Liquid-state NMR in the School of Chemistry @ Edinburgh

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Lorna Murray

Thank you Dusan Uhrin for providing template slides ☺

The NMR crowd @ Edinburgh

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Dr. Dušan Uhrín
Facility director

Mr. Juraj Bella
Facility manager

Dr. Lorna Murray
NMR technician

Where are we?

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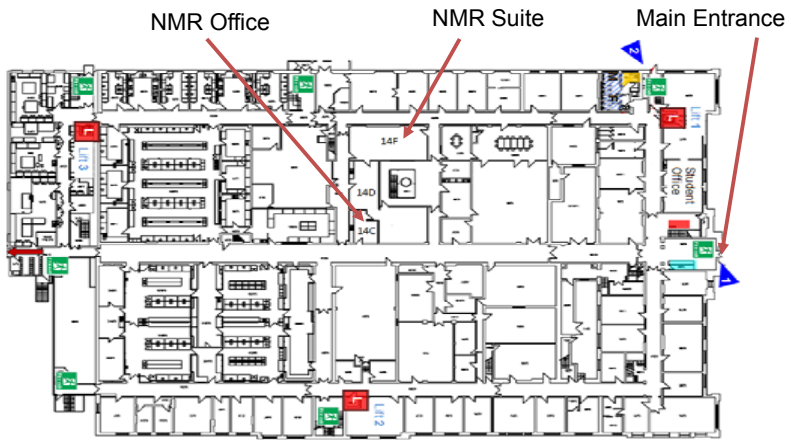

Liquid-state, high-resolution NMR

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- NMR spectroscopy is a powerful analytical technique used in every day research across the whole of chemistry.
- It is a non-destructive method
- NMR provides information on molecular dynamics, conformational behaviour, molecular sizes etc..

NMR at Edinburgh is 51 years old!

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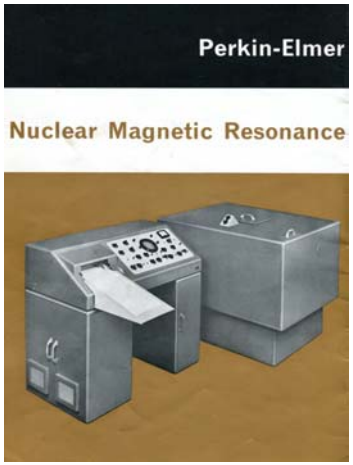

Perkin Elmer R-10 NMR spectrometer

- 60 MHz persistent magnet
- CW mode

Installed in January 1964
Dr. Peter Schwarz

1969 – 2004 Dr. Ian Sadler
Mr. John Millar

David Reed, John Parkinson, Paul Barlow, Peter Sadler

NMR Facility in 2017

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2800 MHz NMR Facility

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Automation is the key

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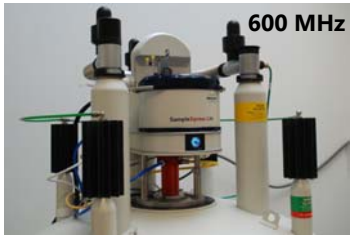
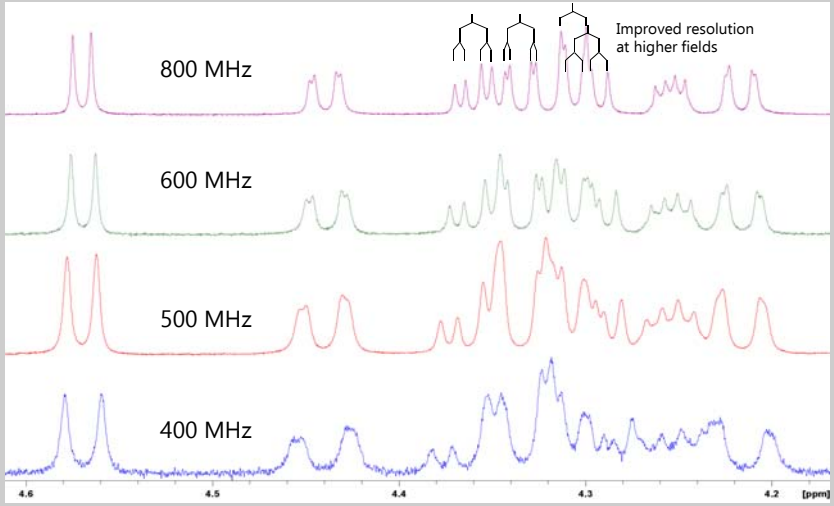
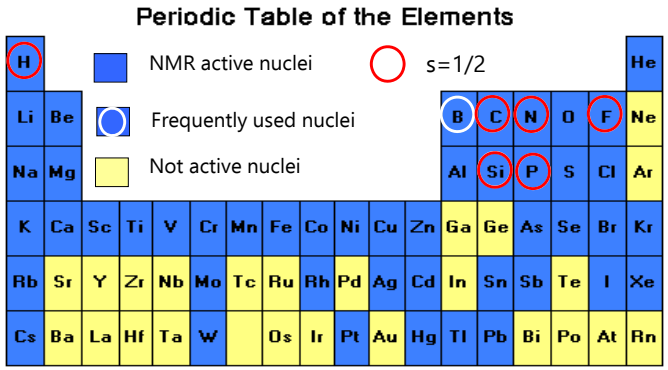


Why do we need higher magnetic fields?



