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

SNUG 2024 Postgraduate Course in NMR Spectroscopy

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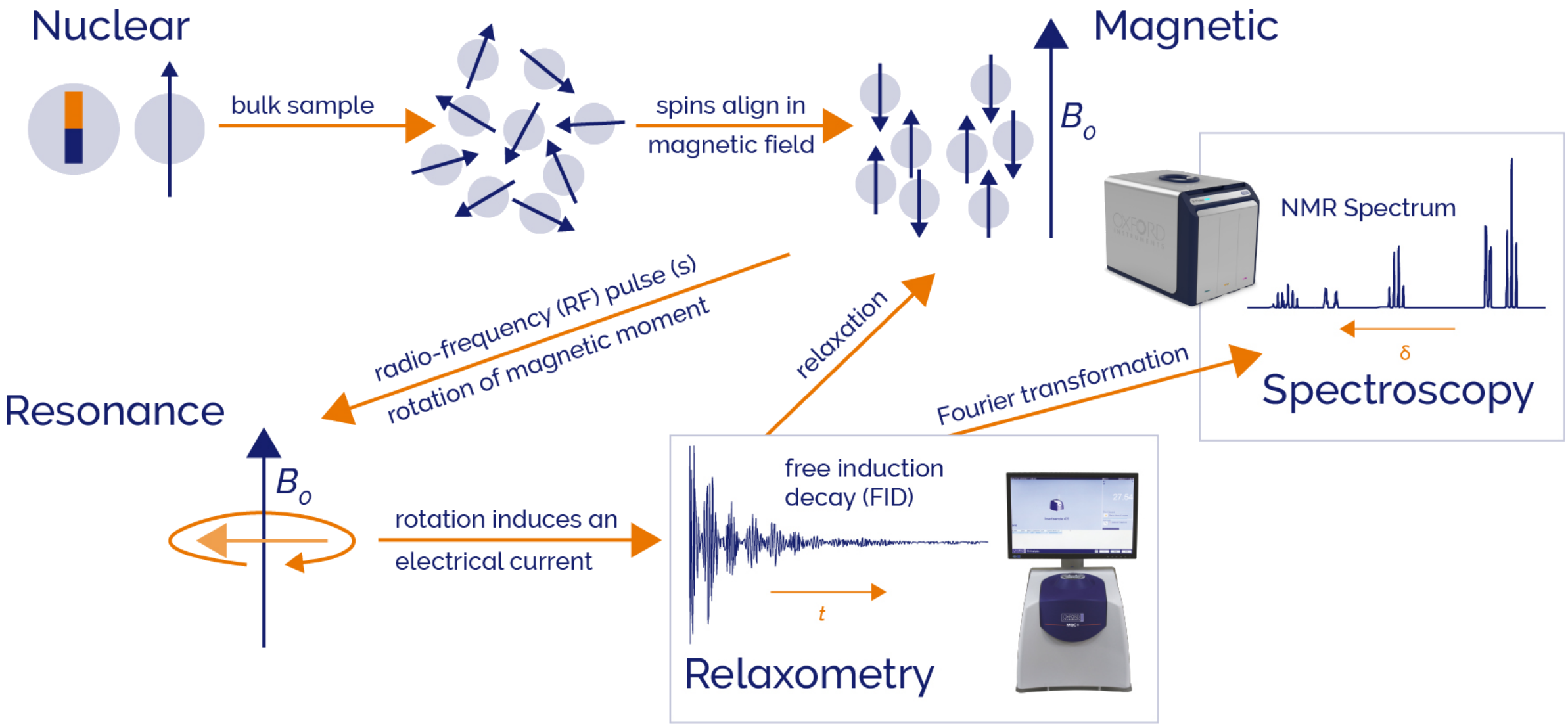
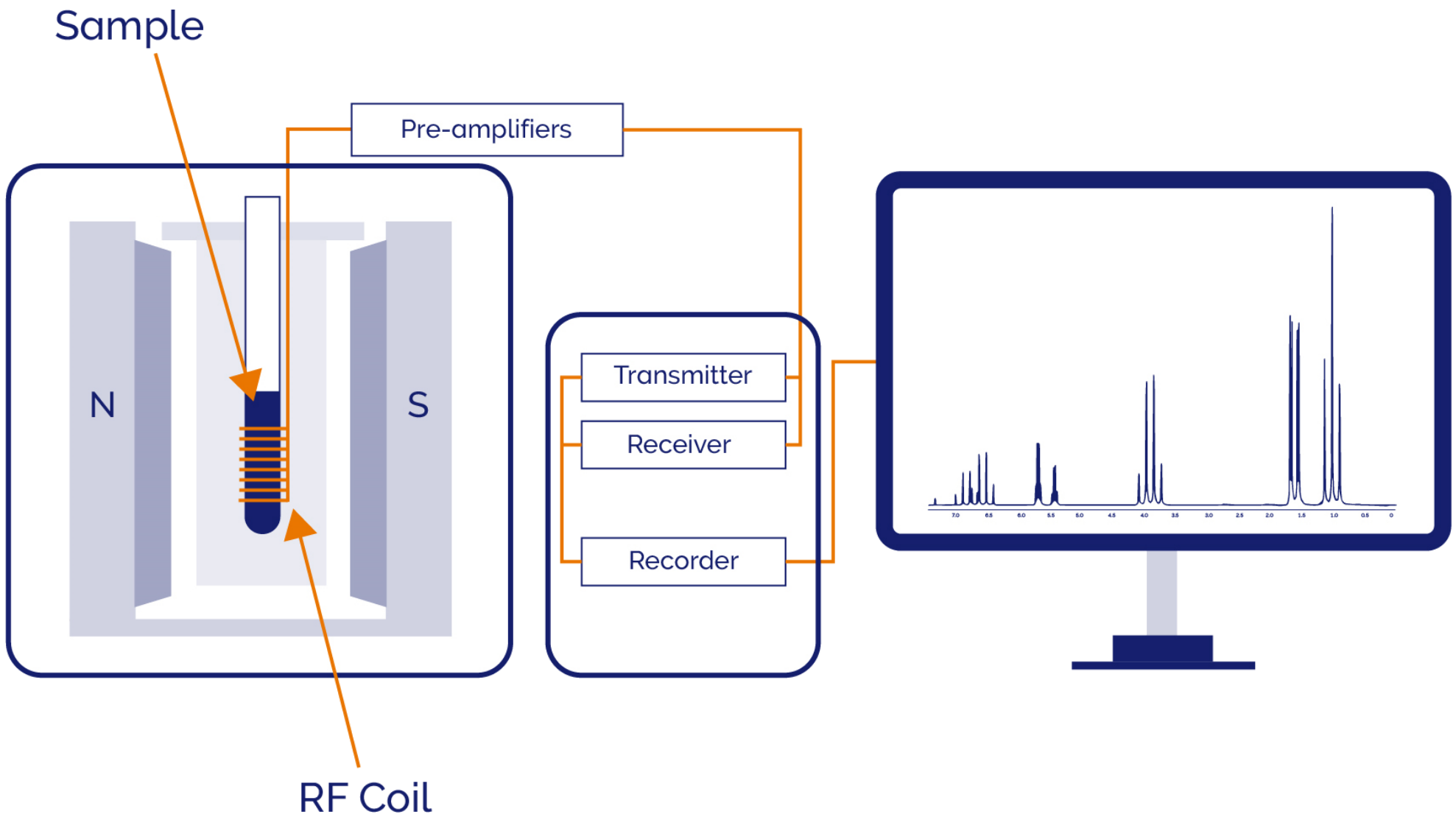
Introduction

- Benchtop NMR
- Benchtop NMR Spectroscopy
- Small Molecule Characterisation / Analysis
- Reaction Monitoring & FlowNMR
- Time-Domain NMR

What is NMR ?



