

### UC Chemistry Nuclear Magnetic Resonance Facility



Three NMR instruments available to UC faculty and students as well as universities and industry in the Cincinnati area:

NEO400: Rm 123A/Crosley Walk-on, <sup>1</sup>H; <sup>19</sup>F; <sup>31</sup>P-<sup>15</sup>N, 2Ds AV400: Rm 108/Crosley Walk-on, <sup>1</sup>H; <sup>19</sup>F; <sup>31</sup>P-<sup>15</sup>N DMX500: Rm 123B/Crosley Triple resonance, longer experiments, non-routine Alex Greenwood Office: Rm 123C/Crosley; Phone: 513-556-9211; Email: greenwa2@ucmail.uc.edu For user training, technical assistance, NMR questions and discussions.





### NMR lab services:

Walk-up NMR spectrometers

 Two 400 MHz spectrometers are available 24/7 for routine spectroscopy to trained users

#### Non-Routine experiments

• Instruments can be reserved for non-routine experiments requiring substantial setup/calibration, temperature regulation, or long run times

Consultation

- Experiment design/planning
- Data interpretation/troubleshooting
- Structure/stereochemistry determination





### Bruker NEO 400 MHz Spectrometer:

Z-Grad BBFO ATM probe: <sup>1</sup>H/<sup>19</sup>F-<sup>15</sup>N Variable temperature capability Automatic sample changer <sup>1</sup>H, <sup>15</sup>N-<sup>19</sup>F 1D and multi-nuclear NMR in one and two dimensions

- State-of the art console, probe and software
- Walk-up instrument, 24/7 availability
- Submit experiment and leave-- data is collected automatically and accessed remotely
- Software: Topspin 4 running ICON-NMR







### Bruker AV 400 MHz Spectrometer:

Z-Grad BBFO ATM probe: <sup>1</sup>H/<sup>19</sup>F-<sup>15</sup>N Variable temperature capability Automatic sample changer <sup>1</sup>H, <sup>15</sup>N-<sup>19</sup>F 1D NMR

- Walk-up instrument, 24/7 availability
- Submit experiment and leave-- data is collected automatically and accessed remotely
- Software: Topspin 2 running ICON-NMR







### Bruker DMX 500 MHz Spectrometer:

Z-Grad <sup>1</sup>H/<sup>13</sup>C/<sup>15</sup>N TXI triple resonance probe Z-Grad BBI probe: <sup>1</sup>H; <sup>31</sup>P-<sup>15</sup>N Variable temperature capability 1H, 13C and multi-nuclear NMR in one, two and three dimensions







- Higher field but older hardware
- Software: XWIN-NMR 3.5
- To operate independently, first get trained on the AV400 and Topspin on the NEO400





# Preparing your NMR sample

- Use a deuterated solvent (needed for lock)
- Use at least 600 µl (4 cm) for good shimming/linewidths
- Use tubes rated for 400 MHz or 500 MHz (for good shimming/linewidths)
- Mark tubes well and use your lab's designated cap color.
- Tubes must not be scratched or broken!
- Tubes should not be dried in ovens hotter than 100 C!







# Preparing your NMR sample

- Solution should be free of particulate- insoluble material will not give signal but **will** disrupt shimming!
- Use appropriate concentration of material!
  - For <sup>1</sup>H 1D: **2 mM** or ~**0.25 mg** gives a SNR of 100 in 16 scans
  - For <sup>13</sup>C 1D: **35 mM** or ~6 mg for SNR of 10 at 1024 scans, or **200 mM** or ~25 mg for SNR of 10 at 32 scans

(masses assume molecular mass of 200 Da)







# Submitting your NMR sample

- 1) Put tube in spinner
- 2) Clean tube and spinner with kimwipe
- 3) Position tube with depth gaugemake sure spinner is flush with top
- Small sample volumes should be centered in coil by bringing tube back up a bit