NMR active nuclei



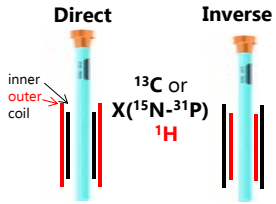
Sensitivity of NMR

$$\frac{S}{N} \propto n\gamma_e\gamma_d^{3/2}B_o^{3/2}t^{1/2}$$

n – number of spins (concentration)
γ_e - gyromagnetic ration of excited spins
γ_d - gyromagnetic ration of detected spins
B_o – external magnetic field
t – experimental time

B_o / MHz	400	500	600	800
Rel. sensitivity	1	1.4	1.8	2.8

Other factors:
1. Coil geometry (direct *vs* inverse)
2. BB *vs* dedicated coils



4. Design of NMR experiments

Excited nucleus	Detected nucleus	NMR experiment	Relative sensitivity
^{13}C	^{13}C	1D ^{13}C spectrum (NOE)	1
^{1}H	^{13}C	DEPT, HETCOR	2
^{1}H	^{1}H	2D HSQC, HMBC	16

3. Temperature of the coil

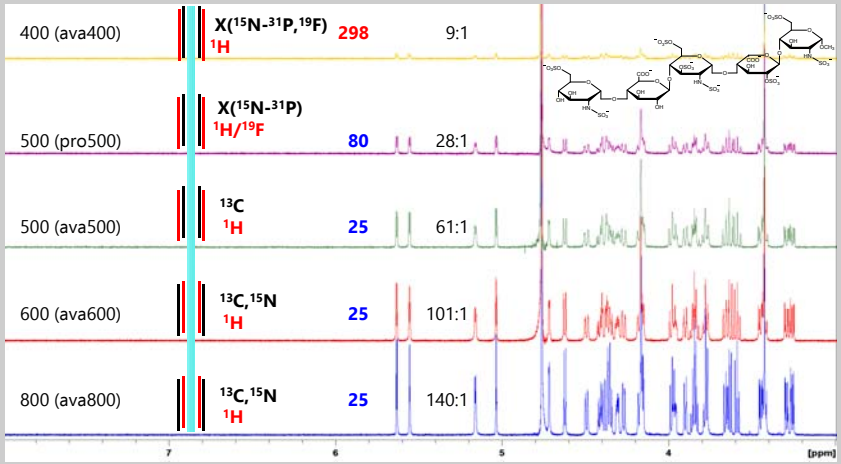
Coil temp/ K	298	80	25
Rel. sensitivity	1	2	4

Spectrometer and sample requirements

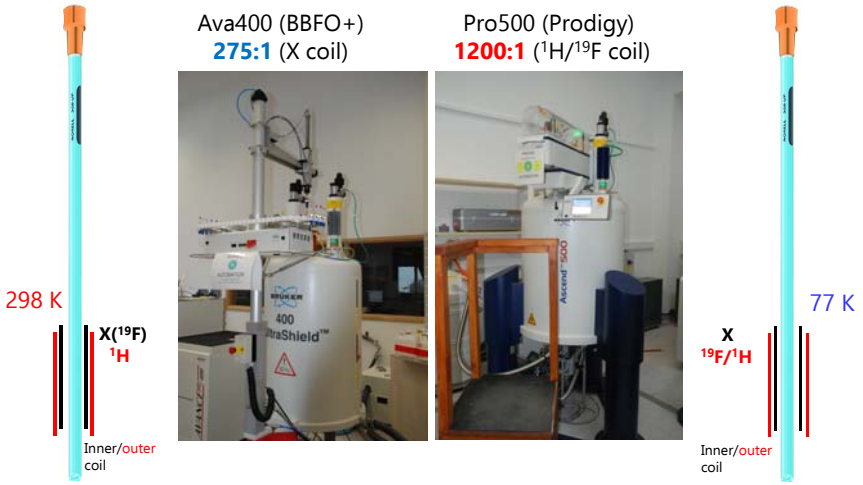
Sample

- Soluble in 550 - 600 μL (water, organic solvents)
- Ten tens of mill ds
- Cho rometer for

