Slow MAS Methodologies Towards to Radioactive Materials???

EURACT 2010

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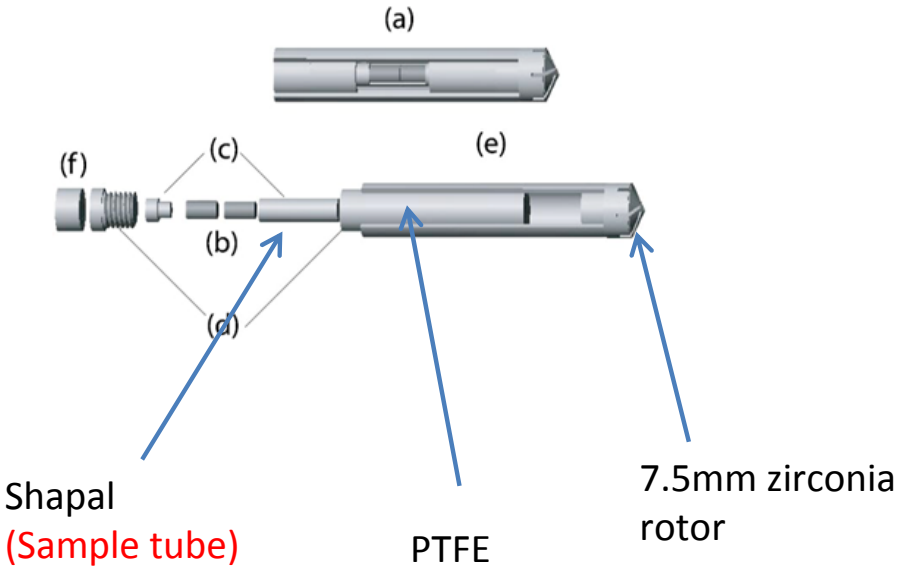
Safety Precaution for MAS Experiments

I Farnan, H Cho, WJ Weber et al. Rev Sci Instr 75, 5232 (2004)

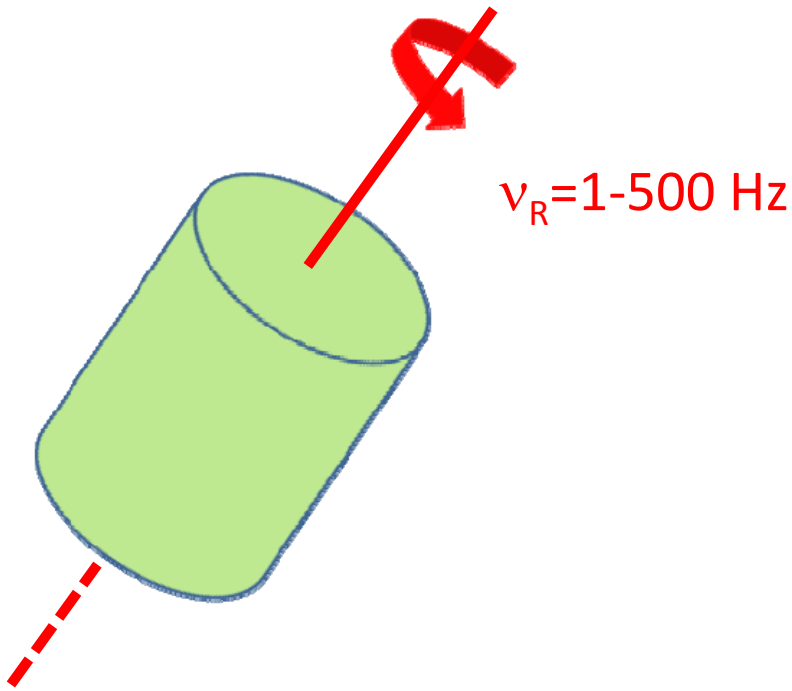
Triple-containment MAS System



Prevent leakage especially during sample spinning !!

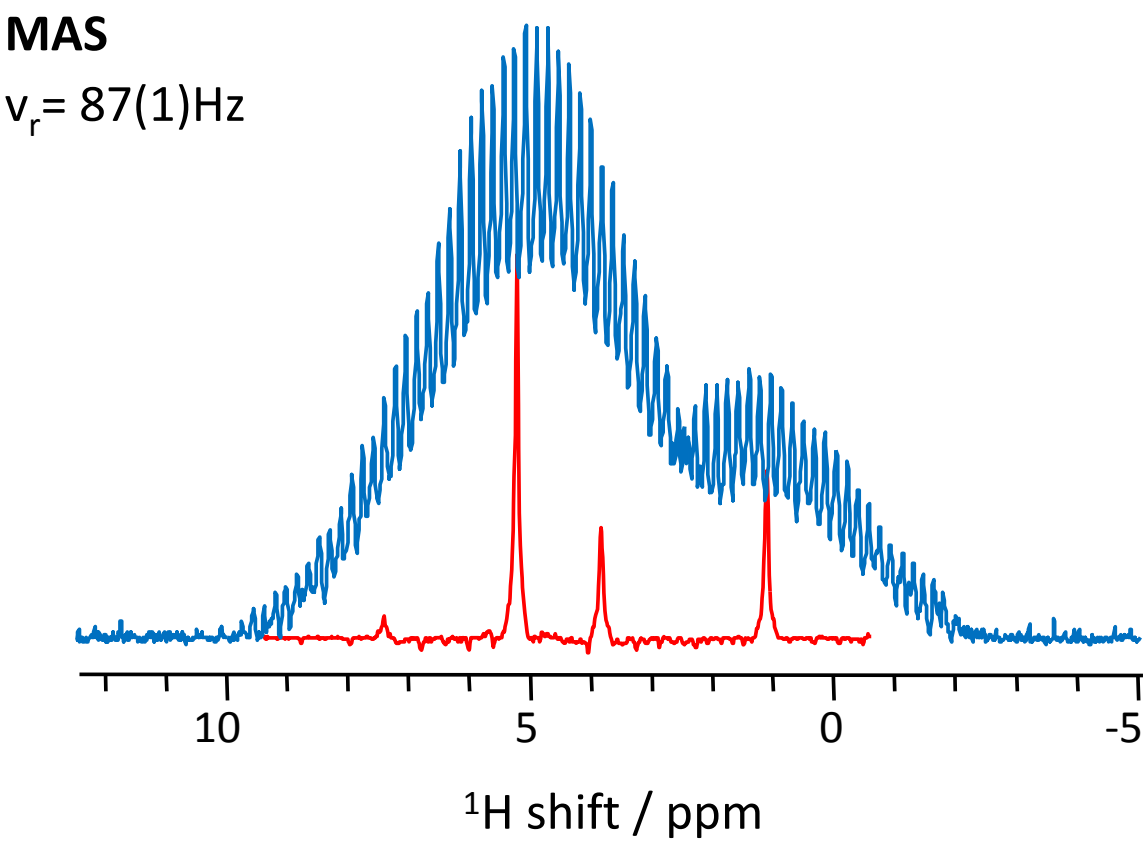
(1) 'SLOW' Magic-Angle Spinning

- Minimized centrifugal force ($F=m\nu^2r$) from spinning
- Enhanced safety
- Lessen sample invasion
- High-resolution



Problem with Slow MAS Spectroscopy

Ethonal/D_2O with Glass Bleads

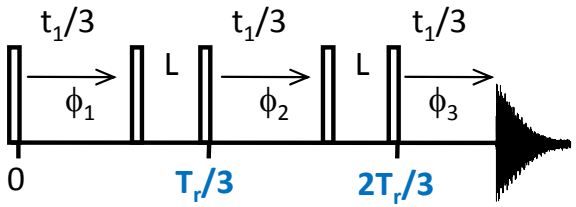


Tool Kits for High-Resolution Slow MAS

MAH: A Bax et al JMR 52, 147 (**1983**)

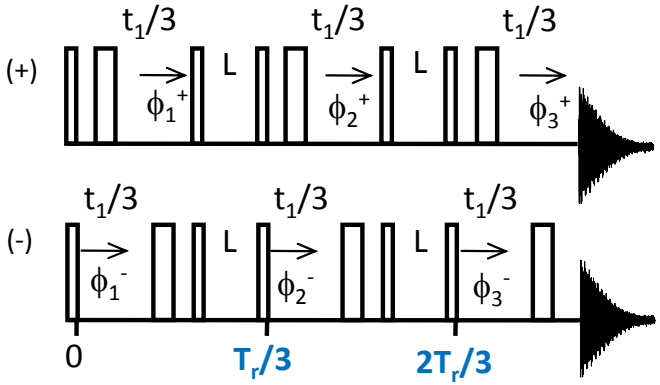
TOSS: WT Dixon JMR 44, 220 (**1981**)

MAT



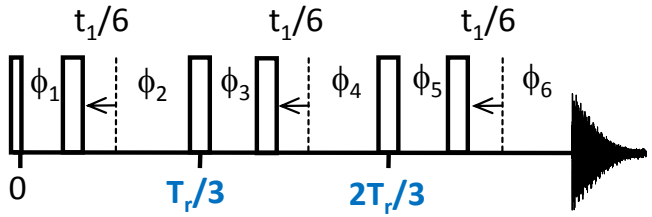
Z Gan JACS 114, 8307 (**1992**)

