HF-NMR
Highfield NMR SRF

UNIVERSITY OF LIVERPOOL

UKMRM June 2025
Tips & Tricks

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University of Liverpool

Tips & tricks
Resources

https://sites.google.com/view/ukmrm2025ttresources/home

What Do I know? / What have I learned?

- Cataloguing experiments - simple shell script
- Pulse sequence libraries - Grenoble & Bruker Users
- Simulated spectra - from mol file to (somewhat) useful peak predictions
- Reference databases - 1D 2D experimental datasets
- NMRbox - virtual machine for NMR software
- Teaching theory to biologists - reson8 NMR week approach
- Favourite au programs - Bruker automations
- Practical things - recycling caps, multi-tube washing, helium recapture

- **Most Importantly all this acquired knowledge is thanks to the openness and expertise of the NMR community!**

Cataloguing experiments

- Simple shell script for scraping experiments
  - Caveats – works for linux (probably mac too) looking for info per dataset (so lots of experiments in one file)

  - Usage

```
mphelan@nmr5: /raid/usr/mphelan/Spectra
mphelan@nmr5:/raid/usr/mphelan/Spectra$ ./dataCheck.sh -e -f ./FEBS_grapes_230905_7/ >example_FEBS.tsv
```

example_FEBS.tsv - LibreOffice Calc

File Edit View Insert Format Styles Sheet Data Tools Window Help

Liberation Sans 10 pt

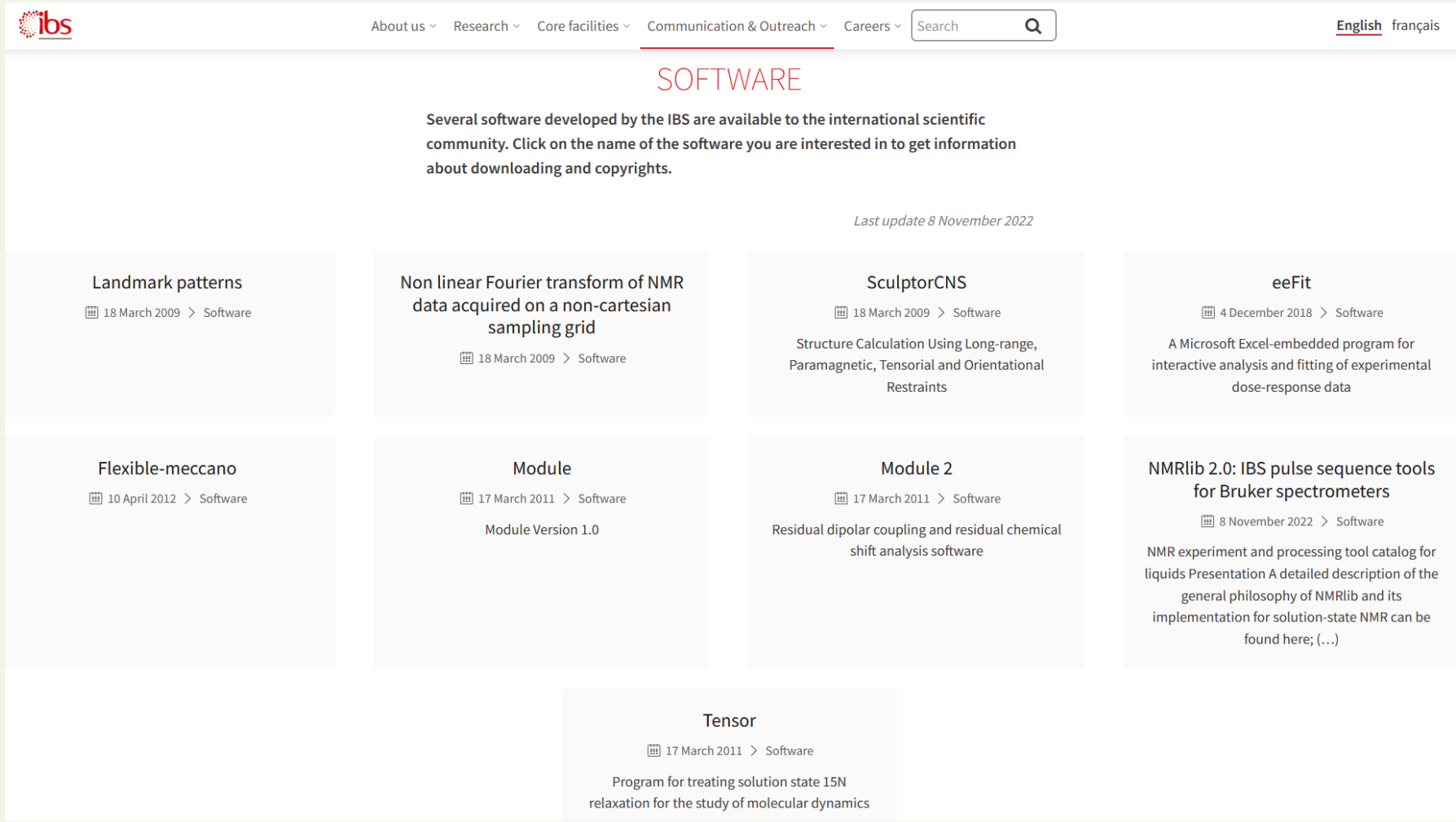
M2 = 32

	A	B	C	D	E	F	G	H	I	J	K	L	M	N
1	dataFolder	Field	ExperimentNo	Timestamp	Title	Holder	Barcode	PulseProgram	Temperature	RG	NS	Overflow	Overflow #	
2	$$ /opt/marie/data/met_test/nmr/FEBS_grapes_230905_7/1080/	700.17	1080	2023/09/05 - 18:51:36	Aderemi_G8_3002119095_[ti5]	103	<Barcode: 3000211909>	<noesygppr1d>	297.8022	79.66	32	YES	32	
3	$$ /opt/marie/data/met_test/nmr/FEBS_grapes_230905_7/1082/	700.17	1082	2023/09/05 - 18:57:15	Aderemi_G8_3002119095_[ti5]	103	<Barcode: 3000211909>	<noesygppr1d>	297.7985	0.98	32			
8	$$ /opt/marie/data/met_test/nmr/FEBS_grapes_230905_7/1270/	700.17	1270	2023/09/05 - 20:07:11	Yasir_G27_30001986221_[ti5]	107	<Barcode: 3000198622>	<noesygppr1d>	297.7994	79.66	32	YES	32	
9	$$ /opt/marie/data/met_test/nmr/FEBS_grapes_230905_7/1272/	700.17	1272	2023/09/05 - 20:12:48	Yasir_G27_30001986221_[ti5]	107	<Barcode: 3000198622>	<noesygppr1d>	297.8009	0.98	32			
10	$$ /opt/marie/data/met_test/nmr/FEBS_grapes_230905_7/2070/	700.17	2070	2023/09/05 - 20:44:57	Emine_R7_30011116508_[ti5]	102	<Barcode: 3001111650>	<noesygppr1d>	297.8013	79.66	32	YES	32	
11	$$ /opt/marie/data/met_test/nmr/FEBS_grapes_230905_7/2072/	700.17	2072	2023/09/05 - 20:50:36	Emine_R7_30011116508_[ti5]	102	<Barcode: 3001111650>	<noesygppr1d>	297.8005	0.98	32			
12	$$ /opt/marie/data/met_test/nmr/FEBS_grapes_230905_7/2100/	700.17	2100	2023/09/05 - 19:48:07	Marike_R10_30002084096_[ti5]	106	<Barcode: 3000208409>	<noesygppr1d>	297.8	79.66	32	YES	32	
13	$$ /opt/marie/data/met_test/nmr/FEBS_grapes_230905_7/2102/	700.17	2102	2023/09/05 - 19:53:44	Marike_R10_30002084096_[ti5]	106	<Barcode: 3000208409>	<noesygppr1d>	297.8026	0.98	32			
14	$$ /opt/marie/data/met_test/nmr/FEBS_grapes_230905_7/2220/	700.17	2220	2023/09/05 - 19:10:26	Beyza_R22_30013109850_[ti5]	104	<Barcode: 3001310985>	<noesygppr1d>	297.7985	79.66	32	YES	32	
15	$$ /opt/marie/data/met_test/nmr/FEBS_grapes_230905_7/2222/	700.17	2222	2023/09/05 - 19:16:07	Beyza_R22_30013109850_[ti5]	104	<Barcode: 3001310985>	<noesygppr1d>	297.7996	0.98	32			
16	$$ /opt/marie/data/met_test/nmr/FEBS_grapes_230905_7/98888/	700.17	98888	1970/01/01 - 01:00:00	Aderemi_G8_3002119095_[ti5]	103	<Barcode: 3000211909>	<noesygppr1d>	297.8	90.5	32			
17														

- Github: https://github.com/LivUniNMR/NMRdataManagement (Rudi Grosman)

Pulse Sequence libraries and Optimisations (1)
IBS Grenoble Protein/RNA suite

- https://www.ibs.fr/en/communication-outreach/scientific-output/software/
- Fully documented



- Works on linux
- Free to academia
- Works on Ts3.5 and above (some probe compatibility checks required for Ts4 but fully documented

Pulse Sequence libraries and Optimisations (1)
IBS Grenoble Protein/RNA suite

- Some useful scripts for optimising pulse lengths / variables per protein / macromolecule

- Quality Control

- Example experiments
  15N TRACT 1D
  Estimation of tumbling

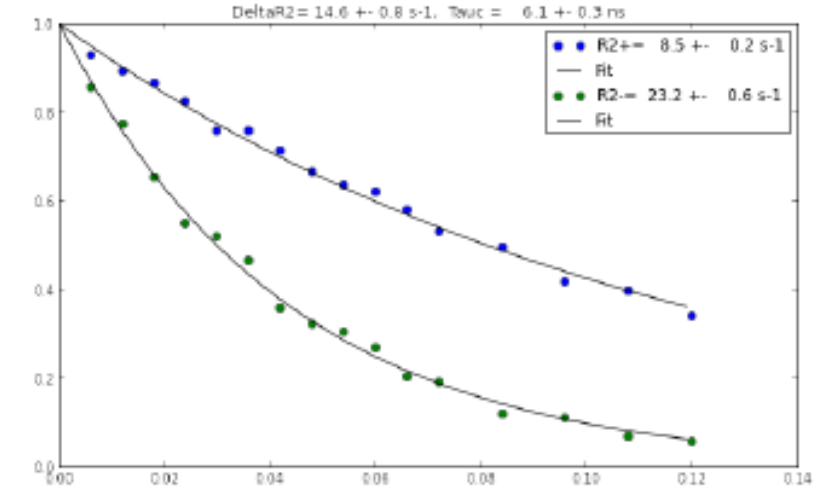
  Sel 1H DOSY
  1H region selected with
  selective pulse, e.g. methyls

  13C 1H DOSY
  13C filter for methyl 1H

1 Ubiquitin

```
libLoc = '/hosts/ibs1300/nmrdata/nmrlib/'
filename1 = '/hosts/ibs700/ibslrmn/afavier/nmr/2019-02-04_ubi-543/15/pdata/1'
filename2 = '/hosts/ibs700/ibslrmn/afavier/nmr/2019-02-04_ubi-543/16/pdata/1'
vclist_file_nl = '/hosts/ibs700/ibslrmn/afavier/nmr/2019-02-04_ubi-543/15/vclist'
vclist_file_bl = '/hosts/ibs700/ibslrmn/afavier/nmr/2019-02-04_ubi-543/16/vclist'
d21 = 0.003
min_val = 9.5
max_val = 8.6

tractAna(filename1, filename2, vclist_file_nl, vclist_file_nl, d21, min_val, max_val )
```



Analysis tool provided

- Python (modular if you wish)
- Authors: Vallet, A., Favier, A., Brutscher, B., and Schanda, P https://doi.org/10.5194/mr-1-331-2020

Pulse Sequence libraries and Optimisations (1)
IBS Grenoble Protein/RNA suite

- Some useful scripts for optimising pulse lengths / variables per protein / macromolecule

- Example experiments
  Optimisations through popt
  Fits and calibrations saved

  15N-filt 1H DOSY
  15N filter for amide 1H

  1D HETSOFAST
  Global structural compactness

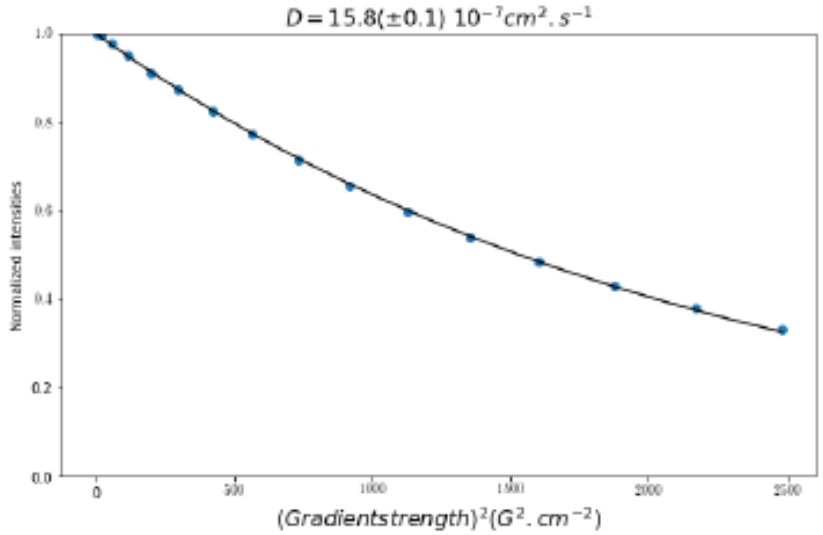
  2D HETSOFAST
  Local structural compactness

2 Ubiquitin

```
1  libLoc = '/hosts/ibs1300/nmrdata/nmrlib/'
2  filename1 = '/hosts/950neo/ibslrmn/afavier/nmr/2019-12-11_S21-LSFubi/2/pdata/1'
3  difflist_file = '/hosts/950neo/ibslrmn/afavier/nmr/2019-12-11_S21-LSFubi/2/difflist'
4  min_val = 2
5  max_val = -1.0
```

```
1  dosyAna(libLoc, filename1, difflist_file,  min_val, max_val)
```

```
Max:  10581648.267089844
Diffusion fitting values saved in: /hosts/950neo/ibslrmn/afavier/nmr/2019-12-11_S21-LSFubi/2/pdata/1/DOSY.fit
Normalized intensities saved in: /hosts/950neo/ibslrmn/afavier/nmr/2019-12-11_S21-LSFubi/2/pdata/1/DOSY.int
```



