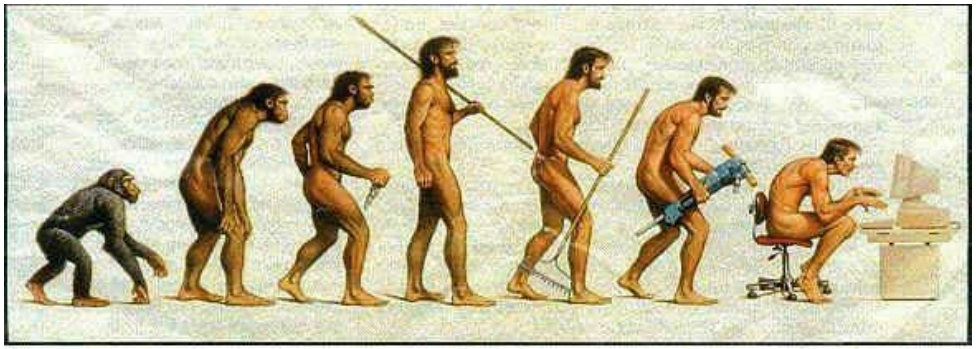


Somewhere, something went terribly wrong

Technology Developments

Recent advances in commercial NMR technology relevant to chemical NMR facilities

Magnets

New developments relating to Helium usage

Helium recycling systems

annual service intervals, power requirements?

Bruker Aeon (400-700 MHz)

1.5(4-500) to 8 (6-700) year He hold times

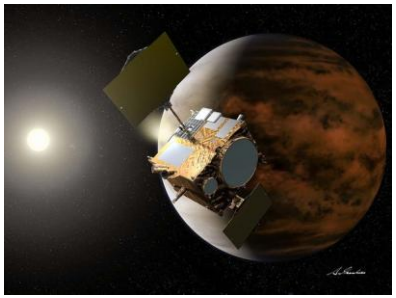
JEOL ZB400 (“zero-boil-off", 400 MHz)

Photo: New Zero Boil Off Magnet for NMR Systems

Also Cryogenic Ltd: “cryogen free” magnet for solid state NMR

Magnet helium efficiency

Magnet	Previous generation magnet, He consumption	Latest magnet, He consumption
400MHz	15 ml/hr	**13 ml/hr**
500MHz	17 ml/hr	**13 ml/hr**
600MHz	28 ml/hr	**16 ml/hr**
700MHz	45 ml/hr	**26 ml/hr**
750MHz	92 ml/hr	**45 ml/hr**
800MHz	250 ml/hr	**140 ml/hr**
850MHz	250 ml/hr	**140 ml/hr**



Probes

Room Temperature probes

- Standard probes for Chemistry support

High-performance broadband configurations

- One Probe: BB+H or F (Agilent)
- Smart Probe: BB.F+H (Bruker)
- Royal probe (JEOL)

Probes

- Cryoprobes-

He cooled:

Standard 5mm:

triple H/C/N (BioLabs), *dual* C/H (ChemLabs), *triple* C observe

Micro

1.7 mm *triple* H/C/N (Bruker)

3 mm *triple* H/C/N (Agilent)- *removable whilst cold*

Multinuclear

BB.F+H or BB+H or F

Quad Nucleus H/C/N/F or P

N_2-cooled (Bruker Prodigy):