PHORMAT



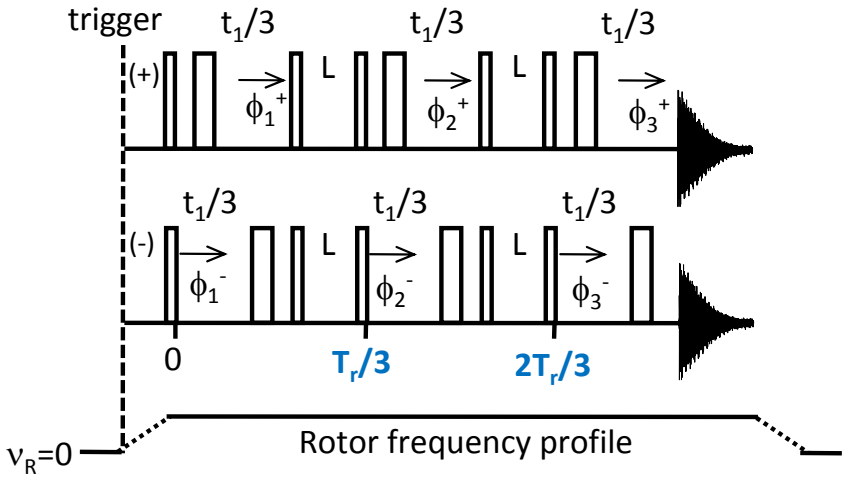
JZ Hu et al JMR A113, 210 (**1995**)

5π-MAT



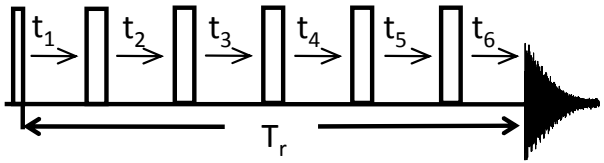
JZ Hu et al JMR A105, 82 (**1993**)

DMAT



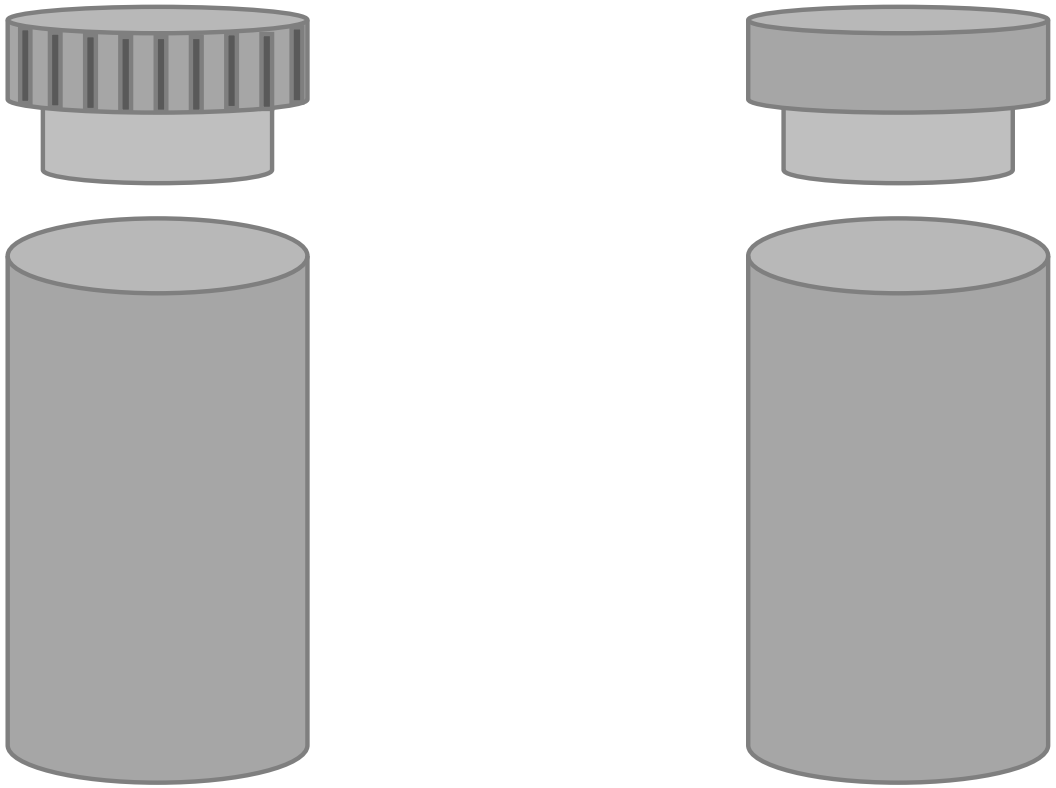
JZ Hu et al JMR 198, 105 (**2009**)

2D-PASS



ON Antzutkin et al JMR A115, 7 (**1995**)

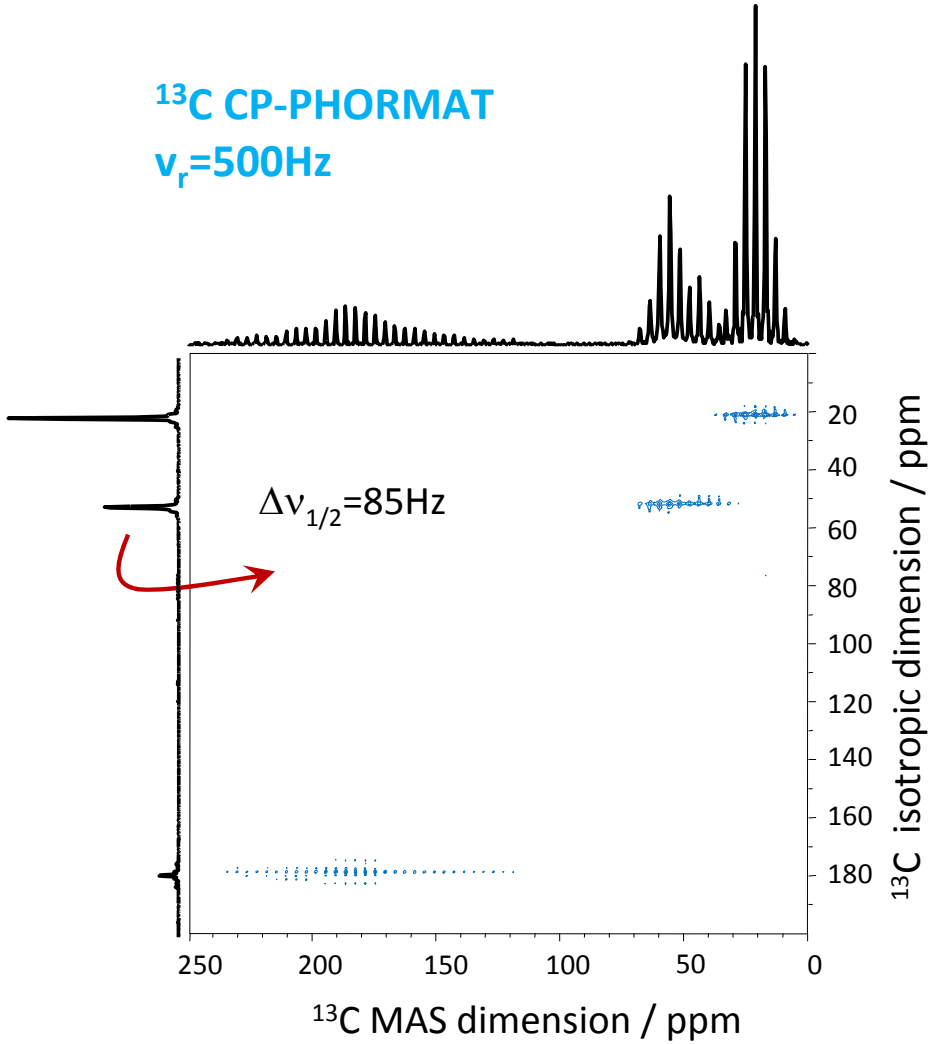
Slow Magic-Angle Spinning

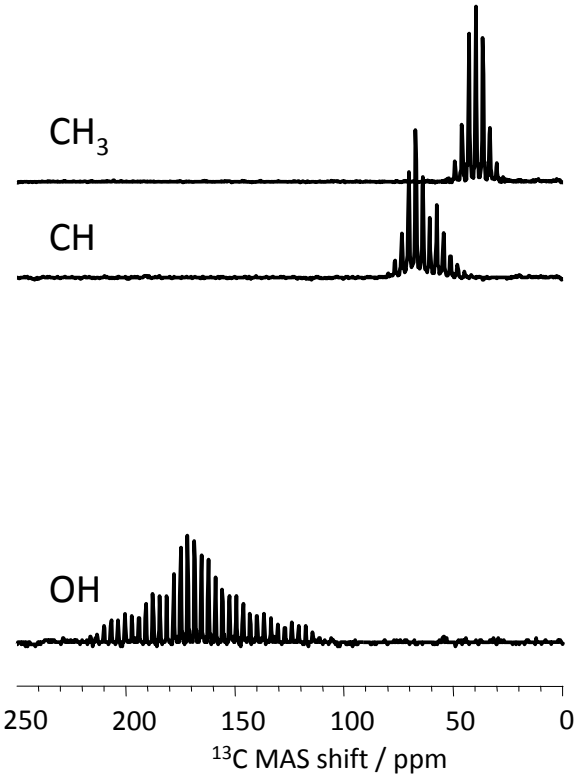


- Smooth grooveless rotor cap
- Big Rotor (if possible)
- Dense Insert (if possible)
- 45 $\pm$ <1Hz

High-Resolution Slow ^{13}C MAS

L-Alanine (3 C sites)

