Vivat Prodigy ? ☹

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500 & 400 MHz nitrogen cooled cryoprobes

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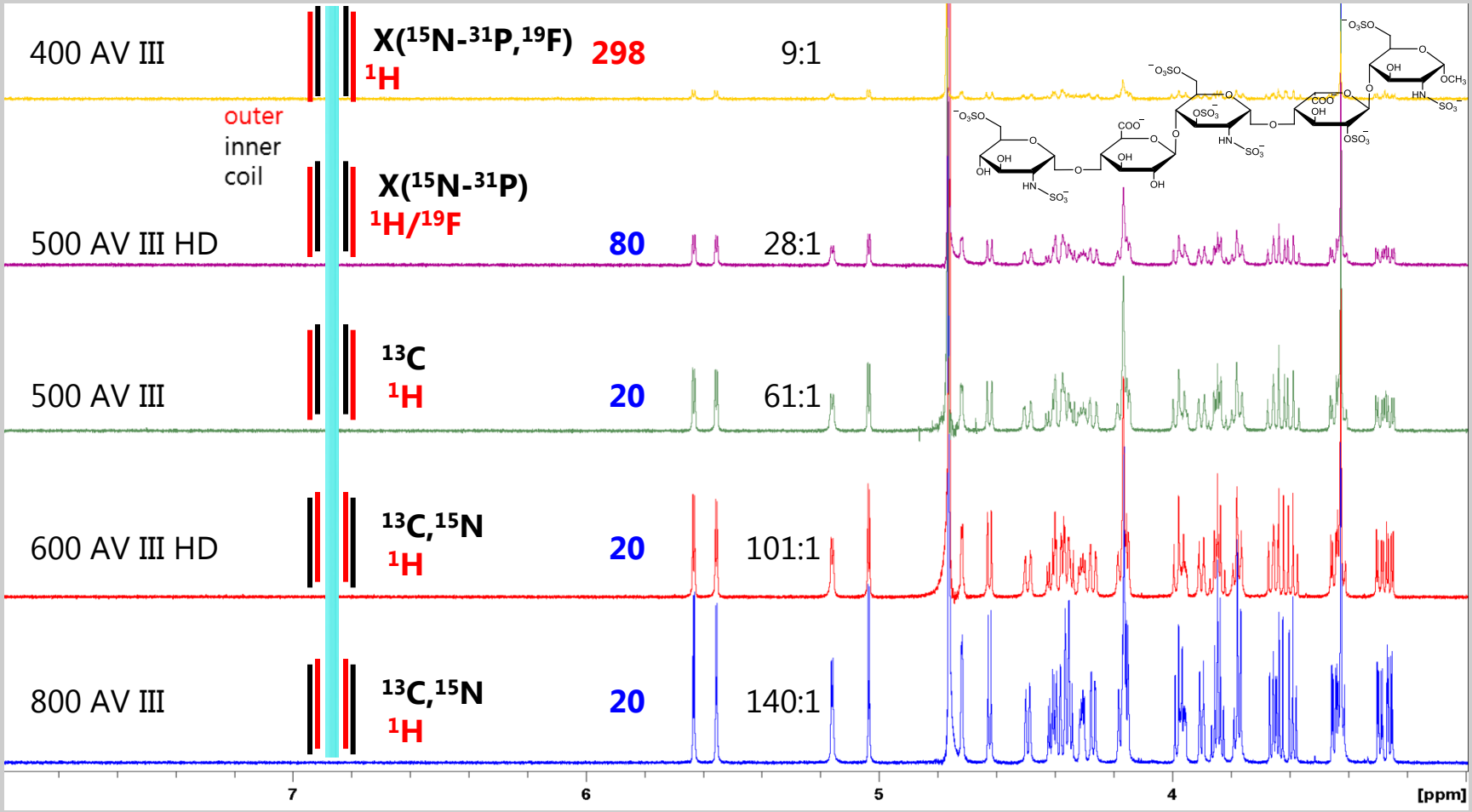
- 500 installed in Dec 2013
- used from Jan 2014
- 400 installed in May 2014
- 5 mm Multinuclear observe
- H&F&D outer coil
- VT 0-135 deg C
- Auto tune/match
- 400 fitted with Cryofit – flow cell equipment

^{1}H sensitivity

^{1}H frequency /MHz	Probe geometry	Coil temp /K	Signal-to-noise (NS = 1, anomerics)	1.4 mg of Fondaparinux, M_w=1726.77 g/mol (1.5 mM)
400 AV III	X(^{15}N-^{31}P,^{19}F) ^{1}H	298	9:1	
500 AV III HD	outer / inner coil; X(^{15}N-^{31}P) ^{1}H/^{19}F	80	28:1	
500 AV III	^{13}C ^{1}H	20	61:1	
600 AV III HD	^{13}C,^{15}N ^{1}H	20	101:1	
800 AV III	^{13}C,^{15}N ^{1}H	20	140:1	



^{13}C sensitivity

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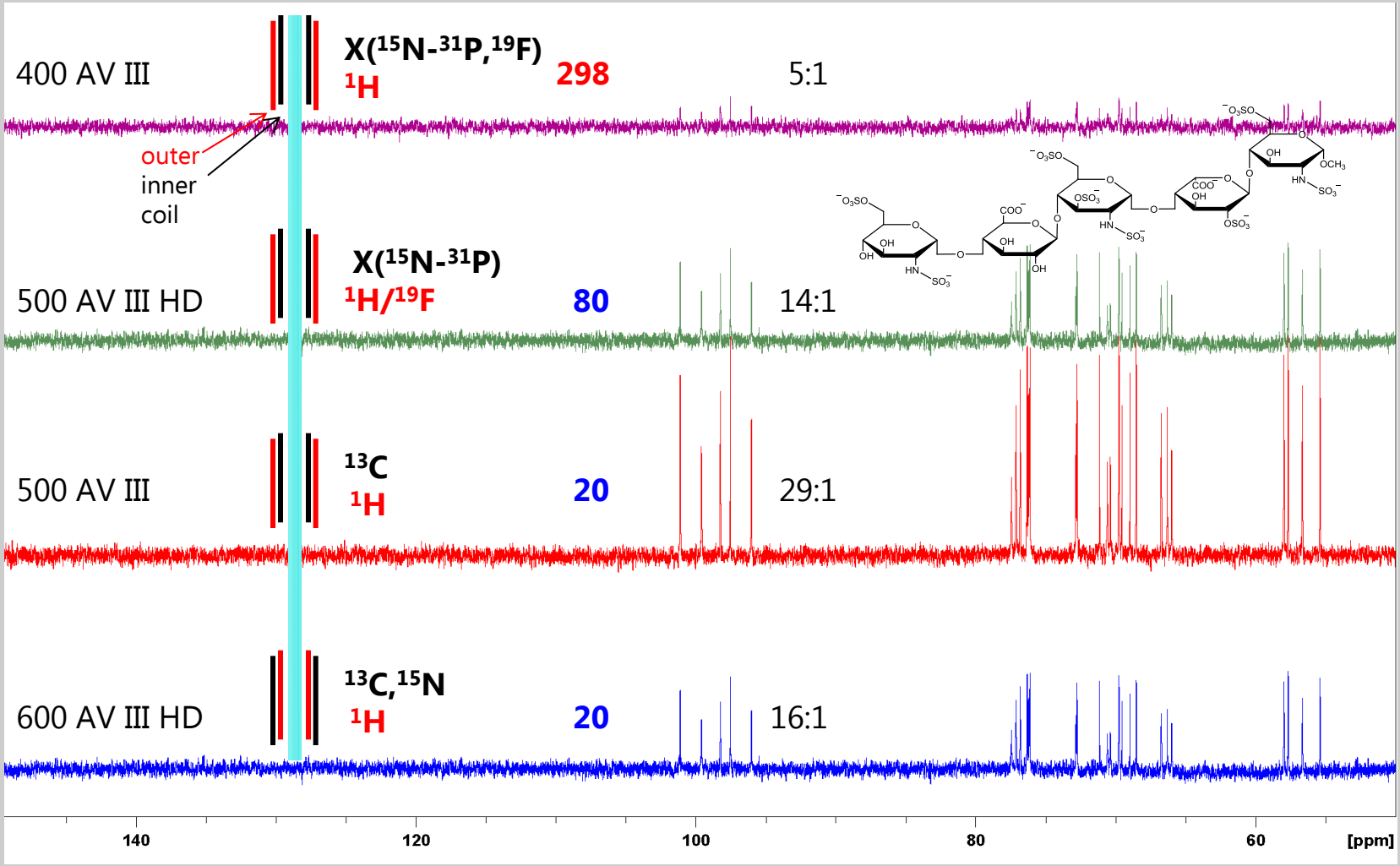
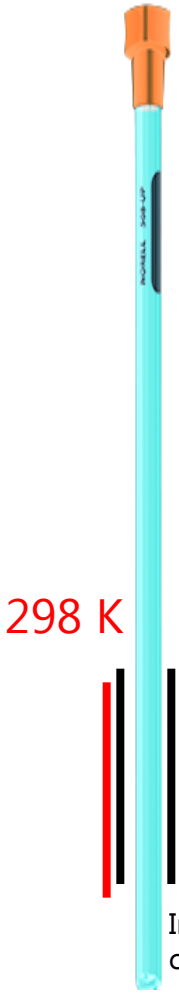
^{1}H frequency /MHz	Probe geometry	Coil temp /K	Signal-to-noise (NS=128 anomerics)	14 mg of Fondaparinux, M_w=1726.77 g/mol (15 mM)



