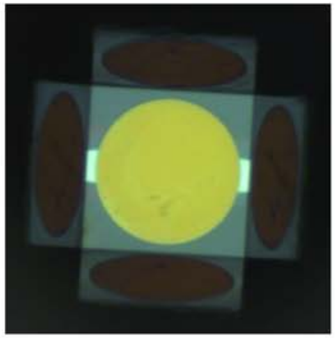
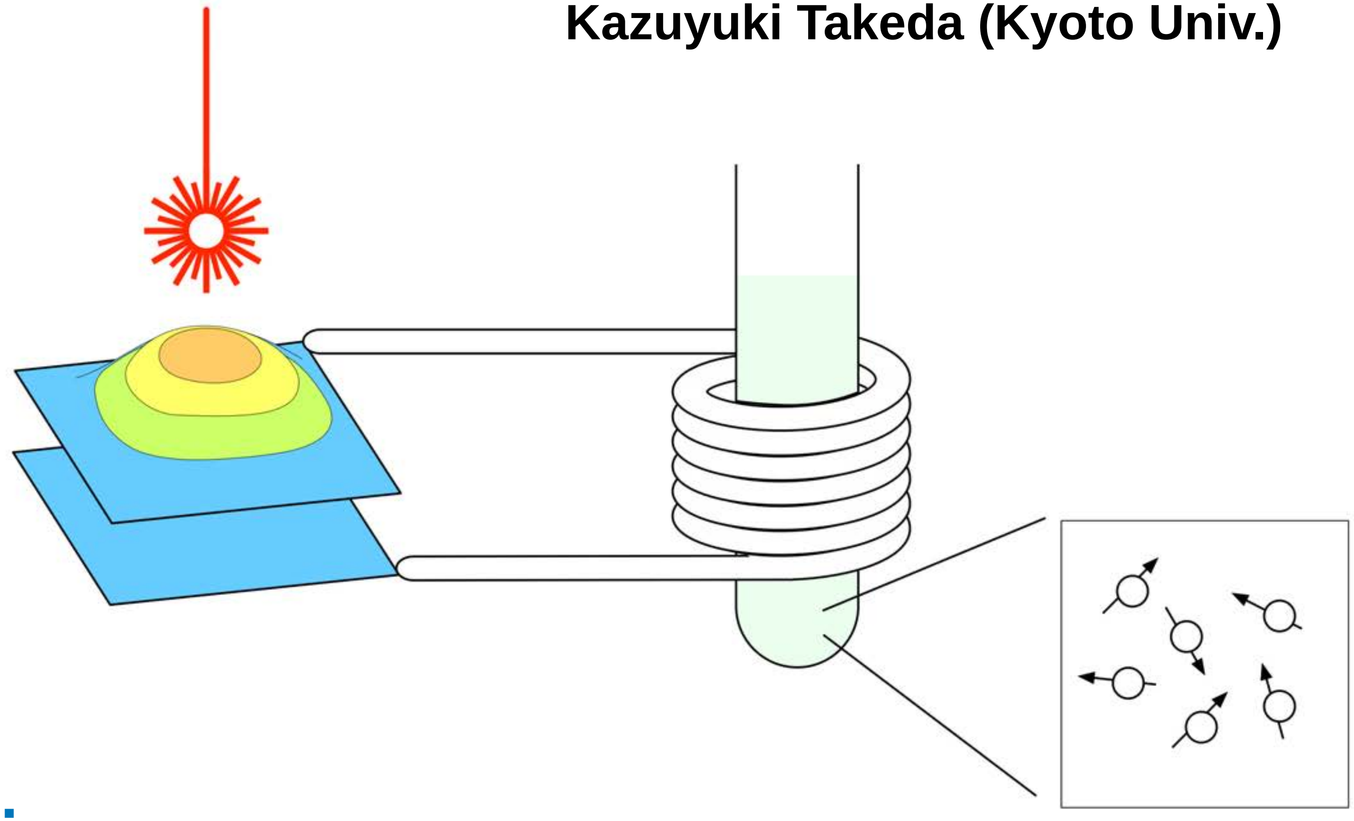
Electro-Mechano-Optical (EMO) Detection of Nuclear Magnetic Resonance

Kazuyuki Takeda (Kyoto Univ.)