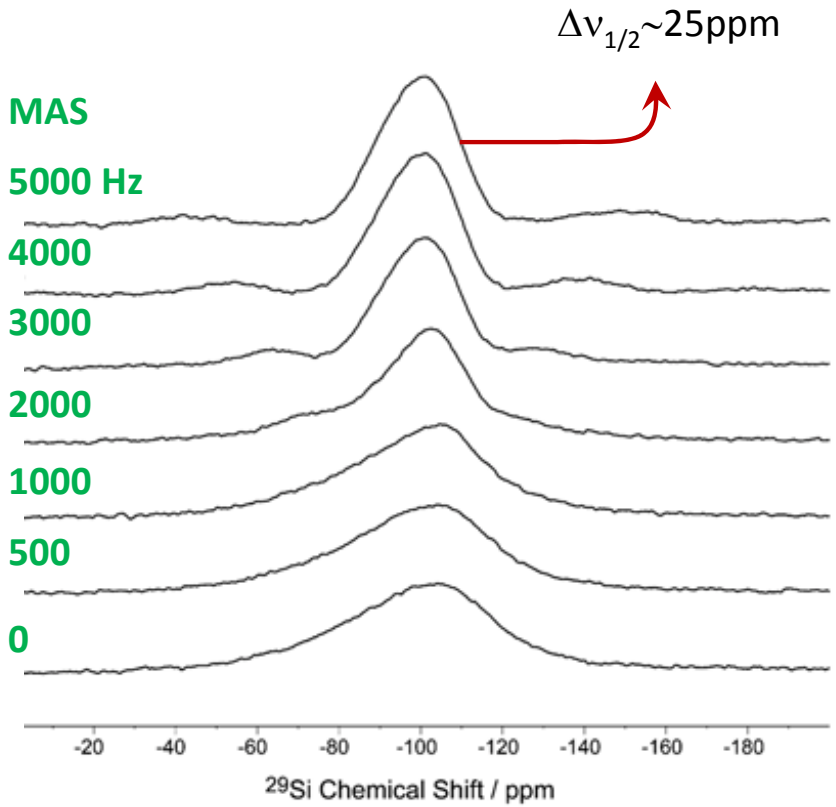




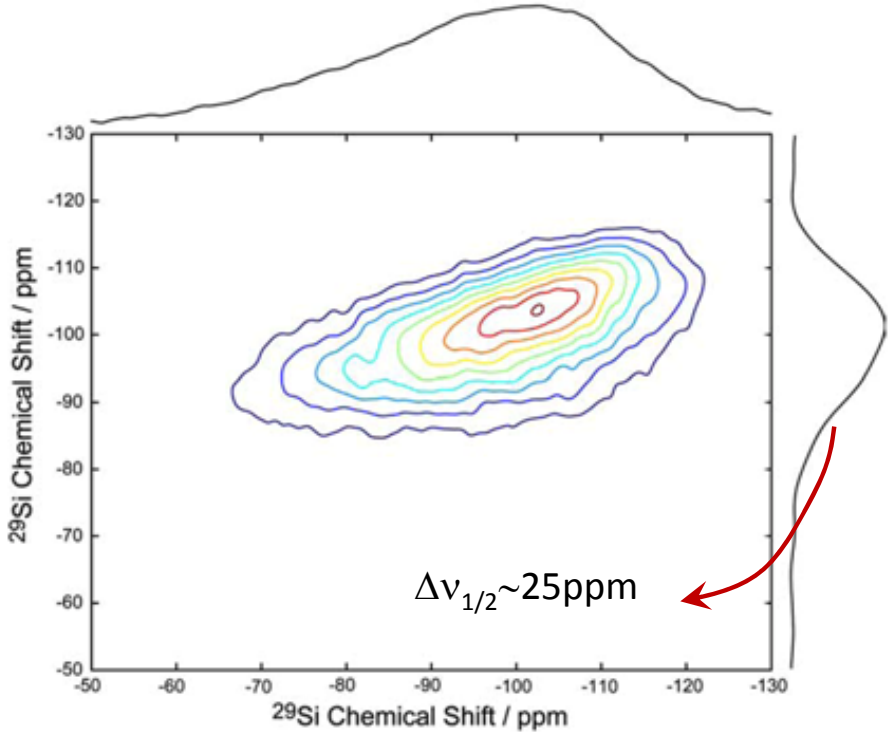
High-Resolution Slow ^{29}Si MAS

Amorphous Borosilicate glass (CE-57)

^{29}Si SP



^{29}Si MAT-CPMG
v_r=300Hz



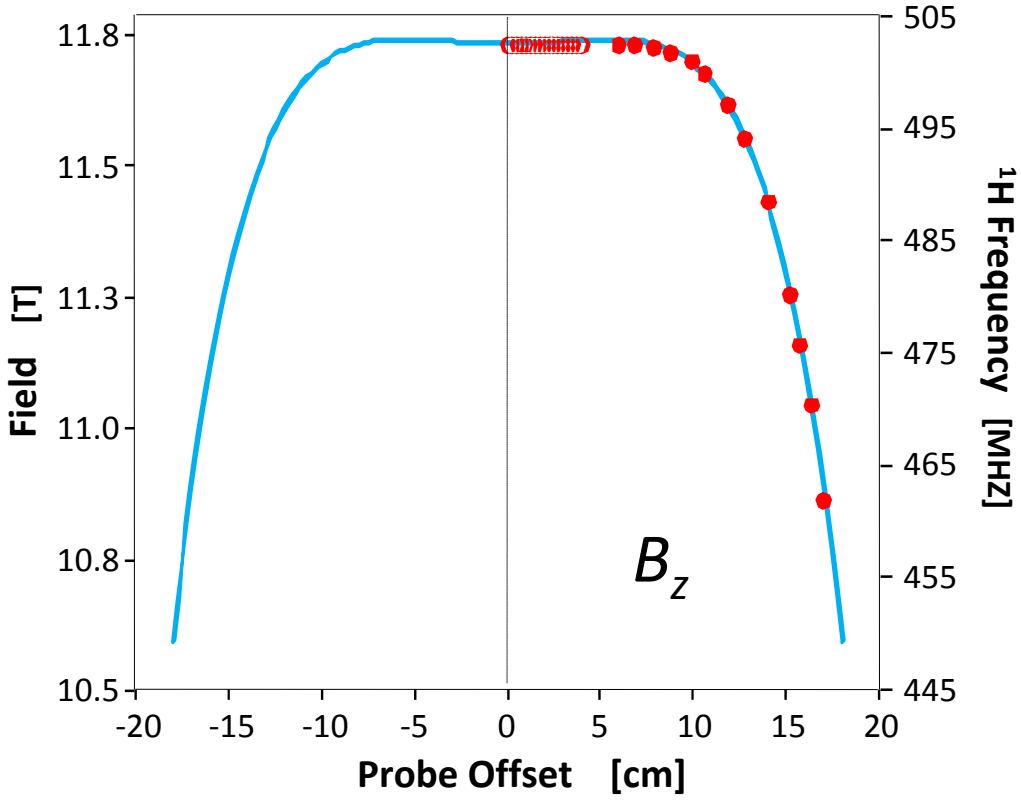
D Sakellariou, JF Jacquinot, T Charpentier, CPL 411, 171 (2004)

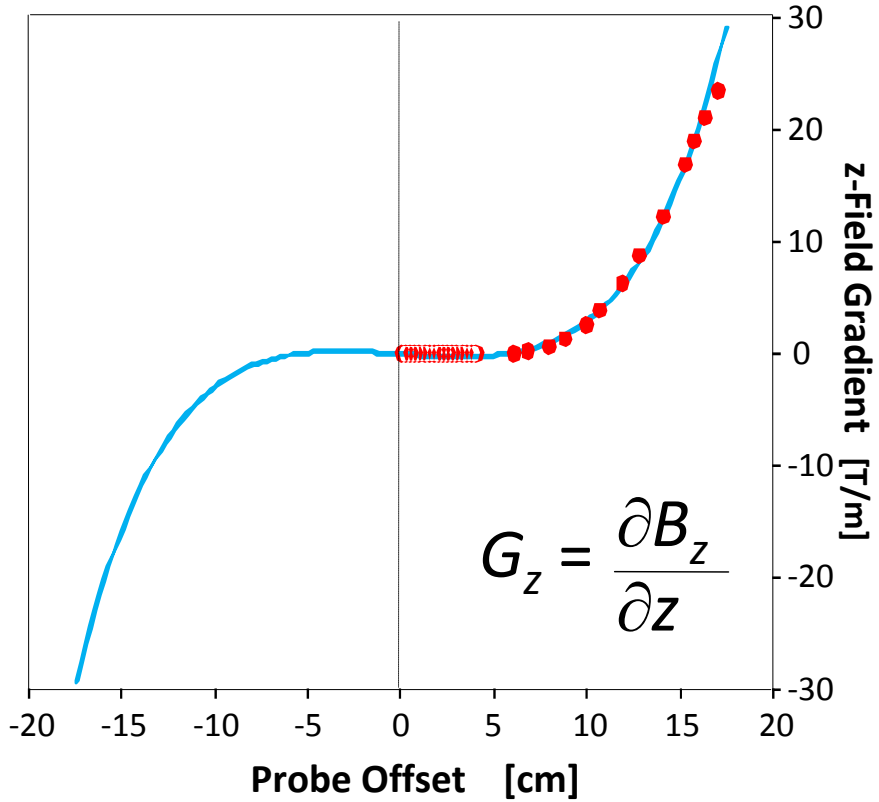
Multi-dimensional Imaging: Combined Stray Field with Slow MAS

JH Baltisberger et al JMR 172, 79 (2005)

An Easy and Effective Imaging Approach !!!