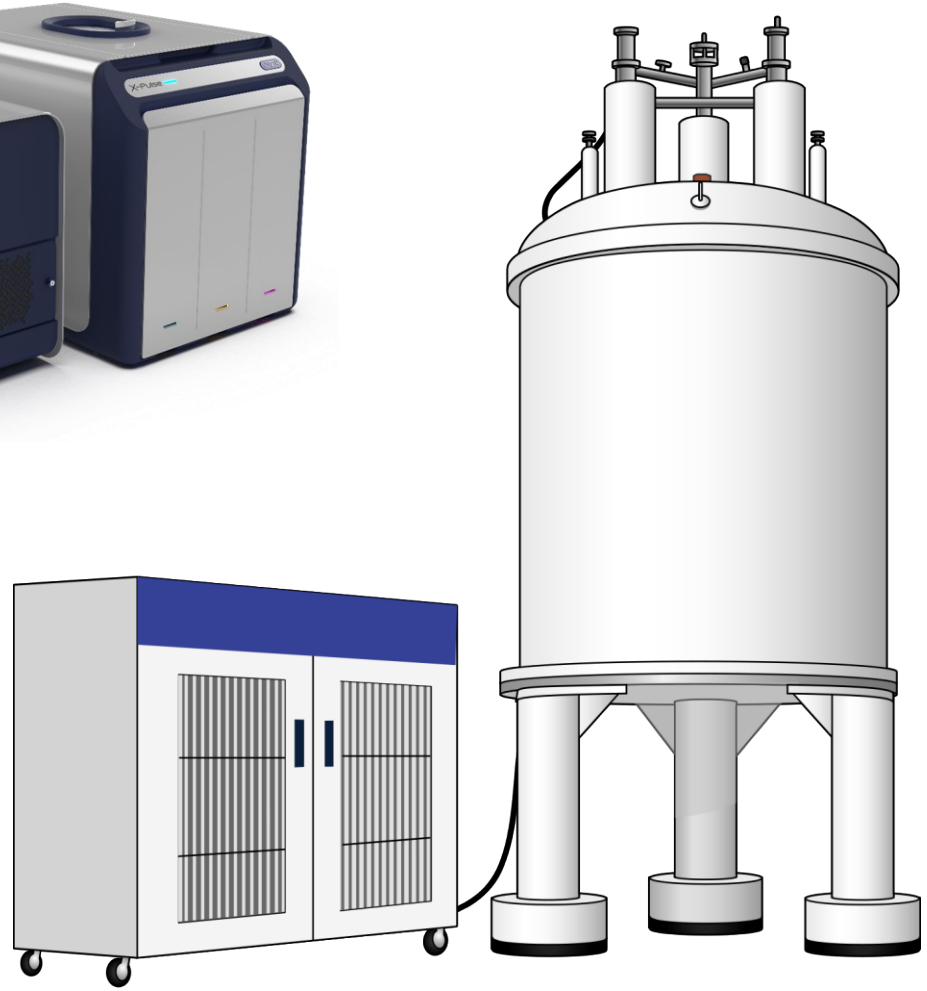
What is an NMR Spectrometer / Relaxometer ?



Benchtop *verses* High-field NMR

- Benchtop NMR
  - up to 120 MHz
  - permanent magnets ($Nd_2Fe_{14}B$)

- 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

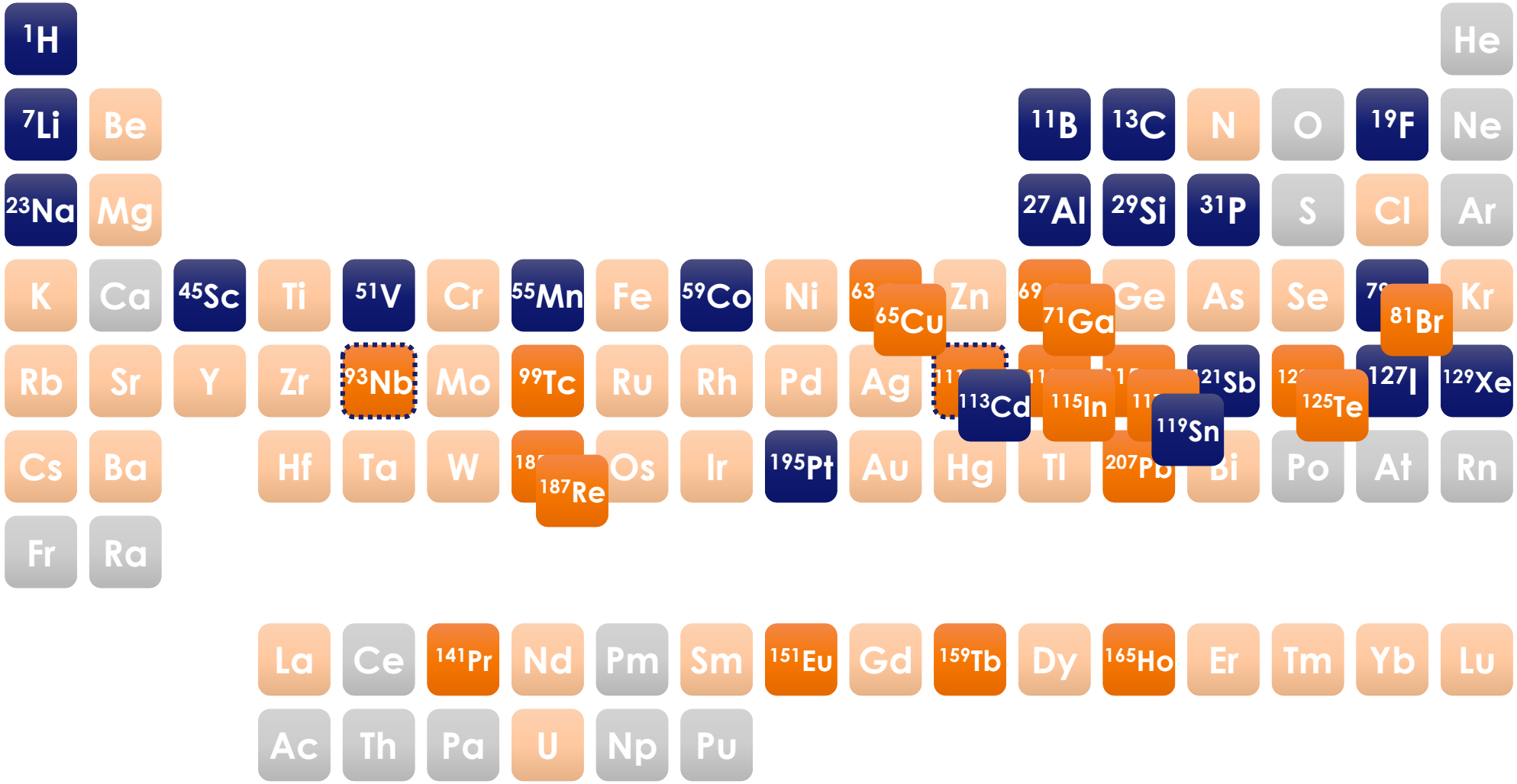
- 1.4 T / 60 MHz permanent magnet
- High resolution ($CHCl_3$ in acetone-d_6)
  - $\nu_{1/2}$ ≤ 0.35 Hz / ≤ 10 Hz @ ^{13}C satellites
- High sensitivity (1% ethylbenzene in $CDCl_3$)
  - ≥ 140 signal-to-noise ratio (SNR)