^{19}F sensitivity



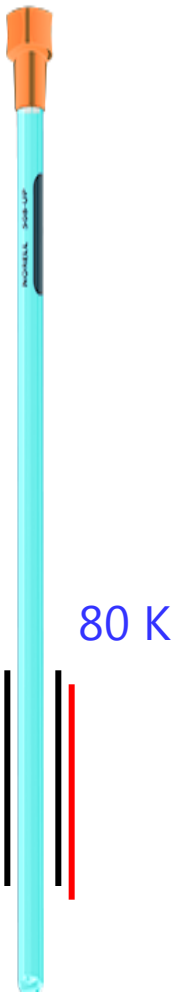
298 K

X(^{19}F)
^{1}H

Inner/outer coil

Ava400 (BBFO+)
275:1 (X coil)

Pro500 (Prodigy)
1200:1 (^{1}H/^{19}F coil)



80 K

X
^{19}F/^{1}H

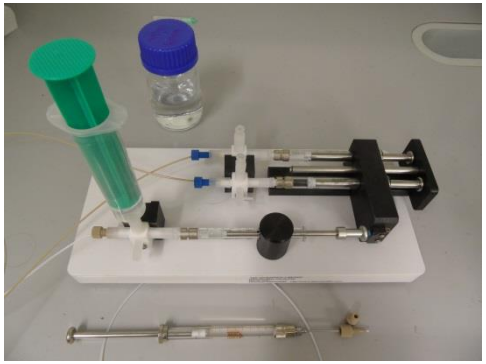
Inner/outer coil

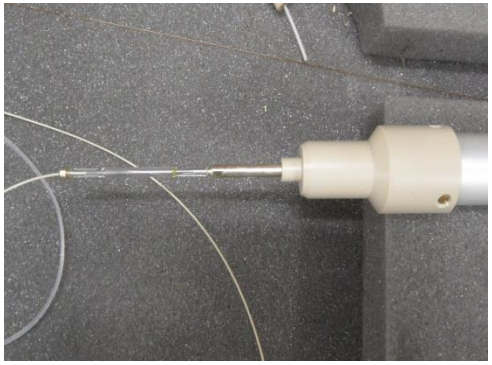
Primary interests / Aplications

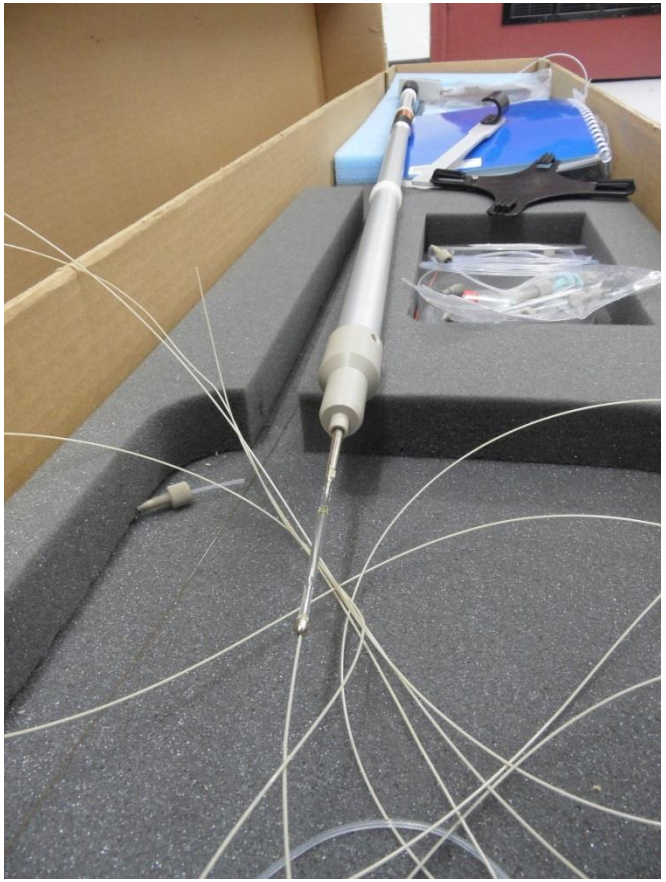
- 500 MHz – Departmental Service
- Predominantly for 1H and X nuclei (other than 13C) for structure characterisation - mostly in automation
- 19F - background suppression, COSY, NOESY, DOSY
- 19F-13C correlations: HSQC, HMBC (conventional and NUS speedy & highres versions)
- 31P – 31P-1H & 1H-{31P}, 2D HMBC (& NUS versions)
- 29Si – background supp. INEPT/DEPT, 2D HMBC
- 11B - background supp. 1H-{11B}, HMQC (NUS ver.)
- 10B – antiring sequence
- 7Li, 59Co,

Primary interests / Alications

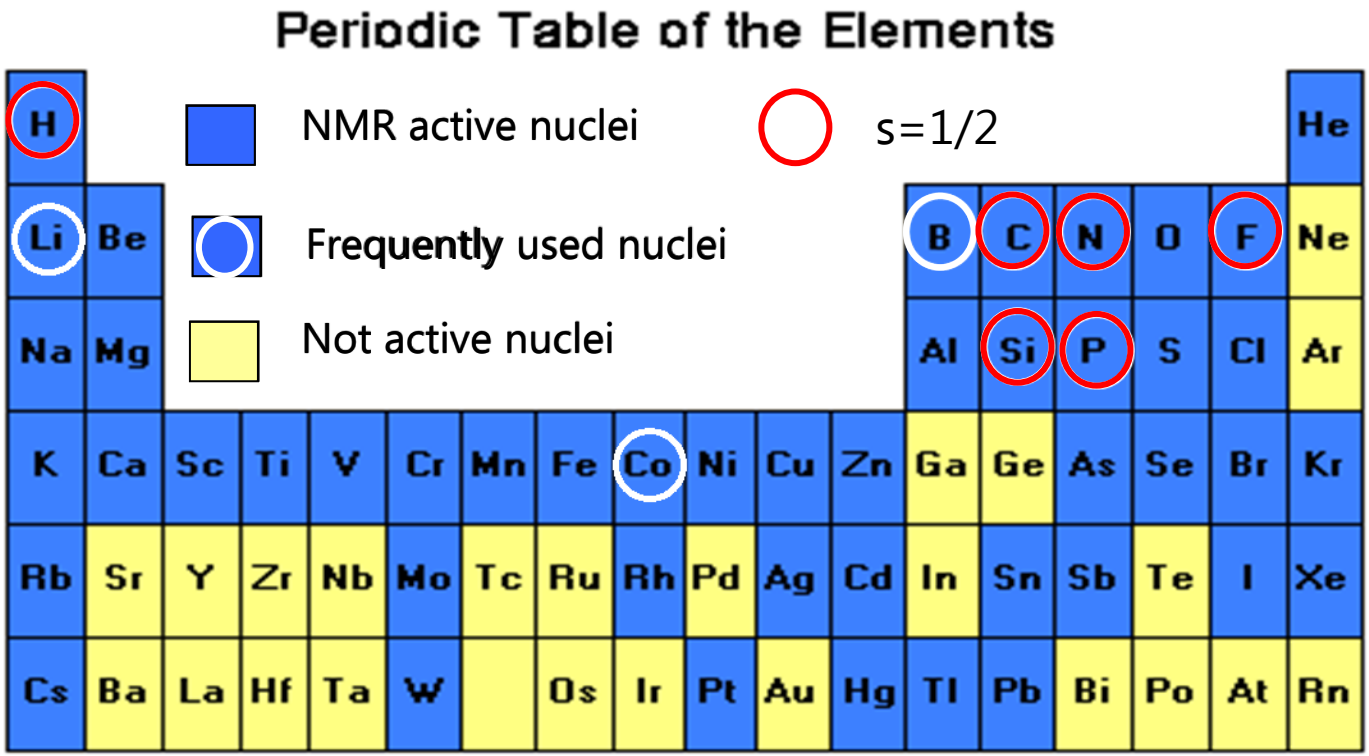
- 400 MHz – Prof Guy Lloyd-Jones group
- 1H/19F & X nuclei
  and reactions monitoring
- The same set up for all nuclei
  as on Departmental 500
- Cryofit – flow cell equipment





