



Everything you wanted to know about Benchtop NMR (but were afraid to ask)

SNUG 2024 Postgraduate Course in
NMR Spectroscopy

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Introduction

+ Benchtop NMR



+ Benchtop NMR Spectroscopy

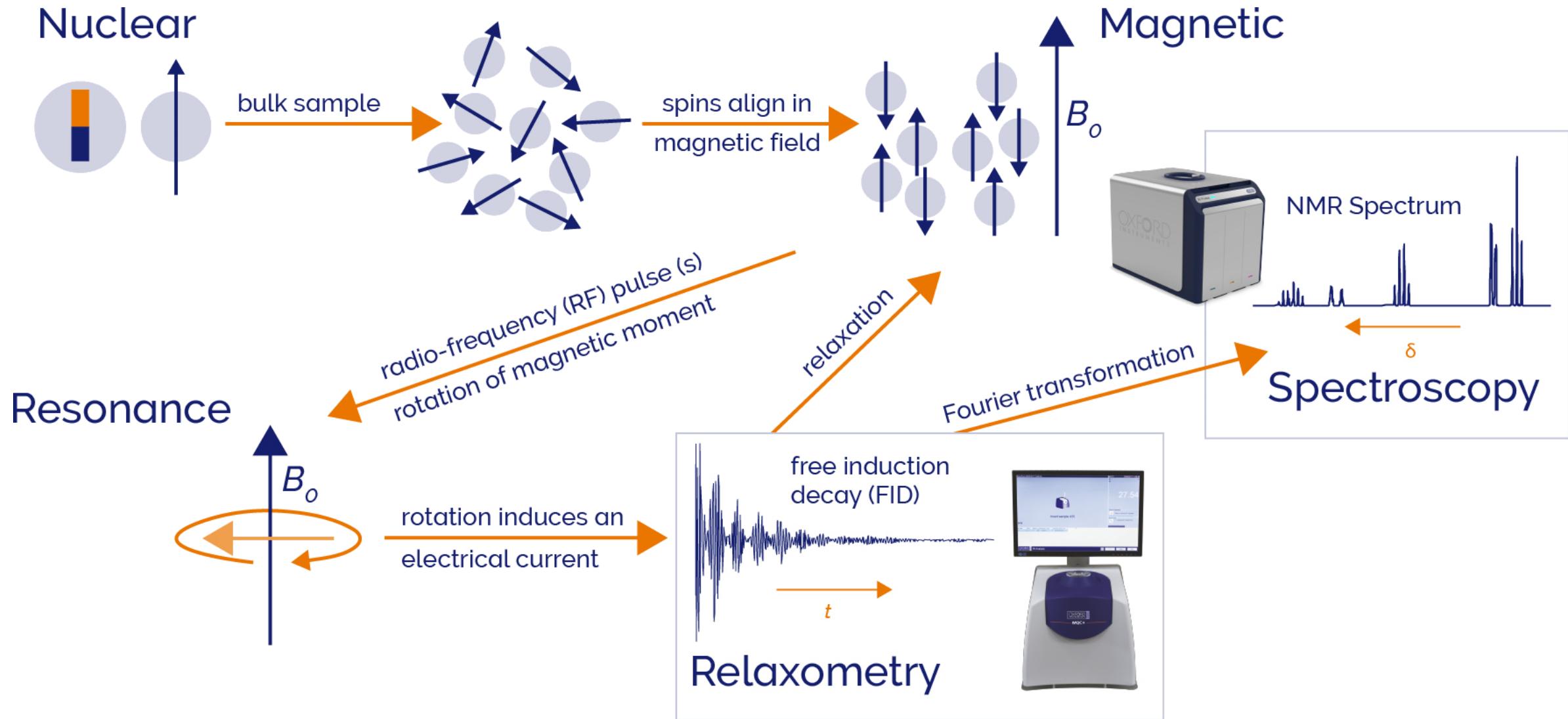
+ Small Molecule Characterisation / Analysis

+ Reaction Monitoring & FlowNMR

+ Time-Domain NMR

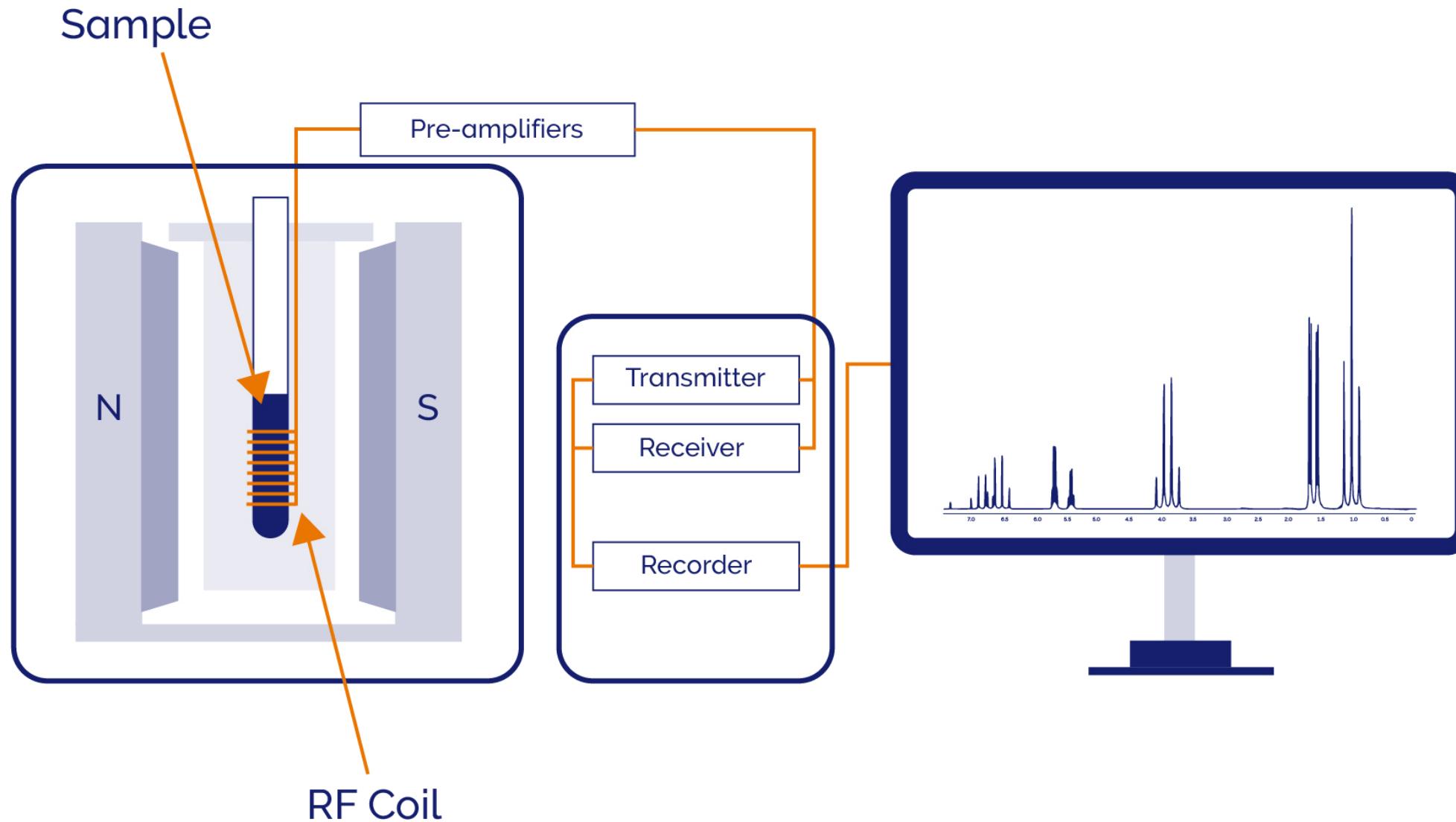


What is NMR ?



