DOTY SCIENTIFIC

The Resonator

NMR - MRI Newsletter Summer 2018

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DEAR COLLEAGUE,

We've been to the ENC in Orlando and at ISMRM/ESMRB in Paris. Some of you saw Laura at Snowbird Utah at the Rocky Mountain Conference. As always we are glad to see old friends and to make some new ones.

At the ENC in Orlando and at ISMRM in Paris we introduced a novel large double-tuned CP MRI coil that we believe our imaging friends found exciting. You can find some information on the coil here http://dotynmr.com/introducing-a-new-generation-of-double-tuned-mri-coils/.

At the Rocky Mountain Conference we introduced a new wide bore MAS probe we call the "WB Ultra Range". We will have more to say about that later in this newsletter. For now we are glad to be home. We have a lot of work to do.

David and Judy Doty

At the Rocky Mountain Conference at Snowbird, Utah, USA, Laura enjoyed this view, at nearly 11,000 feet, with the conference center below.

Serving ice cream in our ENC suite this year proved unusually challenging for Glenn.

(To view more ENC pictures: http://dotynmr.com/enc-2018-photos/)

At ISMRM/ESMRB, David and Judy shared an evening in Paris.

As Promised:

The Doty *WB Ultra-Range* MAS Probe

- **Broad Temperature Range**

-170 °C to +270 °C *(and ultimately higher)*

- **Fully Multinuclear ^{31}P to ^{103}Rh**

Broad Tuning Range with Most Tuning Inserts

- **Double-Tuned ^{1}H/X or Triple-Tuned ^{1}H/X/Y**
- **Broad Range of Spinner Options**

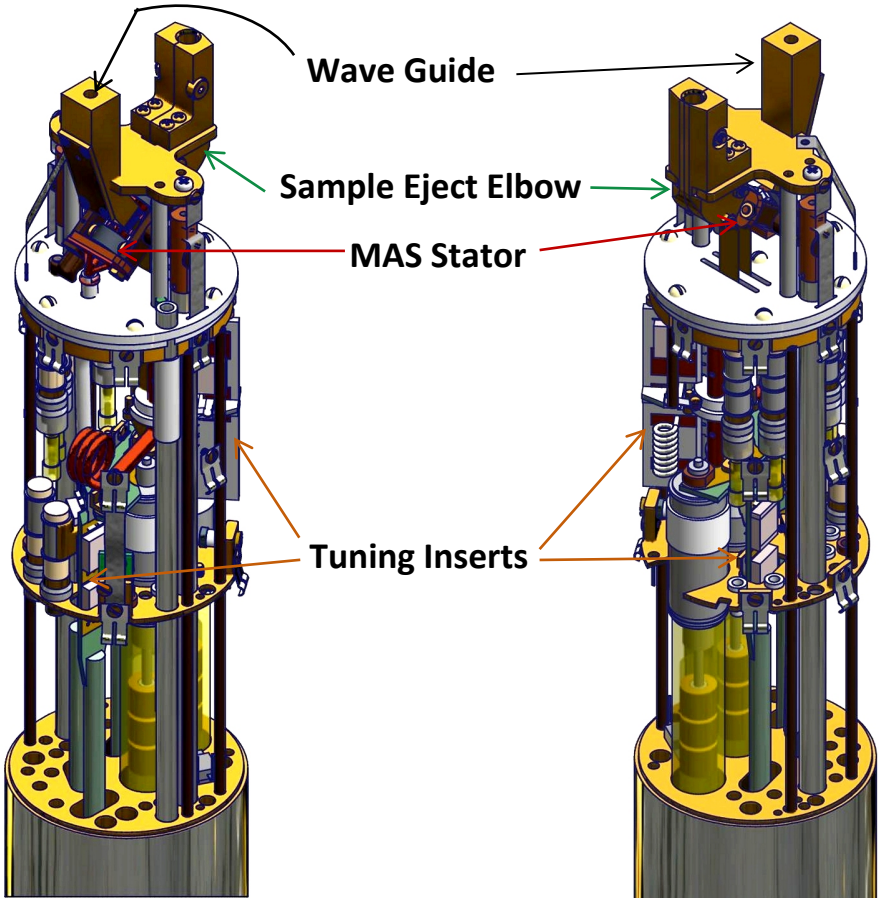
7 mm, 5 mm, 4 mm, or 3 mm

- **A Sample Eject Option Is Available with 3 mm**
- **Compatible with DNP – Microwaves from the Top**
- **For WB Magnets, 300 MHz to 850 MHz**

UPCOMING CONFERENCE

47th Southeastern Magnetic Resonance Conference

October 26 - October 28 2018
Clemson, South Carolina, USA
https://chemistry.sites.clemson.edu/casabiancagroup/semrc.html



To Deuterate Your Proteins – or Not to Deuterate? That is the Question.