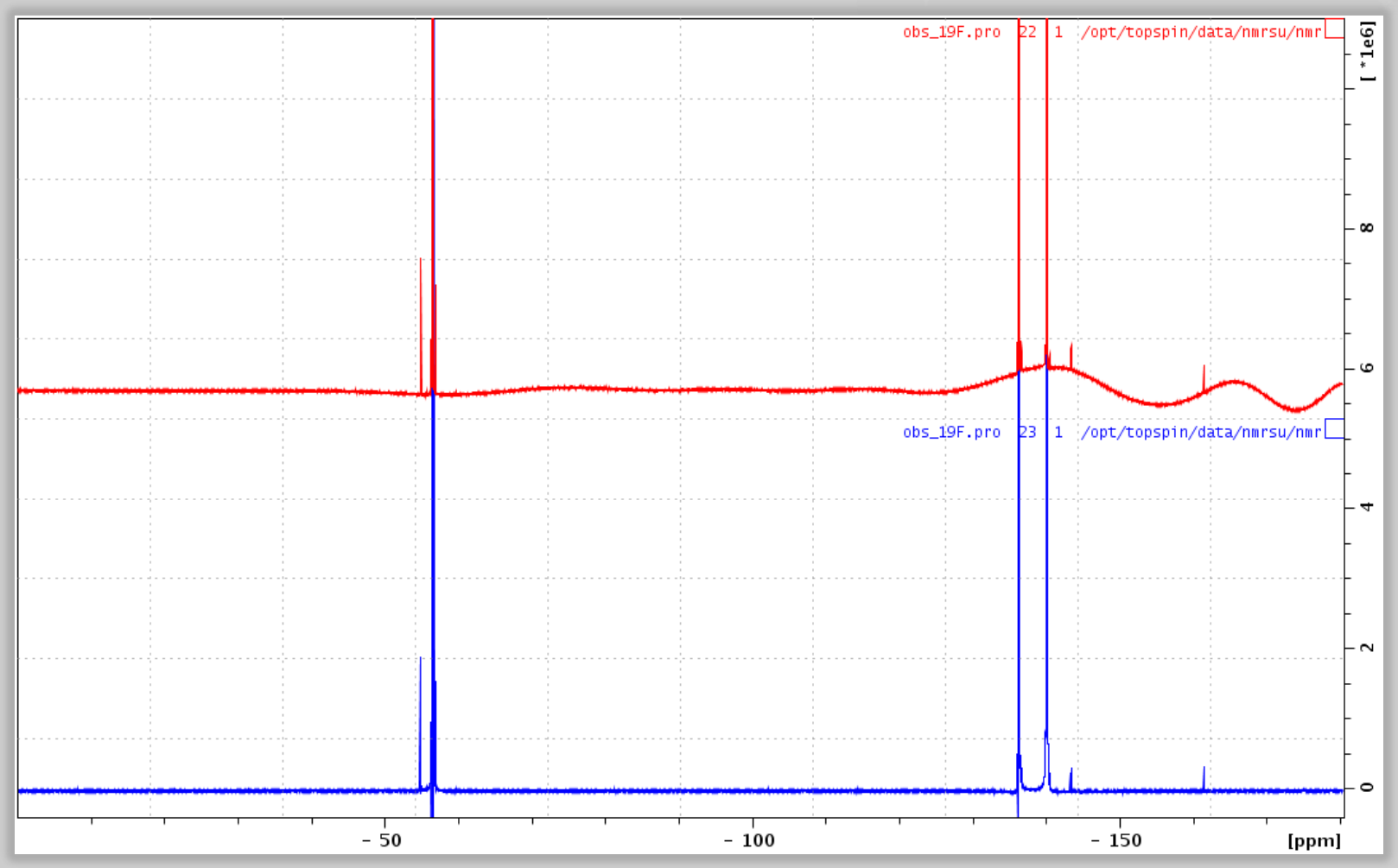


NMR active nuclei

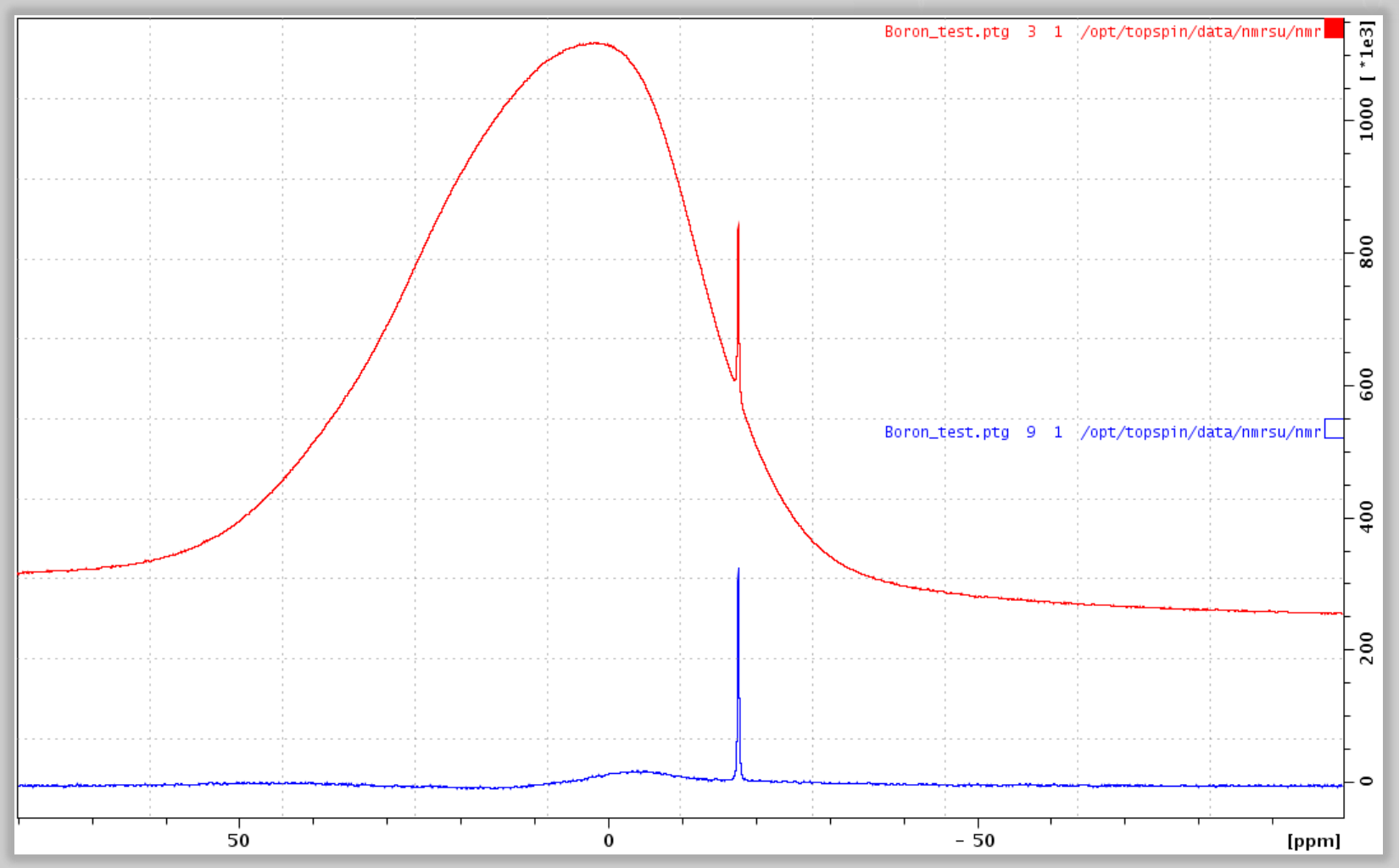


Prodigy except between 199Hg and 153Eu (e.g. 137Ba, 17O, 9Be, 6Li…)