AVANCE Beginners Guide, Bruker





# Submitting your sample in the autosamplers

- 5) Identify the next available position in the autosampler and insert your sample
- 6) Define your experiment in that slot and press "submit"

### Autosampler status light





#### Department of Chemistry NMR Facility



### **ICON-NMR** Interface

	•																
der Type	Status	Disk	Name					No.	Solve	ent E	xperiment	Par	Title / Orig	Pri	Time	User	
ter 1	Finished	i															
the	Finished	/home/nmr1	20201007-s1	L.				1	CDC13	PF	NOTON	84	Sun		00:01:29	hangq	
ter 1	Finished	1															
the	Finished	/home/nmr1	20201007-s2	2				1	CDC13	PF	ROTON	84	Sun		00:01:29	hangq	
ter 1	Finished	i															
er	Finished	/home/nmr1	CHY174A					1	CDC13	PF	ROTON	≡.	Guan	i 🗐 🦲	00:01:29	yueci	
ter 1	Finished	1										_					
the	Finished	i /home/nmr1	CHY174B					1	CDC13	PF	ROTON	84	Guan	III 🤒	00:01:29	yueci	
1 1	Running																
er	Running	/home/nmr1	wy-1007-DTs	8	curr	ently rur	nning sample	1	CDC13	F1	L9_BBOF	<b>14</b>	Liu	III	00:00:50	yanwo	
ter 1	Queued																
er	Queued	/home/nmr1	10-7-ph-BOH	12-S-1h		queued	1	1	CDC13	PF	ROTON	84	Sun		00:01:29	tangjh	
ter 1	Queued					940404											
lec	Queued	/home/nmr1	10-7-Biph-B	30H2-S-:	1h			1	CDC13	PF	NOTON	84	Sun		00:01:29	tangjh	
er 1	Finished	1			nex	t availab	le slot		100000000	a	na secondo a		12.10				
er	Finished	/home/nmr1	10-7-CHO-ph	n-BOH2-1	5-1h			1	CDC13	PF	NOTON	84	Sun		00:01:29	tangjh	
1	Finished	1							120000	12.2		-					
lee .	Finished	/home/nmr1	TJM10072020	J-HPMAK	Q			1	DMSO	PE	RUTUN	Ξŵ	Ayres		00:01:29	mckenztj	
0 1	Finished	1															
Submit	Canc	el <b>I</b> AI <u>E</u> di	it 🕼	Delete	e <u>A</u> d	d 1	Copy 1										() Chan User
eding Exper	iments																
Date	Holder Na	m.Q.		No	Evnanimer	t Load	ATM Rotation I	ock Shim	Aca	Proc	lleen	In	iek   Titla	Onig Re	marke		
2020-10-07		1007 07-	ourror	the ru	nning c	omolo			1100	1							
12:12:54	5 wy	-1007-01s	currer	iuy iu	nning s	ample					yanwo	/	nome/nmri Group I	.10			
2020-10-07	4 CH	Y174B	-	1	PROTON	1	/ .	1 1	,			122	have found from a				
2020-10-07									~	~	yueci	- 1	nome/nmr1 Group	iuan	100 C		
12:01:33						•			~	~	yueci	~	none/nmri Group	iuan			
2020-10-07	3 CH	Y174A		1	PROTON	~	· ·		~	`	yueci yueci	/	home/nmr1 Group	iuan iuan			
2020-10-07	3 CH 2 20	Y174A 201007-s2		1 Finis	PROTON	v vnerimer	v v		, ,	, , ,	yueci yueci hango	л л	home/nmr1 Group	iuan iuan iun	Cu	irrently	
11:55:42	3 CH 2 20	Y174A 201007-s2	-	1 Fini:	PROTON shed ex	v v xperimer	v v		\$ \$	> > >	yueci yueci hangq	л л л	home/nmr1 Group   home/nmr1 Group	iuan iuan iun	Cu	urrently nning sample	
11:55:42 2020-10-07 11:50:17	3 CH 2 20 1 20	Y174A 201007-s2 201007-s1	-	1 Fini: 1	PROTON shed ex PROTON	xperimer	v v		~ ~ ~ ~	, , , , ,	yueci yueci hangq hangq	л л л	home/nmr1 Group home/nmr1 Group home/nmr1 Group	iuan Iuan Sun Sun	Cu	urrently nning sample	
11:55:42 2020-10-07 11:50:17 2020-10-07	3 CH 2 20 1 20	Y174A 201007-s2 201007-s1 M10072020-HPMAK7	F	1 Fini: 1	PROTON shed ex PROTON PROTON	xperimer	v v		* * * * *	* * * * *	yueci yueci hangq hangq mckenzti		home/nmr1 Group home/nmr1 Grou	iuan Sun Sun	Cu	urrently nning sample	
