

**A GUIDE TO  
THE NMR LABORATORY**

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## 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://www.scs.uiuc.edu/chem/molsp.html>.

**TABLE I**  
**Summary of NMR Spectrometers in the NMR LAB**

NMR Instrument	<sup>1</sup> H Freq (MHz)	Probes*	Tube Size/Rotor Size
UI300	300 (7.05 Tesla)	<sup>15</sup> N- <sup>31</sup> P BB <sup>73</sup> Ge- <sup>15</sup> N BB <sup>17</sup> O{X} <sup>183</sup> W{X} <sup>15</sup> N- <sup>31</sup> P BB Solids	5, 10 5, 10, 20 mm 5, 10 mm 5, 10, 20 mm 7, 4, 3.2 mm
U400	400 (9.4 Tesla)	QUAD ( <sup>1</sup> H/ <sup>19</sup> F/ <sup>13</sup> C/ <sup>31</sup> P) QUAD ( <sup>1</sup> H/ <sup>19</sup> F/ <sup>13</sup> C/ <sup>31</sup> P)	5 mm 5 mm
U500	500 (11.75 Tesla)	QUAD ( <sup>1</sup> H/ <sup>19</sup> F/ <sup>13</sup> C/ <sup>31</sup> P)	5 mm
VXR500	500 (11.75 Tesla)	QUAD ( <sup>1</sup> H/ <sup>19</sup> F/ <sup>13</sup> C/ <sup>31</sup> P) PFG Z <sup>13</sup> C Nanoprobe	5 mm 70 μL
UI500NB	500 (11.75 Tesla)	<sup>1</sup> H{ <sup>13</sup> C/ <sup>15</sup> N} PFG Z <sup>1</sup> H{ <sup>31</sup> P/X} PFG Z <sup>1</sup> H{X} switchable <sup>15</sup> N- <sup>31</sup> P BB	5 mm 5 mm 5 mm 5, 10 mm
IP500WB	500 (11.75 Tesla)	<sup>15</sup> N- <sup>31</sup> P BB CPMAS Solids	7 mm
UI600	600 (14.1 Tesla)	<sup>1</sup> H{ <sup>13</sup> C/ <sup>15</sup> N}PFG X,Y,Z <sup>15</sup> N- <sup>31</sup> P BB AutoX ( <sup>15</sup> N- <sup>31</sup> P)	5 mm 5, 10 mm 5mm
Keck750	750 (17.6 Tesla)	<sup>1</sup> H{ <sup>13</sup> C/ <sup>15</sup> N}PFG X,Y,Z <sup>15</sup> N- <sup>31</sup> P BB <sup>13</sup> C{ <sup>1</sup> H} <sup>13</sup> C{ <sup>1</sup> H} <sup>15</sup> N- <sup>31</sup> P BB CPMAS HXY MAS	5 mm 5, 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  $^1\text{H}$  and  $^{13}\text{C}$  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.

$^{13}\text{C}$ : In general, the U500 and VXR500 give the best sensitivity for routine  $^{13}\text{C}$  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.

$^1\text{H}$ : In general, routine  $^1\text{H}$  spectra can be obtained on the U400, U500 and VXR500. All inverse experiments are restricted to the UI500NB, UI600, or Keck750. 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 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	$^1\text{H}$ Sens (1)	$^{19}\text{F}$ Sens (2)	$^{13}\text{C}$ Sens (3)	$^{31}\text{P}$ Sens (4)	Variable Temperature Range
U400 QUAD	~160:1	~150:1	~150:1	~110:1	-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 $^{15}\text{N}$ - $^{31}\text{P}$ 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
Keck750 $^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$ PFG X,Y,Z $^{15}\text{N}$ - $^{31}\text{P}$ 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)  $^1\text{H}$  Sensitivity Standard: 0.1% ethylbenzene/ $\text{CDCl}_3$  (5mm tube)
- (2)  $^{19}\text{F}$  Sensitivity Standard: 0.05%  $\text{CF}_3\text{C}_6\text{H}_5/\text{C}_6\text{D}_6$  (5mm tube)
- (3)  $^{13}\text{C}$  Sensitivity Standard: ASTM, 40% dioxane/ $\text{C}_6\text{D}_6$  (5mm tube)
- (4)  $^{31}\text{P}$  Sensitivity Standard: 0.0485M Triphenylphosphate/  $\text{CDCl}_3$  (5mm tube)
- (5)  $^{13}\text{C}$  Sensitivity Standard: ASTM, 40% dioxane/ $\text{C}_6\text{D}_6$  (10mm tube)

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

**TABLE III**  
**NMR Lab Instrumentation Cost Rate Summary \***

UI300WB, UI500NB, I+500WB	\$10.00
U400, U500, VXR500	\$9.00
UI600	\$12.50 or \$192/day
Keck750	\$15.00 or \$192/day
SUNDS1	\$3.31
PCDS1	\$1.00
Other	\$1.00

\* These rates apply only to UIUC faculty and staff.