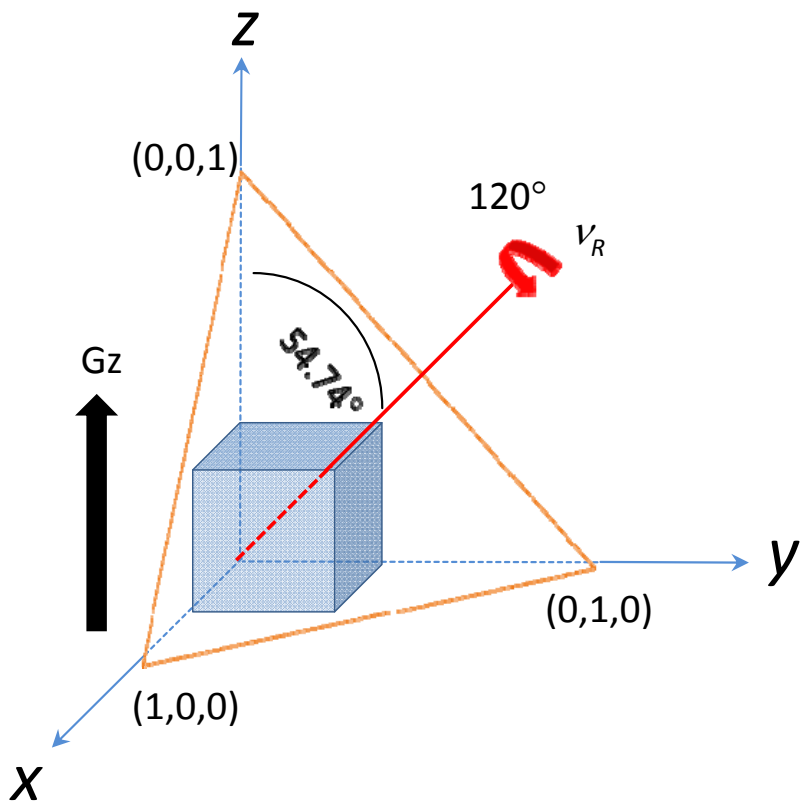
11.75 T Widebore Bruker 500 UltraShield™ Magnet



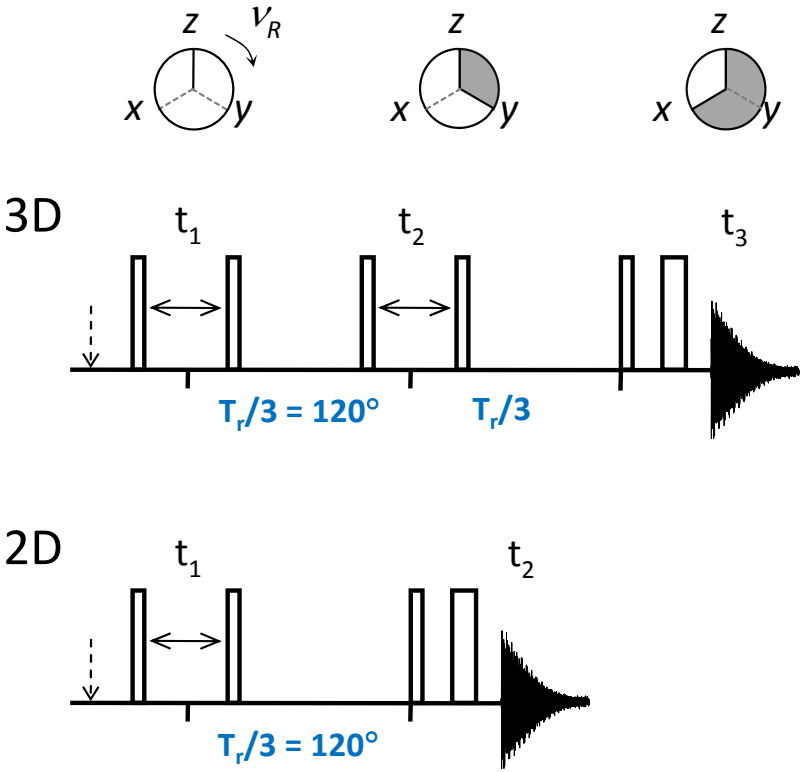


Multi-dimensional Imaging: STRAFI-MAS

Sample Rotation



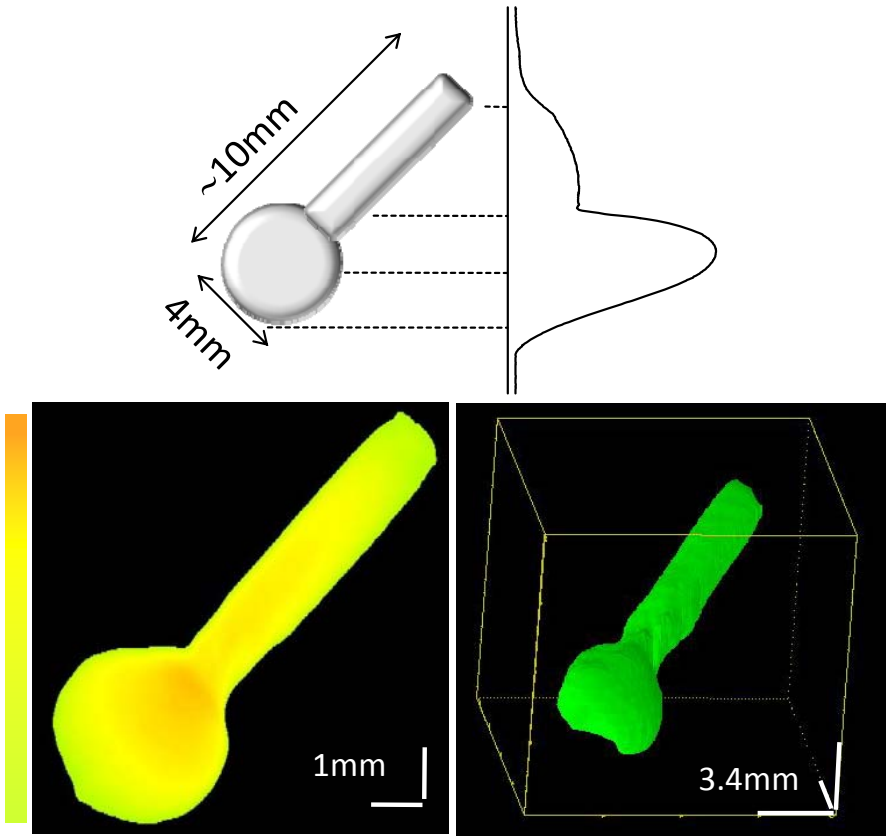
STRAFI-MAS Experiment



JH Baltisberger et al JMR 172, 79 (2005)

STRAFI-MAS Imaging

^{1}H Image



498.88 MHz at 11.75 T

Gz ~ 0.6 T/m

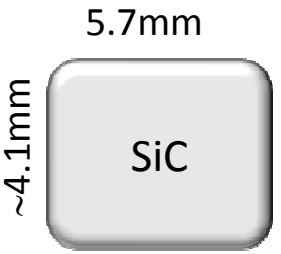
~19 x 380 µm

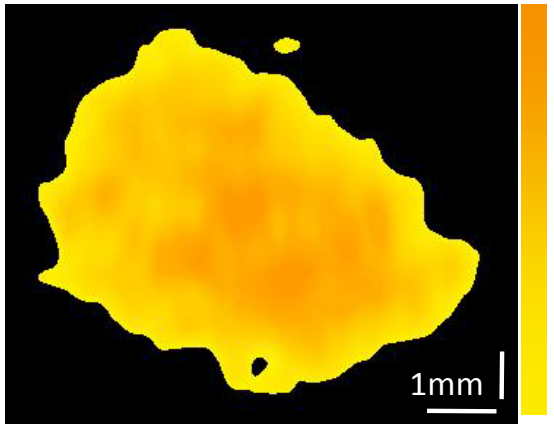
30 min

~19 x 743 x 380 µm;