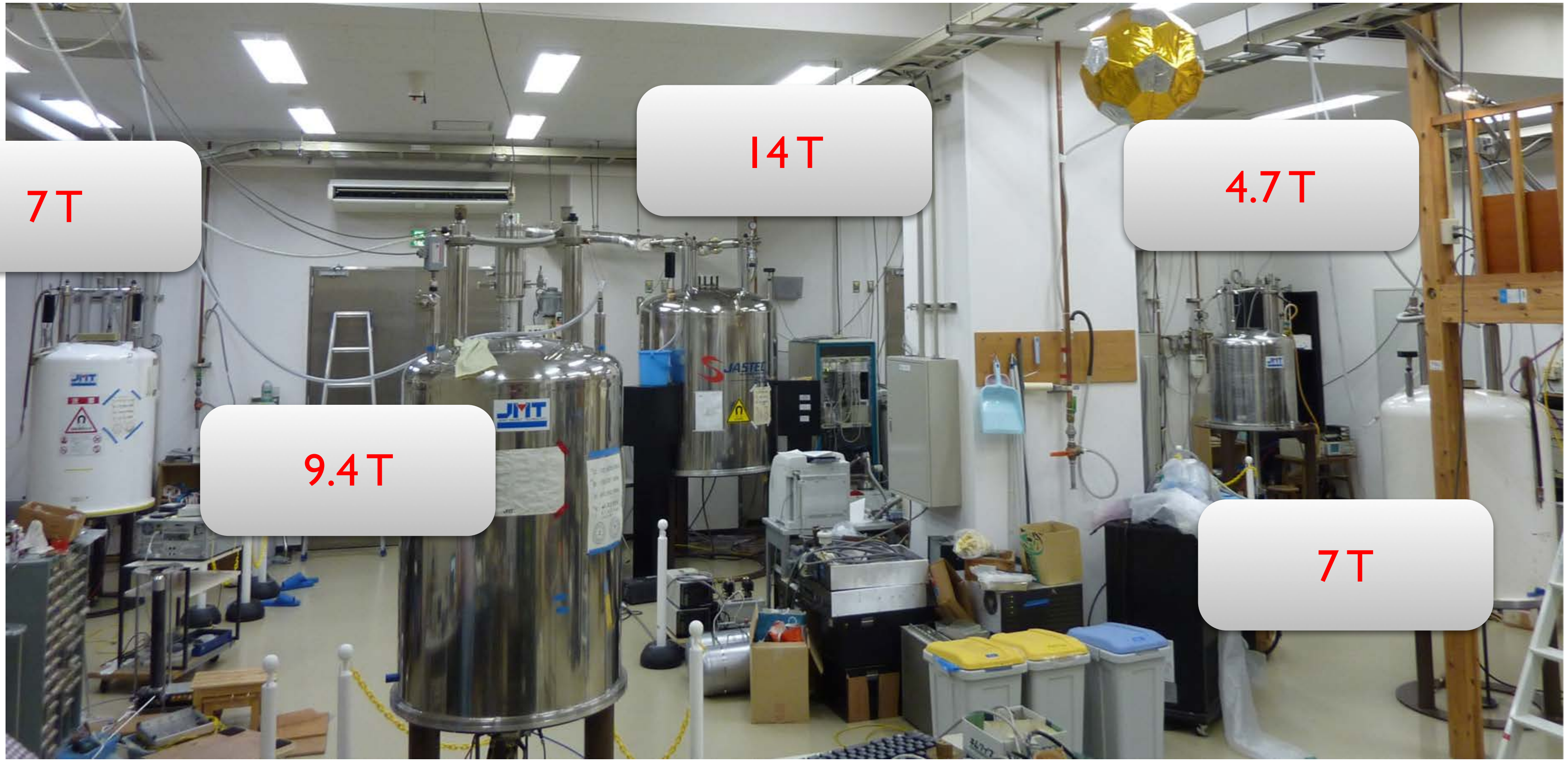


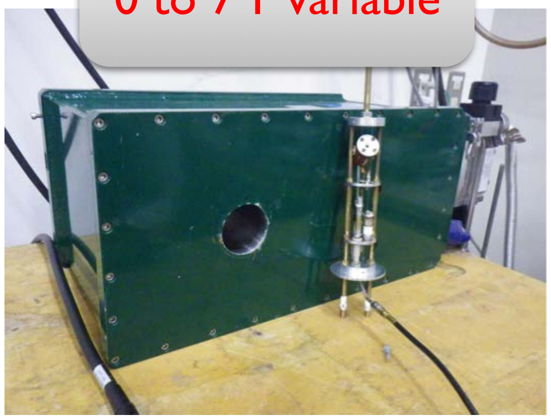


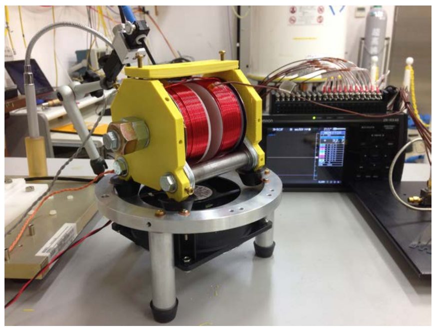
Special thanks to:

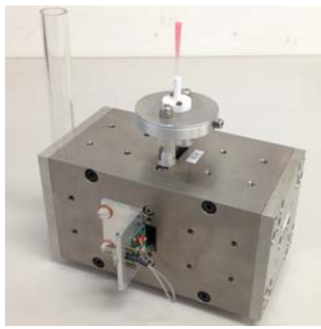
Kazuhiko Yamada, Koji Usami (Organizer), Kentaro Nagasaka, Eiji Iwase, Masato Takahashi, Atsushi Saito, Rekishu Yamazaki, Yasunobu Nakamura, Jacob Taylor, Yusuke Tominaga, Atsushi Noguchi

Solid-State NMR Lab in Division of Chemistry, Kyoto Univ.

