Solid-state nuclear magnetic resonance : from physics to materials

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Solid State Nuclear Magnetic Resonance From Physics to Materials

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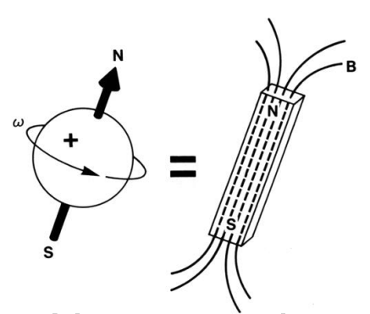
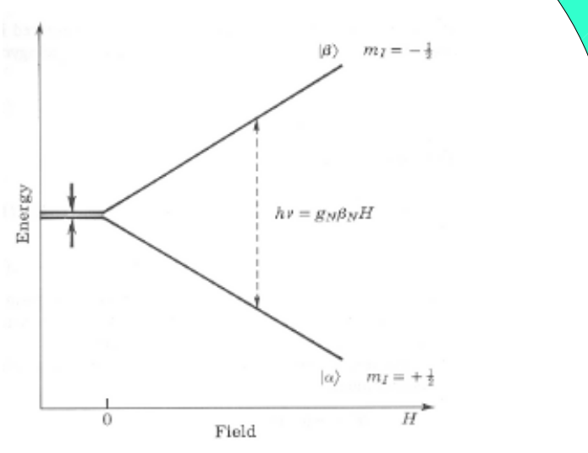
1 Chimie de la Matière Condensée de Paris

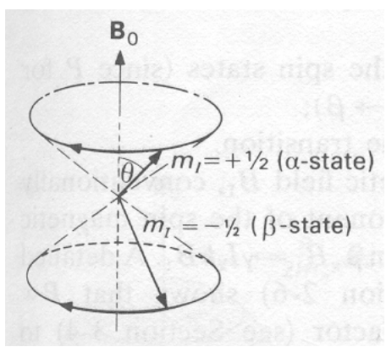
Physics



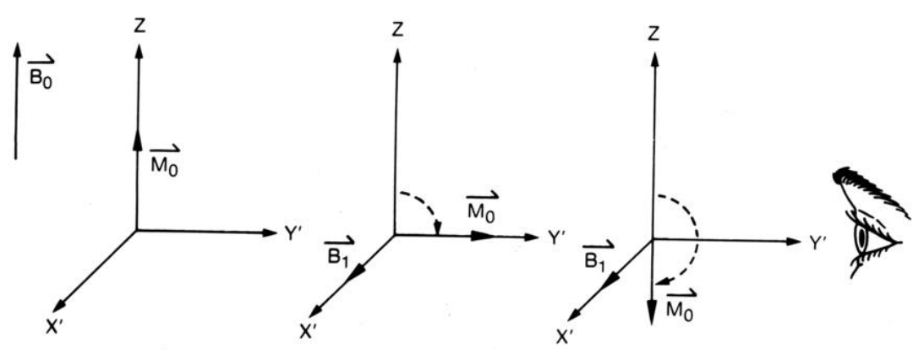
Nuclear spin



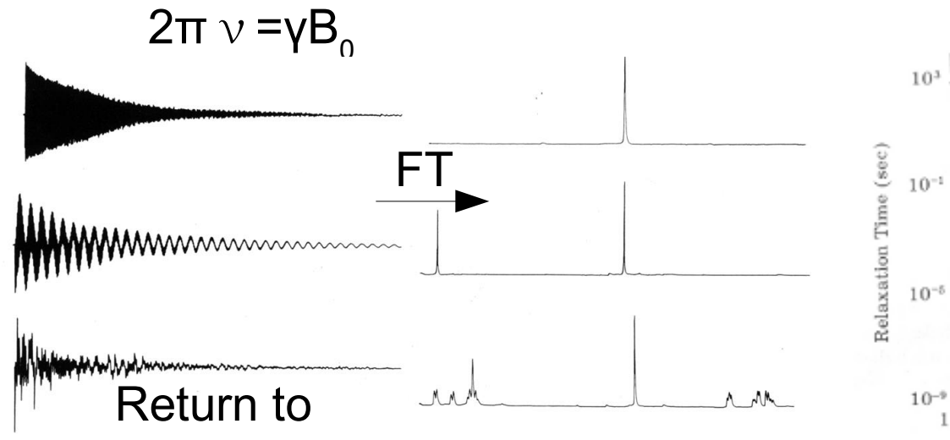
Zeeman effect
NMR is precise but not sensitive



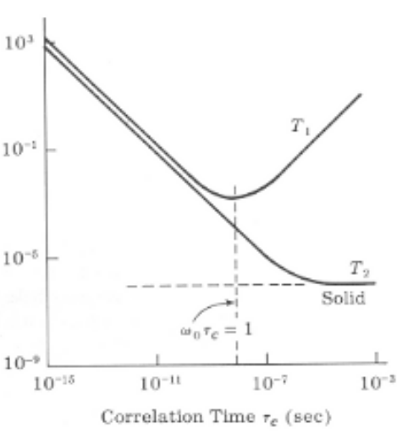
Larmor frequency
$2\pi \nu = \gamma B_0$



