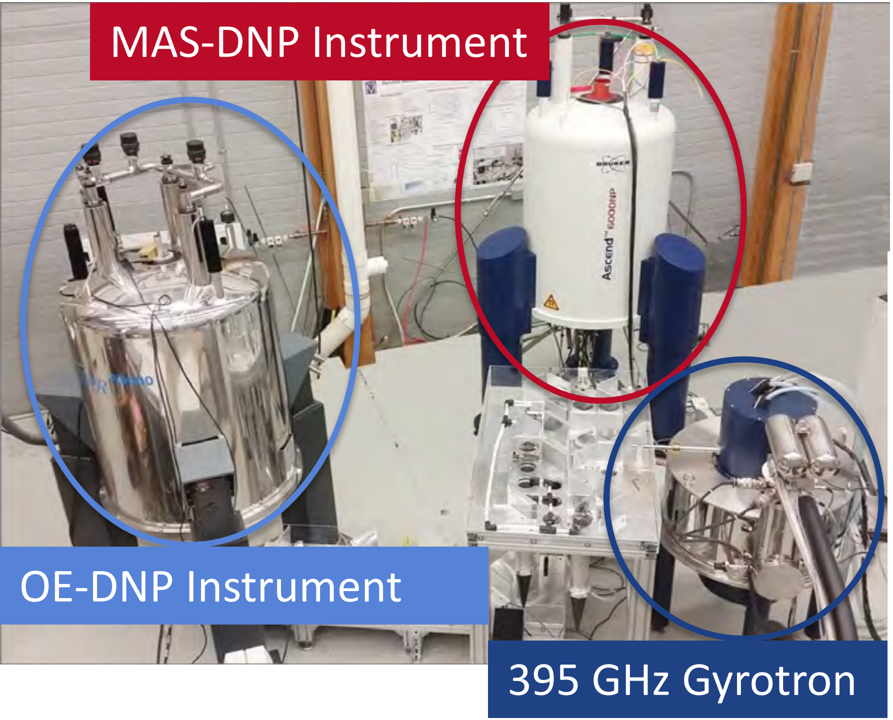
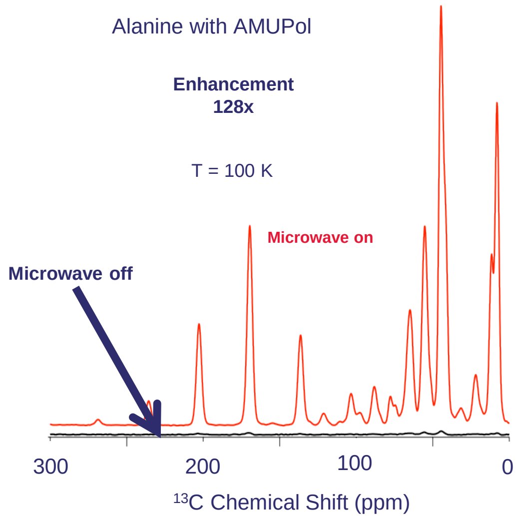
NATIONAL RESOURCE FOR ADVANCED NMR TECHNOLOGY

The Resource is a collaboration among the National High Magnetic Field Lab, Florida State University, and the University of Florida funded by NIH/NIGMS grant 1P41 GM122698.

600 MHz MAS-DNP System



AMUPol (10 mM), D_8-Glycerol/D_2O/H_2O (6/3/1 v%),
→ $\epsilon_{on/off}$ ranging between 110-130 with full microwave power

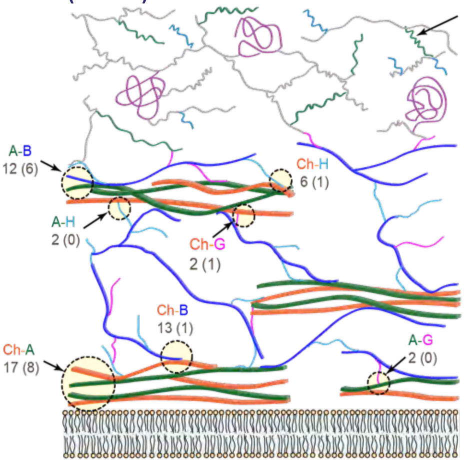


Dr. Frédéric Mentink-Vigier instructs students about gyrotron operation: NHMFL DNP workshop, Tallahassee FL, October 22-24, 2019