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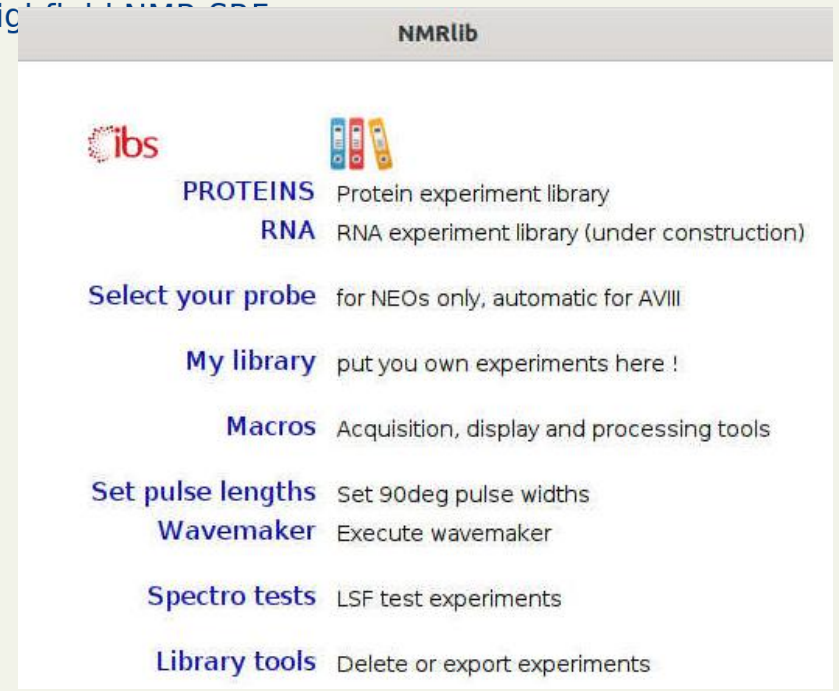
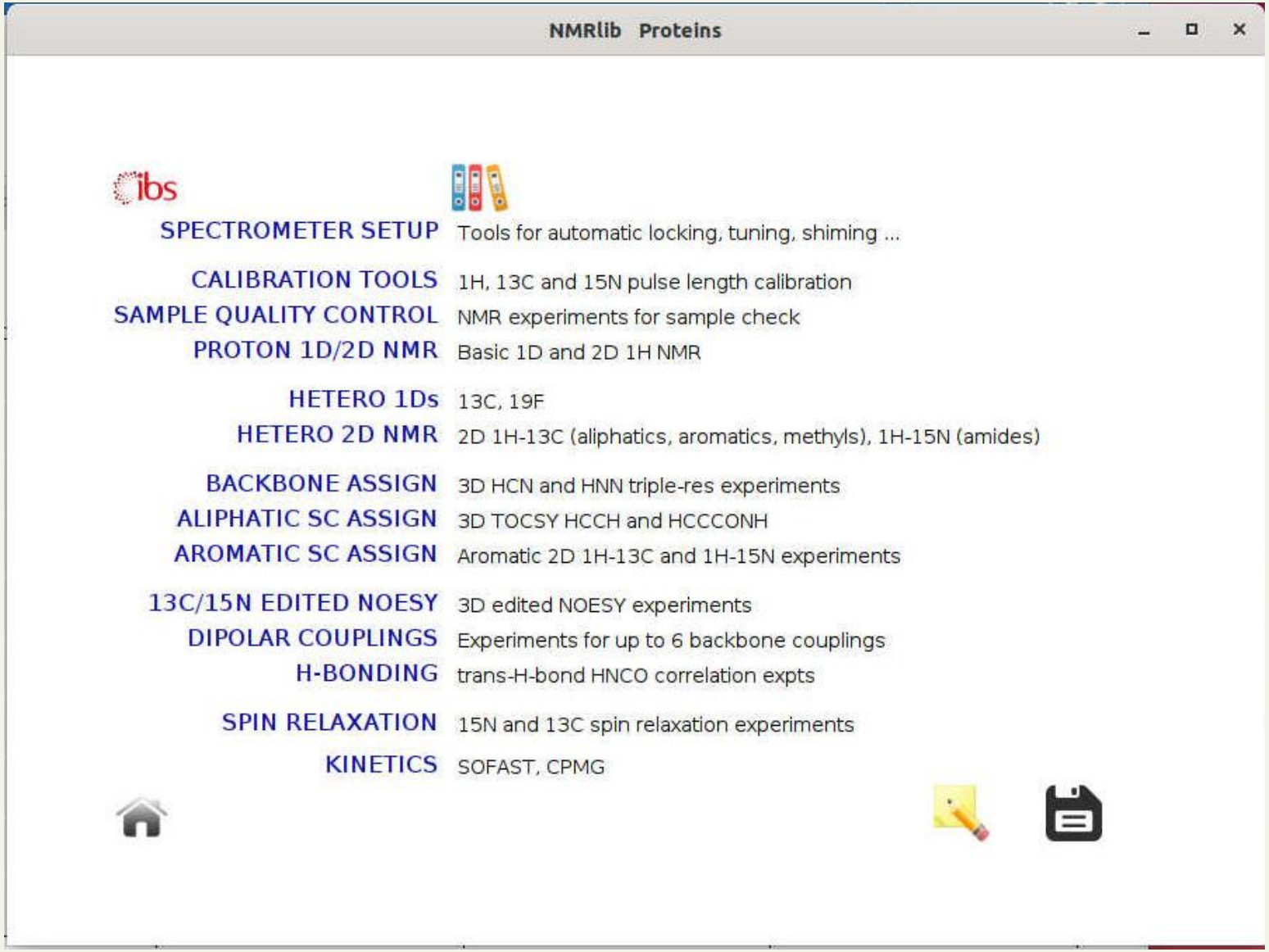
Pulse Sequence libraries and Optimisations (1)
IBS Grenoble Protein/RNA suite



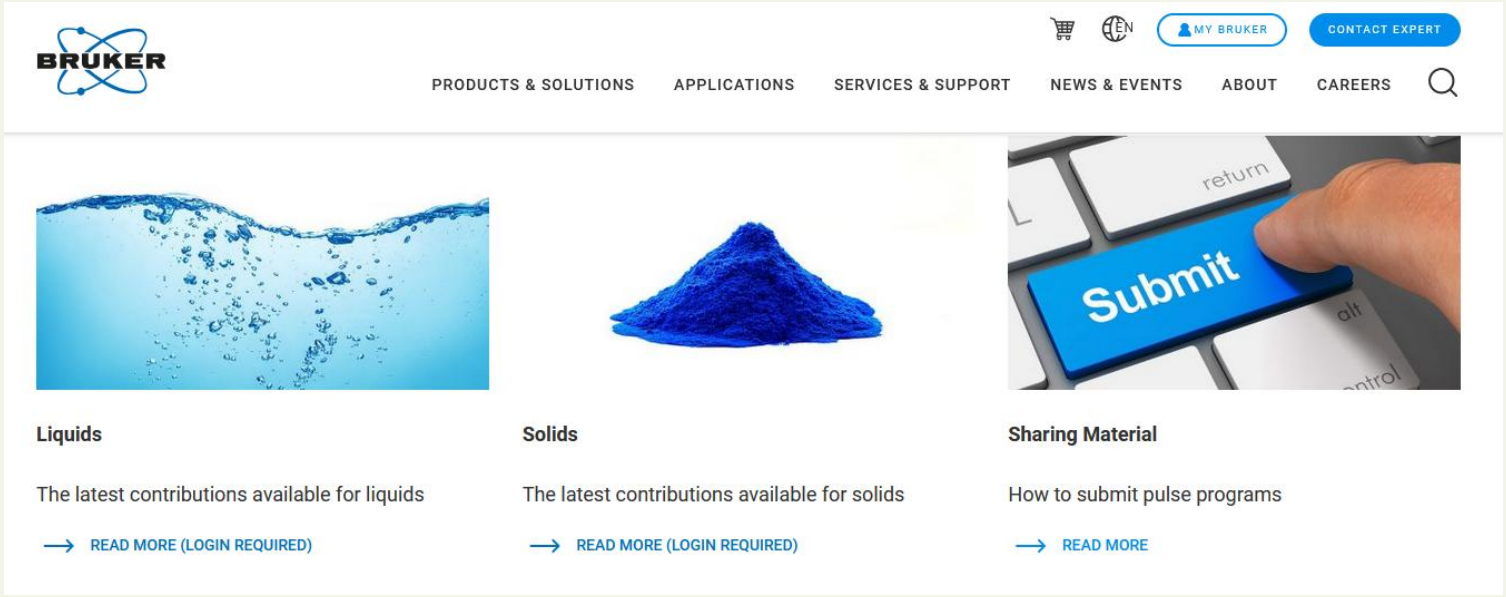


Bruker User Library

https://www.bruker.com/protected/en/services/bruker-user-library/liquids.html

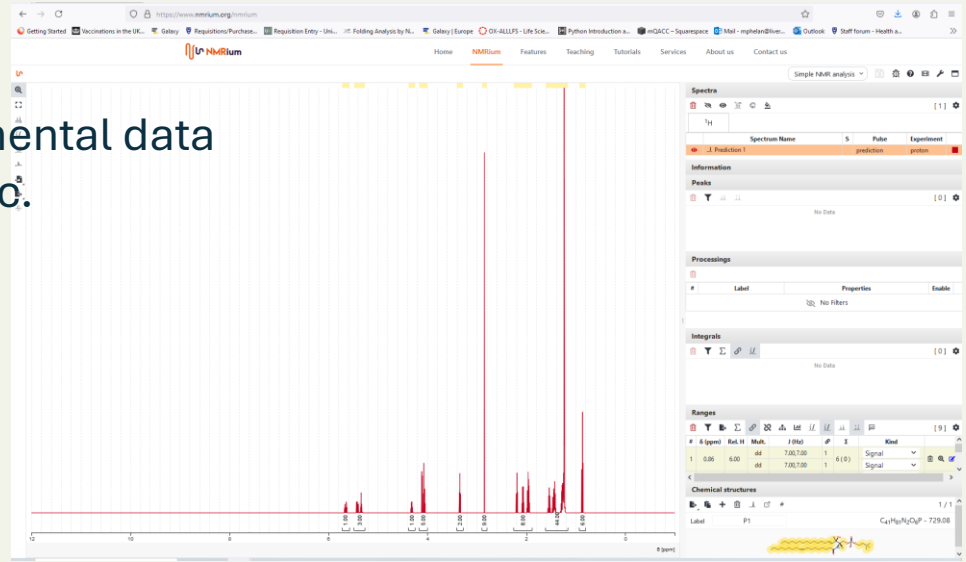
- Suitability for consoles and topspin versions indicated
- With example data
- Links to publication

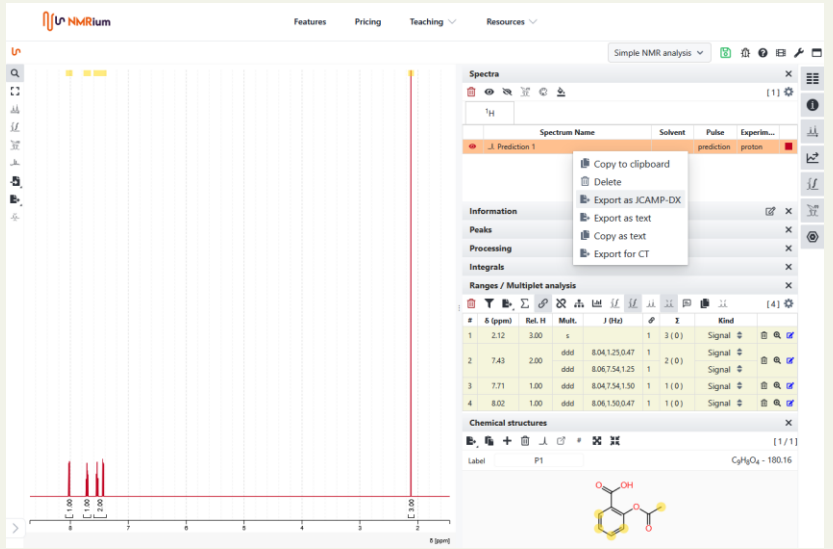
- Ordered into Biomolecules and Small molecules
- Search on name or author
  - Zipped repositories so subdirectories not visible
  - Caveat: I have only used this for DREAMTIME so other datasets may be less complete



Simulated Data

- Nmrium.org
  - Online
  - Overlay experimental data
  - Export jdx tsv etc.