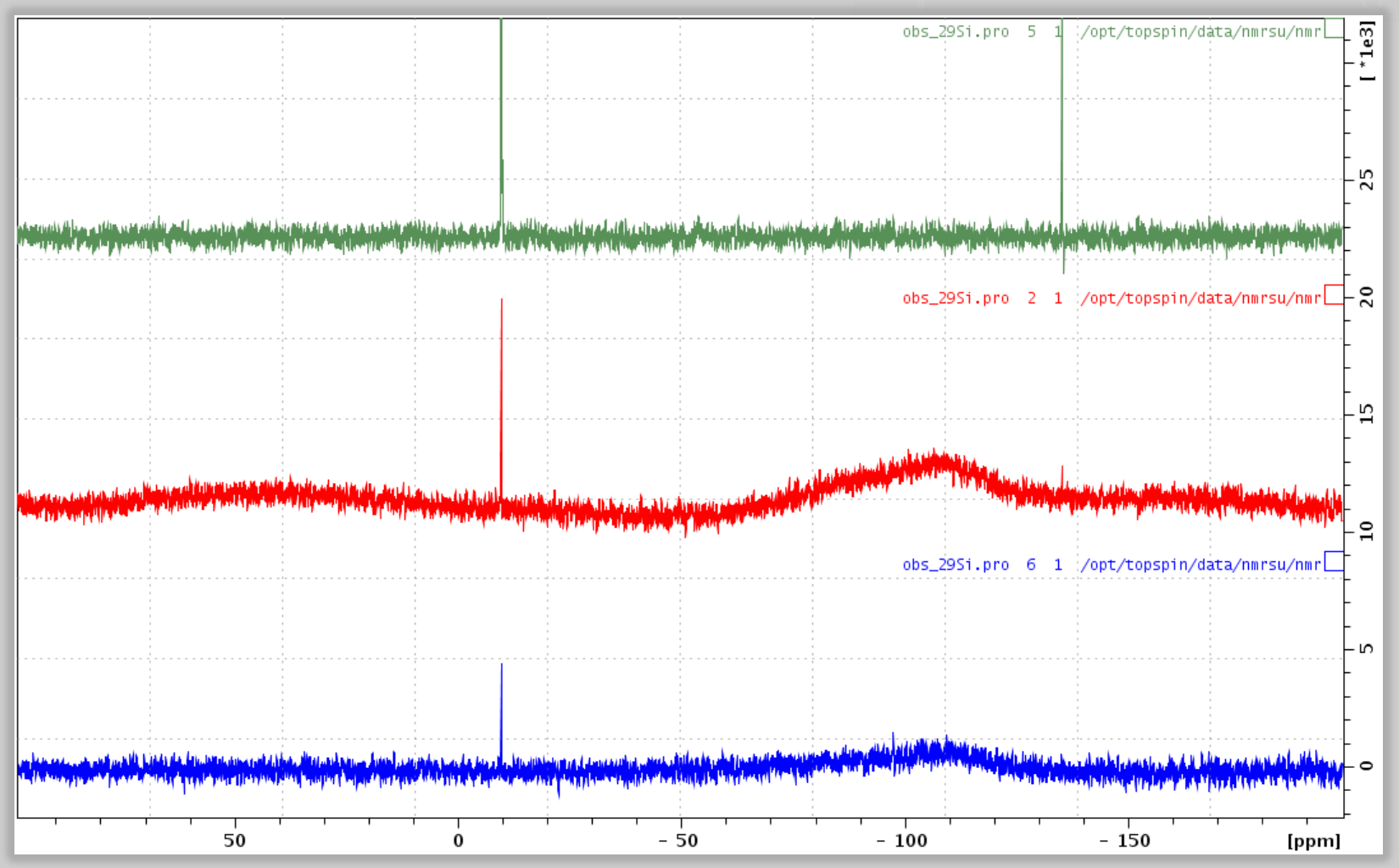
Examples: 19F zgbs_bb background suppression



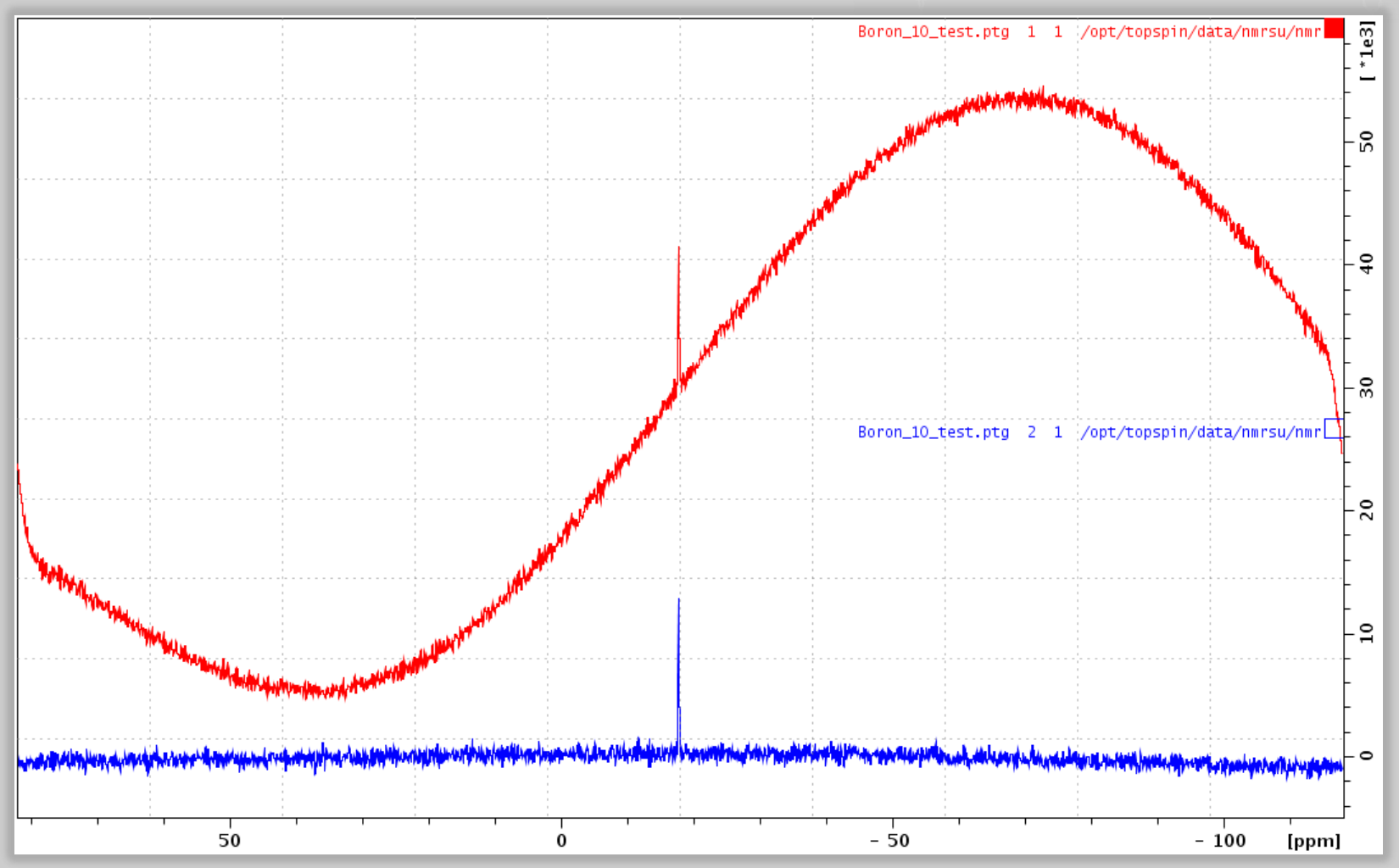
Examples: 11B (zgbs_bb) background suppression