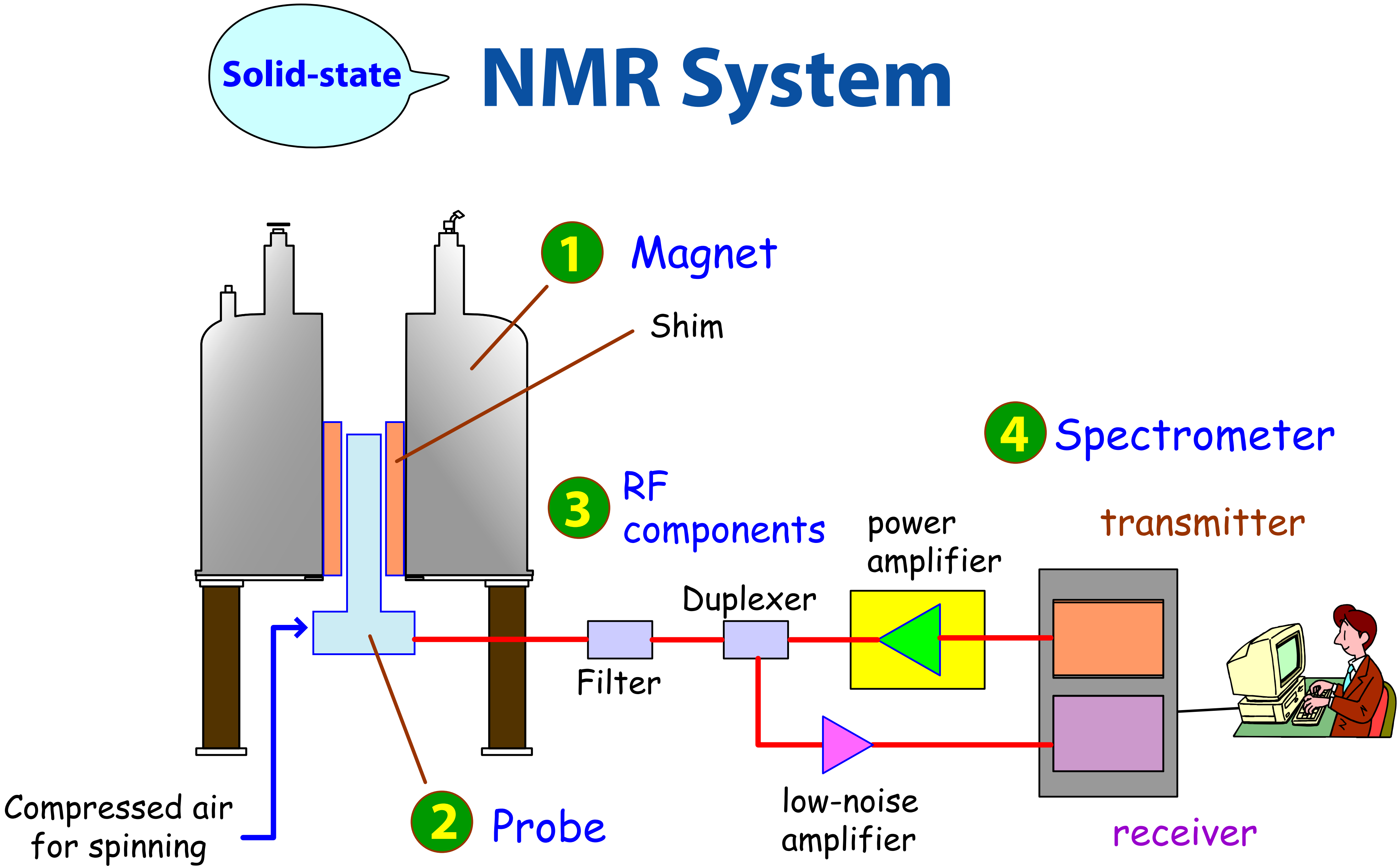




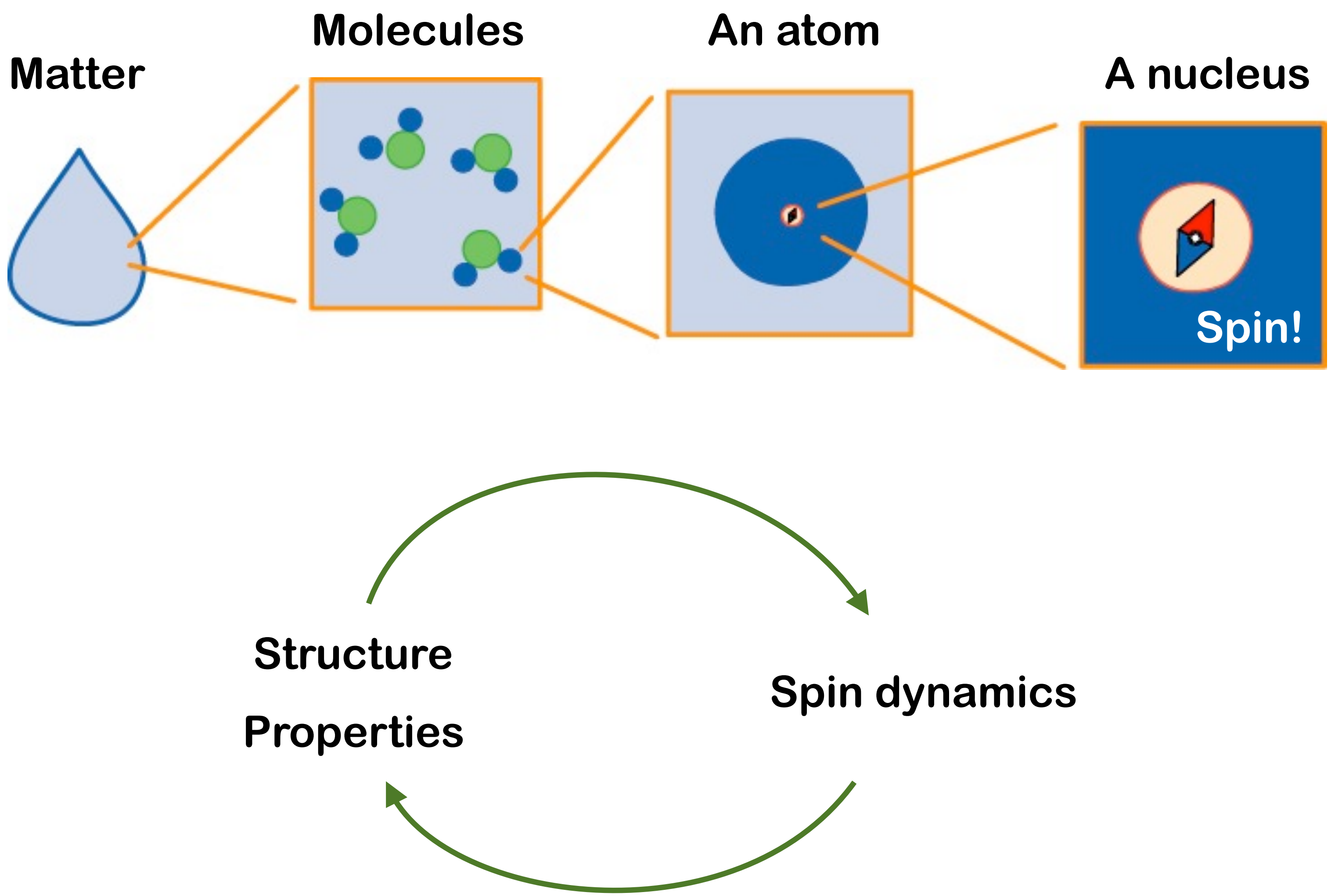




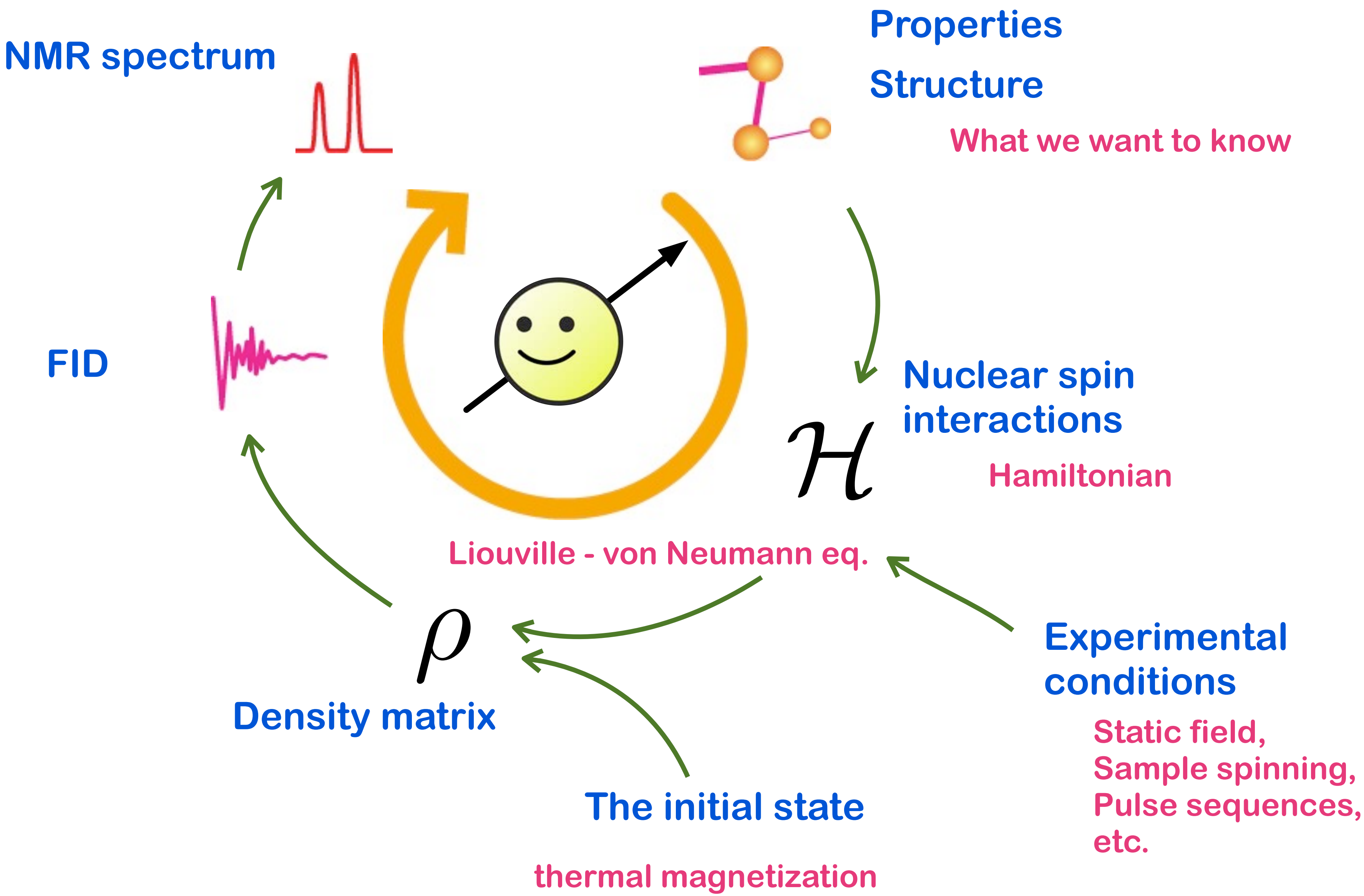
NMR System



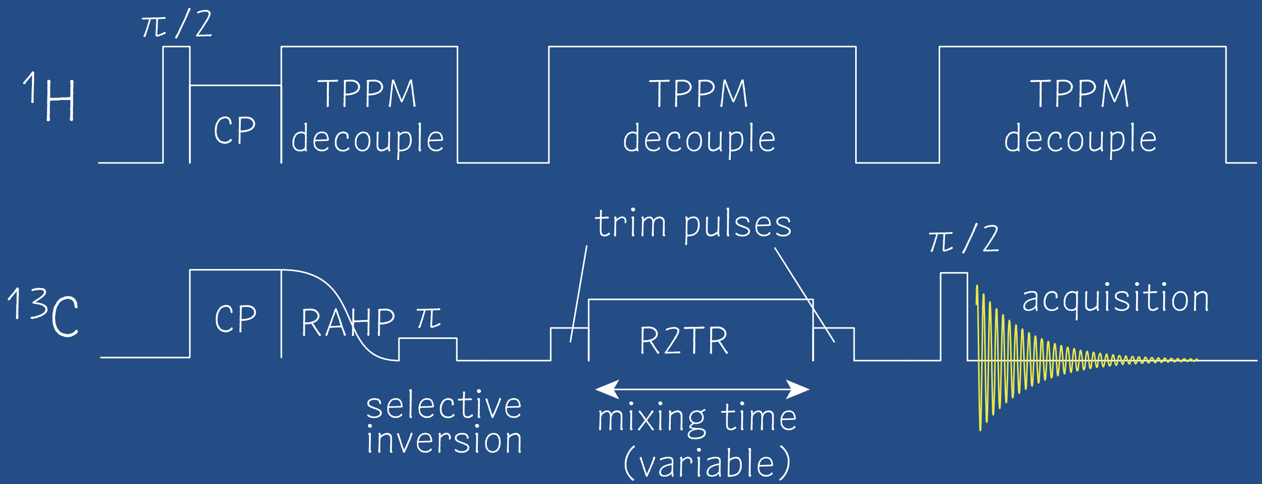
Solid-state
1 Magnet
Shim
4 Spectrometer
3 RF components
power amplifier
transmitter
Duplexer
Filter
Compressed air for spinning
2 Probe
low-noise amplifier
receiver


Matter
Molecules
An atom
A nucleus
Spin!
Structure
Properties
Spin dynamics

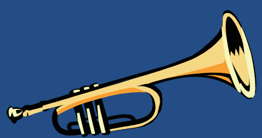
Causality between the NMR spectrum and properties of matter



An Example of Pulse Sequences



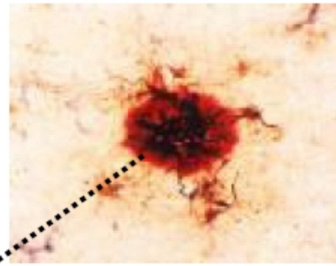
It is a bit like music composition!



