High-Field Solid-State NMR: The Tools and Their Application in Materials Research

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Arno Kentgens
Department of Physical Chemistry / solid-state NMR,
Institute for Molecules and Materials,
Radboud University Nijmegen, The Netherlands.

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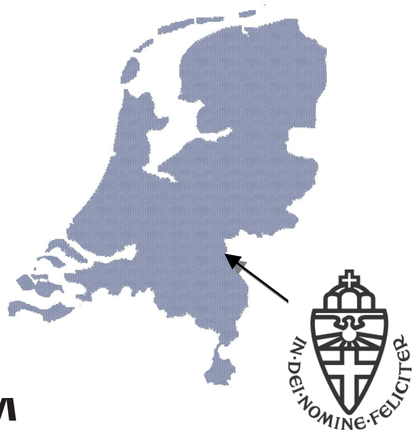
Outline

- **Basic NMR Introduction**
- **Solid-State NMR**
  - NMR tools for spin 1/2
  - Case Study: melaminephosphate flame retardants
  - NMR tools for quadrupolar nuclei
- **NMR tools for the future**
  - NMR above 30 T (1.27 GHz)
  - Microcoil NMR
  - Polarization Enhancement Techniques
  - Mechanical Detection of Magnetic Resonance

Nijmegen Science Faculty & Goudsmit Pavilion for NMR Research



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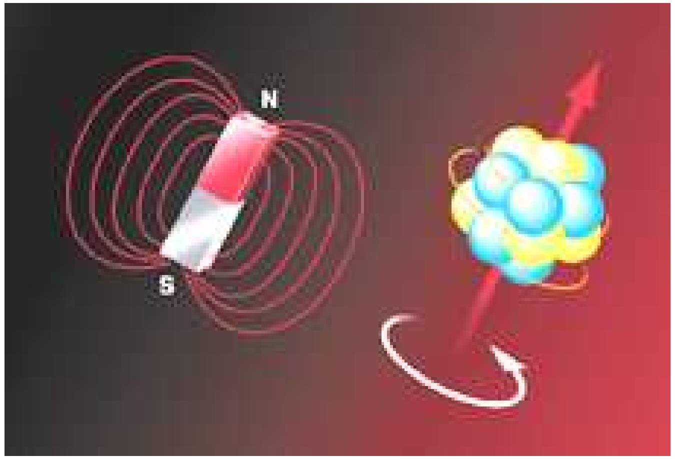
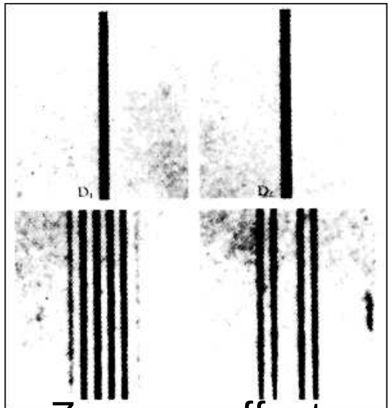
The Concept of Spin

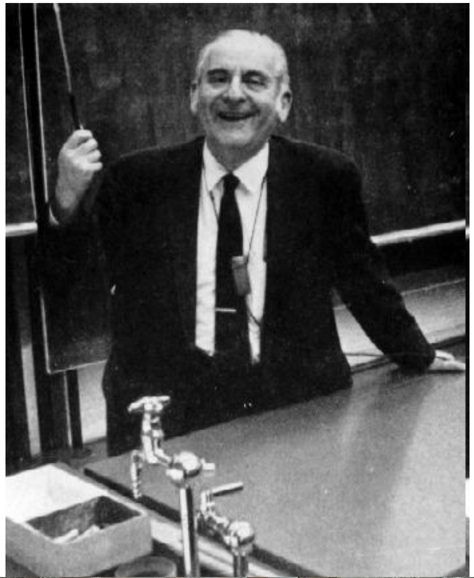


Goudsmit en Uhlenbeck 1925: Electrons have an intrinsic magnetic moment caused by the rotation of the electron



Zeeman effect

Goudsmit - Pauli – Stern 1926: nuclear spin



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Nuclear Spin Hamiltonian

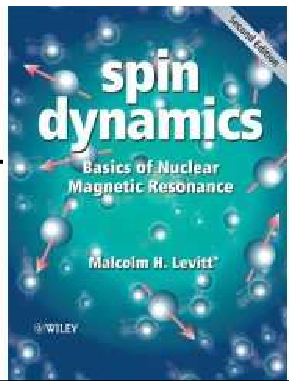
- Quantum state of the entire sample is fully described by a wave function $|\psi_{\text{full}}>$

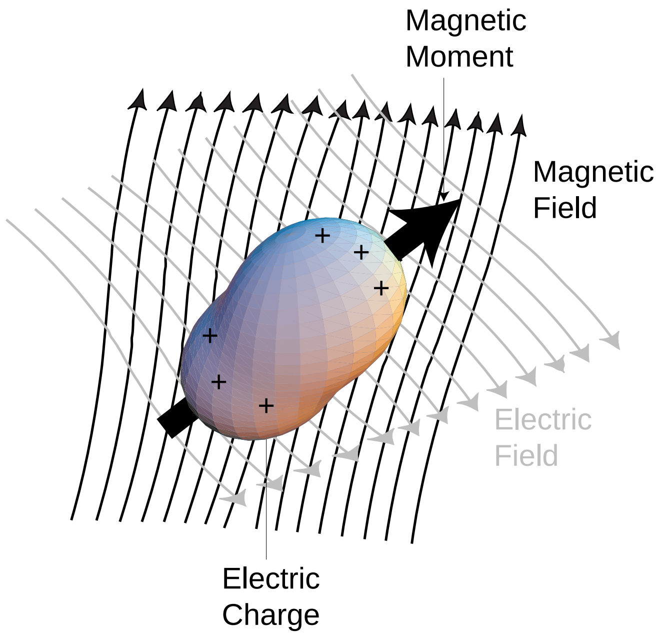
$$\frac{d}{dt}\left|\psi_{full}(t)\right\rangle = -i\hat{H}_{full}\left|\psi_{full}(t)\right\rangle$$

- Effects of rapidly moving electrons is blurred out, their “average” effect is contained in the spin Hamiltonian:

$$\frac{d}{dt}\left|\psi_{spin}(t)\right\rangle = -i\hat{H}_{spin}\left|\psi_{spin}(t)\right\rangle$$

Study Malcolm H. Levitt
Spin Dynamics, Wiley, 2001





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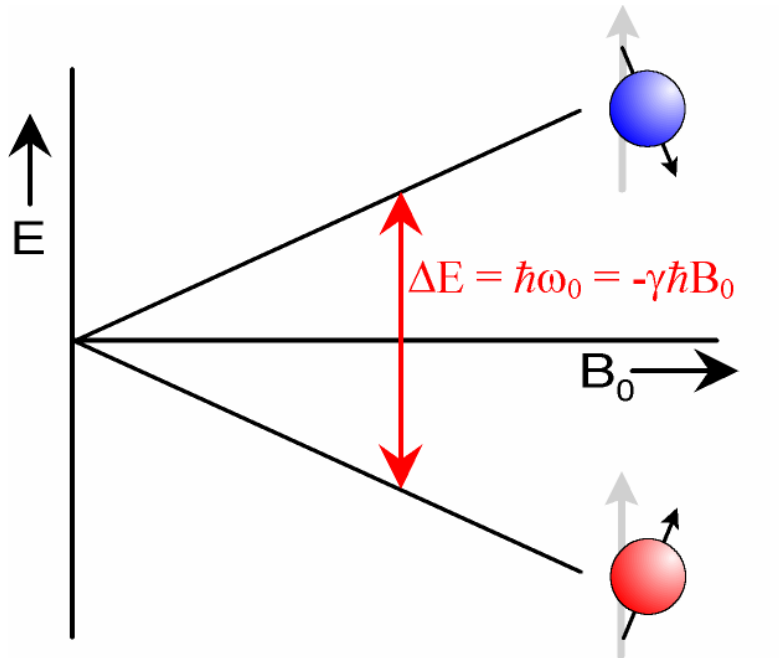
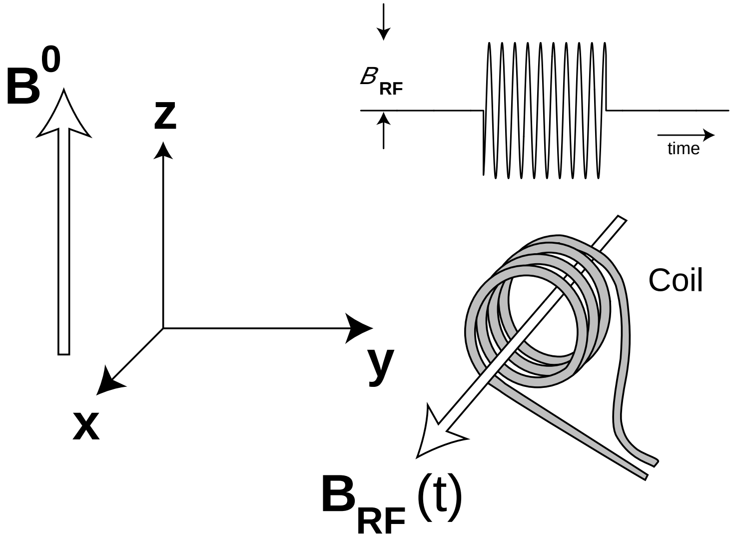
Nuclear Zeeman Interaction

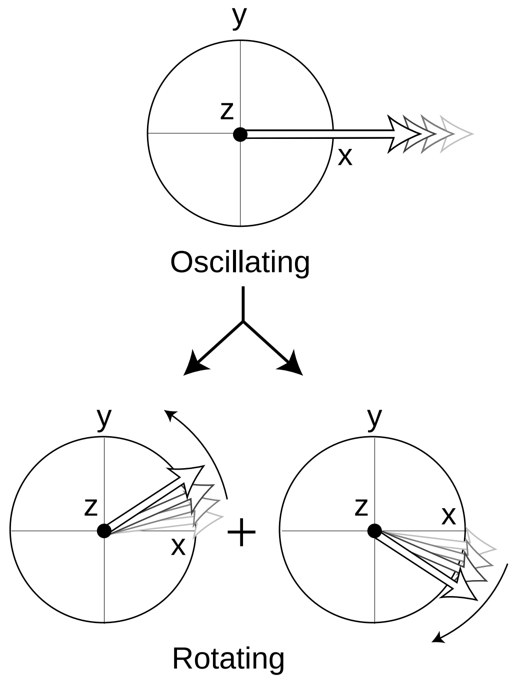
- Spin interacts with external magnetic field:

$$\hat{H}^{j}_{Zeeman} = -\hat{\mu}_j \cdot \vec{B} = -\gamma_j \hat{I}_j \vec{B} \xrightarrow{B_0 // z} \hat{H}^{j}_{Zeeman} = -\gamma_j \hat{I}_{j,z} B_0$$



Transverse RF field

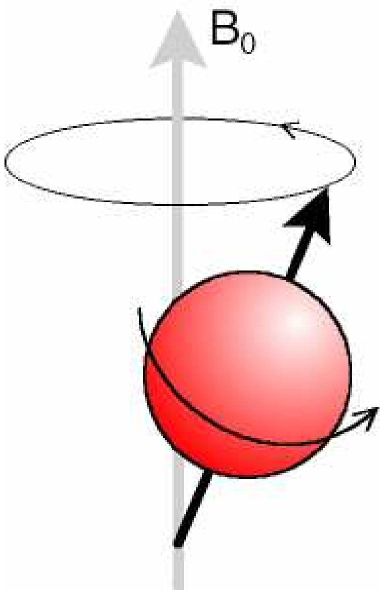




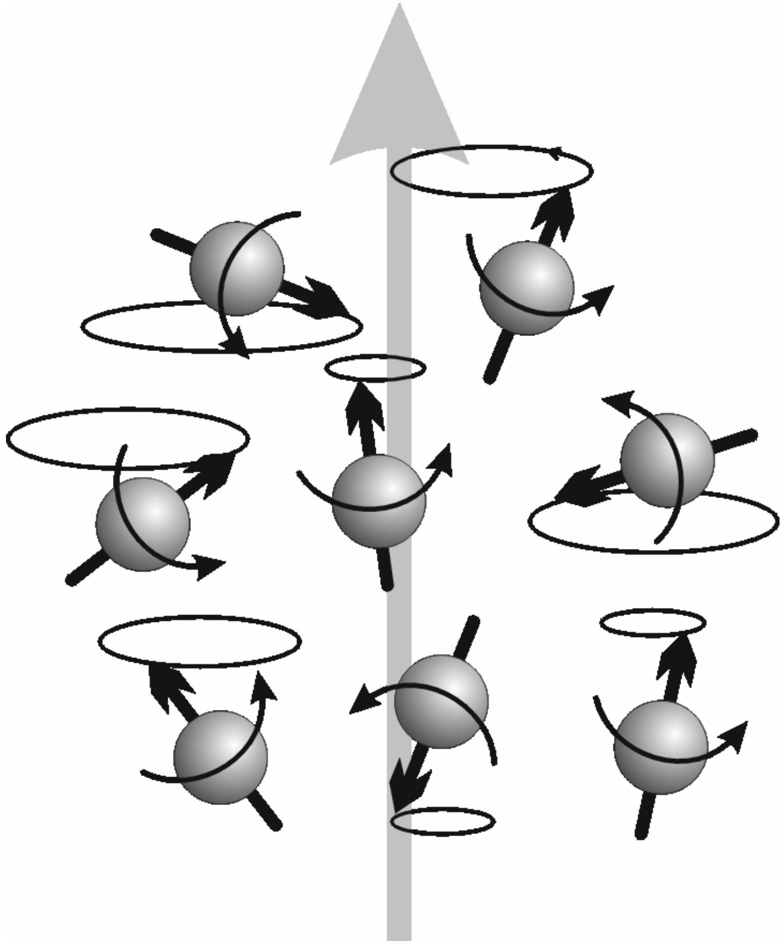
$$\hat{H}^{j}_{RF} = \frac{1}{2}\gamma_j B_1 \{\cos(\omega_0 t + \phi_p)\hat{I}_{j,x} + \sin(\omega_0 t + \phi_p)\hat{I}_{j,y}\}$$

$$\text{nutation frequency } \omega_{nut} = \frac{1}{2}\gamma_j B_1$$

Precession in the Magnetic Field



Larmor precession frequency



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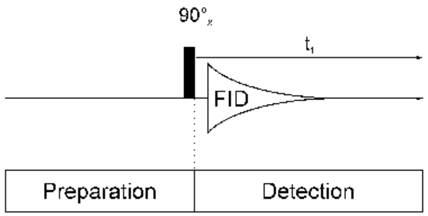
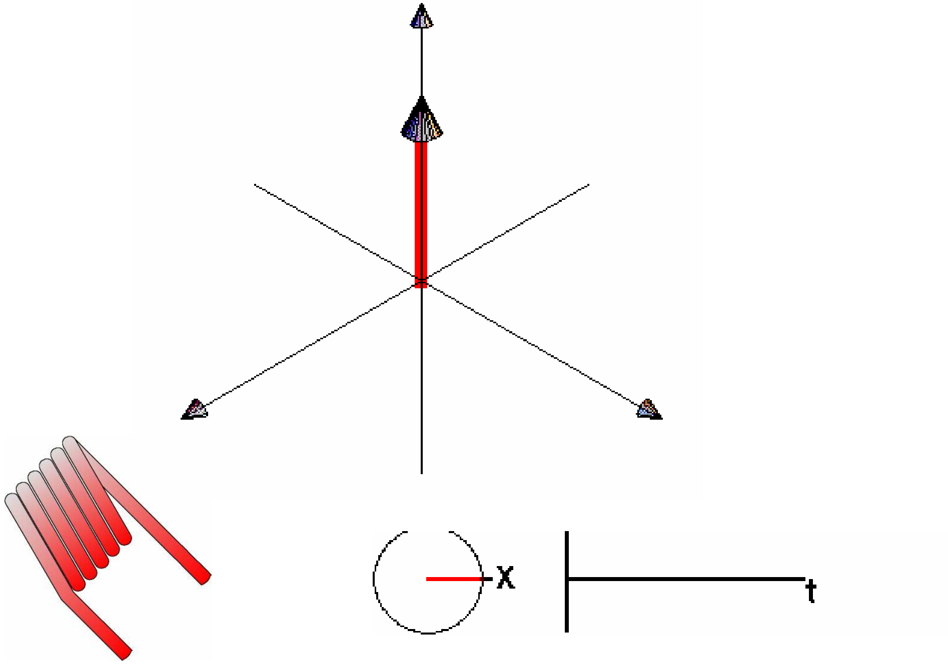
The basic NMR experiment as viewed from the rotating frame

- The magnetization is tipped over by a rf-pulse. The precession of the magnetization in the field induces a voltage in the receiver coil.





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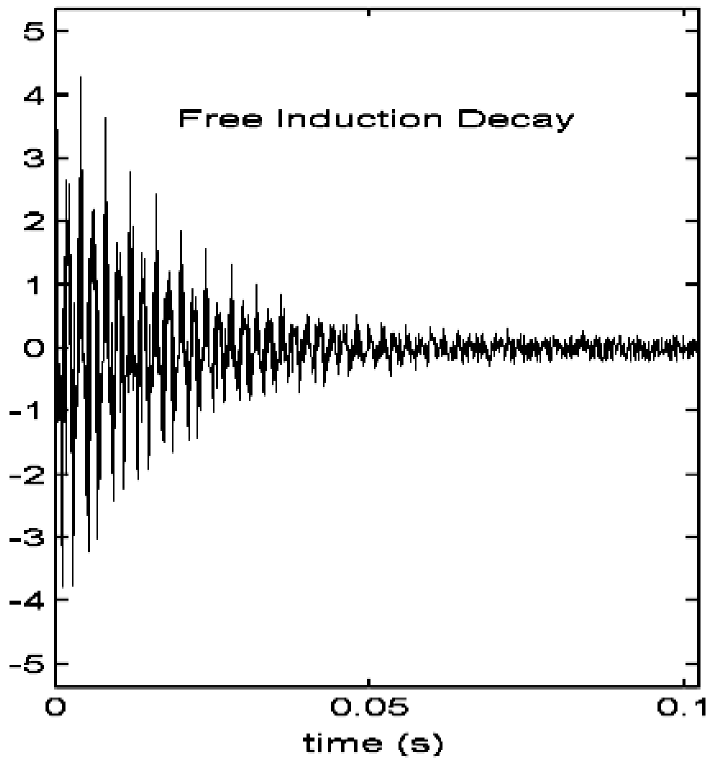
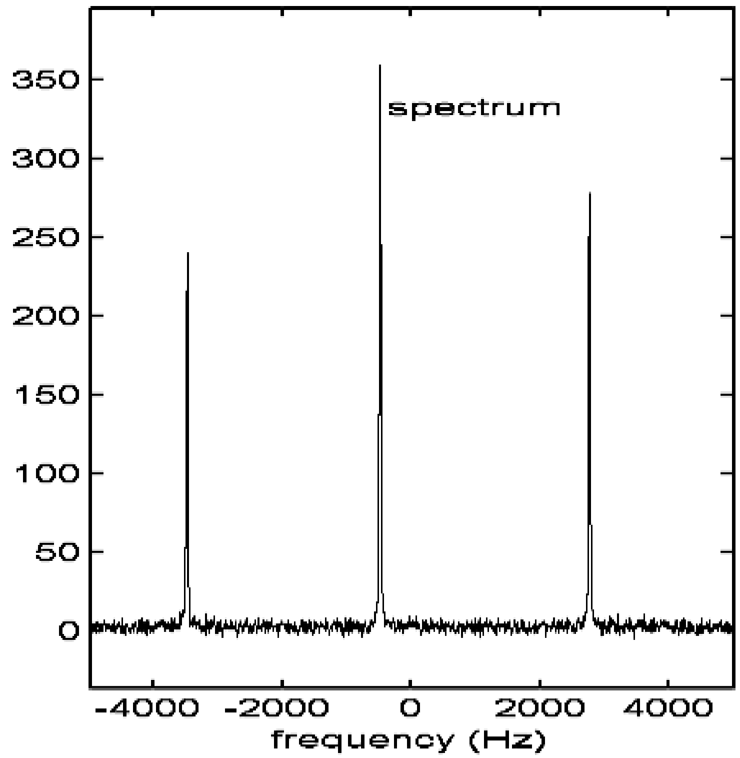
From Free Induction Decay to Spectrum





Fourier Transform

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Chemical Shift

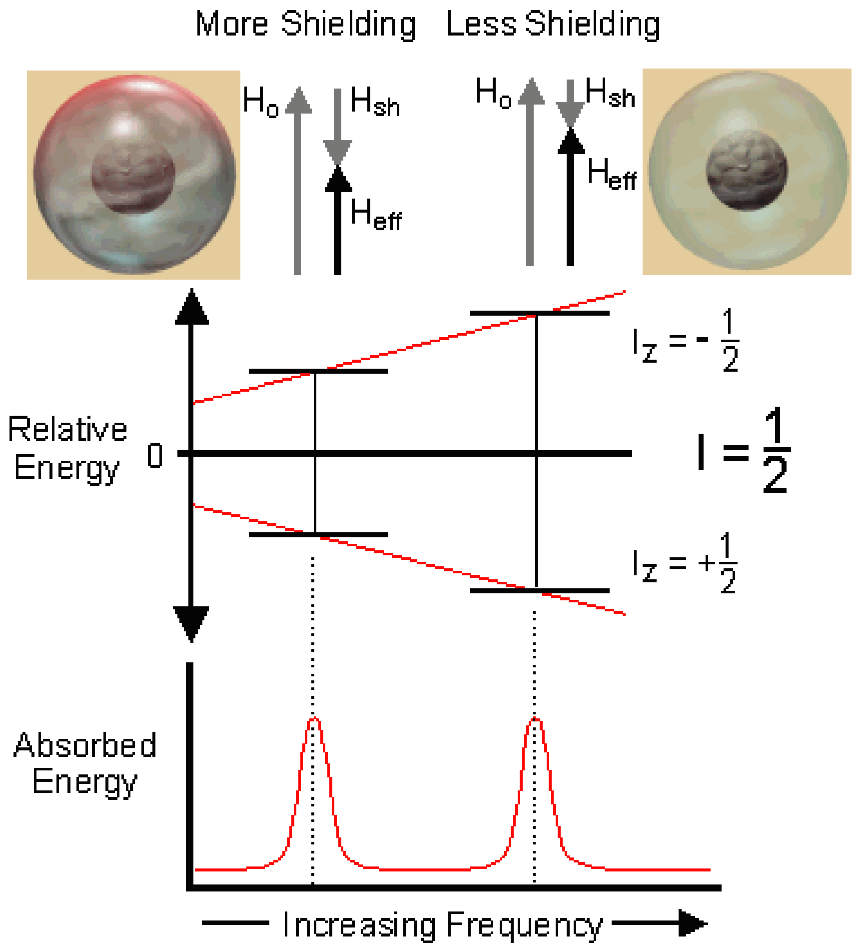
diamagnetism

HFML Nijmegen

$$\vec{B}_{j,loc} = B_0 + \vec{B}_{j,induced}$$

$$\vec{B}_{j,induced} = \begin{bmatrix} \delta_{j,xx} & \delta_{j,xy} & \delta_{j,xz} \\ \delta_{j,yx} & \delta_{j,yy} & \delta_{j,yz} \\ \delta_{j,zx} & \delta_{j,zy} & \delta_{j,zz} \end{bmatrix} \cdot \begin{pmatrix} 0 \\ 0 \\ B_0 \end{pmatrix}$$

$$\text{secular approximation}: \hat{H}^{j}_{CS} \cong -\gamma_j B_0 \delta_{j,zz}(\theta) \hat{I}_{j,z}$$



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Ethanol: CH_3CH_2OH

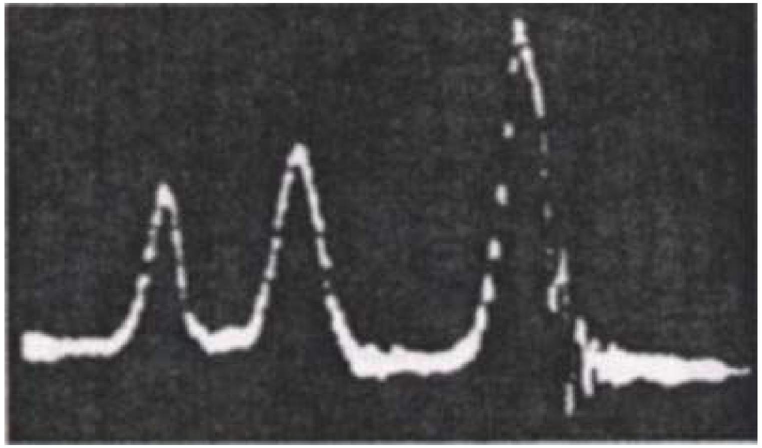
C—H O—H

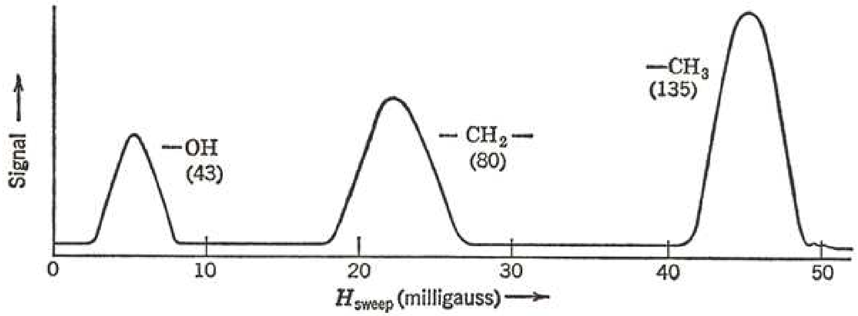
In isotropic liquids motionally averaged chemical shift:

$$\hat{H}^{j}_{CS,iso} \cong -\gamma_j B_0 \delta_{j,iso} \hat{I}_{j,z}$$

$$\delta_{j,iso} = \frac{1}{3}(\delta_{j,xx} + \delta_{j,yy} + \delta_{j,zz})$$

Packard, Stanford 1951

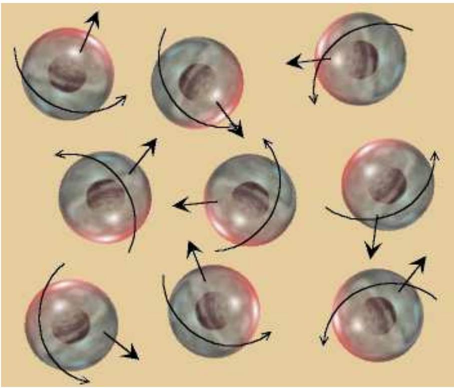


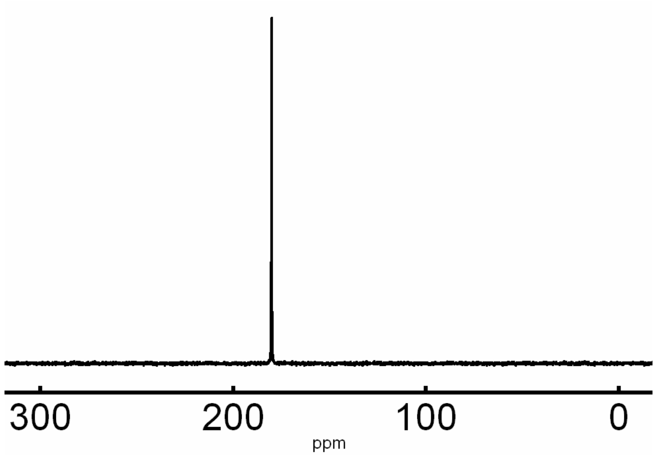


Purcell: "Indeed certain branches of this work are now being pursued in chemical laboratories."