- # Biological Sample
  - Analyzing molecular structure of fungus cell-wall Tuo Wang's group

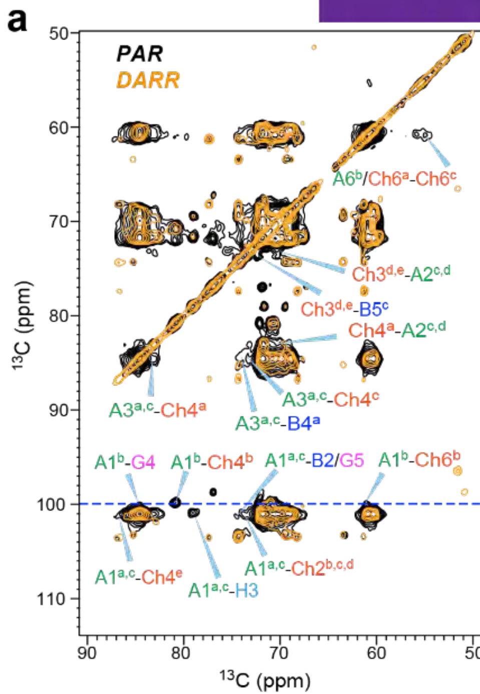
Challenge: **explore the tight packing of chitin and α-1,3-glucans in fungi**

Kang, X. *et al.* Molecular architecture of fungal cell walls revealed by solid-state NMR. *Nat. Commun.* **9,** 2747 (2018).



Illustrative model of the supramolecular architecture of *A. fumigatus* cell walls

DARR collected in 6 hours with no loss of resolution with MAS-DNP

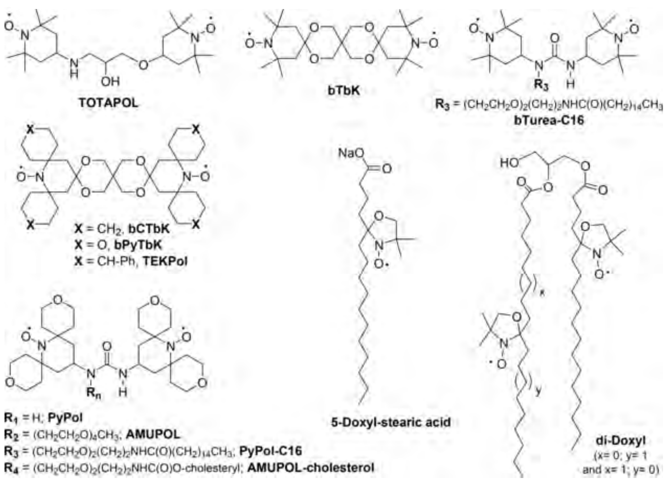



DNP
IS HAVING
A MOMENT

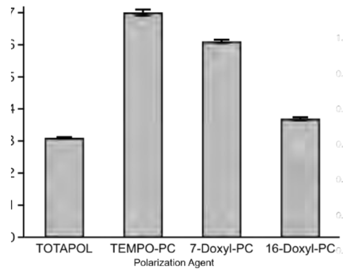
Matrix Free DNP Sample Preparation

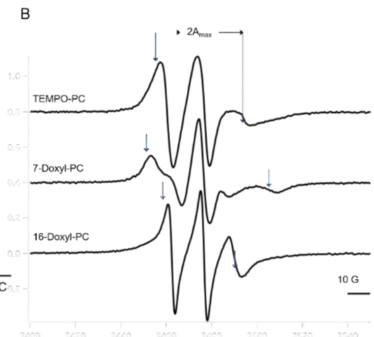
Does *not* require addition of glassing agent (i.e glycerol, DMSO, etc.)