- Three-axis field gradients as standard (z-axis: ≥ 0.5 $T.m^{-1}$)
  - 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 (**W**ater suppression **E**nhanced through T_1 effects)
  - selective one-dimensional TOCSY, NOESY

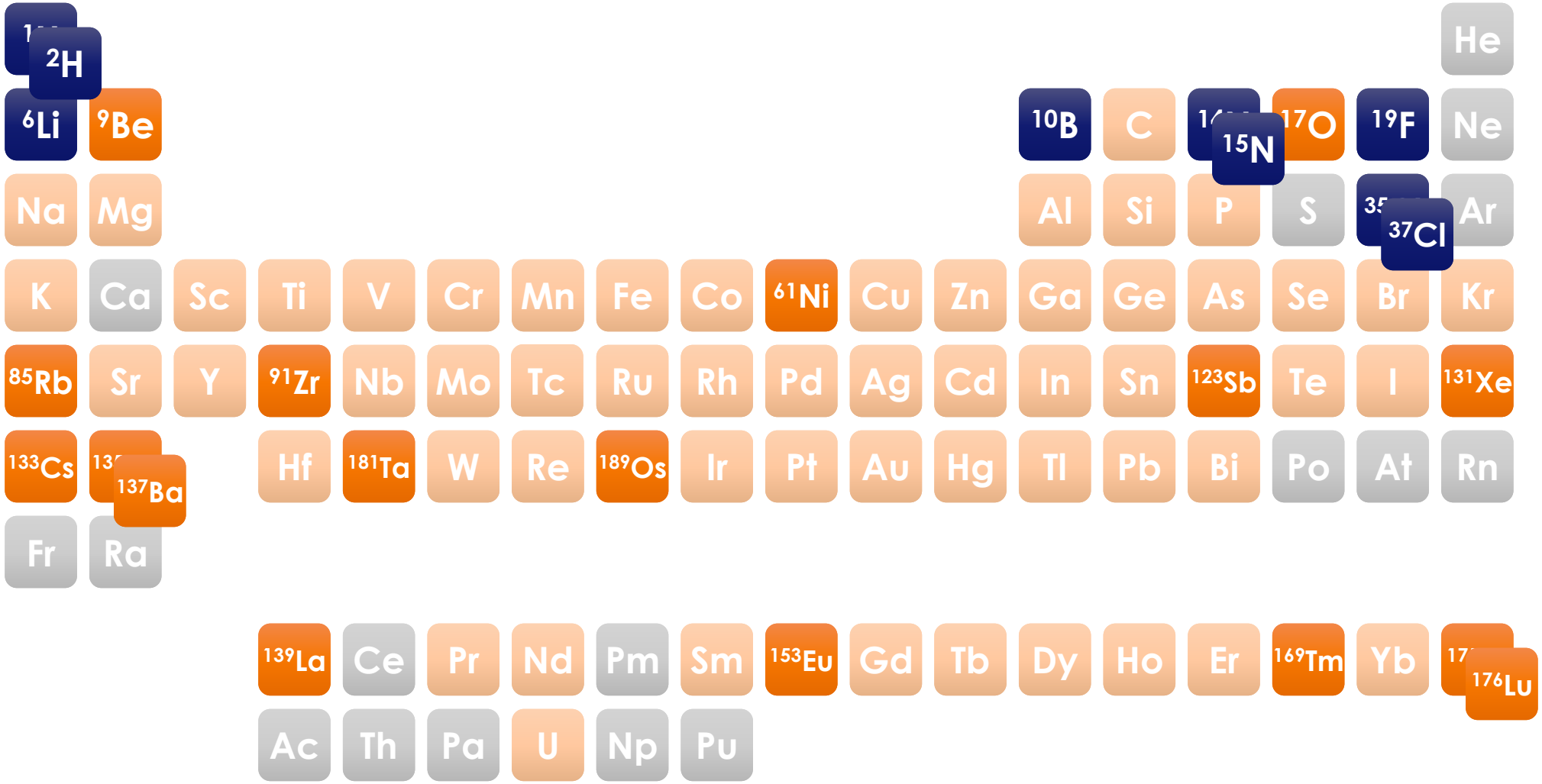
X-Pulse Broadband Benchtop NMR Spectrometer

- Three channel spectrometer
  - Proton/fluorine channel
  - Broadband X-channel *(optional)*
    - ^{7}Li, ^{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)



X-Pulse Periodic Table (low-frequency)



Pulse Sequence Programming / Development

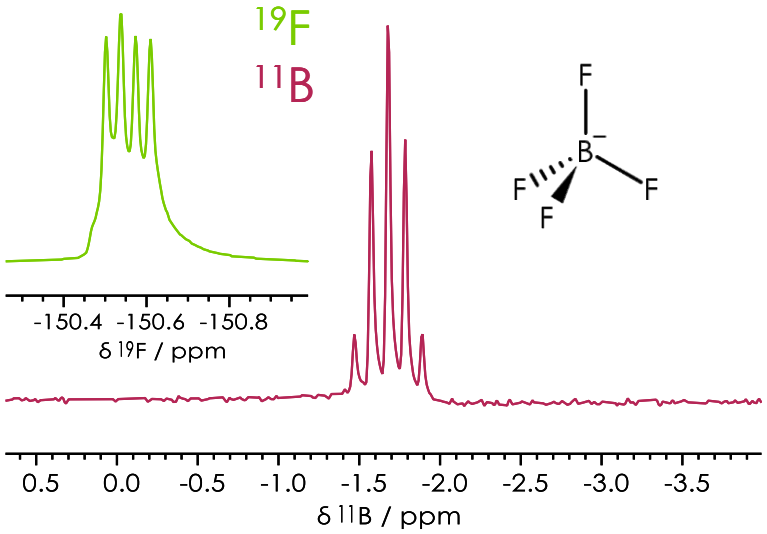
- Sequences programmed in Python
  - complete control of spectrometer hardware
  - pre- / post-acquisition processing