- Gissmo
  - from mol file to (somewhat) useful peak predictions

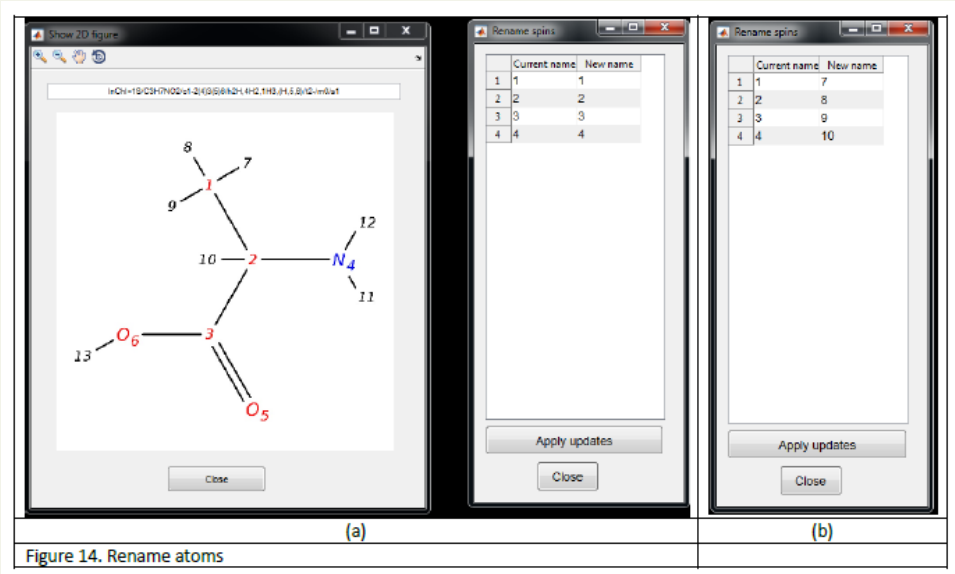


Figure 14. Rename atoms

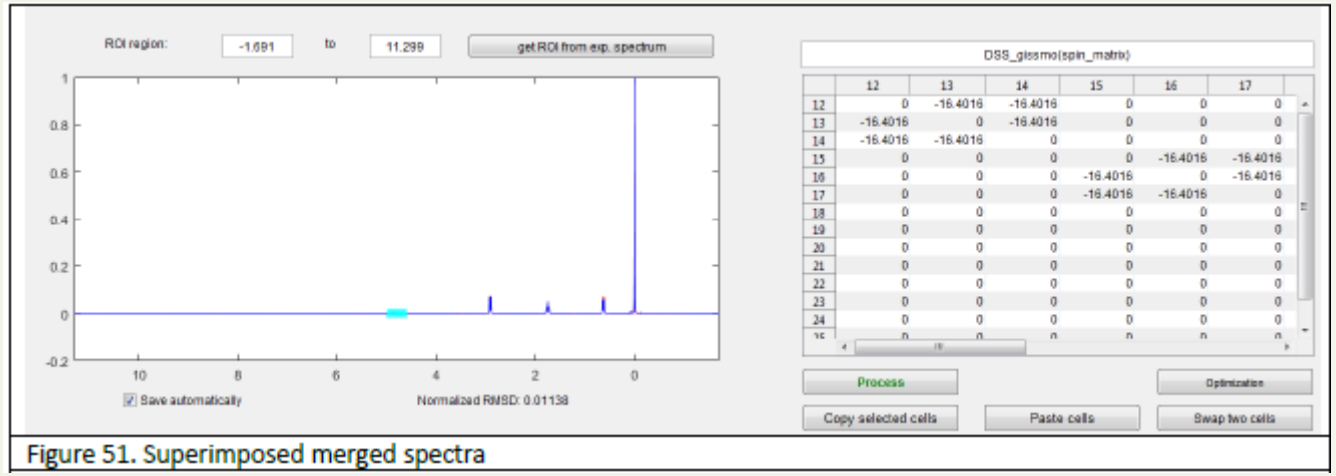
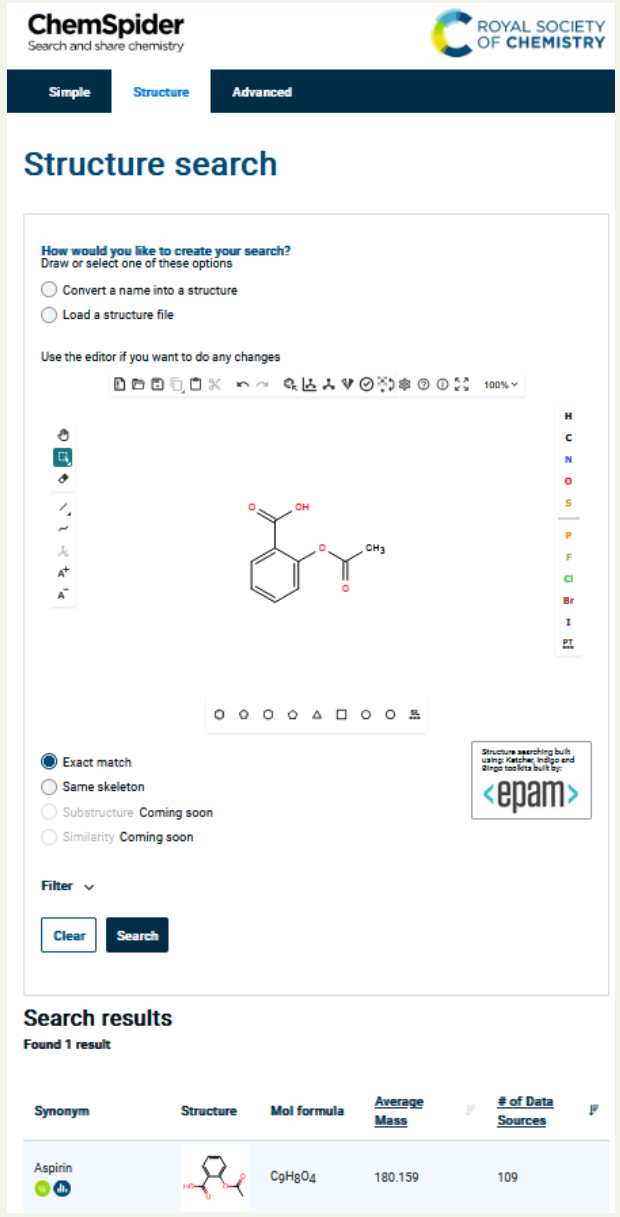


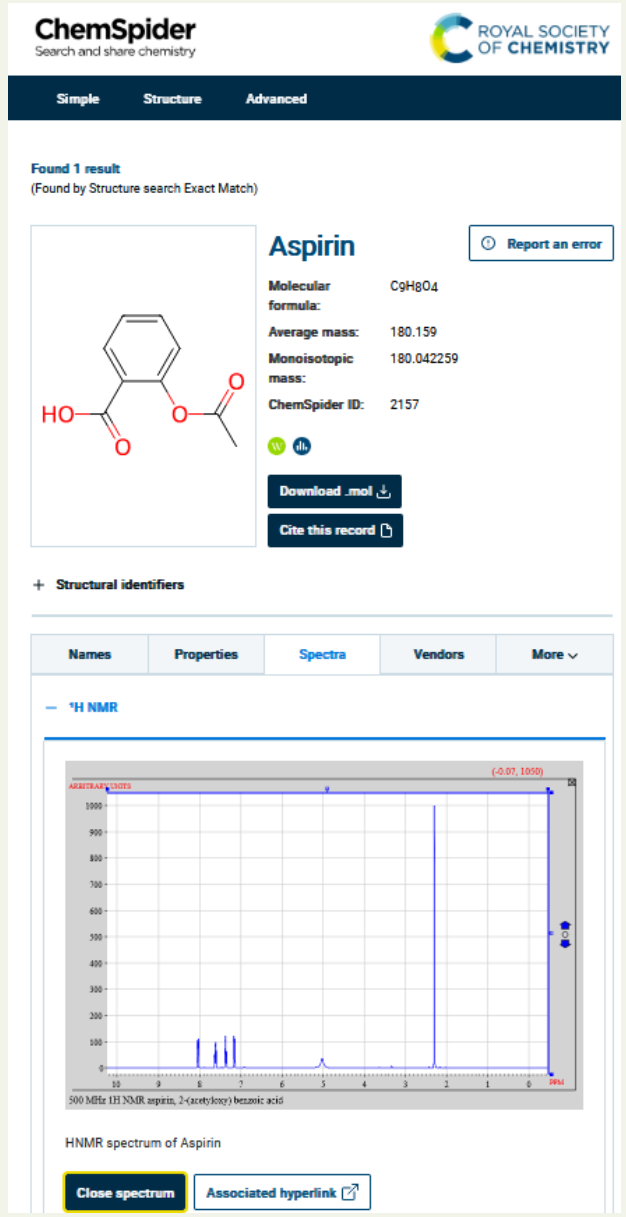
Figure 51. Superimposed merged spectra

- Helpful for teaching and mixture analysis / annotation

Reference Databases (metabolites / natural products)

- Reference databases:
  - **Chemspider** can view spectra but not download?
  - BMRB 1D & 2D data available for download
  - HMDB 1D data available for download
  - BML data available for download (registration required)
  - MetaboLights
  - CASMDB / CCPN





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  - BML data available for download (registration required)
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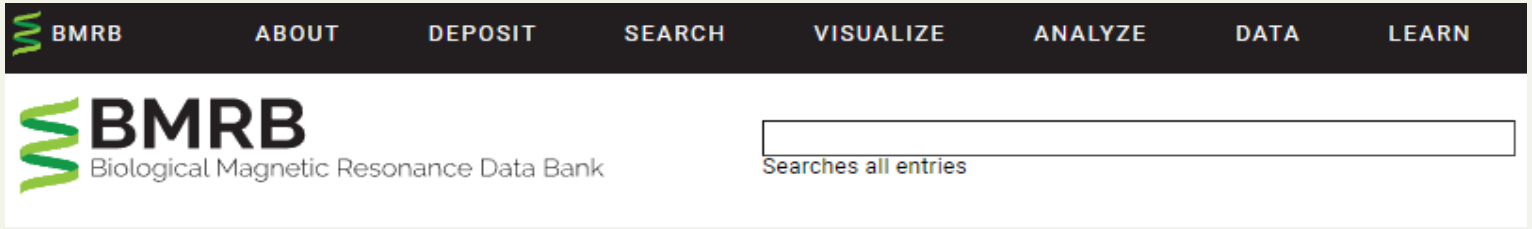
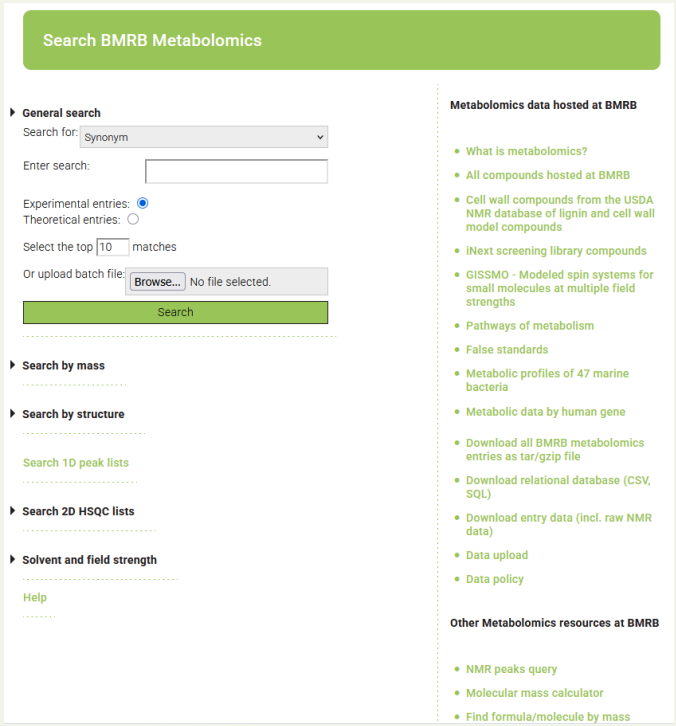


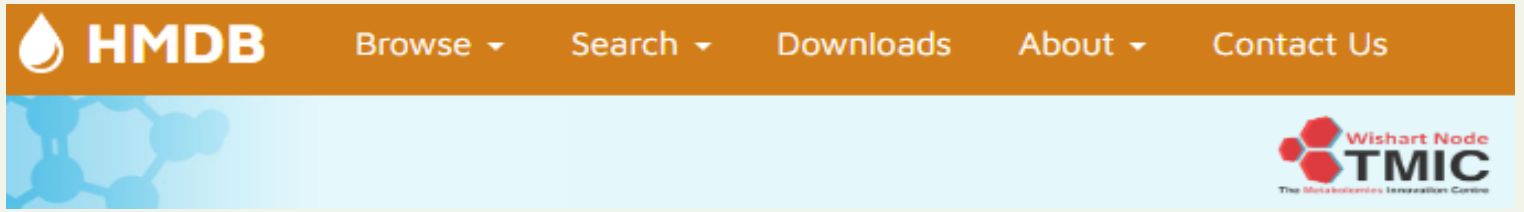


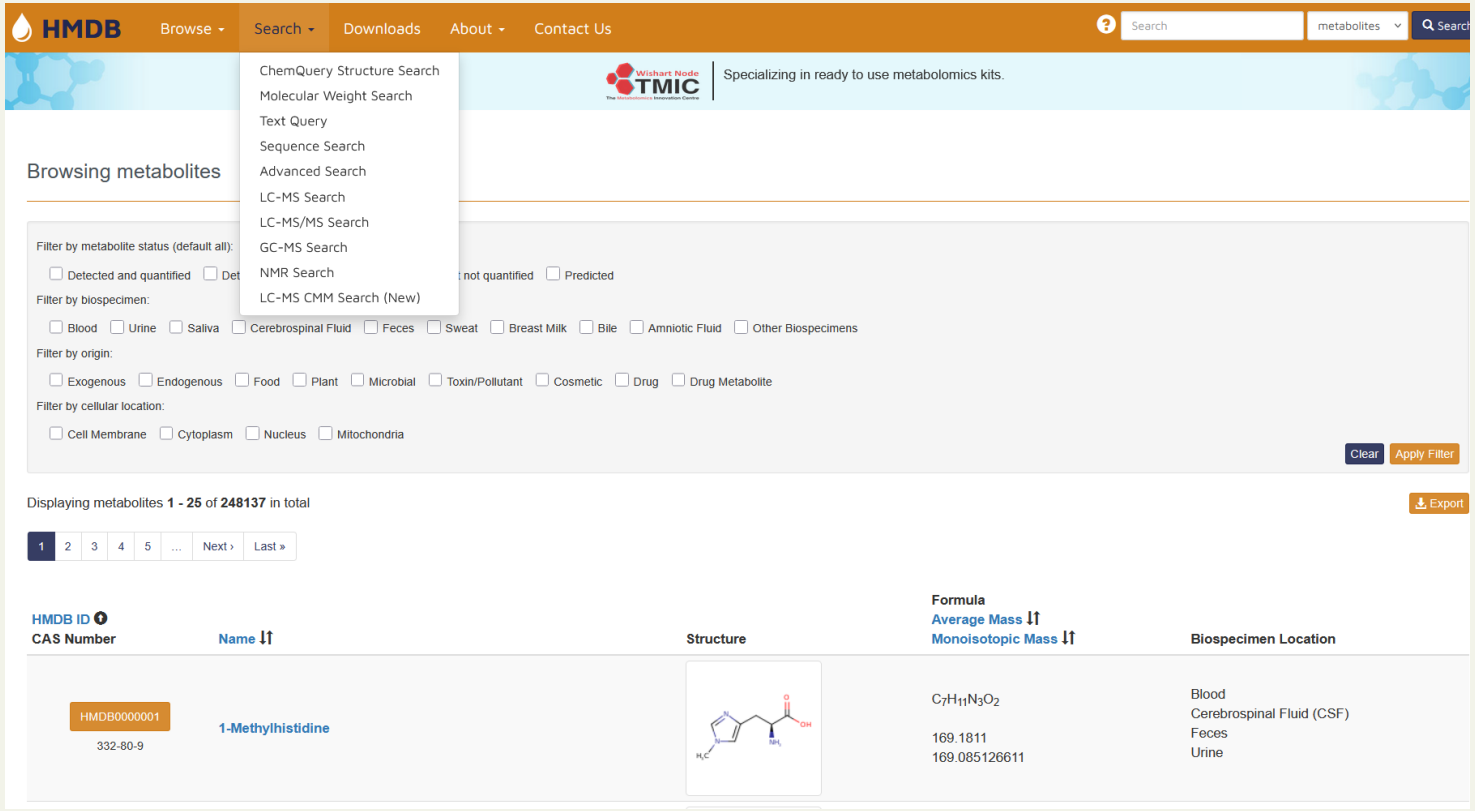
bmrb.io/metabolomics/ Hoch 2023 doi.org/10.1093/nar/gkac1050