11:55:42 2020-10-07 11:50:17 2020-10-07 11:45:48	3 CH 2 20 1 20 16 TJ	Y174A 201007-s2 201007-s1 M10072020-HPMAK7	_	1 Fini: 1	PROTON shed ex PROTON PROTON	kperimer	v o		* * * * *	* * * * * *	yueci yueci hangq hangq mckenztj	л л л л	home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group	uan Guan Gun Gyres	Curu	irrently nning sample	1
1 11:55:42 0 2020-10-07 11:50:17 9 2020-10-07 11:45:48 8 11:41:04	3 CH 2 20 1 20 16 TJ 15 TJ	Y174A 201007-s2 201007-s1 M10072020-HPMAK7 M10072020-HPMAK6	}_	1 Finis 1 1	PROTON shed ex PROTON PROTON PROTON	xperimer ~ ~ ~			* * * * * *	* * * * * * *	yueci yueci hangq hangq mckenztj mckenztj	л л л л л	home/nmr1 Group home/nmr1 Grou	Juan Guan Gun Nyres Nyres	Curu	irrently nning sample	i.
11:55:42 2020-10-07 11:50:17 2020-10-07 11:45:48 2020-10-07 11:41:04 2020-10-07	3 CH 2 20 1 20 16 TJ 15 TJ	Y174A 201007-s2 201007-s1 M10072020-HPMAK7 M10072020-HPMAK6		1 Finis 1 1	PROTON shed ex PROTON PROTON PROTON	kperimer			* * * * * * *	* * * * * * * *	yueci yueci hangq hangq mckenztj mckenztj		home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group	Juan Juan Jun Jyres Jyres	Curui	irrently nning sample	'n_
11:55:42 2020-10-07 11:50:17 2020-10-07 11:45:48 2020-10-07 11:41:04 2020-10-07 11:36:12	3 CH 2 20 1 20 16 TJ 15 TJ 14 TJ	Y174A 201007-s2 201007-s1 M10072020-HPMAK7 M10072020-HPMAK6 M10072020-HPMAK5		1 Finis 1 1 1	PROTON shed ex PROTON PROTON PROTON PROTON	xperimer ~ ~ ~			* * * * * * *	* * * * * * *	yueci yueci hangq hangq mckenztj mckenztj		home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group	Juan Sun Sun Syres Syres	Curu	irrently nning sample	6 7
11:55:42 2020-10-07 11:50:17 2020-10-07 11:45:48 2020-10-07 11:41:04 2020-10-07 11:36:12 2020-10-07	3 CH 2 20 1 20 16 TJ 15 TJ 14 TJ 13 TJ	Y174A 201007-s2 201007-s1 M10072020-HPMAK7 M10072020-HPMAK6 M10072020-HPMAK5 M10072020-HPMAK4		1 Fini: 1 1 1 1	PROTON shed ex PROTON PROTON PROTON PROTON PROTON	xperimer ~ ~ ~ ~			* * * * * * * * *	* * * * * * * * *	yueci yueci hangq hangq mckenztj mckenztj mckenztj		home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group	Juan Sun Sun Syres Syres	Curun	irrently nning sample	6 7
11:55:42 2020-10-07 11:55:42 2020-10-07 11:45:48 2020-10-07 11:41:04 2020-10-07 11:36:12 2020-10-07 11:31:44 2020-10-07	3 CH 2 20 1 20 16 TJ 15 TJ 14 TJ 13 TJ	Y174A 201007-s2 201007-s1 H10072020-HPHAK7 H10072020-HPHAK6 H10072020-HPHAK5 H10072020-HPHAK4	}_	1 Finis 1 1 1	PROTON shed ex PROTON PROTON PROTON PROTON PROTON	xperimer			* * * * * * * * *	* * * * * * * * * *	yueci yueci hangq hangq mckenztj mckenztj mckenztj		home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group	Juan Sun Sun Syres Syres	Curu	irrently nning sample	6 7
1:55:42 2020-10-07 11:55:47 2020-10-07 11:45:48 2020-10-07 11:41:04 2020-10-07 11:36:12 2020-10-07 11:31:44 2020-10-07 11:26:56	3 CH 2 20 1 20 16 TJ 15 TJ 14 TJ 13 TJ 12 TJ	Y174A 201007-s2 201007-s1 M10072020-HPMAK7 M10072020-HPMAK5 M10072020-HPMAK4 M10072020-HPMAK4		1 Fini: 1 1 1 1	PROTON shed ex PROTON PROTON PROTON PROTON PROTON PROTON	xperimer ~ ~ ~ ~ ~ ~			* * * * * * * * *	* * * * * * * * *	yueci yueci hangq hangq mckenztj mckenztj mckenztj mckenztj		home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group	iuan Sun Sun Syres Syres Syres	Curui	urrently nning sample	6 7