Alzheimer's Desease (AD)

AD brain

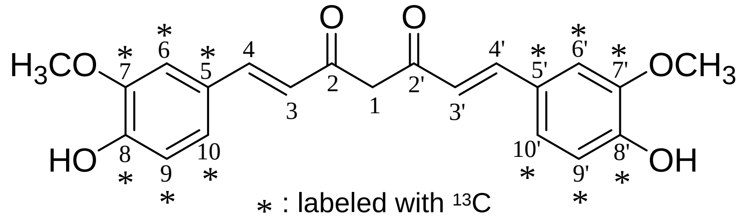
Senile plaque



Amyloid β-protein (Aβ)

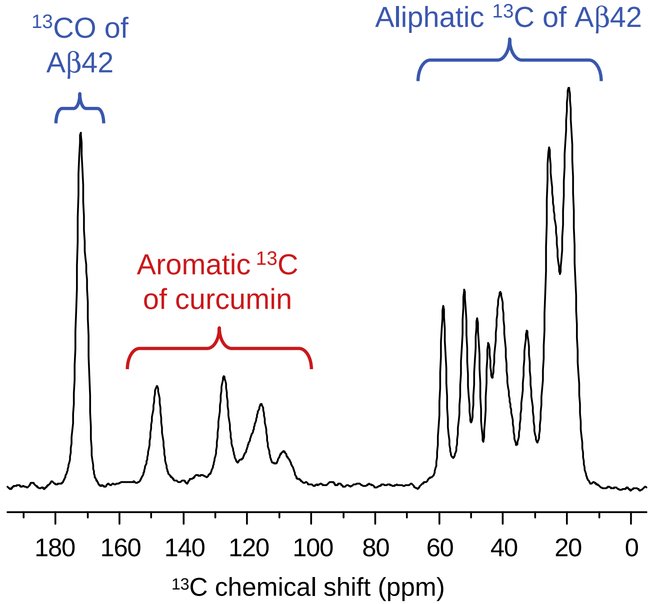
DAEFRHDSGYEVHHQKLVFFAEDVGSNKGAIIGLMVGGVVIA
1 40 42

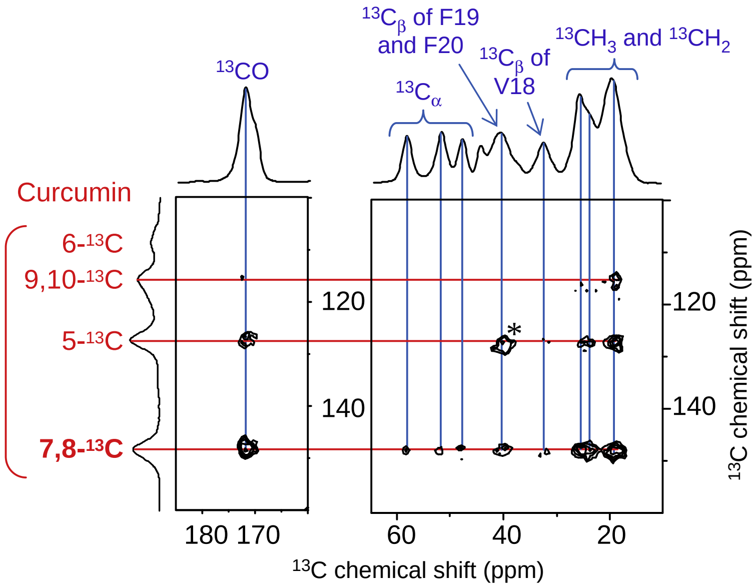
Curcumin
(Curry SPICE !)