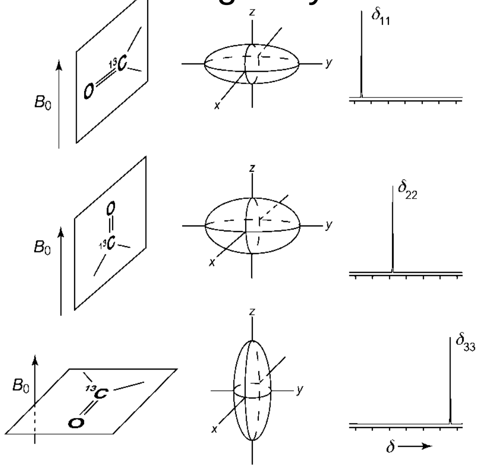
Anisotropic Interactions

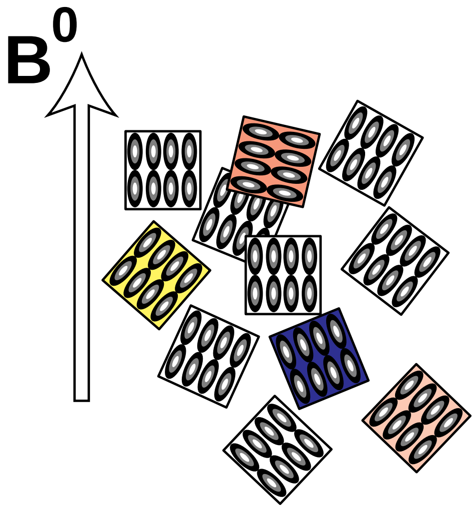
Liquid: rotational and translational motions

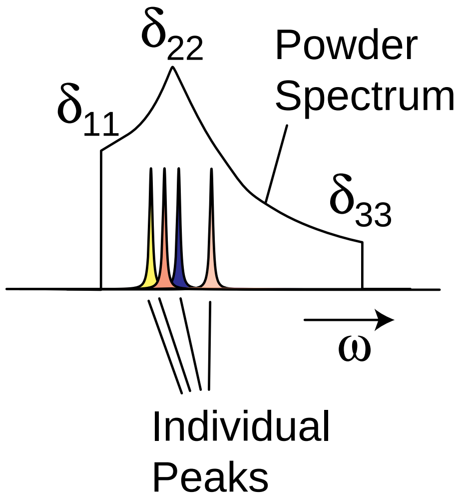




Single crystal







What Information Can NMR Give

Site Identification

- *Chemical Shift*
  - ☞Identification of structural building blocks (^{1}H,^{13}C).
  - ☞Coordination of ^{27}Al, $^{69,71}Ga$, ^{29}Si etc).
  - ☞Hydrogen bonding (^{1}H,^{15}N,^{17}O)
  - ☞Majority of periodic table is accessible

- *Knight shifts, Fermi-contact shifts etc (Lecture Berthier).*
  - ☞Li-ions in paramagnetic battery materials

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Anisotropic ^{13}C interactions

identification

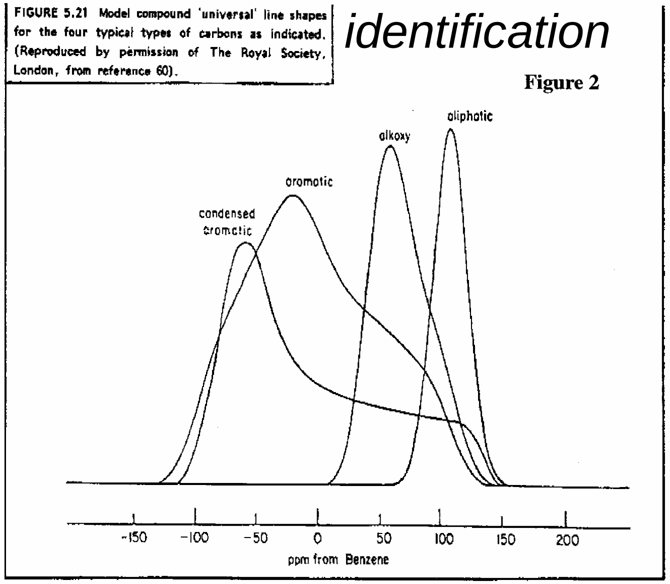

FIGURE 5.21 Model compound 'universal' line shapes for the four typical types of carbons as indicated. (Reproduced by permission of The Royal Society, London, from reference 60).

alignment

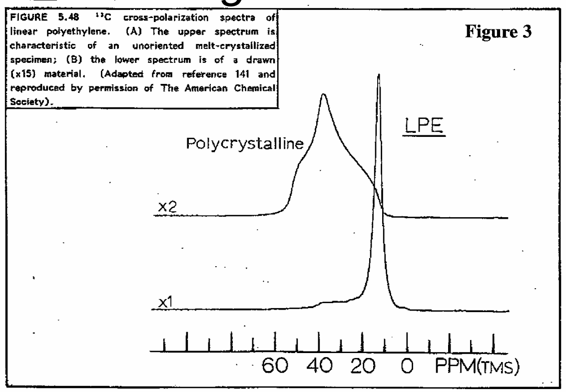

FIGURE 5.48 ^{13}C cross-polarization spectra of linear polyethylene. (A) The upper spectrum is characteristic of an unoriented melt-crystallized specimen; (B) the lower spectrum is of a drawn (x15) material. (Adapted from reference 141 and reproduced by permission of The American Chemical Society).

Molecular motions

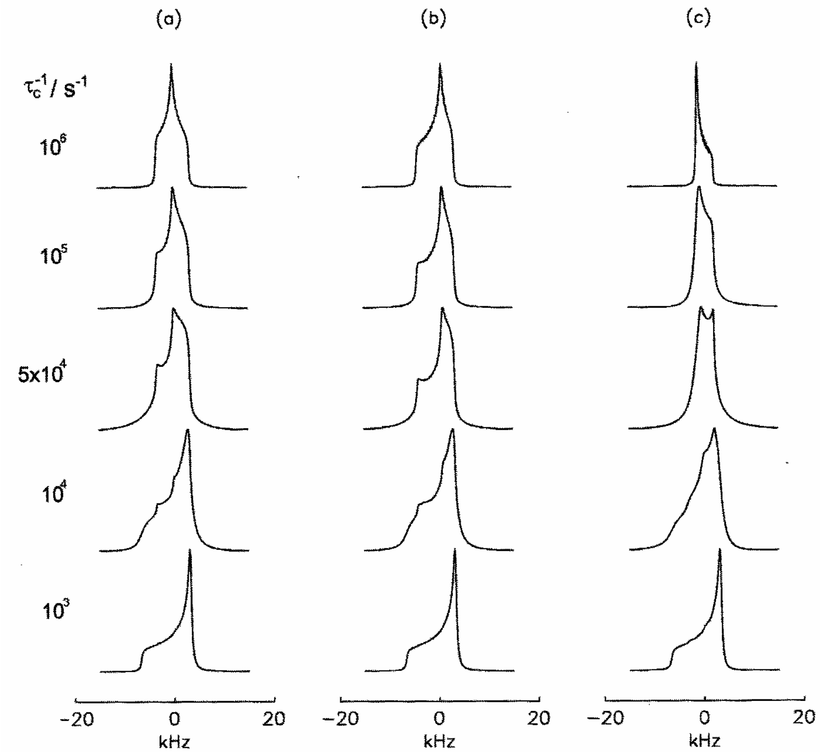

Fig. 6.5 Some chemical shift anisotropy lineshapes under conditions of molecular motion. Three different models of molecular motion are considered: (a) two-site hopping, chemical shift tensor principal *z*-axis reorientates by 109.5°, (b) two-site hopping, chemical shift tensor principal *z*-axis reorientates by 120° and (c) three-site hopping about a rotation axis orientated at 70.5° to the chemical shift tensor principal *z*-axis in each site. In all cases, the chemical shift tensor is axially symmetric and the populations of each site are equal. The τ_c^{-1} (Ω) for each case are given with the spectra. The $\mathbf{\Pi}$ matrices used in the calculations

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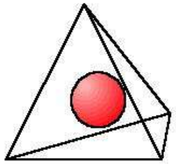
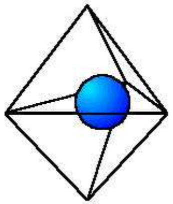
Site Identification

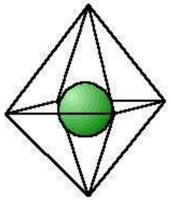
^{27}Al NMR of oxides



4-fold coordinated Al:
80 - 40 ppm

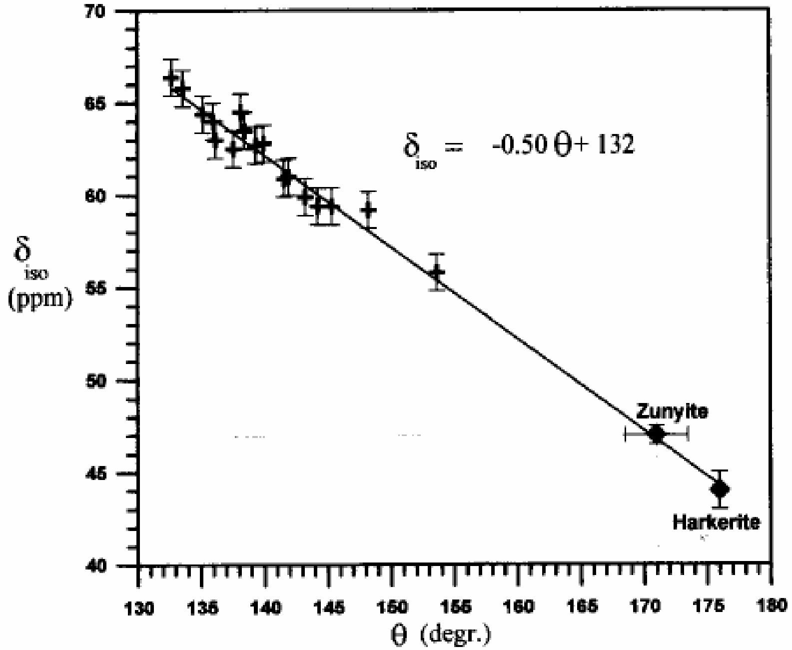


5-fold coordinated Al:
40 - 20 ppm



6-fold coordinated Al:
20 - -10 ppm

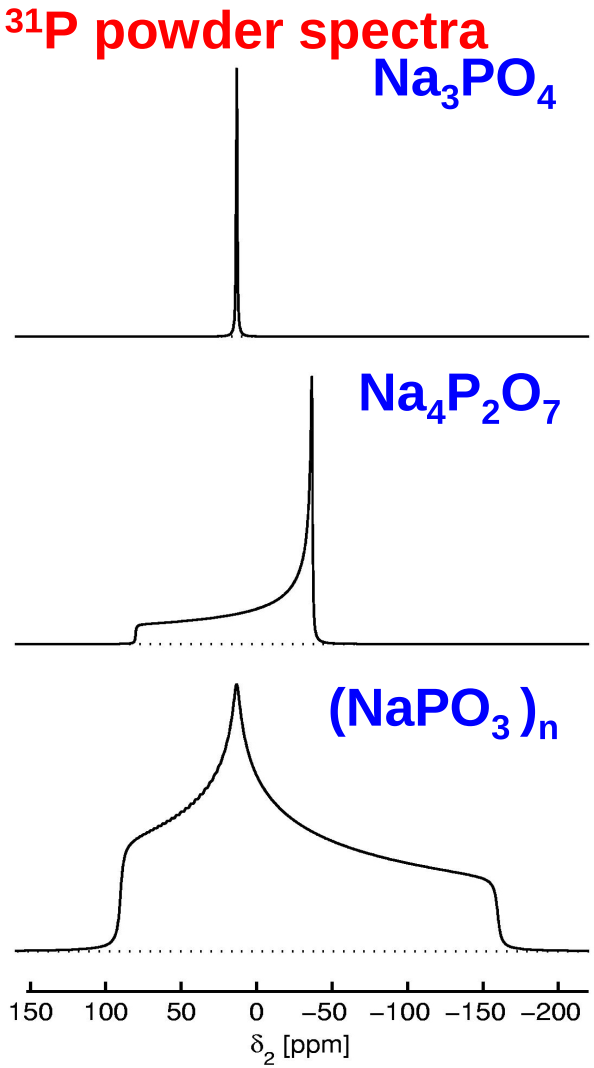
δ_{cs} Tetrahedral <u>Al</u>-O-Si in aluminosilicates



Anisotropic Interactions

- ☹ Broad lines
- ☺ Structural information
- ☺ study dynamics
- ☺ **Manipulation in ordinary and spin space**

✂ use adequate tools



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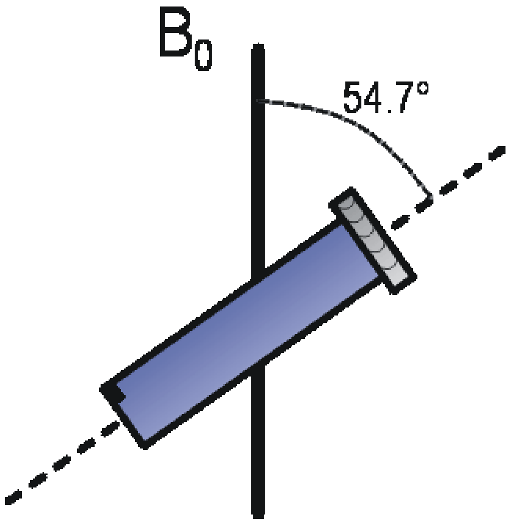
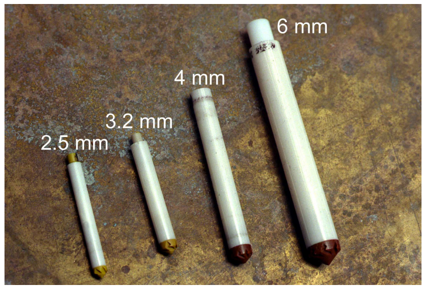
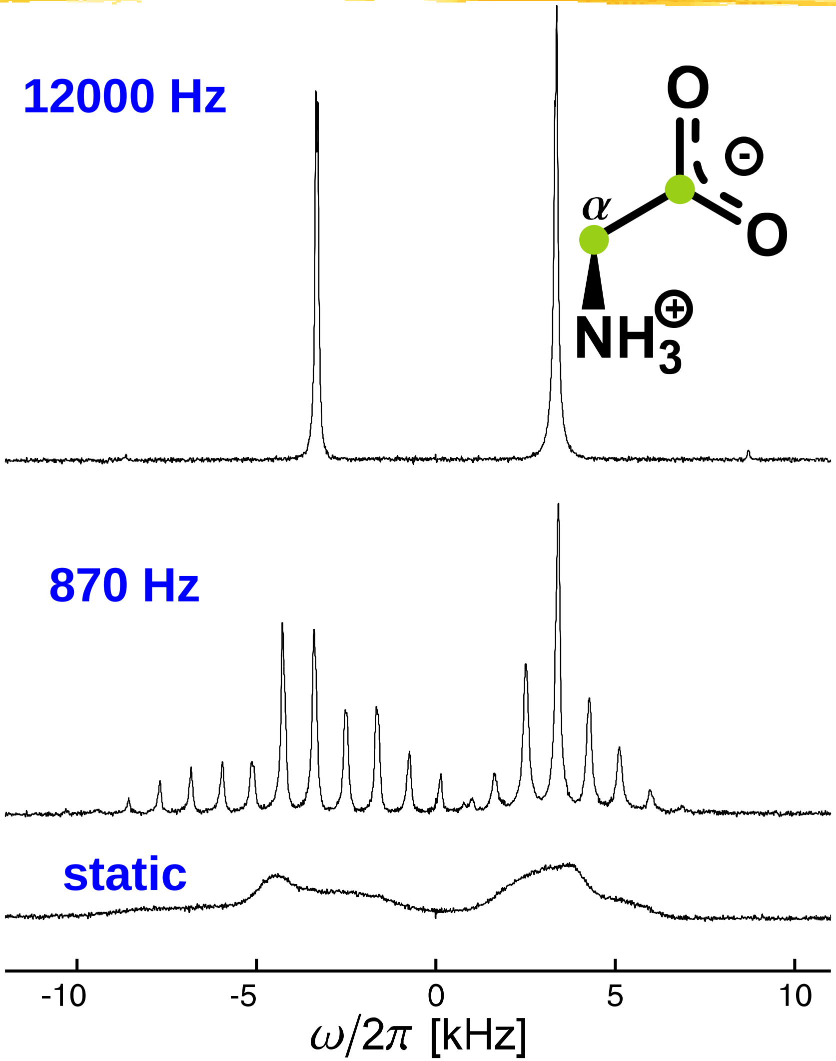
Tool: Magic-Angle Spinning

Averaging of anisotropic interactions in ordinary coordinate space.







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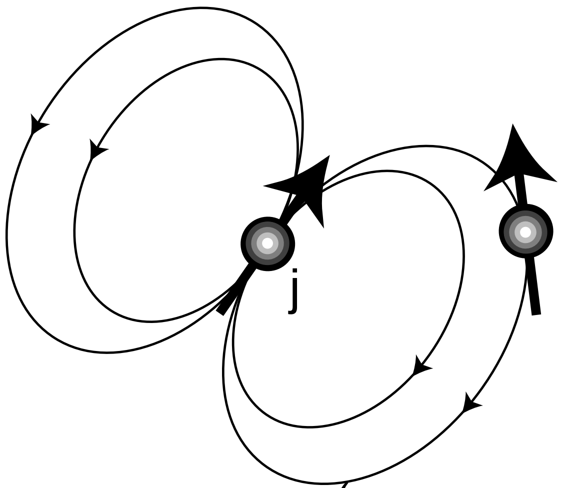
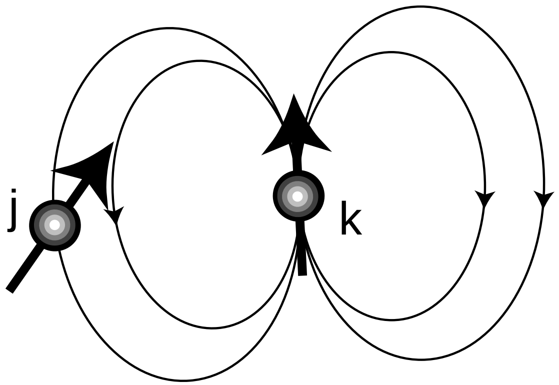
Direct dipole-dipole interactions





$$\hat{H}_{DD}^{jk} = b_{jk}\left(3(\hat{I}_j \cdot e_{jk})(\hat{I}_k \cdot e_{jk}) - \hat{I}_j \cdot \hat{I}_k\right)$$

e_{jk} is the unit vector connecting spin j and k

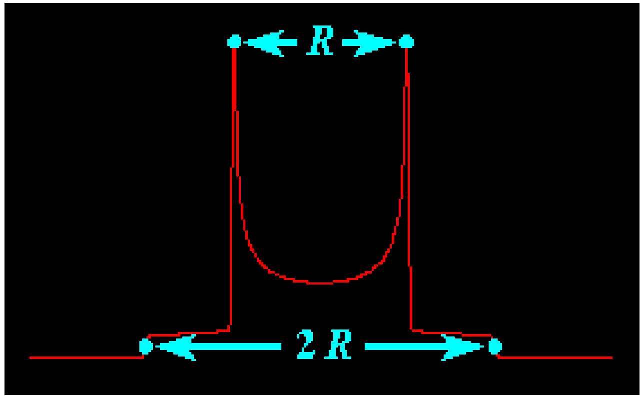
secular approximation :

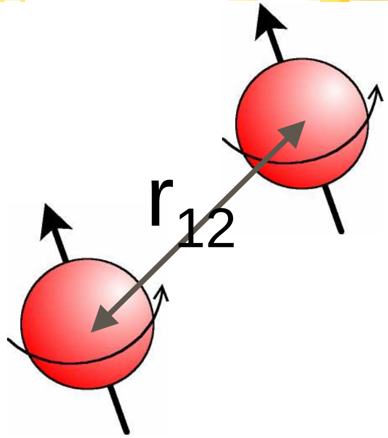
homonuclear : $\hat{H}_{DD}^{jk}(\theta_{jk}) = b_{jk}\left(3\cos^2(\theta_{jk}) - 1\right)\left(3\hat{I}_{jz}\hat{I}_{kz} - \hat{I}_j \cdot \hat{I}_k\right)$

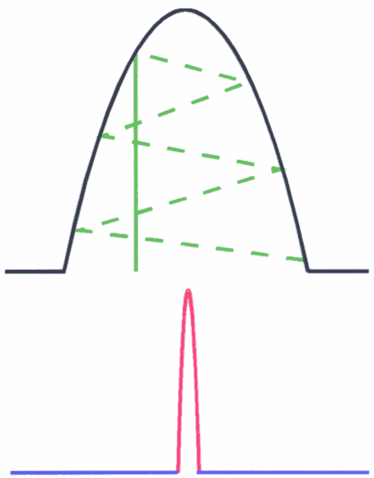
heteronuclear : $\hat{H}_{DD}^{jk}(\theta_{jk}) = b_{jk}\left(3\cos^2(\theta_{jk}) - 1\right)\left(2\hat{I}_{jz}\hat{I}_{kz}\right)$

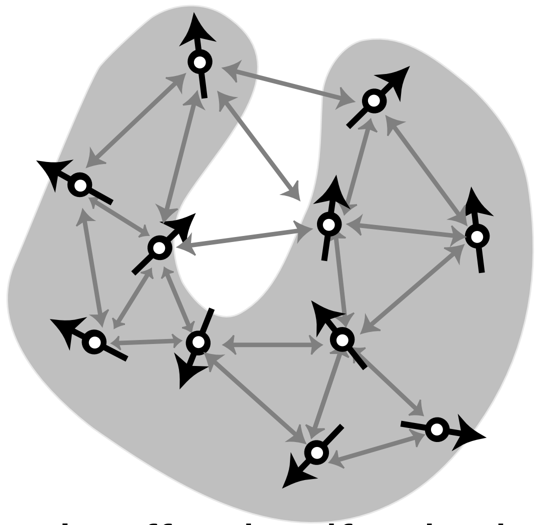
Anisotropic Dipolar Interaction

Pake doublet









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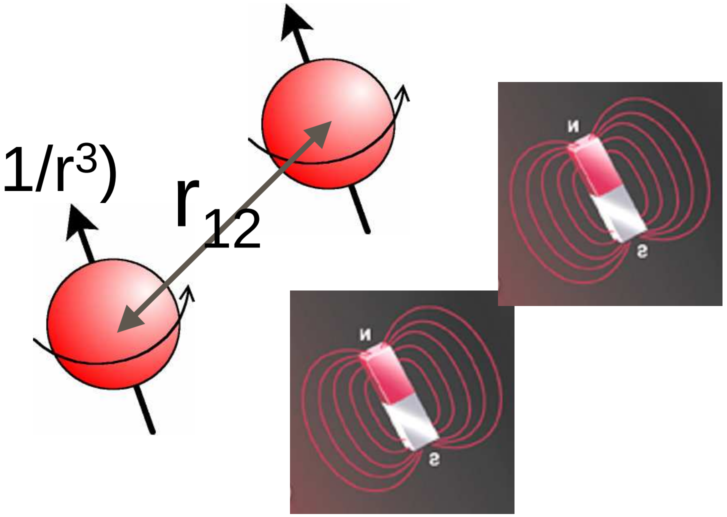
For abundant spins MAS is only effective if spinning speed significantly exceeds the line width

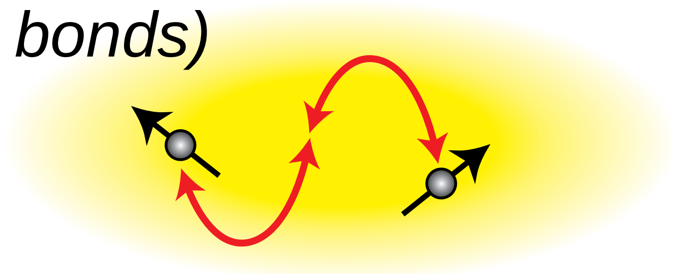
What Information Can NMR Give

Intersite correlations

- *Dipolar Interactions (through space)*
  - ☞Spatial proximity of nuclei (~1/r^3)
  - ☞Homonuclear
  - ☞Heteronuclear



- *J-couplings (mediated through chemical bonds)*
  - ☞Existence of chemical bonds.



- *Hyperfine interactions (coupling to electron spin > Lecture Berthier).*

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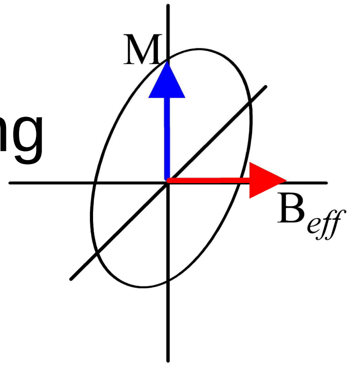
Tool: Radio Frequency Irradiation

- Heteronuclear decoupling of nuclei by CW-irradiation with resonant RF waves. Pulsed alternatives TPPI, XiX etc.

- Homonuclear decoupling by CW irradiation at the magic angle (Lee-Goldburg decoupling). Pulsed alternatives: WAHUHA, MREV-8, FSLG, Dumbo etc.