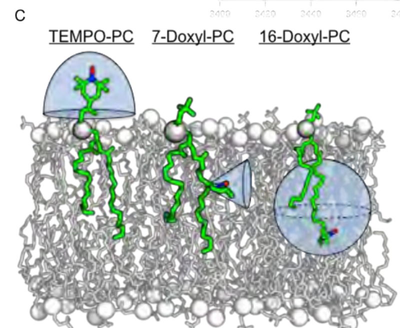
Membrane tethered mono/biradicals



E.Salnikov et al. *ChemPhysChem* 2017, 18,2103 –2113







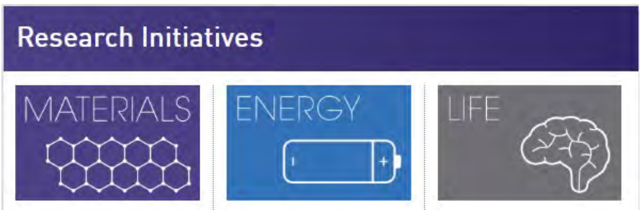
A. Smith et al. *J. Phys. Chem. B.* 2016, 120,32, 7880 –7888

NMRprobe.org

Come work with us!

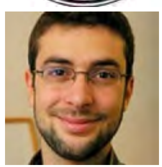
NATIONAL MAGLAB

Nationalmaglab.org



Tim Cross
Steve Hill
Bill Brey
Hans van Tol
Peter Gor'kov
Fred Mentink
Thierry Dubroca

FLORIDA STATE UNIVERSITY 1851



UF UNIVERSITY of FLORIDA

Nhi Tran	Anil Mehta
Adam Smith	Rohit Mahar
Gwladys Riviere	Joanna Long
James Collins	Matt Merritt

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Workshops offer hands-on-experience with biomolecular NMR, hardware development, and operational best practices

NMRprobe.org

Workshop: Biomolecular NMR

4 Days of hands on training

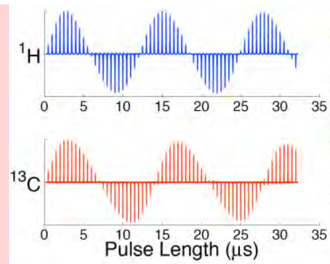
10 Participants: Graduate students
Postdoctoral research associates

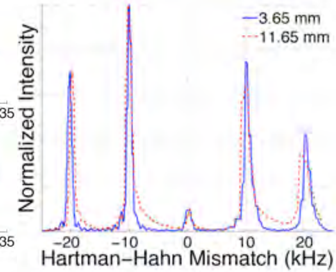
Year 1, 3, etc. $^{13}C/^{15}N/^{2}H$ detected hrNMR

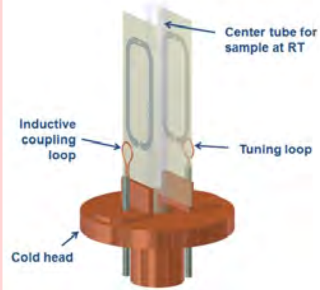
Day 1 Introductory biomolecular NMR
Day 2 $^{13}C/^{15}N/^{2}H$ detection
Day 3a Metabolomics applications
Day 3b Protein applications
Day 4 NMRbox (NMR Data Processing and Analysis BTRR)

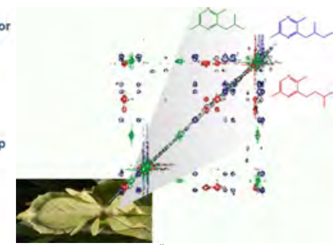
Year 2, 4, etc. DNP MAS Biosolids NMR

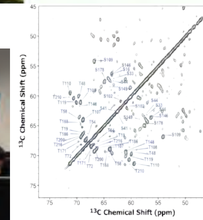
Day 1 Introductory biosolids NMR
Day 2 Structure determination strategies
Day 3 Dynamic nuclear polarization
Day 4 NMRbox











Dr. James H. P. Collins demonstrates good sample preparation techniques to students.