Excitation Radio Frequency pulse

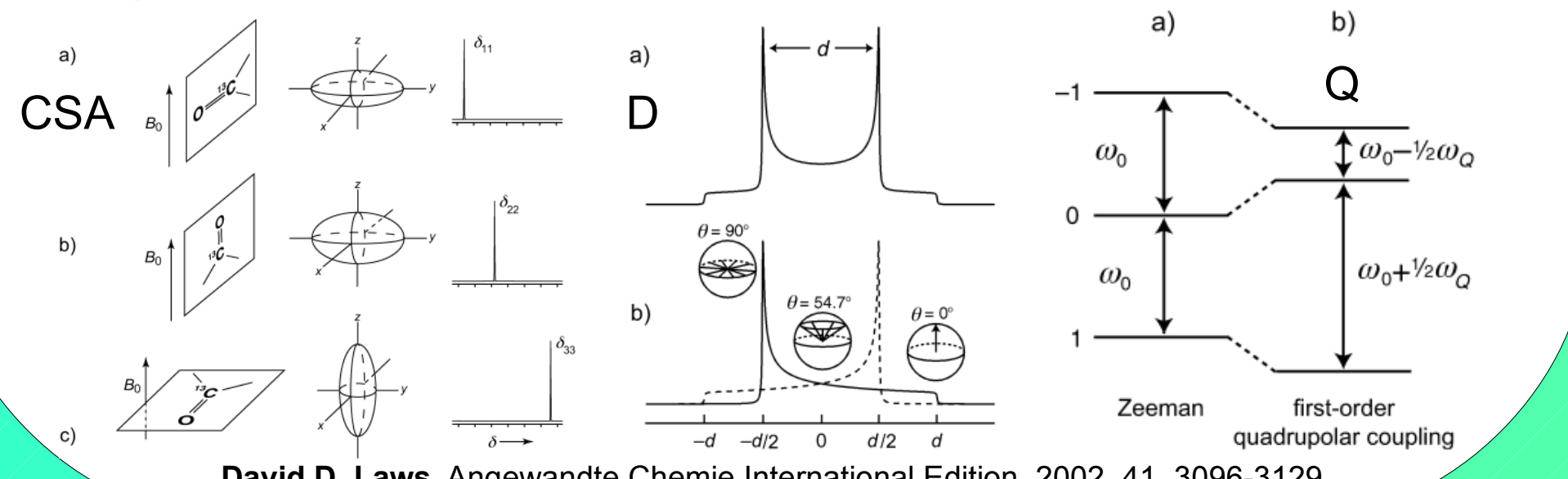


Return to equilibrium



Liquid state :
$T_1 \approx T_2$ →
narrow peaks

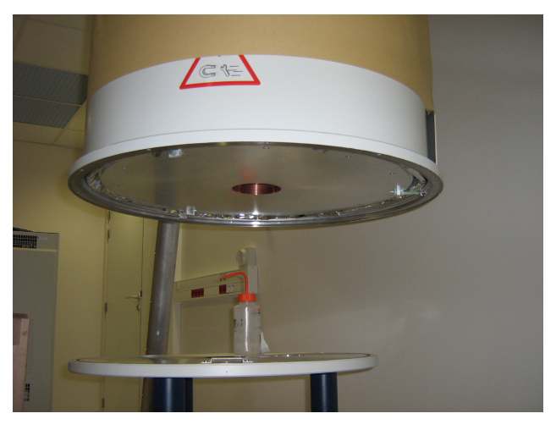
Solid state :
$T_1 > T_2$ →
broad peaks

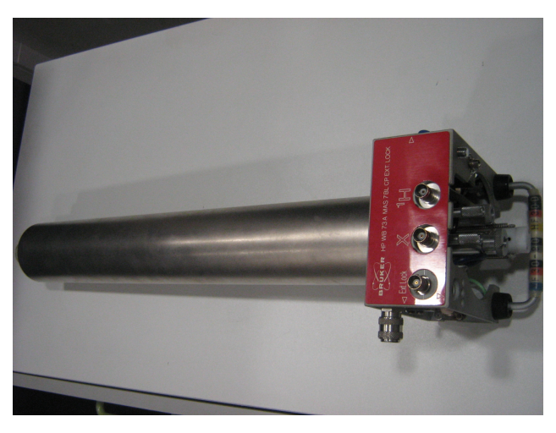


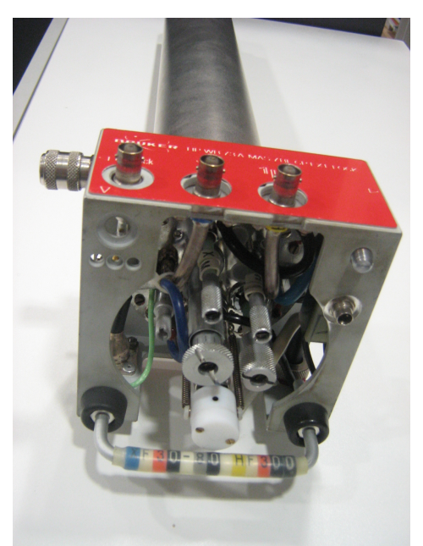
David D. Laws, Angewandte Chemie International Edition, 2002, 41, 3096-3129