BB+H or F

H or F/C/N triple

N_2 cooled probe demo
BB-H[F] @400 & H[F]/C/N @ 600 MHz

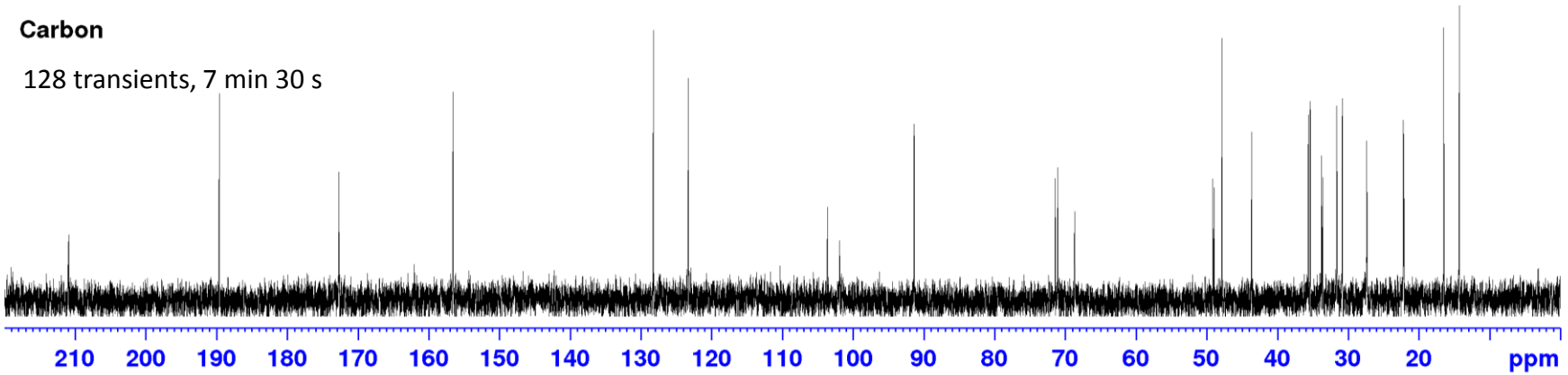
- Primary interest in H/C/F/P studies supporting “biomedical” research projects
  - Identifying & quantifying ^{13}C labelled metabolites
  - Following phosphorylation chemistry & kinetics
  - ^{1}H and ^{19}F ligand binding assays
  - Ligand binding through ^{15}N HSQC protein screening
  - Structural characterisation of synthetic “probes” of biological targets

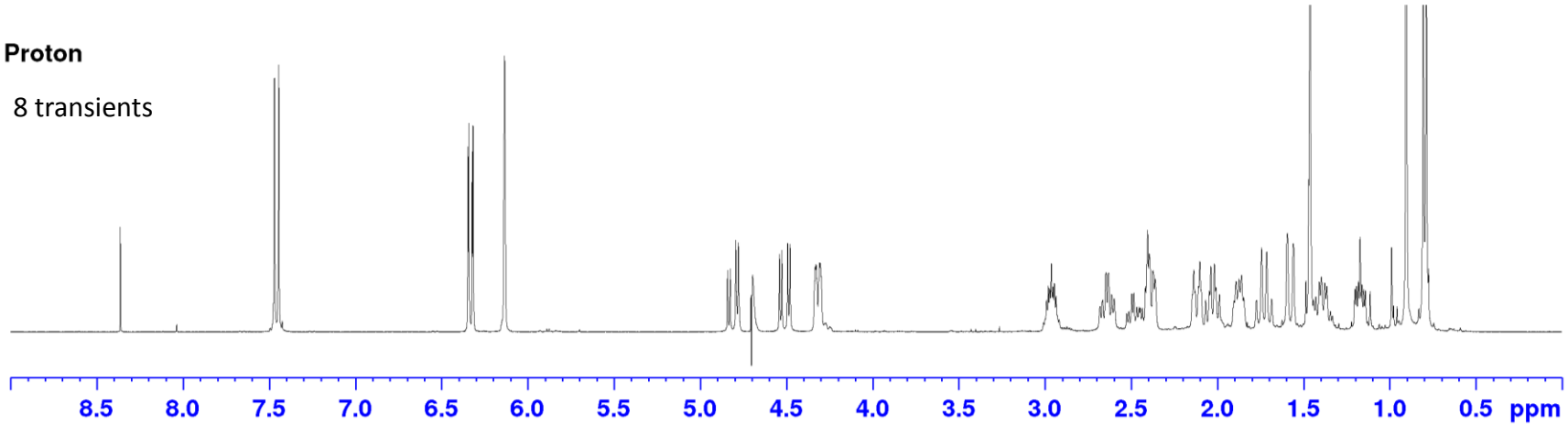
- No capacity to support additional He cryoprobes
  - Dual C/H
  - Triple H/C/N