CW-decoupling

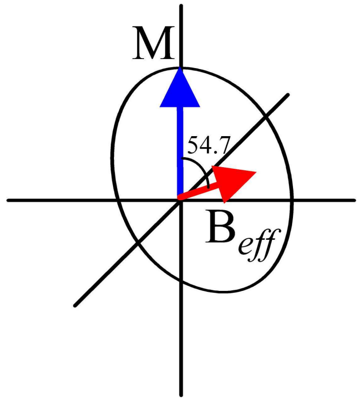
$\langle I_z \rangle = 0$



LG-decoupling

$\langle 3 I_{j,z} I_{k,z} - I_j I_k \rangle = 0$

$\langle I_z \rangle = 1/\sqrt{3}\ I_z$



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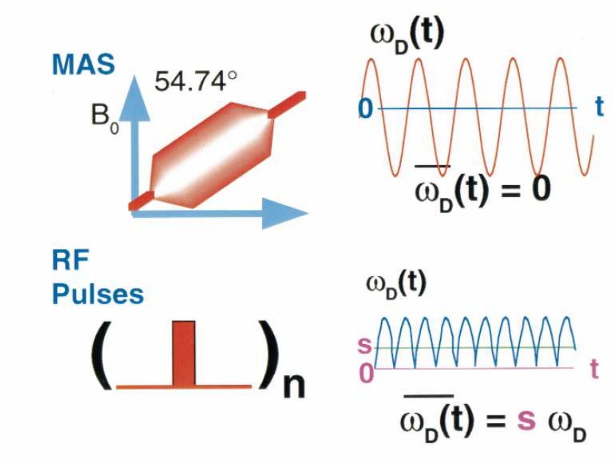
Manipulation of interactions in spin space.

Combining Tools

- Combined Rotational and Multiple Pulse Decoupling

- Recoupling of dipolar interactions using radio-frequency sequences synchronized with sample spinning and matched rf-field strength.



- Transfer of coherence of coupled nuclei

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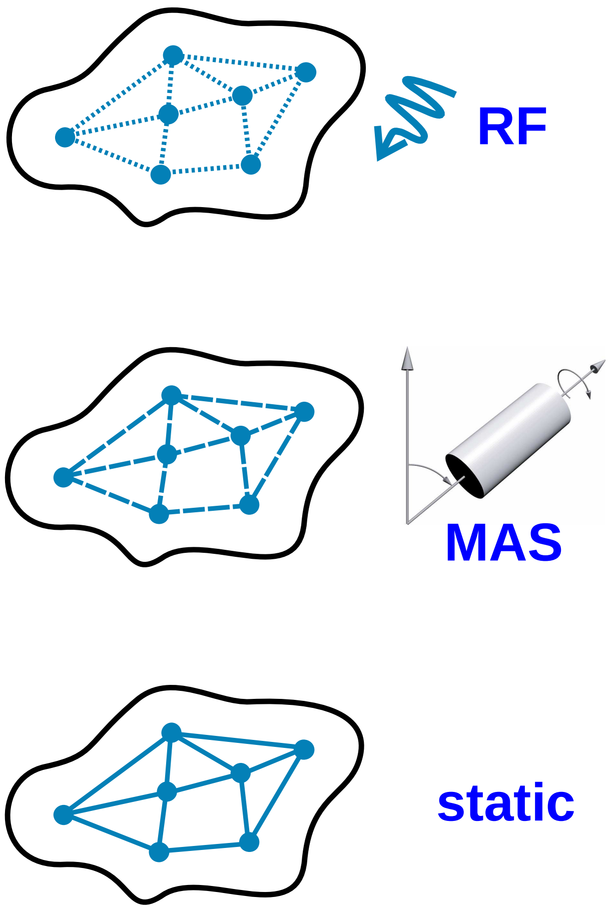
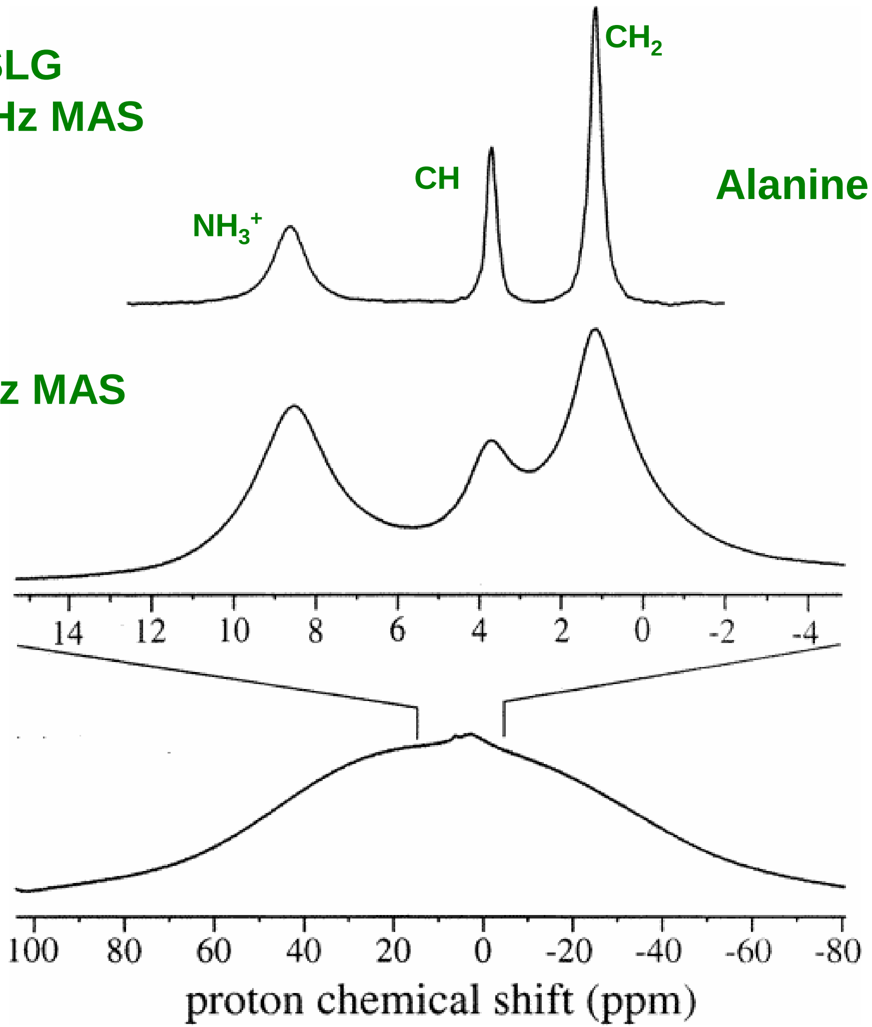
^{1}H Spectroscopy





A. Lesage, L. Duma, D. Sakellariou, and L. Emsley, *J. Am. Chem. Soc.* **123**, 5747 (2001)

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Combining Tools

Adamantane | Natural Rubber

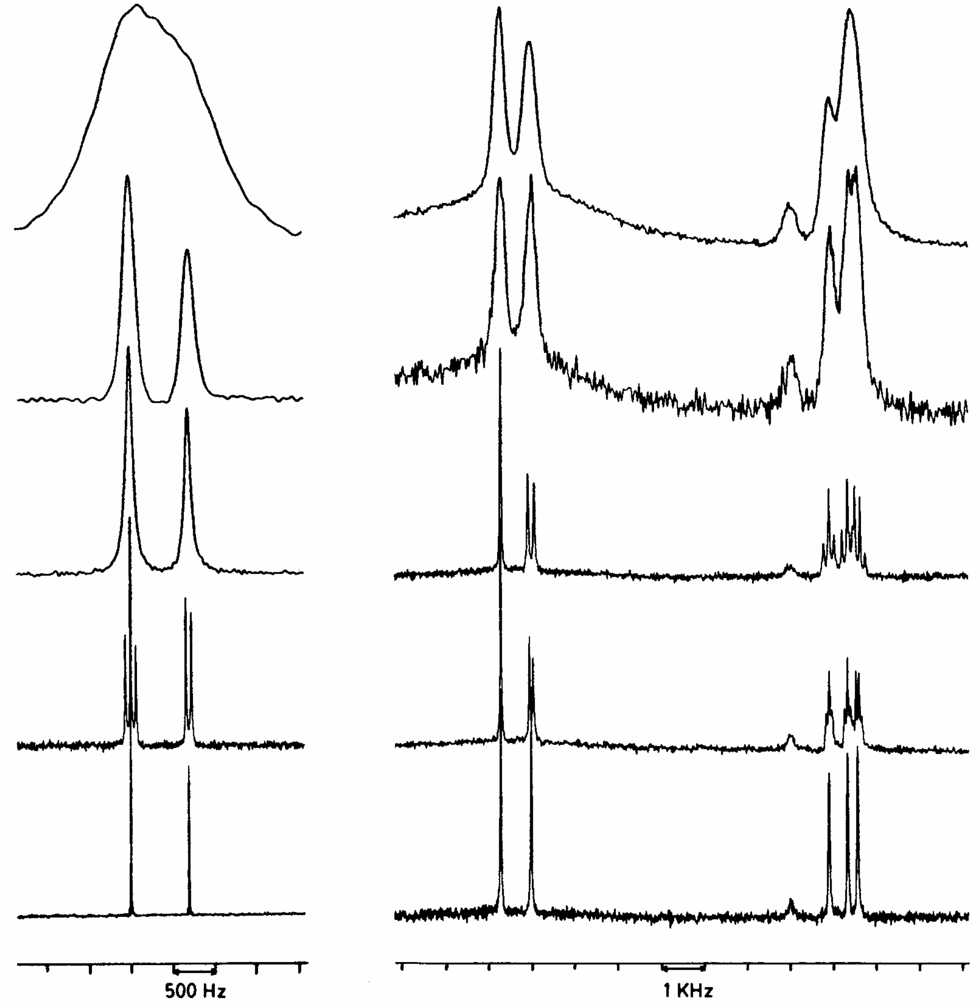
^{13}C Single Pulse Excitation

+ ^{1}H CW Decoupling

+ Magic Angle Spinning

+ MAS + ^{1}H LG decoupling

+ MAS + ^{1}H CW decoupling



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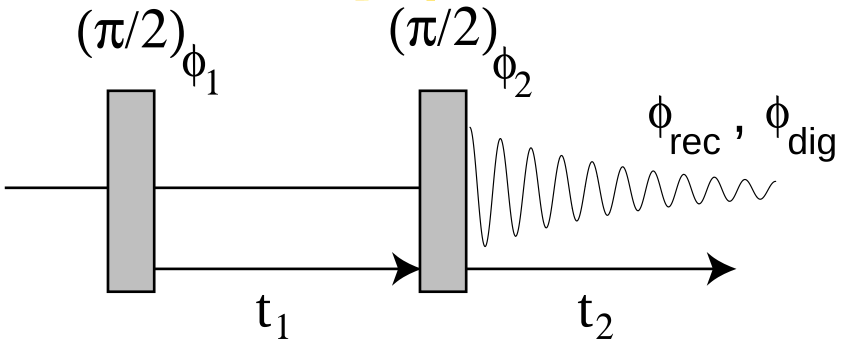
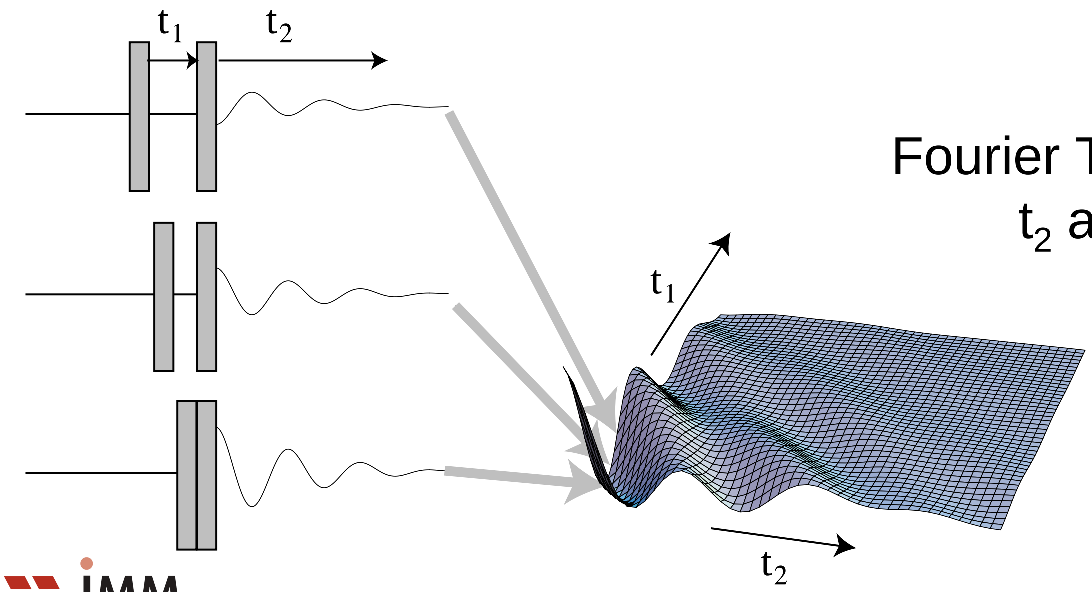
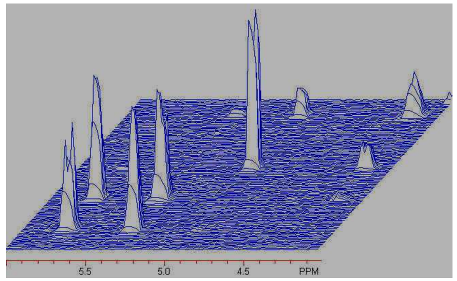
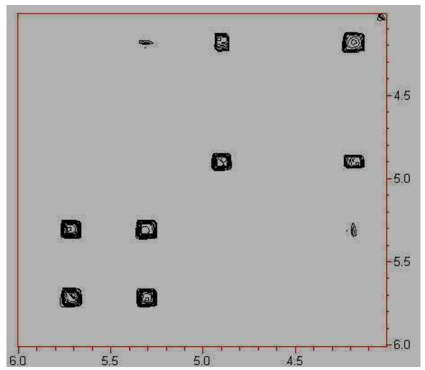
Two-dimensional NMR



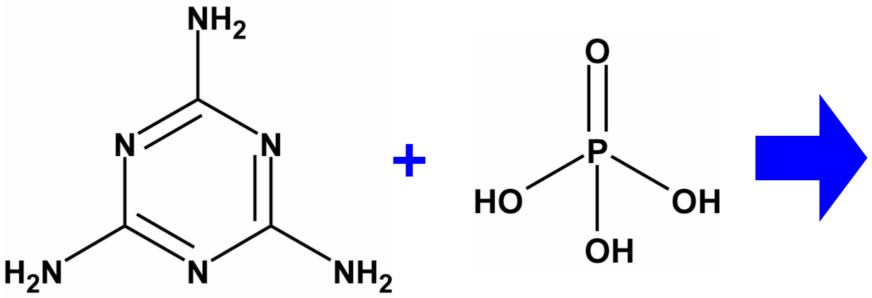


Fourier Transform
t_2 and t_1





Case Study: Environment-Friendly Condensed Phase Flame Retardants



Melamine
M

Phosphoric Acid
P

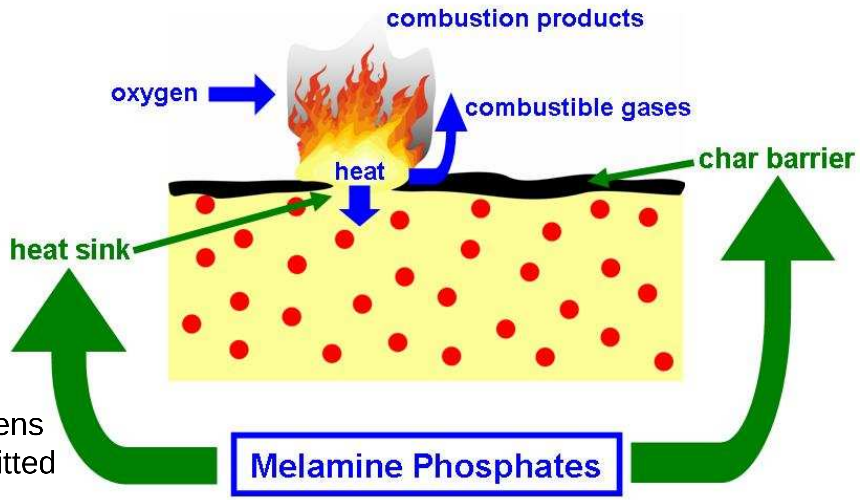
Melamine Phosphates:

MP
$(MP)_2$ (Pyrophosphate)
M Polyphosphate

↓ **Polymerization**

Conversion ~200°C - 260°C
Above 260 °C decomposition

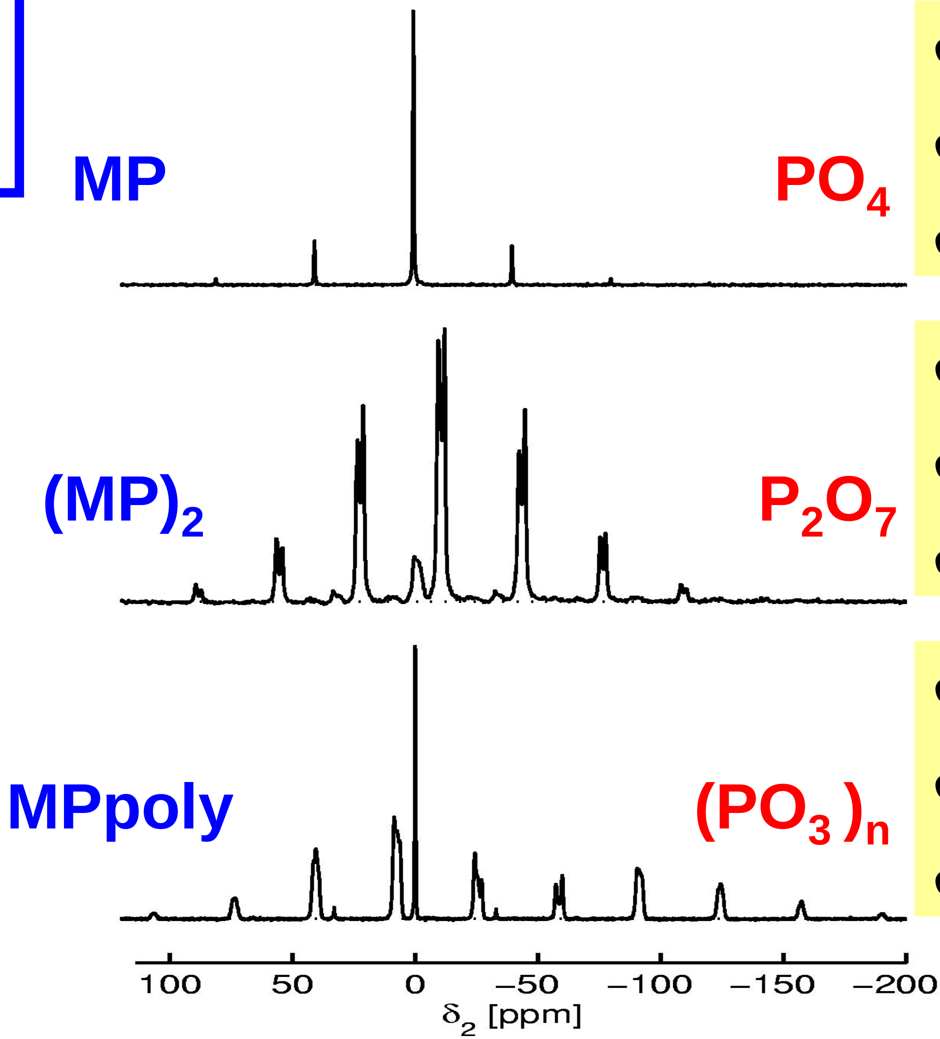
Crystal structures unknown
Polymerization process unknown
FR Mechanism unknown



A. Brinkmann, E.R.H van Eck & A.P.M. Kentgens
Magnetic Resonance in Chemistry, 2007 submitted

Site ID
with added *anisotropic* information

^{31}P Spectra
$\omega_r/2\pi$ = 12 kHz
B_0 = 7.1 T



σ_{11} = 46 ppm
σ_{22} = 4 ppm
σ_{33} = -51 ppm

σ_{11} = 68 ppm | 64 ppm
σ_{22} = -12 ppm | -8 ppm
σ_{33} = -84 ppm |-91 ppm

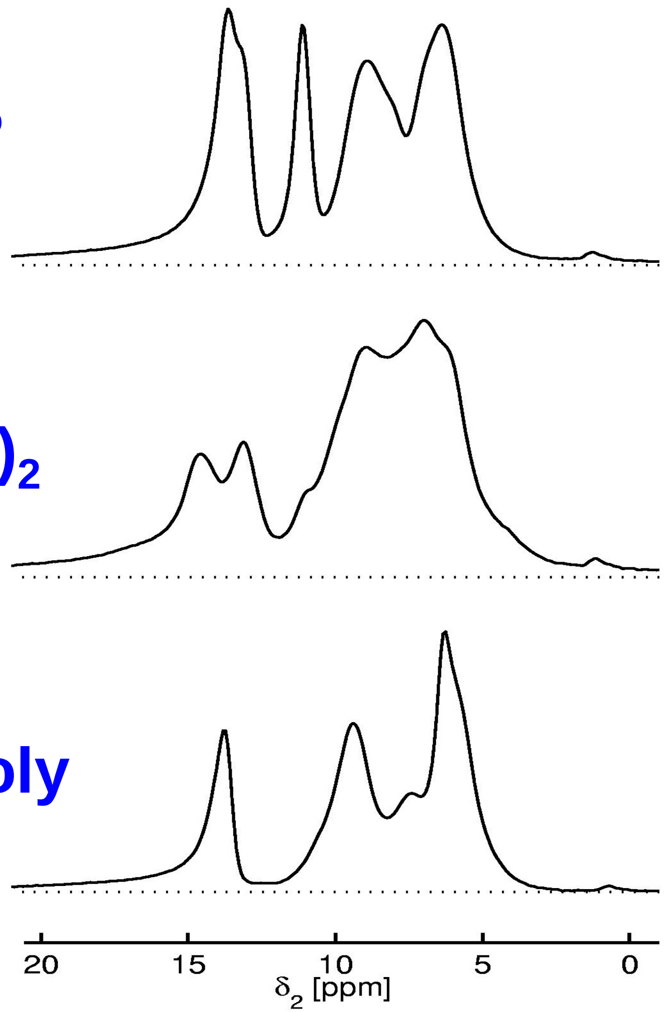
σ_{11} = 77 ppm
σ_{22} = 14 ppm
σ_{33} = -168 ppm