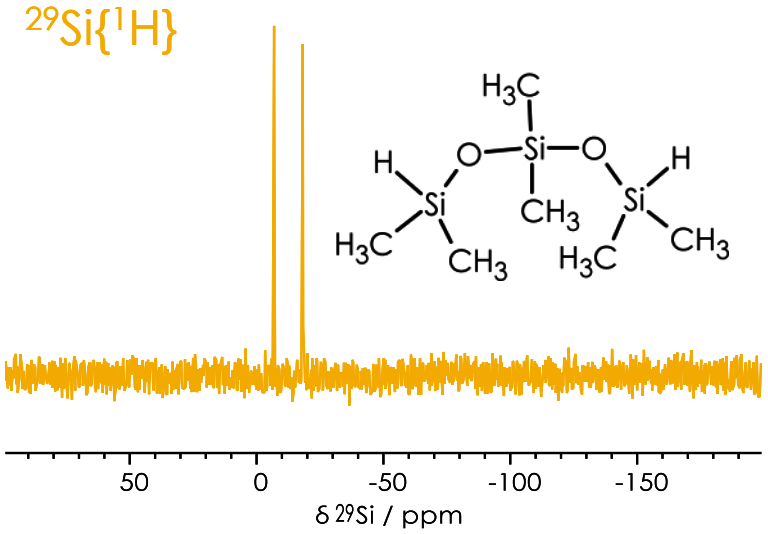
- Two radio-frequency channels
  - high- & low-power
  - shaped / adiabatic pulses
- Three bipolar gradient channels
- External (TTL) triggers
  - input & output
- Customisable JCAMP-DX output files

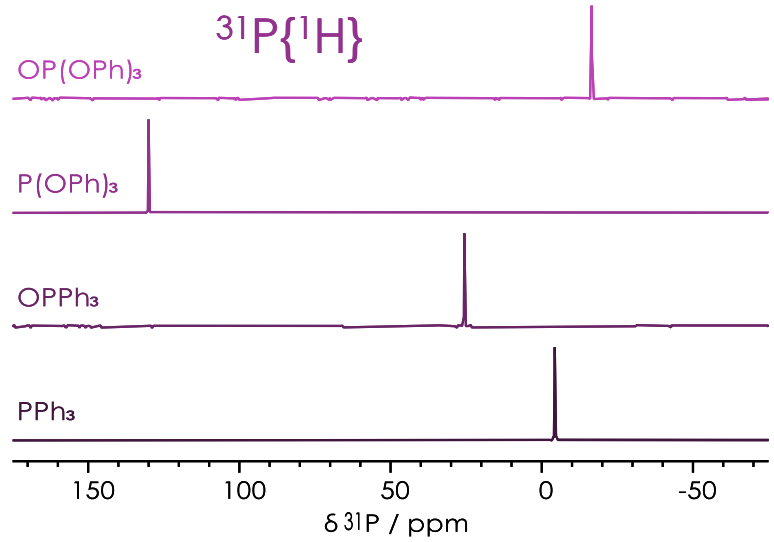
Small Molecule Characterisation / Analysis

Small Molecule Characterisation / Analysis

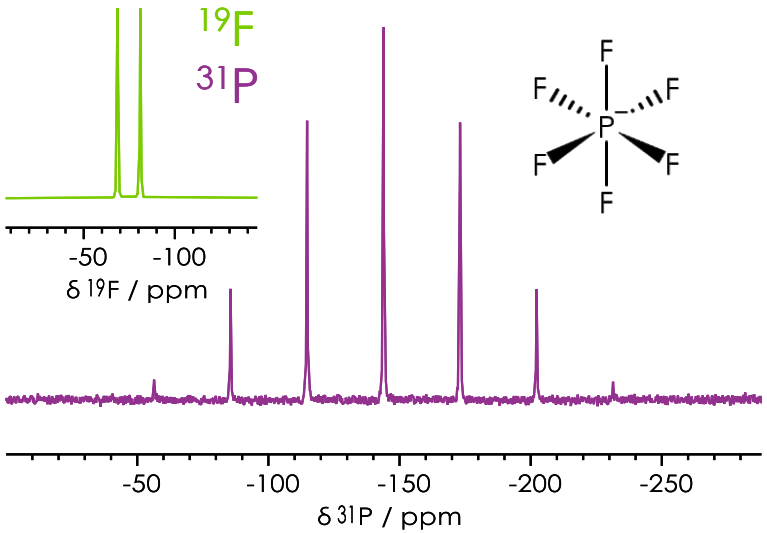
- Characterisation & analysis of pure compounds & mixtures
  - ^{1}H, ^{7}Li, ^{11}B, ^{13}C, ^{19}F, ^{23}Na, ^{27}Al, ^{29}Si, ^{31}P, ^{59}Co, ^{195}Pt …
  - one- & two-dimensional pulse sequences
    - FID, InvRec, CPMG, INEPT, DEPT, PGSE …
    - COSY, TOCSY, NOESY, HSQC, HMBC, HMQC …