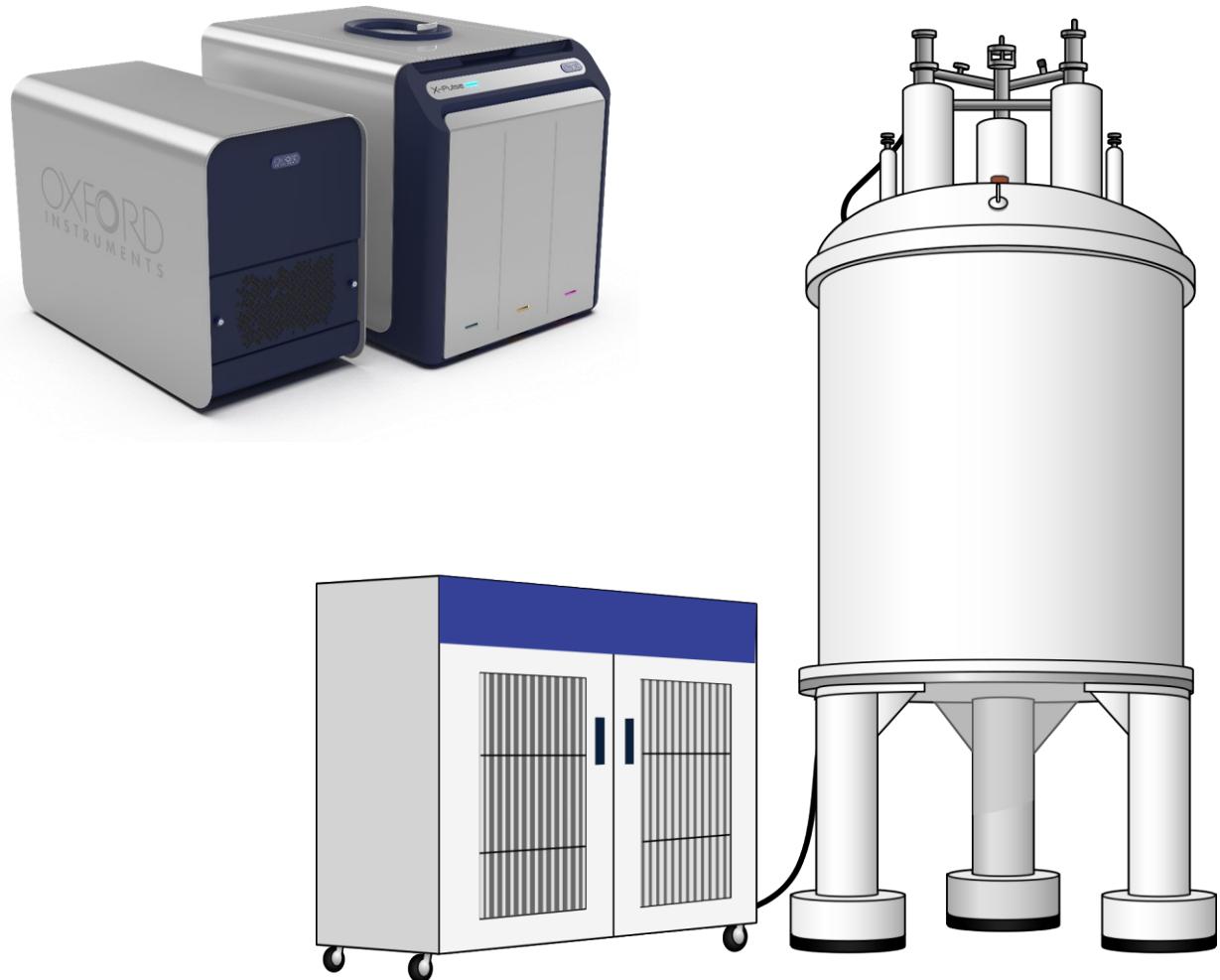
What is an NMR Spectrometer / Relaxometer ?

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Benchtop verses High-field NMR

- Benchtop NMR
 - up to 120 MHz
 - permanent magnets ($\text{Nd}_2\text{Fe}_{14}\text{B}$)
- High-field NMR
 - up to 1.2 GHz (28.2 Tesla)
 - superconducting magnets
 - liquid cryogens (N_2 & He)
 - dedicated facilities / personnel



X-Pulse

Broadband Benchtop NMR Spectrometer

X-Pulse Broadband Benchtop NMR Spectrometer

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- 1.4 T / 60 MHz permanent magnet
- High resolution (CHCl_3 in acetone- d_6)
 - $\nu_{1/2} \leq 0.35 \text{ Hz} / \leq 10 \text{ Hz}$ @ ^{13}C satellites
- High sensitivity (1% ethylbenzene in CDCl_3)
 - ≥ 140 signal-to-noise ratio (SNR)
- Three-axis field gradients as standard (z-axis: $\geq 0.5 \text{ T.m}^{-1}$)
 - gradient-selective two-dimensional homo- & hetero-nuclear correlation
 - pulsed field gradient spin echo, for diffusion measurements
- Shaped pulses and gradients as standard
 - solvent suppression using WET (Water suppression Enhanced through T_1 effects)
 - selective one-dimensional TOCSY, NOESY

X-Pulse Broadband Benchtop NMR Spectrometer



- Three channel spectrometer
 - Proton/fluorine channel
 - Broadband X-channel (*optional*)
 - ^7Li , ^{11}B , ^{13}C , ^{23}Na , ^{27}Al , ^{29}Si , ^{31}P , and many more ...
 - Deuterium lock channel
- User removable / exchangeable probe
- Auto-sampler, 25 positions (*optional*)
- Variable temperature operation (*optional*)
 - minimum range 0 to +65°C
- Flow NMR capable (*optional*)
- Mobile workstation/cart



X-Pulse Periodic Table (standard broadband)

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The periodic table displays elements in a grid. Elements are colored based on their properties:

- Orange:** Li, Be, Na, Mg, K, Ca, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ge, As, Se, Br, Rb, Sr, Y, Zr, Mo, Tc, Ru, Rh, Pd, Ag, Cd, In, Sb, Te, Cs, Ba, Hf, Ta, W, Re, Os, Ir, Pt, Au, Hg, Tl, Pb, Bi, Po, At, Rn, La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Ac, Th, Pa, U, Np, Pu.
- Blue:** H, He, B, C, N, O, F, Ne, Al, Si, P, S, Cl, Ar, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, As, Se, Kr, Sc, Ti, Ru, Rh, Pd, Ag, Cd, In, Sb, Te, Ba, Hf, Ta, W, Re, Os, Ir, Pt, Au, Hg, Tl, Pb, Bi, Po, At, Rn.
- Grey:** Fr, Ra.

Specific isotopes are highlighted in blue boxes:

- Blue Box:** ^{93}Nb , ^{113}Cd , ^{119}Sn .
- Dashed Blue Box:** ^{187}Re .