MAS has long been an integral technique in Solid State NMR, beginning with investigations on typically inorganic samples, but quickly moving to biological samples with ever increasing molecular weight. The resulting complexity of the NMR spectrum has lead to the use of High-resolution inverse ^{1}H detection in Solid State NMR spectroscopy for large molecules. New developments in protein biochemistry and fast spinning have contributed to this rapidly evolving area of NMR. Bibhuti Das, Paul Ellis and David Doty present a white paper in "publications" on the Dotynmr website describing rationales and available techniques for these challenging experiments, while asking the question "To Deuterate Your Proteins or Not to Deuterate? "

Click here to read the full article. "To Deuterate Your Proteins - or Not to Deuterate? That is the Question."

Doty QUAD-FAST-MAS H/X/Y/Z Probe HR Solids Results

Four Independent, Efficient, High-Power Channels.

Solids Quad Resonance.

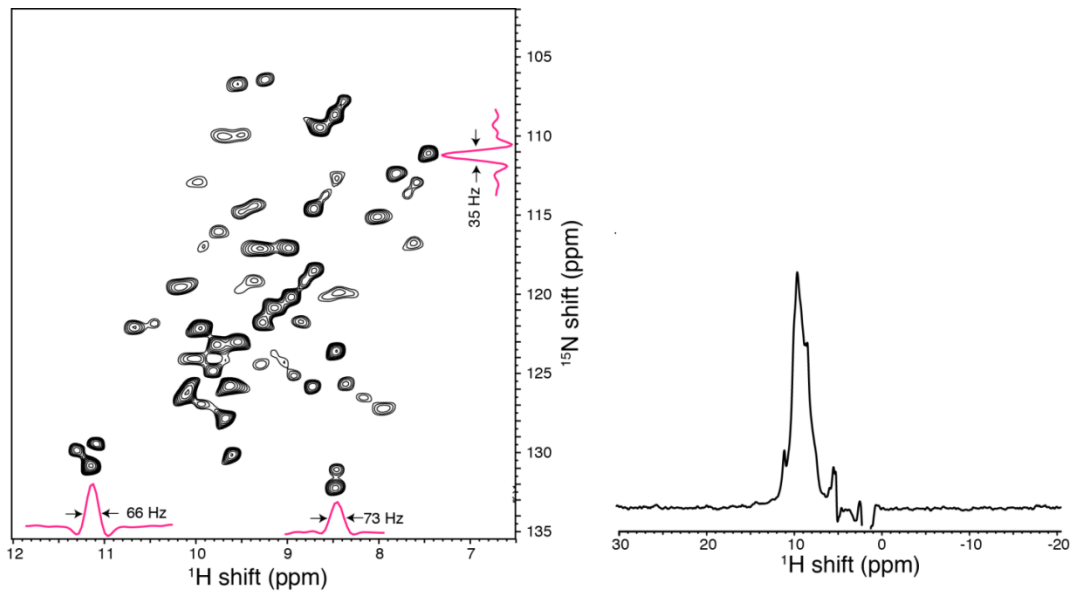


Figure 1.

Inverse proton detected ^{1}H/^{15}N heteronuclear correlation 2D- spectrum of U-C, D, N labeled GB1 obtained from a Doty 3mm Quad-Fast-MAS probe with a 500 MHz JEOL spectrometer. 12-15mg of micro/nano-crystalline protein samples were center packed using spacers and spun at 25 kHz MAS. Two transients were co-added for each indirect increment (total 64) with a 2 second recycle delay. Total experiment time was <5 min. 2D data was zero filled to at least four times and *sine* apodization function was applied prior to Fourier transformation in both dimensions. Proton 1D-spectrum of first FID from the 2D experiment is shown to the right. 1D data was processed with 100 Hz line broadening and measures SNR ~250 per 2 scans.