1D ^{1}H Spectra at 18.8 T

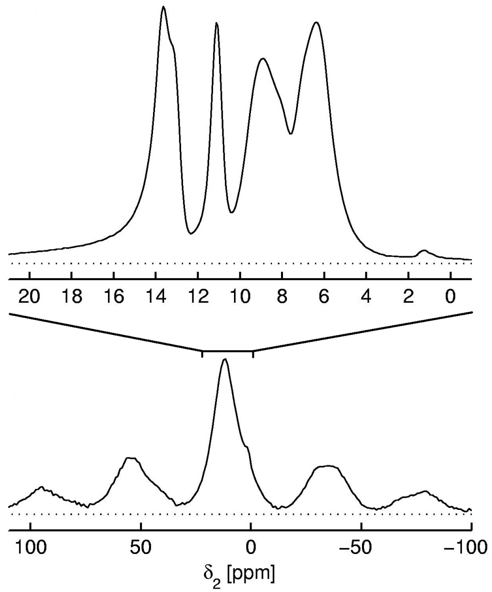
MP

$(MP)_2$

MPpoly



^{1}H Spectra
MAS
$\omega_r/2\pi$ = 49 kHz
B_0 = 18.8 T



$\omega_r/2\pi$ = 12 kHz
B_0 = 7.1 T

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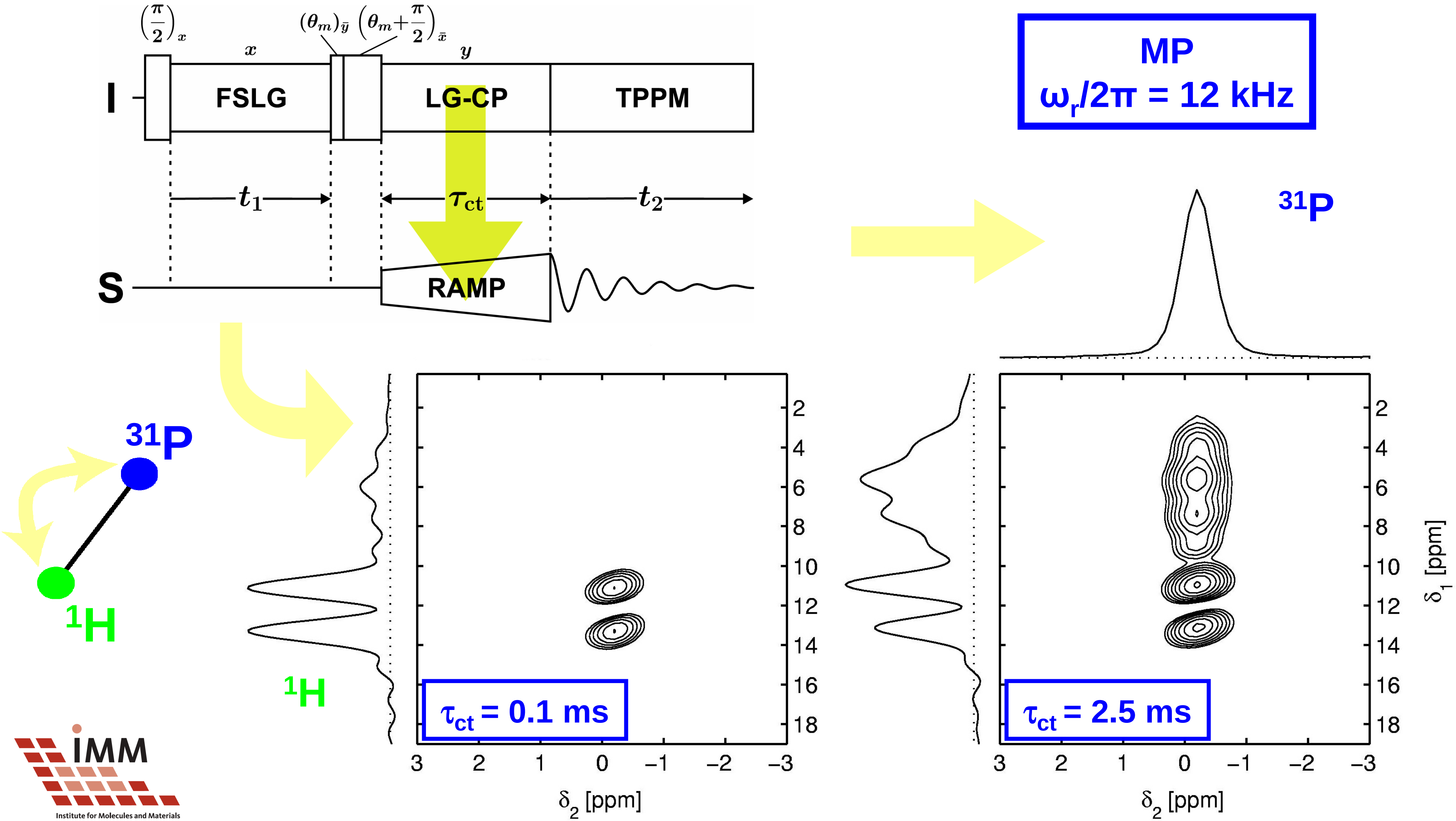
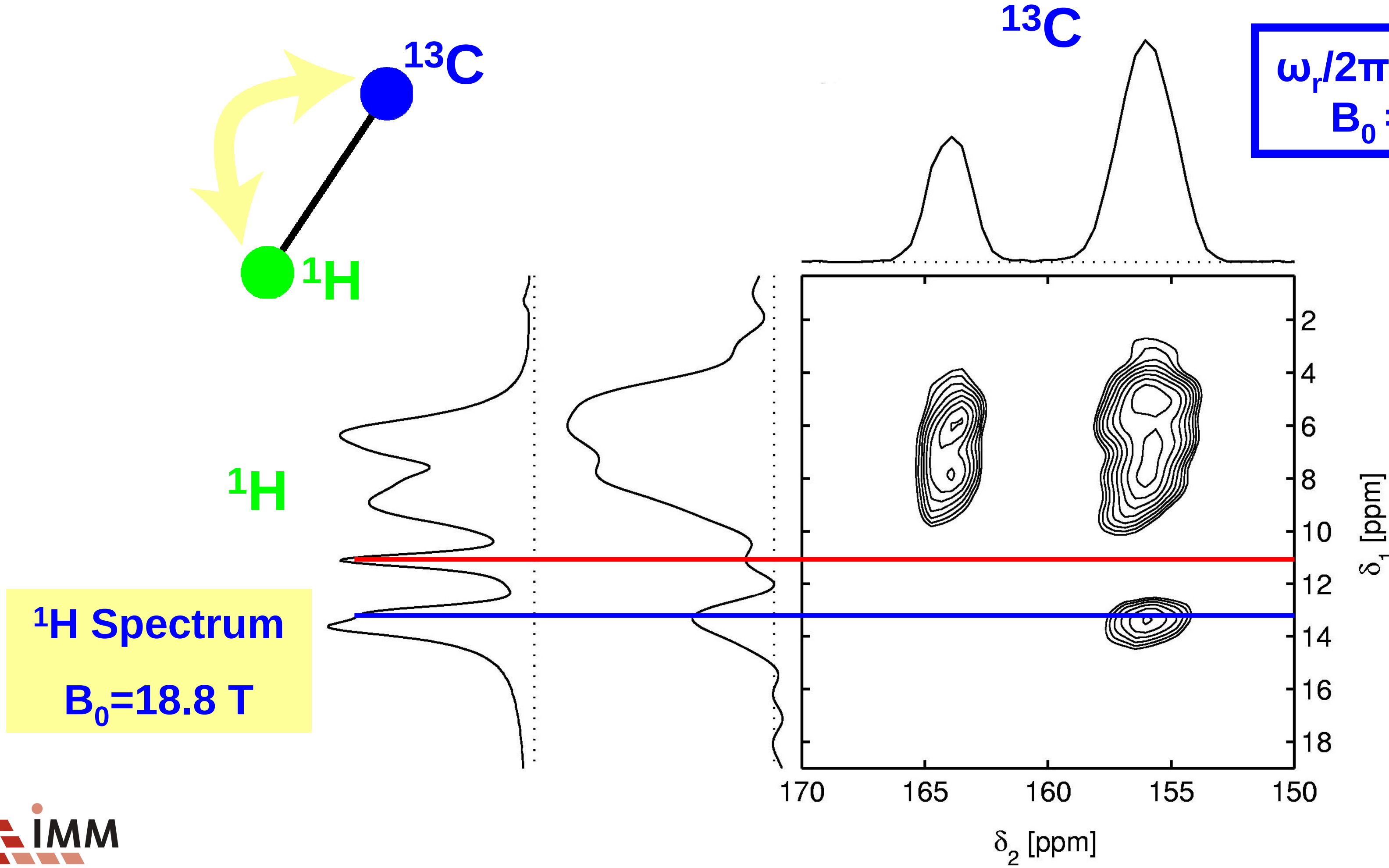
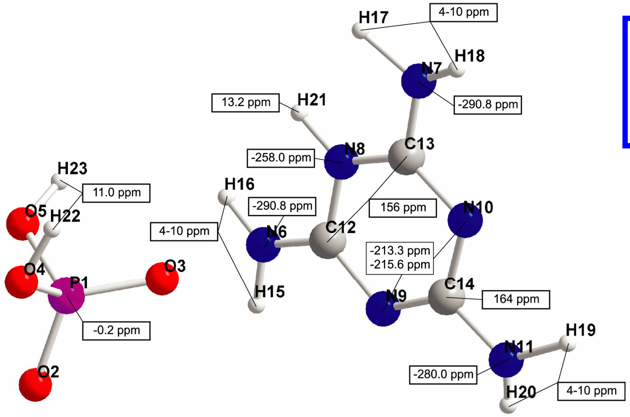
Heteronuclear Correlation ^{1}H-^{31}P



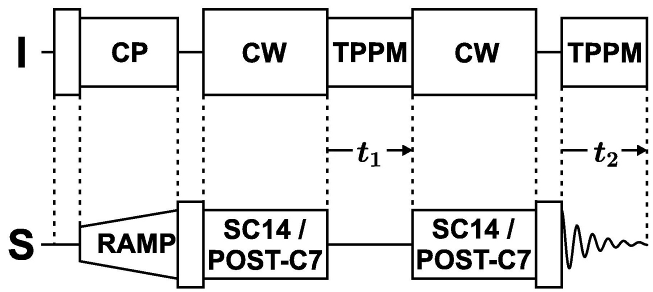
Heteronuclear Correlation ^{1}H-^{13}C

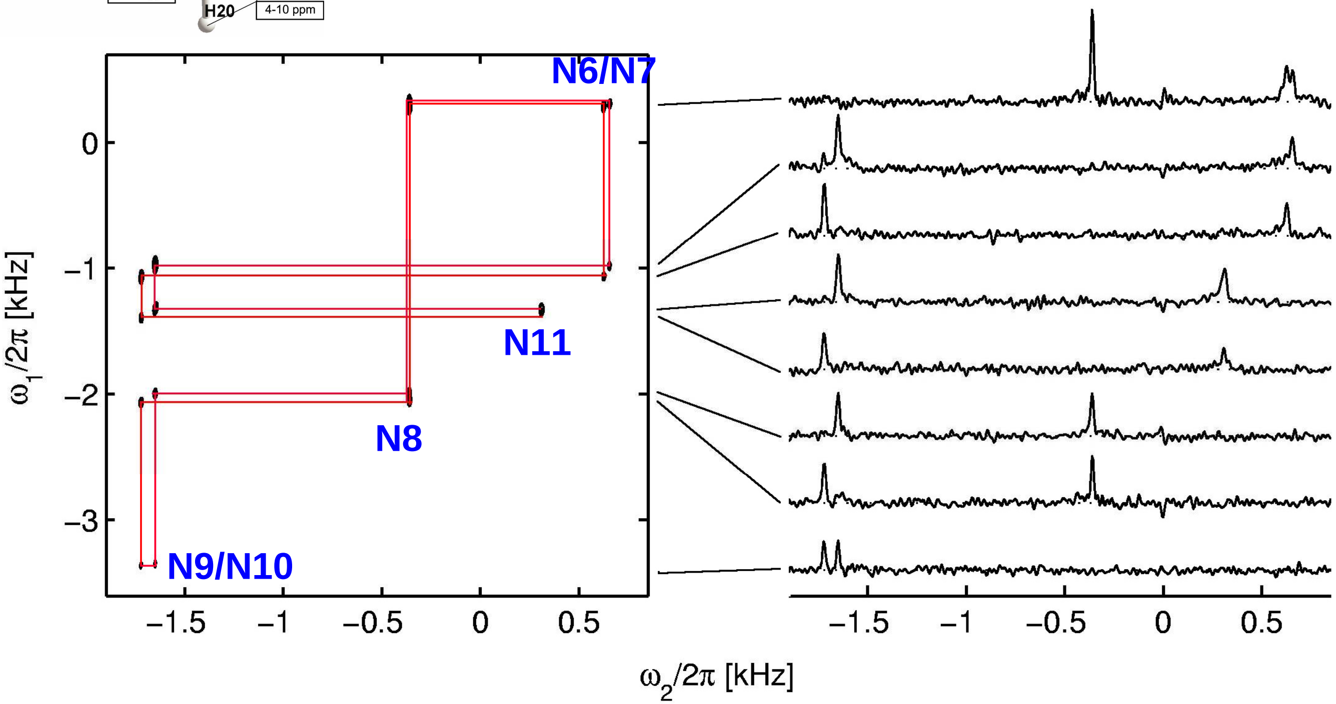


Homonuclear: ^{15}N-^{15}N in MP



$\omega_r/2\pi$ = 12 kHz
B_0 = 7.1 T

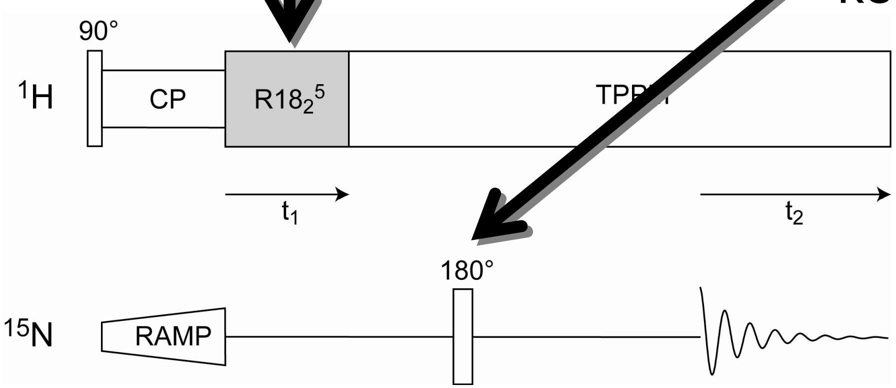


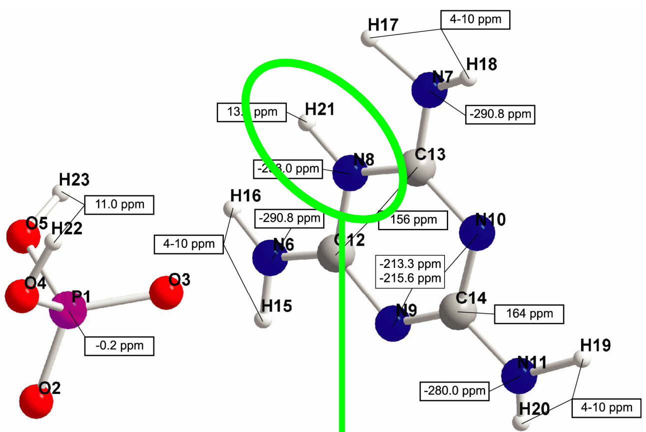


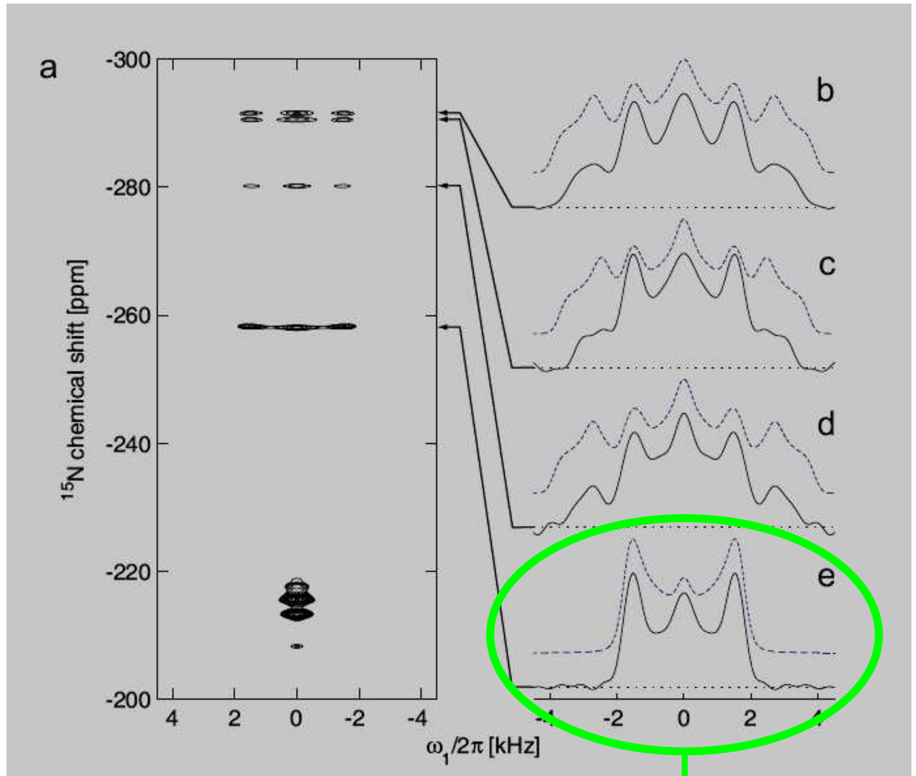
^{15}N-^{1}H distance measurements

Dipolar recoupling

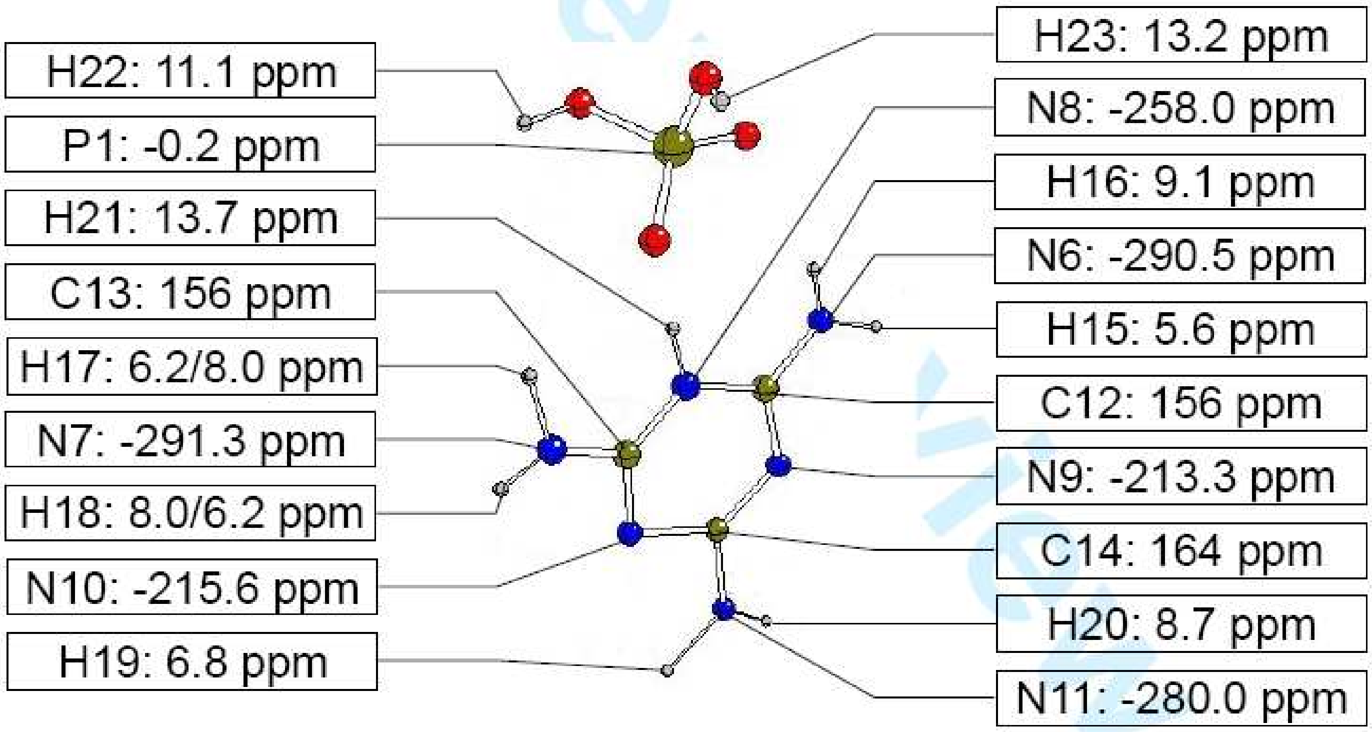
Echo: refocus ^{15}N chemical shift,
keep ^{15}N-^{1}H dipolar interaction







MP: Assignment

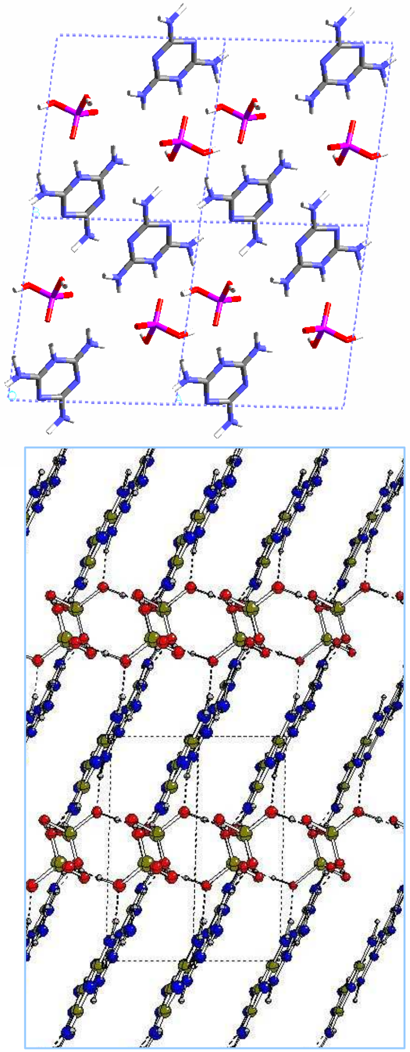


Hydrogen Bonding & π–π Stacking

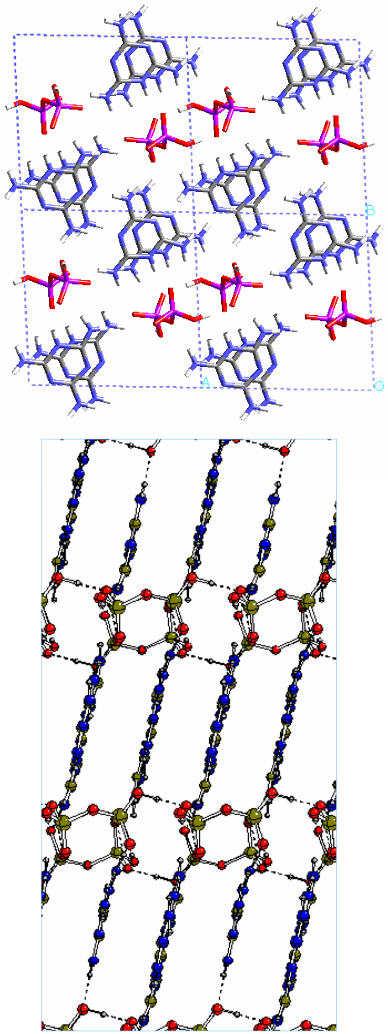
Combined NMR and X-ray Powder Diffraction

V. Brodski, R.Peschar and H. Schenk

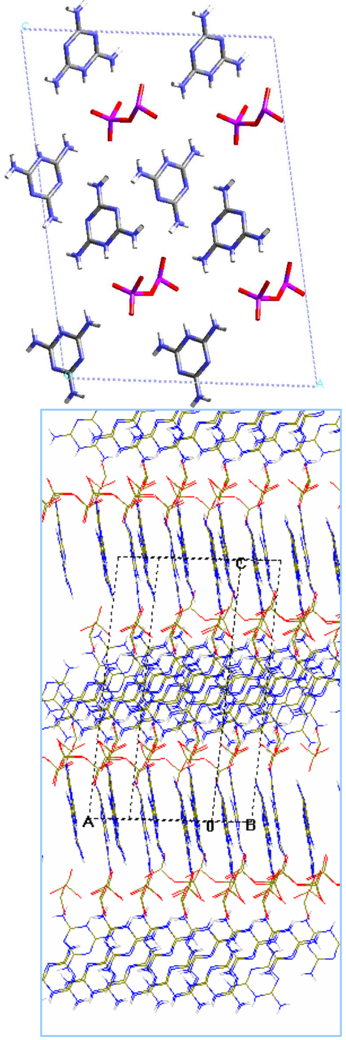
Univ. Of Amsterdam



MP



MPyro

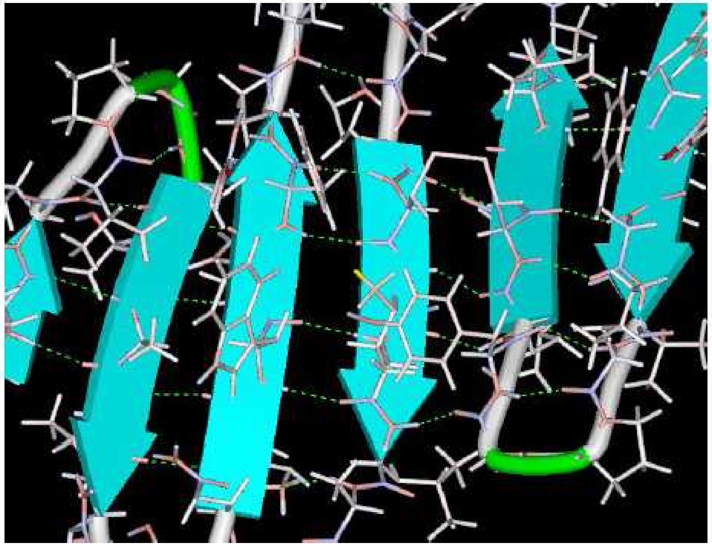


MPpoly

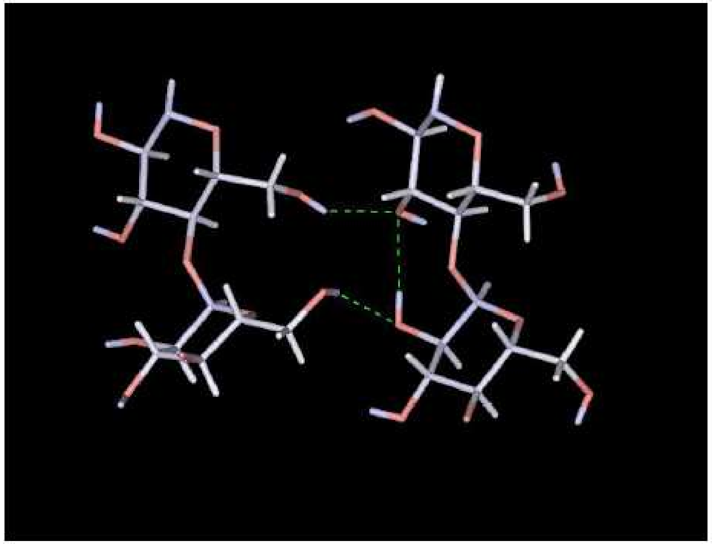
Hydrogen bonding in biological molecules

N-H···O

O-H···O



Proteins and Peptides



Polysaccharides

^{17}O is a quadrupolar I=5/2 nucleus

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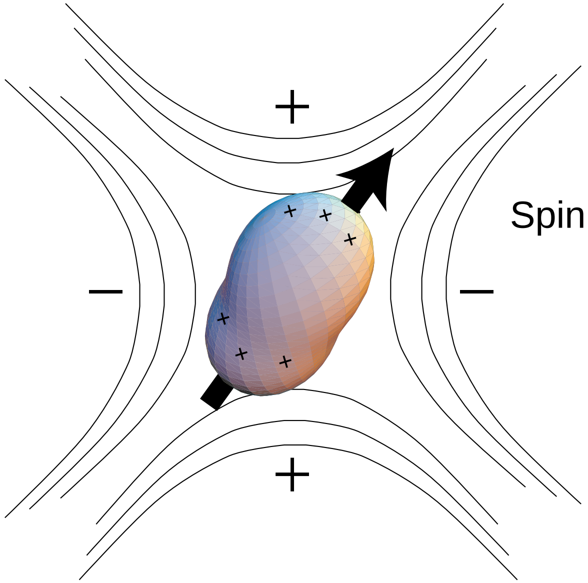
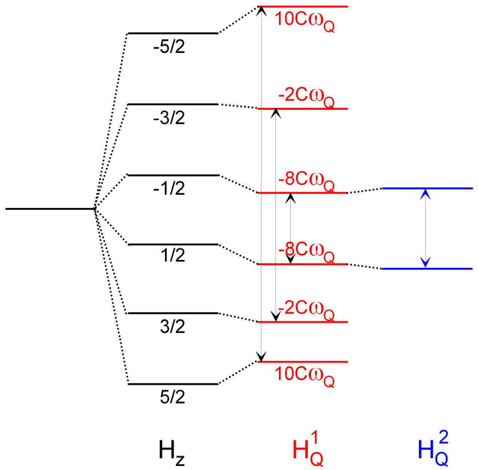
Quadrupolar Interaction



Spin > 1/2



$$\text{secular approximation: } \hat{H}_Q^j(\theta) = \omega_{j,Q}\left(3\hat{I}_{jz}^2 - \hat{I}_j \cdot \hat{I}_j\right)$$

$$\text{with } \omega_{j,Q}(\theta) = \frac{3eQ_j}{4I_j(2I_j-1)} V_{j,ZZ}(\theta)$$