X-Pulse Periodic Table (low-frequency)

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This periodic table highlights various elements used in X-Pulse applications, particularly in low-frequency pulsed neutron sources. Elements are color-coded by category:

- Actinides and Transactinides:** Ac, Th, Pa, U, Np, Pu (Grey)
- Transition Metals:** Ti, V, Cr, Mn, Fe, Co, Ni, Ru, Rh, Pd, Ag, Cd, In, Sn, Te, I (Orange)
- Post-transition Metals:** Sc, Tc, Hf, Ta, W, Re, Os, Ir, Pt, Au, Hg, Tl, Pb, Bi, Po, At, Rn (Light Orange)
- Non-metals:** He, Ne, Ar, Cl (Grey)
- Noble Gases:** He, Ne, Ar, Xe (Light Grey)
- Other:** H, Li, Be, Na, Mg, K, Ca, Rb, Sr, Y, Nb, Mo, Rh, P, S, Br, Kr, Cs, Ba, Fr, Ra, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu (Dark Blue)

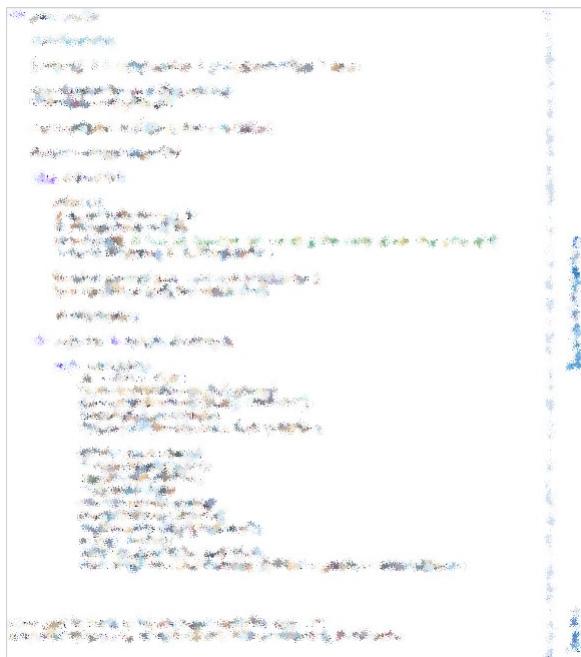
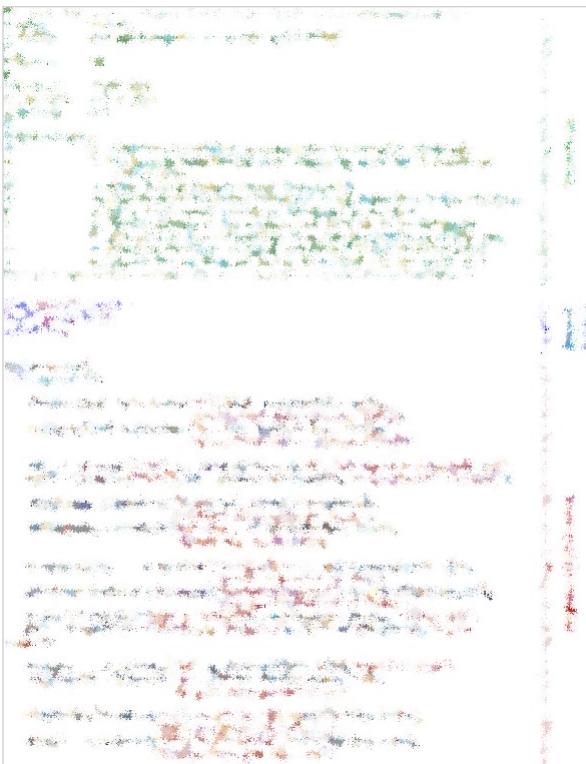
The table also includes specific isotopes highlighted in orange:

- ¹H, ²H, ⁶Li, ⁹Be, ¹⁰B, ¹⁴C, ¹⁵N, ¹⁷O, ¹⁹F, ³⁵Cl, ⁸⁵Rb, ⁹¹Zr, ¹³³Cs, ¹³⁷Ba, ¹³⁹La, ¹⁵³Eu, ¹⁶⁹Tm, ¹⁷¹Yb, ¹⁷⁶Lu.

Pulse Sequence Programming / Development



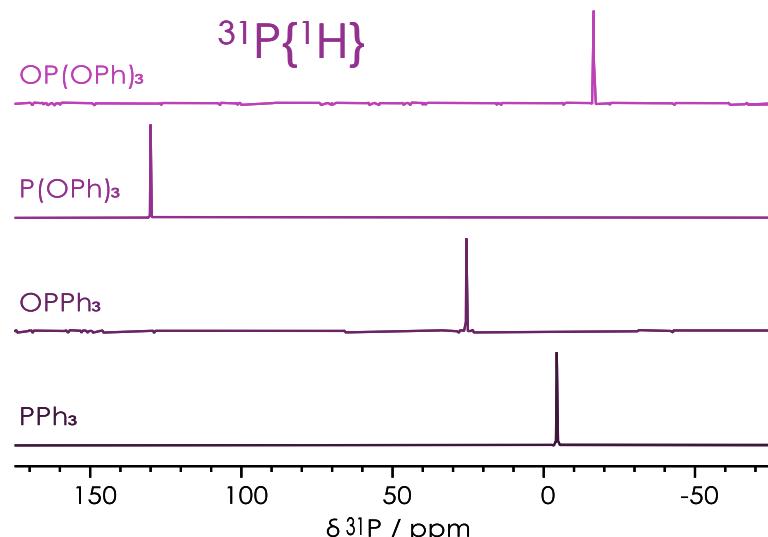
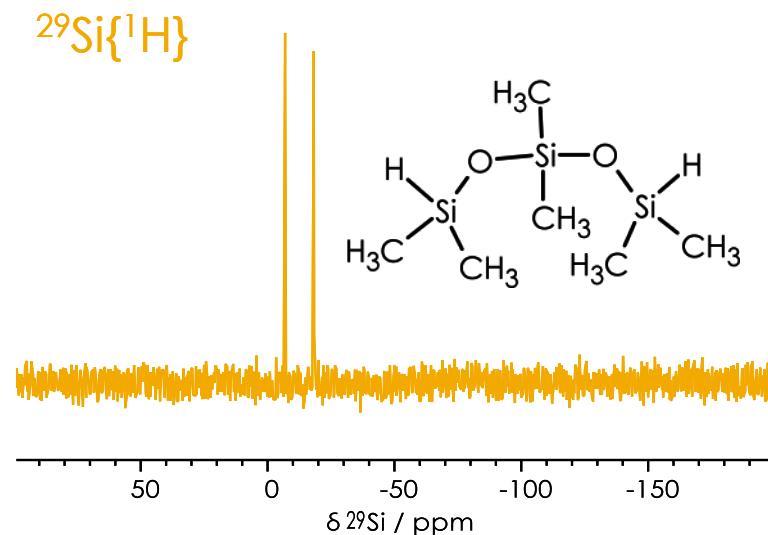
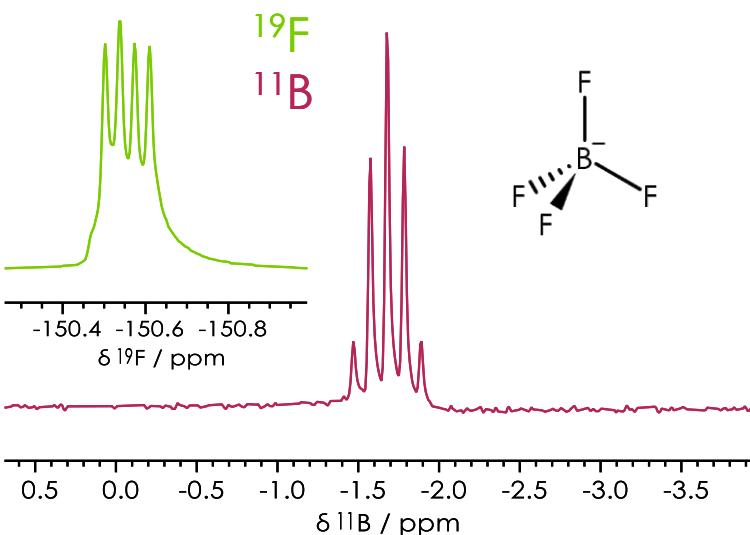
- Sequences programmed in Python
 - complete control of spectrometer hardware
 - pre- / post-acquisition processing
- Two radio-frequency channels
 - high- & low-power
 - shaped / adiabatic pulses
- Three bipolar gradient channels
- External (TTL) triggers
 - input & output
- Customisable JCAMP-DX output files