11:55:42 2020-10-07 11:55:42 2020-10-07 11:45:48 2020-10-07 11:45:48 2020-10-07 11:41:04 2020-10-07 11:31:44 2020-10-07 11:25:56 2020-10-07	3 CH 2 20 1 20 16 TJ 15 TJ 14 TJ 13 TJ 12 TJ 11 TJ	Y174A 201007-s2 201007-s1 H10072020-HPHAK7 H10072020-HPHAK5 H10072020-HPHAK3 H10072020-HPHAK3 H10072020-HPHAK3		1 Finis 1 1 1 1 1	PROTON shed ex PROTON PROTON PROTON PROTON PROTON PROTON	xperimer 			* * * * * * * * * *	* * * * * * * * * *	yueci yueci hangq mckenztj mckenztj mckenztj mckenztj mckenztj		home/nmr1 Group home/nmr1 Grou	Juan Juan Jures Jyres Jyres Jyres	Current	urrently nning sample	6 7
11:55:42 2020-10-07 11:50:17 2020-10-07 11:50:17 2020-10-07 11:45:48 2020-10-07 11:36:12 2020-10-07 11:31:44 2020-10-07 11:22:24	3 CH 2 20 1 20 16 TJ 15 TJ 14 TJ 13 TJ 12 TJ 11 TJ	Y174A 201007-s2 201007-s1 M10072020-HPHAK6 M10072020-HPHAK6 M10072020-HPHAK3 M10072020-HPHAK3 M10072020-HPHAK3	ļ	1 Finis 1 1 1 1 1	PROTON shed e) PROTON PROTON PROTON PROTON PROTON PROTON	xperimer ~ ~ ~ ~ ~ ~			* * * * * * * * *	* * * * * * * * * *	yueci yueci hangq mckenztj mckenztj mckenztj mckenztj	л л л л л л л	home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group	iuan iuan iun iyres iyres iyres iyres	Curun	irrently nning sample	6 7
11:55:42 2020-10-07 11:50:17 2020-10-07 11:50:17 2020-10-07 11:45:48 2020-10-07 11:45:48 2020-10-07 11:35:12 2020-10-07 11:32:55 2020-10-07 11:25:56 2020-10-07 11:22:24 2020-10-07	3 CH 2 20 1 20 16 TJ 15 TJ 14 TJ 13 TJ 12 TJ 11 TJ	Y174A 201007-s2 201007-s1 H10072020-HPHAK7 H10072020-HPHAK6 H10072020-HPHAK5 H10072020-HPHAK3 H10072020-HPHAK3		1 Finis 1 1 1 1 1	PROTON shed e) PROTON PROTON PROTON PROTON PROTON PROTON	xperimer · · · · ·			* * * * * * * * *	, , , , , , , , , , , ,	yueci yueci hangq mckenztj mckenztj mckenztj mckenztj		home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group home/nmr1 Group	iuan iuan iun iyres iyres iyres iyres	Curun	arrently nning sample 5 14 Next av	6 7 ailable
1:55:42 2020-10-07 11:55:17 2020-10-07 11:45:48 2020-10-07 11:45:48 2020-10-07 11:33:44 2020-10-07 11:33:44 2020-10-07 11:26:55 2020-10-07 11:22:24 h	3 CH 2 20 1 20 16 TJ 15 TJ 14 TJ 13 TJ 12 TJ 11 TJ	Y174A 201007-s2 201007-s1 H10072020-HPHAK6 H10072020-HPHAK5 H10072020-HPHAK4 H10072020-HPHAK3 H10072020-HPHAK3	runs	1 Finis 1 1 1 1 1	PROTON shed e) PROTON PROTON PROTON PROTON PROTON PROTON PROTON	xperimer 			* * * * * * * *	* * * * * * * * * *	yueci yueci hangq mckenztj mckenztj mckenztj mckenztj		home/nmr1 Group home/nmr1 Grou	Juan Juan Jun Jures Jyres Jyres Jyres Jyres	Currun	urrently nning sample 5 14 Next av sample	6 7 ailable positio
1:55:42 2020-10-07 11:55:17 2020-10-07 11:45:48 2020-10-07 11:45:48 2020-10-07 11:35:12 2020-10-07 11:31:44 2020-10-07 11:22:24	3 CH 2 20 1 20 16 TJ 15 TJ 14 TJ 13 TJ 12 TJ 11 TJ	Y174A 201007-s2 201007-s1 H10072020-HPHAK5 H10072020-HPHAK5 H10072020-HPHAK3 H10072020-HPHAK3 H10072020-HPHAK3 H10072020-HPHAK3	runs	1 Finis 1 1 1 1 1	PROTON shed ex PROTON PROTON PROTON PROTON PROTON PROTON	xperimer 	Automation		· · · · · · · · · · ·	· · · · · · · · ·	yueci yueci hangq hangq mckenztj mckenztj mckenztj mckenztj		home/nmr1 Group home/nmr1 Grou	Juan Juan Jun Jures Jures Jures Jures Jures		urrently nning sample 5 14 Next av sample	6 7 ailable positio