- *demo data examples….*

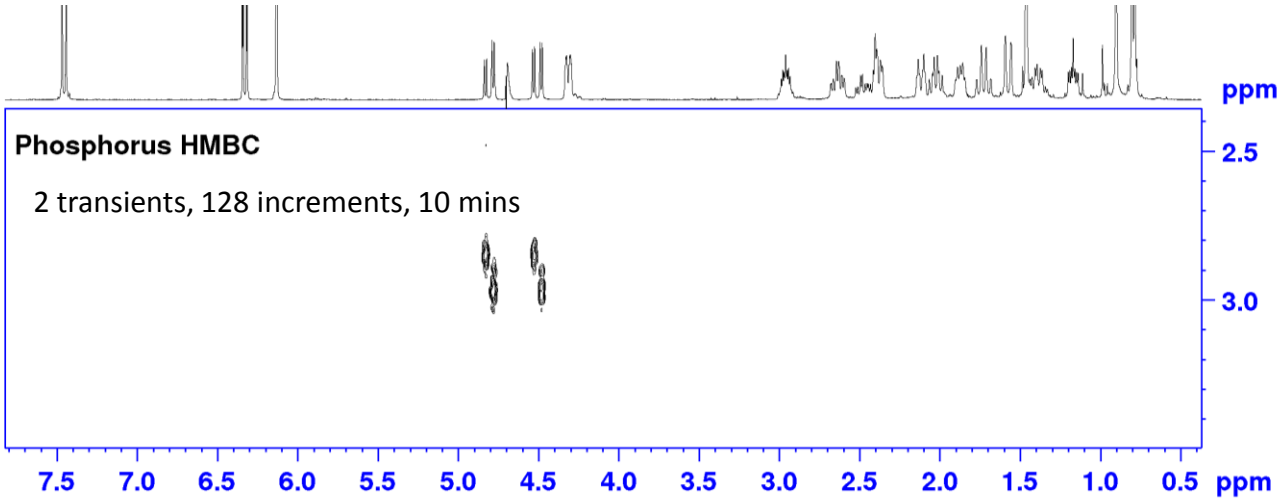
400 MHz BB-H[F] N_2-cooled cryoprobe

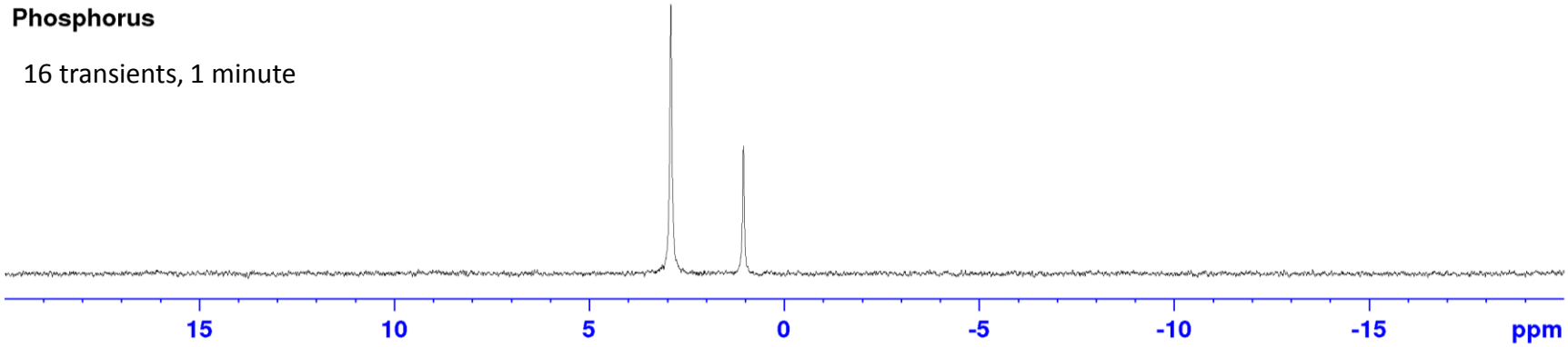
18 mM (6 mg 600 μl), M_r 516, Steroid (dexamethasone phosphate)