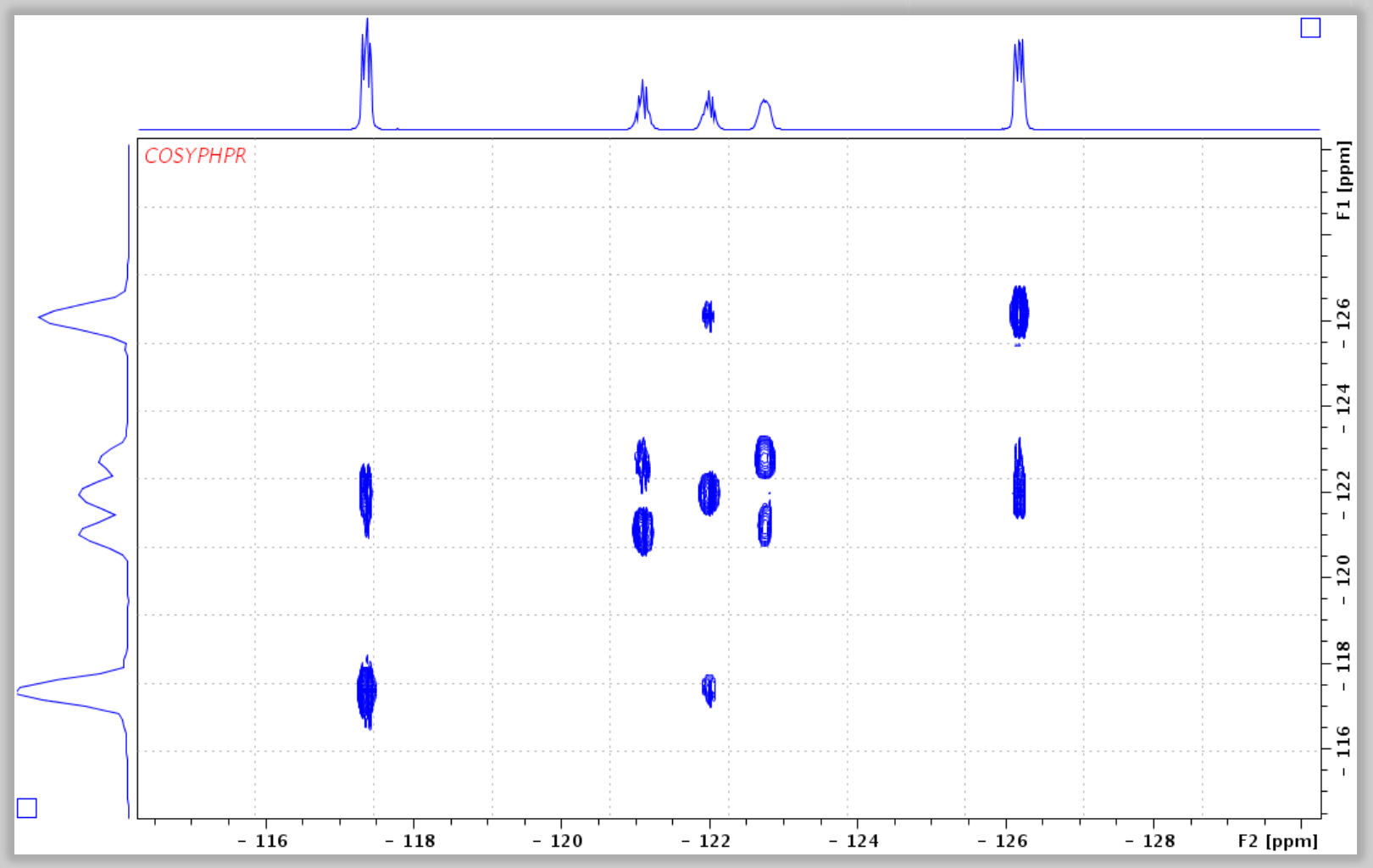
Example: 29Si (zgbs_bb)
background suppression & INEPT



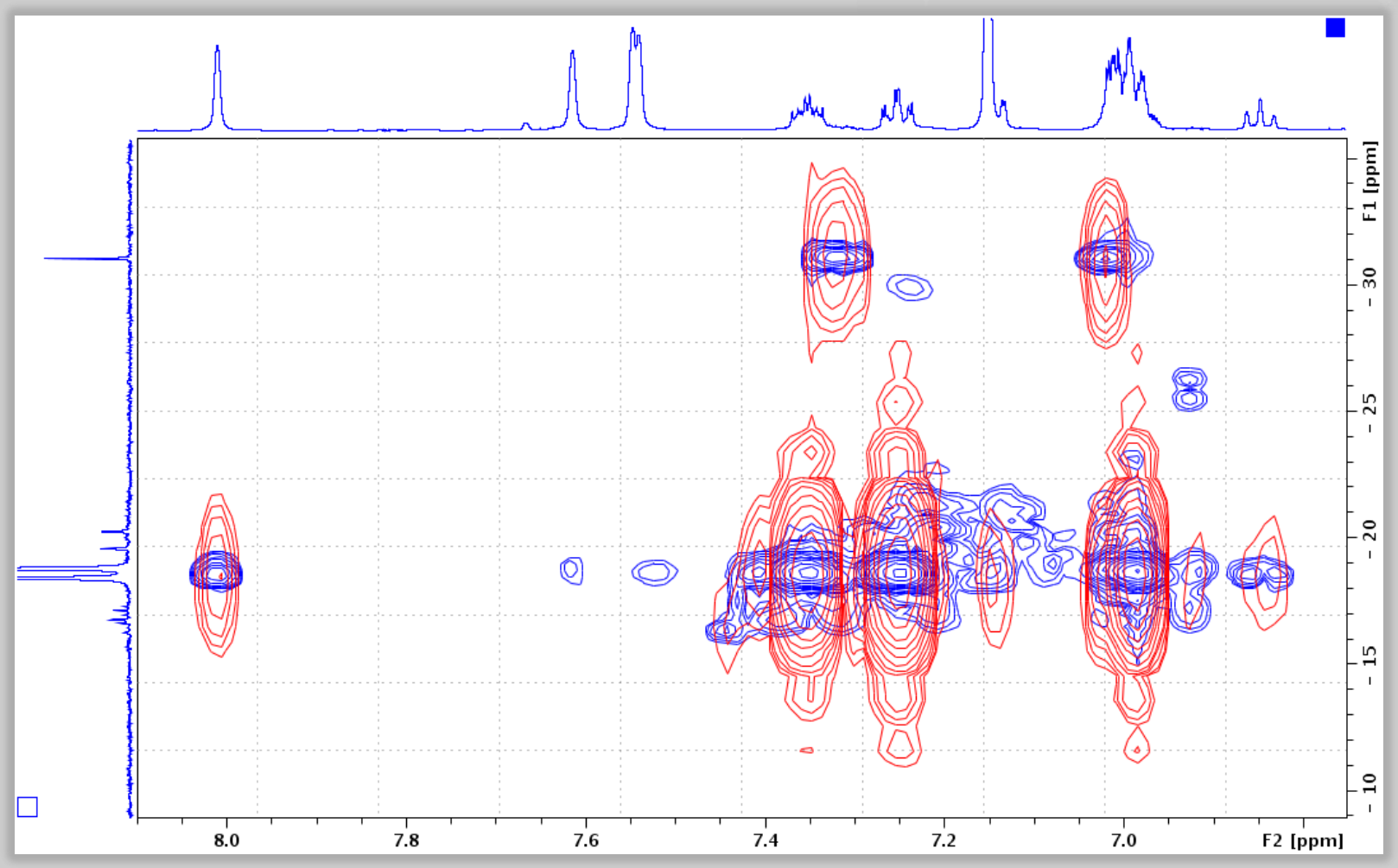
Examples: 10B (aring2_bb) antiring sequence