### **ICON-NMR** Interface

holder positio	n		Experiment number			pa	arame	ters		
/		Sample folder name		so	vent	Experiment		research la	ab expe	eriment length
1 th	Finished /home/nmr1	CHY174A		1	CDC13	PROTON	<b>B</b> 4	Guan	III 🦲	00:01:29
4 <b>4</b> 1	Finished									
te	Finished /home/nmr1	L CHY174B		1	CDC13	PROTON	_ ∎∳	Guan		00:01:29
5 1	Running									
er Hann	Running /home/nmr1	wy-1007-OTs		1	CDC13	F19_BBOF	<b>14</b>	Liu		00:00:50
	Queued (hono (num)	10-7-ph-0040-6-1h		4	CDC17	DDOTON	=4	Cure.		00.01.20
7 4.1	Queued / nome/ nmm	10-7-ph-60H2-5-1H		T	CDCIS	PROTON	<b>•</b>	Sun	day/night queu	Ie
Name you run on the s go into Do not with a p previou data wi	your sample a multiple exp same sample numbered su start or end t period. Do no isly-used nan Il go into the	<b>folder-</b> if beriments e, they will ubfolders he name t use nes or the old folder.	Specify solvent– incorrectly specified solvent will cause loc fail and/or spectrum t badly referenced Set Experime manager to run available on yo	k to o be nt— n ex our a	-Con perin	Some adjust tact NMF nents no	pa ted, ۲	rameters but be ca Ch ler pre	can be areful! neck expe ngth befo essing Se	eriment re ubmit!





