

NMR Sample Service in the Department of Chemistry and Biochemistry, University of Arizona

Any researcher at the University of Arizona (undergraduate, graduate student, postdoc, visiting faculty or faculty) can obtain training and be “licensed” for hands-on use of the Chemistry Department NMR instruments. If you do not wish to learn to use the instruments, you can submit your samples and we will do the NMR analysis for you (“**sample service**”). Our current charges for sample service are \$15 / hour for instrument time and \$40 / hour for operator time for researchers within the University of Arizona. Our charges for outside users are \$45 / hour for instrument time and \$120 / hour for operator time. A typical routine ^1H NMR spectrum requires about 5 minutes of instrument time and 10 minutes of operator time (\$11.25 for University researchers or \$33.75 for outside users) if samples are submitted in approved NMR tubes with deuterated solvent. Sample preparation requires more operator time, plus charges for NMR tubes and solvent. We also train outside users for hands-on use with our automated Bruker AVIII-400 instrument, which costs \$2.50 / minute of automation.

Samples submitted for routine analysis by will be run on the Bruker AVIII-400 (400 MHz) instrument. This instrument is capable of ^1H , ^{13}C , ^{19}F , ^{31}P , ^{11}B , and 2D experiments COSY, HSQC and HMBC. Samples which require higher field (500 MHz or 600 MHz) can be run for the same prices per hour, but may take longer depending on operator availability and demand on the instrument. Please arrange with Dr. Jacobsen or Dr. Dai to drop off samples at their offices, where you will be asked to fill out a sample service request form. Please indicate the amount of sample (in milligrams) and the approximate molecular weight on the request form. Requests for ^{13}C experiments (^{13}C and/or DEPT) will be run the appropriate amount of time for the indicated concentration. Routine ^{13}C or DEPT spectra require 10 mg of typical samples (MW ~ 400) to be run in a short (15 - 20 min.) time on the automated system. ^{13}C -NMR of organic samples in the 1-5 mg range can be run on the DRX-500 for longer times. We have a 3-mm ^{13}C probe on the DRX-600 for ^{13}C of organic samples down to 0.1 mg in 125 μL sample volume.

NMR data from the automated (AVIII-400) instrument will be posted immediately on our web server (aviii400.chem.arizona.edu) under the folder “sampserv”, as a zip file. Site-licensed software (MestReNova) is available to download and install on your computer for data processing. We can also process NMR data for you and send pdf files of the spectra, or meet with you to discuss the results. You will be charged operator time for the data processing, interpretation and discussion.

Available experiments include:

One Dimensional: ^1H , ^{13}C , ^{31}P , ^{19}F , ^{11}B , DEPT-90, DEPT-135 on the automated AVIII-400. Selective 1D experiments (NOE and TOCSY) and water-suppression (90% H_2O) are run on the DRX-500 or DRX-600. We can also do high temperature and low temperature NMR on the Varian Unity-300.

Two Dimensional: DQF-COSY, HSQC, and HMBC can be run on the AVIII-400. TOCSY, NOESY, and ROESY are available on the DRX-600. Biological NMR experiments (^{15}N HSQC, triple-resonance) are run on the Varian Inova-600 (cryogenic probe).

Solid-State: The Bruker AVIII-400 has a CP-MAS solid-state capability. It takes one hour to convert to solid state operation and another hour to go back to automation, so we generally reserve an entire day for solid state experiment.

Samples for routine (solution state) NMR analysis should be submitted in Norell XR-55 7" 5mm tubes, as a solution in a deuterated solvent. NMR tubes and solvents can be purchased from the NMR facility. Samples can also be submitted as a solvent-free oil or powder in a capped vial, but we will charge for the deuterated solvent, the tube and the operator time required for sample preparation. In this case you must indicate the solvent and make sure that the sample is soluble in that solvent. Unless you are a "one-time" user of the facility, it is better to purchase tubes and deuterated solvents and learn how to prepare samples. **YOU ARE RESPONSIBLE FOR PICKING UP YOUR SAMPLE AFTER ANALYSIS IS COMPLETE.** We will charge a disposal fee for any samples that are left for more than one month.

Sample volume should be about 0.7 mL, which gives a sample depth of 3.5 - 4.0 cm. Smaller volumes are very difficult to shim and lead to higher operator costs and lower resolution. The sample solution should be homogeneous with no suspended solid, droplets of water or undissolved oils. Cloudy solutions indicate poor solubility and tend to give very broad lines. A standard which is not overlapped with sample peaks should be included in the sample and indicated on the request form. Recommended standards are TMS (tetramethylsilane) for organic solvents and d₄-TSP (sodium 3-trimethylsilyl-2,2',3,3'-tetradeuteropropionate) for samples in D₂O. In D₂O any single-resonance compound (methanol, acetonitrile, dioxane) which does not overlap with sample peaks can also be used. Usually less than a drop of TMS in a 40 g bottle of CDCl₃ is more than sufficient. You can also purchase CDCl₃ with 0.05% TMS already included.

Clearly label each sample and give it a unique identifying number or name. The best label is a string tag. You can purchase these "merchandise tags" with string loops from an office supply store and wrap the string around the tube many times at the top. Tape labels cannot be used on the automated NMR instrument (Bruker AVIII-400). NMR Data will be permanently archived using your sample identification code and should be accessible at any time in the future.

If you have any questions, please contact:

Neil E. Jacobsen, 621-8146, neil@email.arizona.edu, room 119 Old Chemistry (NW corner of first floor); or Dr. Jixun Dai, 621-2308, dai@email.arizona.edu, room 125 Old Chemistry.