2716 Entries

Reference Databases (metabolites / natural products)

- Reference databases:
  - Chemspider can view spectra but not download?
  - BMRB 1D & 2D data available for download
  - **HMDB** 1D data available for download
  - BML data available for download (registration required)
  - MetaboLights
  - CASMDB / CCPN

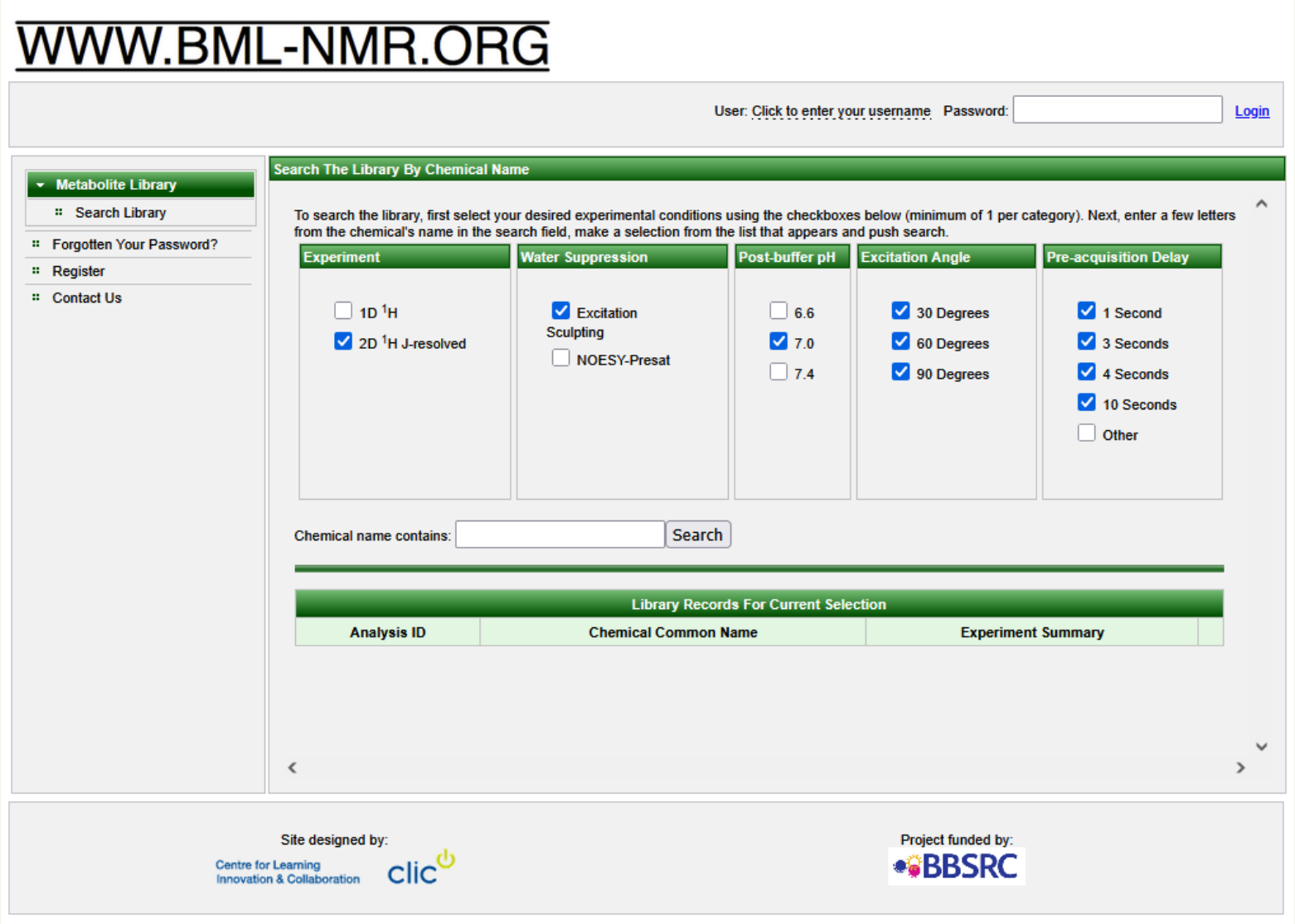




hmdb.ca/metabolites Wishart 2022 DOI:10.1093/nar/gkab1062 **220945 Entries**

Reference Databases (metabolites / natural products)

- Reference databases:
  - Chemspider can view spectra but not download?
  - BMRB 1D & 2D data available for download
  - HMDB 1D data available for download
  - **BML-NMR** 1D & JRES data available for download (registration required)
  - MetaboLights
  - CASMDB / CCPN



bml-nmr.org/

C Ludwig 2012 DOI:10.1007/s11306-011-0347-7

208 Standards

Reference Databases (metabolites / natural products)

- Reference databases:
  - Chemspider can view spectra but not download?
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  - HMDB 1D data available for download
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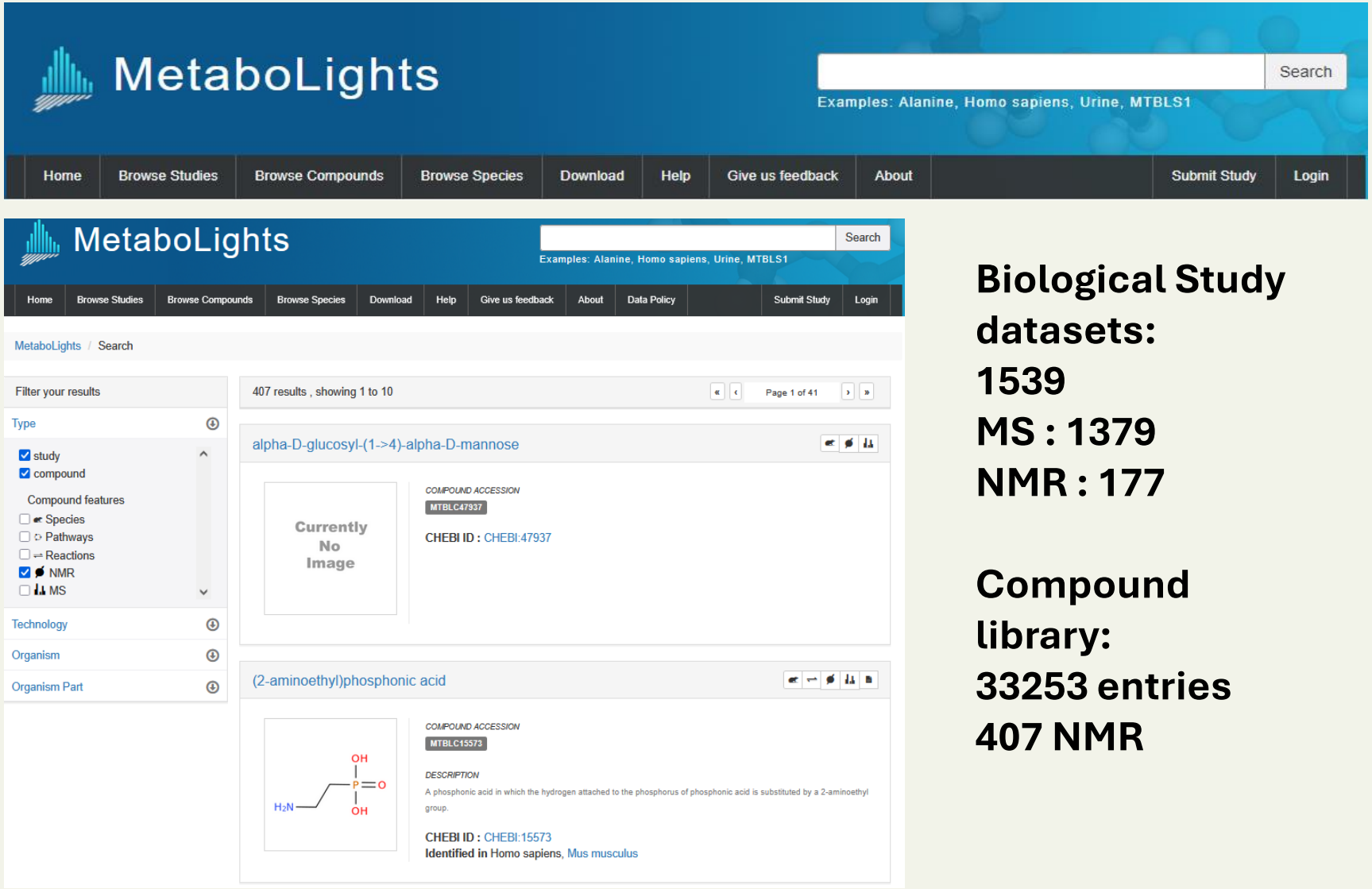
  - CASMDB / CCPN



Biological Study datasets:
1539
MS : 1379
NMR : 177

Compound library:
33253 entries
407 NMR

ebi.ac.uk/metabolights

Yurekten 2023 www.doi.org/10.1093/nar/gkad1045

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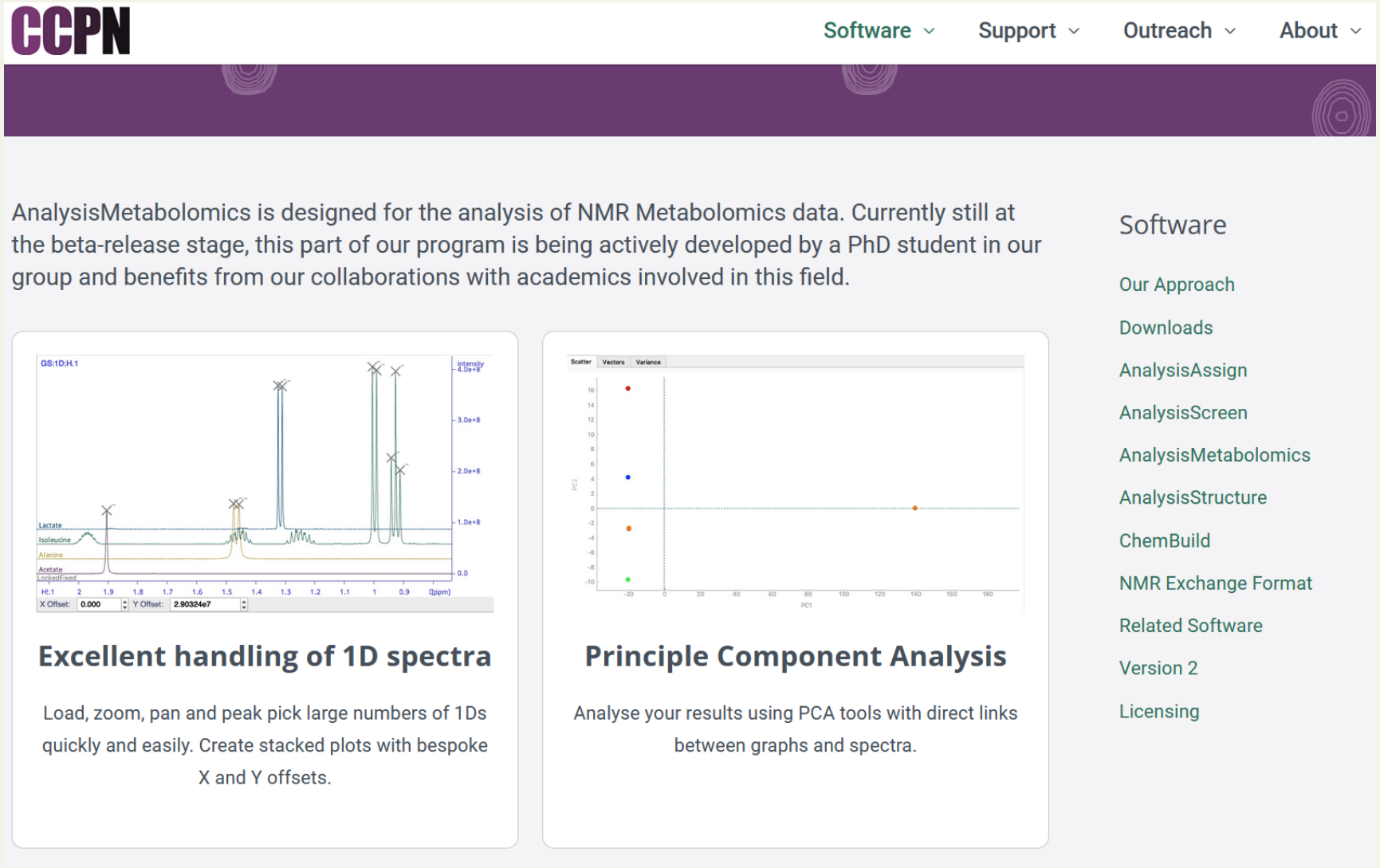
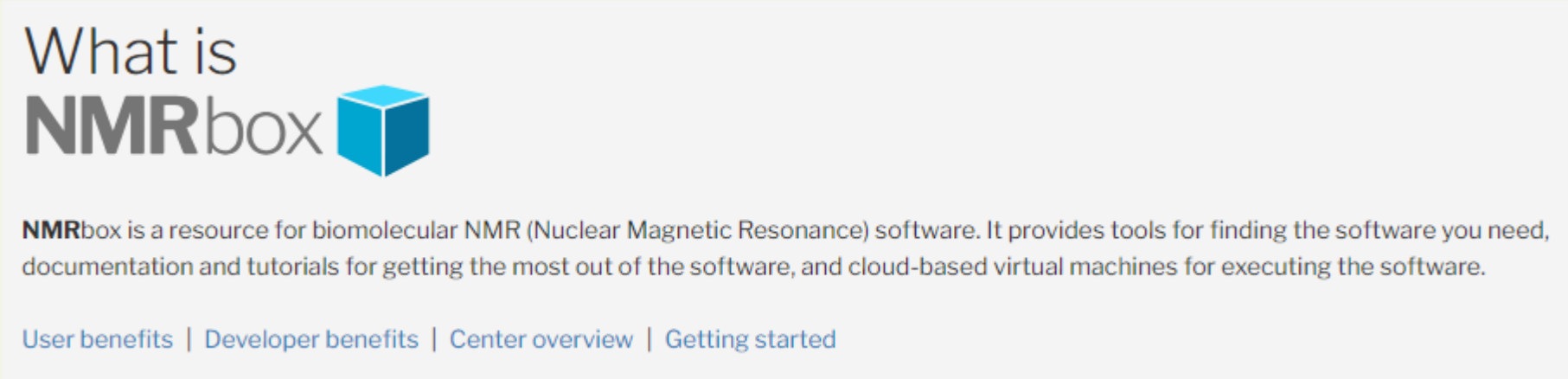


https://ccpn.ac.uk/software/analysismetabolomics/

Virtual Machine – NMRbox / NMRhub

- Virtual machine using VNC viewer (free)