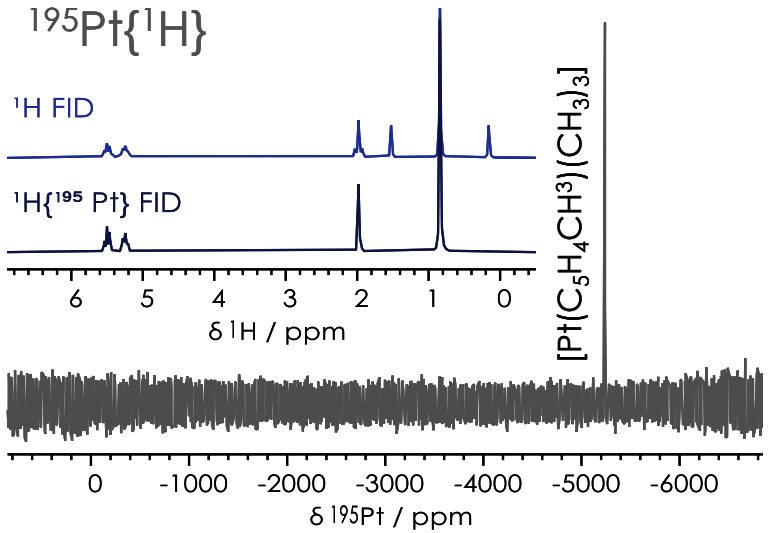


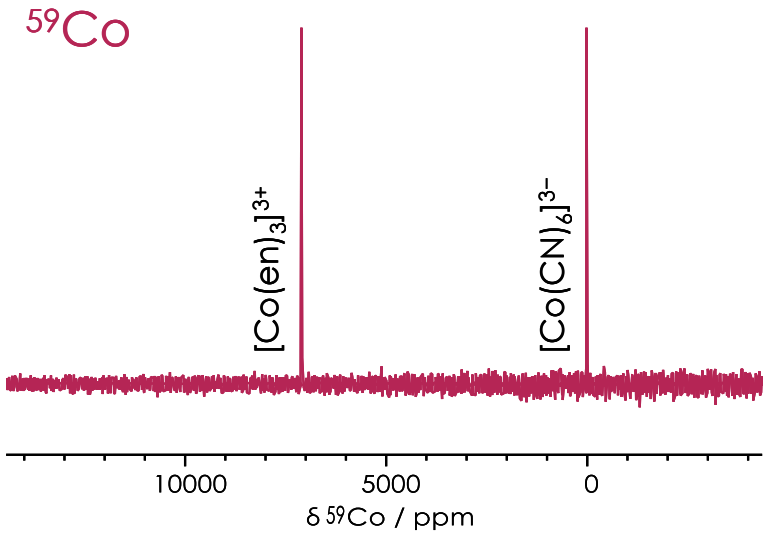


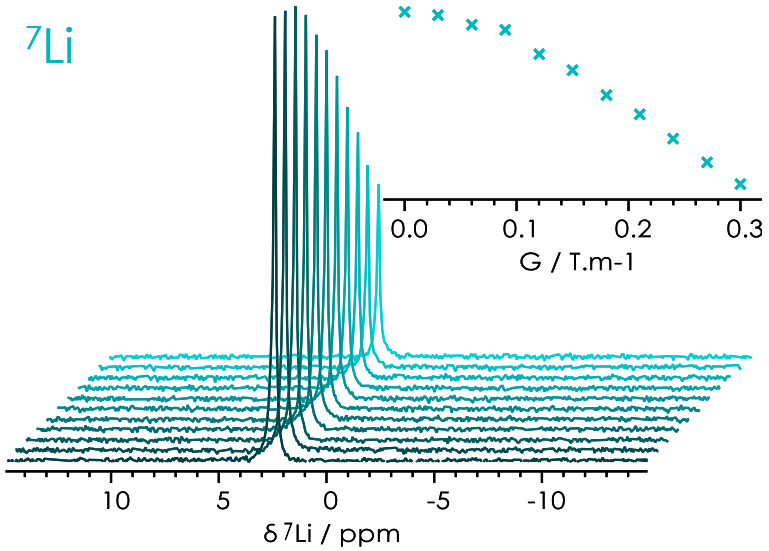


Small Molecule Characterisation / Analysis









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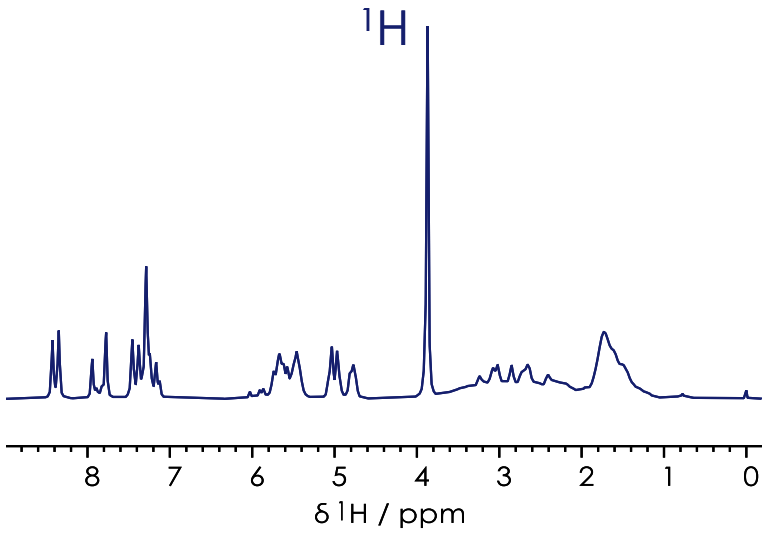