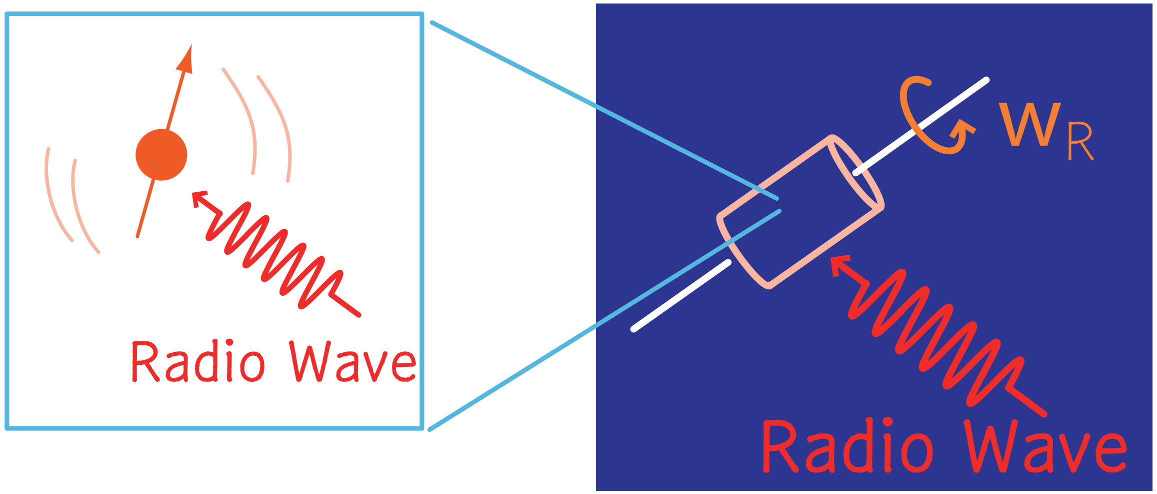
* : labeled with ¹³C

Solid-state ¹³C NMR Spectrum

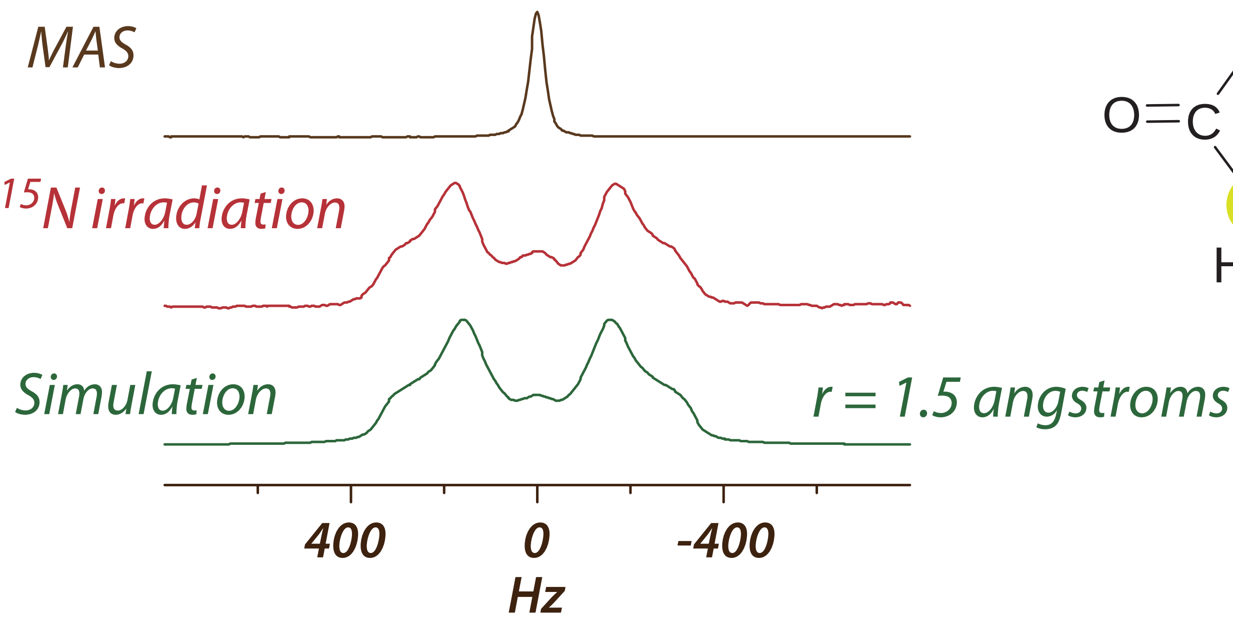


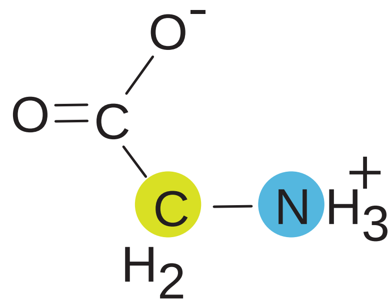


Dipolar recoupling

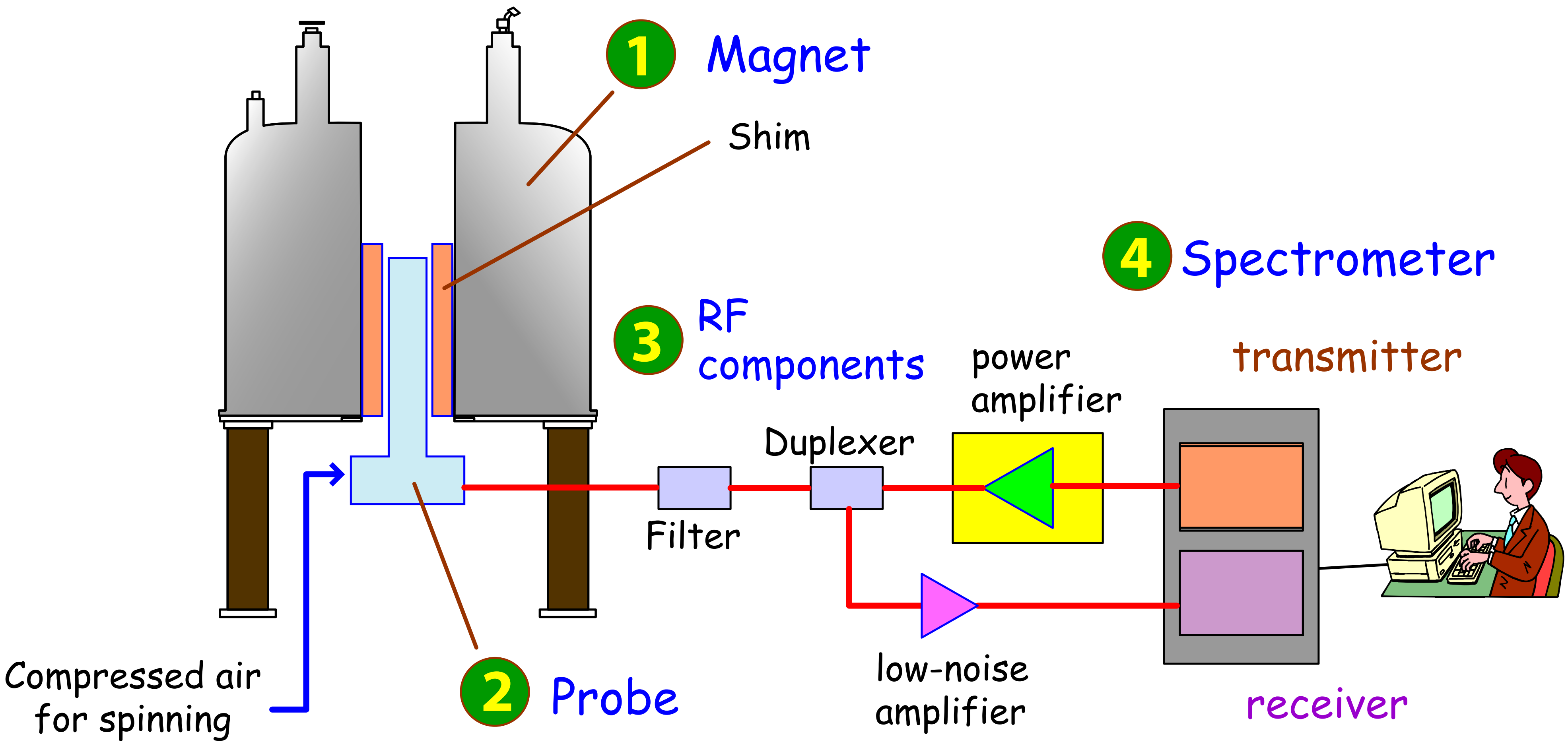


^{13}C NMR of 2-^{13}C-^{15}N-labeled Glycine





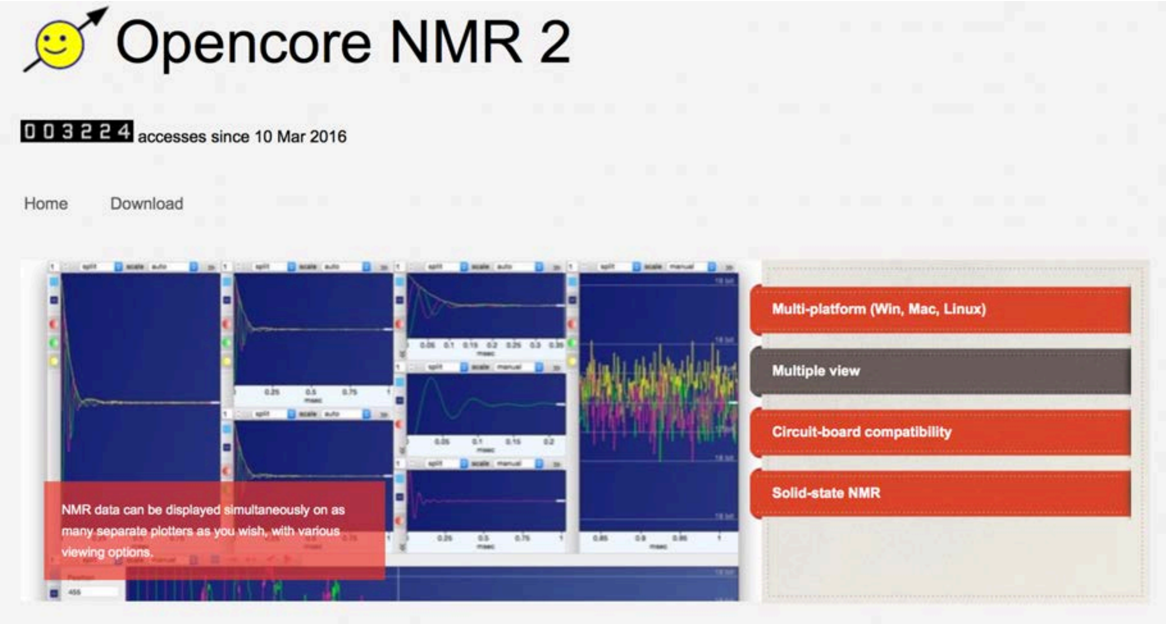
NMR System

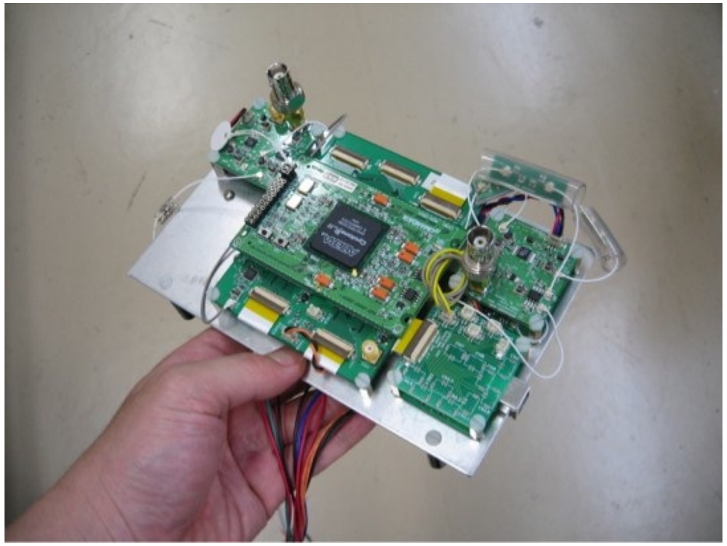


Commercial NMR systems are indeed sophisticated, but too much…

OPENCORE NMR Spectrometer