Mechanics


2.4 tons
Floor reinforcement



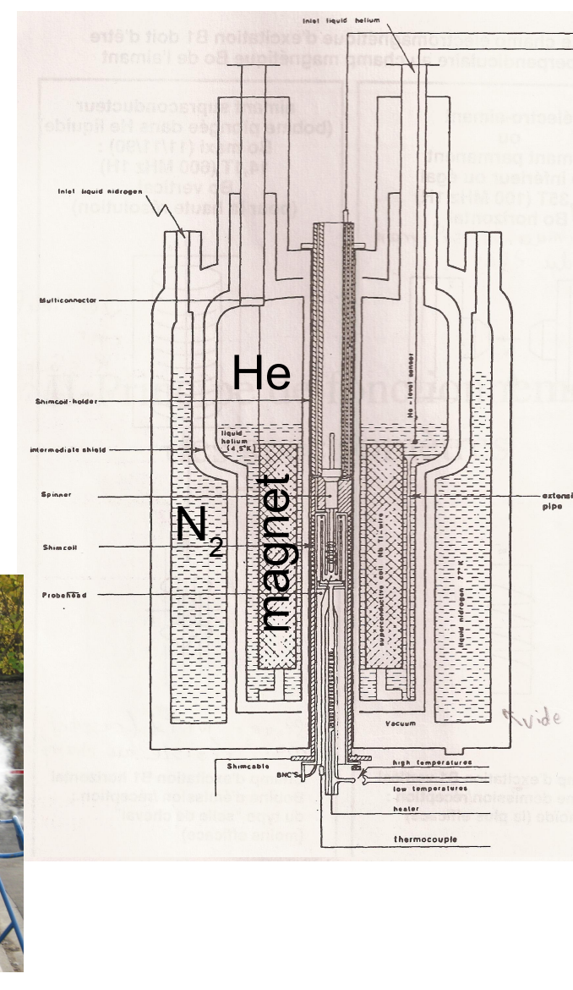


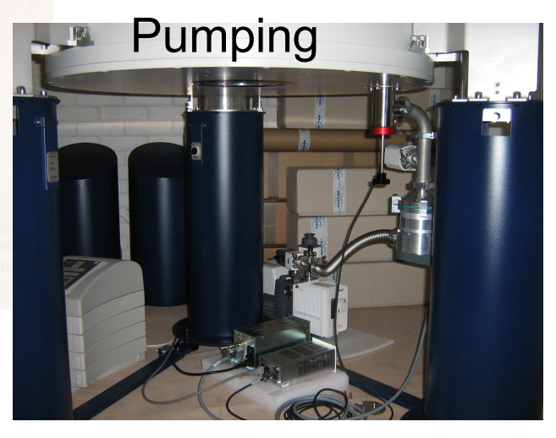

Probe


Spinning rate depending on rotor size

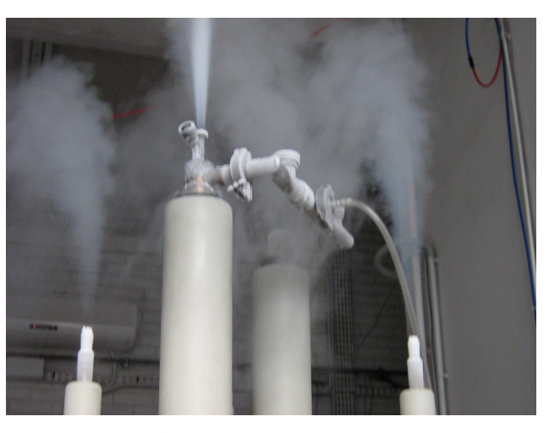
Centrifugation

Cryogenics

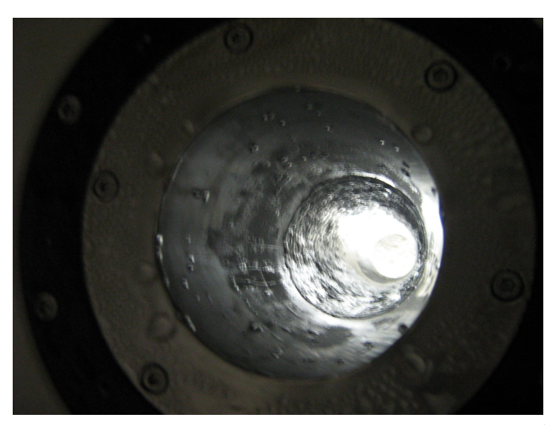



Pumping

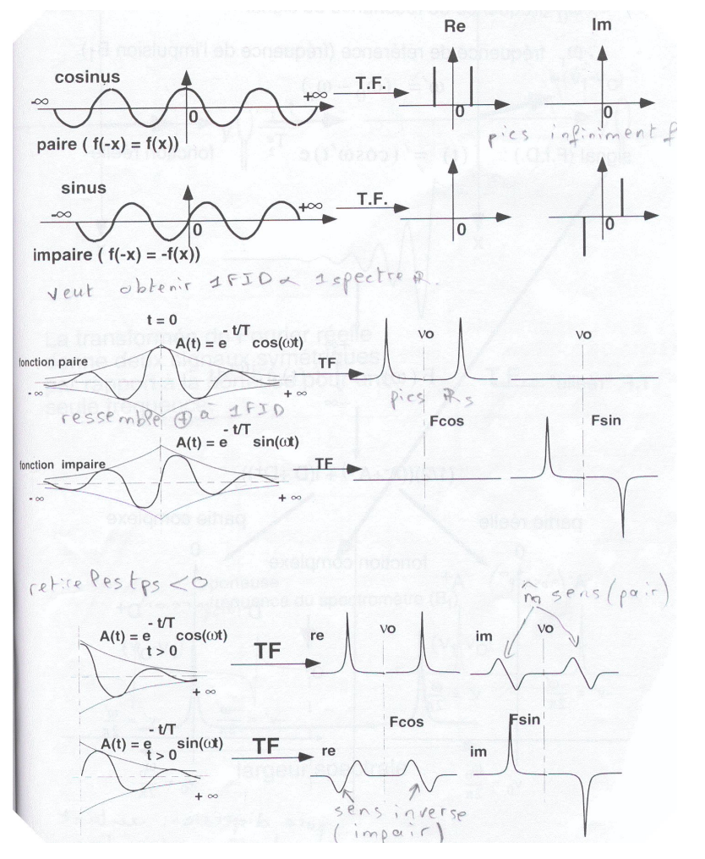
Liquid	Boiling point (K)	Heat of vaporization ($kJ.mol^{-1}$)	Vol. for cool down (L)	Vol. in cryostat (L)	Filling frequency (days)	Cost ($€.L^{-1}$)
Nitrogen	77.4	5.56	1000	286	7	0.50
Helium	4.22	0.08	1000	256	90	7.00