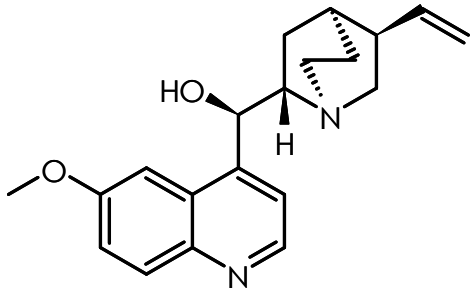

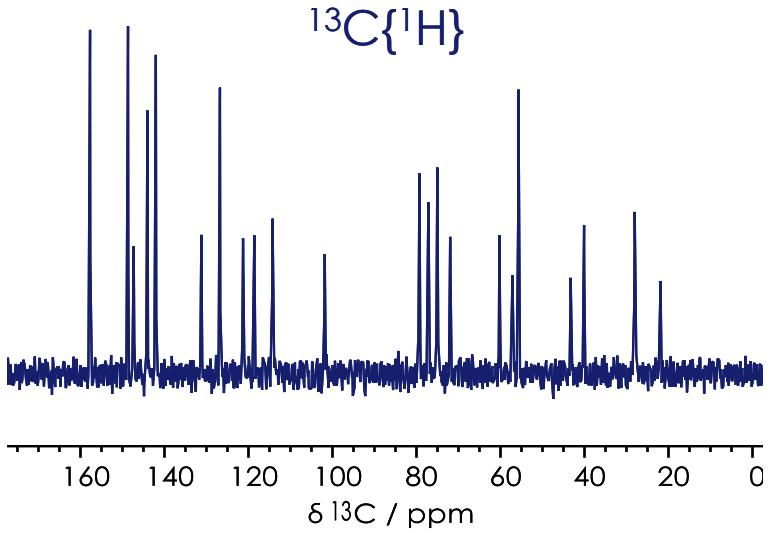

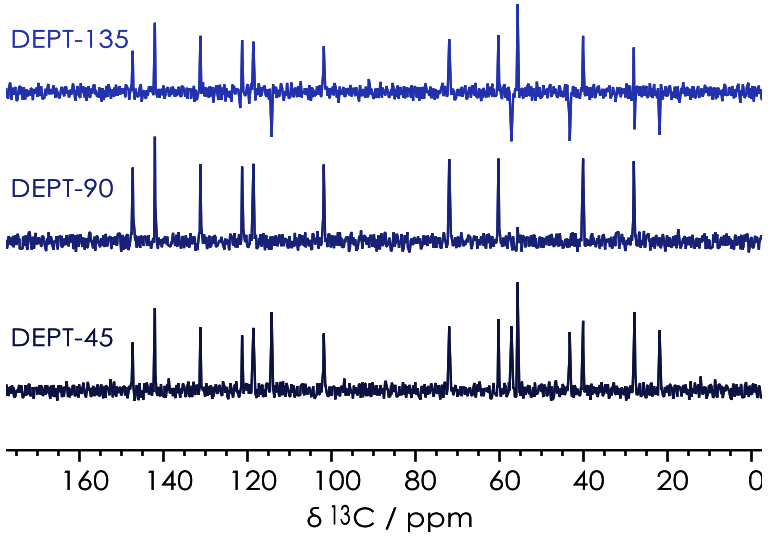

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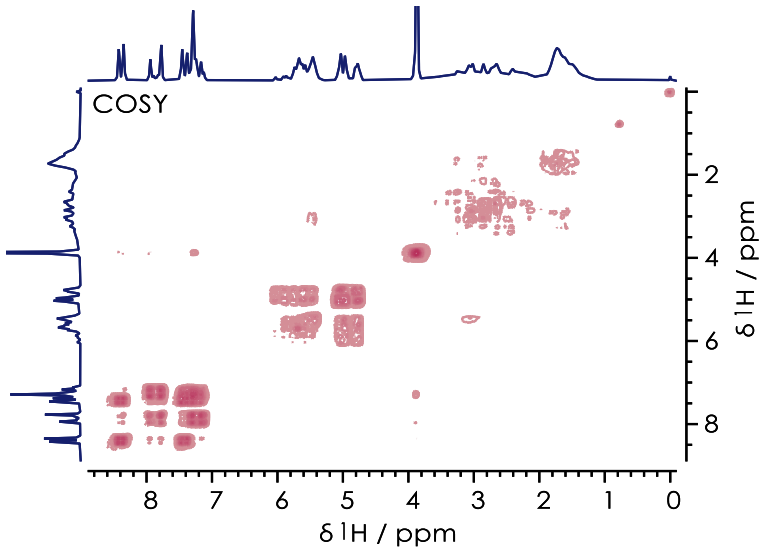
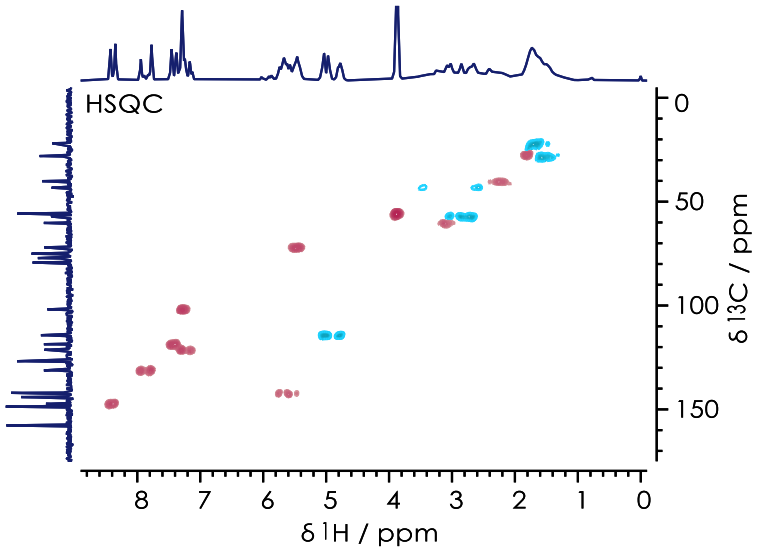
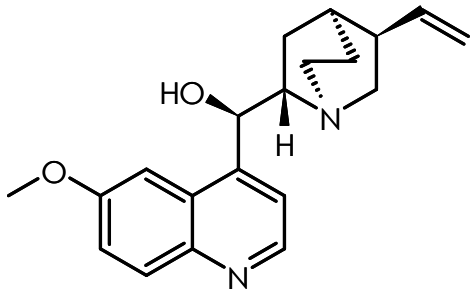
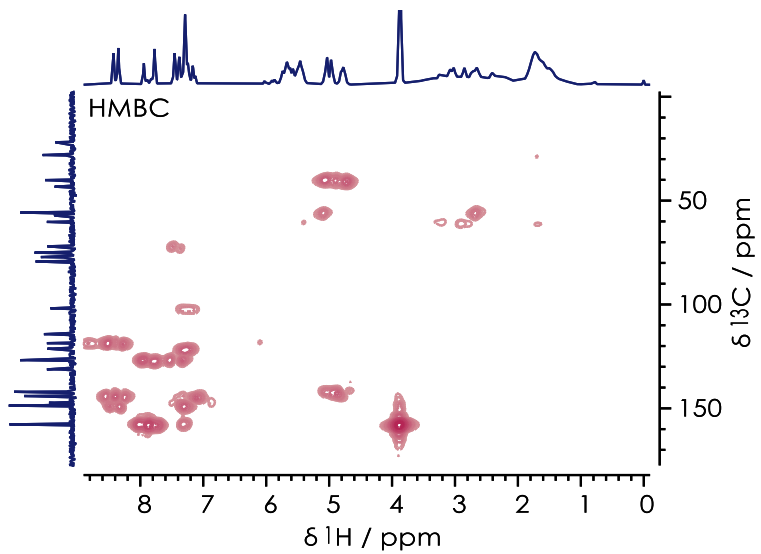