32 hr

^{29}Si Image





99.16 MHz at 11.75 T

Gz ~ 0.4 T/m

~56 x 520 µm

39 hr

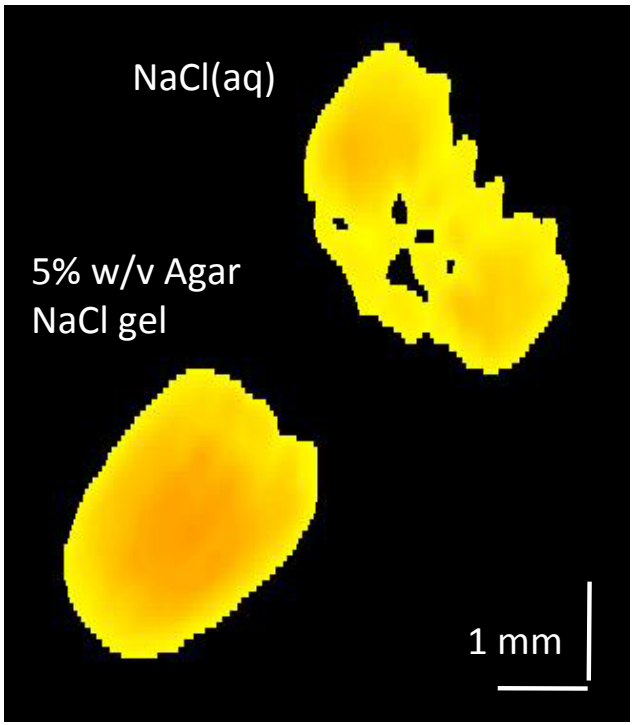
A Wong et al submitted to JMRI

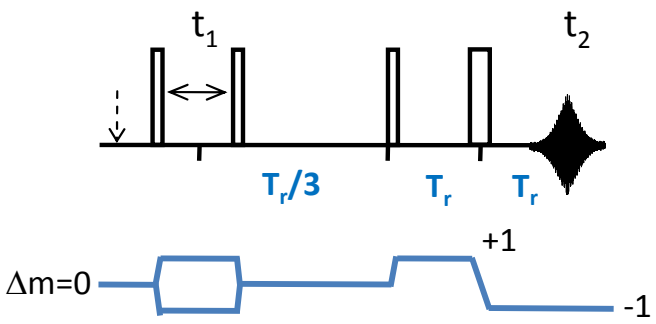
STRAFI-MAS Selective ^{23}Na Imaging

79.2 MHz at 7.05 T

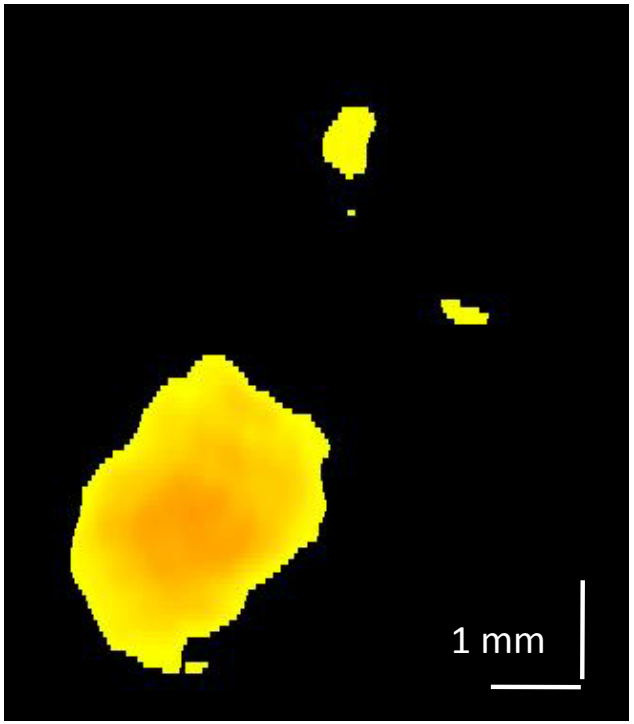
Gz ~ 0.15 T/m

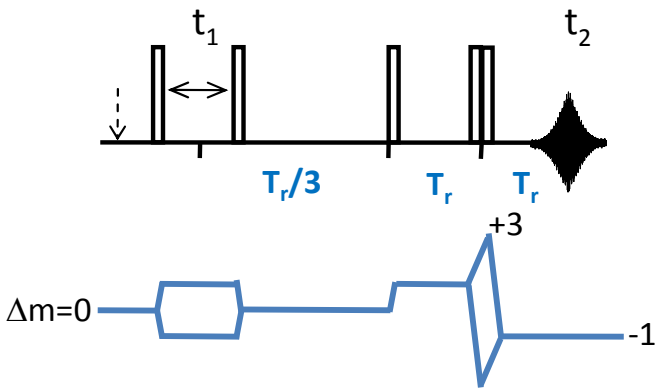
Without TQF





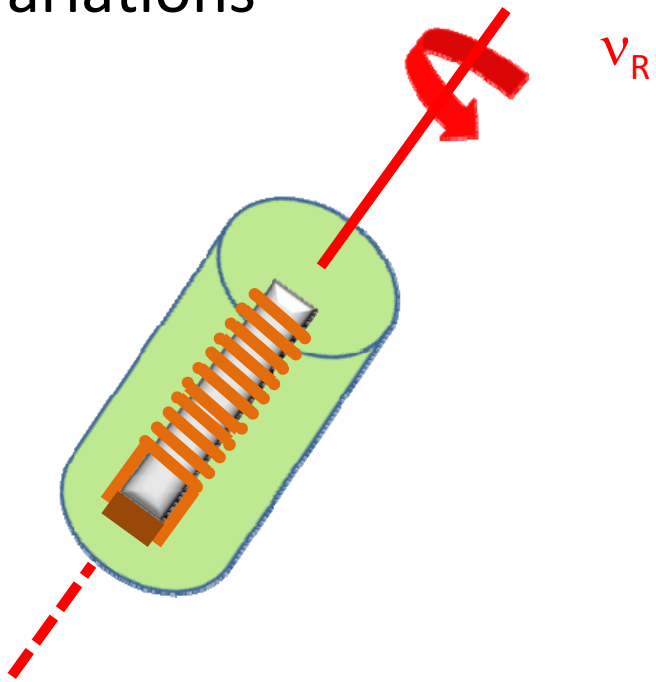
With TQF



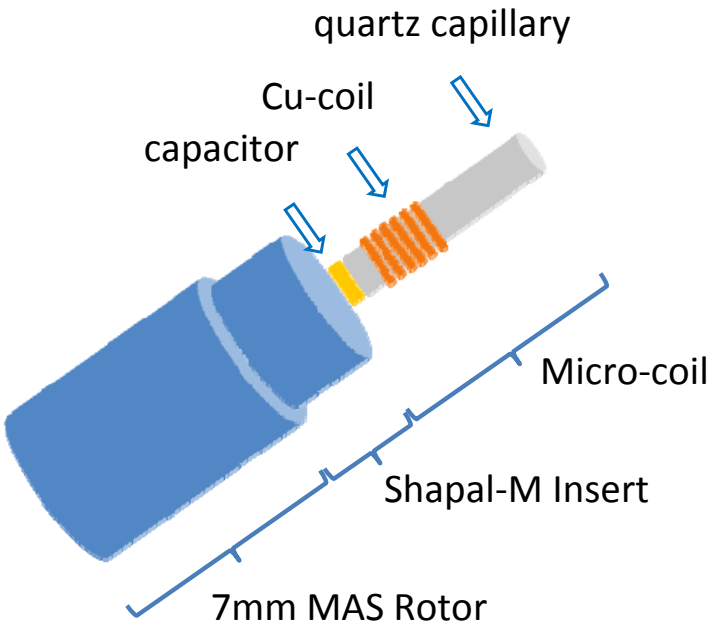


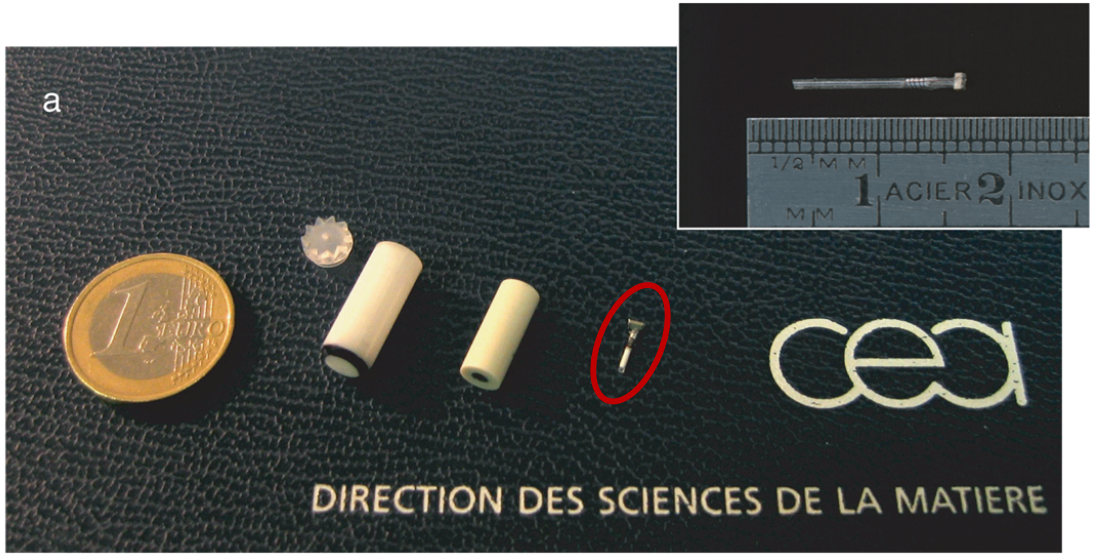
(2) Magic-Angle Coil Spinning (MACS)

- Signal enhancement
- High-resolution
- Size/Volume variations



Magic-Angle Coiled Spinning (MACS)





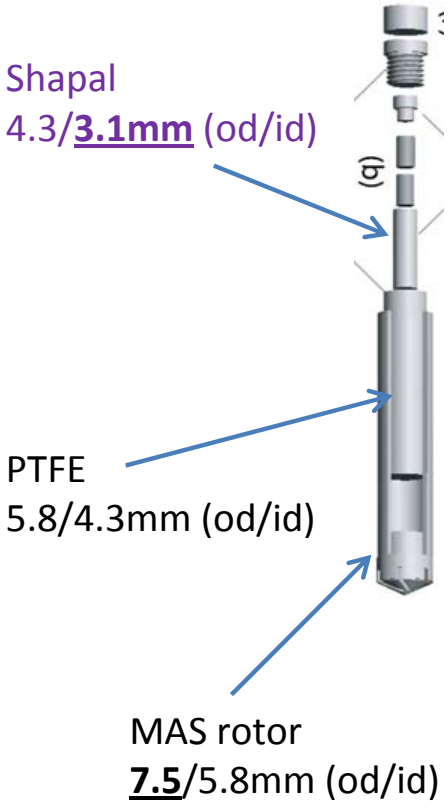
486 MHz

530/660 μm (id/od) capillary

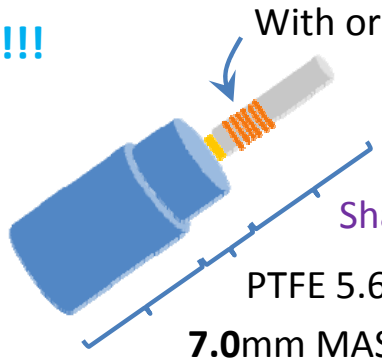
D Sakellariou et al, Nature 477, 694 (2007)