- nmrbox : virtual machine for virtually every nmr software (old, new, and helpful for those with high computational demands)

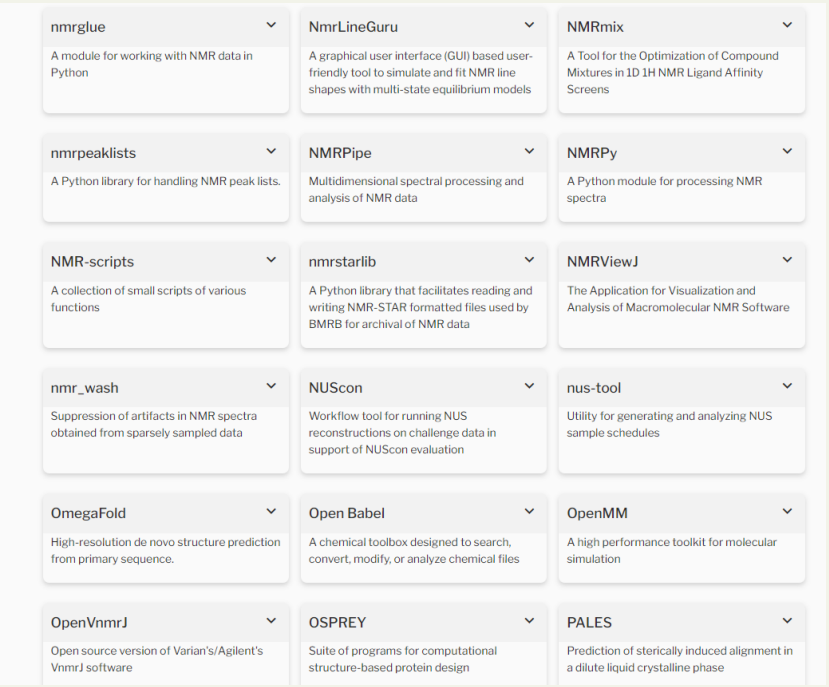


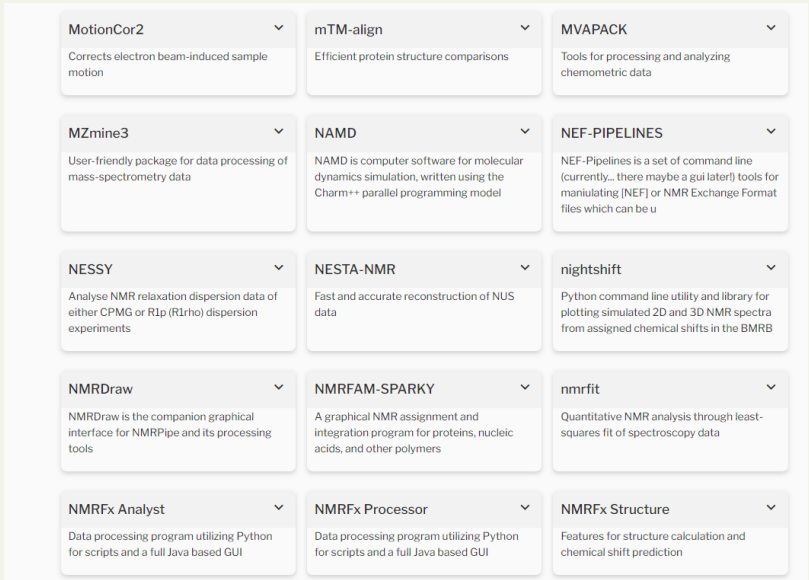
- University of Liverpool & ResoN8 training - Getting started:
  - https://www.youtube.com/playlist?list=PLCfsv-WgdV7KGiPxMGMMjsLAkRnBFQ7F6

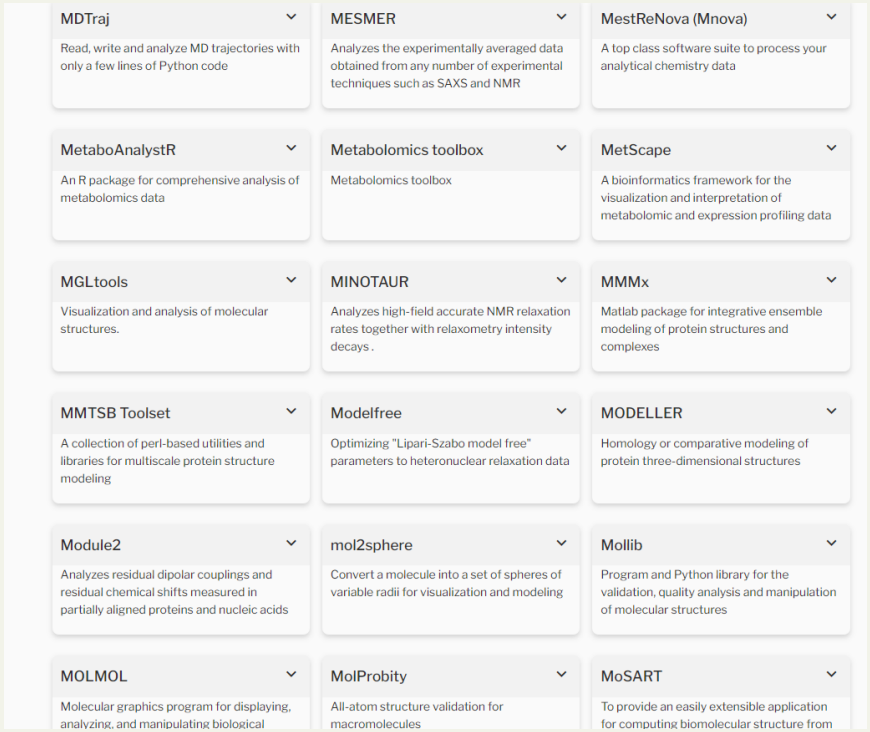
UConn © 2025 NMRbox - Funded by NIH/NIGMS 1P41GM111135

Virtual Machine – NMRbox / NMRhub

- Virtual machine using VNC viewer (free)
- nmrbox : virtual machine for virtually every nmr software (old, new, and helpful for those with high computational demands)
- 274 software packages







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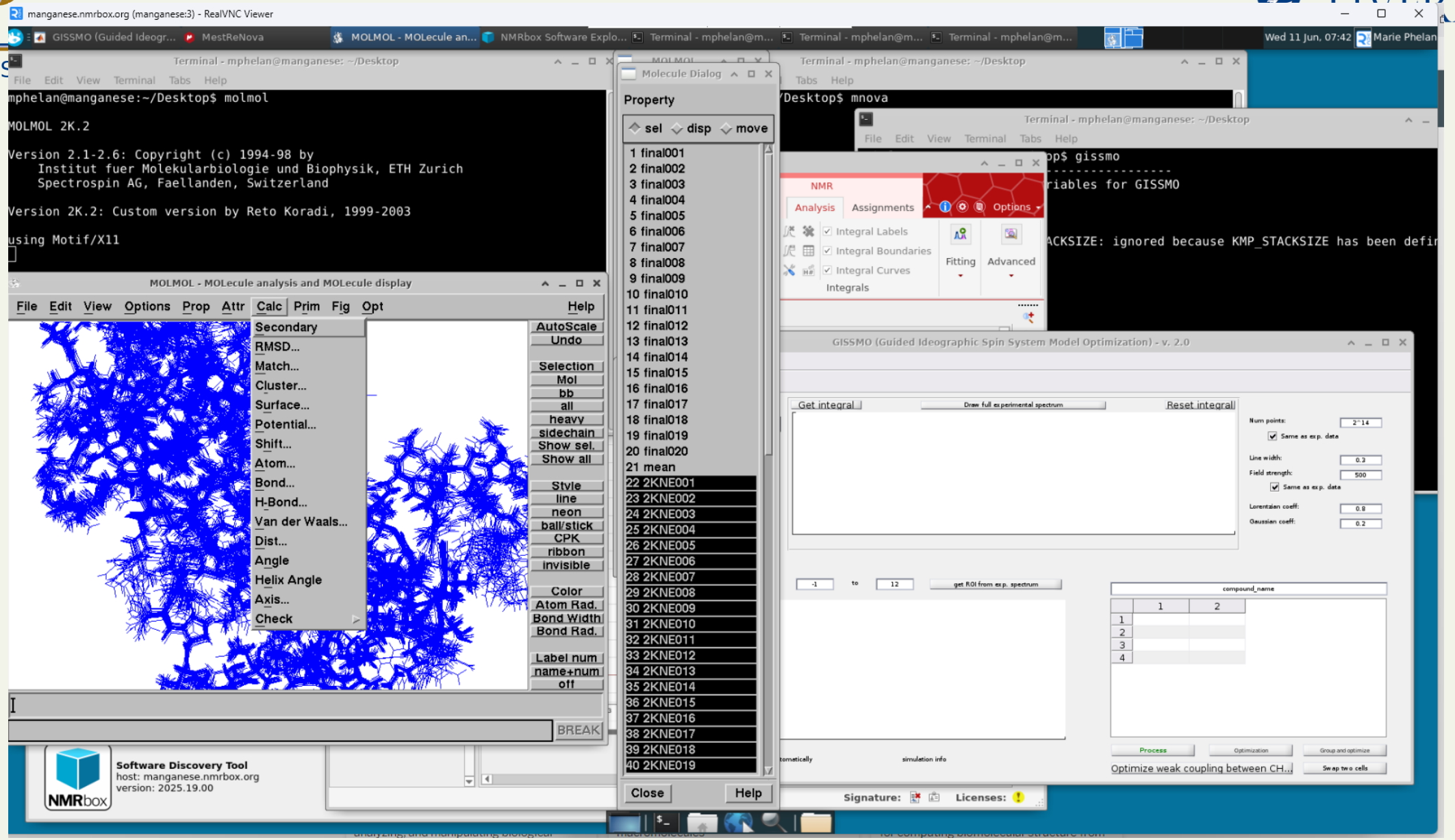
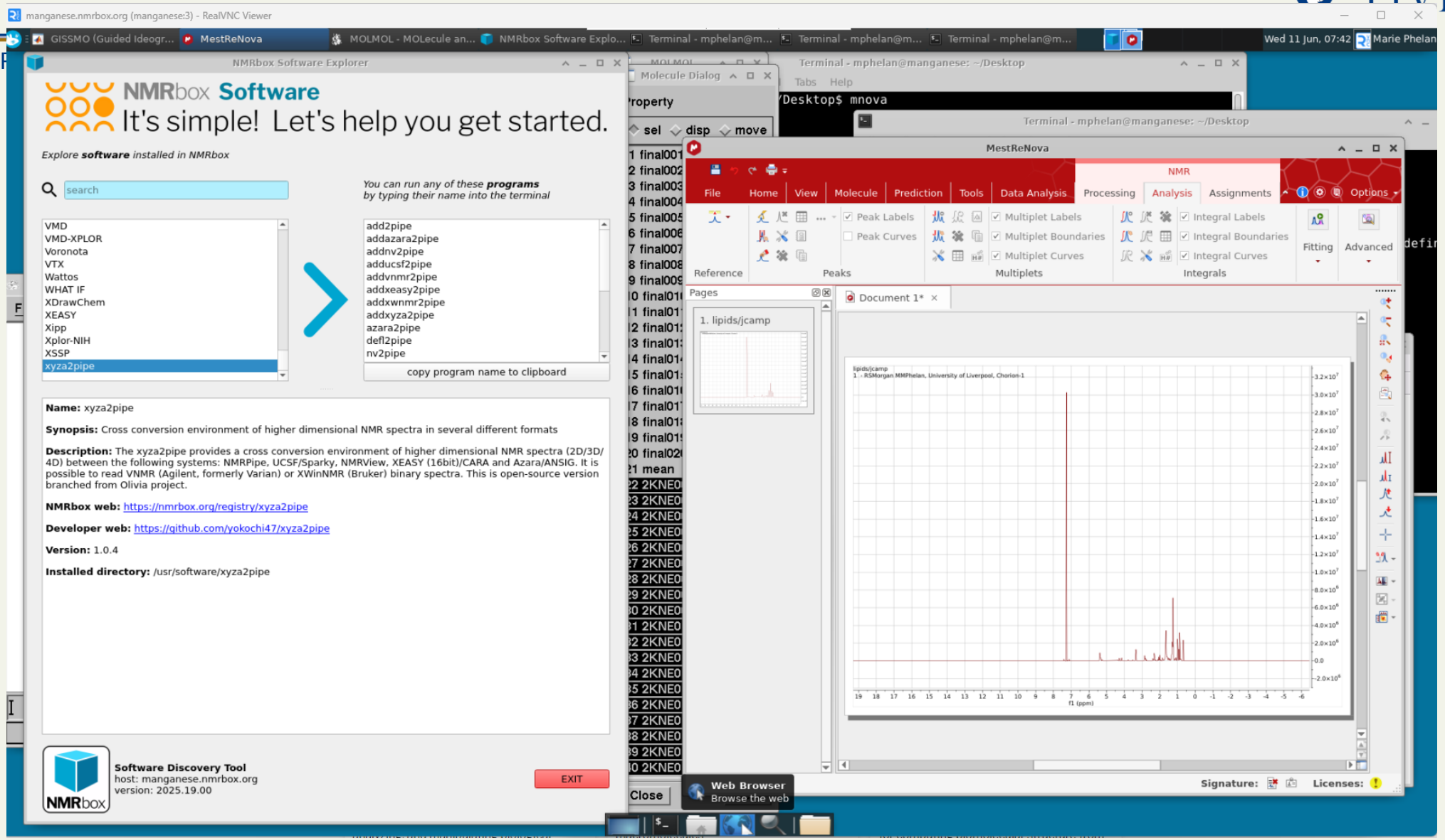
Virtual Machine – NMRbox / NMRhub