# The Night Queue

- On the AV400, <sup>13</sup>C spectra are automatically placed in the night queue. If the experiment time is < 20 min, they will run during idle daytime. Otherwise, they will run starting at 9 PM.
- On the NEO400, 2D spectra and C13CPD experiments will default to the night queue.
   C13CPD32 will default to the day queue, so either **do not adjust the experiment length** longer than 30 minutes or switch it to the night queue.
- Day-queue experiments (such as <sup>1</sup>H 1D) made to run long (> 20 min on AV400, > 30 min on NEO400) should be set to the night queue by clicking on the sun icon:
   It should switch to a moon:
- Mind the total length of the night queue: 9PM-9AM on NEO400, 9PM-10:30 AM on AV400.
   Your experiment will not run if it can not finish within this window. Allow
   approximately 5 extra minutes per experiment for lock/atm/shimming. Before submitting,
   check the current night queue length (from already-submitted experiments) in the bottom
   right corner:







### **Checking Status of NMR Instruments**

- Type <a href="http://chav400.cros.uc.edu:8015">http://chneo400.cros.uc.edu:8015</a> in web browser while on campus (links on NMR lab website)
- Username is your ICON-NMR username, password is "chemistry"
- "Read only" interface

$\leftrightarrow$ $\rightarrow$ C ( $\blacktriangle$ Not	secure   chav4	400.cros.uc.	edu:8015/tem	plate-automation.	htm								\$	A 🔒
	en	W	eb			$\mathbf{M}$								
	Automation - F	Running - Bus	y until : Tue 10:2	6 - Day Experiments	: 00:07 - Night Exp	eriments : 0	0:00							
▶ Help ▶ Logoff	ATM Running	Lock Fiel	I d Shim Rec	🧟 🖬	re Data Process	D Data								
Logged in as nmrl	□9 □10 ♣	1	Completed											•
Instrument Name chav400.cros.uc.edu: spect	10	er.	Completed	/home/nmr1	20201013-s1	1	CDC13	PROTON	<mark>=</mark> 4	Sun		00:01:29	hangq	
	🗆 11 🛛 🖶	<b>**</b> 1	Completed											
	11	the second	Completed	/home/nmr1	wy-1012G	1	CDC13	F19_BBOF	ալիս 💽	Liu		00:00:26	yanwo	
	🗆 12 🛛 🖶	<b>*</b> 1	Completed											
	12	er.	Completed	/home/nmr1	wy-1012H	1	CDC13	F19_BBOF	արին 🔁	Liu		00:00:26	yanwo	
	🗆 13 🛛 🖶	<b>**</b> 1	Completed											
	13	er.	Completed	/home/nmr1	wy-1012I	1	CDC13	F19_BBOF	S alp	Liu		00:00:26	yanwo	
	□ <sub>14</sub> ♣	2 · ···	Running											
	14	1 kr	Running	/home/nmr1	B2-P11-R2-H	1	MeOD	PROTON	<mark></mark>	Merino		00:01:29	senevipp	
	Date Time	Holder N	ame .	/homo/norr1	ם בת 11ת בם No	1 . Experimer	MOD t Load Al	M Rotation Loc	k Shim Acq I	Proc User	Title	00.05.05		Rer -
	2020-10-13 10:18	:26 14 B	2-P11-R2-H		1	PROTON	~			senevipp	Group Merino			
	2020-10-13 09:44	:45 13 w	y-1012I		1	F19_BBOF	~ ~	~	~ ~	<ul> <li>yanwo</li> </ul>	Group Liu			sref .1/c
	2020-10-13 09:40	:46 12 w	y-1012H		1	F19_BBOF	~ ~	~	~ ~	<ul> <li>yanwo</li> </ul>	Group Liu			sref .1/c