400 MHz N_2-cooled cryoprobe

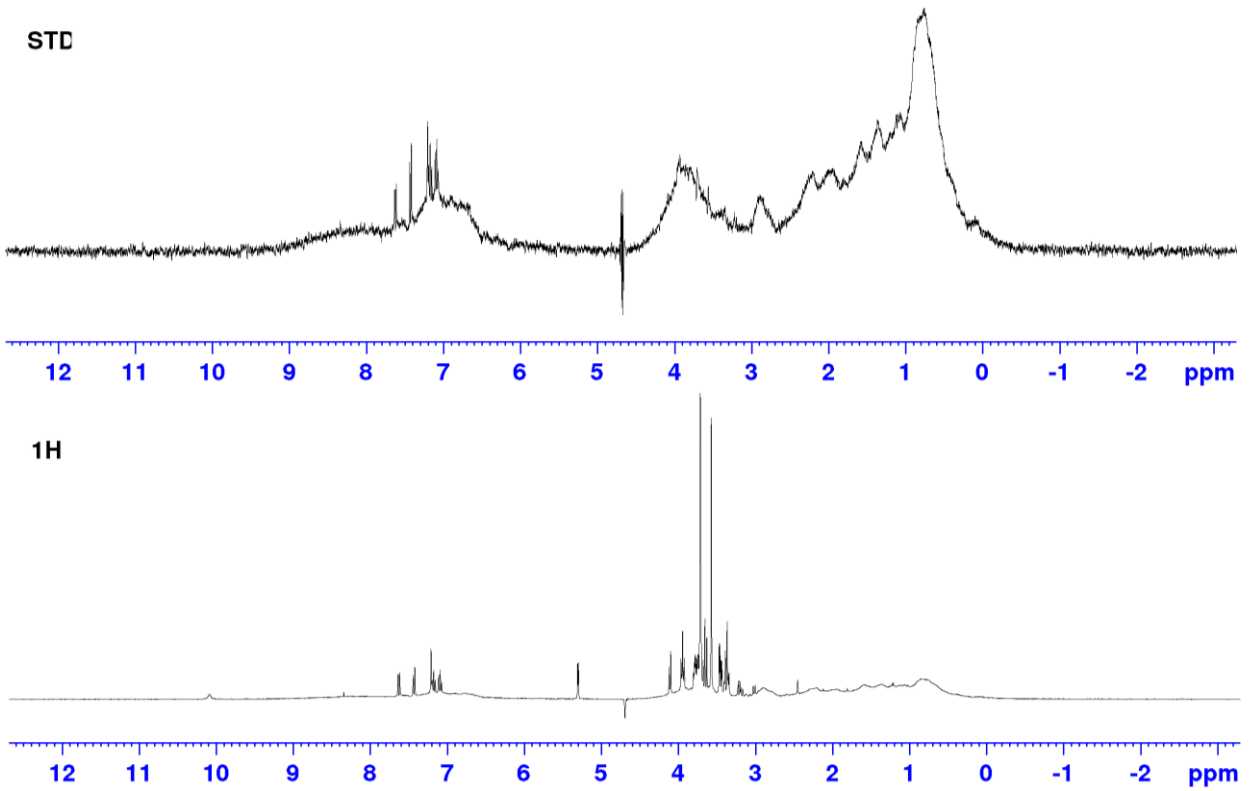




400 MHz N_2-cooled cryoprobe

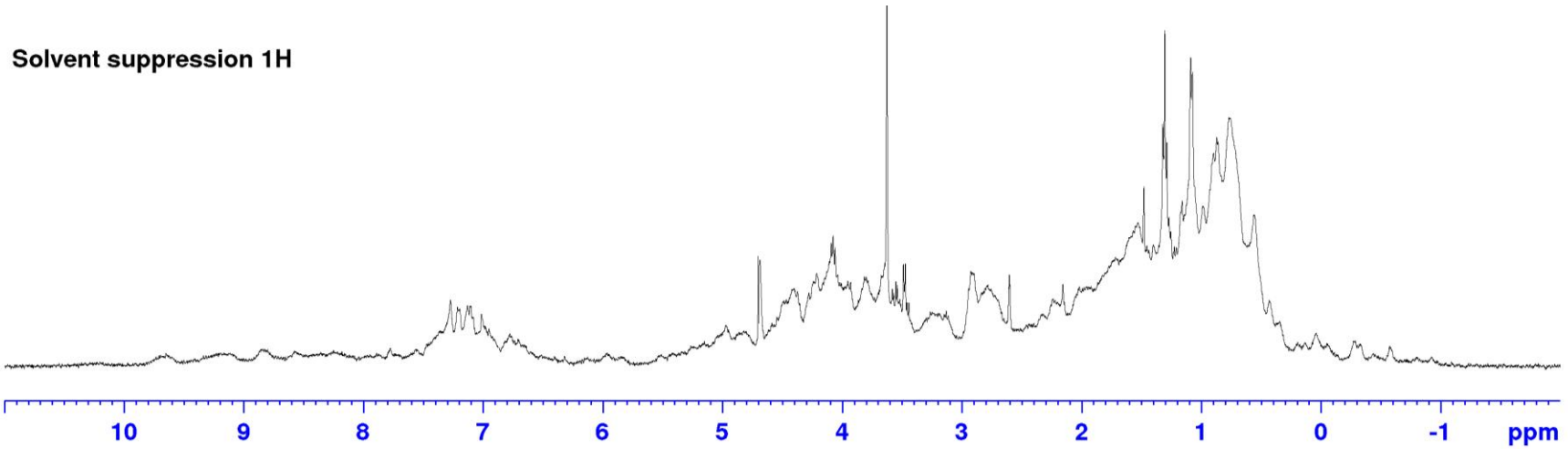
Protein-ligand binding by STD (90% H_2O; excitation sculpting suppression)