Warming up

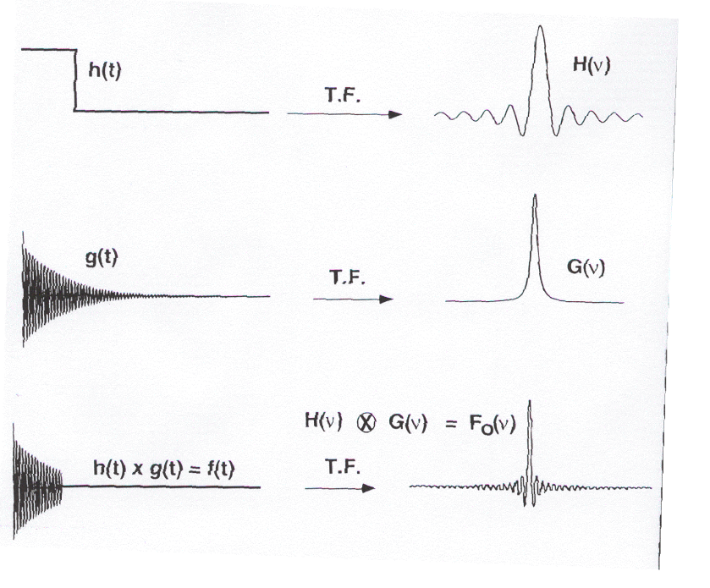

Asphyxia risk


Cryostat frozen

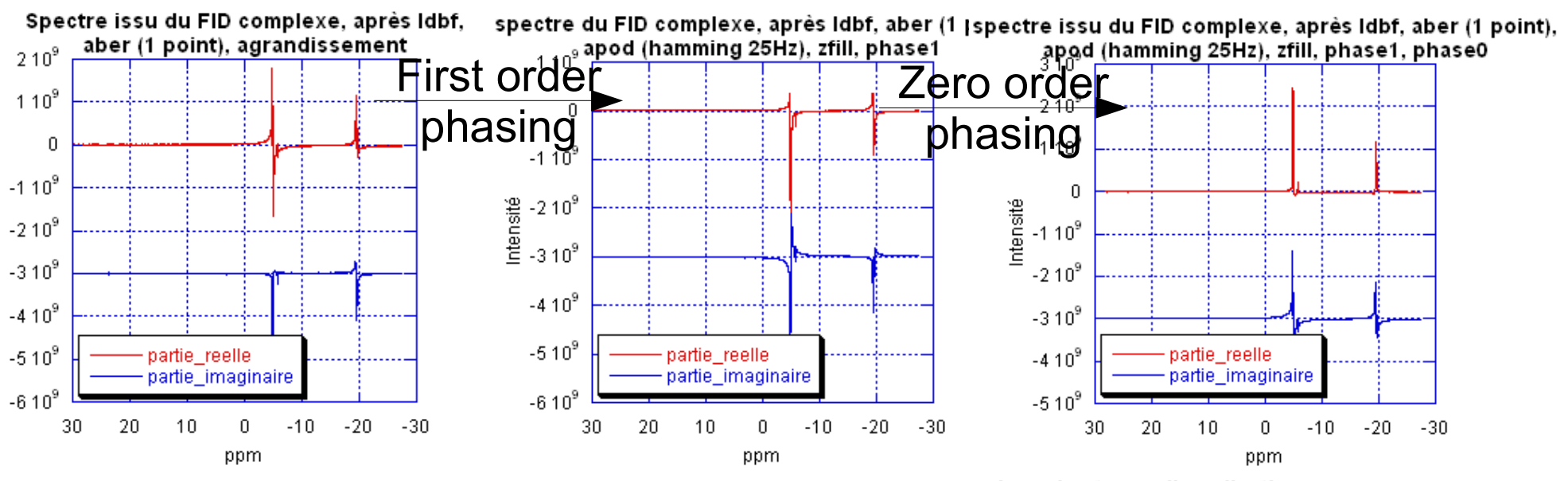
Mathematics

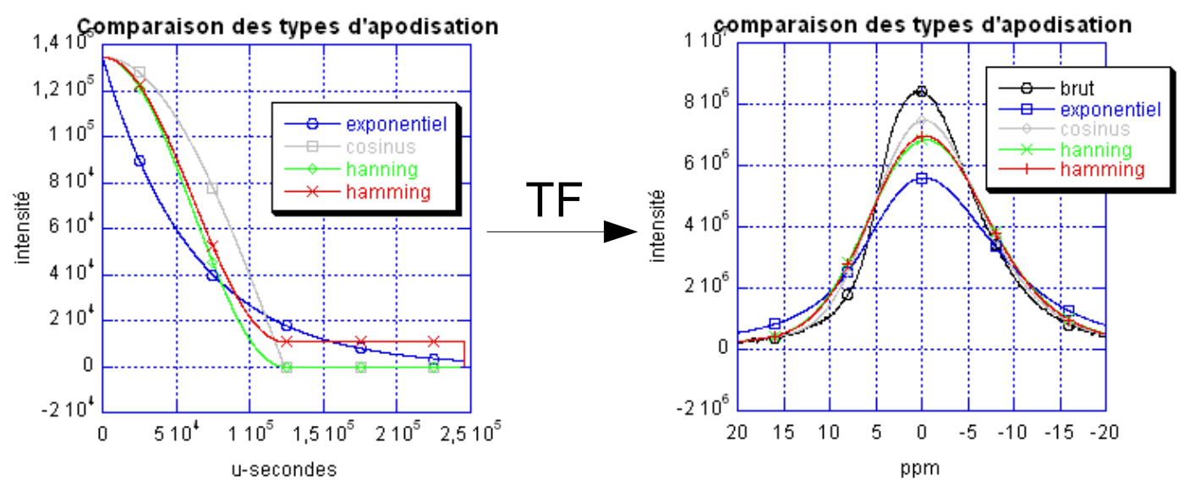


J.-C. BELOEIL, NMR course, 1999, IUT d'Orsay, France

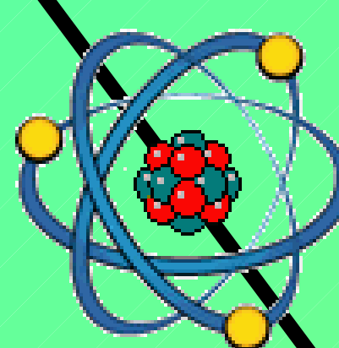


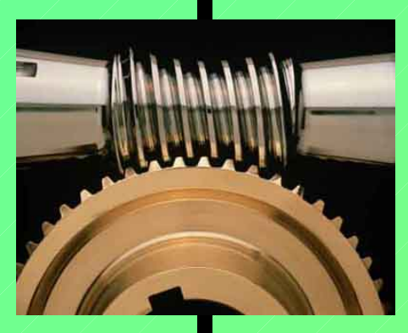
Truncation

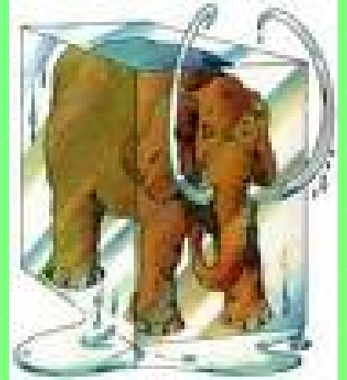

Spectre issu du FID complexe, après ldbf, aber (1 point), agrandissement
spectre du FID complexe, après ldbf, aber (1 point), apod (hamming 25Hz), zfill, phase1
spectre issu du FID complexe, après ldbf, aber (1 point), apod (hamming 25Hz), zfill, phase1, phase0