### **General Rules**

- 1) Wait 10 seconds after your sample is ejected before retrieving it.
  - Removing your sample before the sample changer light turns green again *will freeze the autosampler* and halt data collection.
- 2) Clean tubes with kimwipes and measure their depth with the depth gauge.
  - Tubes with small sample volumes can be placed higher than bottom of gauge to center the sample in the coil.
  - Never place the tube lower than the bottom of the gauge—it may break in the probe.
- 3) Retrieve your samples from the room in a reasonable amount of time.
  - Failure to do so results in our having to dispose of the samples as "unknowns" which is costly and involved.
- 4) Mark your tubes
  - Doing so will allow us to contact you if something happens to your tube, or if it becomes forgotten. Initials should be fine in most cases.
- 5) Log out when you are done
  - This prevents other users from accidentally submitting jobs as you.





# **Instrument-Specific Rules**

### NEO400

#### Queue experiments in numerical order

- This is necessary due to a glitchy autosampler.
- Especially important when the instrument is idle. Failure to do so can cause automation to stop.
- The night queue is still operational.

### Do not adjust the time of C13CPD32 experiments longer than 30 minutes.

- They will run during the daytime regardless of their length!
- For long <sup>13</sup>C, use C13CPD instead.
- Alternatively you can put these in the night queue by clicking the sun icon:

# AV400

### Put experiments with experiment times exceeding 20 minutes in the night queue.

- Large numbers of moderately-long experiments should also be put in the night queue. If they are individually under 20 minutes they will run during idle daytime.
- See me if your account does not allow you to put experiments in the night queue.





# **Chemical Shift Ranges**



Note that the <sup>13</sup>C and <sup>1</sup>H trends tend to match each other! Chemical shift is affected by the same electrons in each case!



<sup>1</sup>H zg, zg30



### Standard Experiments







• High sensitivity

<sup>1</sup>H zg, zg30

- Impurities usually evident if present
- Shows multiplicity, aids in assignments
- Typically requires 100% deuterated solvent
- Relatively fast T1 relaxation, pulse delay can be ~ 2 s
- Overlap sometimes an issue



7.6 7.2 6.8 6.4 6.0 5.6 5.2 4.8 4.4 4.0 3.6 3.2 2.8 2.4 2.0 1.6 1.2 0.4 f1 (ppm)





<sup>13</sup>C CPD, zgig, zgpg, dept







#### <sup>1</sup>H-<sup>1</sup>H COSY 2D

- Provides 3-bond <sup>1</sup>H-<sup>1</sup>H correlations
- Complex multiplicies resolved in cross-peaks
- Option for presaturation of solvent peak
- Most useful for compounds with many protons!







#### <sup>1</sup>H-<sup>1</sup>H NOESY 2D

- Provides through-space <sup>1</sup>H-<sup>1</sup>H correlations
- Unsuitable for compounds between ~1-2 kDa (related ROESY should be performed instead for these)
- Requires setting of a mixing time, usually between 500-800 ms.
- Most useful for compounds with many protons!