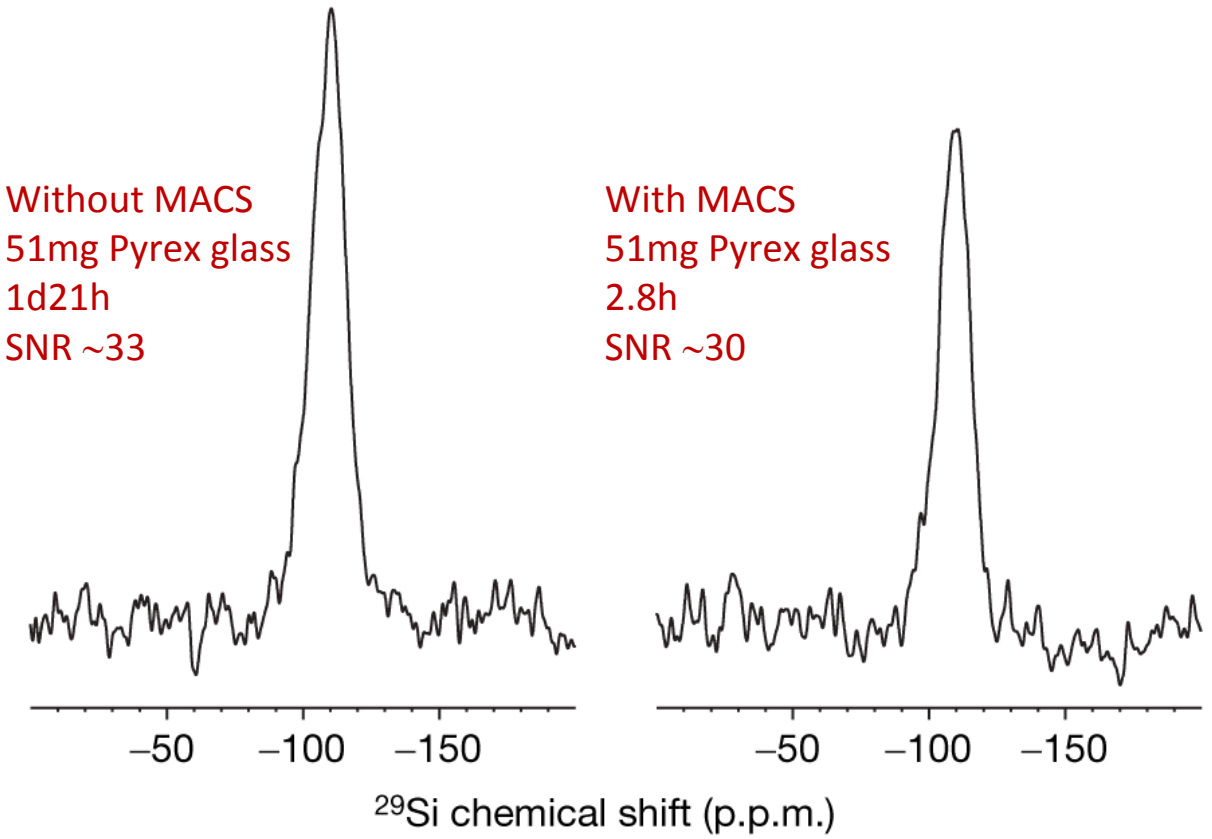
^{29}Si MACS Spectroscopy

Triple-containment MAS System



A Model !!!

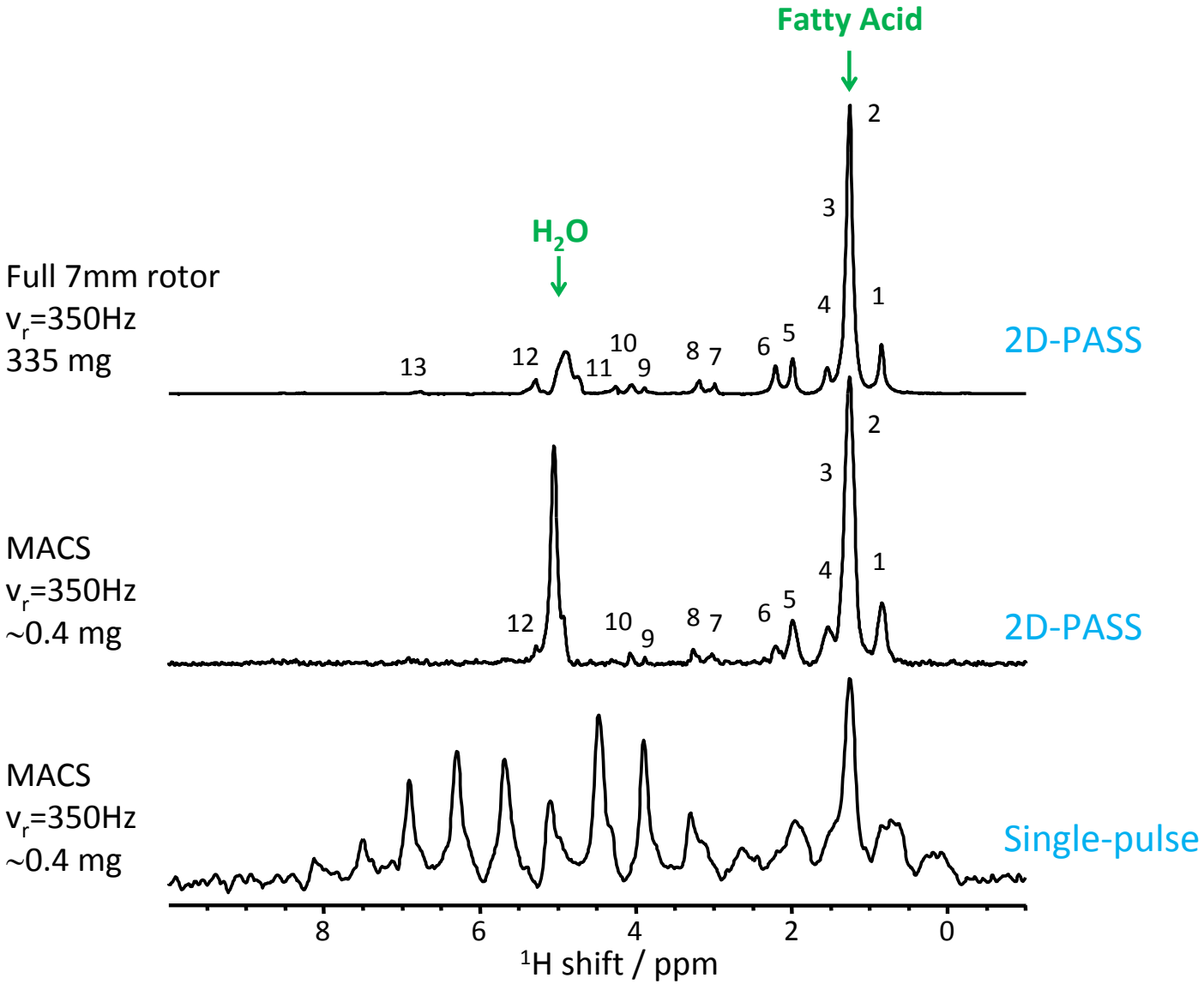




D Sakellariou et al, Nature 477, 694 (2007)

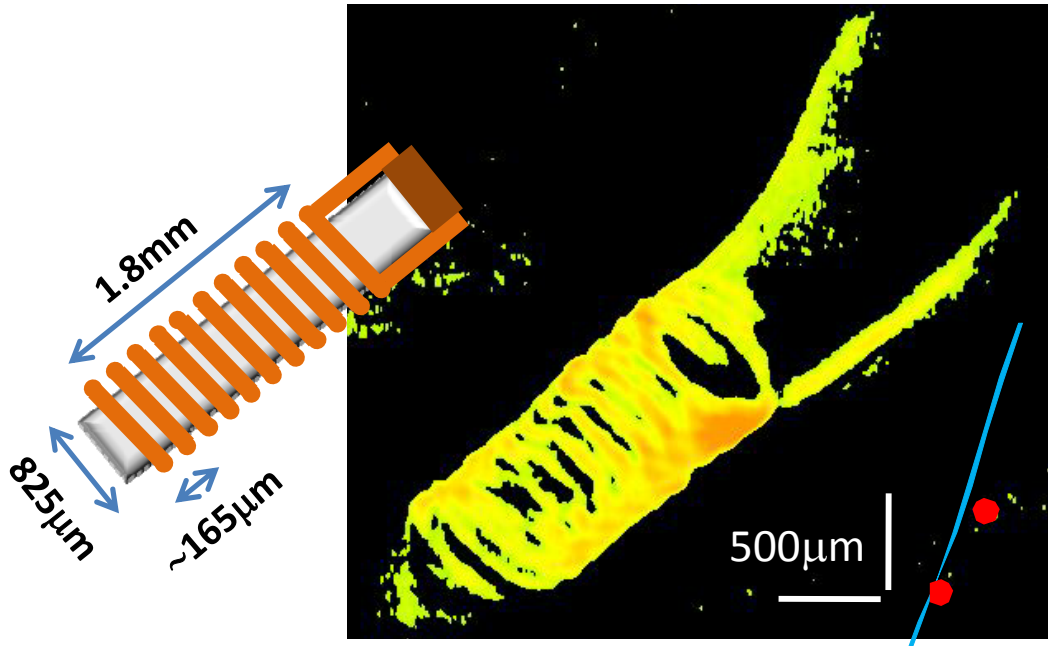
Micro-Sized Slow MACS Spectroscopy

Bovine tissue (H metabolites)



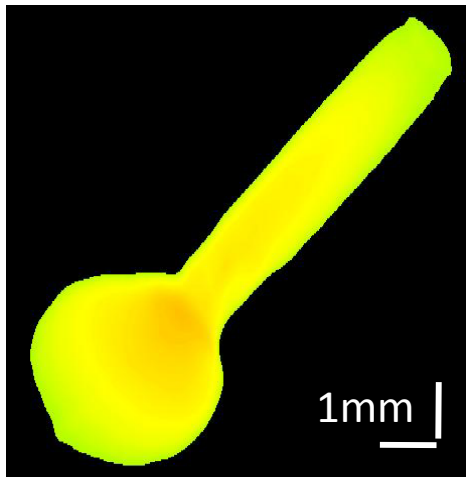
A Wong et al, MRM 63, 269 (2010)