32 transients



400 MHz N_2-cooled cryoprobe
^{19}F on ^{1}H coil

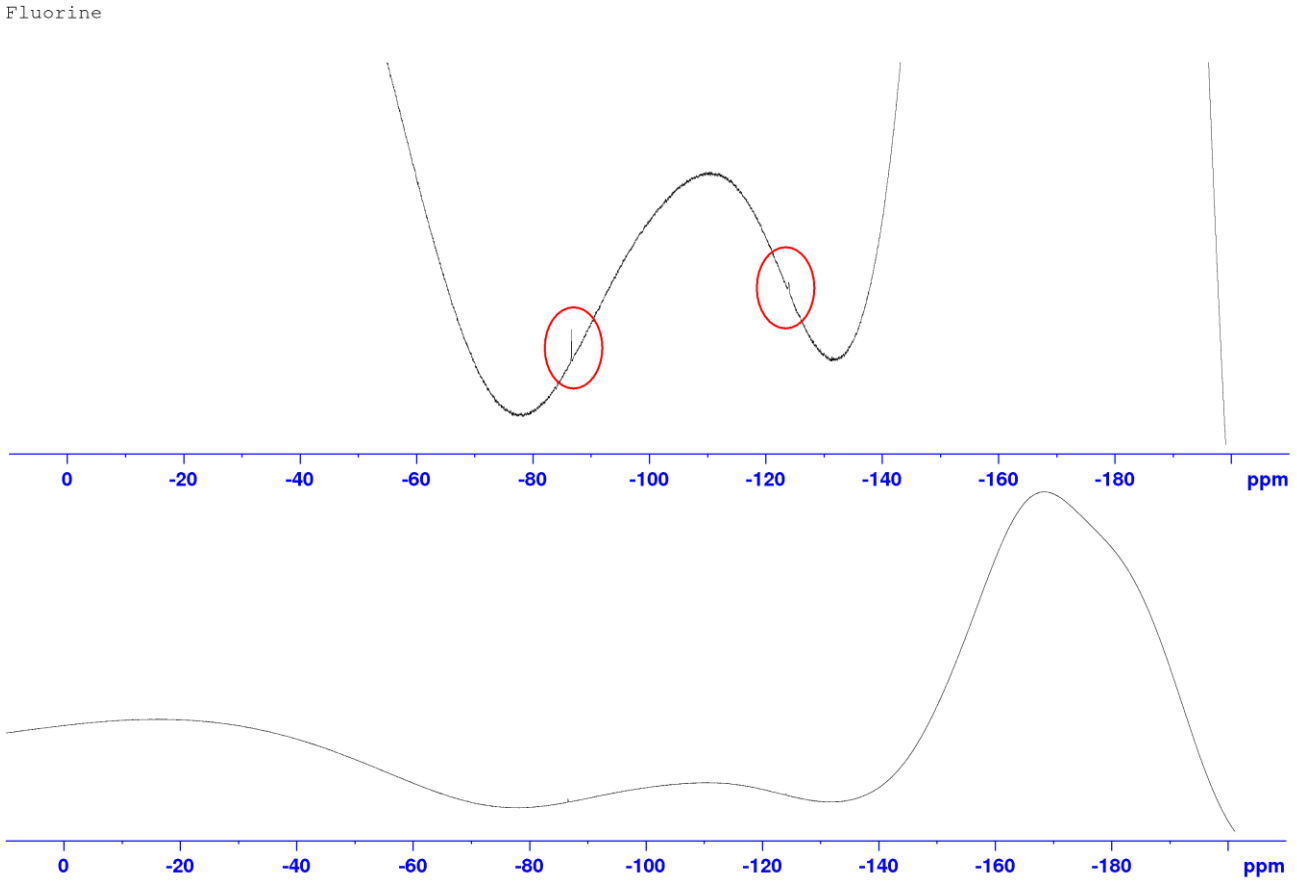
- Protein-ligand binding by ^{19}F NMR (FAXS)
  - 100 uM protein
  - 200 uM fluorinated ligand