Small Molecule Characterisation / Analysis

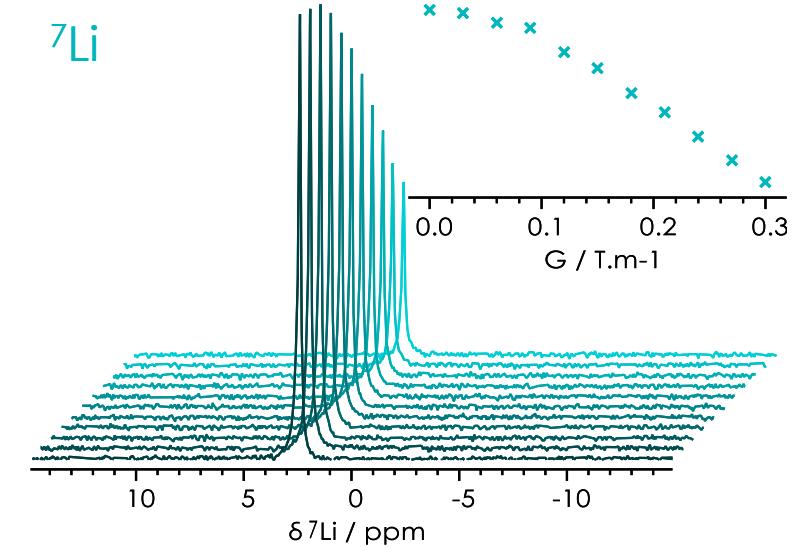
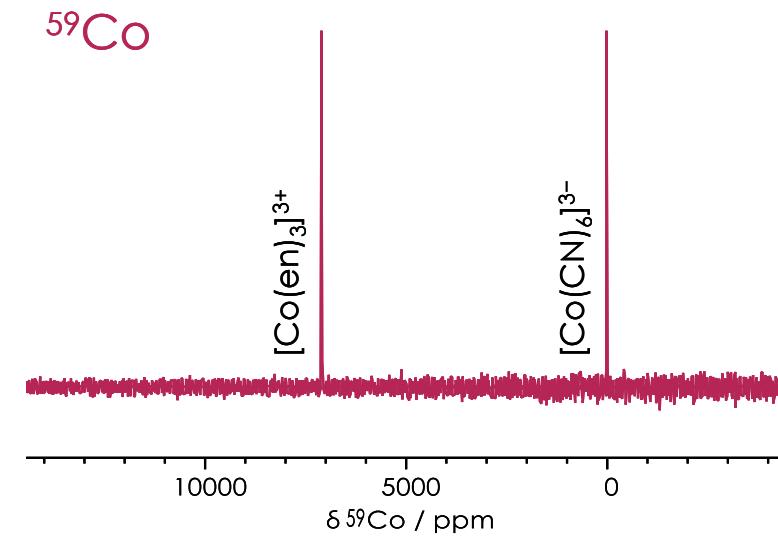
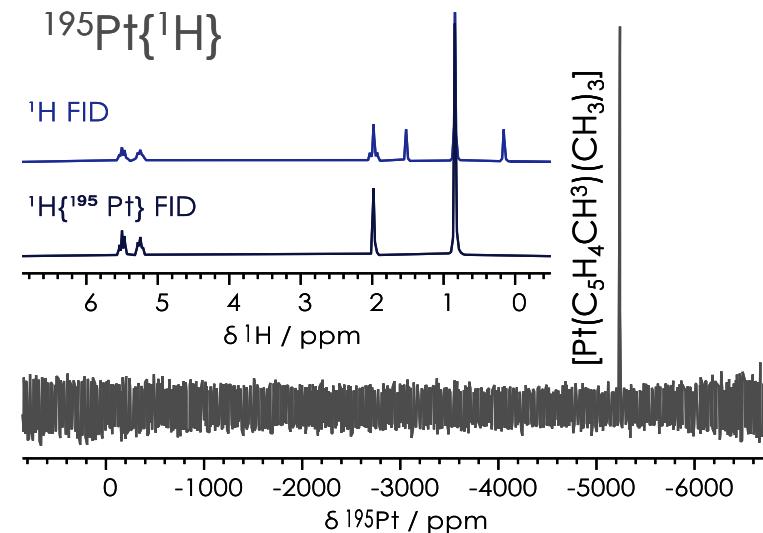
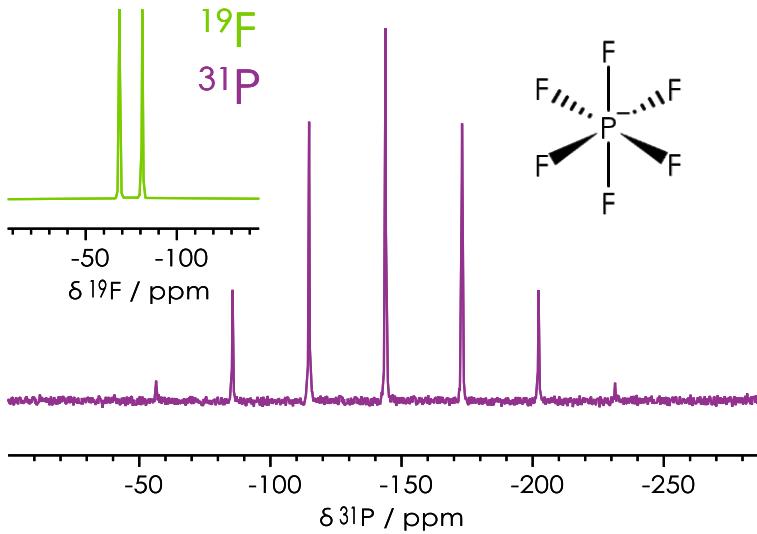
Small Molecule Characterisation / Analysis

- Characterisation & analysis of pure compounds & mixtures
 - ^1H , ^7Li , ^{11}B , ^{13}C , ^{19}F , ^{23}Na , ^{27}Al , ^{29}Si , ^{31}P , ^{59}Co , ^{195}Pt ...
 - one- & two-dimensional pulse sequences
 - FID, InvRec, CPMG, INEPT, DEPT, PGSE ...
 - COSY, TOCSY, NOESY, HSQC, HMBC, HMQC ...