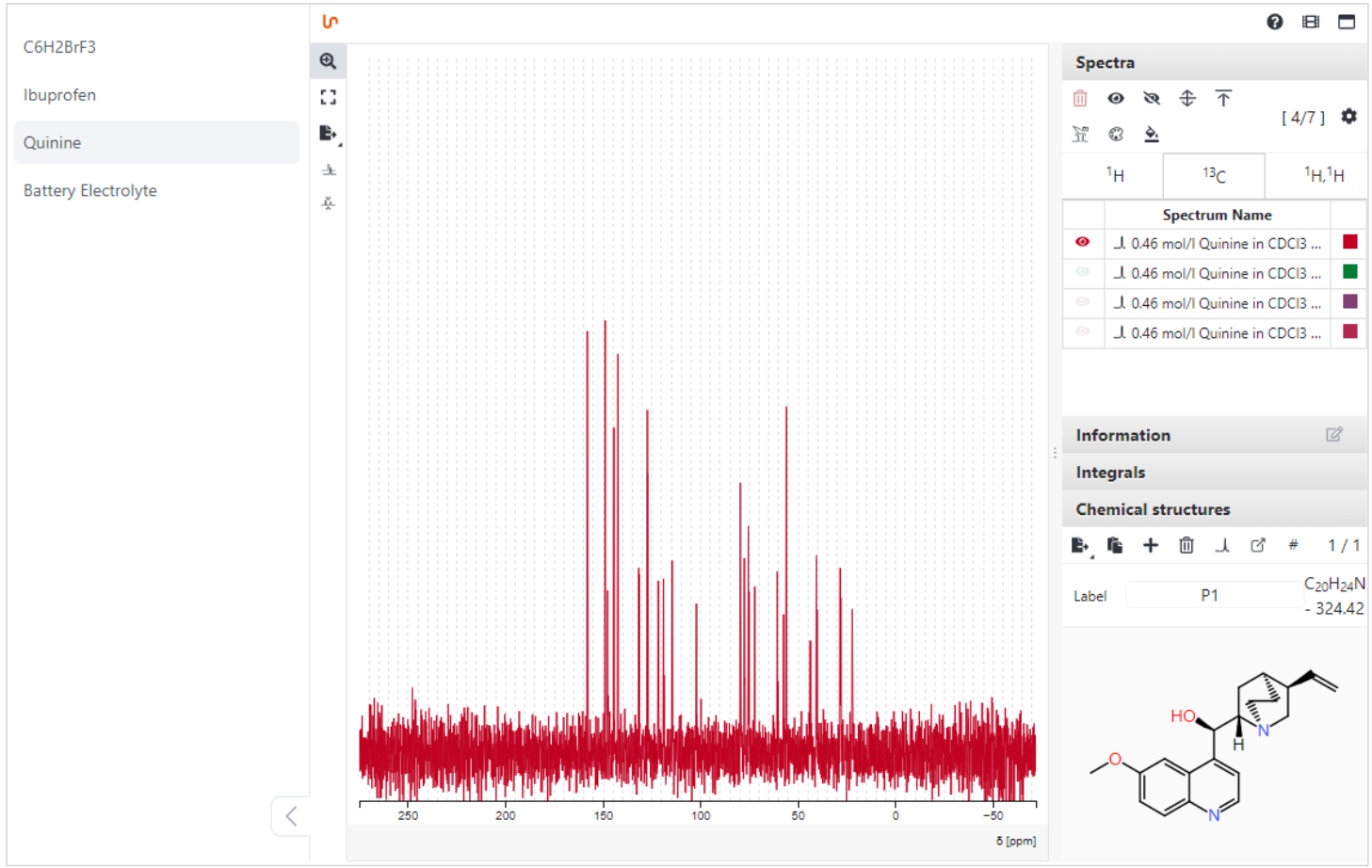






X-Pulse Spectra in NMRium

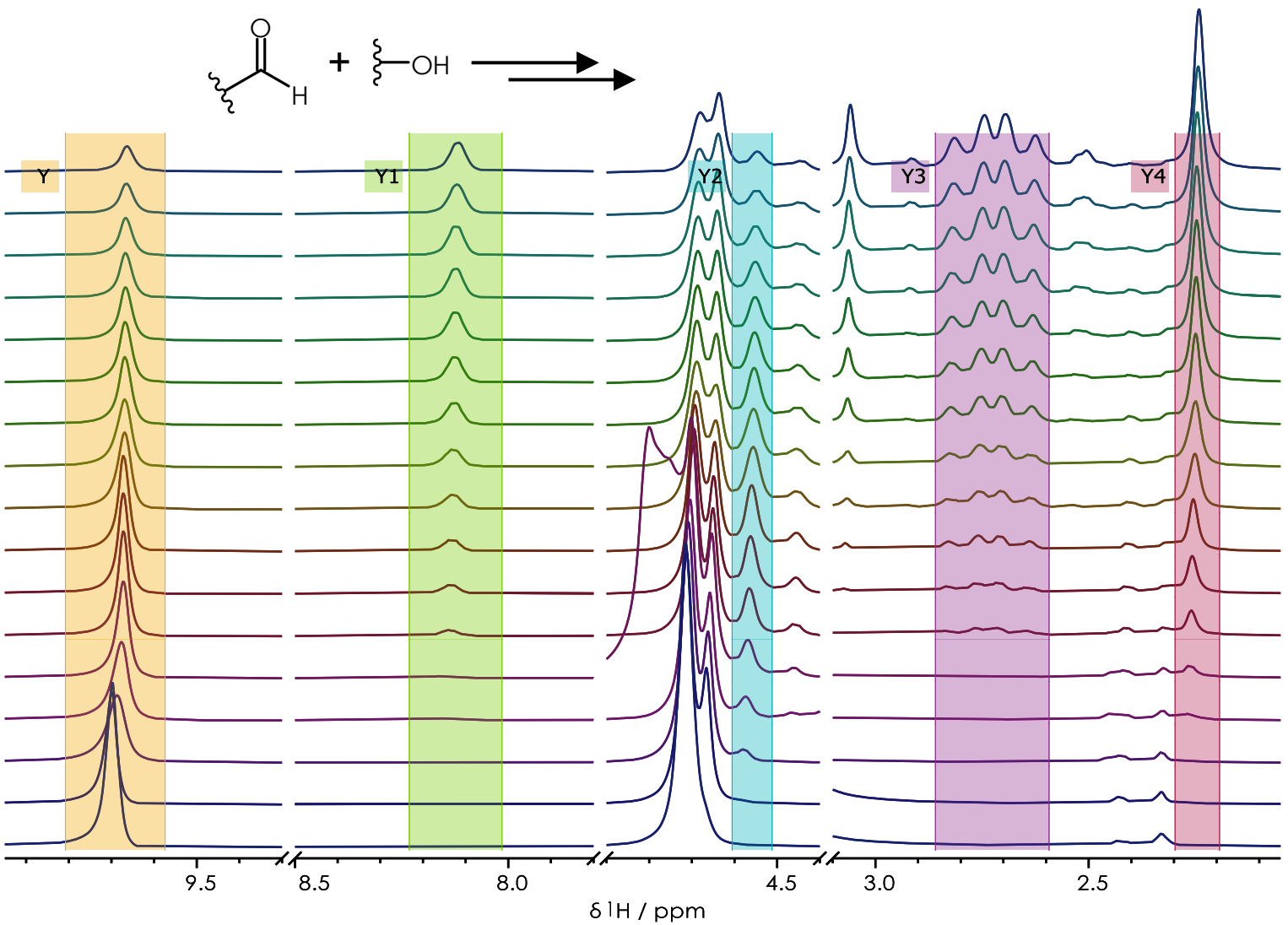
https://nmr.oxinst.com/interactive-spectra-library

