Selective 1D experiments with IconNMR

Minli Xing, U of Michigan BioNMR core lab

- 1. Contact BioNMR core staff Minli Xing (<u>mlxing@umich.edu</u>) to grant supervisor permission to your account on the 600 MHz NMR in Mary Sue Coleman Hall.
- Login to your account, enter your sample information, in the experiment drop down menu, scroll down to the bottom and then select the selective 1D experiment you want to run. SELCOGP for selective 1D COSY, SELNOGPZS.2 for selective 1D NOESY, SELDIGPZS for selective 1D TOCSY and SELROGP for selective 1D ROESY.

	N UM_PROTON_1D 1H P	IOTON 🔹 🖈 💽 No Analysis 🝭 🔳 🧄	\$	000000 📓	00:02:
T	C HMQCGP	sw opt. HMQC with gradients (magn. mode)			
	C HSQCGP	sw opt. HSQC sens. improved with gradients (e/a '	TPPI)		
	C HSQCEDETGP	sw opt. edited HSQC with gradients (e/a TPPI)			
	C HIMQCGPML	sw opt. HMQC-TOCSY with gradients (magn. mode)			
	C HMQCBI	sw opt. HMQC using BIRD pulse (magn. mode)			
	C HMQCBIPH	sw opt. HMQC using BIRD pulse (States-TPPI)			
	CHMQC	sw opt. HMQC (magn. mode)			
	С НМQСРН	sw opt. HMQC (States-TPPI)			
	C HIMBCGPND	sw opt. HMBC with gradients			
	C HMBCLPND	sw opt. HMBC with low pass J-filter (magn. mode)			
	C HSQCETGPML	sw opt. HSQC-TOCSY with gradients (e/a TPPI)			
	C HSQCEDETGPSISP_ADIA	1H-13C multiplicity edited HSQC with gradient se	lectio	n BF1 >= 700	MHz
	C HSQCETGP	sw opt. HSQC with gradients (e/a TPPI)			
	C HSQC_TOCSY_ADIA	1H-13C HSQC-TOCSY with gradient selection BF1 >=	700 M	Hz	
	C HCCOSW	sw opt. CH-correlation			
ł	C HCCOLOCSN	sw ont. COLOC			
	C SELCOGP	selective COSY experiment w/gradients			
	C SELNOGPZS.2	selective NOESY experiment w/gradients			
	C SELDIGPZS	selective TOCSY experiment w/gradients			
-	C SELROGP	selective ROESY experiment w/gradients			

3. Once you select the selective experiment, IconNMR will load two experiments for you, the first one is a 1D 1H experiment, the second one is your selective 1D experiment, you may modify parameters such as number of scans, and then click **submit** to run the experiments. A window will pop up and click **OK** to continue.



4. The 1D 1H experiment will run first, once it is done, double click the 1D 1H experiment under the **Preceding Experiments** window, and topspin will automatically display your 1D 1H spectrum.

	Preceding Experiments																_
	Date	# Holder	Name	No.	Solvent	Experiment	Load	ATM	Rotation	Lock	Shim	Acq	Proc				
	2022-08-16 10:07:58																
Iruker TopSpin 4	1.4 on Isi600pp.isi.umich.e	du as bionmrcore	users														
	uire <u>P</u> rocess	A <u>n</u> alyse	App <u>l</u> ications	<u>M</u> anage										87 🔏	ŝ	? י	BRUKE
Λ Pro <u>c</u> . Spec	trum 🗸 🔨 Adjust Pha	ase 🗸 🔍 🖪a	iseline 🗸 🛛 🧍 Cali	b. A <u>x</u> is 🗸 🗎	A <u>d</u> vanced 🗸	I.							8	ń P) 	Ē	
2D *2 ↓ 3D /2 ↓	() © (N) () @ () = = ⊕ () () () () () () () () () () () () ()	* * ± ⊼.	± ▶ ₩ 3 ■ 🖶 🕂 🖡	≱ ₹ ∾													
SPECTRUM	PROCPARS ACQUPARS	TITLE PULS	EPROG PEAKS	INTEGRALS	SAMPLE ST	RUCTURE PLOT	FID ACQU										
Aug16-2022-	mixing 20 1 /icondata/	data/mlxing/nm	۱r														
PROTON CDC	0000 N3 Acondata mixing 11																F
																	-
					7.2966 7.2839 7.2593	C 7.1886			2.6724 2.6597 2.6343		$-\frac{1.5324}{1.2547}$	-0.0002					-
												I					-
												1					-
									1								-
																	-
																	-
8606						-6											
ēļ		1				_#L			Å		HANK	6 م	۱,				<u> </u>
15			10				5					0					[ppm]