- confuse you! They are separate spectra by default!
- Related: <sup>13</sup>C-<sup>1</sup>H HMBC 2D shows correlations between carbons and protons that are 2+ bonds apart











zg 1	Ds fo	or other nuc	lei		<sup>19</sup> F
<ul> <li>Sor</li> </ul>	netim	ies have long	gT1s		
<ul> <li>Sor che</li> </ul>	netim mical	ies have very I shift ranges	/ large		-113.5' -114.5' -115.5' -116.5' -117.5' -118.5' -119.5' -120.5' -121.5' -122.5' -123.5' -124.5' -12 f1 (ppm) 31P
<ul> <li>Ser that</li> </ul>	nsitivi n <sup>15</sup> N	ties range fro to comparat	om worse ble to <sup>1</sup> H!		10 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 27AI
	Spinl	Natural abundance	Pacantivity $(13C - 1)$	Resonance frequency on	
Hydrogon	1/2		5670	400 00	
Deuterium	1/2	0.015%	0.0082	61 40	
Carbon-13	1/2	1.108%	1.00	100.60	110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -6(
Nitrogen-15	1/2	0.370%	0.022	40.56	
Fluorine-19	1/2	100.000%	4730	376.36	
Aluminum-27	5/2	100.000%	1170	104.32	
Silicon-29	1/2	4.700%	2.1	79.48	
Phosphorous-3'	1/2	100.000%	377	161.92	
Selenium-77	1/2	7.630%	3.15	76.29	
					800 750 700 650 600 550 500 450 400 3! f1 (ppm)



#### Department of Chemistry NMR Facility



### Installing/Activating MNova

UWEBSITE TRANSPORT	DURCES	🕹 DOWNLOAD MNOVA FREE	TRIAL @ QUESTIONS?	😢 Registration Wizarc	t de la constante de	?
> Download Mnova	Download the latest (	Mnova Version		You will ı	need one or several license MestReNova	e files to use
> Download NMR				60 1	FREE	
> Download MS		ć	A		TRIAL	
> Download NMR Predict	Mnova 14.2.0 👻	Mnova 14.2.0	€)€> Mnova 14.2.0 -	If you already have	e If you still need	d to get a license
> Download Verify				your license file(s) press the button to	) please select options:	one of the follo
> Download qNMR	Latest Manual: Please note that manuals are versior	n-specific. If you need a manual for a previ	ous version, please go to the	install it(them)		
	bottom of the page and select the ve	arcion that you are interested in				
> Download RM	page and select the ve	i sion that you are interested in.		<b>X</b> = -1-11	E al la la	D
	Download the latest manual (PDF)	ab com/download	1/mnova	Install Host JD: TEDZW-F	Evaluate	Buy
Download RM     Download SMA     Download M	Download the latest manual (PDF)	ab.com/download	d/mnova	Install Host ID: TEDZW-F	Evaluate	Clo
<ul> <li>Download RM</li> <li>Download SMA</li> <li>Download M</li> <li>Get the cam</li> </ul>	Download the latest manual (PDF)	ab.com/download	d/mnova	Install Host ID: TEDZW-F CSelect a License File ← → ← ↑ ≧ > This F Orcanize * New Folder	Evaluate 22MY5CQ4-29QQQ-T6XSFC82	Buy Cl earch Documents
Download RM Download CMA Get the cam https://www.a es/software.	Download the latest manual (PDF) Inova from mestrela pus license file from artsci.uc.edu/depar html	ab.com/download n rtments/chemistry	d/mnova y/resourc	Install Host ID: TEDZW-F Select a License File ← → ↑	Evaluate 2MY5CQ4-29QQQ-T6XSFC82	earch Documents  earch Documents  Date modified  10/1/2020 3:15 PM  10/1/2020 4:58 PM

• The license needs to refresh every few months by running the software on the campus network



Cance

License Files (\*.lic \*.zip

Open

File name: University of Cincinnati Campus Mn ~



### Resources

#### On the NMR lab website:

- Instructions for running on the instruments
- Instructions for special samples (protonated solvents, small volumes)
- Instructions for accessing data
- Web interfaces for automated instruments (AV400, NEO400)
- Link to MNova and Mnova license





#### Department of Chemistry NMR Facility









Contact:

Alex Greenwood Office: Rm 123C/Crosley; Phone: 513-556-9211; Email: greenwa2@ucmail.uc.edu For user training, technical assistance, NMR questions and discussions.

