values for each of our liquid-state spectrometers for ^{1}H and ^{13}C observation (with ^{1}H decoupling). The s/n values are obtained using industry-standard test samples and conditions.

Instrumental Overview – Approximate Sensitivity (S/N), 5mm Probes

Console/Probe	^{1}H sensitivity	^{13}C sensitivity
Varian 300's, quad-nucleus probes	100:1	90:1
AV-III-HD-400, BBFO probe	450:1	200:1
DRX-500-1 & 2, BBFO probe	450:1	200:1
AV-III-HD-500, BBFO cryoprobe	1350:1	750:1*
AV-III-800, QCI cryoprobe	6200:1	1000:1*

*These probes will show a background signal or rolling baseline when used for ^{13}C acquisition.

PINMRF Staff

Currently PINMRF has a permanent staff of three individuals, supplemented by one or more NMR Facility TAs. The table below lists our permanent staff and their jobs and office locations. For our email addresses, and information on our current NMR TAs, please visit our website at www.pinmrf.purdue.edu. Please be aware that there is a note posted by each spectrometer listing all of the Facility staff contact information. Please refer to these notes if you need to contact someone while you are at the spectrometer.

Name	Position	Office	Phone*
John S. Harwood, D.Sc.	Director	365B WTHR	4-5287
Huaping Mo, Ph.D.	Associate Director	G43 RHPH	4-1480
Donna Bertram[#]	Administrative Assistant	361 WTHR	4-7850

* Off-campus callers please use the area code 765 and the prefix 49, i.e., 765-494-7850.

[#] Part-time PINMRF employee.

If you have a question about the Facility or need assistance with a problem, you may contact any of the individuals listed above, or any NMR T/A.

Spectrometer Access and Usage Charges

PINMRF uses software from iLab Solutions to assist with the management of our facility. Any individual wishing to use our facility in any fashion must first have an account set up with iLabs. You may use the following web address to see the PINMRF homepage at iLabs:

https://purdue.ilabsolutions.com/service_center/show_external/4067

The first time you access our iLabs homepage, click the blue "sign-up" button in the upper-right corner of the page to begin the account setup process. After doing this you also should contact your business office to let them know you have initiated the iLabs account setup process, since your business office will need to correlate your iLabs account with a Purdue account number for the payment of any charges. Please see also the material on pp. 13 – 14.

Users will be charged an hourly rate for spectrometer time. The rate structures are posted by each spectrometer and also are shown on the rates page of the PINMRF website. For spectroscopy service, an operator service fee will be included along with the cost of the instrument time. Time spent using the off-line workstations is not charged to the users. Therefore, we recommend using the off-lines or your own computer for extensive data processing.

Spectrometer Training and Checkout Requirements

User Training - Routine

We have two operating and training programs covering routine spectrometer usage: one for our Bruker spectrometers running TopSpin and one for our Varian 300 MHz spectrometers. These training programs cover the procedures necessary to obtain basic 1D NMR spectra of common nuclei (^{1}H, ^{13}C, ^{31}P, ^{19}F, depending on the spectrometer) using standard PINMRF parameter sets. The training also covers administrative and safety issues related to the use of the facility. The PINMRF training materials also include easy-to-follow instructions for obtaining certain more advanced 1D spectra, such as nOe-difference and DEPT, along with some of the simpler 2D spectra. There are no prerequisites for either the Bruker program or the Varian program. Prospective spectrometer operators will need to successfully complete the course of training, and

pass both a written checkout quiz and a practical checkout exam, before being granted permission to use the spectrometers unsupervised. Training guides are available in hard-copy form at each spectrometer, and are also available for download from http://www.pinmrf.purdue.edu/training. New users may either get training from PINMRF staff or from other, experienced, users within their own research group. If the new user is being trained within their own research group this training **MUST FOLLOW** the current PINMRF training material in use. New users who are trained by other users in their group must still take and pass the written checkout quiz and the practical checkout exam, which will be administered by a member of PINMRF staff.

We are no longer offering training on the Bruker XWinNMR platform for the ARX-300 spectrometer in RHPH as part of our standard new-user training options. User training for this spectrometer may be offered on an ad hoc basis according to need and staff availability.