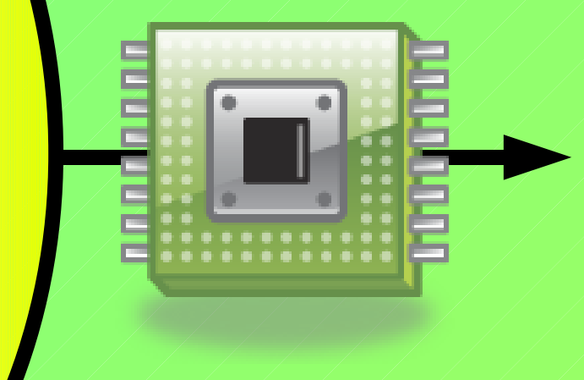

Comparaison des types d'apodisation
comparaison des types d'apodisation

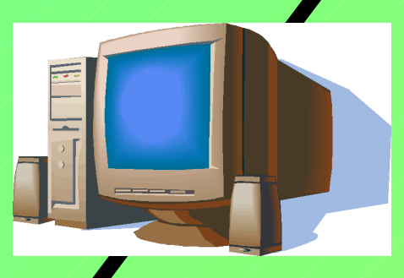
Nuclear Magnetic Resonance

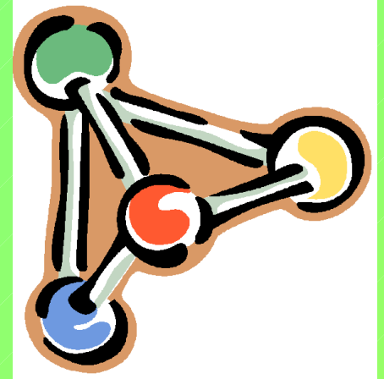












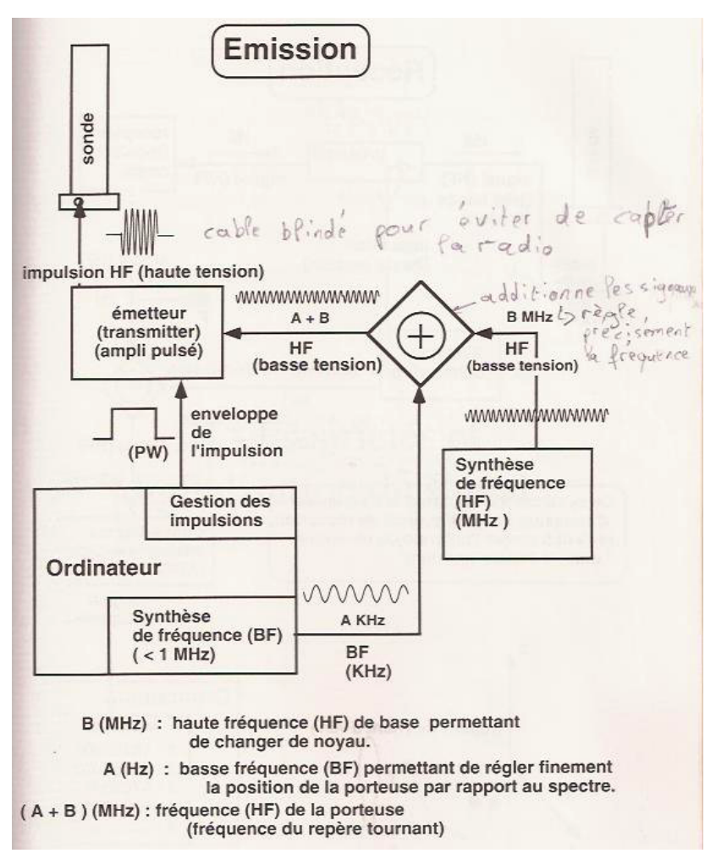
Electronics


Put up to field : 200A !!!
2-3 days needed
Permanent magnet

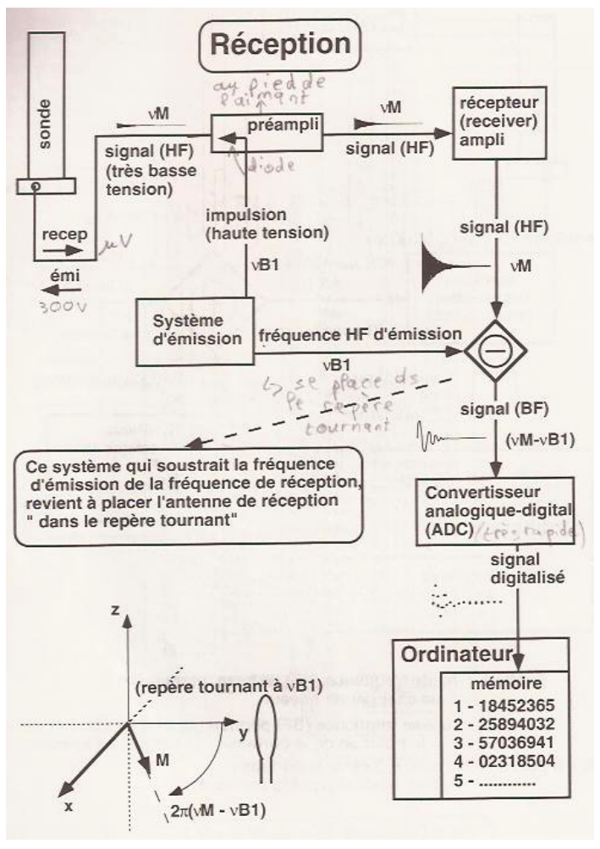

UPS 15 kVA, 1h


Without any maintenance


Radio fréquency control
Temperature unit
Stability control
Pneumatic unit
Power amplifiers (1kW)



B (MHz) : haute fréquence (HF) de base permettant de changer de noyau.
A (Hz) : basse fréquence (BF) permettant de régler finement la position de la porteuse par rapport au spectre.
(A + B) (MHz) : fréquence (HF) de la porteuse (fréquence du repère tournant)



Ce système qui soustrait la fréquence d'émission de la fréquence de réception, revient à placer l'antenne de réception " dans le repère tournant"

J.-C. BELOEIL, NMR course, 1999, IUT d'Orsay, France

Informatics

Spectrometers park administration :
- 3 Linux Redhat workstation 4
- 4 Windows 2000
- 2 Windows XP

Processing software
Backups

Pulse programming

```
;echorot.gul
;theta1-theta2 echo with rotary resonance

;cnst31 :frequence de rotation
;p1 :90 deg on H
;pl1 :H power for hard pulse
;pl10 :H power for spin lock, RF=MAS*2
;l6 :loop counter for echo
;l10 :loop counter for spin lock
```
explications