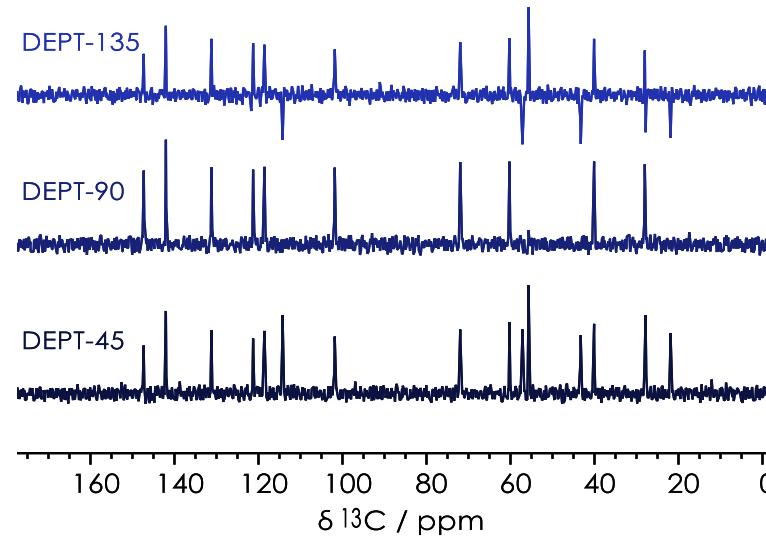
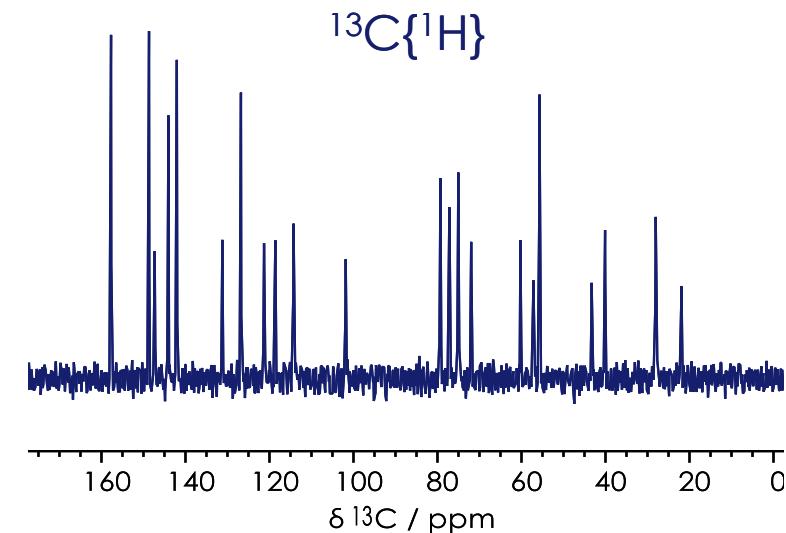
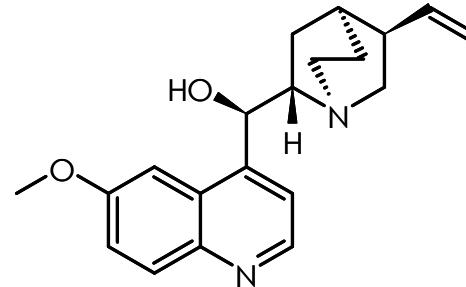
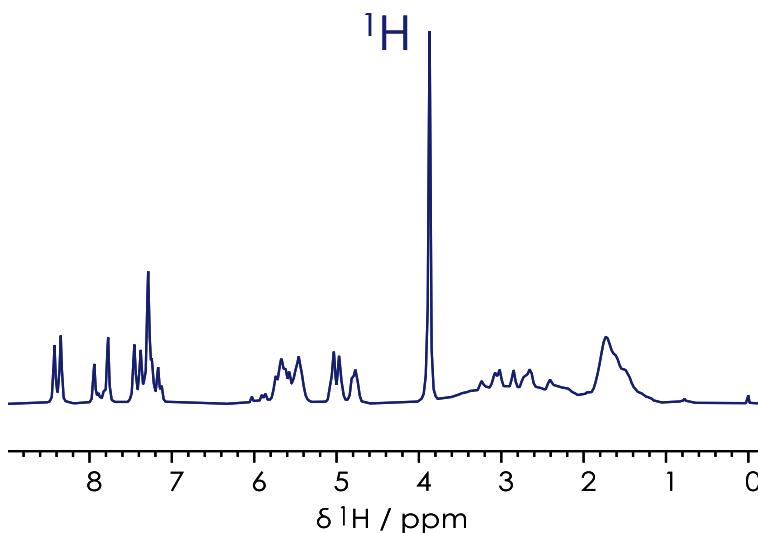
Small Molecule Characterisation / Analysis

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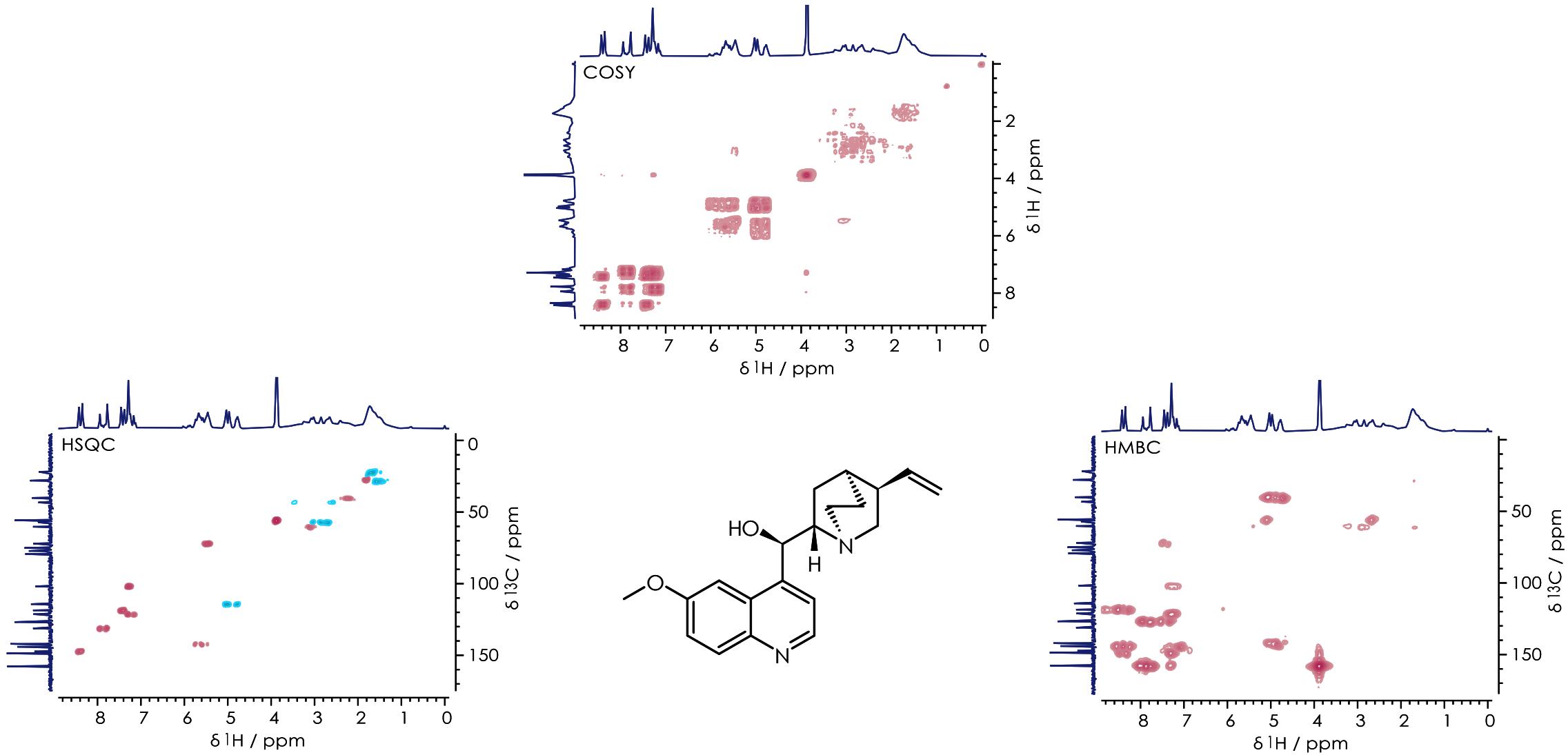
Small Molecule Characterisation / Analysis