www.nmrhub.org | www.nmrbox.org

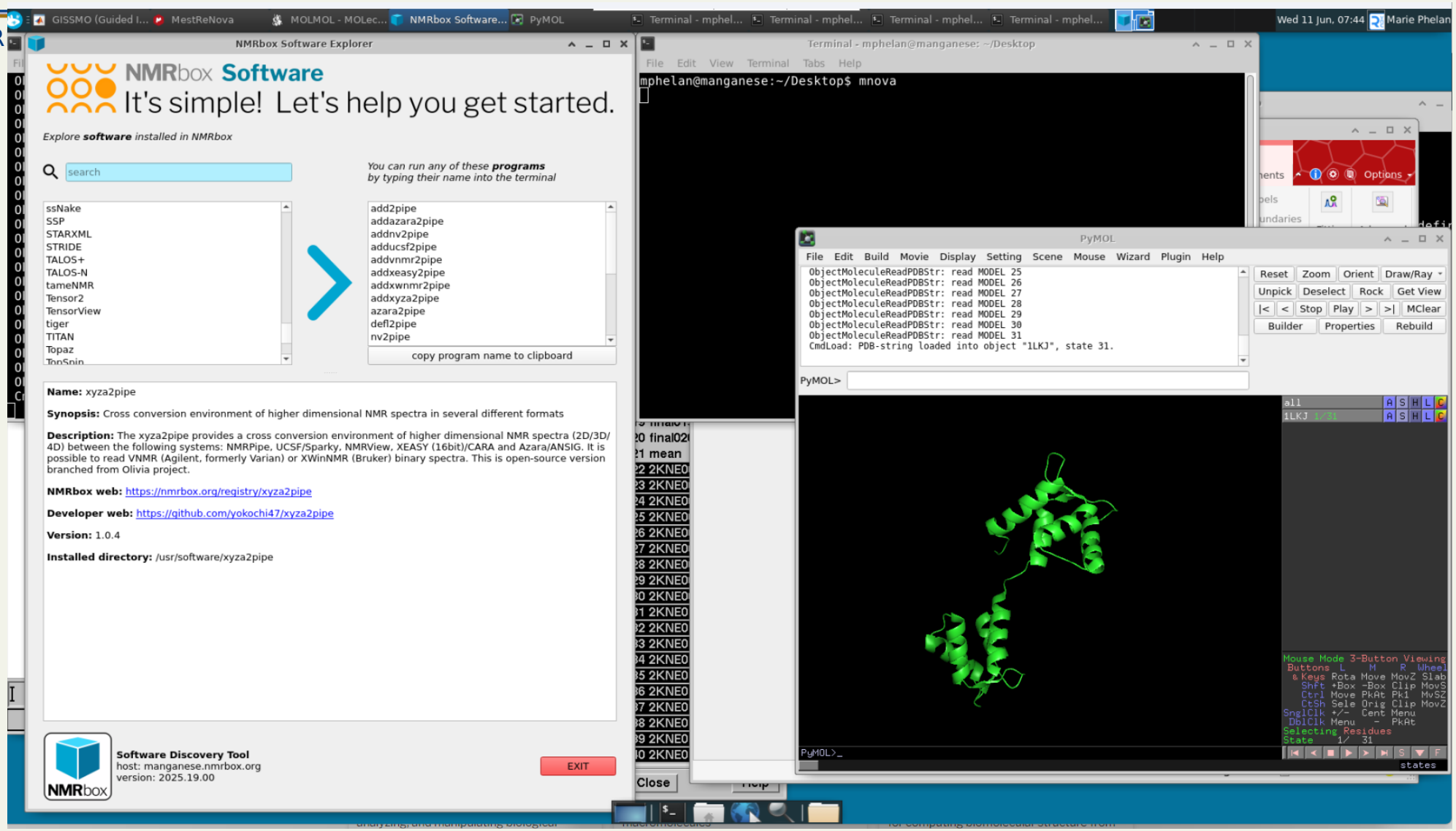
UConn © 2025 NMRbox - Funded by NIH/NIGMS 1P41GM111135

Virtual Machine – NMRbox / NMRhub



UConn © 2025 NMRbox - Funded by NIH/NIGMS 1P41GM111135

Virtual Machine – NMRbox / NMRhub



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Teaching Theory

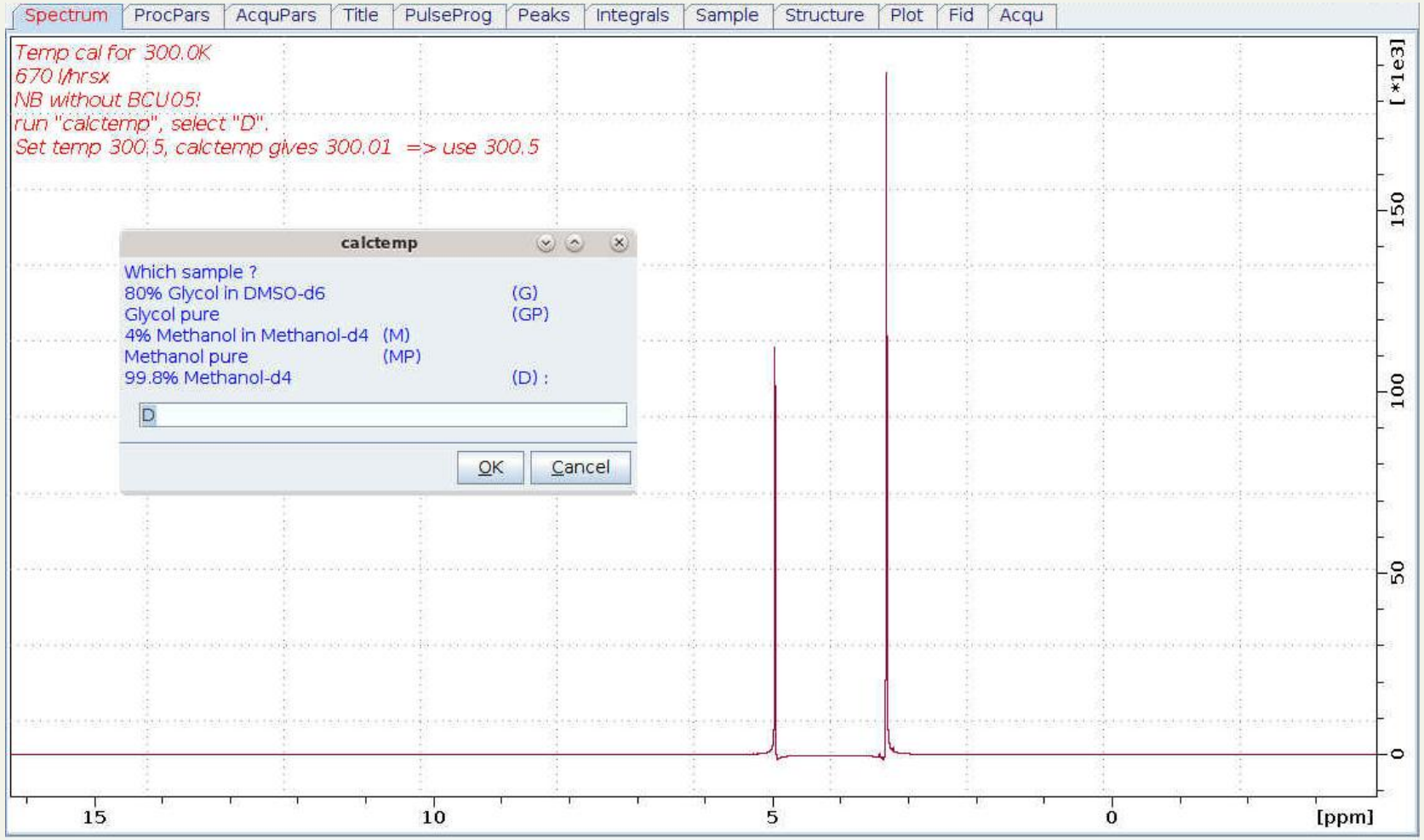
- Teaching theory to biologists: reson8
  - Reson8 NMR week – annual 2 day residential (Northern 8 universities)
    - **Core NMR Concepts** (spin, nuclei (and their frequency ranges), pulses, coupling)
    - **HSQC** (^{1}H sensitivity, spin echos, 2D acquisition, energy level, vectors and product operators)
    - **Relaxation** (effect of rel'n on experiments, T1 & T2 manifestations in pulse sequences, typical T1 T2 values for 1H 13C 15N proteins, T1 & T2 relationship to protein size)
    - **Processing /data analysis** (window functions to improve spectral quality, role of TD, SI, FT and phasing).
  - Local set-up, acquisition, processing practicals

https://sites.google.com/view/reson8nmr

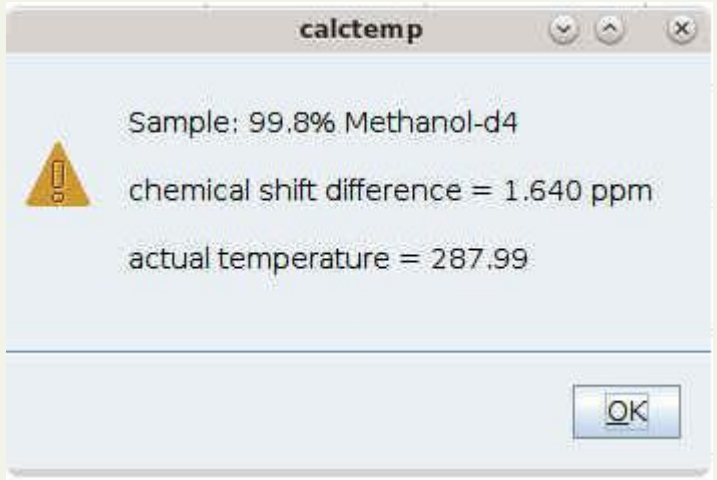
- Linux bootcamp
  - Useful commandline operations
  - Local file organisation (including accessing via VLAN local network)
  - Awk, perl and Python basic scripting