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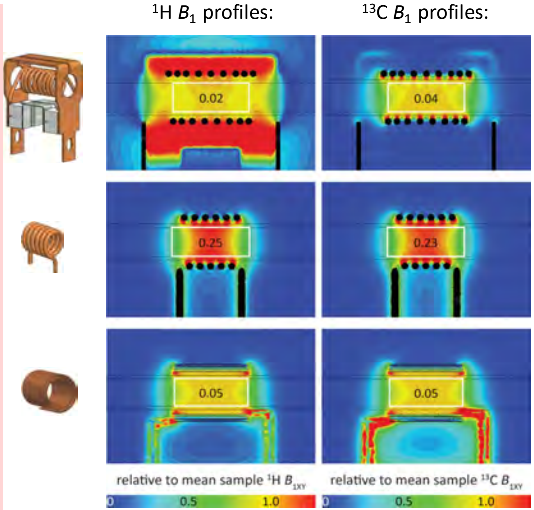
Workshop: Hardware development

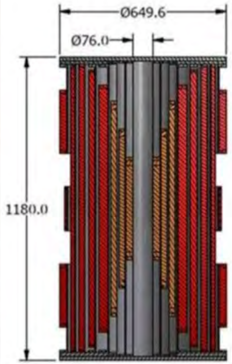
Year 1, 3, etc. NMR probe building

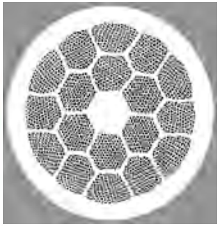
- Day 1 Introductory RF circuitry
- Day 2 Modern NMR probes / coils
- Day 3 In silico design and analysis
- Day 4 Advanced topics (MAS, cryogenics, materials)

Year 2, 4, etc. NMR in the Series Connected Hybrid

- Day 1 Intro to 36T magnet construction
- Day 2 ssNMR of quadrupolar nuclei
- Day 3 NMR in the SCH
- Day 4 NMRbox





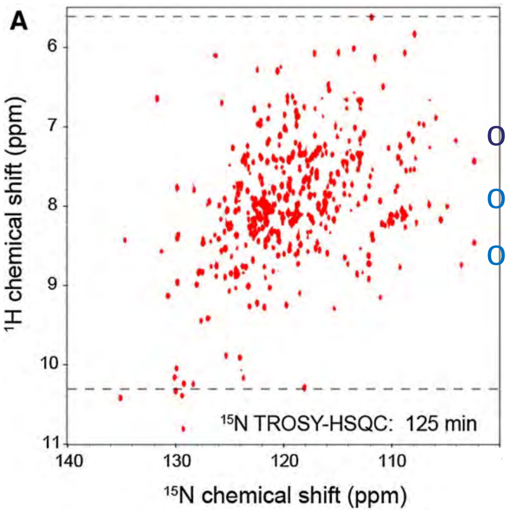


^{15}N-Sensitive 5 mm HTS Probe for 800 MHz

High quality ^{15}N-detected spectra can be obtained at high field.