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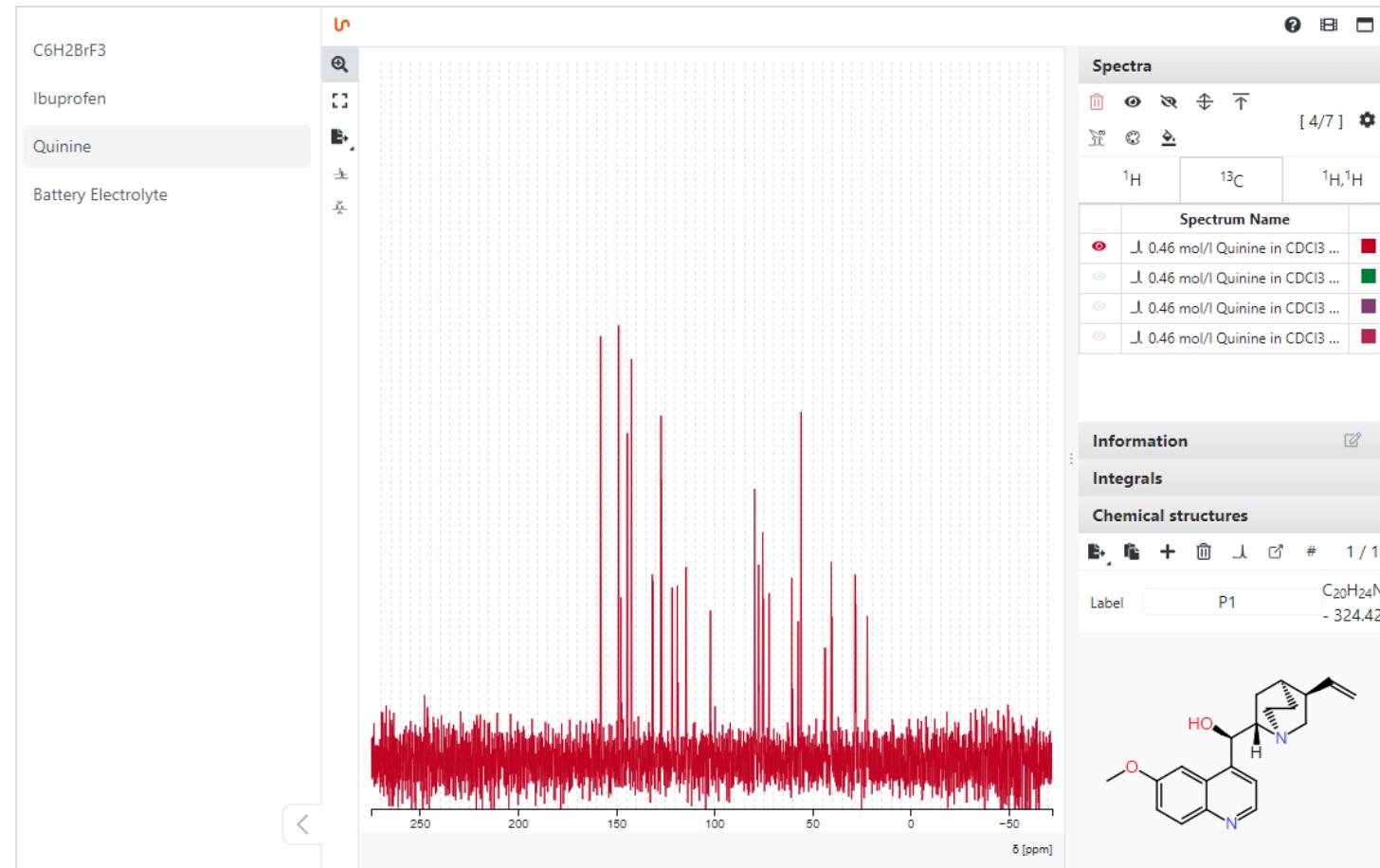
Small Molecule Characterisation / Analysis