If you are an experienced NMR operator you may be able to skip our training program and the practical exam, and become an approved operator after taking only the written checkout quiz. Please check with PINMRF staff if you feel this situation applies to you.

Any prospective facility user may apply for training using the form available on our iLabs website. After logging in to our iLabs website, click the "Request Services" tab. On the Training and Service Requests page, click the "initiate request" button close to the "Training Request" text. If you are an experienced spectrometer operator please provide the information requested so that we can properly process your request.

User Training - Advanced

The Chemagnetics CMX-400 solid-state spectrometer is available for use only on a service basis. We are not currently training students to be operators of this spectrometer.

Other procedures, such as the observation of non-standard nuclei and low-temperature NMR, are considered advanced training. All requests for advanced training should be made through the iLabs PINMRF website as mentioned above.

NMR Spectrometer Practical Exam

Prior to performing this practical exam you must first pass a written exam. The written exam will test you on material in this document as well as spectrometer-related matters.

This is an outline of the tasks you will be required to perform correctly in order to pass the checkout practical exam. This general outline is applicable to either Bruker TopSpin or Varian platforms. You will be allowed up to 30 minutes to complete the following tasks. You may use any training handouts and notes you have. Standard samples will be used for the checkout and will be provided by PINMRF. PINMRF staff may, at their discretion, observe you performing all or part of the checkout exam.

Test Sample - 10% Ethylbenzene in CDCl$_3$ (other samples might be used)

- login to computer, start NMR software program, open lock/shim display
- remove CDCl$_3$ standard, insert new sample, lock and shim
- create/read new dataset/experiment
- run ^{1}H spectrum, process, integrate and plot
- set up/read new dataset/experiment for ^{13}C spectrum
- run ^{13}C spectrum, process and plot

Standard Sample - CDCl$_3$ (w/CHCl$_3$ & TMS)

- remove test sample and replace with CDCl$_3$ standard sample, lock and shim
- initialize spectrometer to ^{1}H
- shut down NMR software program, and logout from UNIX/Linux

NMR Sample Preparation Tips

The following suggestions will help you prepare good samples for NMR analysis:

1. Sample Tube. Only good-quality NMR sample tubes such as Wilmad 527-PP or other vendor's equivalents should be used. The 500 MHz spectrometers and higher require the use of a 528-PP tube or better. Appropriate NMR tubes are available in the Chemistry and RHPH storerooms.

2. Sample Volume. For conventional samples in organic solvents the best results will be obtained using a sample volume of 0.75 - 0.8 mL. Smaller volumes can be used but will require much more shimming. If you must use a smaller volume make sure it is centered about the coil centerline marked on the sample depth gauges.

3. Sample Quality. When making up the sample, place the sample in a small vial and add the solvent to the vial. Make up a filter using a small piece of Kimwipe inserted into a Pasteur pipette. Put the NMR tube in a holder and insert the filter-containing pipette into the NMR tube. After dissolution, draw the solution up with another pipette (not the one with the filter), and then introduce the solution into the NMR tube through the filter-containing pipette.

4. Sample Solvents. Solvents should be kept dry and free from impurities. DMSO in particular can pick up large amounts of water if a bottle is left open, which can lead to a large peak in the NMR spectrum. Molecular sieves can be used to dry solvents; however, these create particulates which must be removed using the filtering method described above.

5. Sample Referencing. For conventional ^{1}H and ^{13}C NMR, our NMR equipment does not need TMS or any other reference materials in the NMR tube. We ask that these not be added to the sample unless you are specifically requested to do so. If you must include TMS use a very low concentration.

<u>Access to the Facility Laboratories</u>

Normally the laboratories are open during business hours. If you need access at other times you can obtain keys to the labs as described below:

1. Keys for PINMRF laboratories in WTHR and BRWN. Each NMR approved research group will be issued keys on paddles according to the number of currently trained operators. There will be one key paddle issued per 8 trained users in the group. **These key paddles are issued to the faculty member.** To obtain a group key paddle, please contact Donna Bertram (dbertram@purdue.edu) in 361 WTHR.   Please note that the current Chemistry department protocol does not allow the issuance of building keys for BRWN and/or WTHR to individuals who are not members of the department.