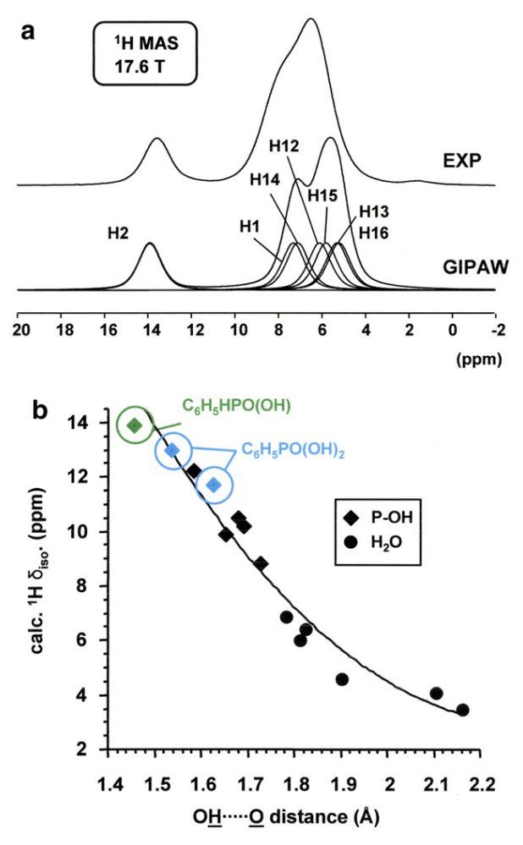
```
#include <Avancesolids.incl>

"p2=p1*2"
"p10=l10*1s/cnst31"
"d10=1s/cnst31-p2/2"
```
definitions

```
1 ze
2 d1
3 10u pl1:f1
  (p1 ph1):f1
  0.3u
  (p10 pl10 ph10):f1 ;RF=MAS*2 rotary resonance echo
  d10
  (p2 ph2):f1
  d10
  (p10 pl10 ph10):f1 ;RF=MAS*2 rotary resonance echo
  go=2 ph31
  wr #0
exit
```
main program

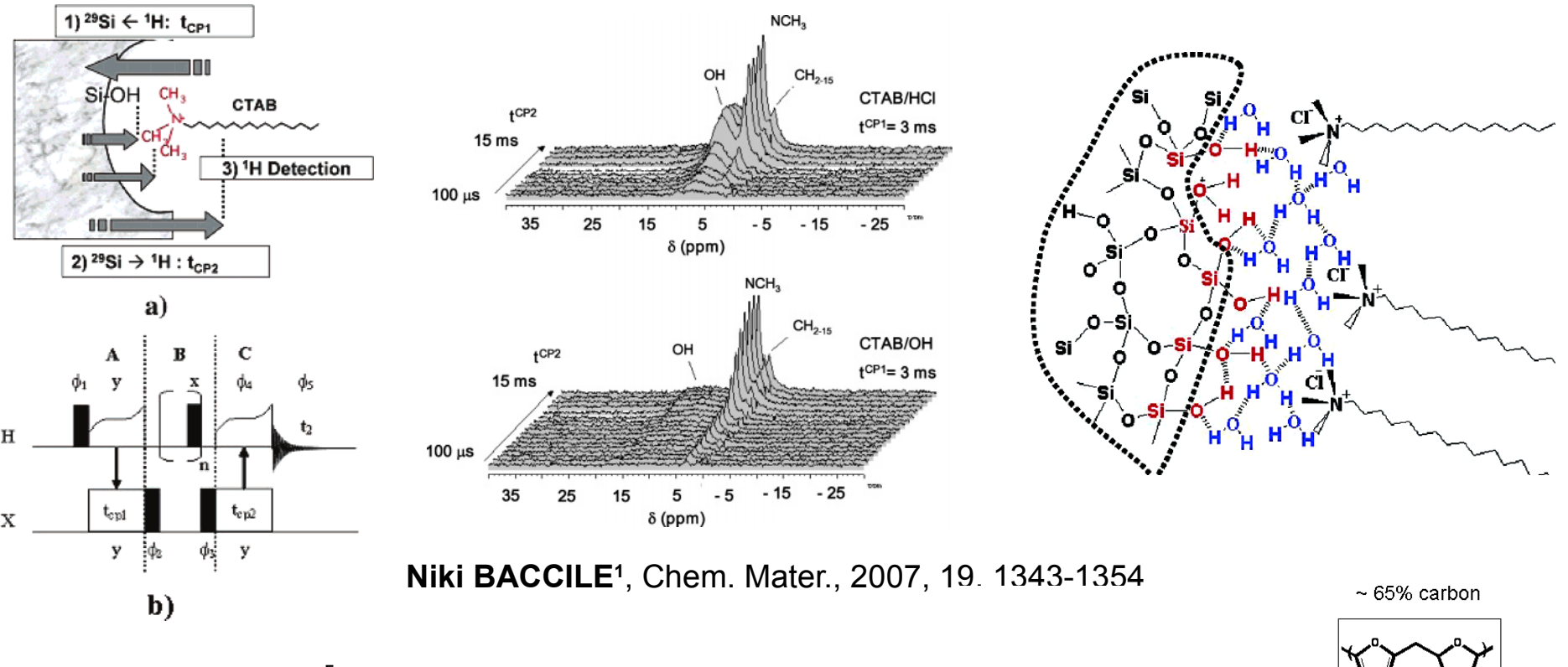
```
ph0=  0
ph1=  0 1 2 3
ph2=  0 0 0 0  1 1 1 1  2 2 2 2  3 3 3 3
ph10= 3 0 1 2
ph31= 0 3 2 1  2 1 0 3
```
phase cycling