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X-Pulse Spectra in NMRium

<https://nmr.oxinst.com/interactive-spectra-library>

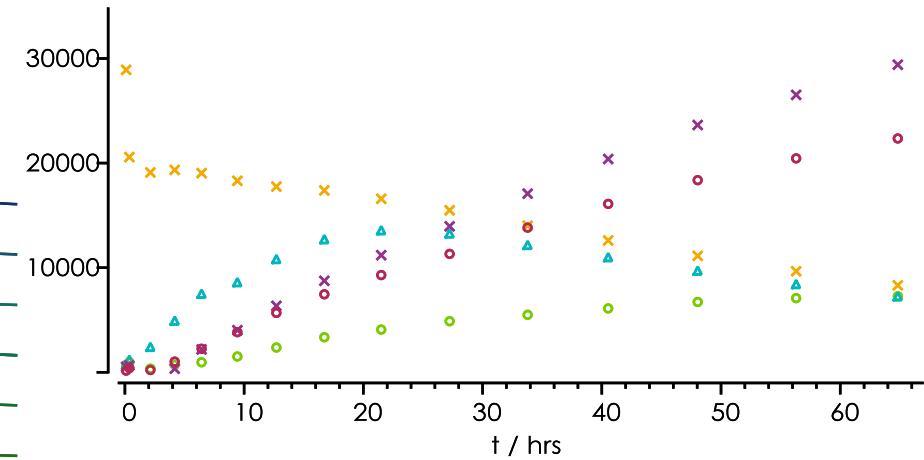
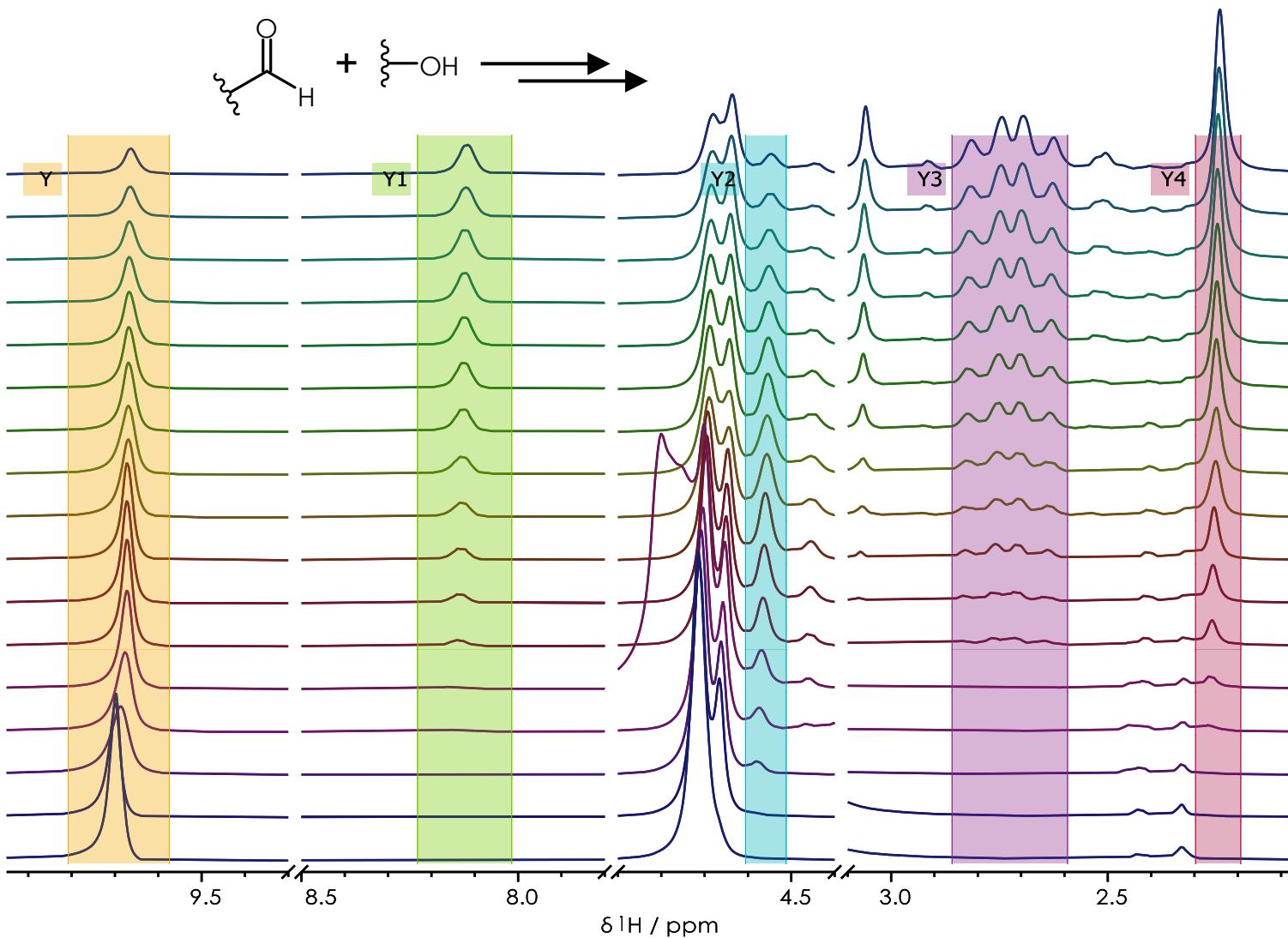


Reaction Monitoring & FlowNMR

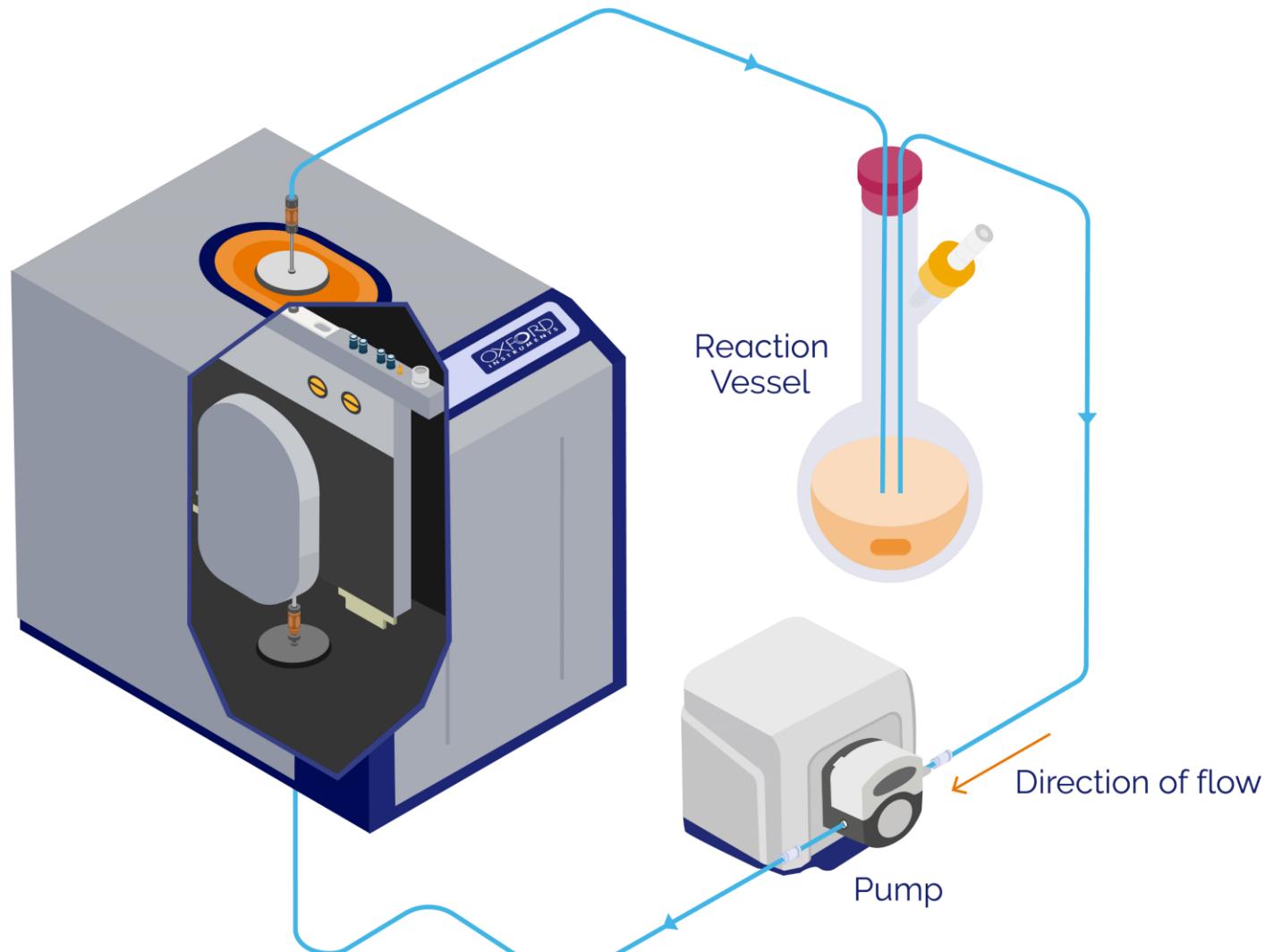
www.oxinst.com/webinars/advances-in-reaction-monitoring-with-flownmr