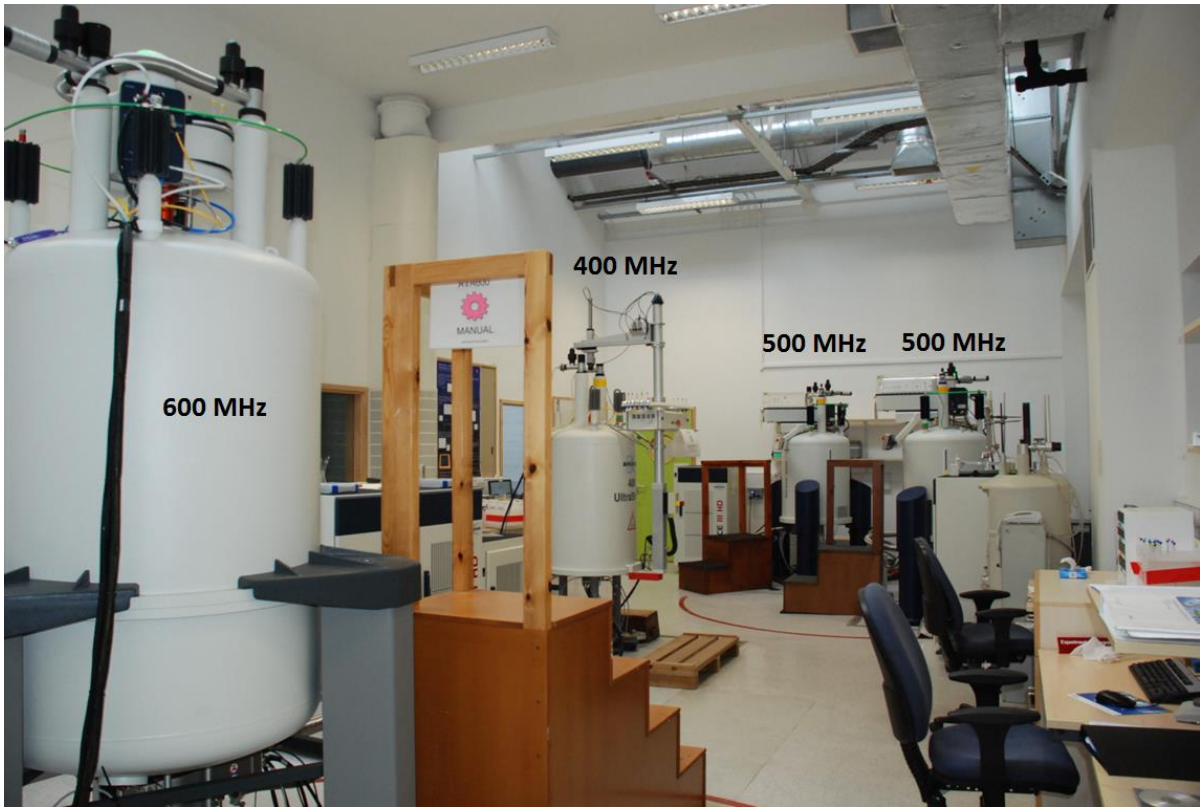
Examples: 19F COSY



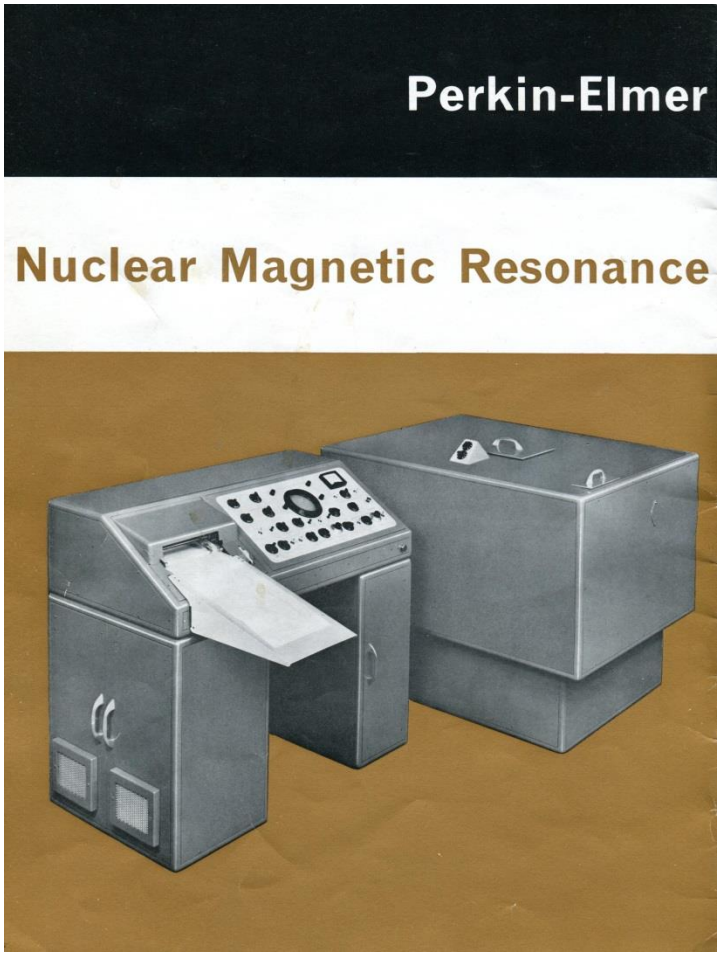
Examples: 31P HMBC (NUS) structure characterisation



Operational aspects



NMR at Edinburgh is 50 years old this year!



Perkin Elmer R-10 NMR spectrometer

- 60 MHz persistent magnet
- CW mode

Installed in January 1964
Dr. Peter Schwarz

Operational aspects

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- With shielded magnet very compact
- Room 3m x 3m is sufficient
- Installation straightforward versus He probes
- 16A power socket, N2 gas (6 bar) supply & backup N2
- No mess with flexlines, cables, compressor etc…
- Quality (dry) of N2 gas is critical !
- No He filters ☺
- Issues – Yes ☹
- PROdigy = PROblems free – NO! ☹

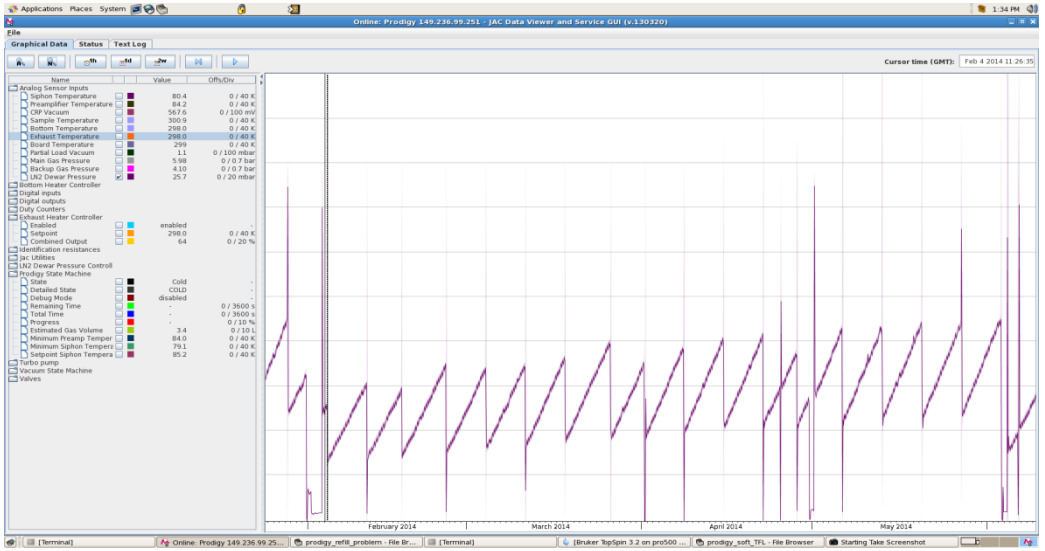
Issues
Soft TransFer Line (TFL)

- Poor vacuum in TFL decreases hold time efficiency from about 10 days to less than one week.
- In LN2 consumption - from 8 kg/day to up 10 kg/day
- Easy monitoring by In-build gui service monitor

- LN2 Dewar Pressure
- Initial DP after refill:

26 mbar – good vacuum

50 mbar – poor vacuum



- Had to pump FTL twice (every four months)
- Bruker is working on new improved version of TFL

Issues – LN2 refill problem under investigation

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- Example of normal LN2 refill:

Dewar pressure:

DP 6-8 mbar

(purple)

Heater – low

(yellow)

Exhaust temp:

298 K

(orange)

Issues – LN2 refill problem
Ice blockage ?

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- Example of “acceptable” LN2 refill:

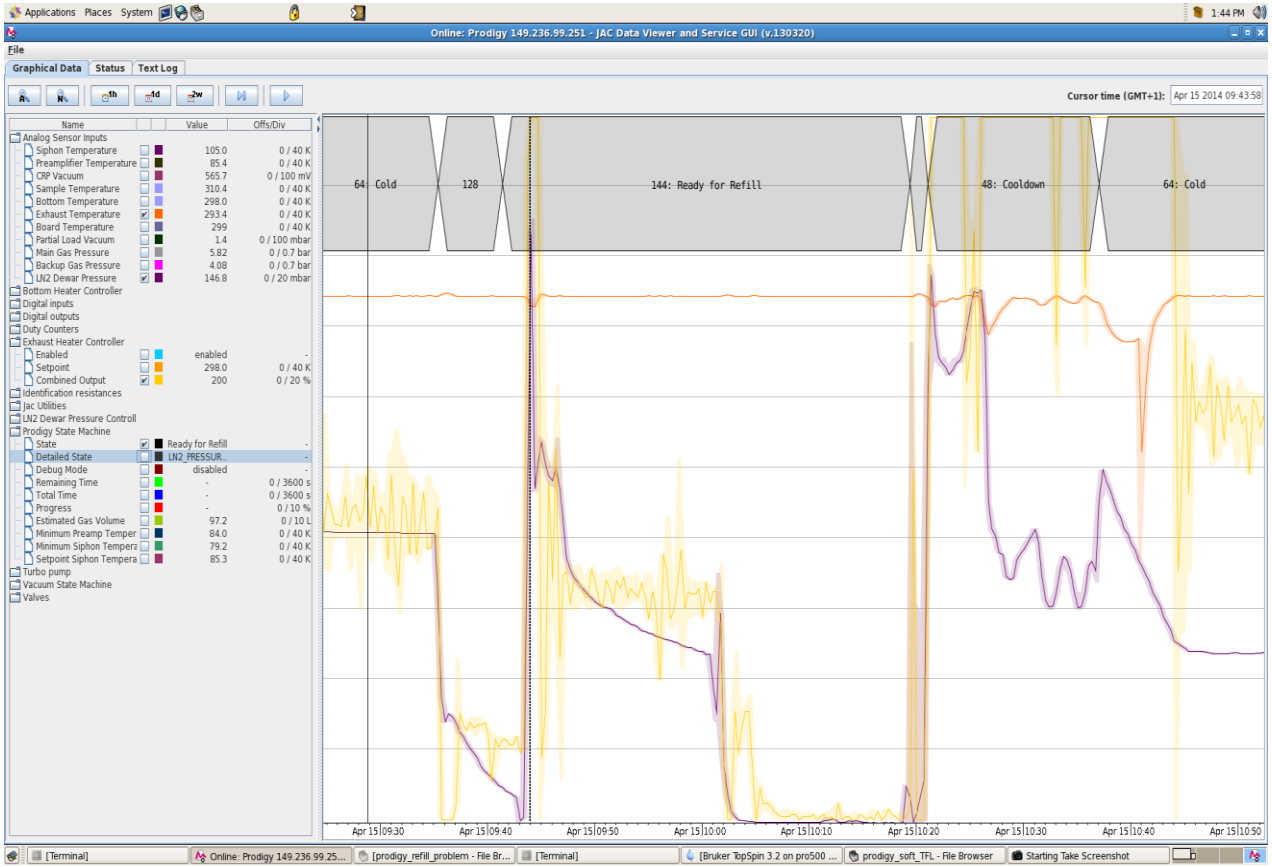
Dewar pressure:

DP 150 mbar !