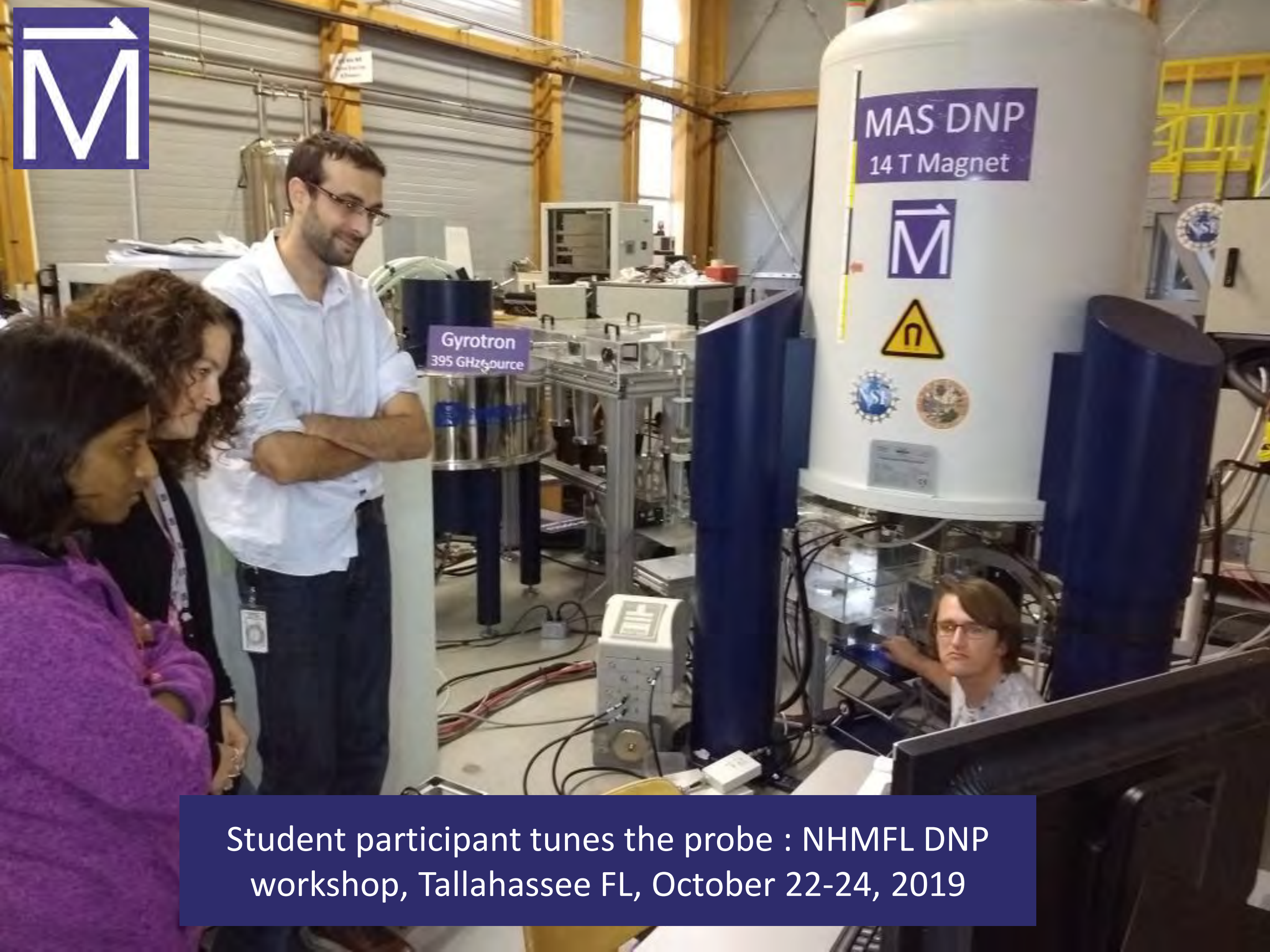
- **Narrow ^{15}N linewidth reduces spectral overlap**
- **No deuteration needed**
- **Low ^{15}N frequency -> less sample loading**
- **No water suppression**

} **Ideal for HTS coils!**



- **Unshielded 800 AV2 available in Tallahassee**
- ***800 US^2 NMR System installed at UF this fall***
- ***With supplement we have ordered <u>two</u> probe bodies and cryocooler***

Takeuchi *et al.* (2015) *J. Biomol. NMR*, **63**, 323.



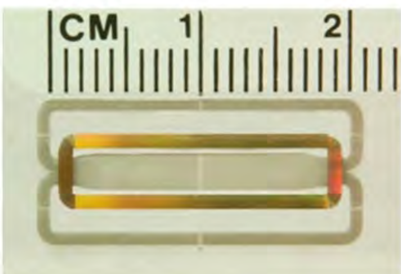
Student participant tunes the probe : NHMFL DNP workshop, Tallahassee FL, October 22-24, 2019

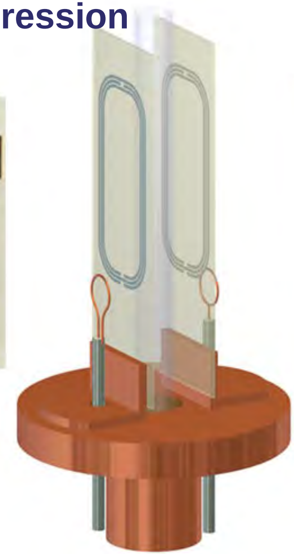
^{13}C-^{2}H sensitive 3-mm HTS probe for 600 MHz

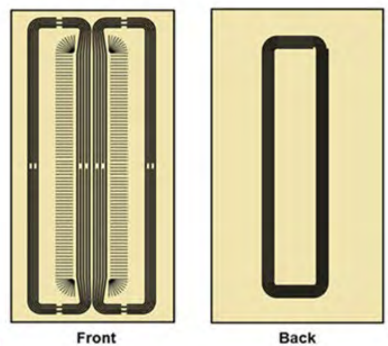
Direct detection is essential for metabolic flux studies and exploits the large spectral dispersion of ^{13}C.