The QUAD-FAST-MAS probe and multinuclear detection

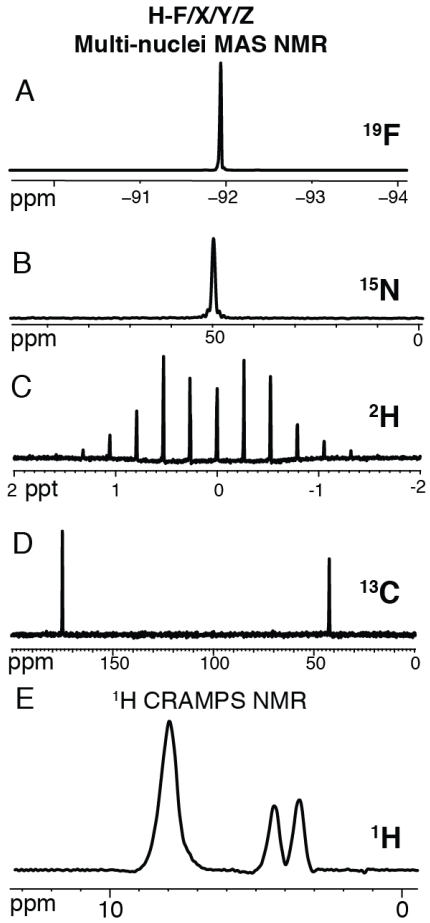
Doty Scientific offers a 3mm H-F/X/Y/Z quad tuned MAS probe that enables state-of–the-art NMR technology. The

MAS probe is designed with a single solenoid RF resonator that is capable of tuning to four different frequencies ranging from 10 MHz to 800 MHz (and eventually to 1.3 GHz with 1.3-mm rotors). The maximum spinning speed can reach up to 28 kHz while pulsing on four channels with duty cycle such that accommodates an average power up to 10 W. For the purposes of the present discussion, we demonstrate the performance of the four channel MAS probe, QUAD-Fast-MAS, on a variety of samples (liquids, viscous liquids, and solids) in Figures 2 and 3, including a microcrystalline protein sample in Figure 1. The protein sample is a 56 residues long U- C, D, N isotope labeled and perdeuterated (100% back exchanged) immunoglobulin G binding domain 1 (GB1) in micro/nano-crystals.

We demonstrate the resolution and sensitivity of the QUAD-Fast-MAS probe by applying tailored experiments (single pulse to CPMAS) for multi-nuclear detection. The probe was configured with the four channels tuned to ^{1}H-^{19}F/^{13}C/^{15}N/^{2}H. The NMR

Figure 2.

Multi-nuclear NMR spectra obtained from a Doty 3mm Quad-Fast-MAS probe with a 500 MHz JEOL spectrometer.



A) ^{19}F, PFCE, one scan. 5 kHz spinning

B) ^{15}N CPMAS, ^{15}N D-Alanine. 22 kHz spinning,

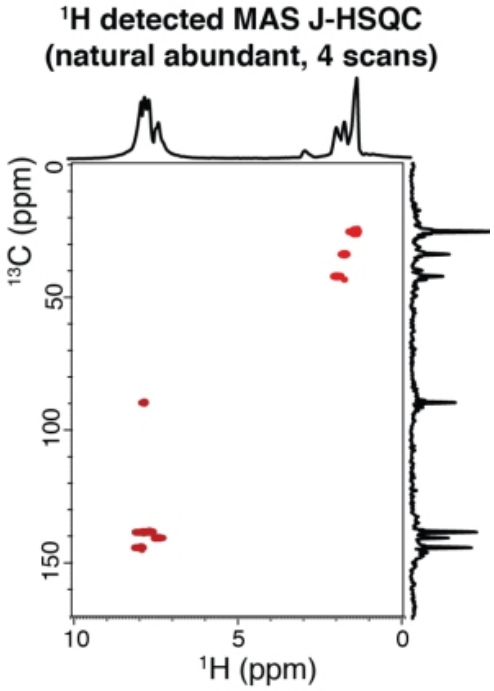
C) ^{2}H D-Proline, one scan. 20 kHz spinning

D) ^{13}C CPMAS, Glycine.

E) ^{1}H detected CRAMPS, Glycine, one scan.

Figure 3.

Inverse proton detected 2D HSQC spectrum of 5cb in natural abundance.



4-cyano-4' pentyl biphenyl (5cb) liquid crystal was used to record a proton detected 1H/13C HSQC-2D spectrum. The 30 µL fully packed rotor was spun at 5 kHz MAS to obtain a high-resolution proton spectrum from 5cb in nematic phase at room temperature (^{1}H linewidth is ~28 kHz without MAS). Standard inverse detected 2D HSQC pulse sequence (similar to solution NMR) was applied to record the 2D spectrum with four scans per increment. Each cross-peak corresponds to chemical shift of singly bonded ^{1}H and ^{13}C nuclei.

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