Heater – high

Exhaust temp:

298 K



Issues – LN2 refill problem complete ice blockage ?

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- LN2 dewar does NOT accumulate any LN2 at all

Dewar pressure:

DP 260 mbar !

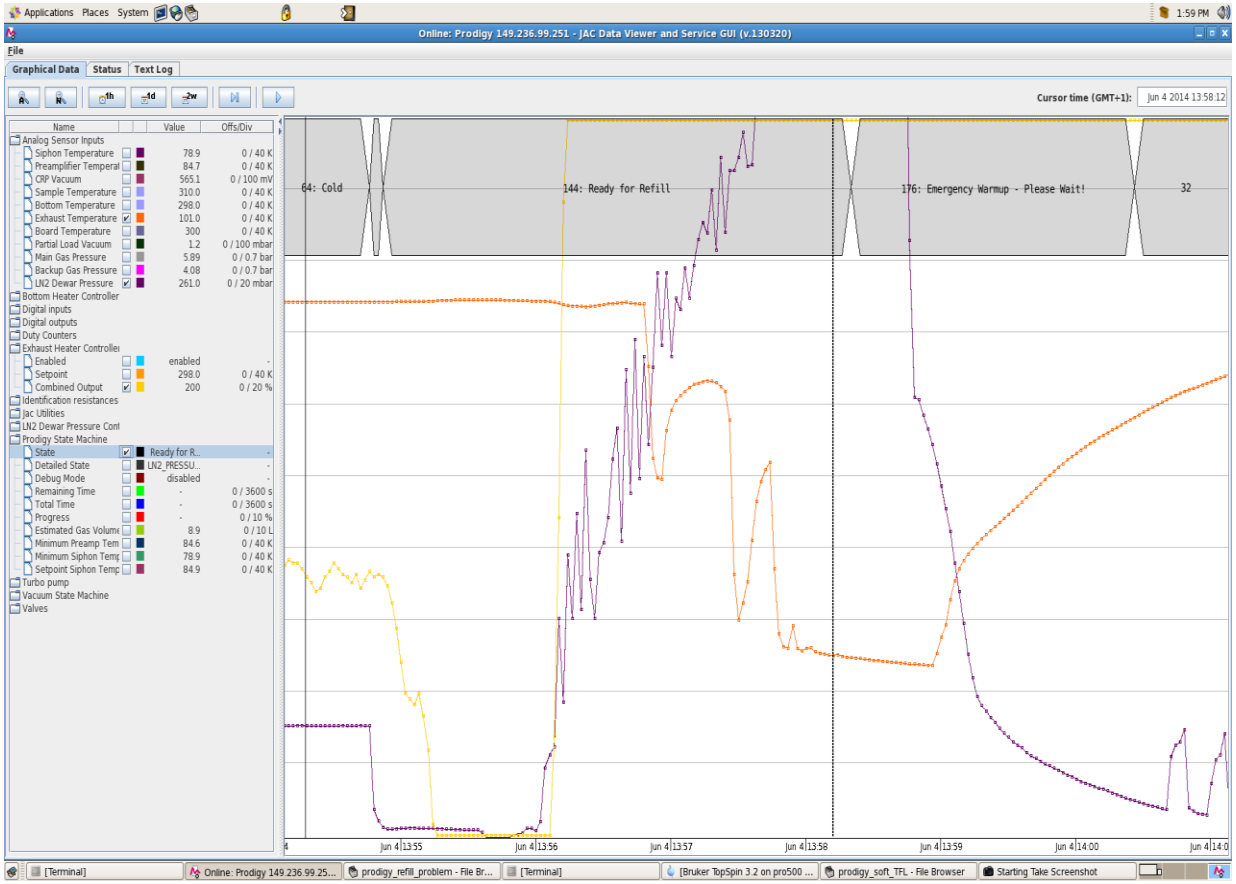
Heater – 200 % !

Exhaust temp:

Below 100 K !

Emergency

warmup !



Issues – LN2 refill problem how it looks like

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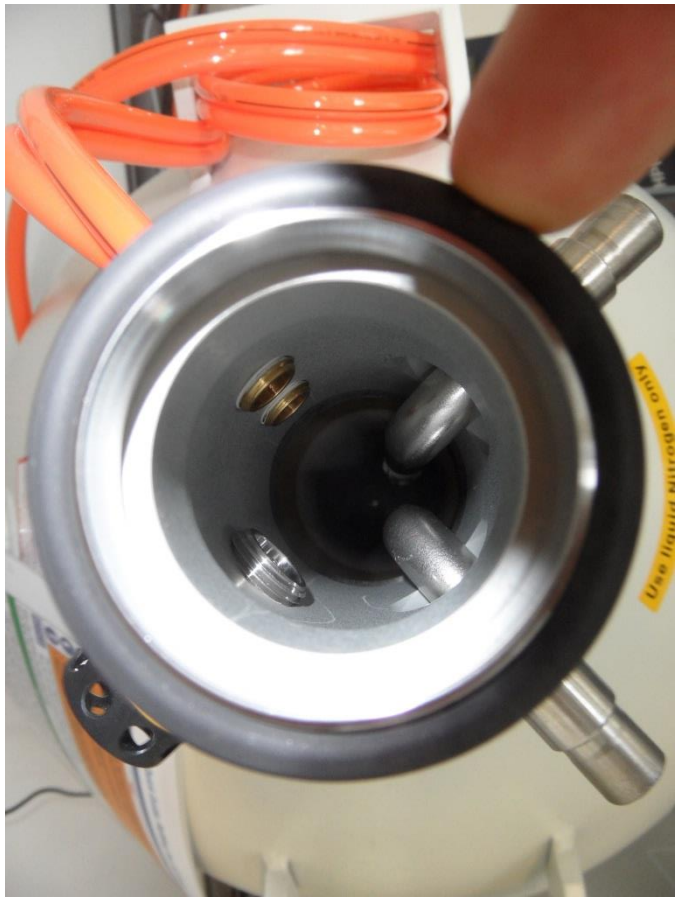
This is something you do not want to see ☹

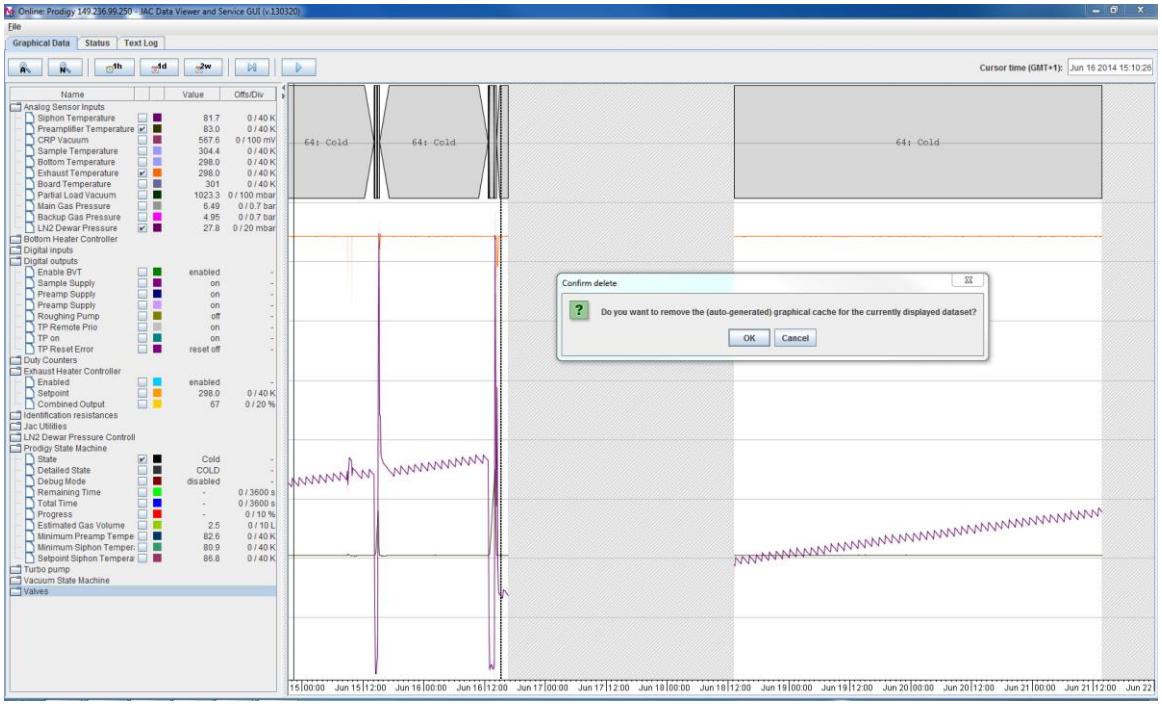
Issues – LN2 refill problem and what's behind