2. Keys for the laboratory in B055 DRUG. The DRUG NMR lab does not require a key but the DRUG building requires your Purdue ID card to be coded to allow after-hours access. An approved NMR user may obtain access to DRUG by contacting Dr. Harwood who will forward your request to the DRUG building deputy Mr. Stu Michael (michaesd@purdue.edu).

3. Keys for the laboratory in G43 RHPH. An approved NMR user may obtain a key to G43 RHPH by completing a RHPH building key request form. Key request forms can be picked up in 130 RHPH. Please note that keys are NOT issued to undergraduates.

4. Temporary NMR keys for loan. Any approved user may check out a key for temporary use from Donna Bertram (dbertram@purdue.edu) in 361 WTHR.

PINMRF RULES

You should know and adhere to the following rules in order to maintain user privileges on the Facilty spectrometers. Failure to adhere to these rules will result in the suspension of your NMR privileges until you have completed training and checkout again.

1. Sign into the iLabs kiosk and start a session before attempting to log in to the spectrometer computer. If there is a problem with the spectrometer, indicate it in the log book (you will <u>not</u> be charged for this entry!), put an "Out of Service" sign on the spectrometer computer keyboard, and contact PINMRF staff.

2. **ABSOLUTELY NO FOOD OR DRINKS** are allowed in the NMR room, or near any other computers or spectrometers.

3. **NEVER** attempt to use a spectrometer for which you are not trained.

4. Some NMR experiments require additional hardware training beyond the standard training required for checkout. Examples of such experiments include low-temperature VT, experiments involving nuclei other than ^{1}H, ^{19}F, ^{31}P and ^{13}C, and 2D and 3D experiments not discussed in the standard PINMRF training materials. Please contact PINMRF staff for assistance with such experiments.

5. Small metal objects (e.g. loose staples, staplers, metal pens, and paper clips) **MUST NOT** be brought into the NMR spectrometer room.

6. Credit cards, magnetic data storage tapes, diskettes, and analog watches should not be brought near the magnet because loss of data or damage to them will occur. The safe region is indicated by the yellow chain - do not bring these items within the zone formed by the chain and back of the spectrometer.

7. Observe the sign-up rules given at the training session and posted on the scheduler pages.

Warnings

Some magnets are fitted with a pneumatic anti-vibrational platform. The magnet can be easily damaged while the anti-vibration system is engaged.

NEVER lean against the magnet or the anti-vibration system.

NEVER hold or pull any part of the magnet or the anti-vibration system.

NEVER stand on the anti-vibration system while it is engaged.

Safety Procedures

DO NOT allow persons with pacemakers or metallic implants WITHIN THE MAGNET'S 5 GAUSS FIELD LINE.

DO NOT take metal objects (keys, credit cards, metal chairs, tools, etc.) WITHIN THE MAGNET'S 5 GAUSS FIELD LINE.

DO NOT allow unauthorized persons to approach the magnets.

OBEY ALL the safety notices posted in the Facility and especially those around the magnets.

In Case of a Magnet Quench

When a superconducting magnet quenches all of the cryogens in the magnet will be rapidly boiled off. This will generate large amounts of He and N_2 gas, and these cold gases will condense the water in the air and will generate a large cloud of vapor. In the event of a magnet quench, **PLEASE IMMEDIATELY LEAVE THE ROOM IN A CALM AND ORDERLY FASHION**. Do not waste time attempting to retrieve your NMR sample! If the room quickly fills with vapor kneel down and crawl along the floor. The oxygen level will be higher down low and visibility will be better. Notify PINMRF staff as soon as possible. Please note that a quench is an extremely rare occurrence, but in the extremely unlikely event that one occurs we do not want anyone to panic.

RULES FOR RESERVATION AND USE OF
WALK-ON TIME ON:

Varian 300s (365 & 369 WTHR)
AV-III-HD-400 (369 WTHR)
AV-III-HD-500 (B055 DRUG - weekdays 8:00 a.m. - 6:00 p.m. only)
ARX300 (G43 RHPH)

The rules for the use of walk-on NMR time on these instruments are as follows:

1. Each user must reserve his/her own time. No one may reserve time for another person.

2. Please do not sign up for time unless you are sure you will use it. If you cannot use time you have reserved, please remove your name from the time slot as soon as possible so someone else may use it. People not using time they reserve may lose access to the instrument.