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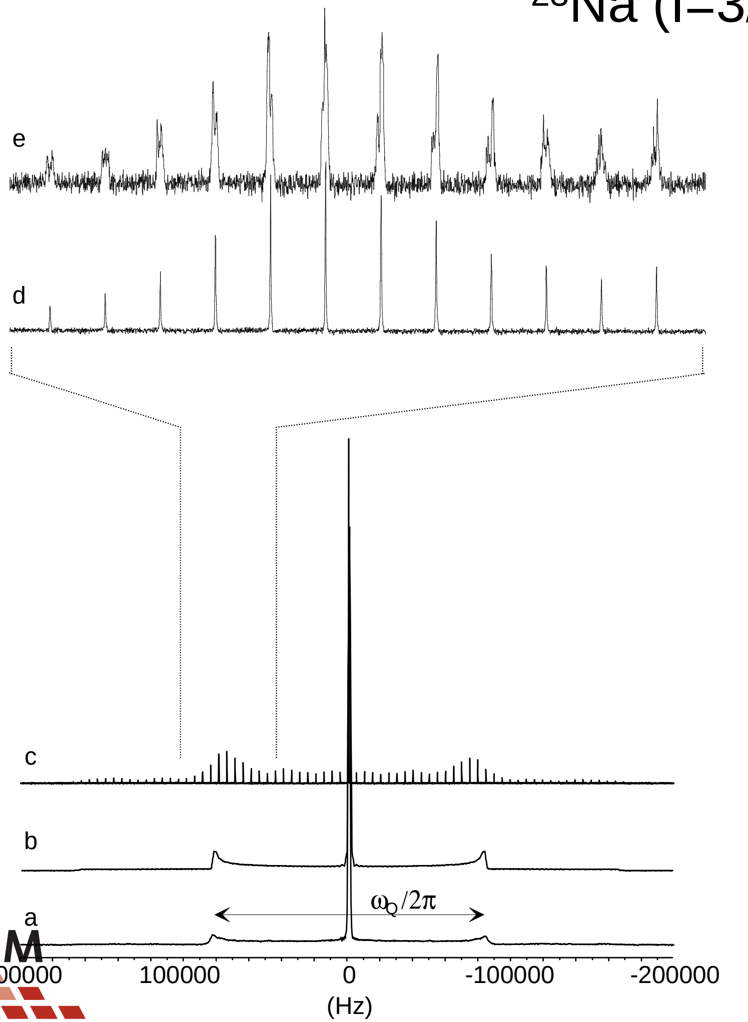
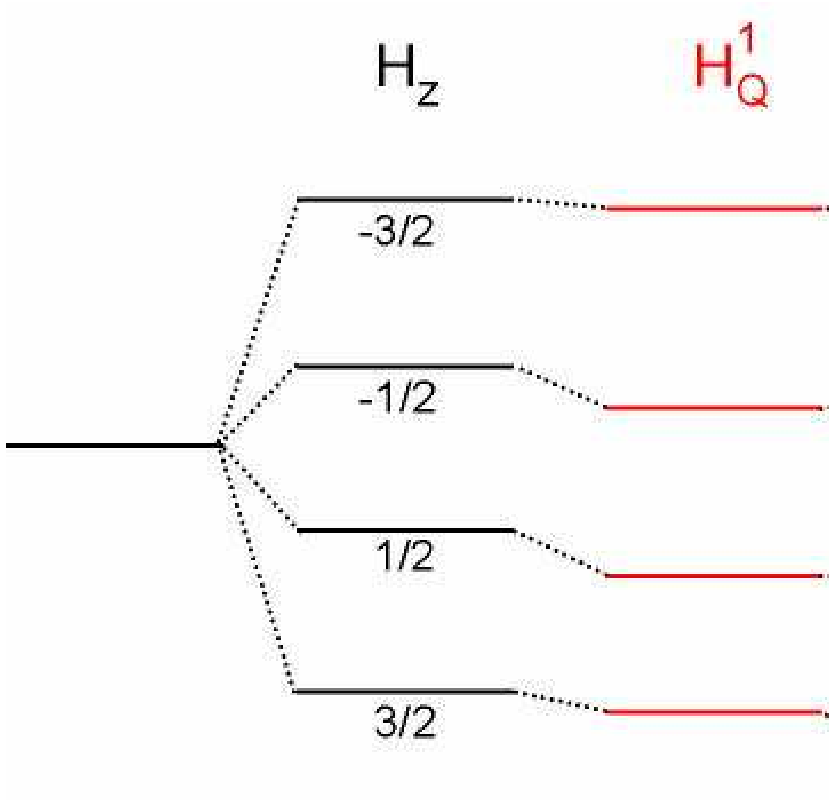
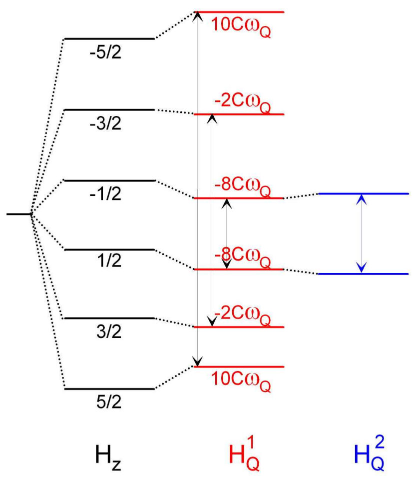
First order quadrupolar interaction

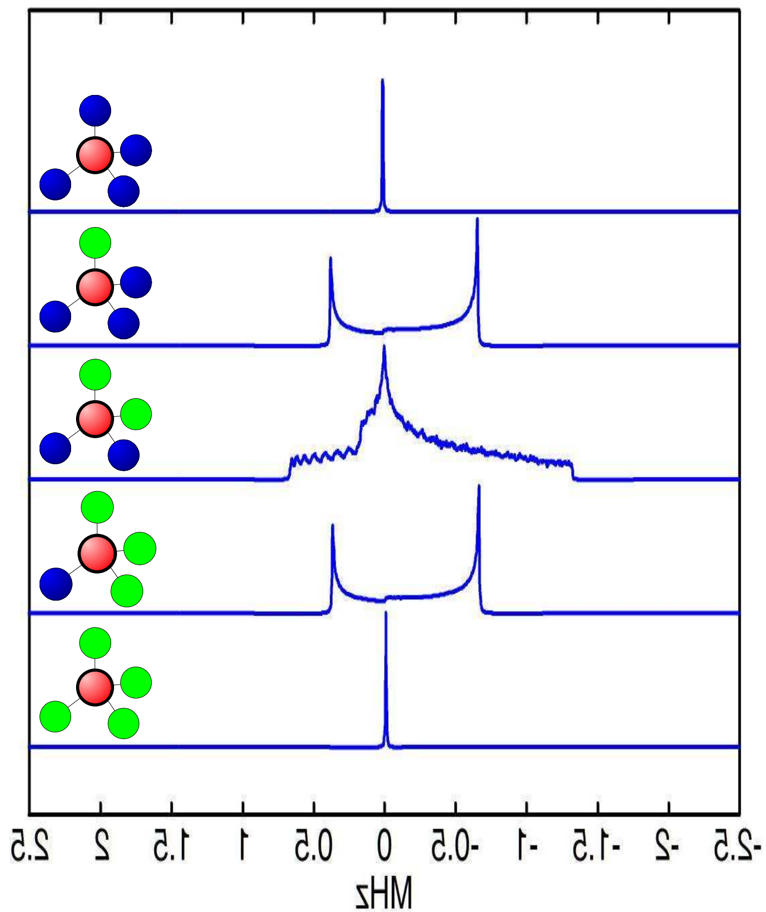
^{23}Na (I=3/2) in $NaNO_3$





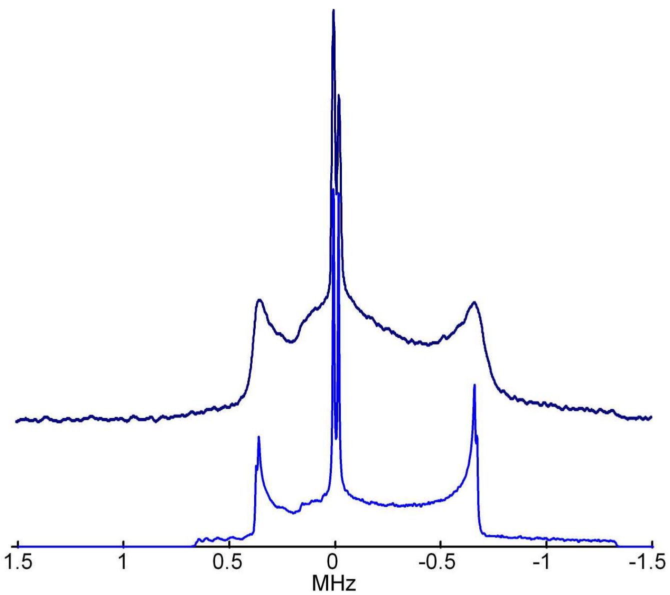
Second order quadrupolar interaction





As Coordinations in AlGaAs

^{75}As NMR @ 18.8T
$Al_{0.47}Ga_{0.53}As$



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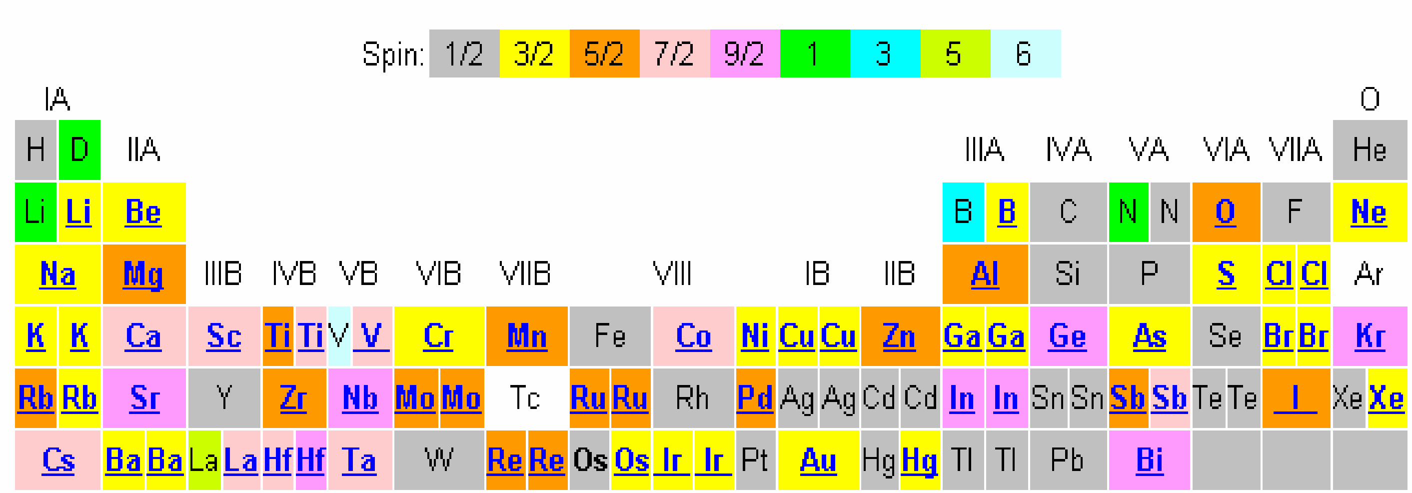
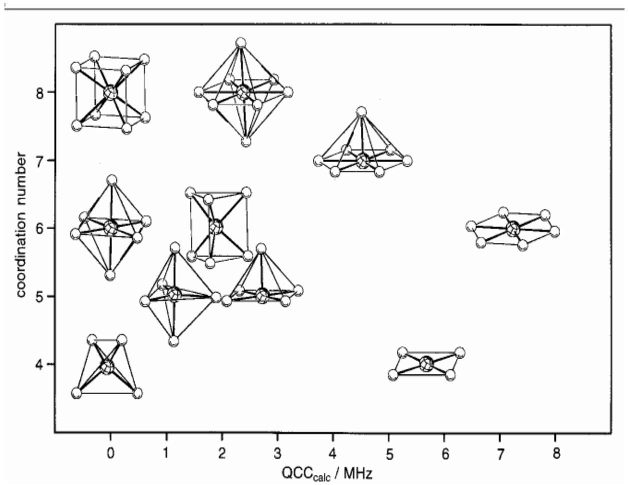
What Information Can NMR Give

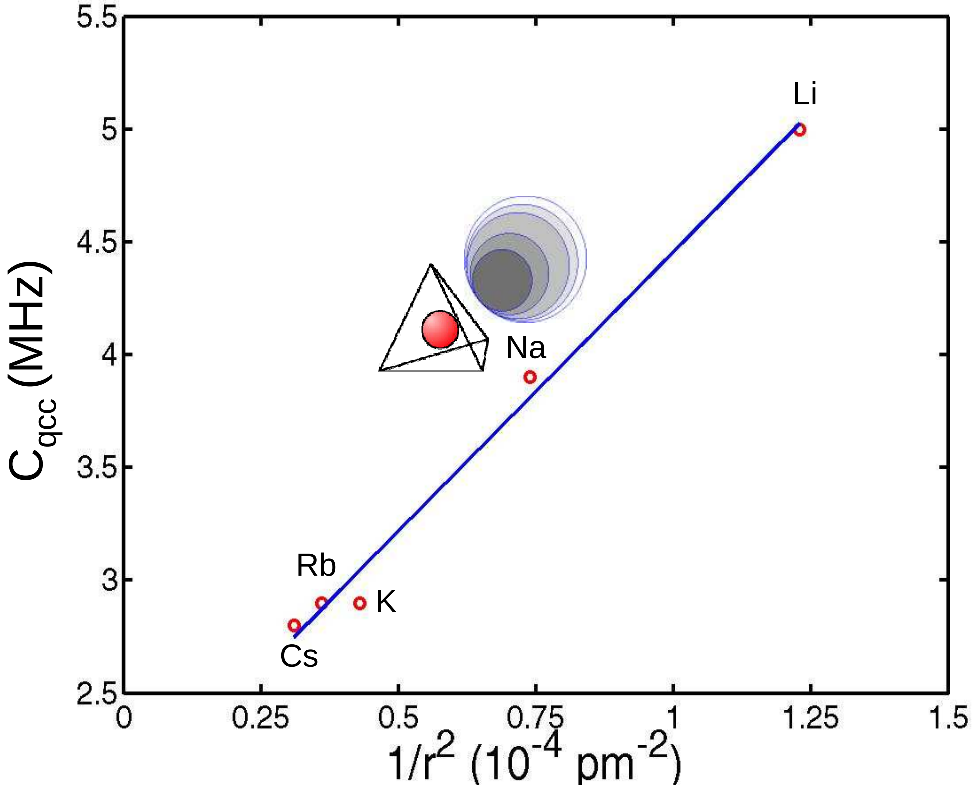
Site Identification

- *Quadrupolar Interaction*
  - ☞Determination of local symmetry (distortions).
  - ☞^{17}O NMR parameters are sensitive to H-bond formation.
  - ☞Majority of periodic table has I>1/2



Quadrupolar Interaction: Site Symmetry





Framework aluminosilicate glasses with varying charge-balancing cations (Li, Na, K, Rb, Cs)
Dirken, Nachtegaal and Kentgens, Solid State Nucl. Magn. Reson. 5 (1995) 189.

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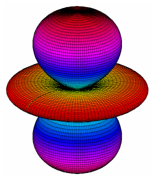
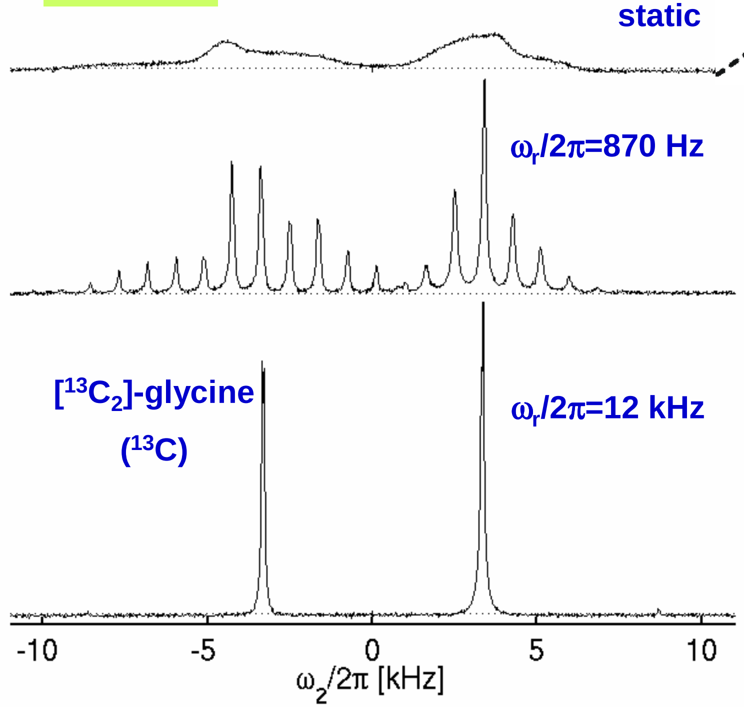
Tool: Magic Angle Spinning

averaged

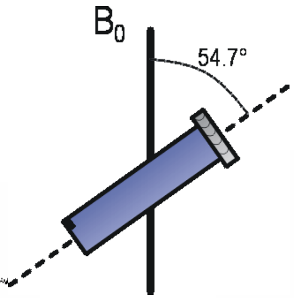


P_2

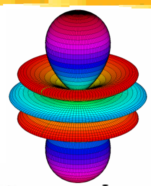
spin-½



fast magic-angle spinning

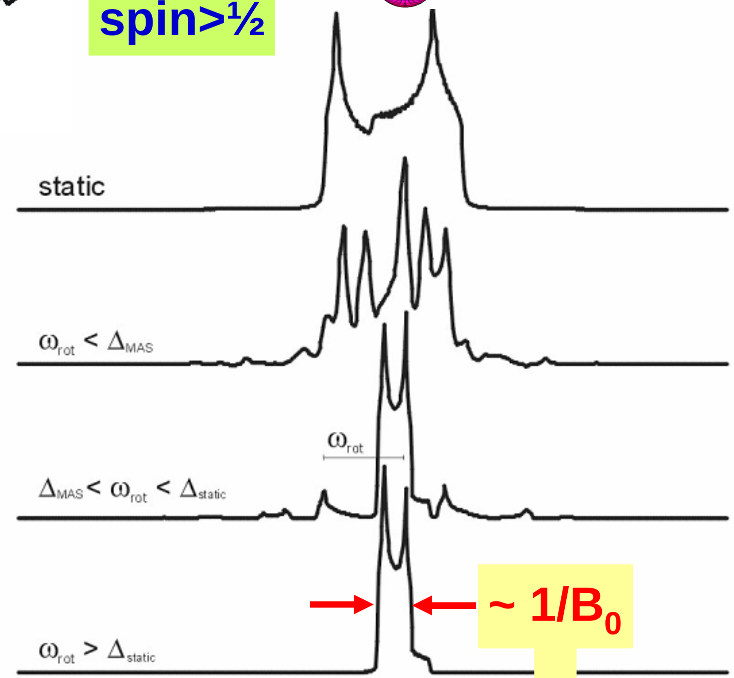


P_4



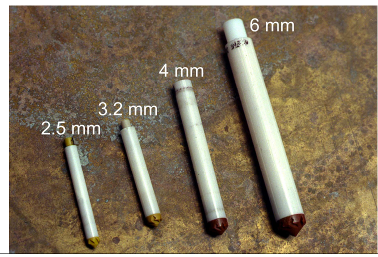
not averaged

spin>½



~ 1/B_0

Use high fields !



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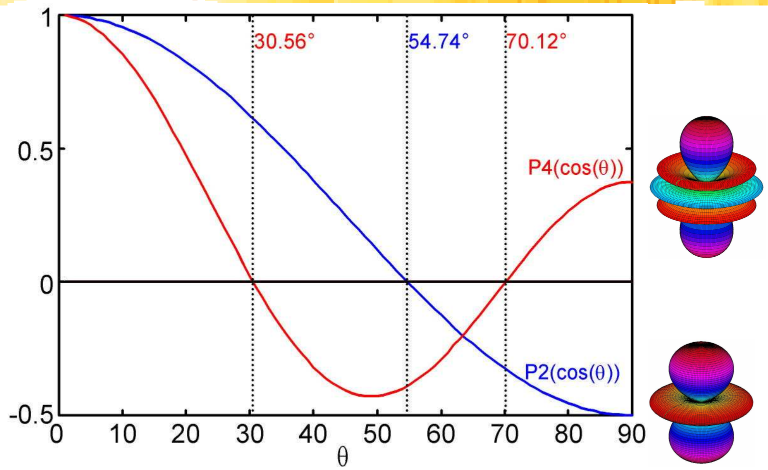
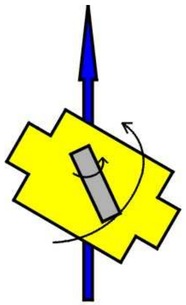
Tools: Double Rotation (DOR) Dynamic Angle Spinning (DAS)



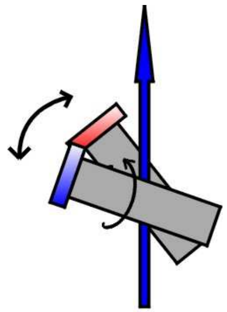
$$\upsilon_{m,-m} = C_0(I,m)\cdot\upsilon_0 + C_2(I,m)\cdot\upsilon_2(\alpha,\beta)\cdot\underline{P_2(\cos(\theta))} + C_4(I,m)\cdot\upsilon_4(\alpha,\beta)\cdot\underline{P_4(\cos(\theta))}$$



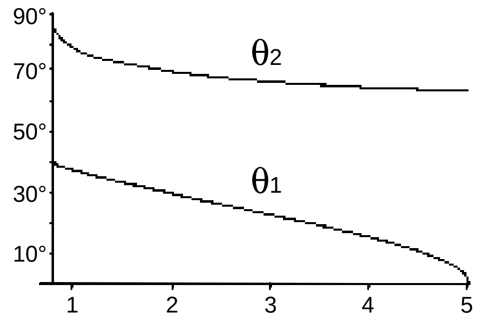
DOR

Mechanically involved

Dedicated Hardware needed



DAS



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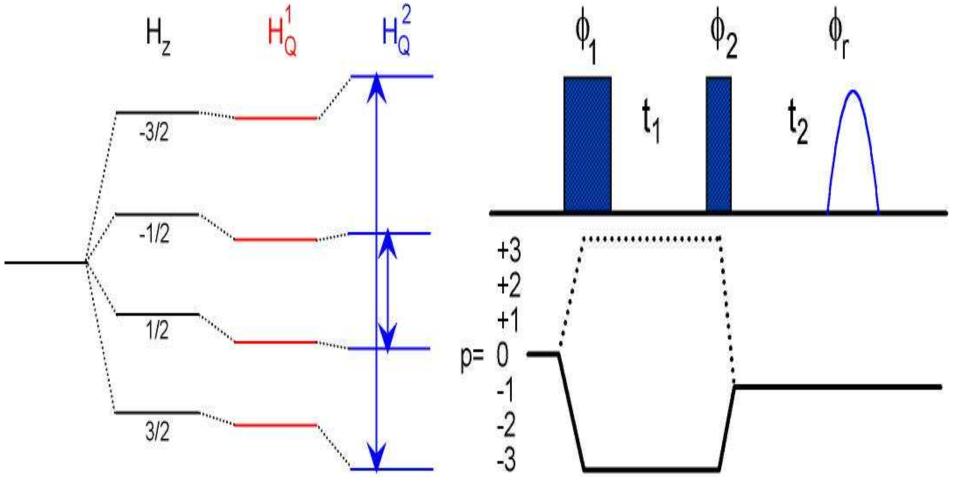
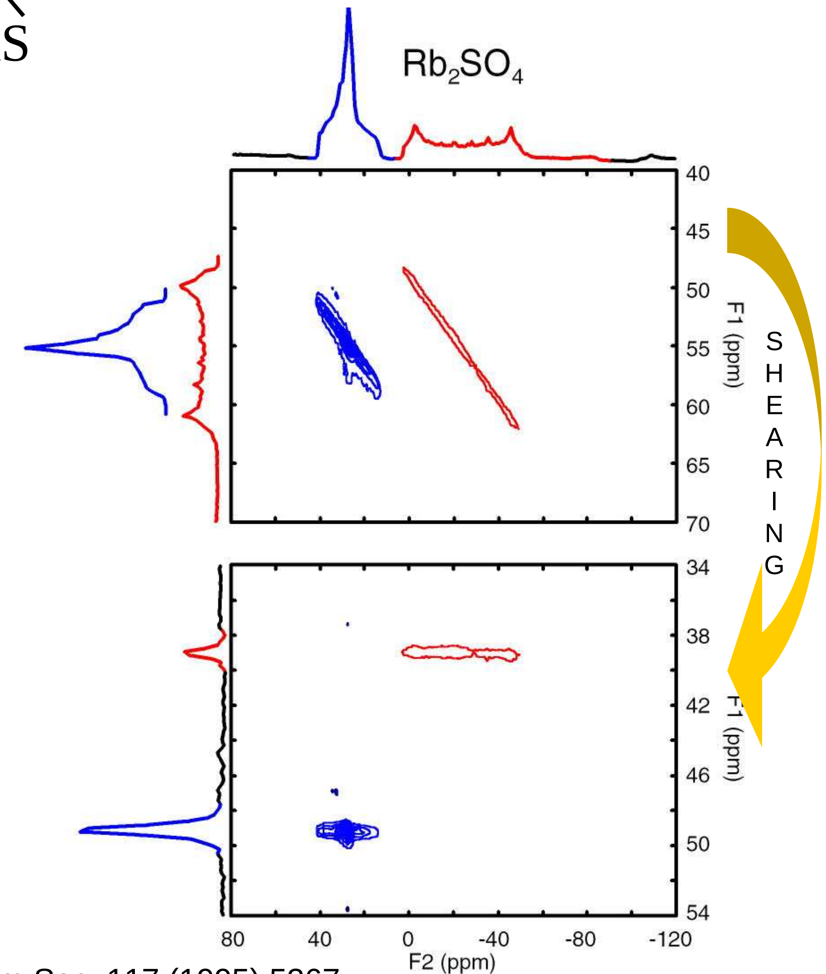
Samoson, Lippmaa and Pines, Mol. Phys. 65 (1988) 1013. / Llor and Virlet, Chem.Phys.Lett. 152 (1988) 248.
Mueller, Baltisberger, Wooten and Pines, J. Phys. Chem. 96 (1992) 7001.

