



Guidelines for NMR Sample Submission

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NMR spectra can be obtained in two different ways: (a) by registering on-line as a NMR user at <http://nmr.chem.ualberta.ca> and measuring your own spectra, or (b) by submitting samples to the NMR support group. There are 8 spectrometers in the Department: five with full general access (i300, i400, m400, ibd5, u500) and three with limited access (s400, i600, v700). Three work stations (d500, ibdw, d601) are also available for data processing, analysis and plotting. A description of the capabilities of each instrument can be obtained from the NMR web site (link: Spectrometers Overview).

In general, samples requiring any of the following should be submitted to the NMR staff:

- **variable temperature** experiments
- **kinetic studies** requiring frequent access to the spectrometer
- nuclei such as ^{11}B , ^{15}N , ^{27}Al , ^{45}Sc , ^{63}Cu , ^{171}Yb , ^{195}Pt
- **shift reagent studies**

NMR-related forms are available in WB-13 as well as at <http://nmr.chem.ualberta.ca> in PDF format (link: NMR forms).

For samples to be submitted to the NMR support group the following policies apply:

1. Reservations are accepted to a **maximum of two weeks in advance** and only after consultation with staff.
A reservation for a particular time does not guarantee that the sample is measured exactly at that time. Efficient spectrometer use sometimes requires that similar samples are grouped together, e.g. low temperature samples.
2. Samples should be **submitted one day in advance** in WB-13 together with a completed *Request for NMR Service* form. For unstable sample it is imperative to make arrangements with staff ahead of time to accommodate you and indicate where the sample can be found, e.g. fridge or freezer in WB-13.
3. If your **sample is not ready**, PLEASE **cancel** your reservation. This courtesy applies to reservations with staff and on-line. Failing to provide a sample altogether or more than 5 minutes delay for reserved time, puts your sample at the end of the queue for the day.
4. Individuals submitting samples are **responsible to pick up their samples and vials** together with the spectra (WB-13). NMR staff are not responsible for samples after 48 hours. Failing to remove old samples/vials repeatedly can result in refusal of service in the future.
5. Try to submit a high quality sample (you can expect much better results) by paying attention to the following:
 - free of particles (filter the solution through cotton)
 - correct sample height (55 mm, equivalent to 0.7 mL)
 - appropriate concentration: not too concentrated for ^1H and ^{19}F (< 100 mM), not too dilute for ^{13}C and ^{31}P (>10 mM)

$$\text{for 0.7 mL samples: } \text{conc. [mM]} = \frac{\text{weight in mg}}{(\text{mol. weight}) \times 0.7} \times 1000$$

- fill in the service request form properly and legibly and attach a proper label to the NMR tube (see instructions on the form); do not write directly onto the NMR tube.

Additional information:

- a) **Data storage.** NMR data obtained by NMR staff and data acquired by individuals in the *gennmr* account are **deleted automatically after 180 days**. NMR data of research groups with login accounts are not subject to automatic data removal, hence it is the group's responsibility to manage their disk space and to ensure that disk usage stays below the assigned quota.
- b) All NMR data are stored on a data server and can be processed/plotted on any NMR spectrometer or data station immediately after they are saved.
- c) PC, Mac and UNIX/LINUX computers in the Department can do remote data processing on NMR data stations after they have passed a safety check and are approved by the IT and NMR supervisors.
- d) All data stations are equipped with a **CD/DVD burner** and USB connections for memory sticks to facilitate data archiving. Furthermore, data can be transferred to a computer of your choice through the network. Note that security restrictions will not allow computers to connect without explicit approval by the IT and NMR supervisors.
- e) The **s400** spectrometer is used by NMR staff during regular working hours. Persons wishing to be trained individually in the operation of the s400 spectrometer need written approval from their research director. Variable temperature experiments are only allowed with the explicit agreement of NMR staff and the student/PDF must remain in the lab during the experiment(s).
- f) To ensure fair and equal access to **s400** overnight time, each research group is limited to one s400 overnight reservation per week. However, if the overnight time is not reserved by 13:00, it can be taken by anybody irrespective of previous overnight use.
- g) For the **walk-up spectrometers** you should consult with experienced NMR users in your research group. In general, there are no individual check-outs, however if you feel uneasy with the use of a spectrometer and wish to obtain training, consult the NMR staff to organize a training session.
- h) Beginners are strongly encouraged to use the EZ NMR system to reduce the slope of the learning curve. It covers a large variety of 1D and 2D experiments for 1H, 13C and 31P. Instructions on how to use it are available on the NMR web pages (link: Course Material).
- i) The on-line reservation software enforces the booking rules automatically. They can be reviewed on the web by clicking the spectrometer symbol on the Spectrometer Reservations and Schedules page.
- j) If you are new to the Department and need a key to the NMR labs, print out the relevant form, fill it in, **understand what you are signing for**, then have your supervisor sign the sheet and finally obtain the signature of the NMR supervisor. The key will then be given to you in the E3-44 general office.