Micro-Sized Slow MAS Imaging



v_R = 110(1) Hz

Gz ~ 0.6 T/m



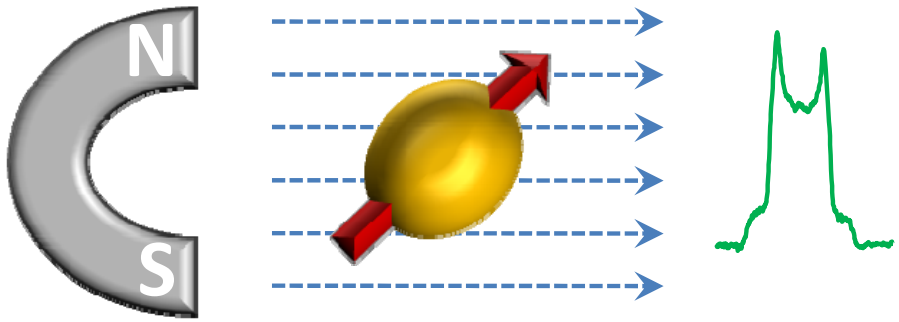
v_R = 463(1) Hz

Gz ~ 5.2 T/m

(3) Portable Permanent Magnet

- On-site (*ex-situ* and *in-situ*) measurement
- Low cost

Cedric Hugon, Guy Aubert and Dimitris Sakellariou



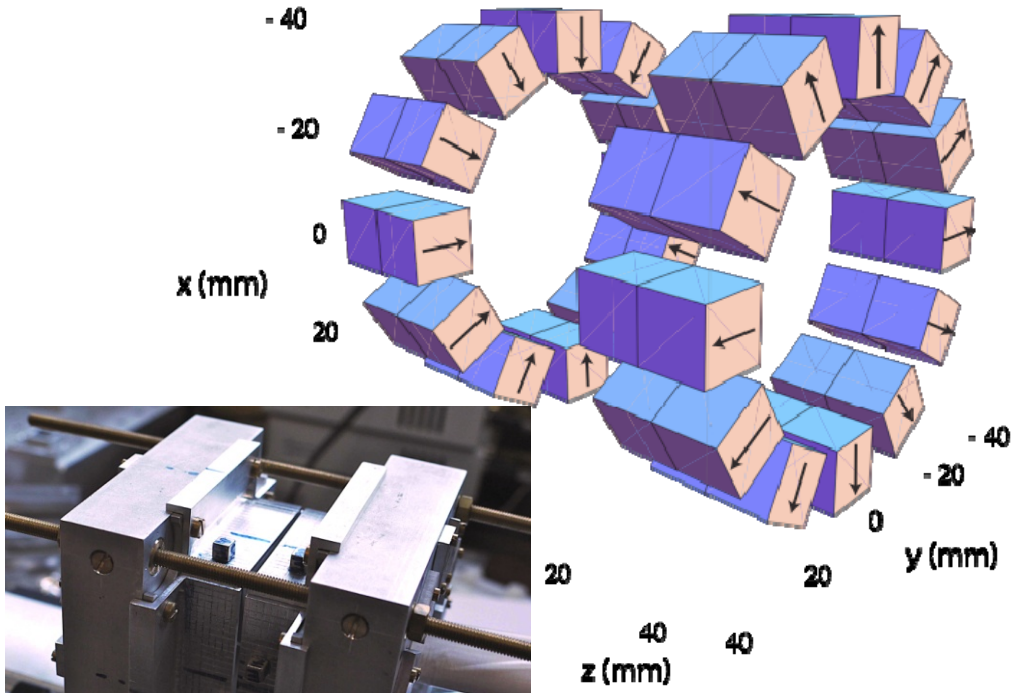
Approach to Magnet Design

- Field variation
- Good field homogeneity
- Low cost & portability

$$B_z(r,\theta,\emptyset) = Z_0 + \sum_{n=1}^{\infty} r^n \left[Z_n P_n(\cos\theta) + \sum_{m=1}^{n} (X_n^m \cos m\emptyset + Y_n^m \sin m\emptyset) P_n^m(\cos\theta) \right]$$