Predictive computations

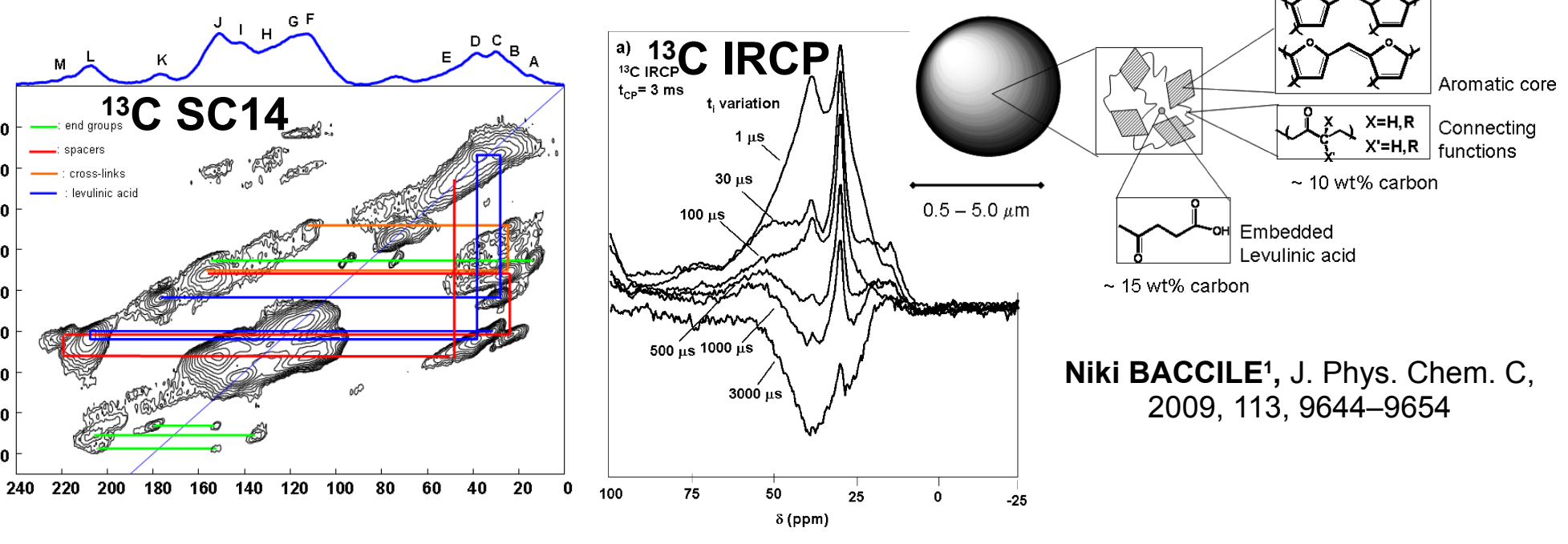


Christel GERVAIS[1]
Journal of Magnetic Resonance, 2007, 187, 131–140

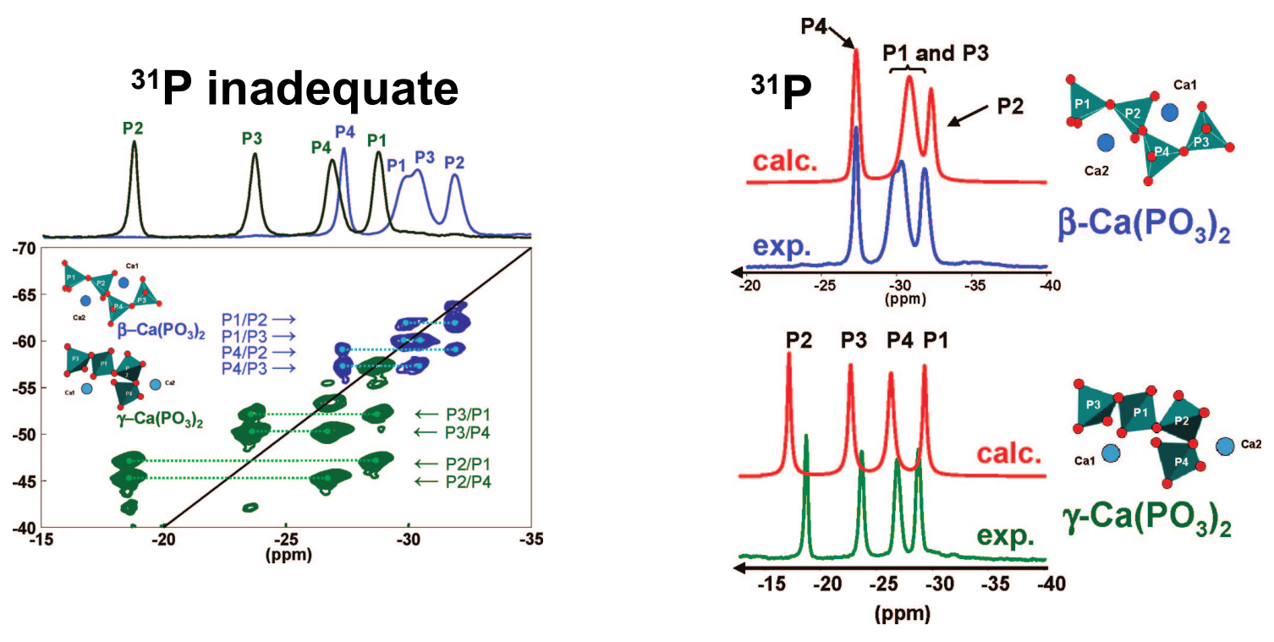
Chemistry



Niki BACCILE[1], Chem. Mater., 2007, 19, 1343-1354

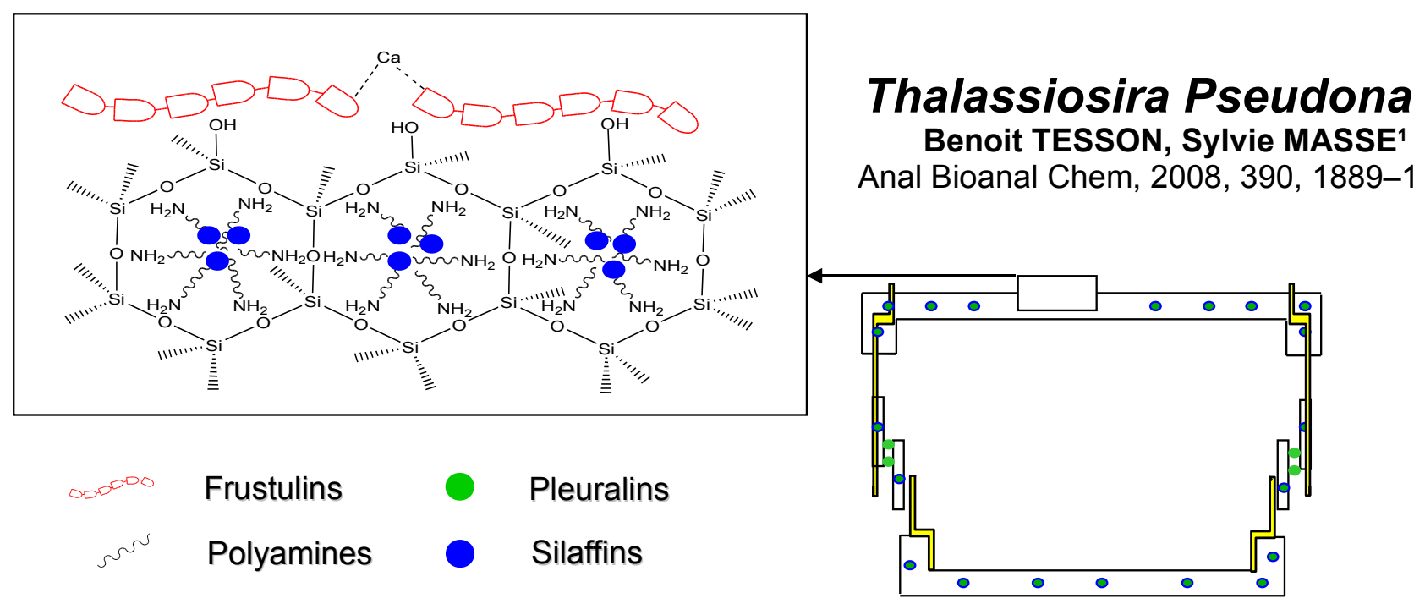


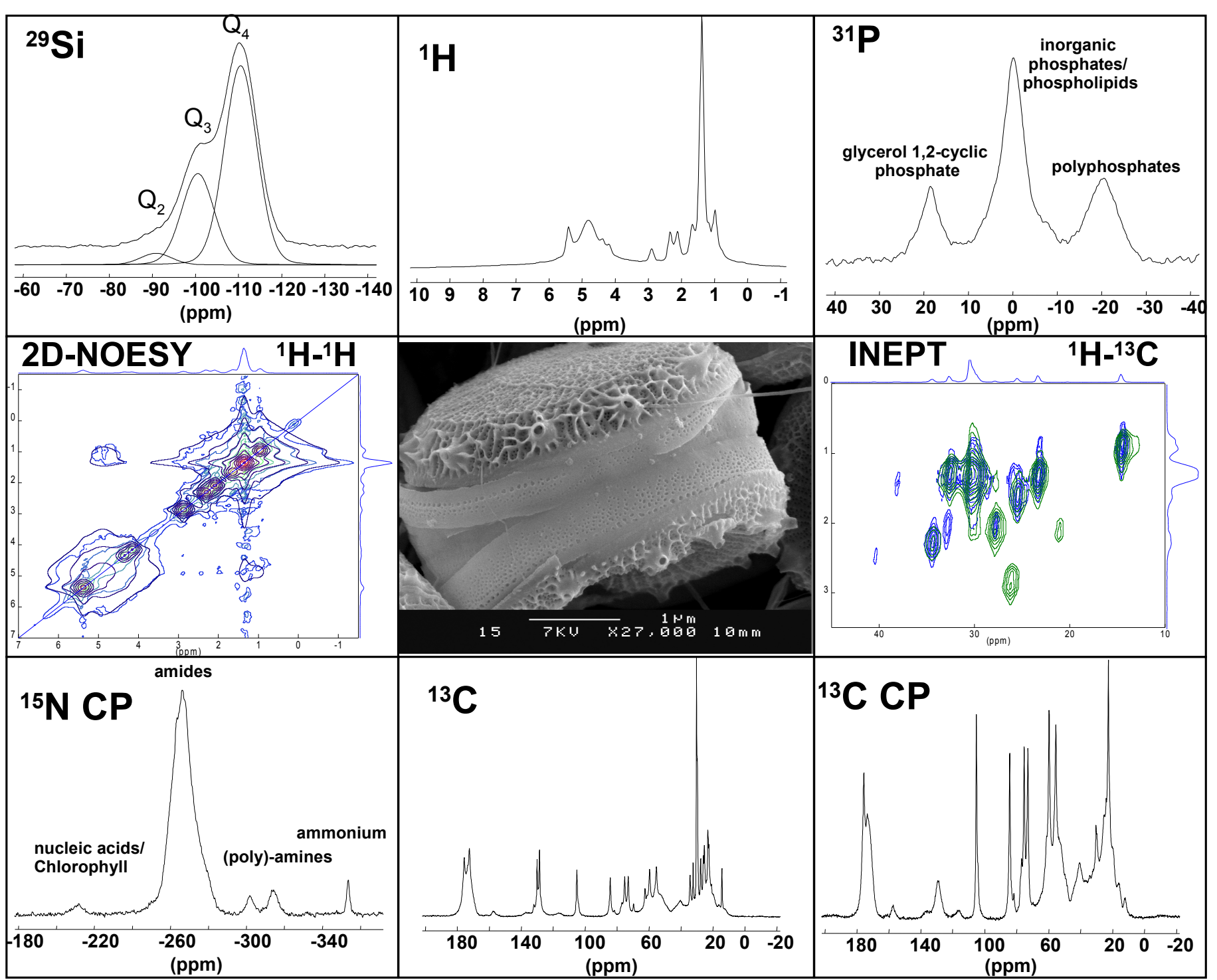
Niki BACCILE[1], J. Phys. Chem. C, 2009, 113, 9644–9654



Frédérique POURPOINT[1]
Chem. Mater., 2007, 19, 6367–6369

Biology


Thalassiosira Pseudonana
Benoit TESSON, Sylvie MASSE[1]
Anal Bioanal Chem, 2008, 390, 1889–1898



Conclusions

Nuclear Magnetic Resonance is a powerful technique that interacts with many fields, for instance physics, mechanics, cryogenics, electronics, mathematics, informatics, and of course chemistry and biology. In liquid state, NMR is sometimes used as a black box, just to check if synthesis works. However, in solid state it is difficult to use it this way. Indeed, physical interactions are not averaged anymore, leading to signal broadening. Some tools can be used to narrow the signals and / or to manipulate interactions either in the laboratory frame or in the rotating frame. Solid state NMR can be used on a wide range of nucleus to quantify species, study their mobility, check proximities between different parts of the sample, either by dipolar coupling or by chemical bonding. One sometimes need to avoid physical artifacts such as dead time in order to get a correct spectrum. In this case, linear prediction and other mathematic tools can be very useful. Finally, one has also to keep in mind that the sample itself can induce difficulties, especially when studying nanoparticles where the side effects become not negligible at all.