- **Applications in natural products, mixture analysis, metabolomics**
- **Low frequency -> less sample loading (small R_S)**
- **No need for water suppression**

} Ideal for HTS coils!







- o **Builds on our unique expertise in dual-sensitive HTS probes**

Ramaswamy *et al.* (2016) *IEEE Trans. on Appl. Supercon.*

NMRprobe.org

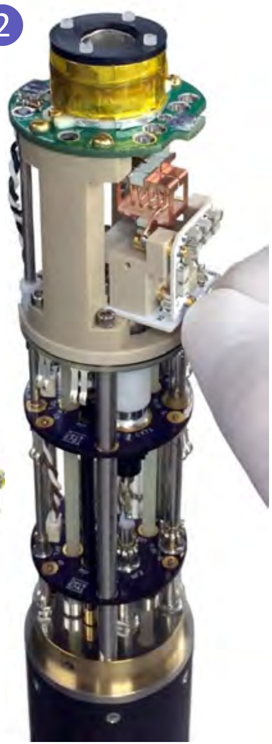
New NMR User Probes

1.5 GHz Ultrafast MAS

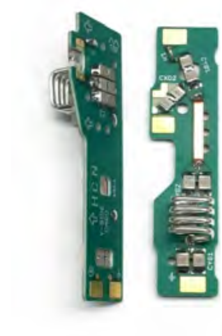
1

1-resonance 3.2 mm MAS
Low-γ, quadrupolar NMR
22/36 μL, 18/24 kHz speed

2



2-resonance ^{1}HX Static Probe
Materials, Biological Solids
Larger volume 3, 4, 4x4 mm Low-E coils

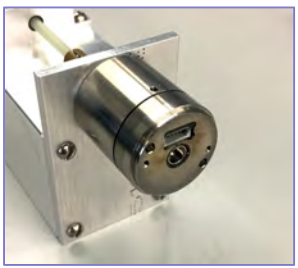


3

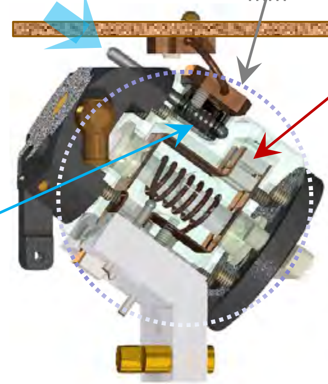
3-resonance 2.0 mm ^{1}HXY MAS
Multidimensional NMR
9 μL, 37 kHz speed

3.2 mm ^{1}HX MAS Probe, 2-resonance, 1.5 GHz

- Addresses lack of ^{1}H decoupling and large samples for low-γ NMR
- 3.2 mm will rotor pack up to **4X** more sample than 2.0 mm
- Broad range of nuclei, low- and mid-gamma
- Materials and some biological spin-½ exp-ts
- 7Li lock sample + coil are integrated into MAS stator
- Improve sensitivity for mid-gamma X ≥ ^{27}Al
- Remote magic angle adjustment



Piezo motor for remote magic angle adjustment



DNP workshop, NHMFL, Tallahassee FL, October 22-24 2019

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3.2 mm [1]HXY MAS Probe, 3-resonance, 1.5 GHz

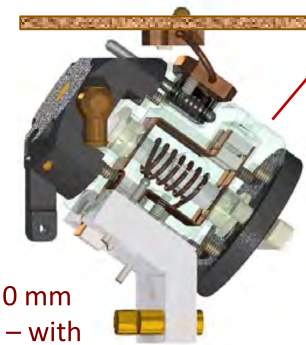
More sample volume than in existing 2.0 mm [1]HXY probe