<b>Cancellation Charges for Reservations on RESLOG:</b>		
Type of Cancellation	Time Period	Charge
Auto-cancel/No show	<1 hr	2 hrs instrument time
Auto-cancel/No show	>1 hr	Time reserved + 2 hrs
Auto-cancel/Late show	All	Actual time used + 1 hr
Auto-cancel/Leftover time	All	instrument time (additional charge for unused reserved time will be at Lab staff discretion)
<u>Emergency Log-off</u>	1 hr	instrument time

## **II. DATA BACKUP**

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

- 1. 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 instructions below). 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, etc.). An automatic daily or weekly backup is recommended.
- 2. On PCDS1 in 146 RAL.** For users who belong to a group or groups who can't/won't map their data on their own PC's, you will be grouped into the "Orphans" directory, which will be mapped and accessible only on the PCDS1 in 146 RAL. Backup options there are Zip disks (100MB and 250MB capacity), Optical disks (128MB and 230MB capacity), CD's, and USB flash drives.

### III. INSTRUMENTATION

**VARIAN UNITY INOVA UI300:** (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:  $^{15}\text{N}$ - $^{31}\text{P}$  (10 mm),  $^{73}\text{Ge}$ - $^{17}\text{O}$  (20 mm),  $^{17}\text{O}\{\text{X}\}$  (10 mm),  $^{183}\text{W}\{\text{X}\}$  (20 mm). Available probes for solids cover the range  $^{29}\text{Si}$ - $^{31}\text{P}$  (7 mm) and  $^{15}\text{N}$ - $^{29}\text{Si}$  (7 mm),  $^{15}\text{N}$ - $^{31}\text{P}$  (4, 3.2 mm). A broadline  $^2\text{H}$  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.

**VARIAN UNITY 400 (U400):** (Located in 150 RAL) A multinuclear solution FT-NMR instrument. Available probes: Nalorac QUAD probe ( $^1\text{H}$ ,  $^{19}\text{F}$ ,  $^{13}\text{C}$ ,  $^{31}\text{P}$ ) (5 mm), Inverse Detection (5 mm), and  $^1\text{H}$ -X broadband ( $^{31}\text{P}$ - $^{15}\text{N}$ ) (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.

**VARIAN UNITY INOVA 500 (U500):** (Located in 150 RAL; console upgraded to an Inova console in March 2008) A multinuclear solution FT-NMR instrument. Available probes: Nalorac QUAD probe ( $^1\text{H}$ ,  $^{19}\text{F}$ ,  $^{13}\text{C}$ ,  $^{31}\text{P}$ ) (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.

**VARIAN UNITY INOVA 500 (VXR500):** (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 ( $^1\text{H}$ ,  $^{19}\text{F}$ ,  $^{13}\text{C}$ ,  $^{31}\text{P}$ ) (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.

**VARIAN UNITY INOVA 500NB (UI500NB):** (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.

**VARIAN INFINITY PLUS 500WB (IP500WB):** (Located in 55NL; console upgraded from Inova console to Infinity Plus in 2002) A multinuclear solid-state FT-NMR four channel instrument. Available probes:  $^{15}\text{N}$ - $^{31}\text{P}$  BB CPMAS (7 mm). 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.

**VARIAN UNITY INOVA 600 (UI600):** (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  $^{15}\text{N}$ - $^{31}\text{P}$  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.

**VARIAN UNITY INOVA 750 (Keck750; console upgraded in 2002 to allow for biomolecular solid state NMR experiments):** (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),  $^{15}\text{N}$ - $^{31}\text{P}$  BB (10 mm),  $^{13}\text{C}\{^1\text{H}\}$  (3 mm), and  $^{15}\text{N}$ - $^{31}\text{P}$  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.

**SUN Data Station (SUNDS):** (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.

**PC Data Station (PCDS1):** (Located in 150 RAL) This is a data station based on a PC with Windows XP operating system. See Section II for information on accessible directories. Supported data devices are 3-1/2" floppies, 3-1/2" optical disks (128MB or 230MB), and 100MB Zip disks and USB memory keys. The Acorn NMR NUTS NMR processing program is supported.