

**A GUIDE TO
THE SCS NMR LABORATORY**

Dean Olson, Director

SCHOOL OF CHEMICAL SCIENCES
UNIVERSITY OF ILLINOIS
600 South Matthews, Urbana, Illinois 61801

FACILITY PERSONNEL:

Dean Olson, 244-0564; U400, UI400, U500, VXR500, UI500NB; Lab Director

Prof. Scott Denmark, Faculty Advisor

Tracie Hubert, 333-2041; Business Functions, Reslog

Jennifer Rapp, 333-0327; VNS500WB, UI300WB-solids, VNS750WB

Feng Lin, 333-6283; UI300WB-solutions; UI600; System Administrator

I. INTRODUCTION

This guide provides information about the instruments and techniques available in the NMR Laboratory (NMR Lab). Scheduling and sign-up policies are described in handout UGL016. Most of the information contained herein is of a fairly general nature. If you have any questions or problems you should consult the NMR Lab handouts addressing specific topics or consult laboratory personnel. Any new user of the NMR Lab should read **Handout #011 - New User Orientation to the NMR Lab**. For full description of instrumentation, see our webpage at <http://scs.illinois.edu/nmr/>.

TABLE I
Summary of NMR Spectrometers in the NMR LAB

NMR Instrument	¹ H Freq (MHz)	Probes*	Tube Size/Rotor Size
UI300	300 (7.05 Tesla)	¹ H/X switchable BB ⁷³ Ge- ¹⁵ N BB ¹⁷ O{X} ¹⁸³ W{X} ¹⁵ N- ³¹ P BB CPMAS (ChemMag)	5 20 mm 10 mm 20 mm 7, 4, 3.2 mm
U400	400 (9.4 Tesla)	QUAD (¹ H/ ¹⁹ F/ ¹³ C/ ³¹ P) ¹ H/X switchable BB	5 mm 5 mm
UI400	400 (9.4 Tesla)	QUAD (¹ H/ ¹⁹ F/ ¹³ C/ ³¹ P)	5 mm
U500	500 (11.75 Tesla)	QUAD (¹ H/ ¹⁹ F/ ¹³ C/ ³¹ P)	5 mm
VXR500	500 (11.75 Tesla)	QUAD (¹ H/ ¹⁹ F/ ¹³ C/ ³¹ P) PFG Z ¹³ C Nanoprobe	5 mm 70 μL
UI500NB	500 (11.75 Tesla)	¹ H{ ¹³ C/ ¹⁵ N} PFG Z ¹ H{ ³¹ P/X} PFG Z ¹ H/X switchable BB ¹⁵ N- ³¹ P BB	5 mm 5 mm 5 mm 10 mm
VNMRS500WB	500 (11.75 Tesla)	¹⁵ N- ³¹ P BB CPMAS (Doty)	5 mm
UI600	600 (14.1 Tesla)	¹ H{ ¹³ C/ ¹⁵ N}PFG X,Y,Z ¹⁵ N- ³¹ P BB AutoX (¹⁵ N- ³¹ P)	5 mm 5, 10 mm 5mm
VNS750NB (magnet is down as of 16Jan2011)	750 (17.6 Tesla)	¹ H{ ¹³ C/ ¹⁵ N}PFG X,Y,Z ¹⁵ N- ³¹ P BB ¹³ C{ ¹ H} ¹³ C{ ¹ H} ¹⁵ N- ³¹ P BB CPMAS (Doty) HXY MAS (Varian)	5 mm 10 mm 3 mm 5mm 5 mm 4 mm

* BB = Broad Band; PFG = pulsed field gradient; X, Y, Z = available gradients; CPMAS = cross polarization magic angle spinning

Table II gives a useful comparison of ^1H and ^{13}C resolution and sensitivities for the various spectrometers and probes in the NMR Lab, as well as the variable temperature range available on the various probes.

^{13}C : In general, the U500 and VXR500 give the best sensitivity for routine ^{13}C measurements on 5mm samples. For example, a sample of 15 mg of a material with molecular weight <1000 in 0.5 mL solvent requires 5-10 minutes to achieve a reasonable S/N. If you are mass limited, please read the discussion in handout UGL015 for guidance in choosing the best probe for your needs. If you are solubility limited, consider using a 10 mm probe on the appropriate spectrometer.

^1H : In general, routine ^1H spectra can be obtained on the U400, UI400, U500 and VXR500. All inverse experiments are restricted to the UI500NB, UI600, or VNS750NB. Table II serves as a guide to the most appropriate instrument for your data collection. If you have 10-15 mg of material with molecular weight <1000, you should be able to achieve a reasonable routine ^1H S/N within 1 minute on *any* of these spectrometers (time for shimming is, however, not included!).

TABLE II
Comparison of Sensitivity (Sens) for NMR Spectrometers
(Numbers in () indicate sample used, see list following Table.)

NMR Instrument, Probe	^1H Sens (1)	^{19}F Sens (2)	^{13}C Sens (3)	^{31}P Sens (4)	Variable Temperature Range
U400, QUAD 5mm switchable	~160:1 ~120:1	~150:1	~150:1 ~150:1	~110:1 ~150:1	-100 C to +100C -100 C to +100C
UI400, QUAD	~120:1	~120:1	~130:1	~90:1	-100 C to +100C
U500 , QUAD	~260:1	~270:1	~210:1	~300:1	-100 C to +100C
VXR500 , QUADG $^1\text{H}\{\text{X}\}$ switchable	~260:1 ~300:1	~280:1	~230:1 ~210:1	~200:1	-80 C to +100C -100 C to +100C
UI500NB $^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$ PFG Z $^1\text{H}\{^{31}\text{P}/\text{X}\}$ PFG Z	~750:1 ~700:1		~80:1 NA		-30 C to +80C -30 C to +50C
UI600 $^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$ X,Y,Z AutoX ^{15}N - ^{31}P BB (10mm)	~1000:1 ~370:1 NA	~475:1 NA	~125:1 ~320:1 ~1100:1	~200:1 ~550:1	-20 C to +80C -80 C to +130C -150 C to +150C
VNS750NB $^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$ PFG X,Y,Z ^{15}N - ^{31}P BB (10mm) $^{13}\text{C}\{^1\text{H}\}$ 5 mm $^{13}\text{C}\{^1\text{H}\}$ 3 mm	~1350:1		~180:1 ~1200:1 ~530:1 ~220:1	~780:1	-20 C to +80C -150 C to +150C -100 C to +100C -100 C to +100C

- (1) ^1H Sensitivity Standard: 0.1% ethylbenzene/ CDCl_3 (5mm tube)
- (2) ^{19}F Sensitivity Standard: 0.05% $\text{CF}_3\text{C}_6\text{H}_5/\text{C}_6\text{D}_6$ (5mm tube)
- (3) ^{13}C Sensitivity Standard: ASTM, 40% dioxane/ C_6D_6 (5mm tube)
- (4) ^{31}P Sensitivity Standard: 0.0485M Triphenylphosphate/ CDCl_3 (5mm tube)
- (5) ^{13}C Sensitivity Standard: ASTM, 40% dioxane/ C_6D_6 (10mm tube)

* See Handout UGL015 - ^{13}C Sensitivity for Mass Limited Samples

TABLE III
NMR Lab Instrumentation Cost Rate Summary. See the SCS NMR Website for the current rates.

Cancellation Charges for Reservations on RESLOG:		
<u>Type of Cancellation</u>	<u>Time Period</u>	<u>Charge</u>
Auto-cancel/No show	<1 hr	2 hrs instrument time
Auto-cancel/No show	>1 hr	Time reserved + 2 hrs
<u>Emergency Log-off</u>	1 hr	<u>instrument time</u>

II. DATA BACKUP

The NMR Lab does not take responsibility for backing up your data. You should backup your own data:

On the computers in your own lab. If your group has provided the NMR Lab the IP addresses of the computers in your lab, then you should be able to map your group's NMR data as network drives on these computers (see handout UVU403 – Data Backup Options). Backup should then be done on these computers either locally by individual users or centrally as a group, by whatever means that are available on these computers (e.g., hard disk, CD/DVD, USB memory stick, etc.). An automatic daily or weekly backup is recommended.

If you need help with data backup, contact Feng Lin.

III. INSTRUMENTATION

UI300WB - VARIAN UNITY INOVA: (Located in 55 NL; console upgraded in 2002 to Inova from a GE GN console.) A multinuclear solution and solids FT-NMR instrument. Available probes for solutions: ^{15}N - ^{31}P (10 mm), ^{73}Ge - ^{17}O (20 mm), $^{17}\text{O}\{\text{X}\}$ (10 mm), $^{183}\text{W}\{\text{X}\}$ (20 mm). Available probes for solids cover the range ^{29}Si - ^{31}P (7 mm) and ^{15}N - ^{29}Si (7 mm), ^{15}N - ^{31}P (4, 3.2 mm). A broadline ^2H probe and fast digitizer allow solid state deuterium studies to be performed in the temperature range of -150 to 200 °C. Deuterium lock and unlocked mode of operation. Homonuclear and heteronuclear proton and X nucleus decoupling for solutions. Variable temperature with a range covering -100 to +100 °C. A variety of software is available for both one and two dimensional NMR experiments.