Design is based on 2.0 mm probe with tune cards

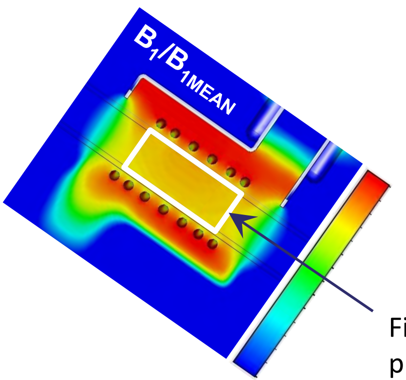
Tune cards will be made on demand: HCN, HCO, HCD, etc…

Biological and materials applications

Remote magic angle adjustment



Based on 2.0 mm [1]HXY probe – with new 3.2 mm spinner



Field simulations by W. Mao predict homogeneous rf fields

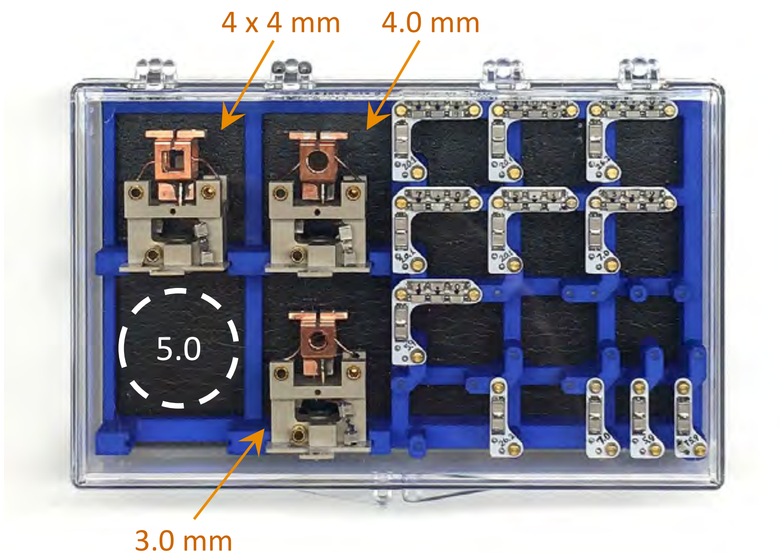
5.0 mm Static RF Coils, 1.5 GHz – aligned proteins

Larger volume coils for ^{1}HX static probe – more S/N for aligned samples

5.0 mm coils pack 65% more sample than 4.0 mm

Hope for 50 kHz B_1 on both ^{1}H and ^{15}N

No need to build new probe



Currently available RF coil inserts

Students test new hardware outside the magnet in Gainesville FL

NMRprobe.org

Ultrafast 100 kHz MAS Probe, 800 MHz, 0.75 mm

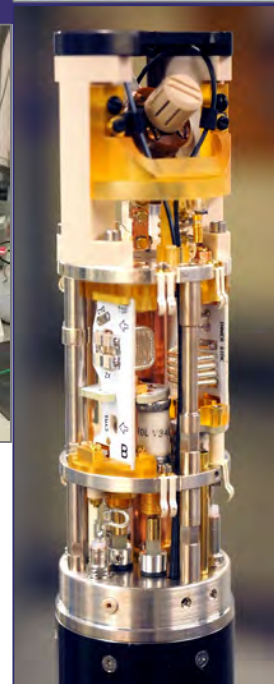
RF development for fast MAS took place at 800 MHz

We aim to optimize ^{1}H sensitivity at higher B_0 fields

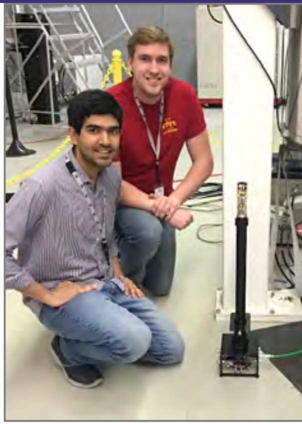
0.75 mm JEOL spinner = 100 kHz speed

Our ^{1}H power efficiency = **2X** of commercial JEOL probe

Wide range of ^{1}HXY isotopes via tune cards



Aaron Rossini group



GB1 protein, 800 MHz, 95 kHz MAS, I. Hung: