CCPN Conference 2018

CCPN

University of Kent

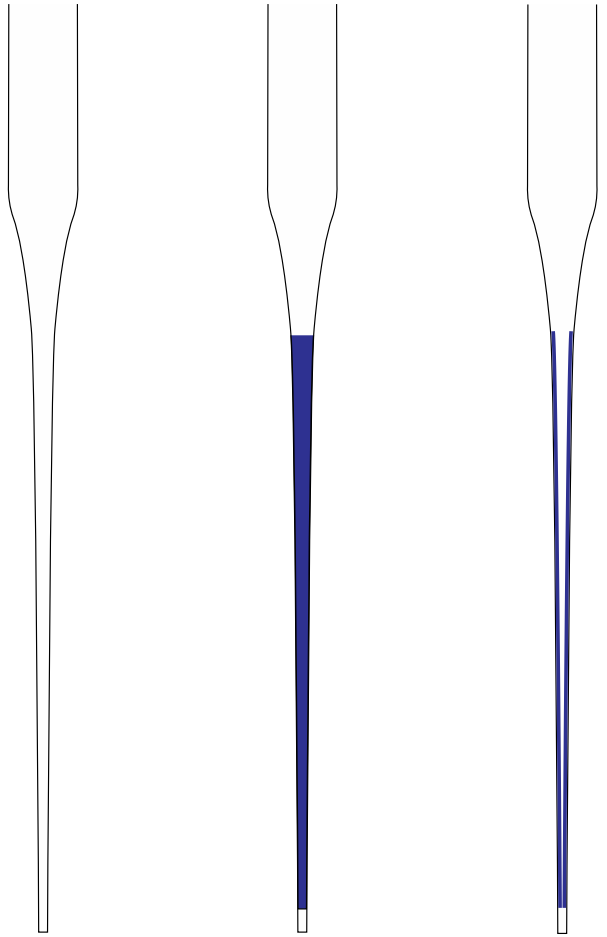
CCPN 2018

- Thursday 6th – Saturday 8th September
- Preliminary Program
  - Thursday PM Very & Ultra High Field + technical aspects of biomolecular NMR
  - Friday AM Protein-Ligand Interactions
  - Saturday 1Day Joint CCPN CCP - EM Meeting
  - Saturday AM Integrated Structural Biology with cryo-EM and NMR
  - Saturday PM EM in Biological Science, The resolution revolution

- Speakers: Brian Smith, Michele Vendruscolo, Sebastian Hiller, Marielle Walti, Enrico Luchinat

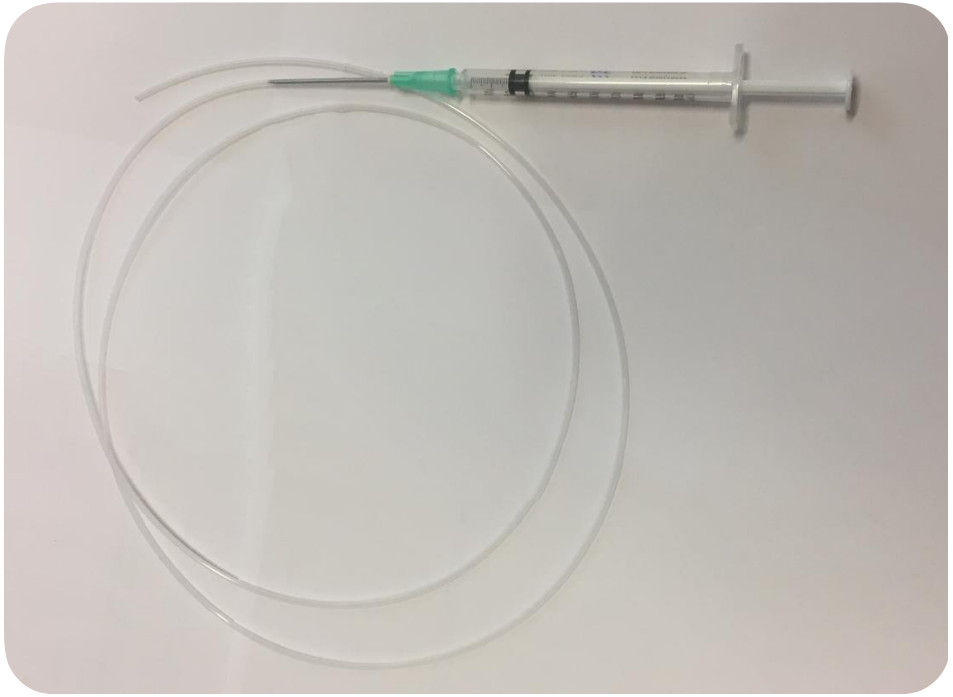
Saving Water: a tale of narrow tubes and water

- manipulating aqueous samples in small tubes (3mm bruker shaped etc) leads to losses
- Water sticks to glass pipettes (Si-OH$\shortparallel\shortparallel\shortparallel\shortparallel$OH$_2$)
- Glass pipette? draw as little sample into a long glass pipette as possible & expel
- Disadvantage still sticks to glass so some loses



Of narrow tubes and water II

- Solution
- use 1ml syringe 21G blunt needle (OD =0.8mm) ptfe tube (ID 0.8mm)
- Cutoff a length equal to 2mm/µl (area of 0.8mm ID = 0.5mm^2)
- Fill liquid into ptfe tube not syringe



Of narrow tubes and water: Details

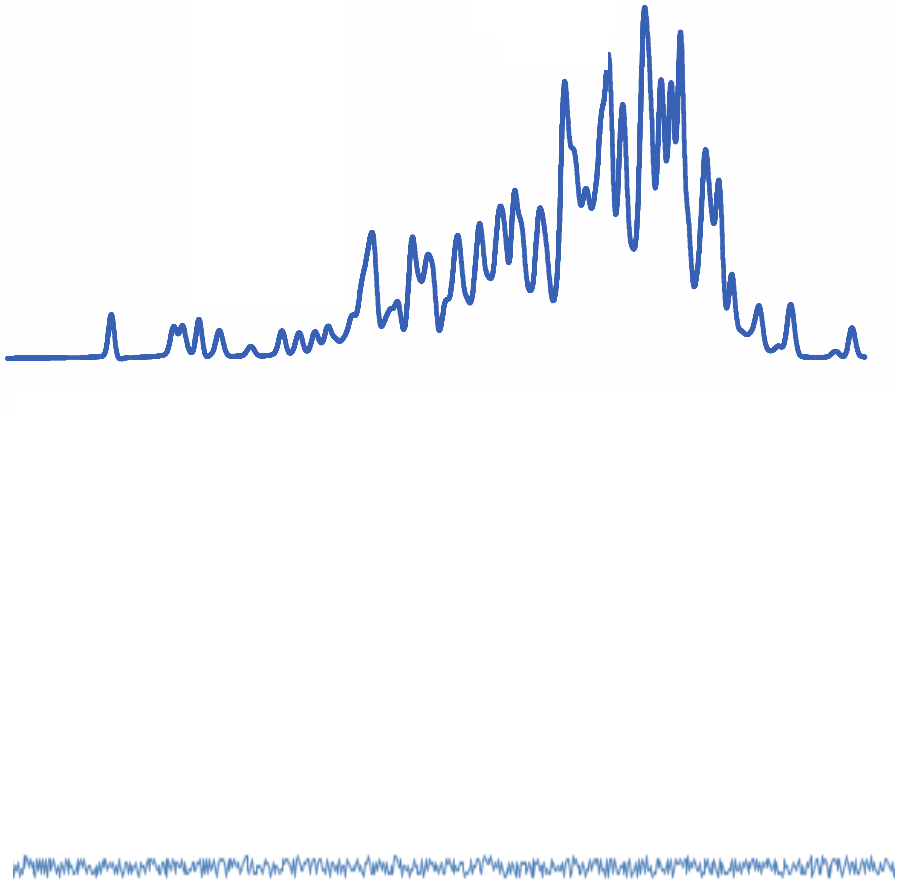
- Tubing kinesis 008T16-080-20 0.8mm ID 20M £35.00
- Needles https://www.needlez.co.uk/product/21g-blunt-needle-1-5inch-38mm

- Note Hilljenberg NMR Pasteur 1000 £245.60
  24p each! normally 80p-£1

- Original idea from Rainer Kümmerle Bruker

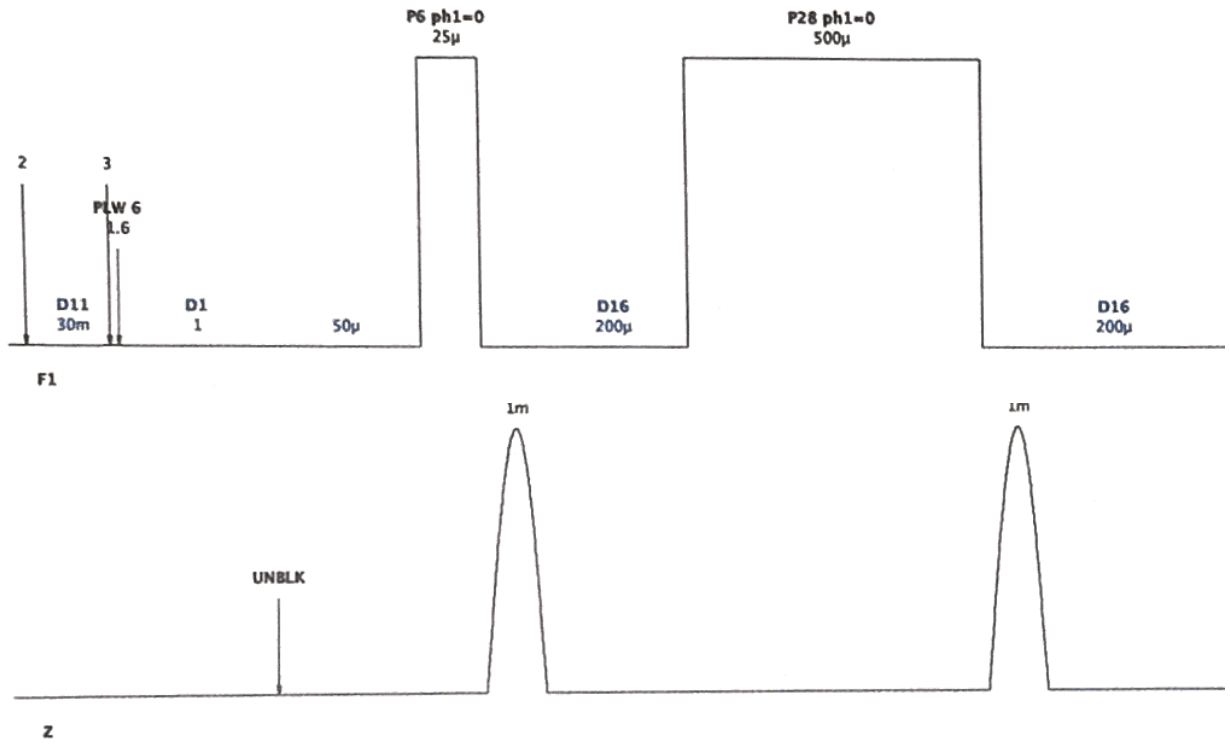
How to delete your spectrum Zuiderweg Trick

- How to completely saturate a spectrum

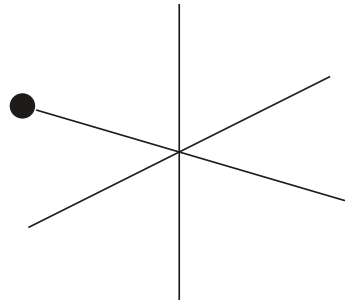


Zuiderweg Trick

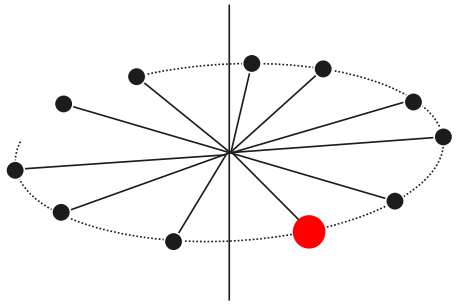
- Hard 90 x
- Z gradient
- Spin lock x
  (really a long
  dephasing pulse
  6dB down 500uS
- X/Z gradient

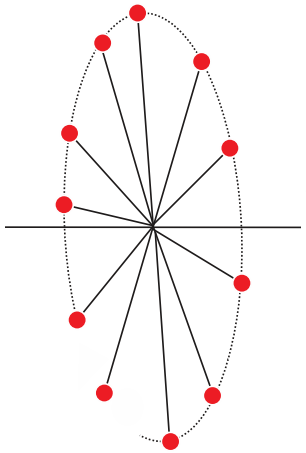


Zuiderweg



Z PFG





Dephasing pulse

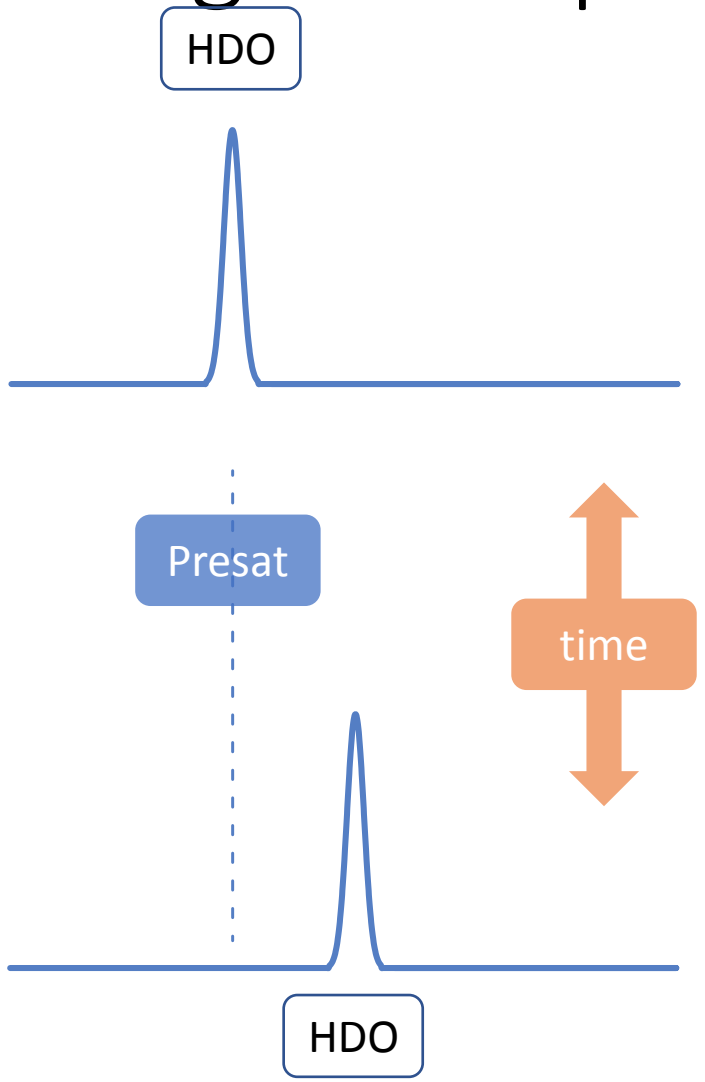
Multiple revolutions field inhomogeneous

Zuiderweg

- Uses reset a spectrum before relaxation experiments when pulsing too fast
- Saturation recovery e.g. solvent PREs
- Ref Yip, G. & Zuiderweg, E., 2005. Improvement of duty-cycle heating compensation in NMR spin relaxation experiments. J. Magn. Reson, 176(2), pp.171–178.