5. Under the Acquire tab, click More, and select Setup Selective 1D Expts. in the drop down menu.

\equiv <u>Acquire</u> <u>Process</u> Analyse Applications <u>Manage</u>	
Create Dataset 🚽 Sample → 轢 Lock V Tune → 븿 Spin → 幦 Shim → औ Prosol → Gain → ▶ Ru	n ↓ M <u>o</u> re ↓
2DI*2 ♦ I II ® IN O ® IN III IIIIIIIIIIIIIIIIII	IconNMR Automation (icona)
3D /2 호 ⊕ ⊖ ← ⊡ ⊥ ← → ↔ ± 1	<u>S</u> etup Selective 1D Expts.
	TopSoli <u>d</u> s (topsolids)
SPECTRUM PROCEARS ACQUEARS THE POLSEPROG PEARS INTEGRALS SAMPLE STRUCTURE PLOT FID ACQU	BioTop
Aug16-2022-mixing 20 1 /icondata/data/mixing/nmr Ishortcode 000000	TopGuide (topguide)
PROTON CDCI3 licondata mixing 11	Shape <u>T</u> ool (stdisp)
	APSY (apsy)
(000mm-1000	NMR Thermometer (nmrtemp)
	NOL4 4400

6. A new menu will appear, click **Define Regions.**

Bruker TopSpin 4.1.4 on Isi600pp.lsi.umich.edu as bionmrcoreusers											
≡	<u>A</u> cquire	<u>P</u> rocess	A <u>n</u> alyse	App <u>l</u> ications	<u>M</u> anage						
G <u>B</u> acl	< 1D Selectiv	ve Experiment :	Setup 🦺 D	efine <u>R</u> egions	Create <u>D</u> atase	ets 🗸					
2D *2 3D /2		♥ \$ [©] , ₩ 5 € <u>1</u> . ₩	+) ‡ ∓ ; + ↔ ± ;	╗╨╎╸ ╫╴╎╸┉╺╴	3∎ ↓ ≪	ICON-NMR					
SPEC1	RUM PROCP	ARS ACQUPAF	S TITLE P	ULSEPROG PEAKS	INTEGRALS	SAMPLE STRU	ICTURE				

Delete integrals. (1) Disable define region by clicking the **define region** icon. (2) Delete current integrals:
2a. Select all the integrals by moving the mouse to the bottom of each peak and click, once selected, a green box will appear at the bottom of the peak. Press the ctrl button and click to select multiple integrals.
2b. Click the **delete region** icon to delete all integrals.



8. Define the region to selectively irradiate (1) Zoom into the region: click and drag over the region you are interested in. (2) Enable define region by clicking the **define region** icon. (3) Click and drag over your peak to define the region. (3) Click the **show full spectrum** icon. (4) Repeat above to define multiple regions.



9. Click the **Save Regions To 'reg'** icon to save the regions you defined. Then click the **return**, **save regions** icon.



10. Go back to IconNMR, if you defined more than one regions, add more selective 1D experiments so that the number of the selective 1D experiments matches the number of regions you defined. The F2 reference spectrum for all your selective experiments is the 1H experiment you just collected. Click on **submit** to submit all selective 1D experiments.

▽ 11	K 3									
	er.	Finished	/icondata	Aug16-2022-mlxing		20	CDCI3	chloroform-d	N PROTON	1H experiment
	the states	Available	/icondata 🚽	Aug16-2022-mixing	-	21	CDCI3	chloroform-d 🗸	C SELDIGPZS	selective TOCSY experimen -
		🚣 F2	/icondata 🚽	Aug16-2022-mlxing	-	20	•			
	fer .	Available	/icondata 🚽	Aug16-2022-mixing	-	22	CDCI3	chloroform-d 🗸	C SELDIGPZS	selective TOCSY experimen 🗸
		₄ F2	/icondata 🚽	Aug16-2022-mlxing	-	20	- -			
				·			_			