U400 - VARIAN UNITY INOVA 400: (Located in 150 RAL; console upgraded to an Inova console in August 2008.) A multinuclear solution FT-NMR instrument. Available probes: Nalorac QUAD probe (^1H , ^{19}F , ^{13}C , ^{31}P) (5 mm), Inverse Detection (5 mm), and ^1H -X broadband (^{31}P - ^{15}N) (5 mm). Deuterium lock and unlocked mode of operation. Homonuclear and heteronuclear proton decoupling. Variable temperature range varies with the probe. A variety of software is available for both one- and two-dimensional NMR experiments.

UI400 - VARIAN UNITY INOVA 400: (Located in 146 RAL) A multinuclear solution FT-NMR instrument. Available probes: Nalorac QUAD probe (^1H , ^{19}F , ^{13}C , ^{31}P) (5 mm). Deuterium lock and unlocked mode of operation. Homonuclear and heteronuclear proton decoupling. Variable temperature range varies with the probe. A variety of software is available for both one- and two-dimensional NMR experiments.

U500 - VARIAN UNITY INOVA 500: (Located in 150 RAL; console upgraded to an Inova console in March 2008) A multinuclear solution FT-NMR instrument. Available probes: Nalorac QUAD probe (^1H , ^{19}F , ^{13}C , ^{31}P) (5 mm). Deuterium lock and unlocked mode of operation. Homonuclear and heteronuclear proton decoupling. Variable temperature with a range covering -100 to +100 °C. A variety of software is available for both one- and two dimensional NMR experiments.

VXR500 - VARIAN UNITY INOVA 500: (Located in 150 RAL; console upgraded to an Inova console in October 2007) A multinuclear solution FT-NMR instrument. Available probes: Nalorac QUAD gradient probe (^1H , ^{19}F , ^{13}C , ^{31}P) (5 mm). Deuterium lock and unlocked mode of operation. Homonuclear and heteronuclear proton decoupling. Variable temperature with a range covering -80 to +100 °C. A variety of software is available for both one- and two dimensional NMR experiments.

UI500NB - VARIAN UNITY INOVA 500NB: (Located in 146 RAL) A multinuclear solution FT-NMR three channel instrument. Available probes: Varian $^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$ Z (5mm). Deuterium lock and unlocked mode of operation. Homonuclear and heteronuclear proton decoupling. Variable temperature with a range covering -50 to +80 °C. A variety of software is available for both multi-dimensional NMR experiments.

VNMRS500WB - VARIAN VNMRS500WB: (Located in 55NL; console upgraded from Infinity Plus in 2002 to a VNMRS console in August 2009.) A multinuclear solid-state FT-NMR four channel instrument. Available probes: ^{15}N - ^{31}P BB CPMAS (5 mm), solution probes. Deuterium lock and unlocked mode of operation. Homonuclear and heteronuclear proton

decoupling. Variable temperature with a range covering -100 to +100 °C, depending upon the probe. A variety of software is available for multi-dimensional NMR experiments.

UI600 - VARIAN UNITY INOVA 600: (Located in 55 NL) A multinuclear solution FT-NMR three channel instrument. Available probes: Varian $^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$ X,Y,Z (5 mm) and $^{15}\text{N}\text{-}^{31}\text{P}$ BB (10 mm) probes. Deuterium lock and unlocked mode of operation. Homonuclear and heteronuclear proton decoupling. Variable temperature with a range covering -100 to +100 °C, depending upon the probe. A variety of software is available for multi-dimensional NMR experiments.

VNS750NB - VARIAN UNITY INOVA 750 (Located in A151 CLSL; console upgraded in 2002 to allow for biomolecular solid state NMR experiments; console upgraded to VNMR5 console in October 2009): (Located in 151A CLSL) A multinuclear solution FT-NMR three channel instrument. Available probes: Varian $^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$ X,Y,Z (5 mm), $^{15}\text{N}\text{-}^{31}\text{P}$ BB (10 mm), $^{13}\text{C}\{^1\text{H}\}$ (3 mm), and $^{15}\text{N}\text{-}^{31}\text{P}$ CPMAS (5 mm) probes. Deuterium lock and unlocked mode of operation. Homonuclear and heteronuclear proton decoupling. Variable temperature with a range covering -100 to +100 °C, depending upon the probe. A variety of software is available for multi-dimensional NMR experiments.

SUNDS - SUN Data Station: (Located in 150 RAL) This is a data station based on the SUN SPARCstation Ultra 10 computer. It has the Varian VNMR software installed for data workup. Data sets may be transferred to the SPARC via Ethernet. Data may be plotted on an HP LaserJet 5SiMX for 8.5" x 11" or 11" x 17" plots.