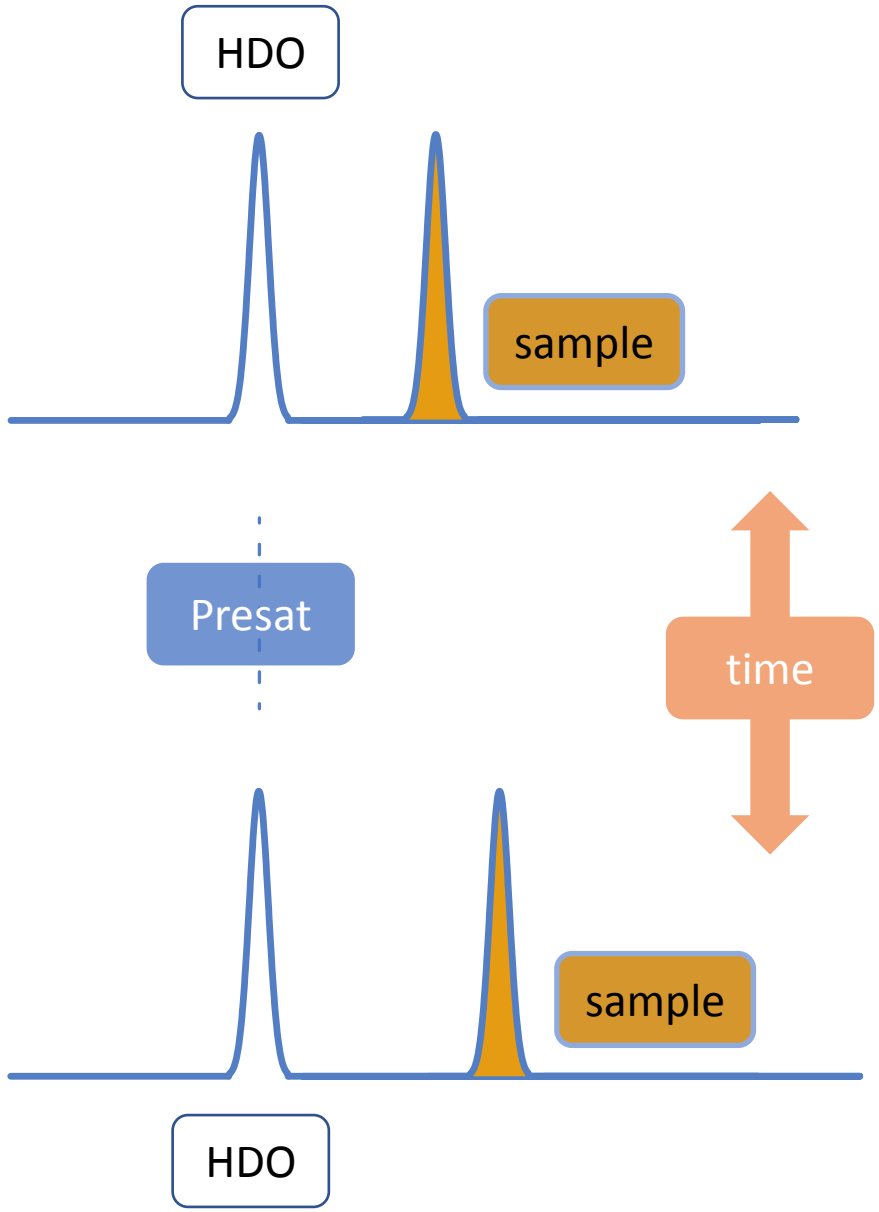
Wacka peak suppressing a moving water peak

- Problem you have a reaction in an organic solvent that produces water
- The water position depends on hydrogen bonding and [H_2O]
- You want to presat/suppress the water but it must be exactly in the middle of the spectrum to work with your sequence