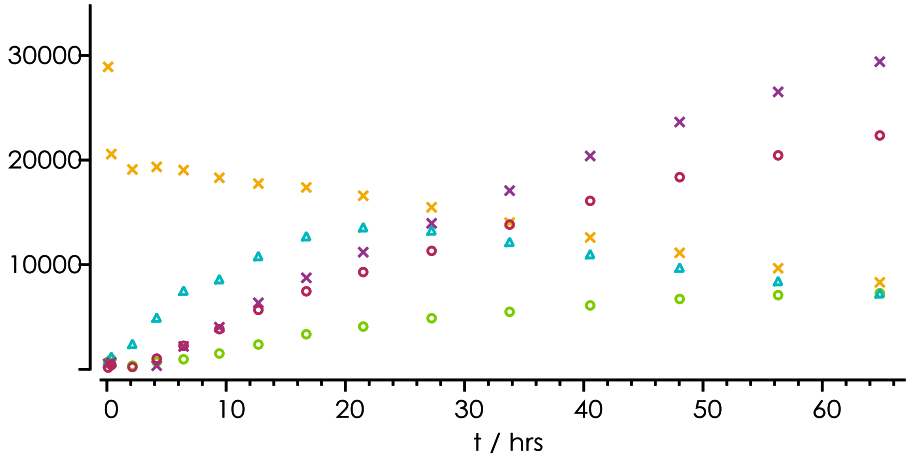


Reaction Monitoring & FlowNMR

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

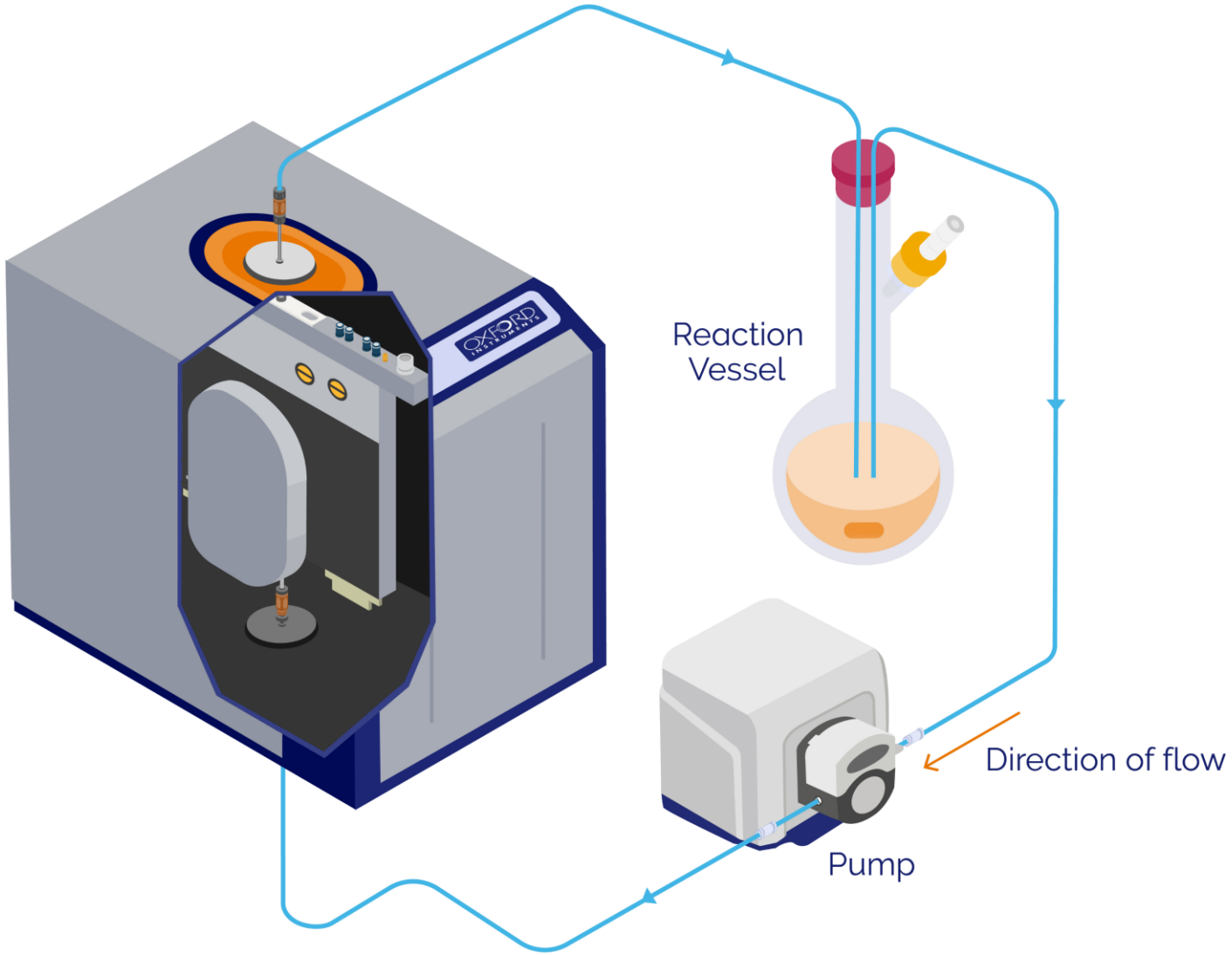
Reaction Monitoring





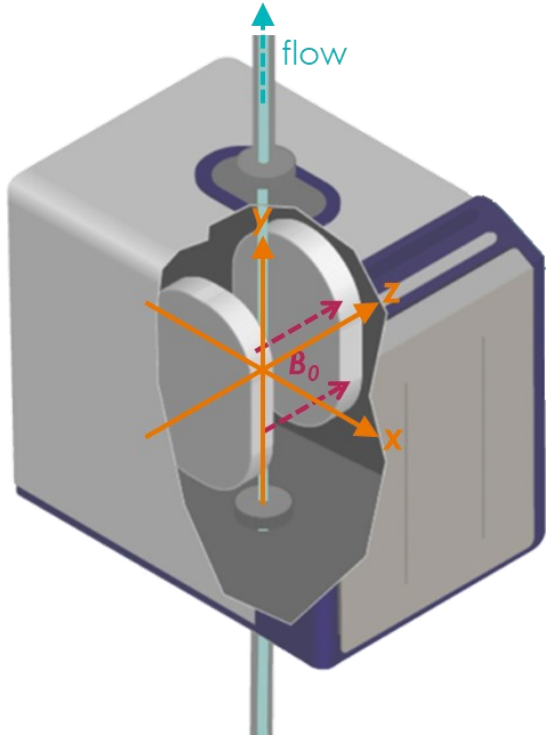
- The aldehyde signal at δ_H **+9.7** ppm, decreases as the starting material is consumed
- An intermediate species is observed at δ_H **+4.6** ppm
- Signals corresponding to product species can be identified, including singlets at δ_H **+8.1** and **+2.3** ppm, and a quartet at δ_H **+2.7** ppm

FlowNMR



FlowNMR

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

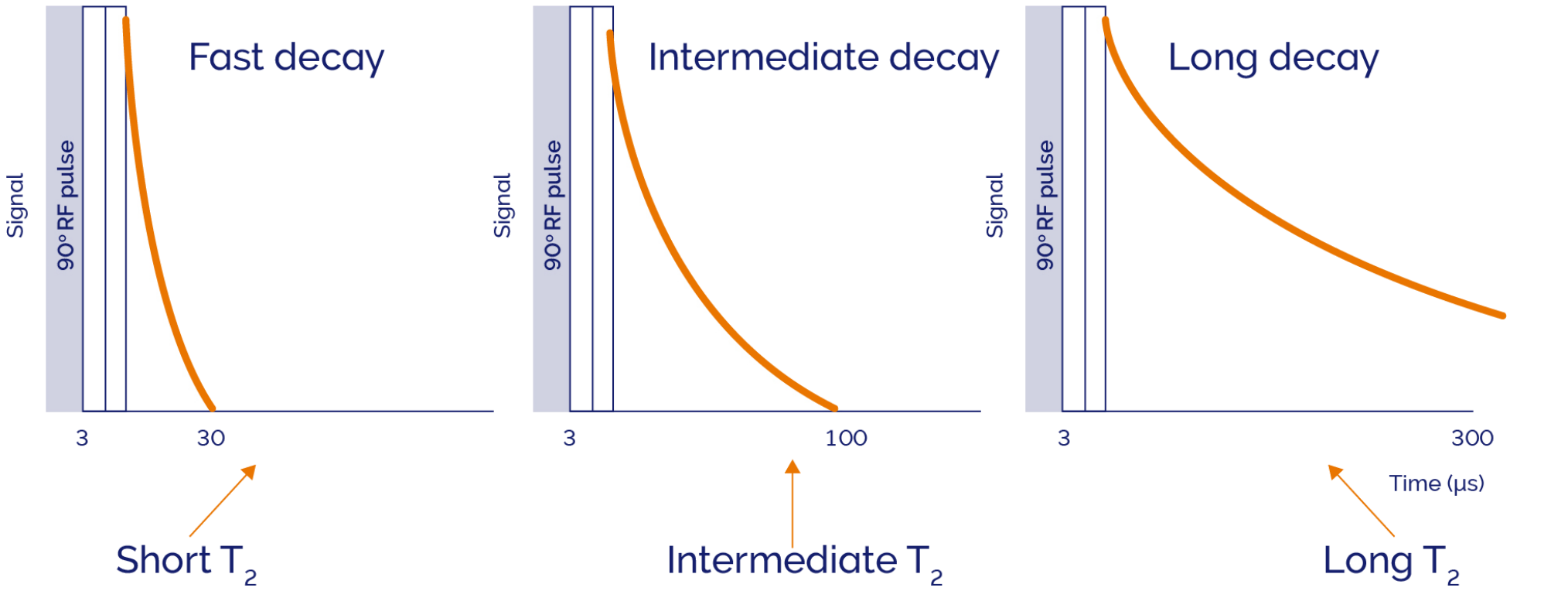


OXFORD INSTRUMENTS

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

Time-domain NMR

- Oxford Instruments MQC family of TD-NMR Instruments
  - 5 MHz or 23 MHz permanent magnet
  - user removeable / 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 ?

nmr.oxinst.com

www.linkedin.com/showcase/magnetic-resonance/

magres@oxinst.com

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