http://kuchem.kyoto-u.ac.jp/bun/indiv/takezo/opencorenmr2/index.html

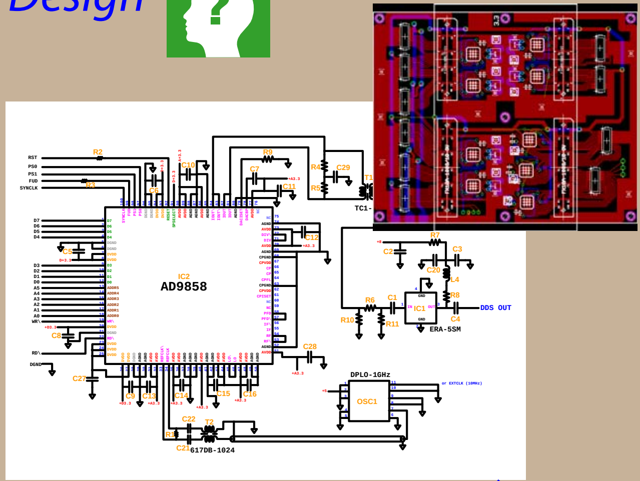




Takeda, Review of Scientific Instruments, 78 (2007) 033103.
Takeda, JMR, 192 (2008) 218-229.
Takeda, Annual Reports on NMR, 74 (2011) 355-393.

Design

CAD



Board manufacturing

Parts mounting & soldering

Garbage boards

Do over!

Eventually...

Productions

#1

#2

#3

K. Takeda

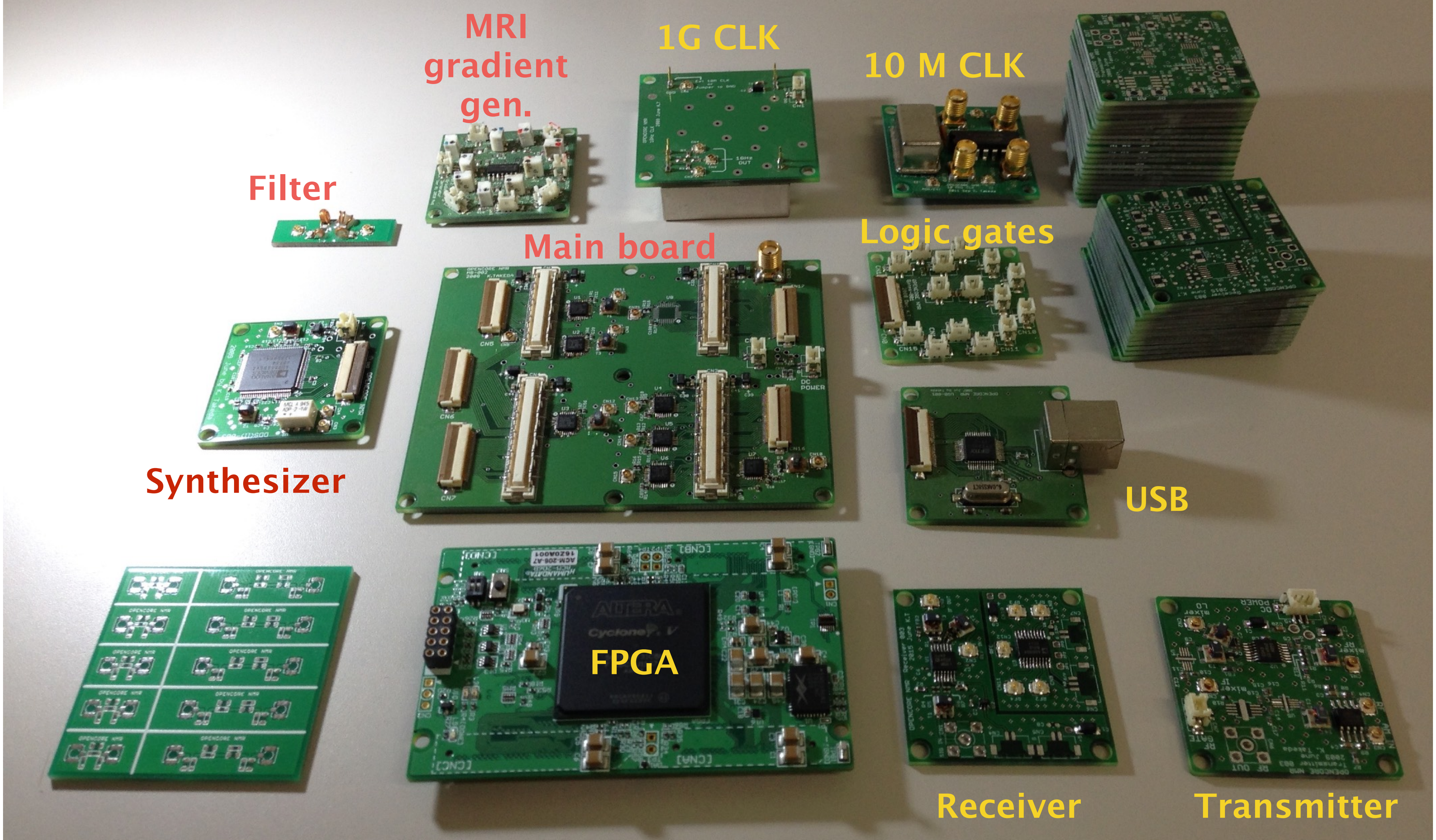
Rev. Sci. Instrum. 78, 033103 (2007)

J. Magn. Reson., in press.

Design available on the web:

http://kuchem.kyoto-u.ac.jp/bun/indiv/takezo/opencorenmr

NMR Spectrometer Modules

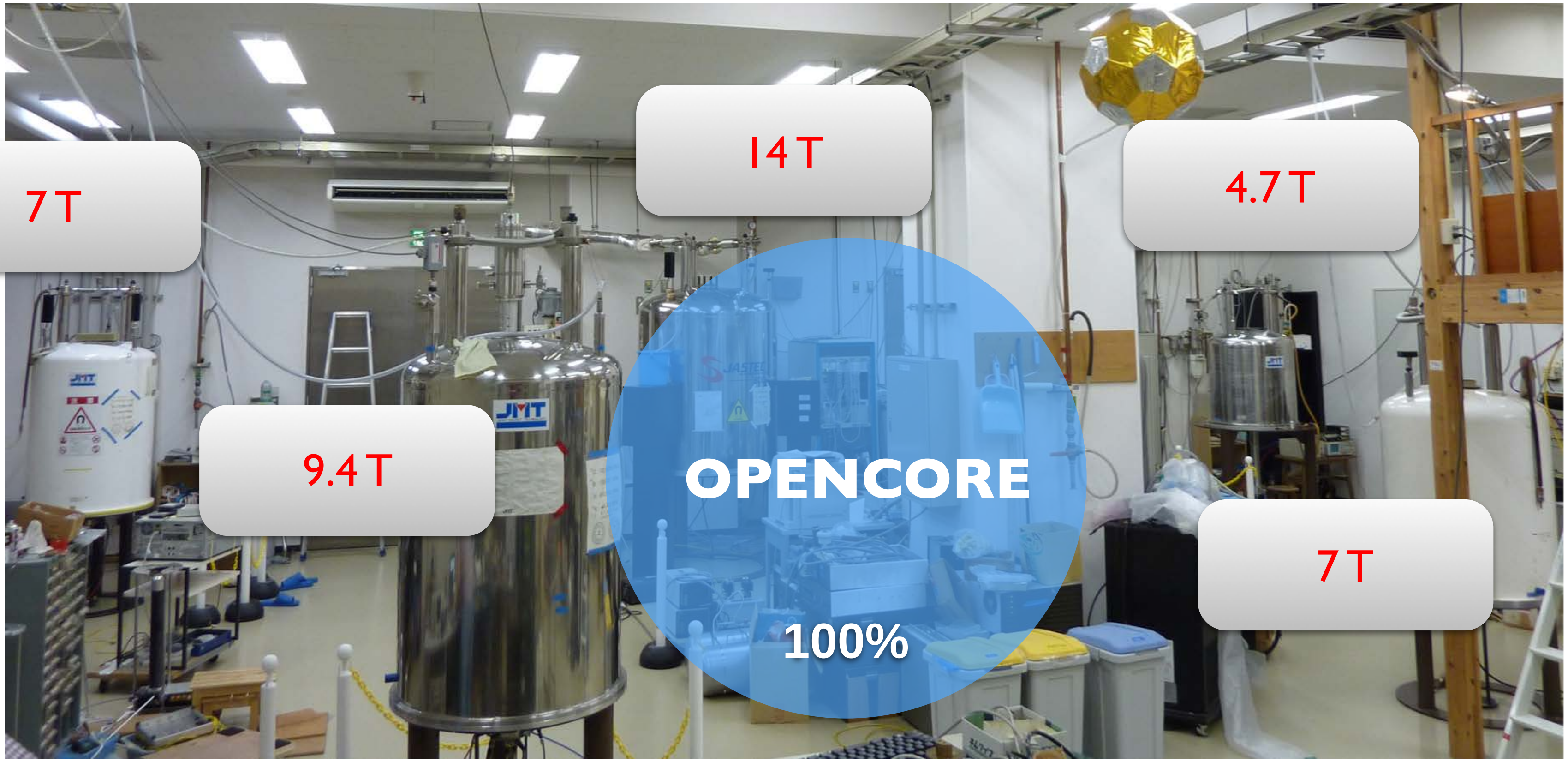


Board drawings are available.

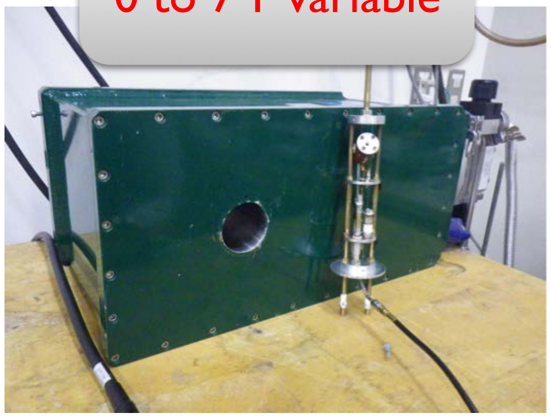
Specs

- 3 rf channels (synchronous & asynchronous modes)
- amplitude, phase, frequency modulation (up to ~600 MHz)
- minimum pulse width: 25 ns
- mininum pulse-width step: 6.25 ns
- 80 Ms ADC
- digital quadrature demodulation
- FPGA integration

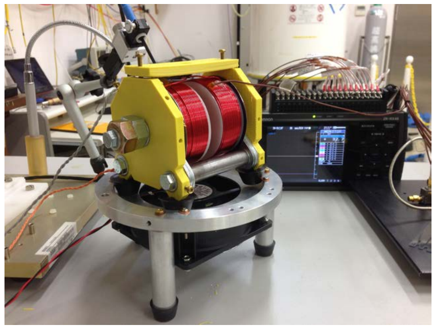
Our NMR systems are running on…



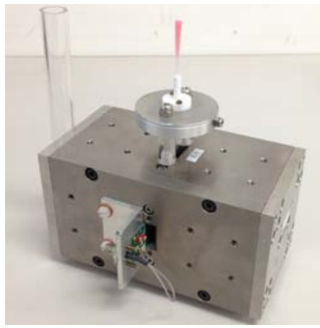
0 to 7 T variable



(partially) 3D-printed electromagnet

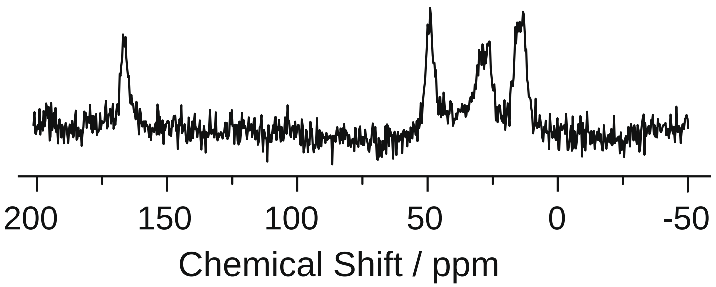


1 T permanent

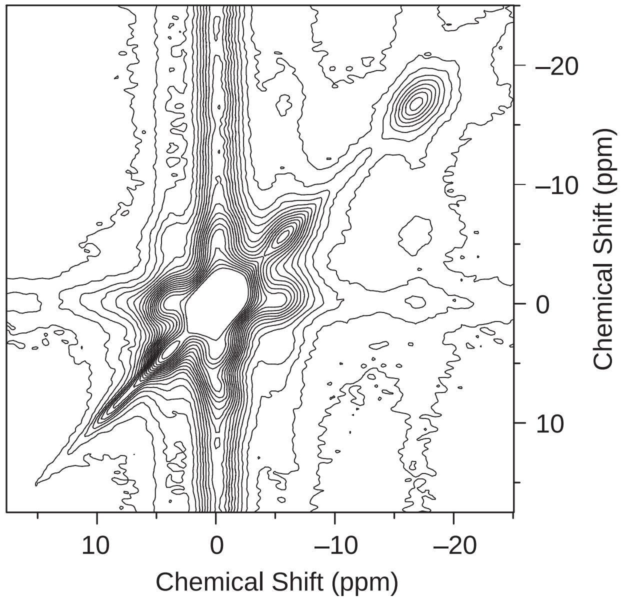


Sensitivity demanding experiments

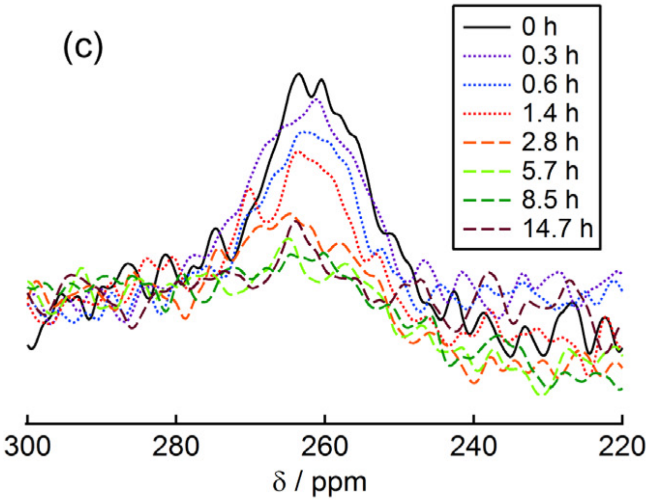
- ^{13}C microcoil CPMAS & TPPM decouple in Aβ42 (~0.1 mg)

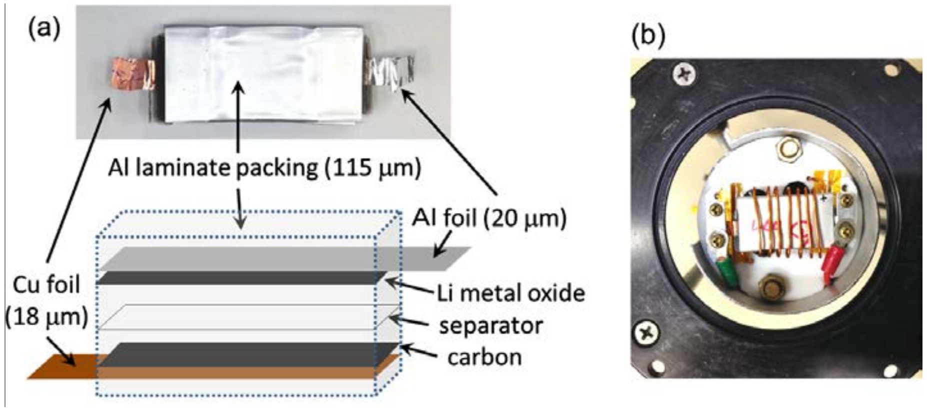


- ^{6}Li exchange in ^{6}Li-labeled $LiCoO_2$ (cryo-coil MAS)