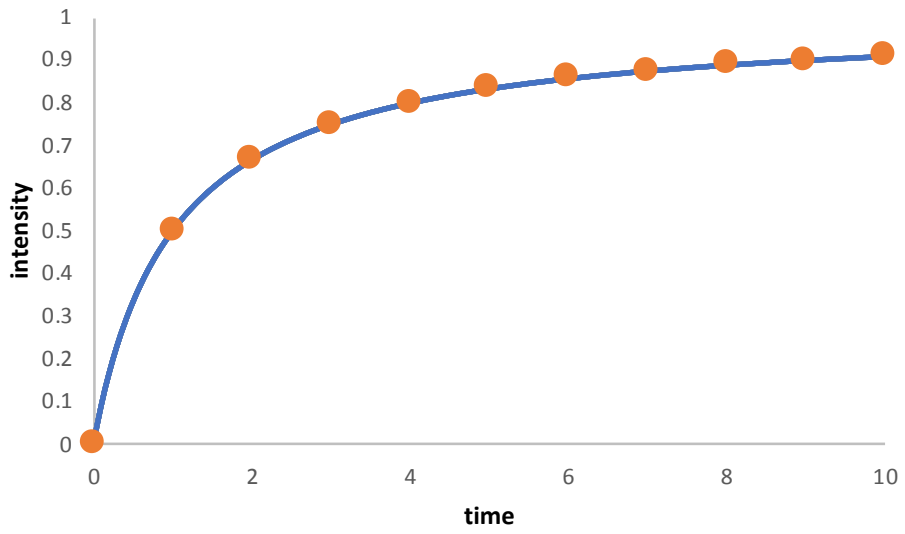
Moving water II

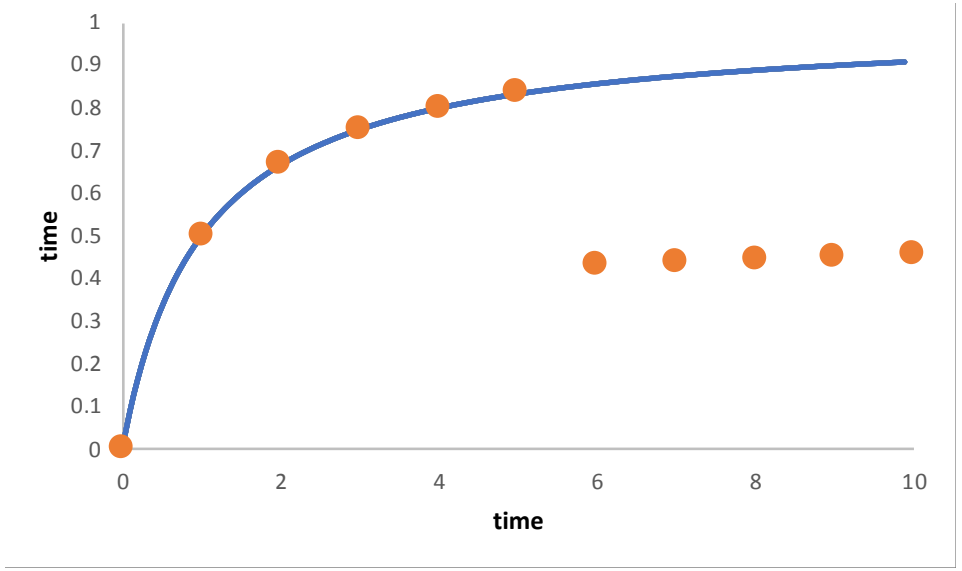
- Solution add some D_2O, put HDO in middle of spectrum, setup water suppression
- Lock to HDO
- ^1HDO peak moves in lock with H^2DO peak so water stays in middle of spectrum exactly
- However your spectrum moves relative to water so add a reference compound