3. Times at which sign-up may begin are clearly indicated on the sign-up sheets and must be followed.

4. Limits on reservation of time (per person per machine per indicated time period) are as follows:
   a) 8 AM to 6 PM – reservations not currently allowed;
   b) 6 PM to 11 PM - one reservation per day of length 15 to 60 minutes;
   c) 11 PM to 8 AM - one reservation per week of length 1 to 10 hours;
   d) Persons more than five minutes late for a reserved time block forfeit that block if another user arrives before them; forfeited time still counts against the allotment totals given above.
   e) **IF THE MACHINE IS UNRESERVED AND NOT IN USE AT THE BEGINNING OF A TIME BLOCK, THE ABOVE LIMITS MAY BE SURPASSED UNTIL ANOTHER USER WHO HAS NOT EXHAUSTED THEIR TIME ALLOTMENT FOR THE DAY WISHES TO USE THE INSTRUMENT**.

5. Note that, on days scheduled as "maintenance/development," the instruments are available for use but are subject to the rules posted for maintenance and development days.

6. Please try to make reservations between 6 PM and 8 AM (evening/nighttime blocks) contiguous with those of other users. Please try not to fragment the schedule to the point where there are a lot of small blocks open but no one-hour or larger blocks for the people who really need them.

7. If you are leaving a sample running unattended, please leave a readily-observed note at the spectrometer with your contact information (e.g., lab phone number, cellular phone number, office/lab location) so that you can easily be reached if necessary.

8. Advance reservations of time for training and other special situations (such as spectrometer usage in support of teaching laboratories) may be made only by PINMRF Staff. Exceptions to these rules are available for special situations by permission of PINMRF at least two business days in advance of the time needed. These reservations will show up as red time blocks on the iLabs scheduling software pages.

9. Non-approved reservations violating these rules are invalid and should be brought to the attention of PINMRF staff.

Guide to Starting, Extending and Finishing a Spectrometer Session using the iLab Software

NOTE: The following assumes you have already set up your iLab account and had it approved by your business office. If you have not done this, go to https://purdue.ilabsolutions.com/service_center/show_external/4067 and click on the blue "sign-up" button in the upper left-hand corner of the page. Follow the instructions in the pop-up window.

Starting your iLab session

1. Using a web browser on either a laboratory kiosk, your own computer, or your smart device, navigate to the PINMRF iLab kiosk webpage:

https://purdue.ilabsolutions.com/service_centers/4067/equipment_kiosk/dashboard

2. Log in to iLab using your Purdue or iLab credentials.

3. Use the menus under the "Equipment by category" heading to select the spectrometer you wish to use.

4. Use the green "Create Session" button to initiate your session. Select the time period to match the estimated duration of your session.

5. Click the green "Create Session" button.

6. When the session menu comes up, select the account you want to use for billing using the selection tool. If you have access to only one account you may skip this step.

7. Click the green "Start" button to begin. This will allow the spectrometer to be used.

8. If you are using a kiosk, click the "Log out & use equipment" button to log out of the session so that another user may have access to the kiosk. If you are using your own computer or smart device you may skip this step.

NOTE: if you are using a PINMRF Bruker AV-III spectrometer, you may access iLab to start your session after logging in to the spectrometer host computer. Simply start the Firefox browser on the host computer and follow the instructions steps 1 – 7, above. You must start your iLab session before attempting to start TopSpin.

Extending your iLab session

1. If you need more time for your current active session, click the "Extend" button on the session page. In the pop-up window which follows, select the amount of time you need and click the green "Extend" button.

Finishing your iLab session

1. If you are using a kiosk and have logged out as described in step 8, above, log back in to iLab.
2. Click the "Finish" button on your session page. Confirm that you want to finish your session by clicking on the green "Finish session" button in the pop-up window.
3. You may then log out of iLab.