Reaction Monitoring

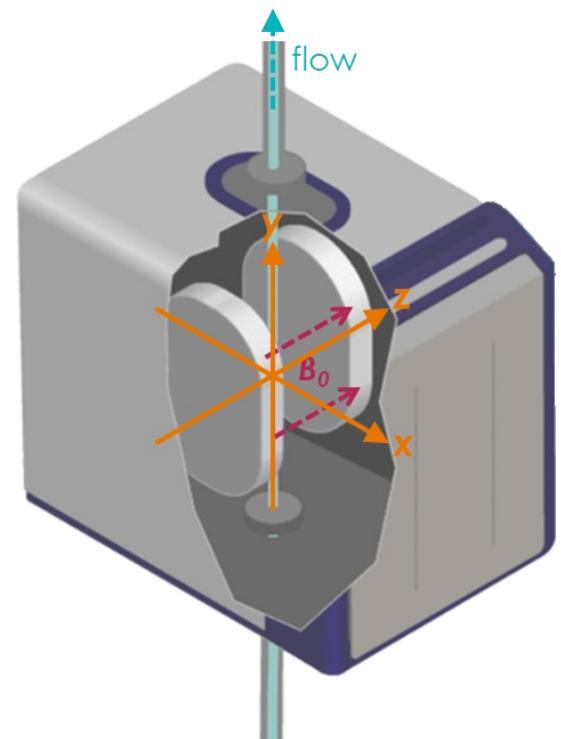
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- The aldehyde signal at $\delta_{\text{H}} +9.7$ ppm, decreases as the starting material is consumed
- An intermediate species is observed at $\delta_{\text{H}} +4.6$ ppm
- Signals corresponding to product species can be identified, including singlets at $\delta_{\text{H}} +8.1$ and $+2.3$ ppm, and a quartet at $\delta_{\text{H}} +2.7$ ppm



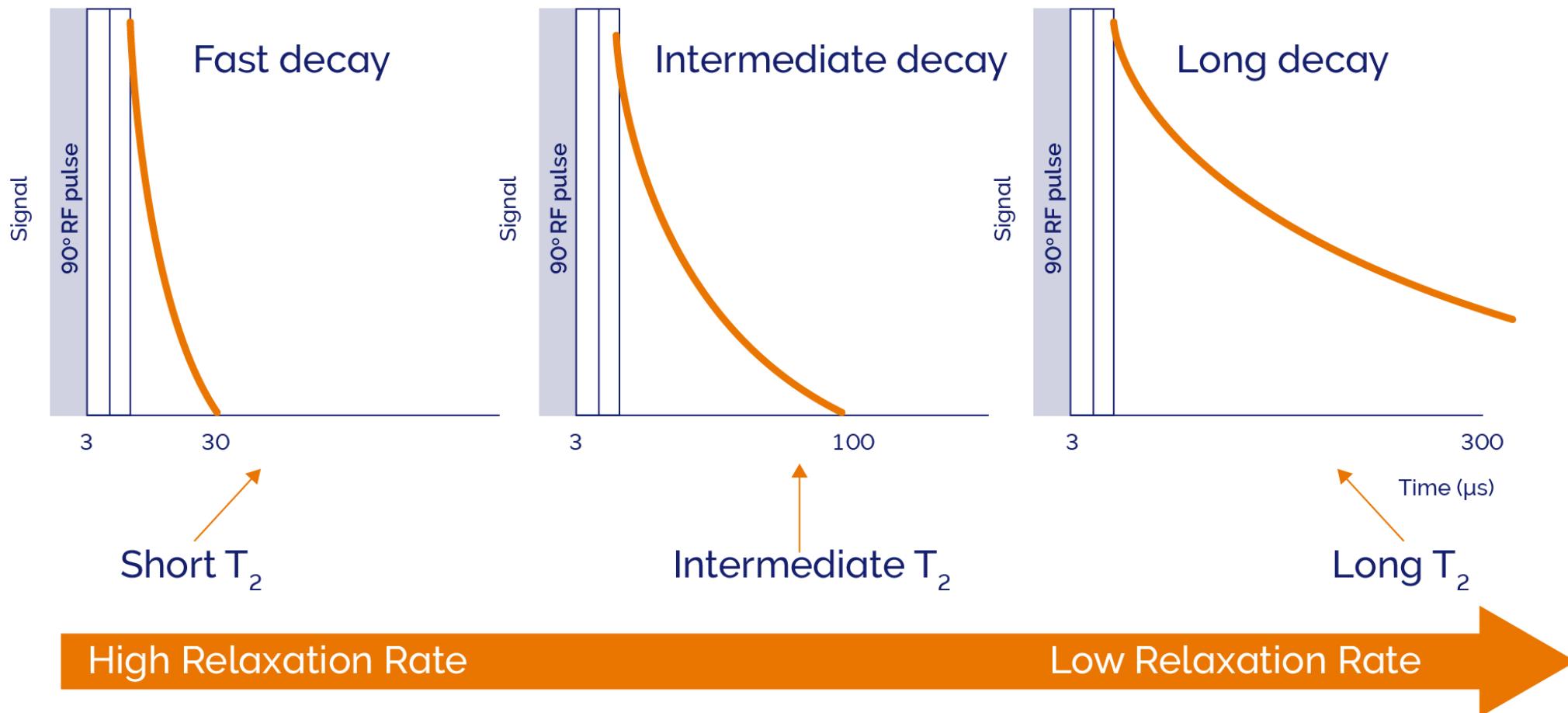
- Chemical reactions & processes can be monitored by flowNMR
 - calculation of rates
 - identification of intermediates / reaction completion
 - optimisation of reactions
- Wide range of applicable nuclei
 - ^1H , ^7Li , ^{11}B , ^{19}F , ^{23}Na , ^{31}P ...
- Quantitative NMR (qNMR) is possible in flow
 - quantification of $\text{Li}^{+}(\text{aq})$ and $\text{Na}^{+}(\text{aq})$ – extraction of lithium
- Three-axis field gradients allows for advanced experiments
 - solvent suppression (WET) in flow
 - measuring diffusion constants / polymer sizing in flow



Time-Domain NMR

MQC+

What is Time-Domain NMR ?



Solids

- proteins, carbohydrates and fibre in foods
- polymer/biopolymer matrices of samples
- crystalline polymer components

Solid-like components

- bound water
- residual moisture
- amorphous polymer components

Liquids

- oils/fats
- spin-finish lubricants



- Oxford Instruments MQC family of TD-NMR Instruments
 - 5 MHz or 23 MHz permanent magnet
 - user removable / exchangeable probes
 - Hydrogen-1 (proton) or Fluorine-19
 - 10 – 60 mm sample tube diameter
 - solid and/or liquid samples
 - optional variable temperature configuration (-5 to +70°C)
- QC/QA and full research grade systems
- Applications often replace ‘wet chemistry’ methods
- ISO & ASTM standard methods

Summary

Summary



- A good benchtop NMR spectrometer can (from an NMR point-of-view) do everything a high-field NMR spectrometer can do
 - that **doesn't** mean they can be used for all applications, though probably for more than you think ...
- Benchtop form-factor gives more system flexibility
 - fume cupboards, glove boxes, pilot plants ...
- NMR isn't just spectroscopy (and MRI) !
 - time-domain NMR / NMR relaxometry



Any Questions ?

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magres@oxinst.com

inclusive • innovative • trusted • purposeful