Multiple-Quantum MAS

$$\upsilon_{m,-m} = C_0(I,m)\cdot\upsilon_0 + C_2(I,m)\cdot\upsilon_2(\alpha,\beta)\cdot P_2(\cos(\theta)) + C_4(I,m)\cdot\upsilon_4(\alpha,\beta)\cdot P_4(\cos(\theta))$$

MAS





Sensitivity issue (multiple-quantum excitation and conversion)

Frydman and Harwood, J.Am.Chem.Soc. 117 (1995) 5367

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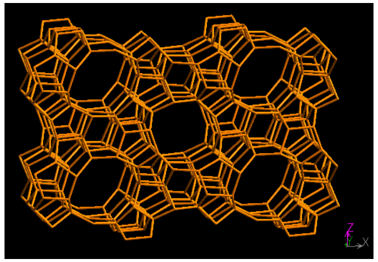
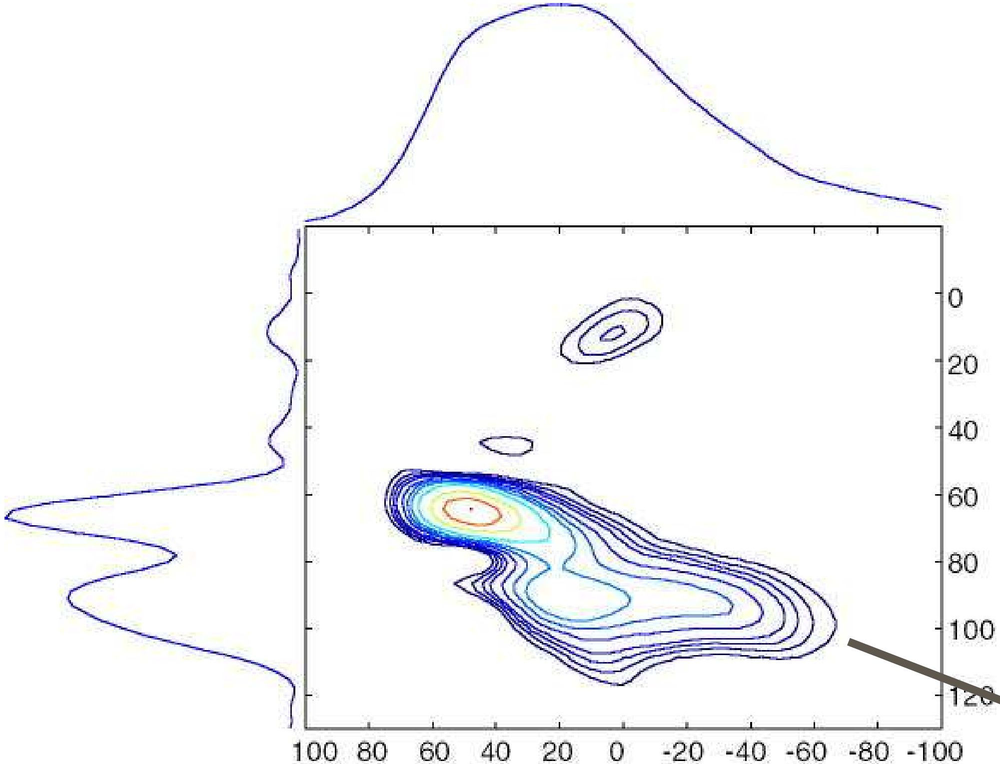
Dehydrated H-ZSM5



^{27}Al MQMAS



☑High Field (14.1 T)

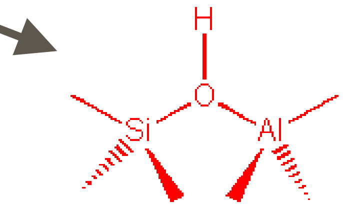
☑Fast MAS (27 kHz)

☑MQ→1Q DFS conversion

$\overline{\sigma}_{iso} = 55\,ppm$

$\overline{C}_{qcc} = 16.4\,MHz$

$\eta = 0.1$



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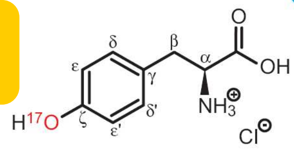
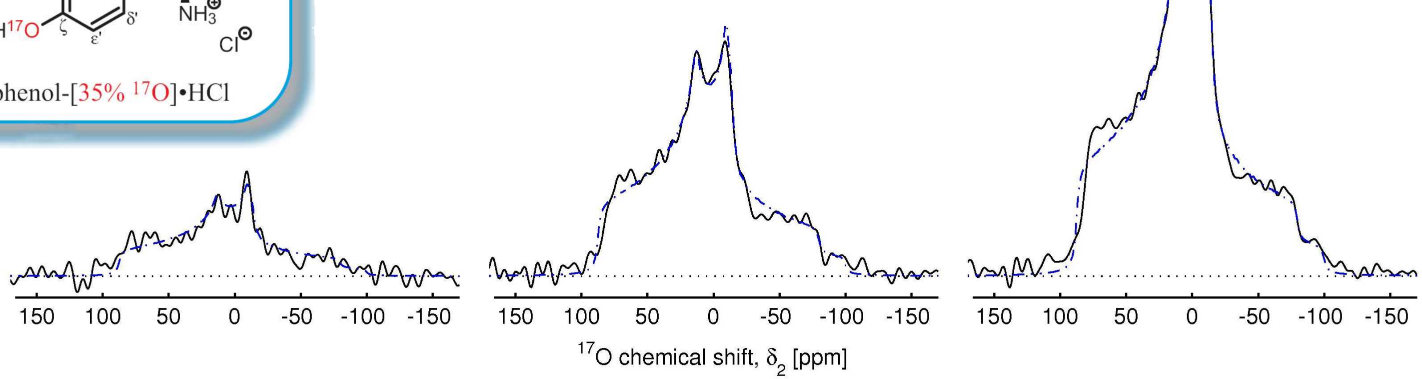
Kentgens, Iuga, Kalwei and Koller, J.Am.Chem.Soc. 123 (2001) 2925.

rDFS ^{17}O MAS NMR

Experiments:
δiso= 87 ppm
C_{QCC}= 8.52 MHz
η=0.74

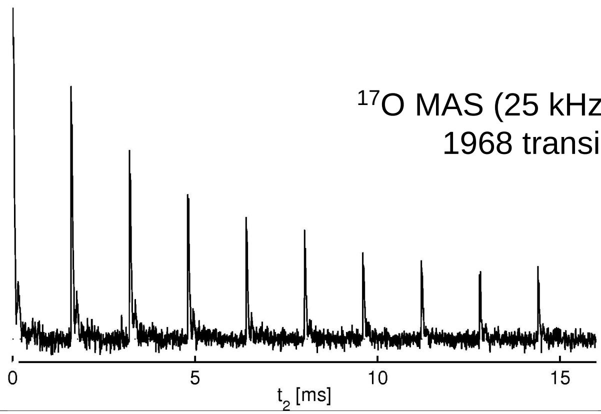


L-tyrosine-phenol-[35% ^{17}O]•HCl

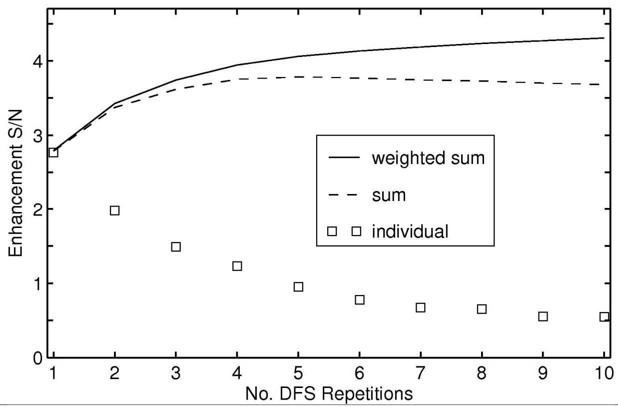


^{17}O challenging because of low γ and large C_Q

~**4.3 S/N Enhancement** for biologically relevant material



^{17}O MAS (25 kHz) @ 14.1T
1968 transients



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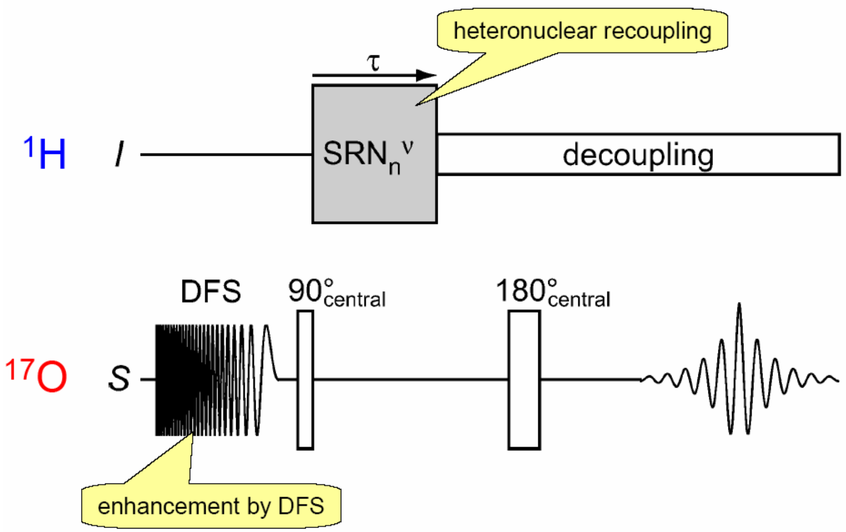
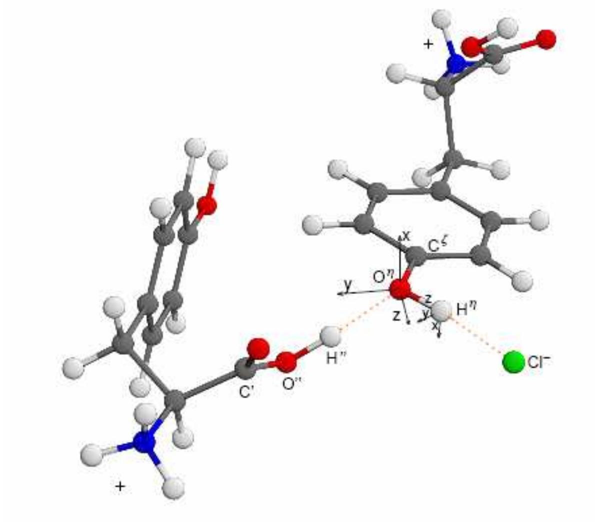
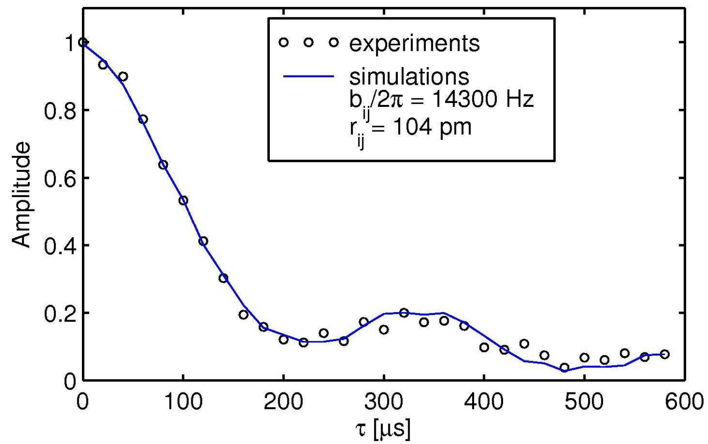
Heteronuclear Recoupling: ^{17}O-^{1}H distance measurement







r_{OH} distance 104 pm is within 5% of the distance determined by neutron diffraction (99 pm).

O-H libration slightly averages dipolar interaction.

A. Brinkmann & A.P.M. Kentgens
J. Phys. Chem. B 110 (2006) 16089

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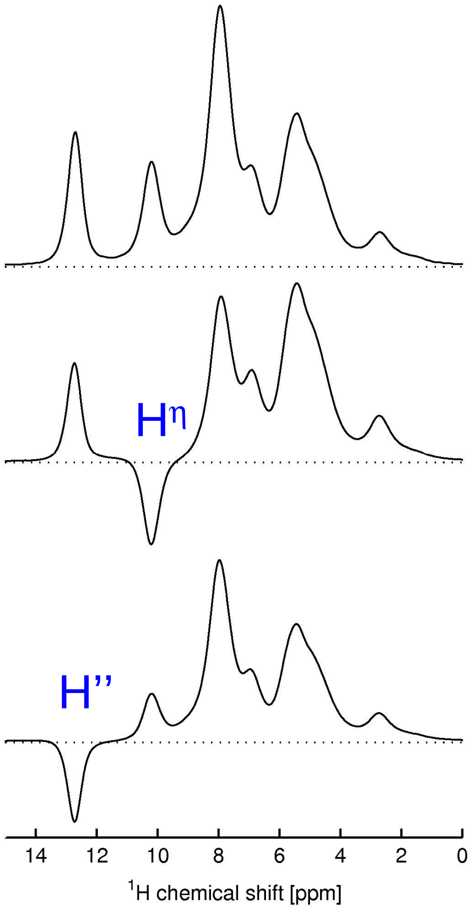
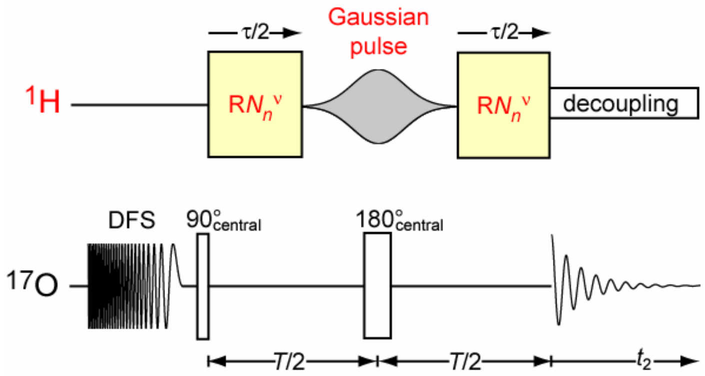
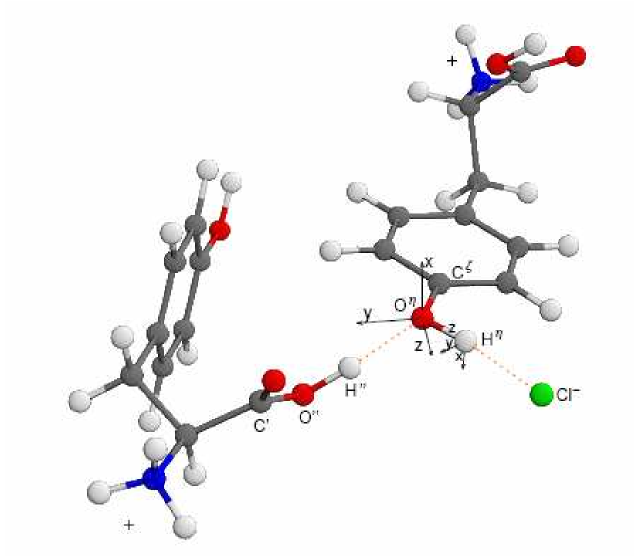
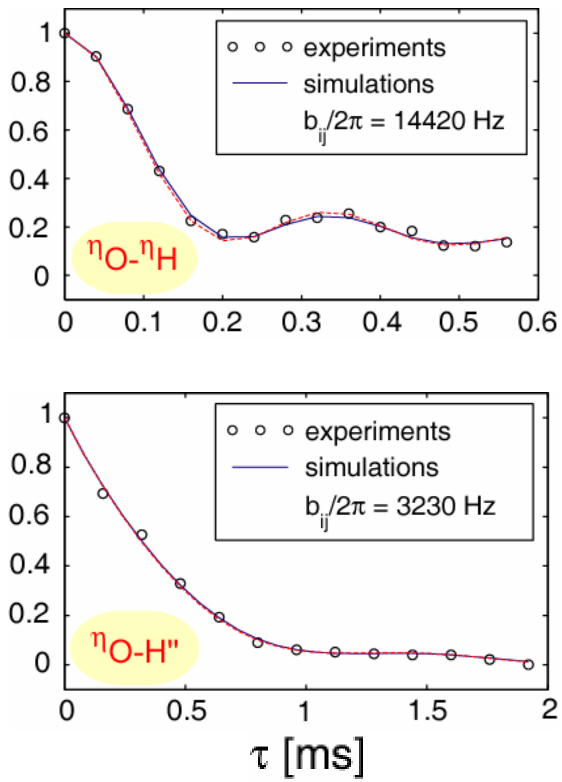
Heteronuclear Recoupling: ^{17}O-^{1}H distance measurement









$r_{O\text{-}H''}$ distance 161 pm

A. Brinkmann & A.P.M. Kentgens
J. Am. Chem. Soc. 128 (2006) 14758

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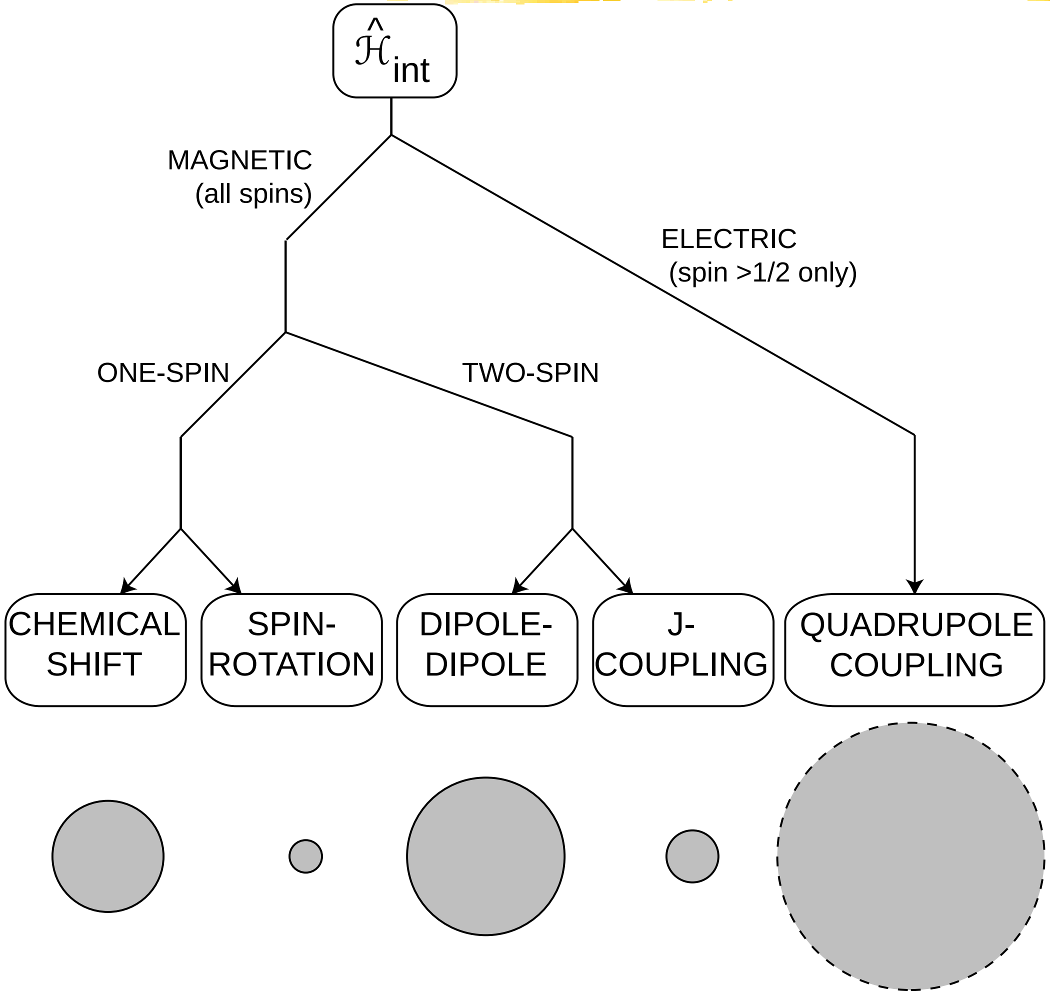
Summary of Internal Hamiltonians



Malcolm H. Levitt, *Spin Dynamics*, Wiley, 2001

Conclusions

- Solid state NMR is a powerful analysis technique
  - Probes microscopic interactions (1-100Å)
  - Study structure and dynamics
  - Works in crystalline, partly disordered and amorphous compounds
  - Non-destructive technique needing no special sample preparation
- Novel methodological developments will open new applications in advanced materials science

Sensitivity enhancement is driving methodological developments

Options for signal enhancement:	Potential gain
• *Double B_0*	*3*
• Cryo-cooled rf coils	3
• *Population transfer in coupled or quadrupolar spin systems*	*2-5*
• *Low temperature MAS*	*10*
• *Microcoil detection*	*100*
• Dynamic Nuclear Polarization (DNP)	10^3
• Optical polarization (ODMR / OPMR)	10^4
• Hyper polarized Xe, He, Kr	10^4
• Para-Hydrogen	10^4
• *Force detection*	*10^3-10^6*

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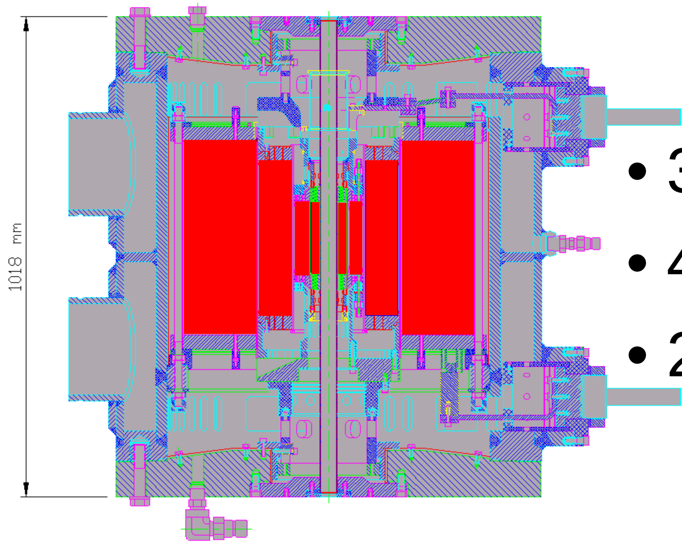
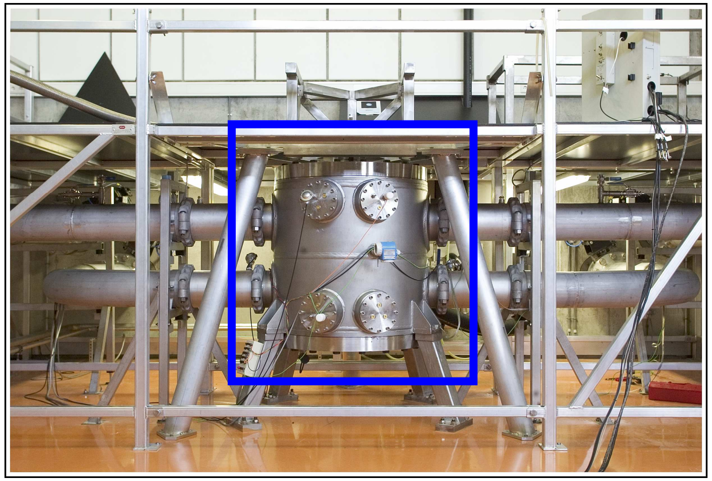
SSNMR Beyond 1 GHz



- 33 T
- 40 kA
- 20 MW



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Opportunities and Problems

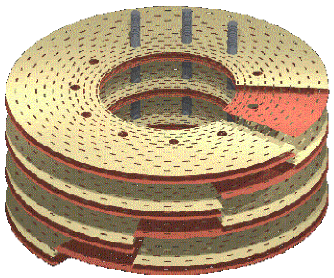
Opportunities

- Sensitivity (~$B^{7/4}$)
- Resolution (~B - B^2)
- High speed (proton) MAS
- Quadrupolar nuclei

Problems

- Intrinsic homogeneity (~10^{-3}/cm)
- Temporal stability (~10^{-5})
  - power supply
  - temperature and flux changes
- Operation time

- Ferro-shims
- High speed MAS
- Reference deconvolution
- Follow-B



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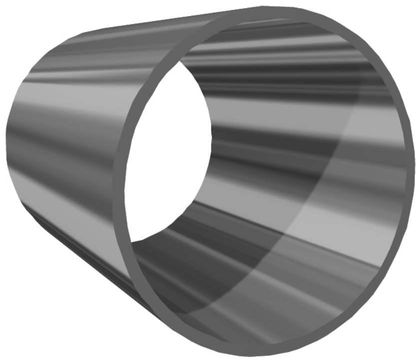
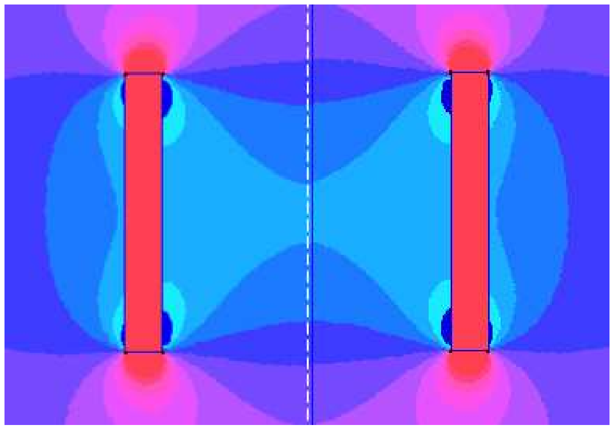
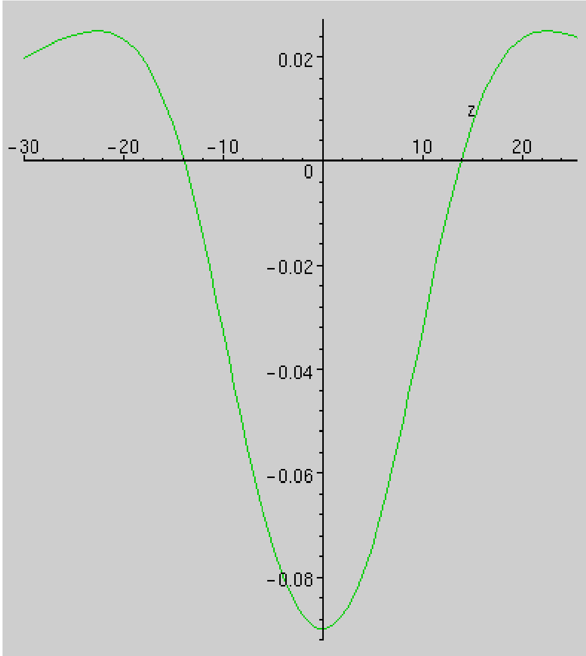
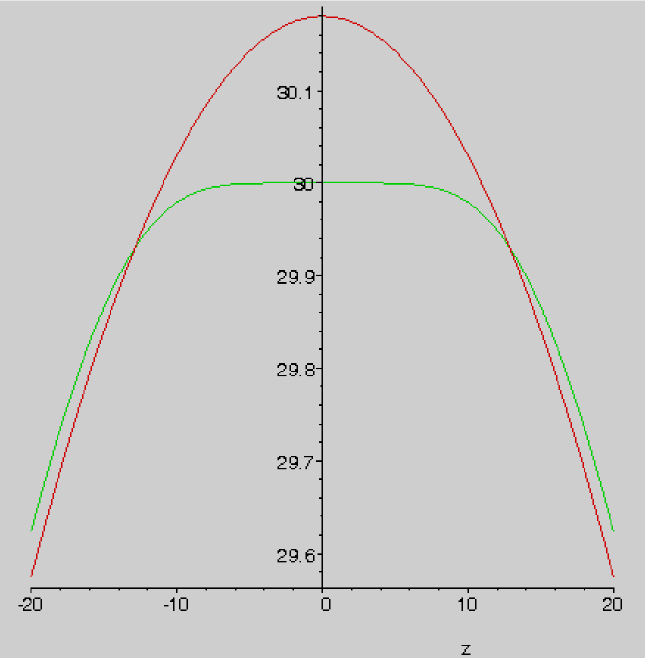
Field profile of a uniformly magnetized cylinder