Topspin – Favourite au programs

- calctemp



Findeisen 2007 DOI 10.1002/mrc.1941



Methanol standard
(99.8% MeOD)
Bruker Z10627

Topspin – Favourite au programs

- au_lc1d

BRUKER

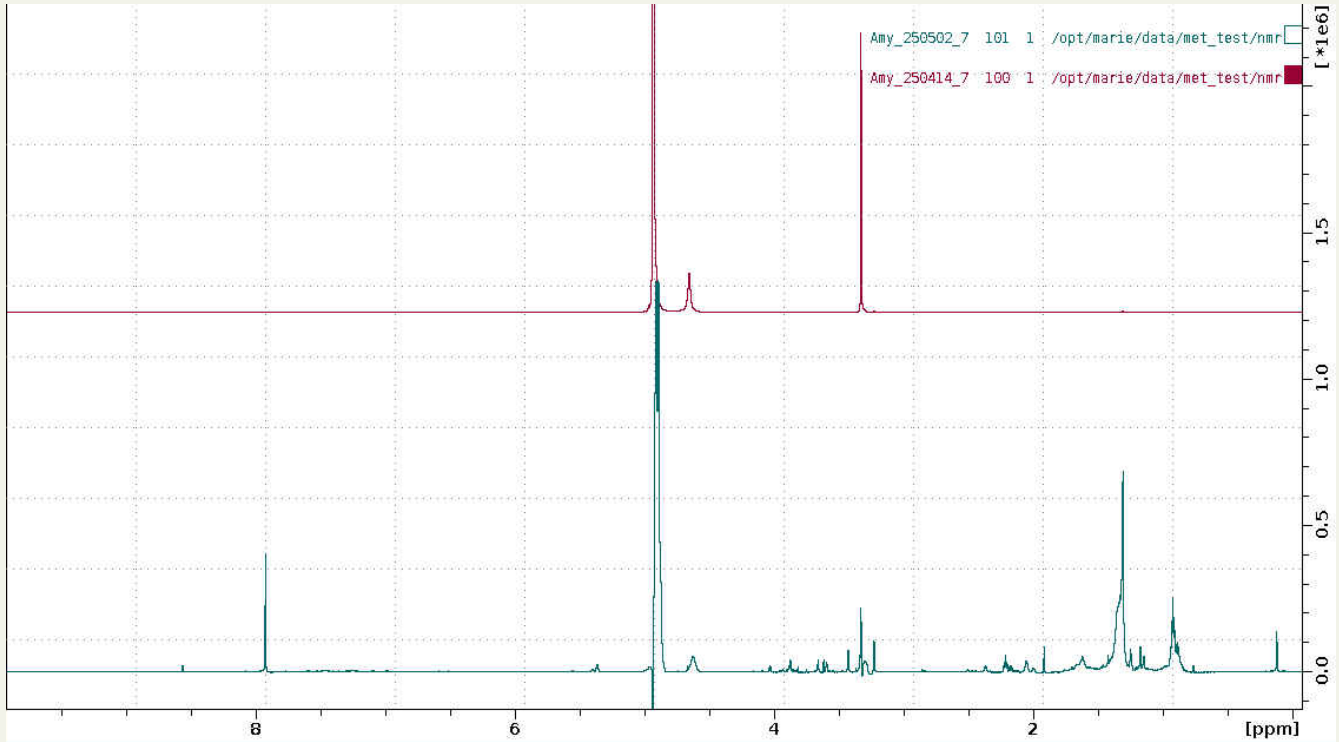
TOPSPIN 3.5 & LC-NMR

Software Manual

Version 140903

LCNMR

Written by
Dr. Ulrich Braumann
ulrich.braumann@bruker-biospin.de



5.6 AU-Program au_lc1d: 1D spectra with solvent suppression

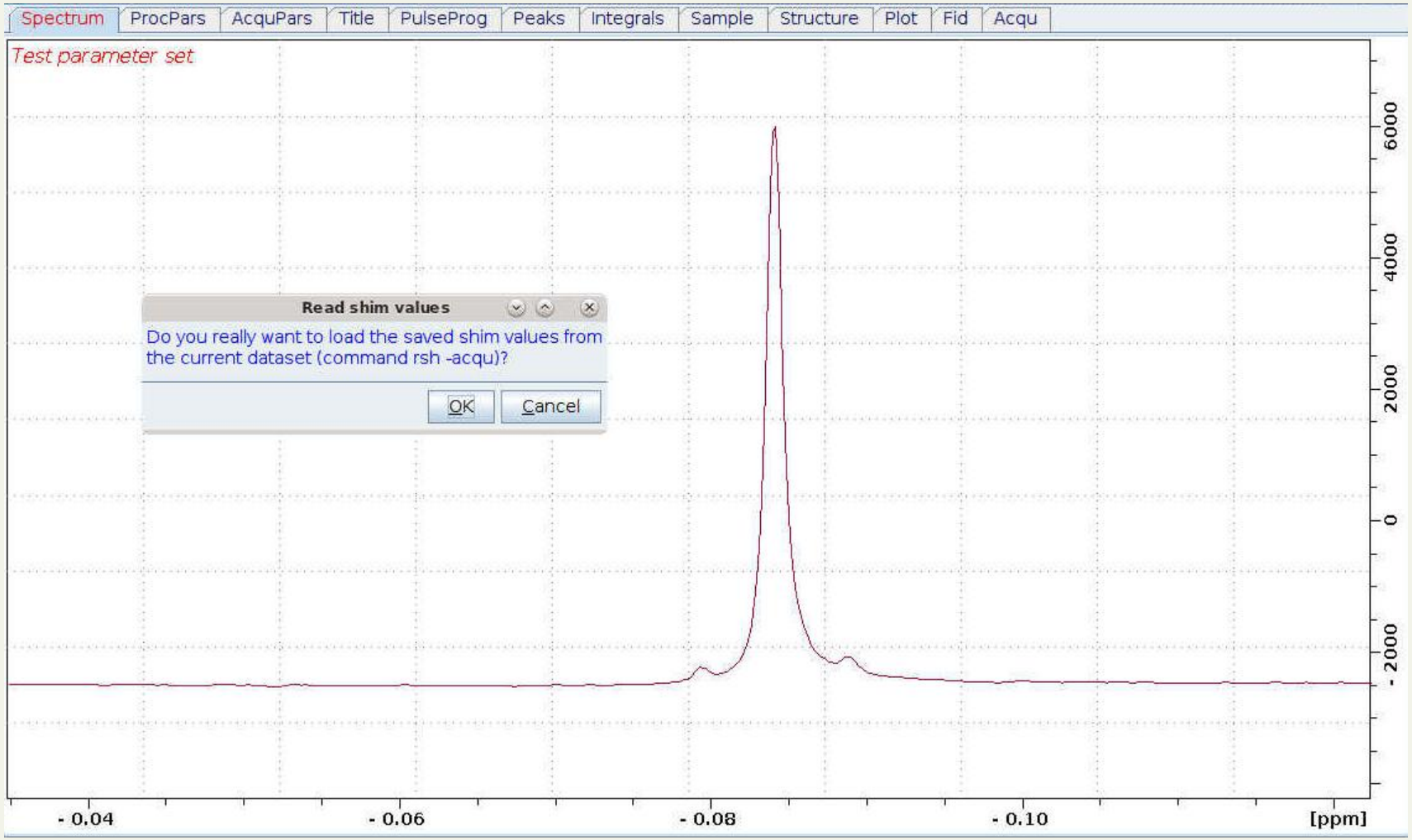
The AU program **au_lc1d** controls the acquisition of 1D spectra with all kinds of solvent suppression techniques. The acquisition can be carried out step wise with signal to noise control.
In fact **au_lc1d** use the AU-programs **lcprep** to run a preparation experiment and prepares all parameters for the solvent suppression using the AU-program **lcsetsup**.
The program can be started on any 1D data set with xau au_lc1d or normally with xaua.

Actions carried out by au_lc1d

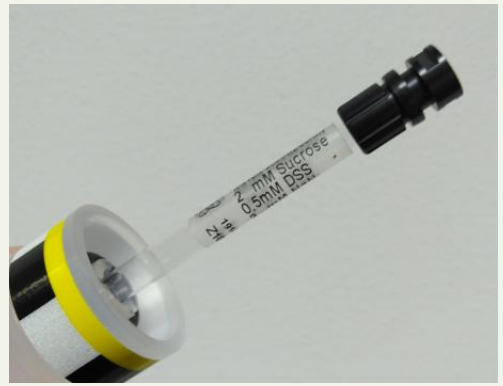
- Run the AU-program **lcprep** to acquire a 1D solvent search spectrum.
- Run the AU-program **lcsetsup** to find the desired number of solvents and adjust all parameters for solvent suppression on the current data set.
- Determine the RG for the experiment. Either a fixed **RG** can be used, or RGA is executed and the result is correct by **CNST29**.
- The acquisition is always started with the maximum number of scans, determined by **NS** and **TD0**.
- If the signal to noise control is activated the SINO is first checked after **L31** scans and further in iterations of **NS** scans until **TD0** loops or the requested SINO is reached.
- A basic processing is executed after each increment, the signal to noise is determined with AU program lcsino.

Topspin – Favourite au programs

- rsh -acqu



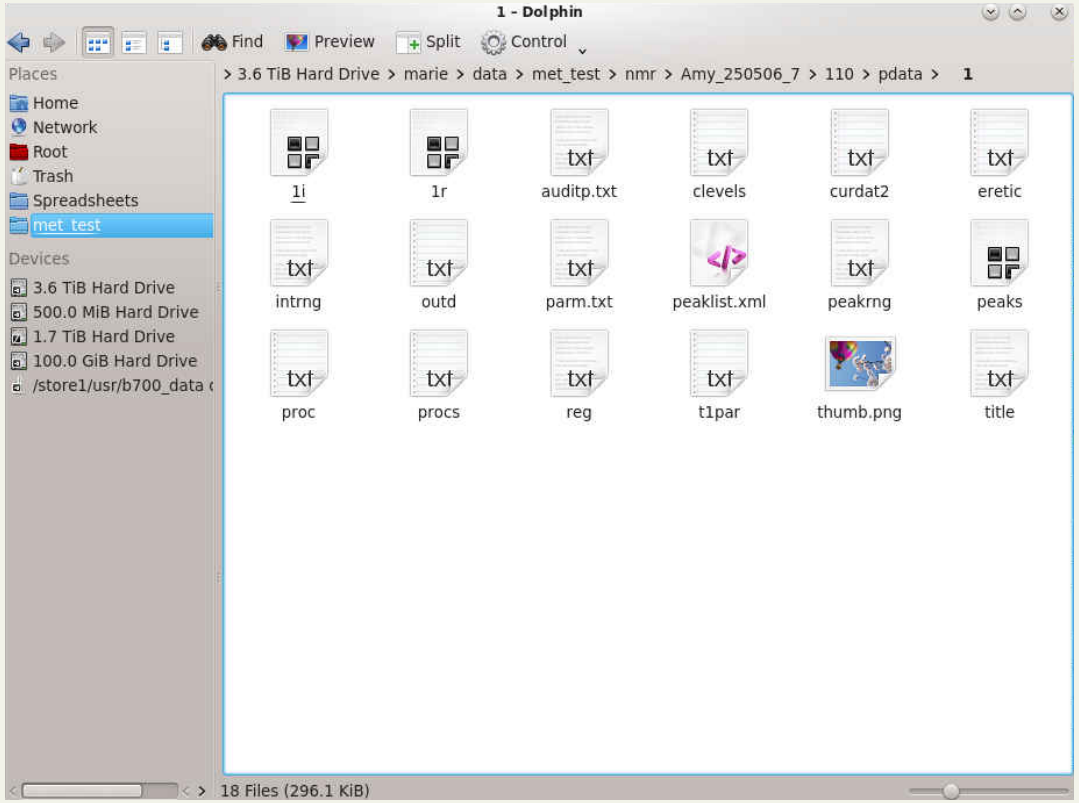
Good reference material for 3D shimming (topshim gui and select 3D)

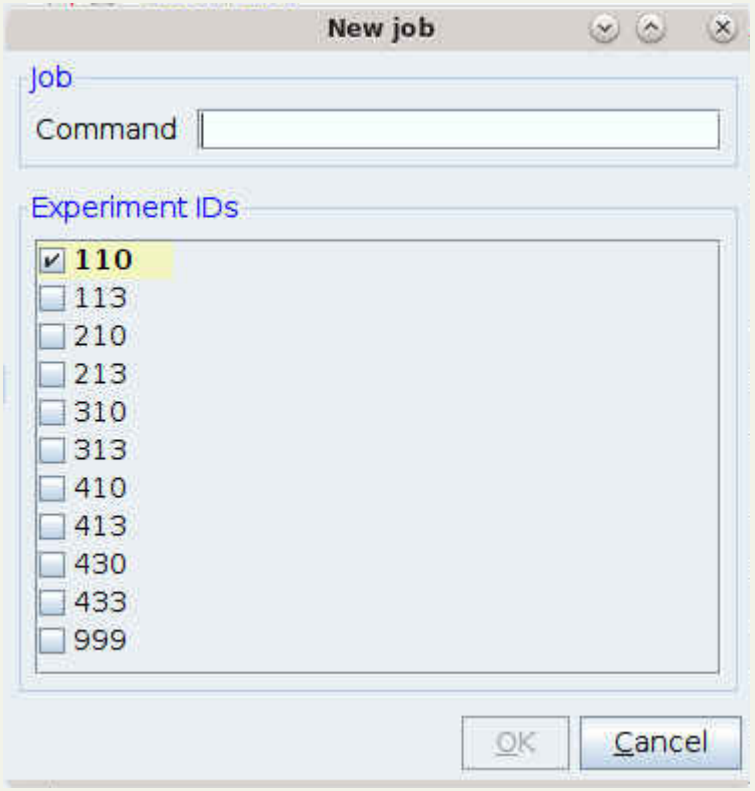


Sucrose & DSS standard
(10% D_2O 90% H_2O)
Bruker Z10902

Topspin – Favourite au programs

- expl & qumulti (thanks Nikita!)





Some manuals here: https://www.bruker.com/protected/en/services/user-manuals/nmr/general.html
More supplied with Bruker Topspin install

Practical Things

- SampleJet / high throughput tube usage
  - Use Bruker caps (we recycle caps)
  - Use of 108mm and 178mm tubes
  - Soak in decon90 over night and wash
  - Home made multi-tube (10) washing station
  - https://rgmetab.github.io/NMRmetab/Miscellaneous/Tube%20washing/

Practical Things

- SampleJet
  - Training to prevent incorrect operation
  - Recalibrate periodically (every 6 months)
  - Replace gripper periodically (1-2 years)

- User operation
  - https://www.youtube.com/watch?v=MnFWzC7FDtE

- Maintenance & Calibration etc:
  - https://www.youtube.com/playlist?list=PLCfsv-WgdV7JcOa89KUNC-rzHiF-1kVlk

Practical Things

- Helium recapture –
  - UoL-specific written manual available

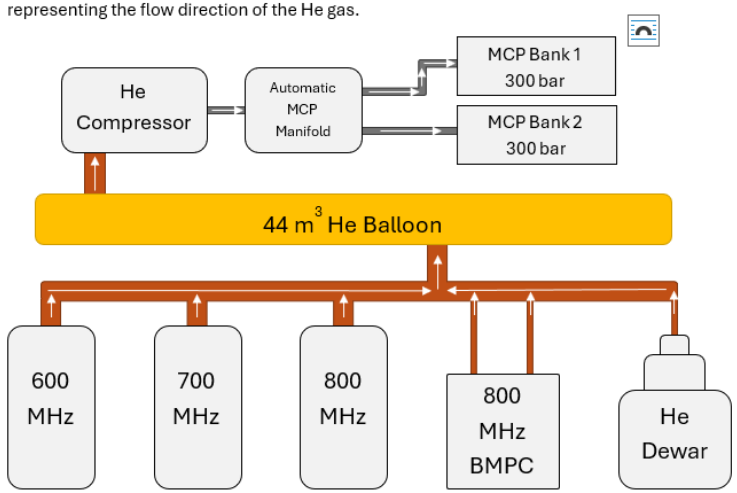
He Recovery System SOP

Overview

Helium Recovery system aims to collect the He boiloff from the magnets during the normal operation and fills and compress them into cylinders to be collected by BOC for liquification. Liquified He will then be purchased by the facility to be used during helium fills. This scheme ensures the facility required 500 L allocation from BOC during shortage periods.

High level system architecture

The recovery system comprises of 6 collection points into a balloon that acts as intermediate reservoir which is then transferred into two manifold cylinder pallet (MCP) banks using a compressor. The switching between the MCP banks is performed by an automatic manifold. The overall schematic of the system is shown below with white arrow representing the flow direction of the He gas.