Bruker's Enigma:



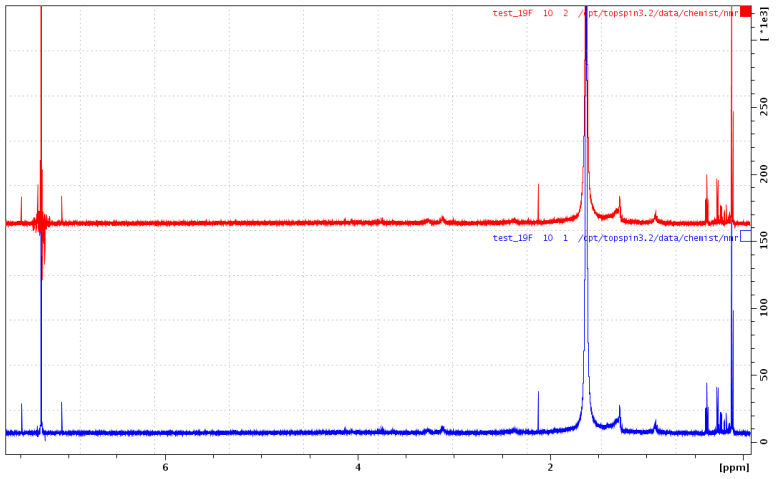
Issues / inconveniences
Sudden loss of memory

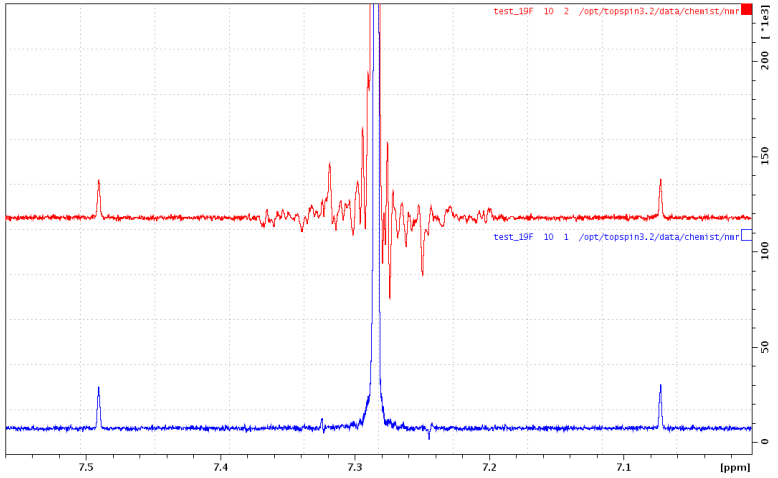
- Greyed areas / missing data ?
- There is a cure - Go to File & Renew Graphic Cache (Quick)



- Nice tool but software is a bit temperamental

Issues / inconveniences
Automation is the key





- Resistive touch screen

It requires quite a firm touch - sometimes even shaking magnet

- Capacitive touch screen

would be welcome

Vivat Prodigy ?

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Bionmr:

two fold sensitivity of
He cryo-probes still makes
significant difference in
3D and nD expriments
duration (4 times).
Interactions (2D) – Prodigy!

Chemistry:

you really do not need
sensitivity of He cryoprobes
unless you already have one.

Other stuff

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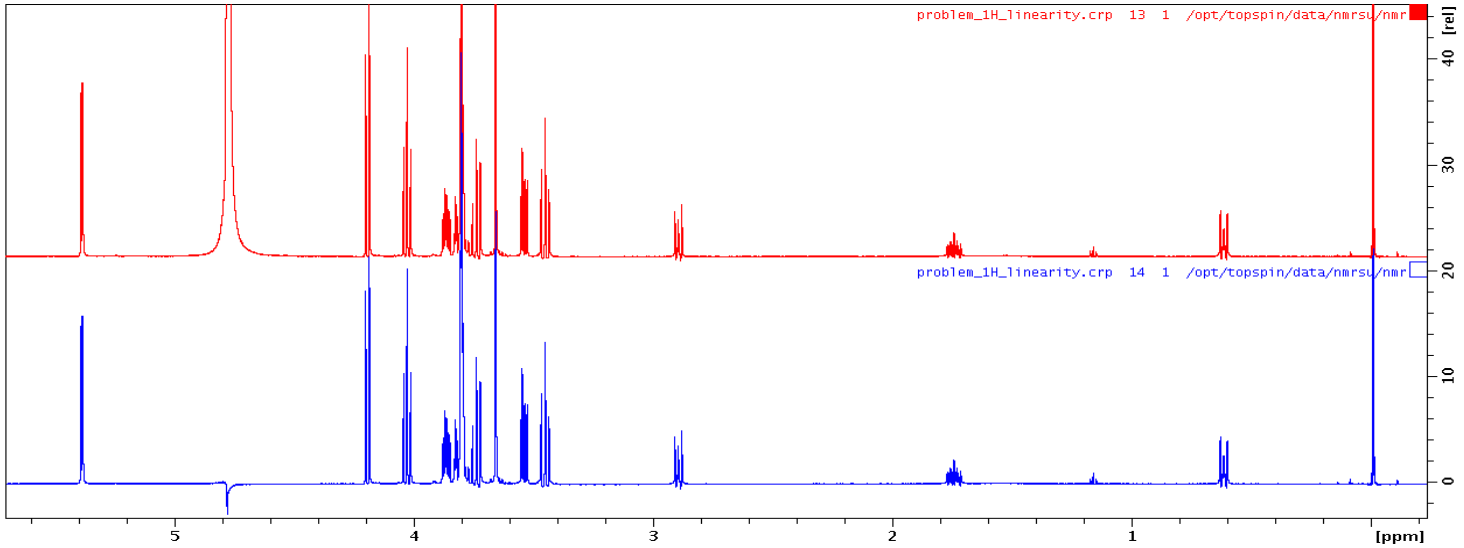
- Bruker: Windows 7 professional – NO Server for NFS (Network File System) – no way to mount Linux PC hard drive
- 600 MHz TCI (Z44896/009) 1H non linearity - about +0.9 dB zgesgp



- 600 MHz AV I (2001) 3 channel working console available
- Additional 600 MHz AV I spare parts available too

Liquid-state NMR in the School of Chemistry @ Edinburgh now

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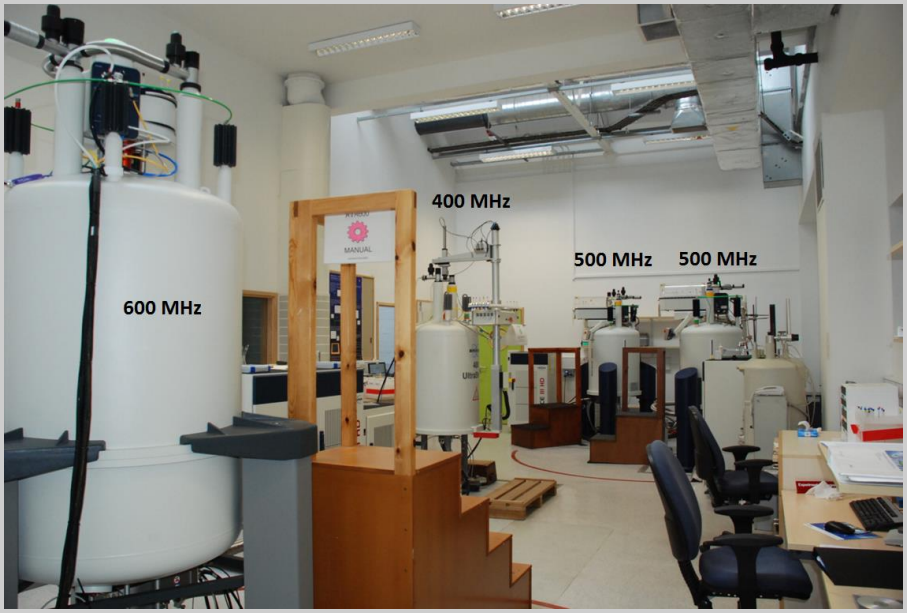

Core capability for Chemistry Research – EPSRC & The University of Edinburgh