Field profile
Ferroshim



Magnet field
profile before and
after Ferroshim

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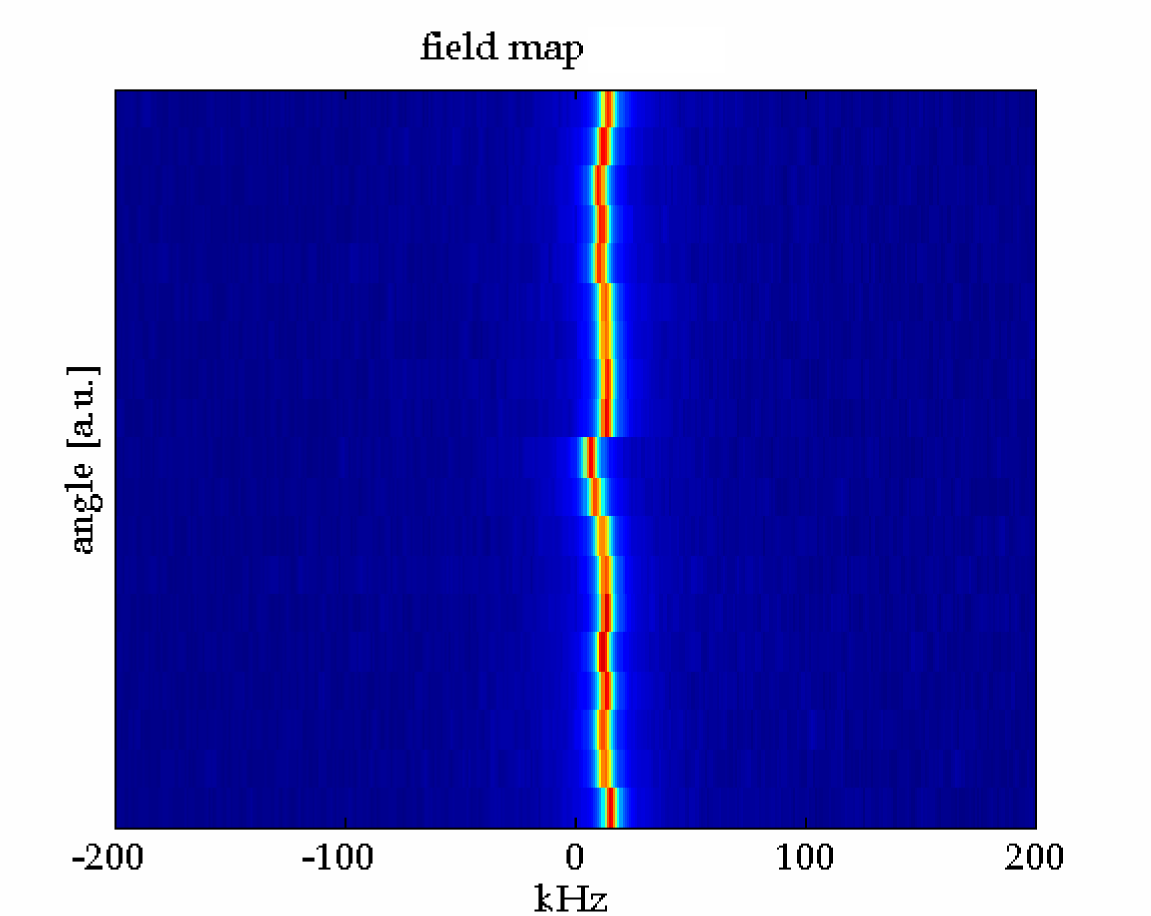
Shifting the shim off-axis allows reduction of radial gradients
Magic Angle Spinning averages residual gradients

Field map with ferroshim

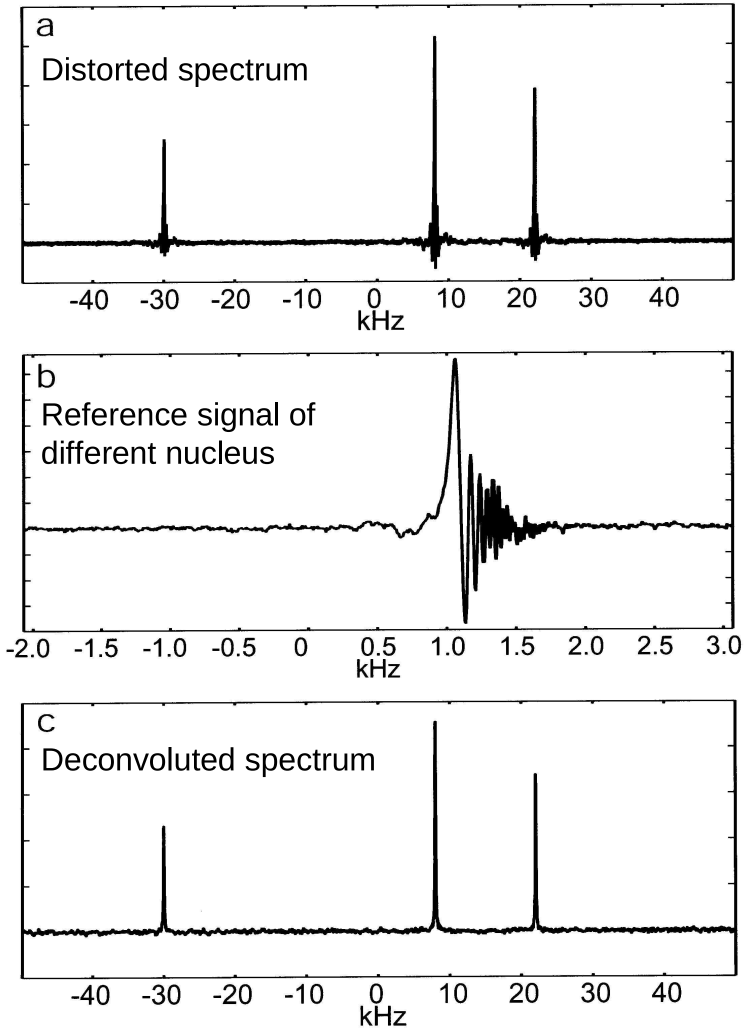


Field map D_2O, after x-y optimization ferroshim
Field x-y gradient < 5 kHz (drift dominated) <25 ppm/cm

Reference Deconvolution

$$c(t) = \left(\frac{S_{ideal-ref}(t) \times W(t)}{S_{exp-ref}(t)} \right)^{\frac{\gamma_I}{\gamma_S}}$$

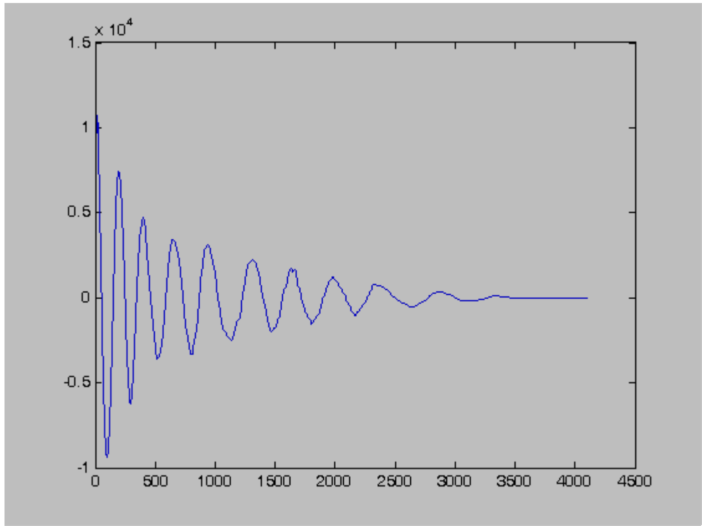
$$S_c(\omega) = FT[S_{exp}(t) \times c(t)]$$

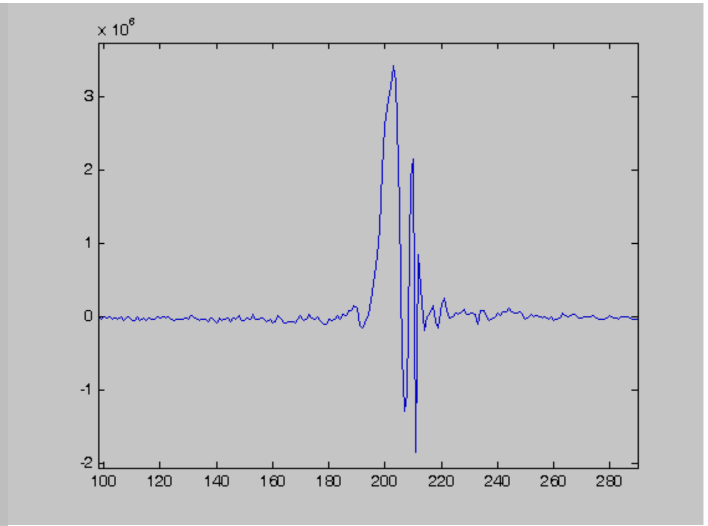


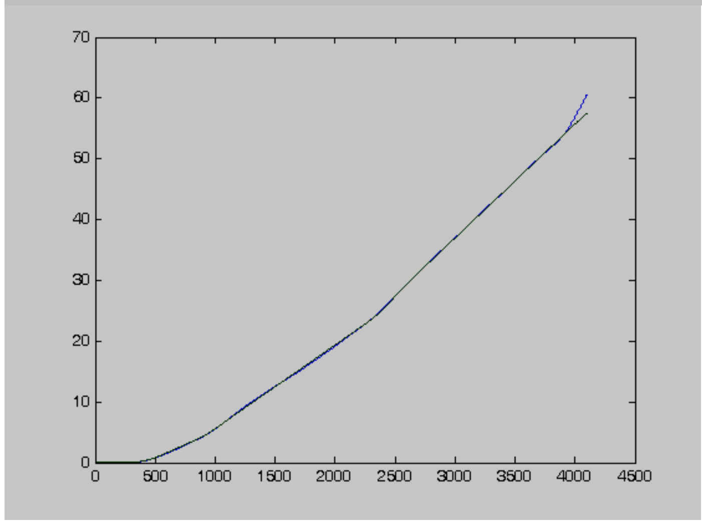
Morris, Barjat and Horne, PNMRS 31 (1997) 197-257.

Metz, Lam and Webb, Concepts Magn. Reson. 12 (2000) 21-42.

Reference Deconvolution







- Discrete field steps
- MAS: resolution is stability limited
- Fourier spectrum distorted (chirp)
- Length of FID determines resolution

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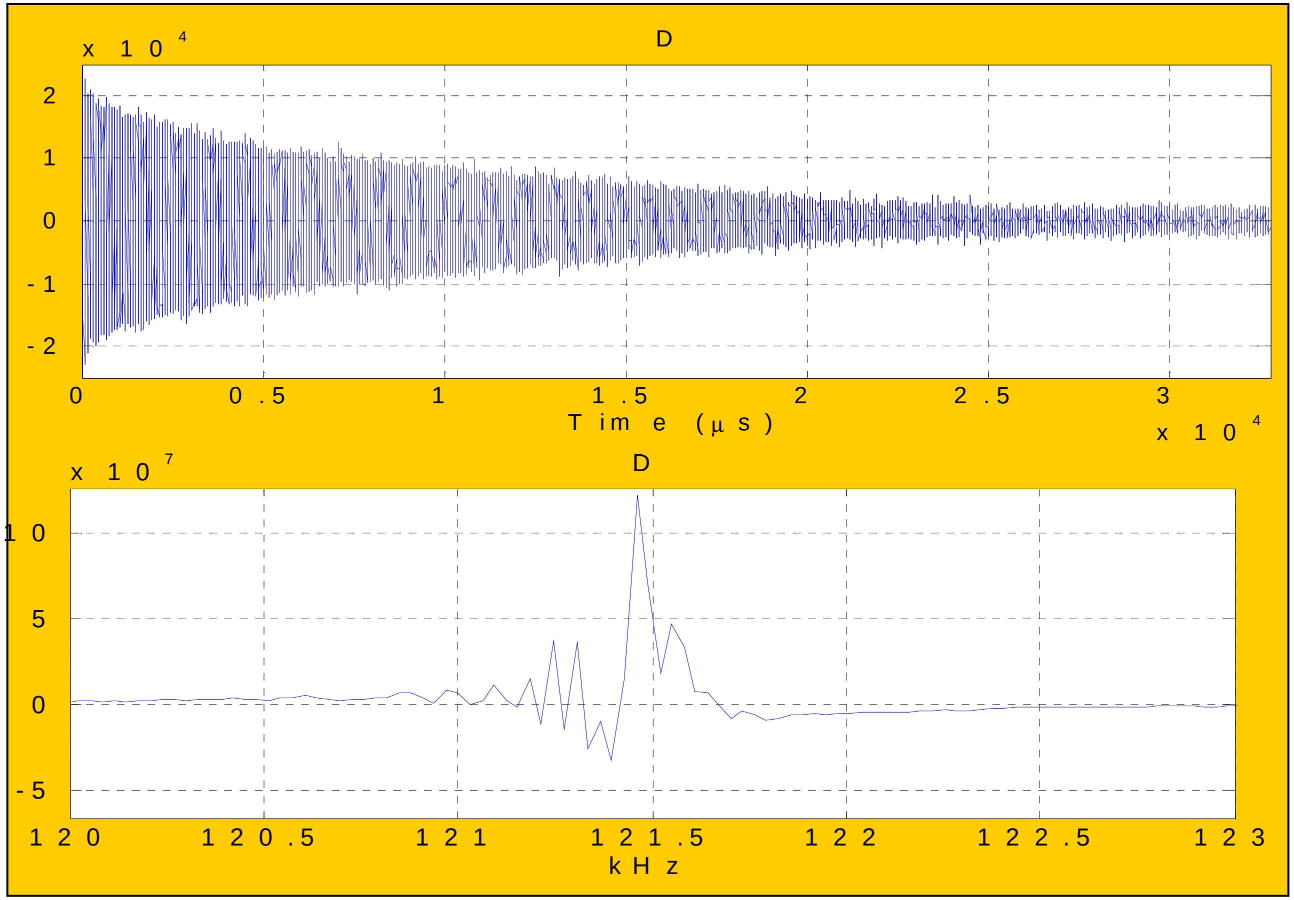
Triple-tuned MAS probe
^{2}D reference channel

Jan van Bentum
Ernst van Eck
Jan van Os

Probe technology:
Ago Samoson



ND_4Cl reference signal lasts 30 - 50 msec,
i.e. intrinsic homogeneity of about 0.15 ppm.

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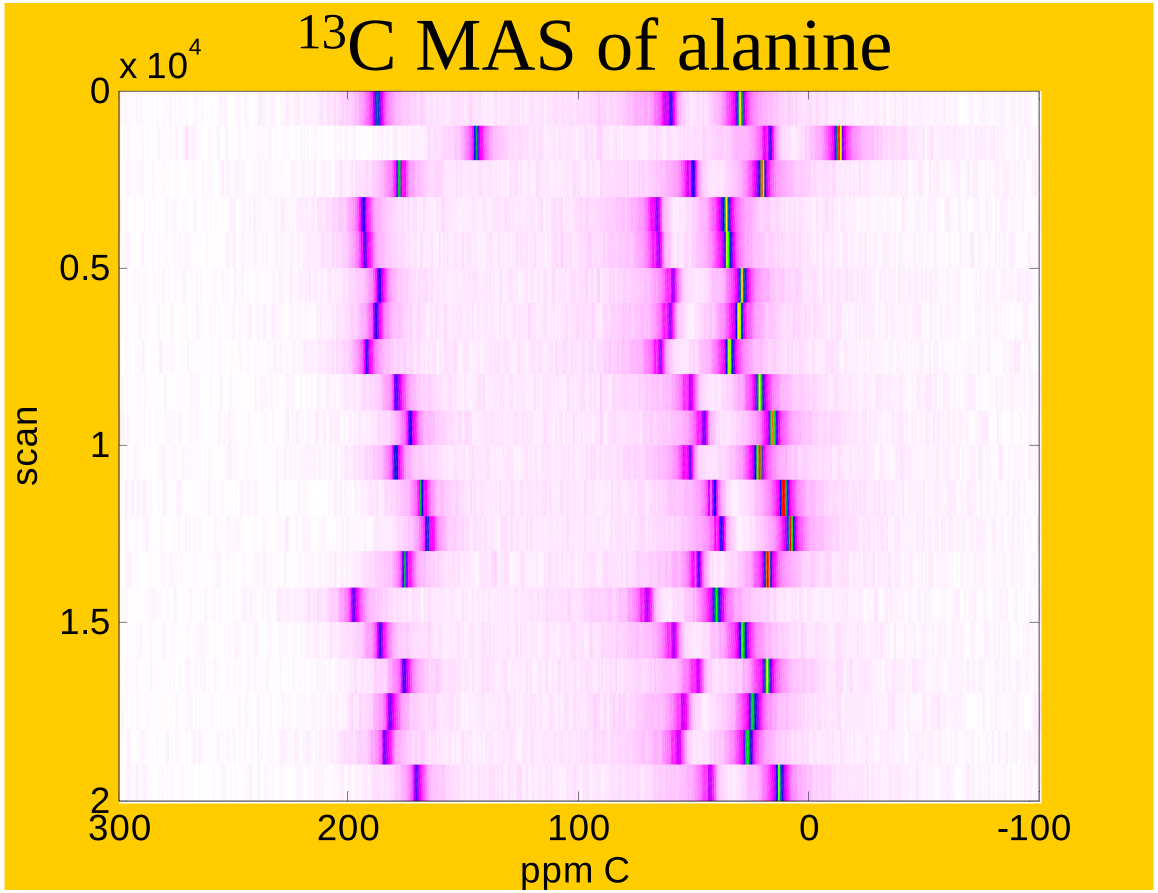
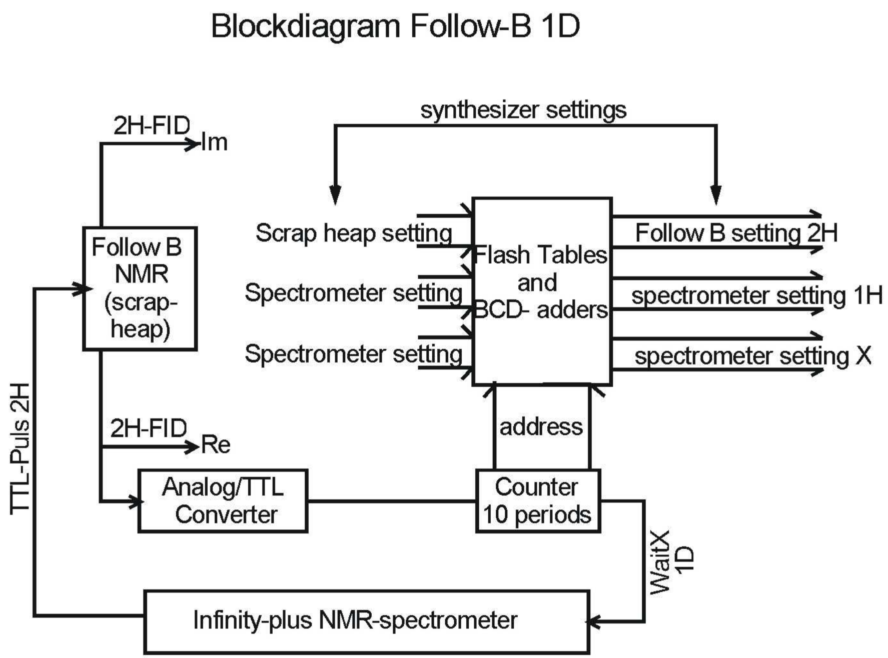
Field Stability



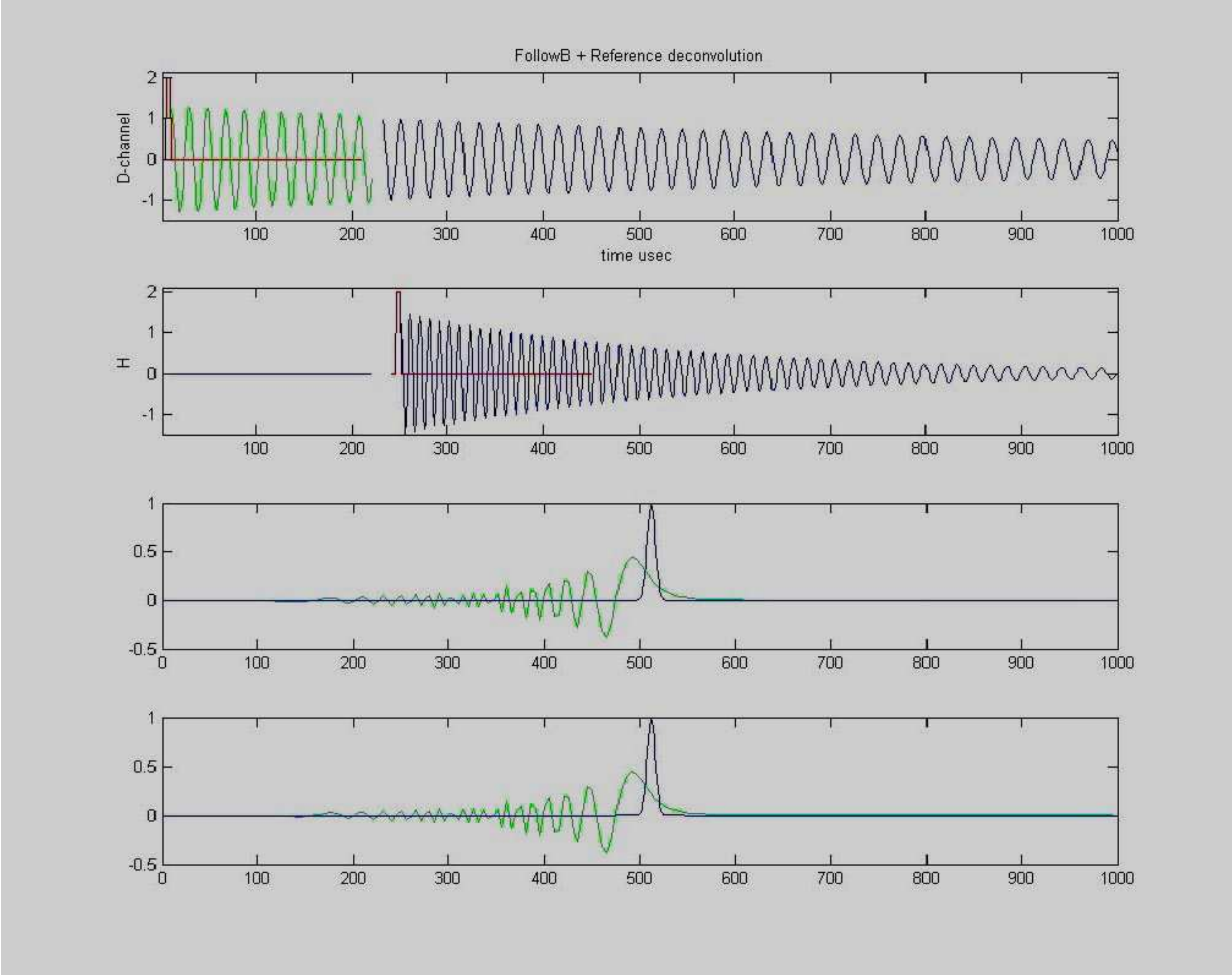
Field stability 10-50 ppm on ^{13}C

©Follow B option

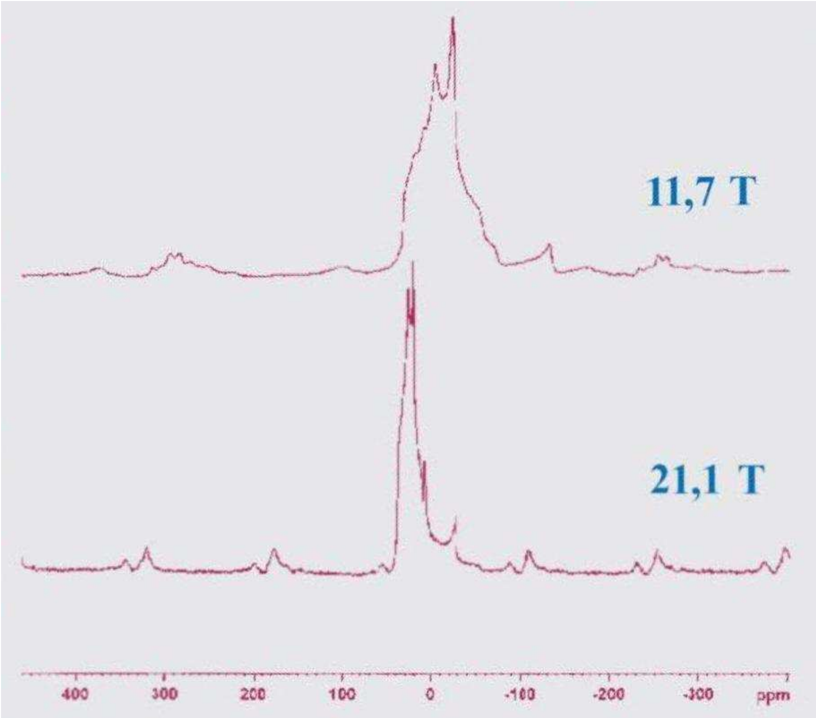


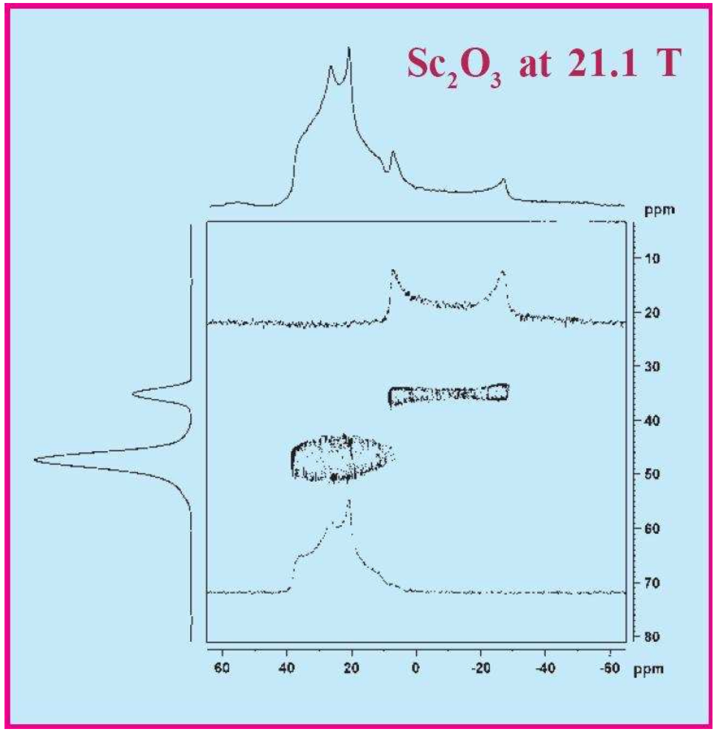
- Use first 200 μsec of reference FID to determine field
- Reset spectrometer frequencies
- Use remaining part of ref-FID for deconvolution