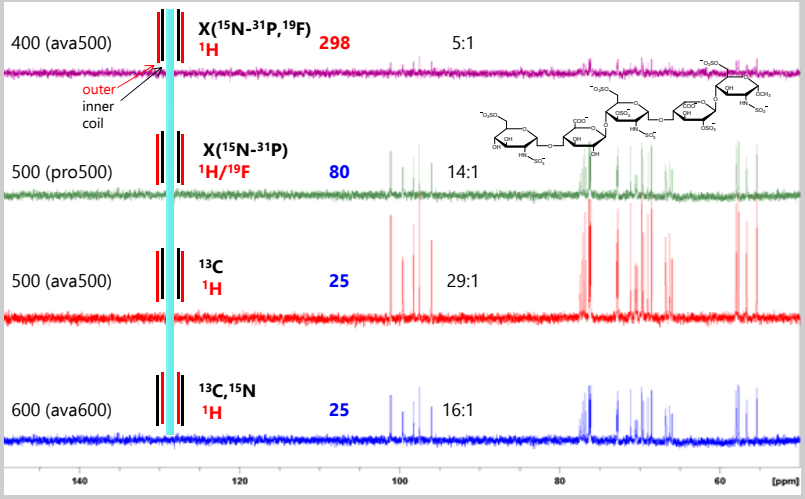
^{1}H sensitivity



^{19}F sensitivity



^{13}C sensitivity



NMR Summary

B_o (MHz)	Spectro-meter	Coil temp	Optimised for	Best for	Sample temp. range /^{o}C
400	ava400	room	^{15}N-^{31}P,^{19}F/^{1}H	X	-150, +150
500	ava500	He	^{13}C/^{1}H	^{13}C	0, 55
500	pro500	N_2	^{15}N-^{31}P/^{1}H,^{19}F	X, ^{19}F	0,135
600	ava600	He	^{1}H/^{13}C/^{15}N	^{1}H, ^{13}C/^{15}N HSQC	0, 55
800	Ava800	He	^{1}H/^{13}C/^{15}N	^{1}H, ^{13}C/^{15}N HSQC	0, 55

Not covered here:

- HR MAS @ 600 MHz – high-resolution spectra of compounds on solid support

Manual

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AVA500

MANUAL

BOOKING REQUIRED

NMR in Automation

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AVA400

AUTOMATION

**** DO NOT TOUCH BUTTONS ****

**** IF THE SAMPLE CHANGER IS MOVING ****

Lab Safety

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- No gloves/lab coasts!!
- Safety glasses
- No broken tubes
- Report breakages/spillages

Sample Preparation

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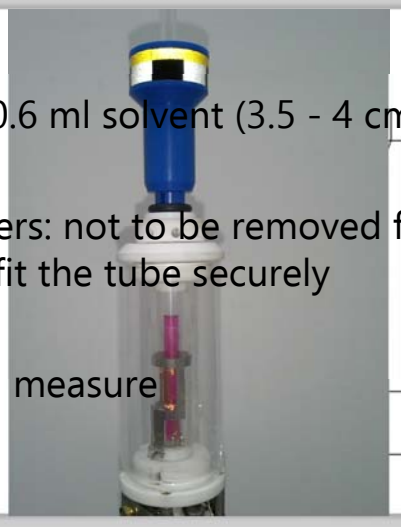
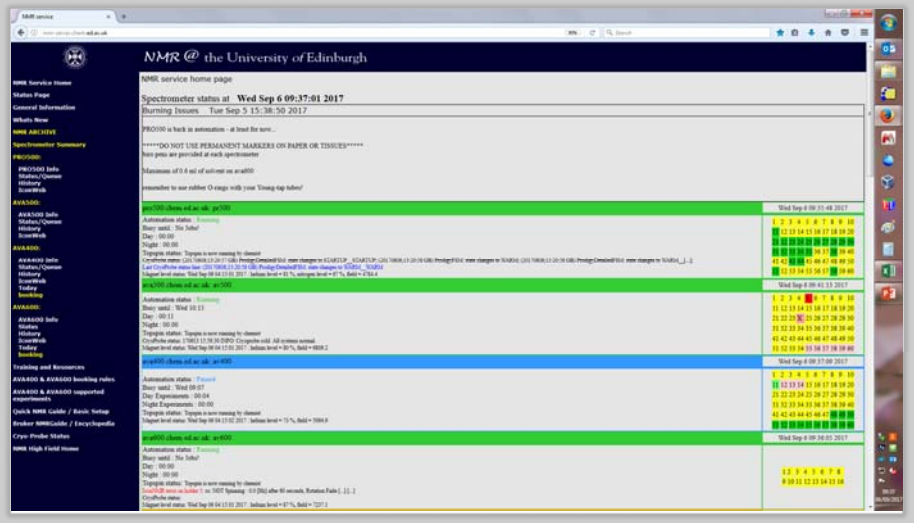
- 0.5 – 0.6 ml solvent (3.5 - 4 cm)
- Spinners: not to be removed from NMR lab, must fit the tube securely
- Depth measure



Website



Thank you

Updates and information available on university network:

http://nmr-server.chem.ed.ac.uk

Sign up for automation training
Sign up for email updates: mnr-updates

In December we will run a data interpretation workshop and a hardware workshop depending on demand. Look out for updates!!