400 MHz N_2-cooled cryoprobe

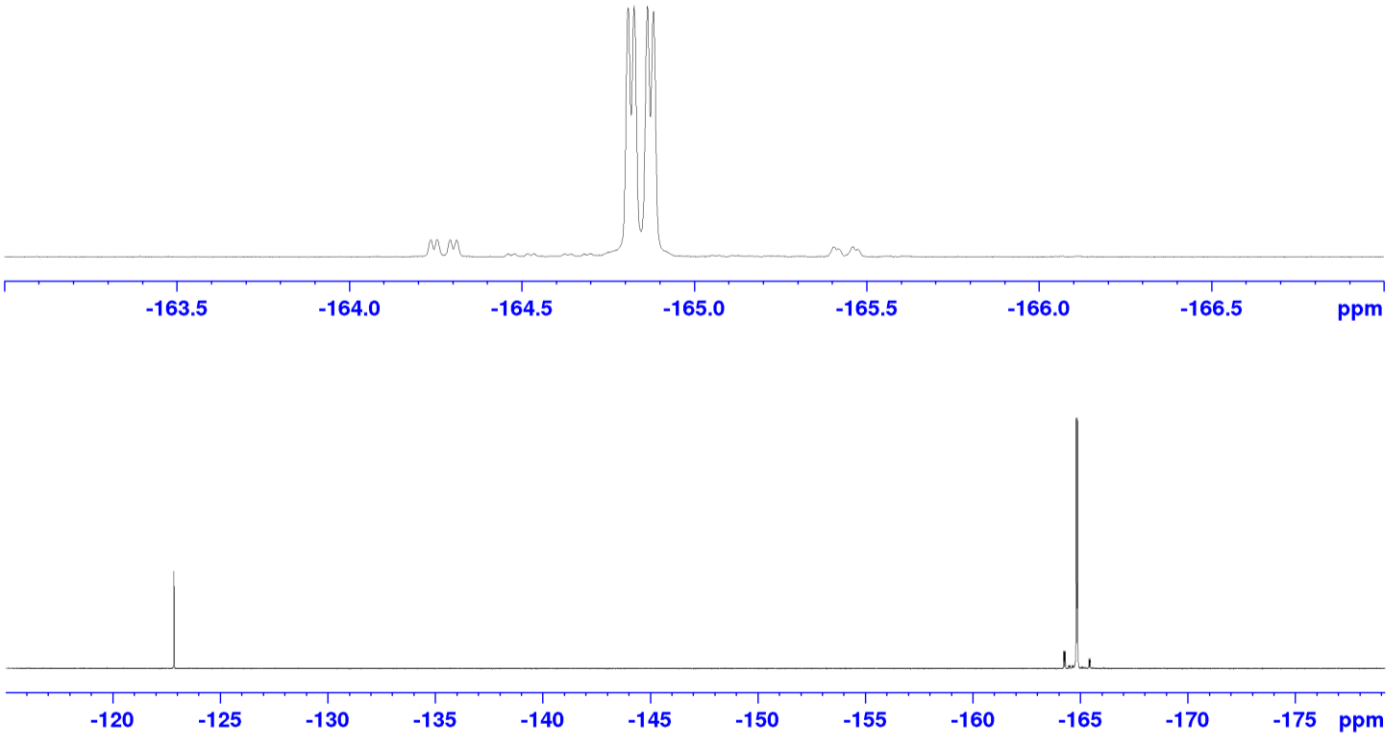
Protein-ligand binding by ^{19}F NMR (FAXS)

100 uM protein, 200 uM fluorinated ligand



600 MHz H[F]/C/N N_2-cooled cryoprobe
^{19}F on ^{1}H coil

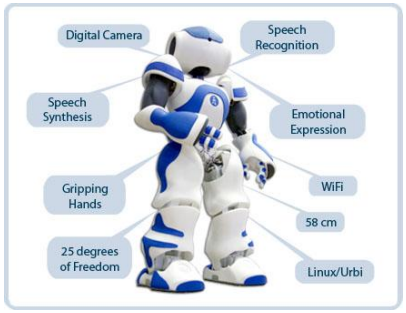
No ^{19}F background on H-observe coil
…but no ^{1}H pulsing/decoupling with ^{19}F



N_2-cooled cryoprobes

- Benefits *vs* He cryoprobes
  - Siting & installation easier
  - Costs: purchase, maintenance and running

- Issues
  - Reduced sensitivity gains
  - Need for regular N_2 fills
  - N_2 dewar hold time does not match magnet N_2 hold time!



Robotics

Sample handling robotics

- Bruker SampleExpress (60/16), Sample Case (24), Sample Jet (480)

- Agilent: AS7510 (12), AS7600 (96),

- Jeol: 24 top-mounted models, 64 carousel