Follow B + reference deconvolution



Bruker News and Events
AVANCE 900, first results





At 21.1 T separation of the two Sc sites is nearly achieved…

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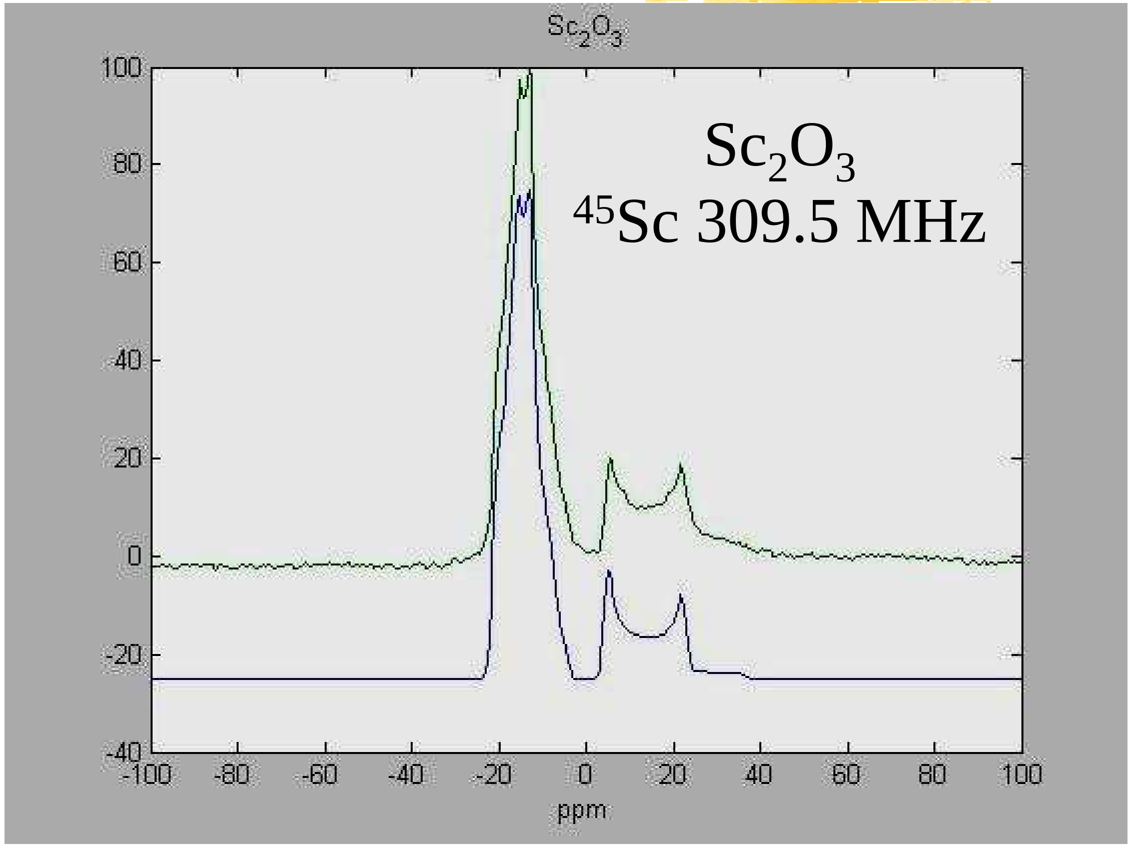
Systems with large quadrupolar interactions



64 scans
MAS 38 kHz
Fixed phase
Averaging
=> followB
=> Ref. dec.

1: C_q=15.4 MHz η_Q=0.61
2: C_q=23.4 MHz η_Q=0.10

Intensity ratio
exp 1:2.995
theor 1:3

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Hydrogen storage

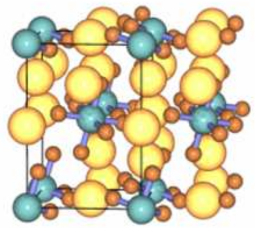
- Important technical issues are weight, volume, discharge/recharge rates, reaction heat, safety and cost
- IEA (International Energy Agency) targets:
  - at least 5-10 wt.%
  - H_2 recoverable at < 80℃
  - Loading/unloading at 1 atm absolute pressure.
- Solid H_2 storage
  - (Complex) Metal hydrides, like $NaAlH_4$, $NaBH_4$, $LaNi_5H_6$.

$$NaAlH_4 \leftrightarrow Na_3AlH_6 + 2\ Al + 3\ H_2$$

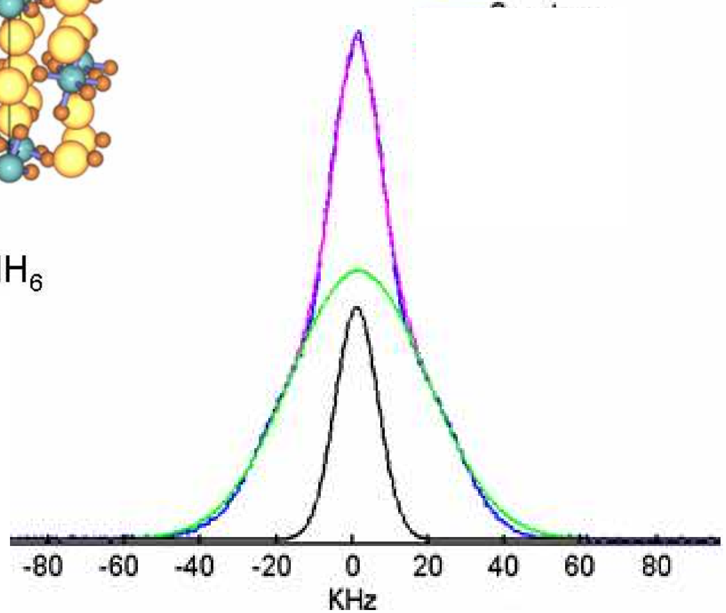
$$Na_3AlH_6 \leftrightarrow 3\ NaH + Al + 3/2\ H_2$$

Static [1]H NMR on Ti-doped $NaAlH_4$

- Partly release of H_2 -> $NaAlH_4$ and Na_3AlH_6 are present
- Hahn-Solid-Hahn Echo to avoid spectral distortions
- Two fractions with different relaxation times T_1 and different line widths.
- Na_3AlH_6: Narrowing of the line shape -> proton mobility in the crystal -> fast rotating AlH_6 clusters



Na_3AlH_6



$NaAlH_4 \leftrightarrow Na_3AlH_6 + 2\ Al + 3\ H_2$

$Na_3AlH_6 \leftrightarrow 3\ NaH + Al + 3/2\ H_2$

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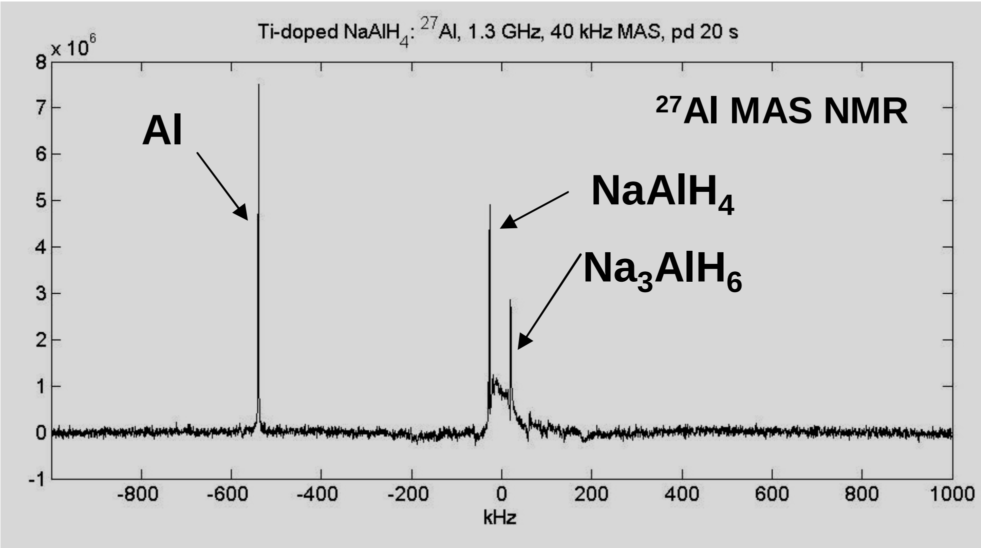
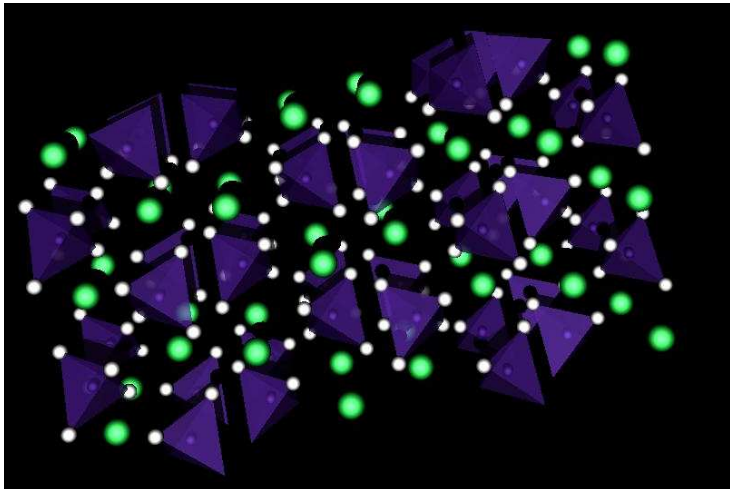
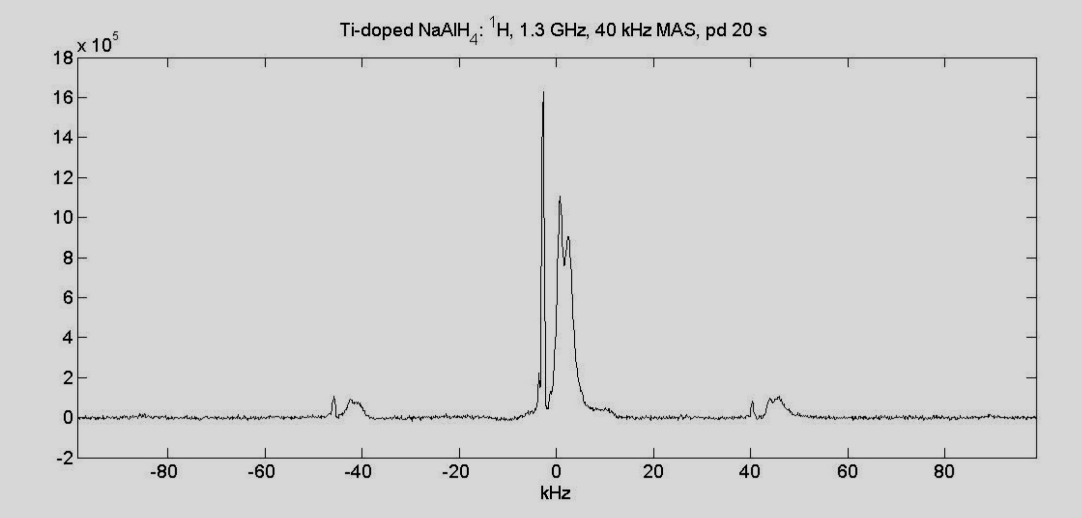
Margriet Verkuijlen, Jan van Bentum

^{1}H and ^{27}Al high-speed (40 kHz) MAS of Ti-doped Alanates at 30 T





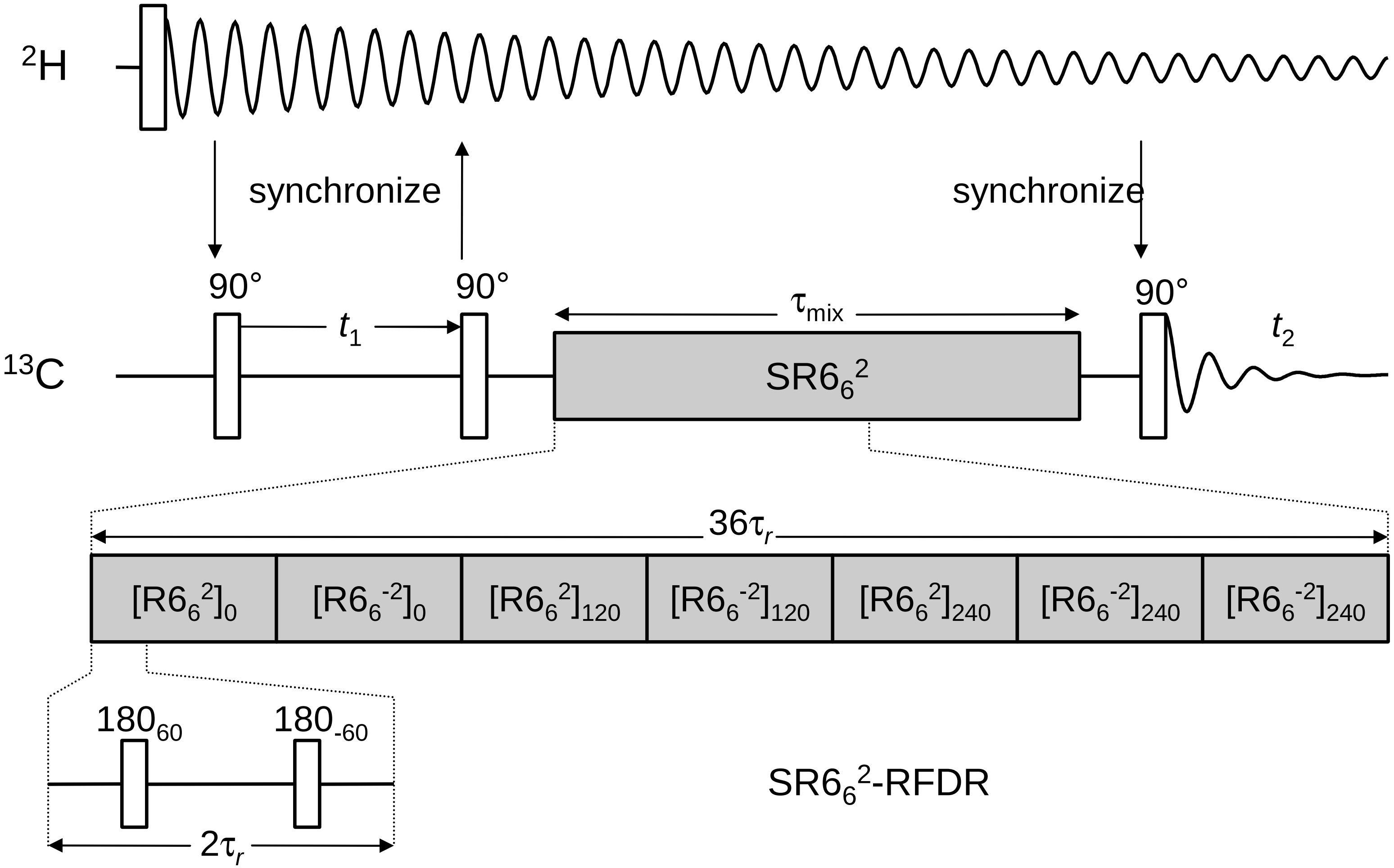


$NaAlH_4 \leftrightarrow Na_3AlH_6 + 2\ Al + 3\ H_2$

$Na_3AlH_6 \leftrightarrow 3\ NaH + Al + 3/2\ H_2$

M. Verkuijlen, E. van Eck, J. van Bentum,
B. Dam (Free University of Amsterdam)
C. Baldé, K. de Jong (Utrecht University)

Broadband homonuclear recoupling



$SR6_6^2$-RFDR

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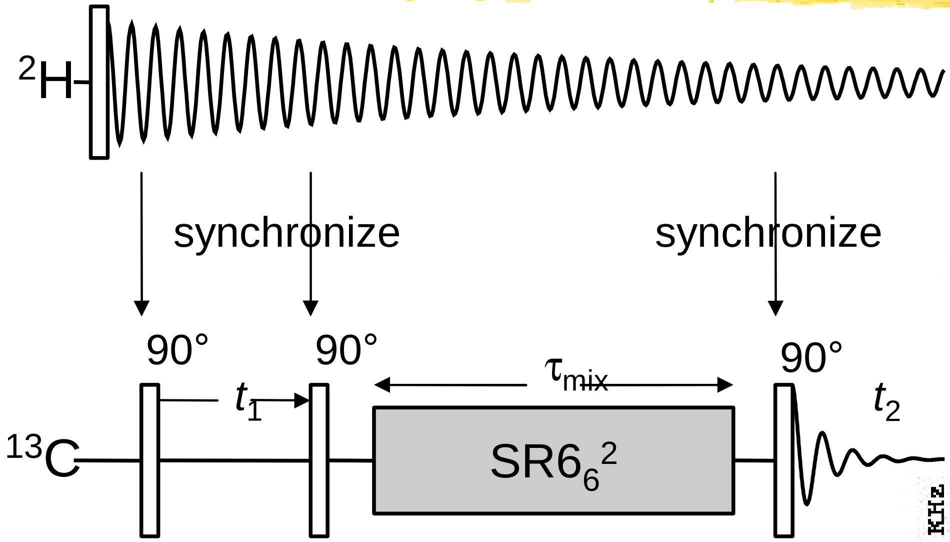
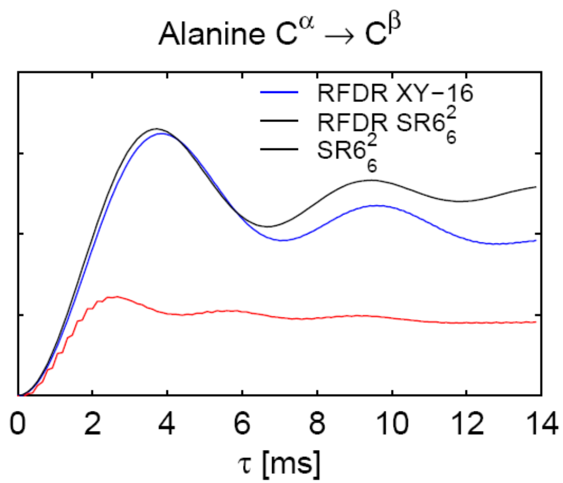
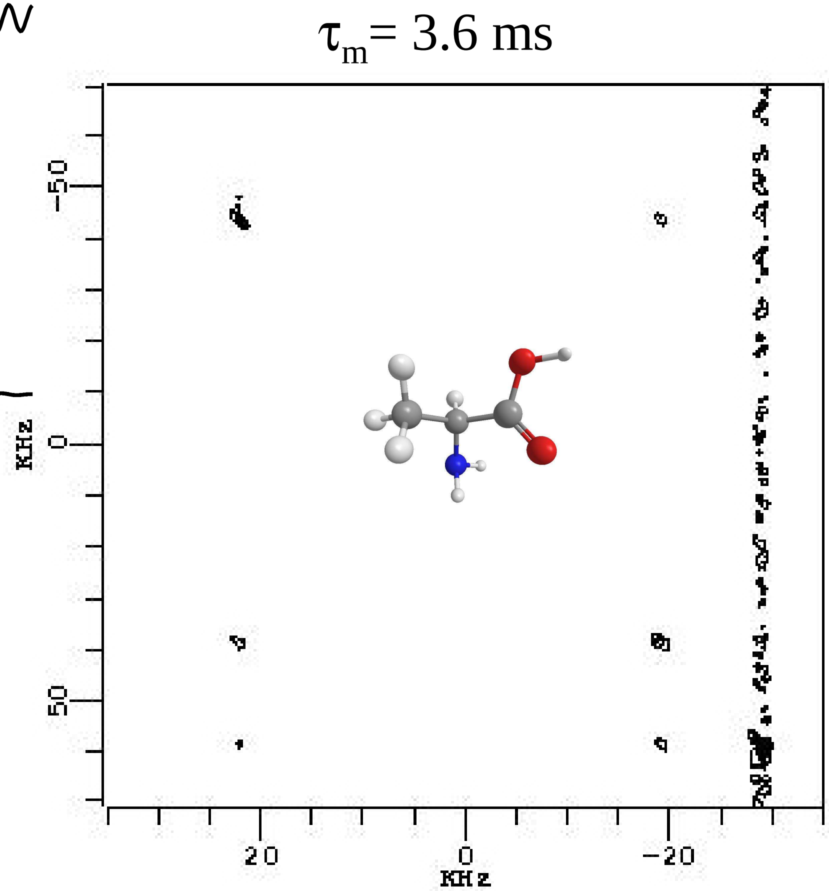
Andreas Brinkmann, Jorge Garibay

^{13}C-^{13}C homonuclear correlation ^{13}C labeled L-alanine







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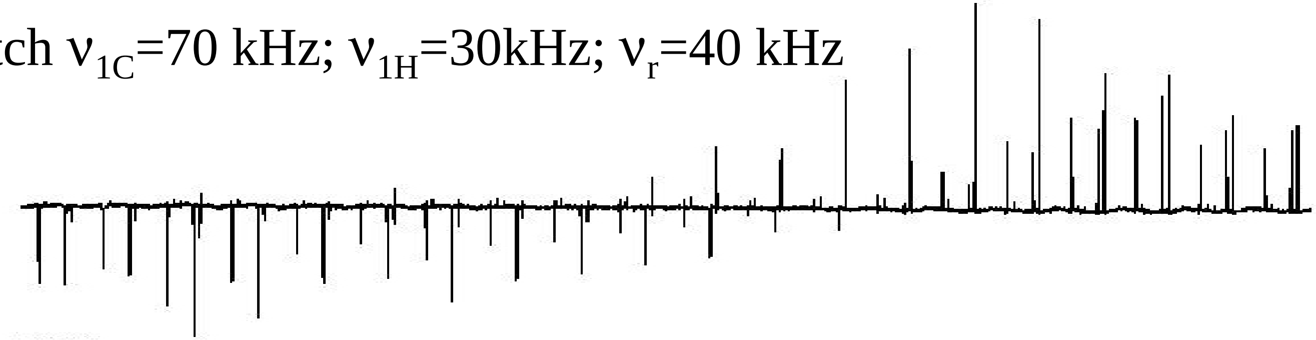
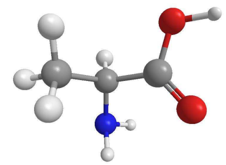
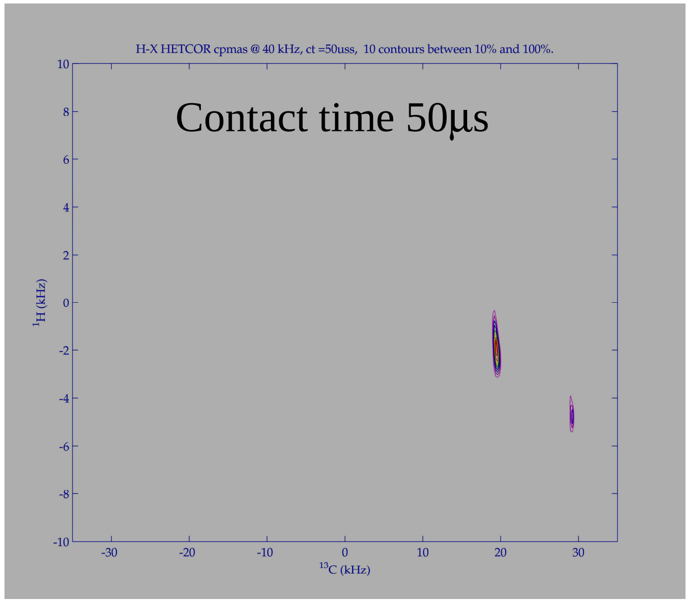
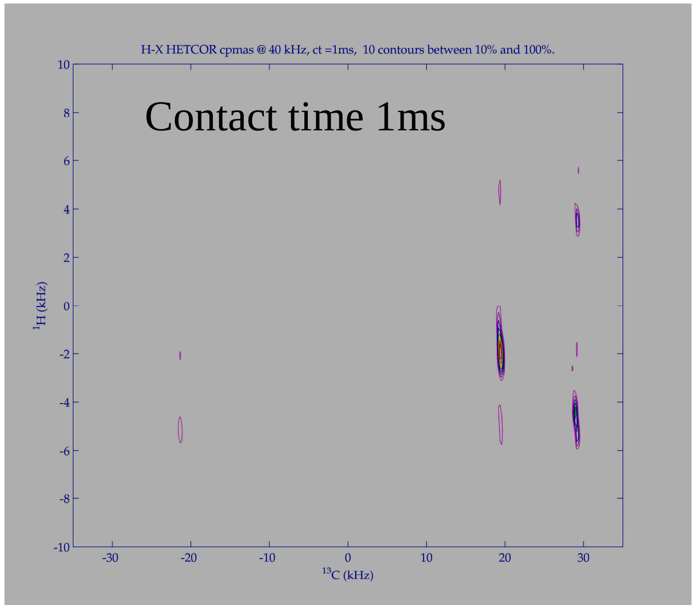
^{13}C-^{1}H heteronuclear correlation of L-alanine

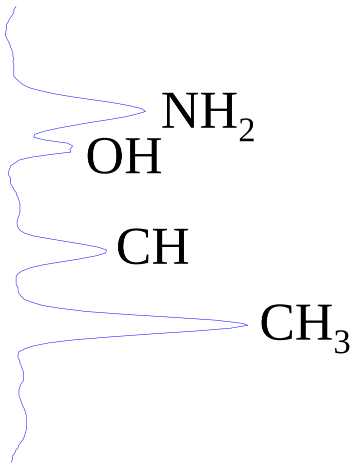
CP match ν_{1C}=70 kHz; ν_{1H}=30kHz; ν_r=40 kHz











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Conclusion High Field NMR

- Using a combination of hardware solutions and NMR tricks, one- and two-dimensional solid-state NMR at 30 Tesla is feasible.
- Quadrupolar systems with either very large or very small quadrupolar interactions
- High resolution proton NMR

Acknowledgements

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