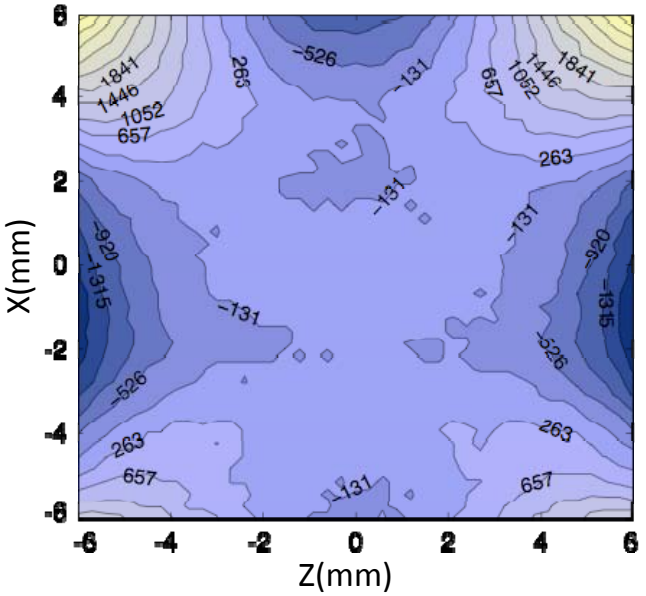
Cylindrical *In-situ* Magnet

Design

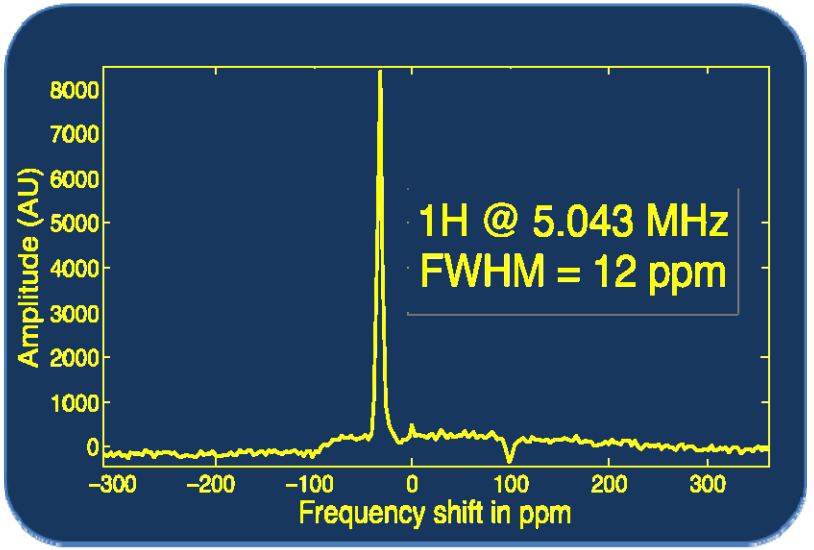


- low cost <200 €
- B_0=120 mT
- ΔB = 6 ppm in 2mm diameter

Field Measurement

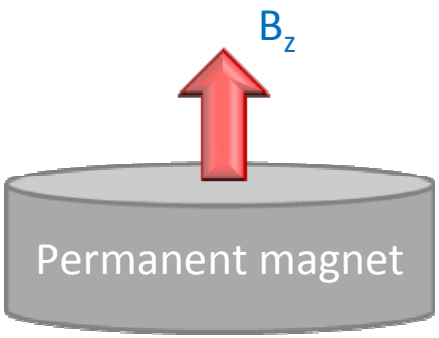


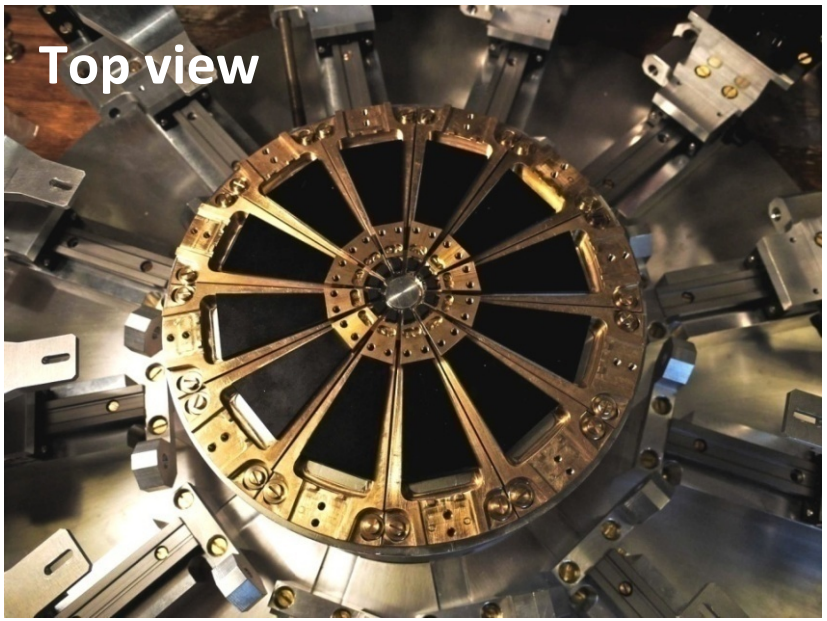
^{1}H NMR Measurement

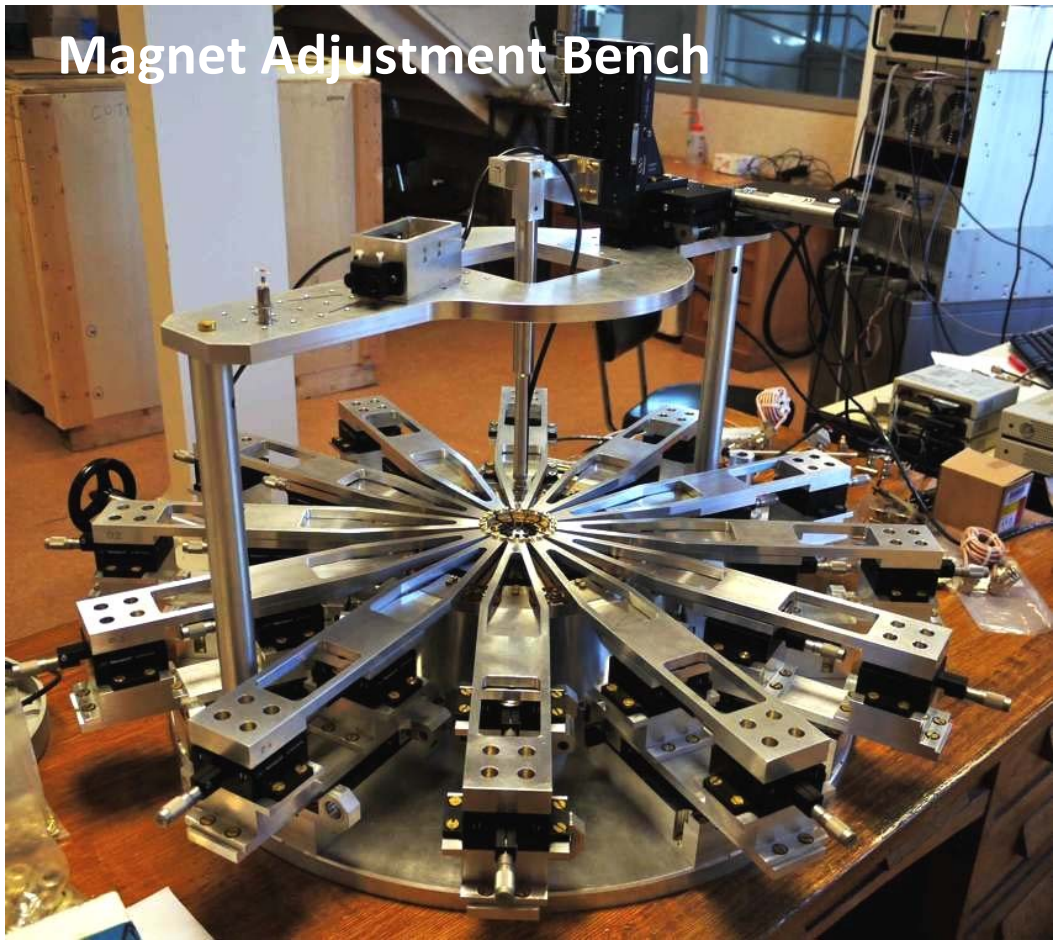


C Hugon et al CR Chimie 2010 in press
C Hugon et al JMR 2010 submitted

Single-Sided *Ex-situ* Magnet

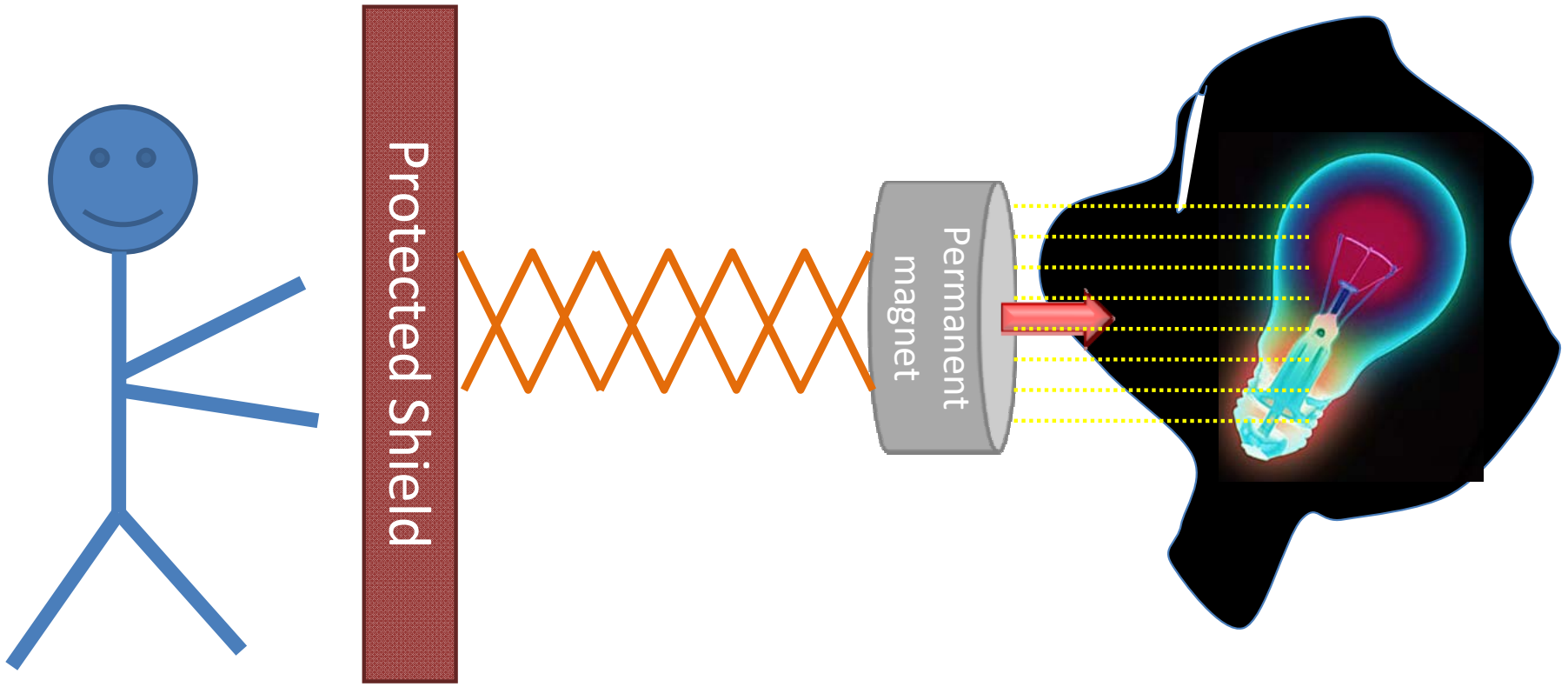






- B_0= 0.3 T
- ΔB = 3.3 T/m
- Sweet spot @ 2cm from surface
- 1cm diameter spherical volume (with ppm uniform gradient)
- Penetration up to 7cm (with uniform gradient)

Imagine


Protected Shield
Permanent magnet
You
Radioactive site
A safe distance

Conclusions

1. Slow-MAS spectroscopy and imaging
    a. Enhanced safety precaution from spinning

2. Magic-angle coil spinning
    a. Signal enhancement
    b. Capable of micro-sized sample

3. Portable permanent magnet
    a. On-site (*in*- or *ex-situ*) NMR/MRI experiments

Guy Aubert

Jacques-François Jacquinot

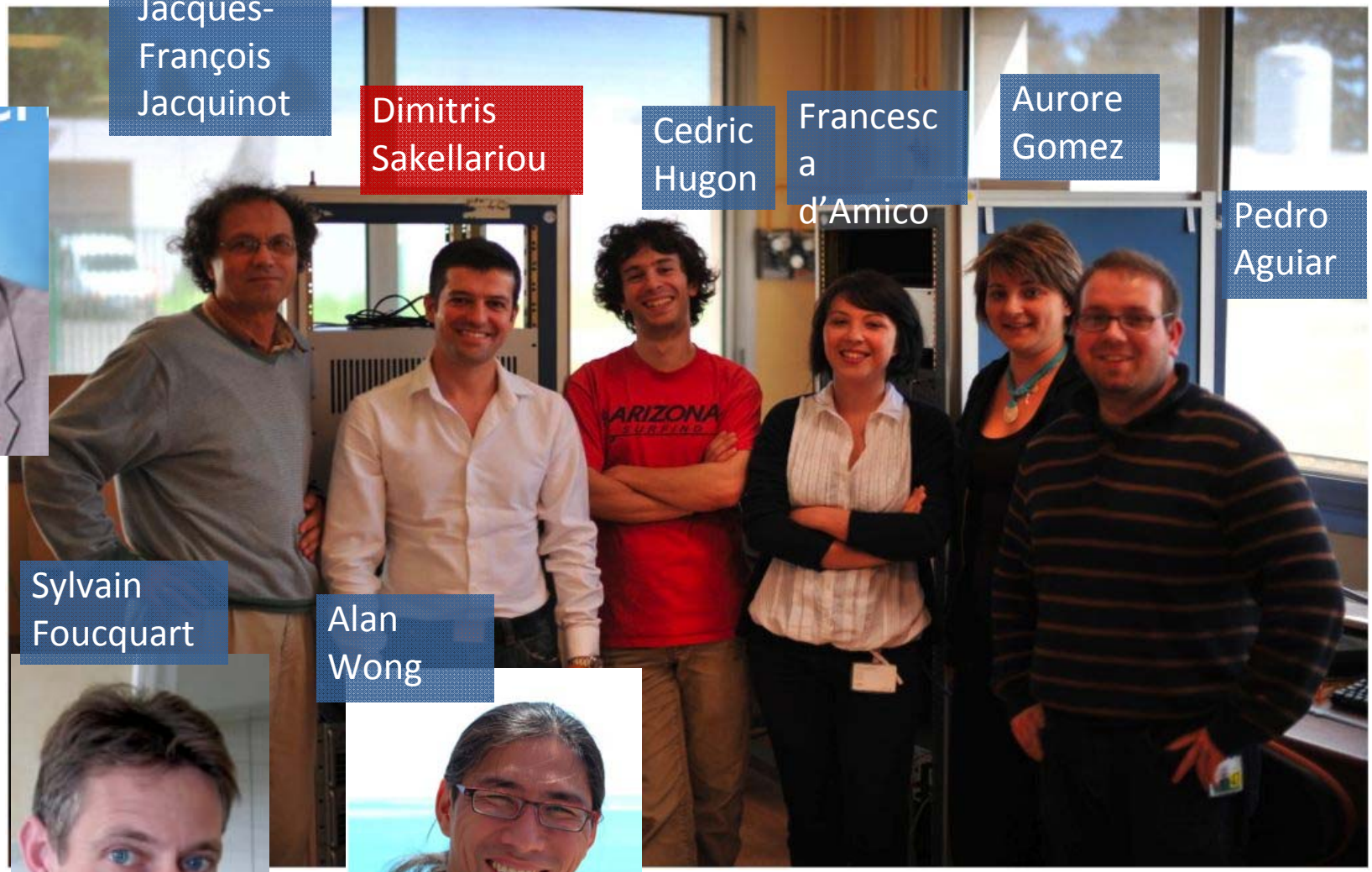
Dimitris Sakellariou

Cedric Hugon

Francesca d'Amico

Aurore Gomez

Pedro Aguiar



Angelo Guiga

Sylvain Foucquart

Alan Wong

THANK YOU !!!