

NMR Sample Service in the Chemistry Department, University of Arizona

Neil E. Jacobsen, Ph. D. NMR Facility Manager

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. Our current charges for sample service are \$10 / hour for instrument time and \$40 / hour for operator time for researchers within the University of Arizona. Our charges for outside users are \$30 / hour for instrument time and \$120 / hour for operator time. A typical routine ^1H NMR spectrum requires about 20 minutes of instrument time and an equal amount of operator time (\$16.67 for University researchers or \$50 for outside users). We are experimenting with hands-on use for outside users with our Bruker AM-250 instrument (routine ^1H and ^{13}C), which costs \$75.00 / hour.

Samples submitted for routine analysis by noon Wednesday will be run on Friday mornings on the Varian Unity 300 instrument. Samples which require higher field (500 MHz or 600 MHz) or more sophisticated experiments (2D or selective 1D) can be run for the same price, but may take longer depending on operator availability and demand on the instrument. Samples should be submitted and picked up from room 118 Old Chemistry (SE corner of the first floor). This room will be open from 8 a.m. to 5 p.m. Monday-Friday except holidays, and will be locked at other times. Fill out a request form and leave the sample in the "Submitted" rack. Please indicate the amount of sample (in milligrams) and the approximate molecular weight on the request form. Requests for ^{13}C experiments (^{13}C , APT or DEPT) will be run the appropriate amount of time for the indicated concentration and no longer, even if the signal-to-noise ratio is inadequate. Requests for ^{13}C , APT or DEPT spectra require 30-40 mg of typical samples to be run in a short (30 - 90 min.) time on the Unity-300. ^{13}C -NMR of organic samples in the 3-5 mg range can be run on the DRX-500 with the direct ^{13}C ("dual") probe. 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.

Routine samples submitted by noon Wednesday should be finished by Friday afternoon unless they require long ^{13}C acquisitions. You may submit on Friday morning as long as you notify us by Wednesday noon that samples will be coming. We only reserve (one week in advance) enough time on the instrument to complete a "normal" number of samples, so unexpected increases in demand can delay service for a week or more. Completed samples can be picked up from the "Completed" rack in 118 Old Chem. and spectra can be found in the metal "In/Out" rack.

Available experiments include:

One Dimensional: ^1H , ^{13}C , ^{31}P , ^{19}F , D_2O shake to identify exchangeable (OH and NH) protons, and DEPT can all be run on the Unity-300. Selective (shaped-pulse) experiments (NOE and TOCSY) and water-suppression (90% H_2O) are run on the DRX-500 or DRX-600. More exotic nuclei (^{29}Si , ^{11}B , ^{77}Se , etc.) can be run on the Unity-300 (low sensitivity inverse probe) or the DRX-500 (10 mm tubes with direct probe). We can also do high temperature and low temperature NMR on the Unity-300.

Two Dimensional: DQF-COSY, TOCSY, NOESY, ROESY, HSQC, and HMBC are run on the DRX-500 or DRX-600. Requests for 2D experiments should be accompanied by copies of the 1D spectra. 2D data is difficult to put on paper, since many different plots at different contour levels are required. We recommend that you learn to use our 2D graphics software package ("Felix") which can be accessed from any PC on campus.

Solid-State: We currently have no solid-state capability at the University of Arizona. Right now we are trying to acquire a new or used solid-state instrument, or upgrade an existing instrument for solid-state (CP-MAS) capability.

Samples can be accepted in Wilmad 507-PP or Kontes 33-220 NMR tubes only. Samples submitted in any other tube will be transferred to a new Kontes 33-220 tube and you will be charged for the tube. 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.

Sample volume should be about 0.6 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. Use very small amounts of standard, since pulsed-FT NMR is very sensitive! 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. If samples require heating to be solubilized, please specify the temperature required.

Clearly label each sample and give it a unique identifying number or name. The best label is a paper label placed as high as possible on the tube and secured with transparent tape. You can also purchase "merchandise tags" with string loops from an office supply store and wrap the string around the tube many times at the top. Data will be saved on CD using your identifying name or number and should be accessible at any time in the future. If you need a different presentation of data (e.g., expansions, resolution enhancement, etc.), just fill out a request and attach a copy of your spectrum.

If you have any questions, please call Neil E. Jacobsen at 621-8146, email at neil@email.arizona.edu or come to his office, room 119 Old Chemistry (NW corner of first floor).

March 15, 2006

TO: NMR Sample Service and Hands-On Customers

FROM: Neil E. Jacobsen, Ph.D., NMR Facility Manager

CONCERNING: NMR Rate Increase

Our last rate increase was January, 2003 (20%). In recent years we have been losing money in the NMR facility, with estimated losses of \$13,000 for the first half of this fiscal year. This is due to a combination of aging equipment requiring more maintenance and repairs, increased benefits costs for our two graduate assistants, and lower revenues due to declining usage. As a result we have decided to institute an across-the-board increase of 66% to all of our charges in the facility. The new rate structure is as follows (all rates are per hour):

	Old Rate	New Rate
U of A hands-on use:	\$6.00	\$10.00
U of A service (instrument):	\$6.00	\$10.00
U of A service (operator):	\$24.00	\$40.00
Industry hands-on use:	\$45.00	\$75.00
Industry service (instrument):	\$18.00	\$30.00
Industry service (operator):	\$72.00	\$120.00

These rates are the same for all six instruments at all times of day and all days of the week, with one exception: the Varian Inova-600 with cryogenic probe will cost \$15.00 per hour for U of A hands-on and service use and \$45.00 per hour for Industry service. This is due to a \$13,000 per year mandatory service contract and higher overall maintenance risks for the cryogenic probe.

It is hoped that these new rates will bring the facility into the black and allow us to provide the highest standard of service and reliability. I think you will find that these rates are still quite reasonable compared to commercial NMR sample services, especially in view of our ability to provide specialized services and the convenience of a local provider. The new rates will be effective starting March 22, 2006.