A sudden loss of intensity

- You see this

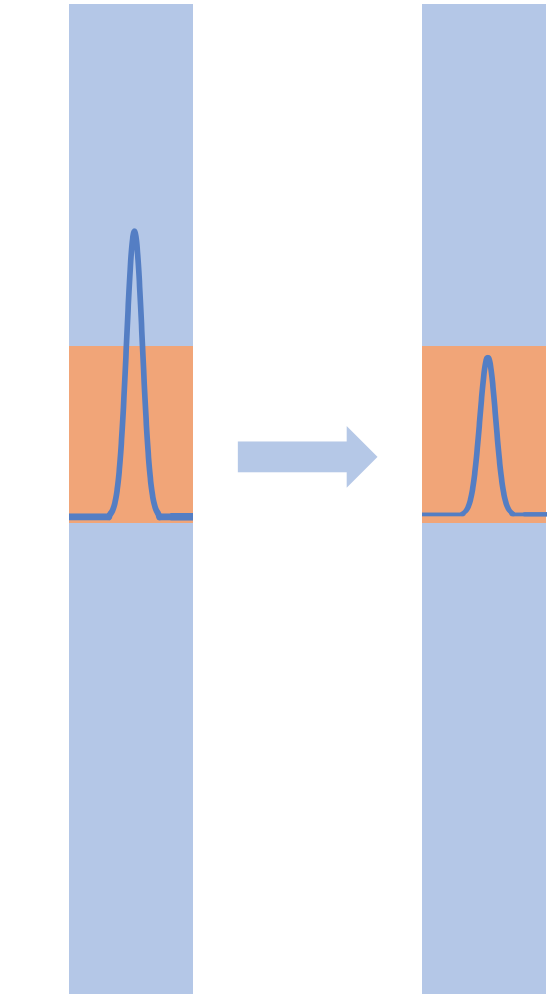




- But expected this

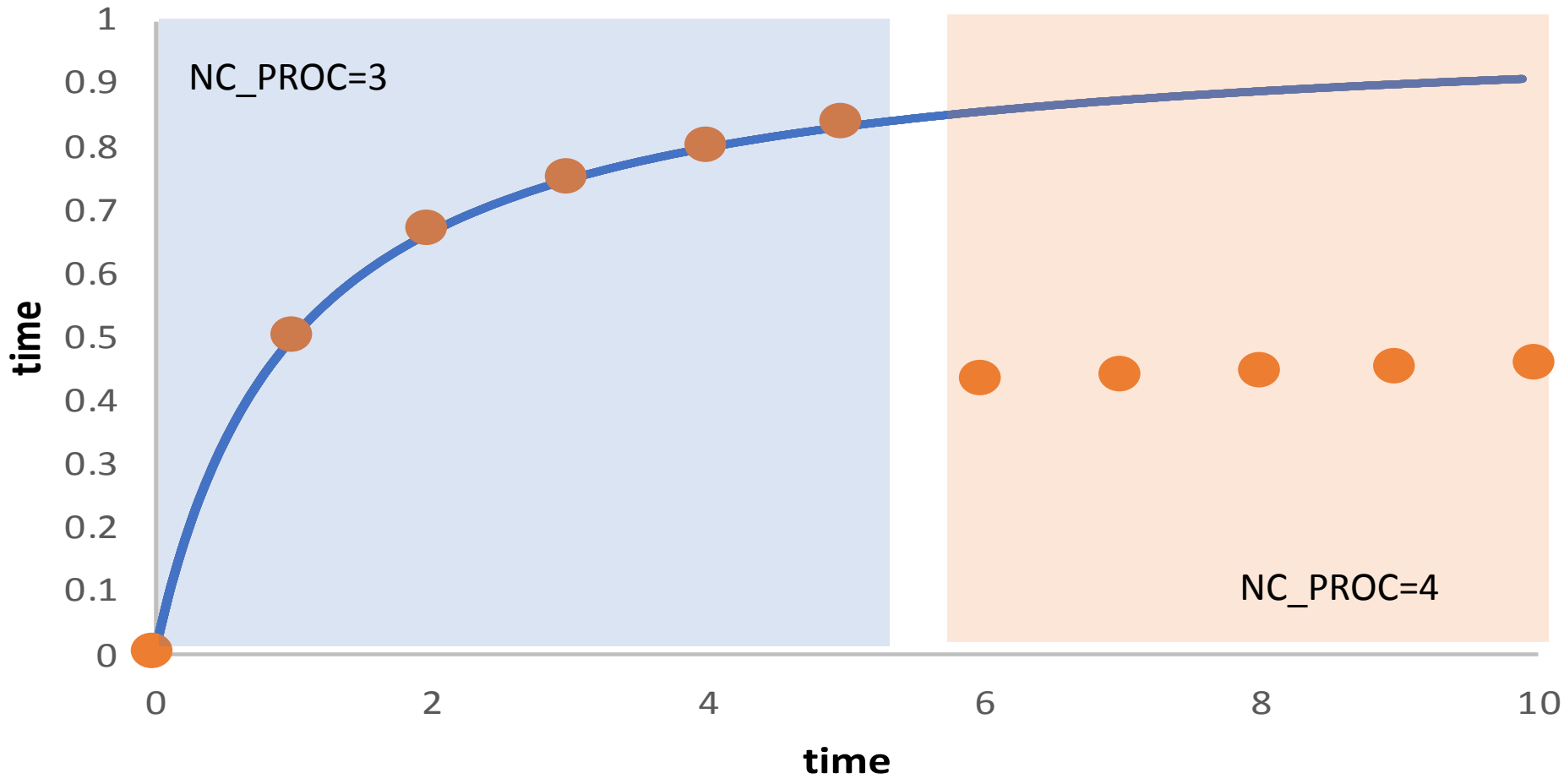
Explanation

- You are using non Bruker software
- Bruker data is saved as integers (dynamic = 2^{32})
- Topspin processes in doubles (dynamic range 2^{2046} - 2^{1023}) and divides data on saving so it doesn't overflow
- On save sets a multiplier NC_PROC
- Your software doesn't take note of the multiplier
  ccpn analysis, old version of nmrglue, azara
- Different on neo / topspin 4?



NC_PROC=N NC_PROC=N-1

Why is my data stunted



From the horses mouth

NC_proc

Processing in TOPSPIN performs calculations in double precision floating point but stores the result in 32-bit integer values. During double to integer conversion, the data are scaled up or down such that the highest intensity of the spectrum lies between 2^{28} and 2^{29}. This means the 32 bit resolution is not entirely used. This allows for the highest intensity to be increased, for example during phase correction, without causing data overflow. NC_proc shows the amount of scaling that was done, for example:

NC_proc = -3 : data were scaled up (multiplied by 2) three times
NC_proc = 4 : the data were scaled down (divided by 2) four times

Supporters