



## Using Spectrometers in NMRFAM

If you have a dilute (1 mg or less for a small molecule) sample, running your experiments in NMRFAM is recommended. All NMRFAM's spectrometers are equipped with cold probes, which provide 3 to 4 times of better sensitivity compared to a regular room temperature probe.

### 1) NMRFAM's accounts

Please contact Lai Bergeman (NMRFAM administrator) for accounts. Each user will need 3 accounts: NMRFAM account for spectrometer login, NMRhub account for data access and iLab account for NMR time reservation and billing.

### 2) Spectrometers in NMRFAM

Most of the Pharmacy School users often use the following three spectrometers in NMRFAM.

1. Kerry, Bruker 500 MHz, 5 mm TXO probe, 5 X 96 SampleJet sample changer
2. Dexter, Bruker 600 MHz, 5 mm QXI (1H/31P/13C/15N or 1H/19F/13C/15N) probe
3. Kurgan, Bruker 600 MHz, 5 mm TXI probe or 1.7 mm TXI probe, 5 X 96 sample changer

Kerry is the best choice for 1D carbon, and it is also the least busy instrument. Usually it is not difficult to book NMR time for the next day on Kerry, while on other spectrometers, you may have to make the reservation several days or longer in advance. Dexter and Kurgan (with 5 mm TXI probe) are better for multidimensional experiments (such as HSQC and HMBC). If you have very dilute sample, Kurgan with the 1.7 mm probe is the best.

### 3) Running experiments in NMRFAM

Although both Kerry and Kurgan are equipped with the SampleJet autosampler, NMRFAM does not use the software IconNMR (which we use on our AV400) for fully automatic data acquisition. Users do not submit the experiments to the queue. Instead, NMRFAM uses TopSpin for data acquisition, and most of the users run their samples manually. As a result, users have to reserve their NMR time online in advance to run their samples. NMRFAM will provide detailed training before users can use the spectrometers. The following briefly describes the procedures on how to run experiments in NMRFAM.

1. Log into your NMRFAM account. For each user, all spectrometers share a single user account.
2. Launch TopSpin software.
3. Type "ej" to eject air. If there is any sample lifted up, take it away.

4. Put your sample on the top of the magnet and type “ij” to insert.
5. Lock your sample on the solvent you used.
6. Start automatic tuning.
7. Start gradient shimming.
8. Create a new experiment (dataset). You can modify any parameter (for example, changing the number of scans) in this step.
9. Type “rga” to adjust the receiver gain and type “zg” to start acquisition.
10. After the experiment done, type “ej” again to remove your sample, and type “ij” again to insert (even without any sample).
11. Close the TopSpin software and logout.

#### **4) Common experiments you can choose when creating a new dataset**

1. PROTON (default 1D <sup>1</sup>H experiment)
2. C13CPD (default 1D <sup>13</sup>C experiment)
3. COSYGPSW (magnitude mode COSY with gradient)
4. COSYGPDPHSW (phase sensitive DQF-COSY)
5. MLEVPHSW (phase sensitive TOCSY)
6. HSQCETGPSISP\_ADIA (HSQC with adiabatic pulses, no <sup>13</sup>C multiplicity edited)
7. HSQCEDETGPSISP\_ADIA (HSQC with edited multiplicity and adiabatic pulses)
8. HMBCGP (1H-<sup>13</sup>C magnitude mode HMBC)
9. NOESYPHSW (phase sensitive NOESY)
10. HSQCETGP\_15N (1H-<sup>15</sup>N HSQC)
11. HMBCGP\_15N (1H-<sup>15</sup>N HMBC)

#### **5) Running multiple samples**

Two of the NMRFAM’s spectrometers, Kerry and Kurgan, are equipped with the SampleJet autosampler. However, NMRFAM uses a home-made program NMRbot (instead of IconNMR on our AV400) to run multiple samples automatically. To do that, you have to create a new dataset for the first sample, modify the parameters, then save it as the parameter file for all your samples. If you like to run multiple experiments for each sample, you need to create a parameter file for each experiment. Please note that SampleJet uses a special 5 mm NMR tube. You can purchase these NMR tubes in advance or directly from NMRFAM.

#### **6) Other capabilities in NMRFAM**

For solution state NMR, NMRFAM only provides <sup>1</sup>H, <sup>13</sup>C, <sup>15</sup>N, <sup>31</sup>P and <sup>19</sup>F experiments. For other nuclei (such as <sup>11</sup>B), NMRFAM has even better capabilities. You can run solid state NMR for almost any NMR active nucleus in NMRFAM, including quadrupolar nuclei, such as <sup>35</sup>Cl and <sup>14</sup>N. Please contact NMRFAM for details.