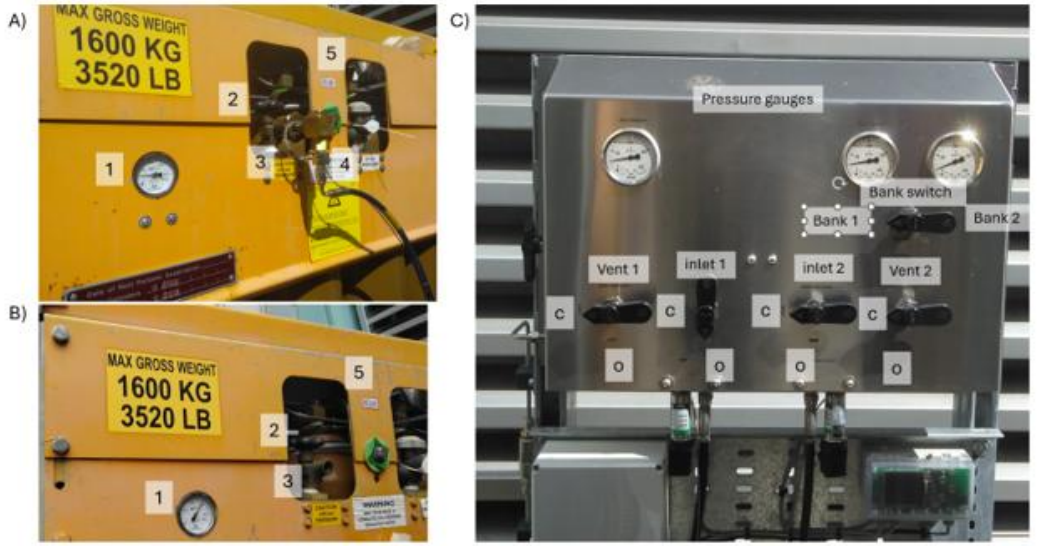
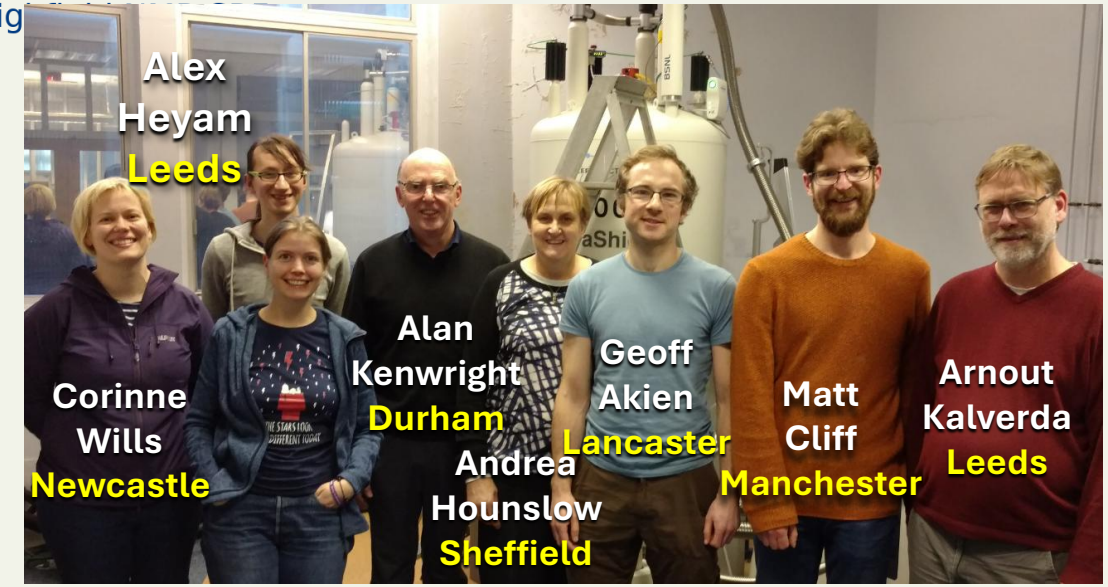


Figure MCP A) bank connected and not ready for collection, B) bank disconnected and read for collection C) external control panel –valve configuration shown for collection in bank 1.

Acknowledgments

RESON8 | NMR Group

UNIVERSITY OF LIVERPOOL



Reson8 (past & present)

Tips & tricks
Resources / links

Peter Gierth
(then Bruker)

Mark Howard
(now Bruker)

Robin Stein
(Bruker)

Juraj Bella
Edinburgh

Rudi Grosman
(now Bruker)

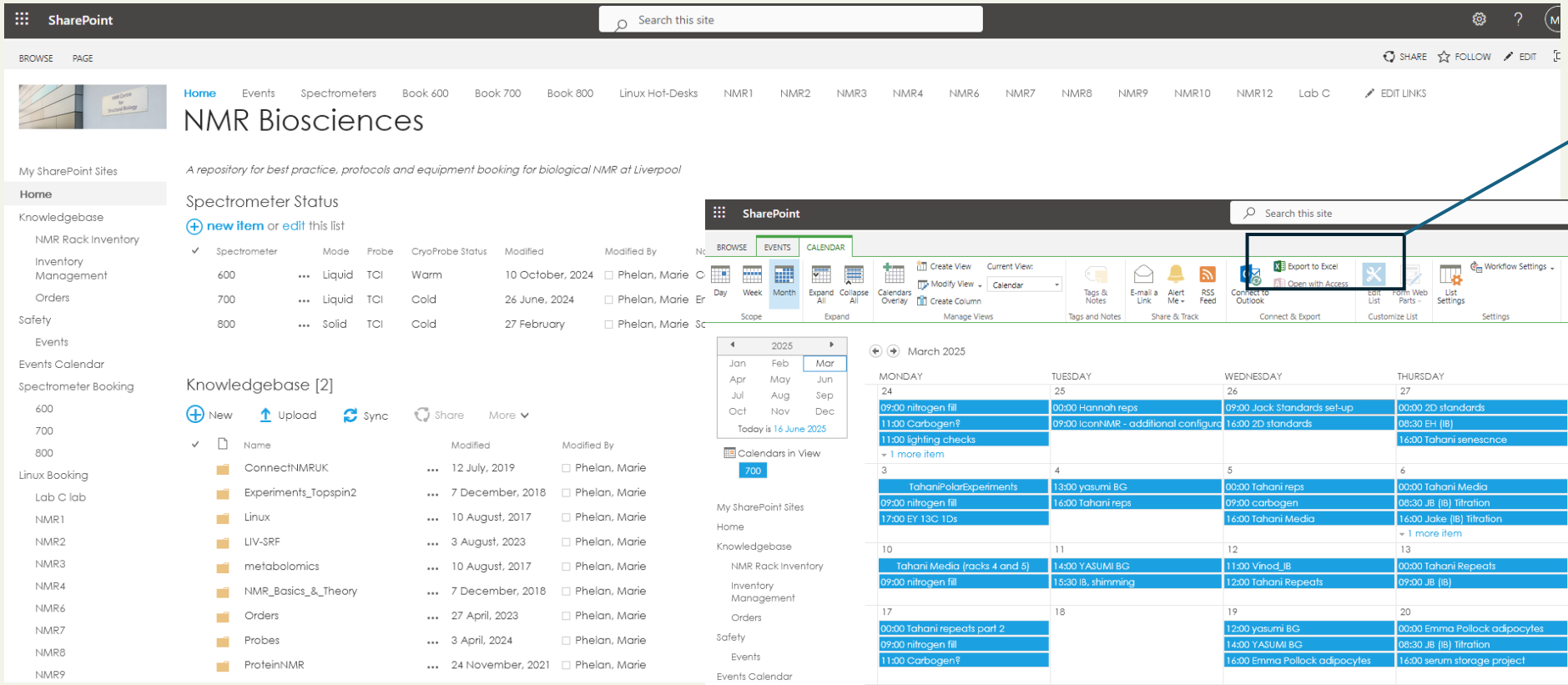
Liv-SRF
Liverpool
Shared
Research
Facilities

UKMRM
RSC
UCL
thanks Nikita Harvey

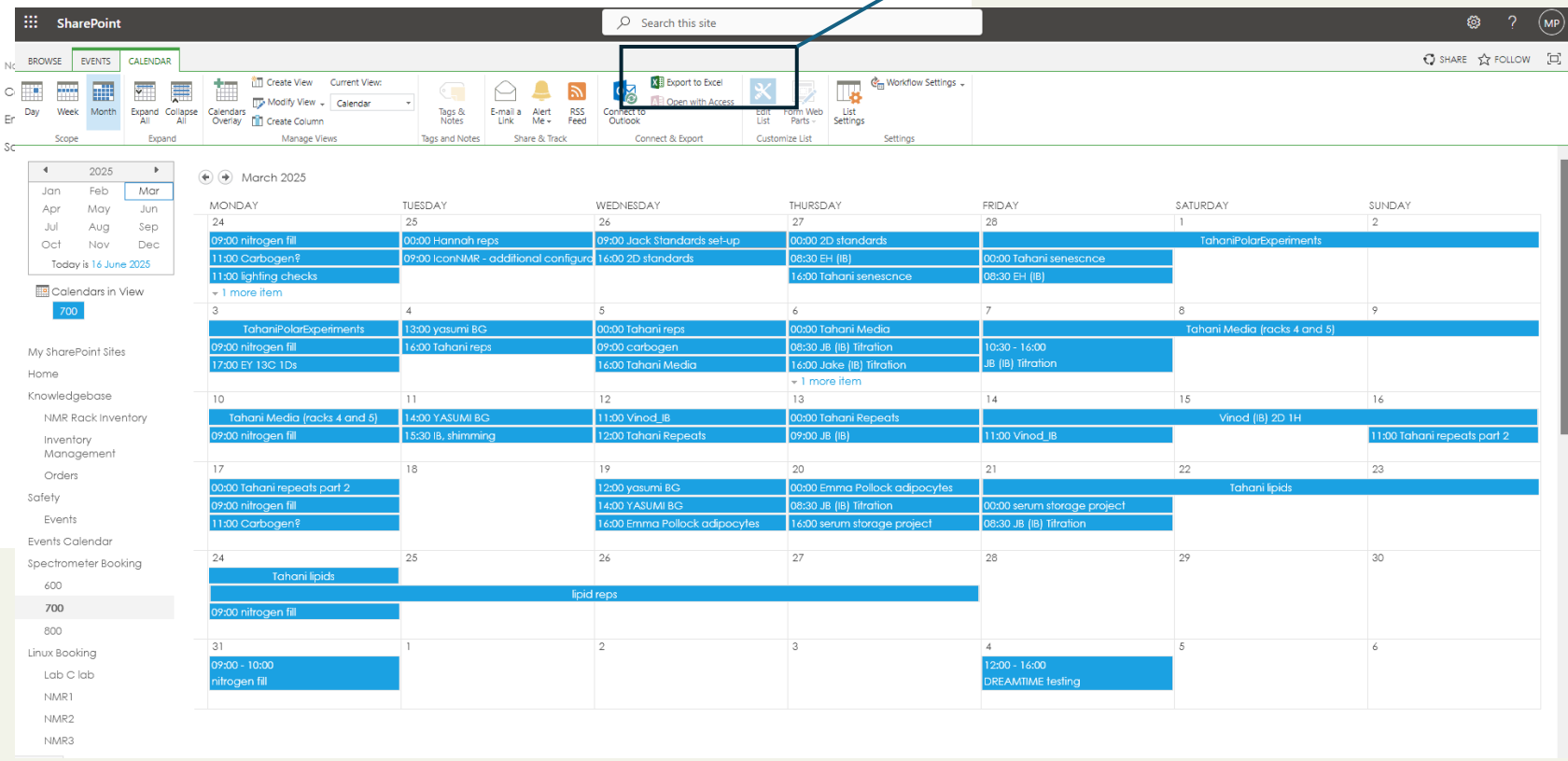
Most Importantly all this acquired knowledge is thanks to the openness and expertise of the NMR community!

Practical Things

- Booking / managing
  - Sharepoint

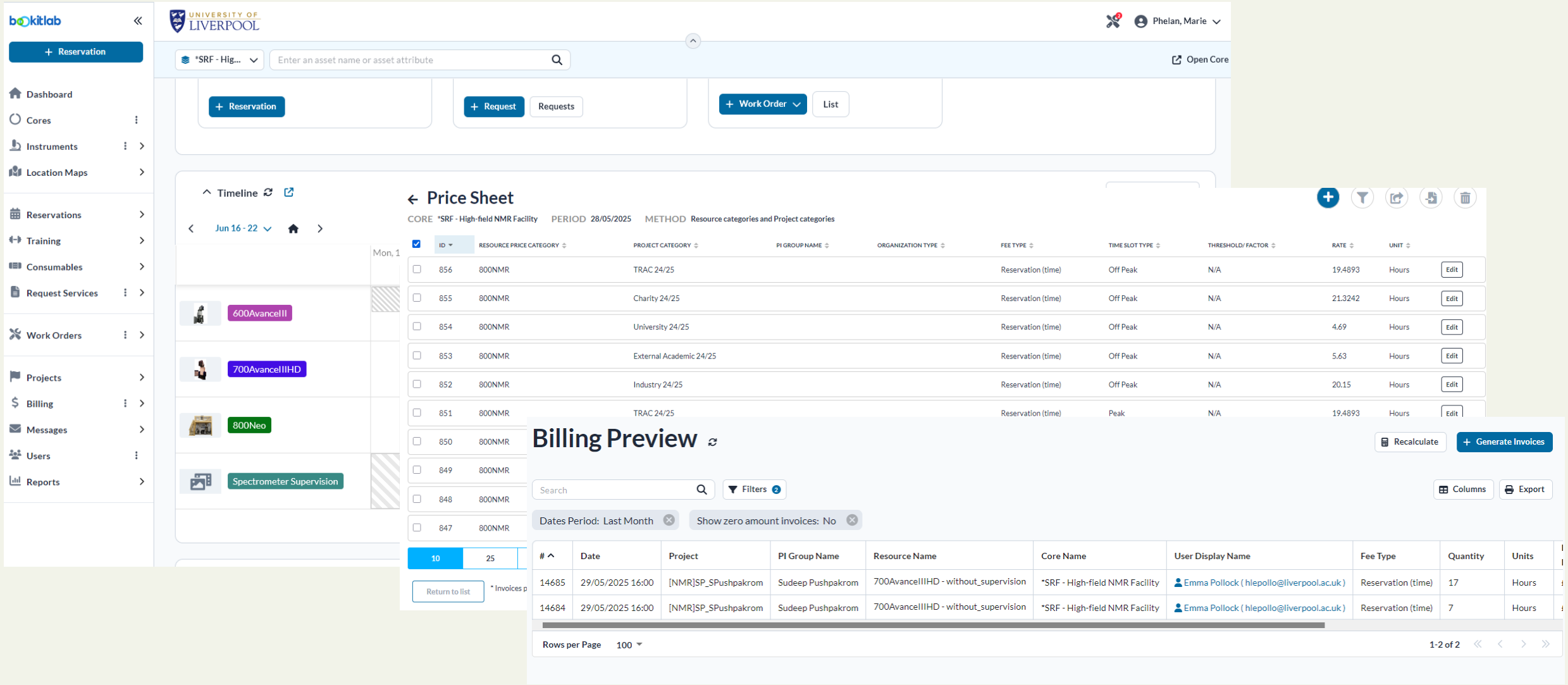


Export as spreadsheet



Practical Things

- Booking / managing
  - BookitLab (Prog4Biz)



Practical Things

- Safetynet.liv.ac.uk
  - Safety Induction, training etc..