- in-situ ^{7}Li NMR of thin-film batteries





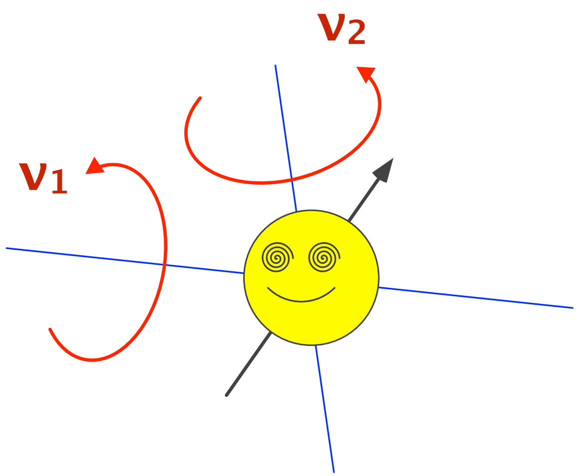
Journal of The Electrochemical Society, 162 (2015) A952

Carbon 79 (2014) 380

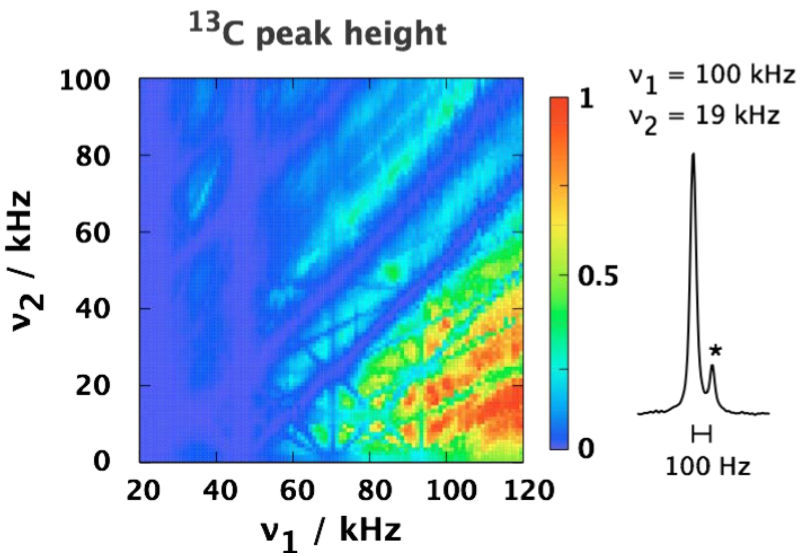
rf modulation

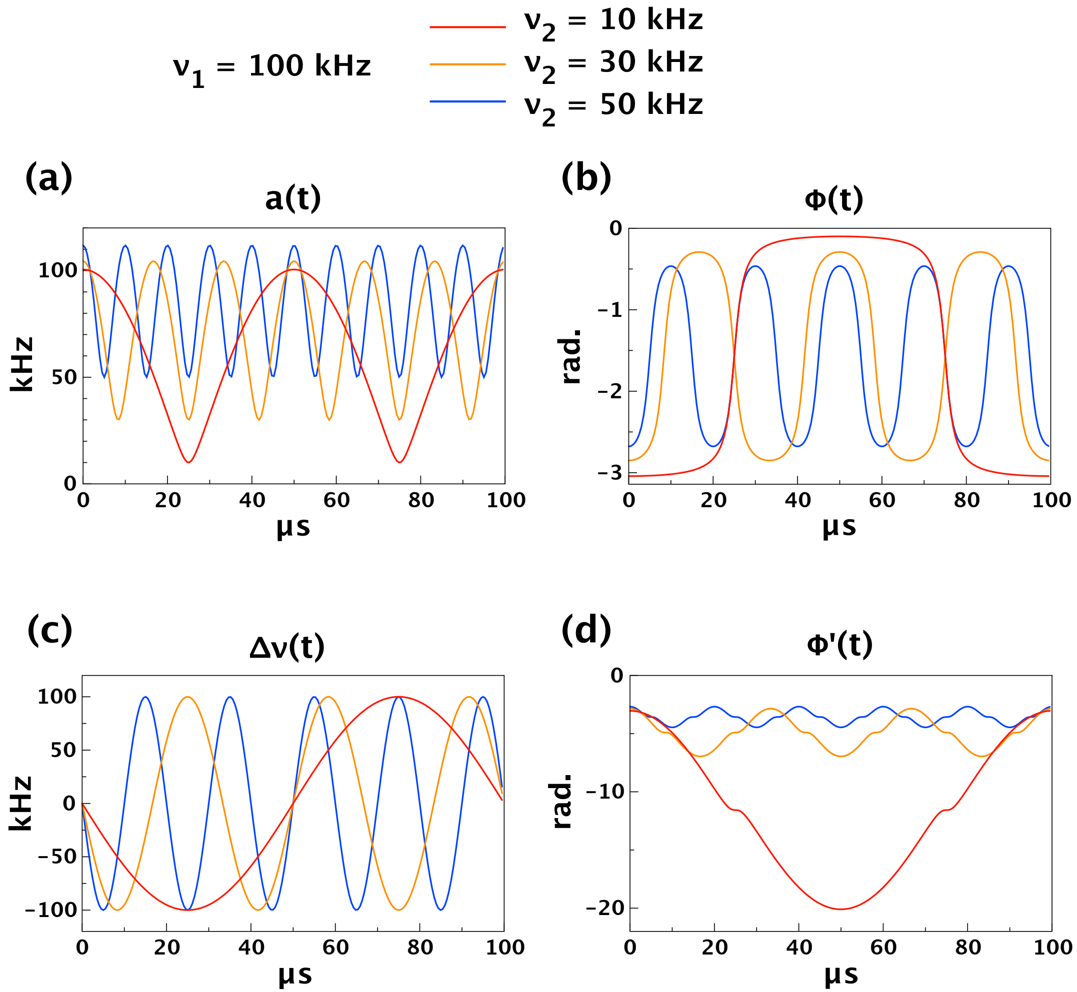
Double Nutation (DONUT) ^{1}H decoupling

Takeda, Wakisaka, Takegoshi,
Journal of Chemical Physics, 141 (2014) 224202



$$U = e^{2\pi i \nu_2 t I_y} e^{2\pi i \nu_1 t I_x}$$



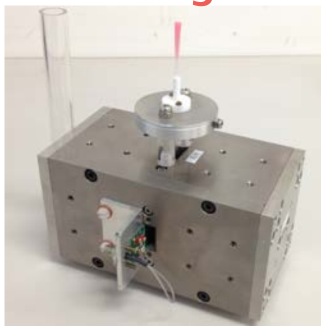


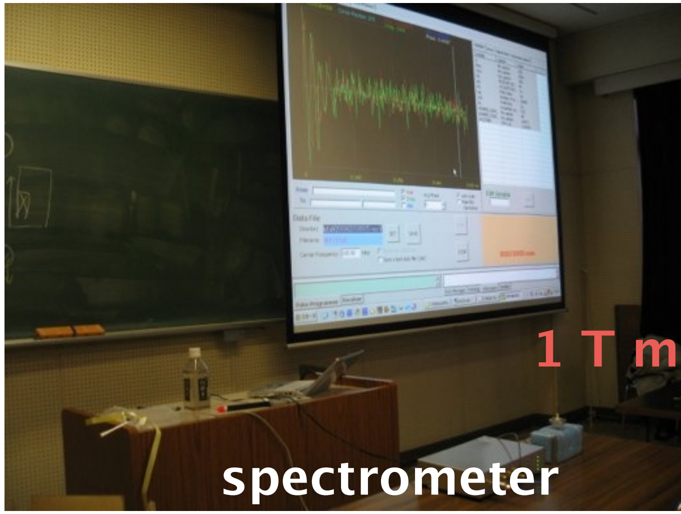
Portable hand-made NMR

NMR in a hospital

NMR in a classroom

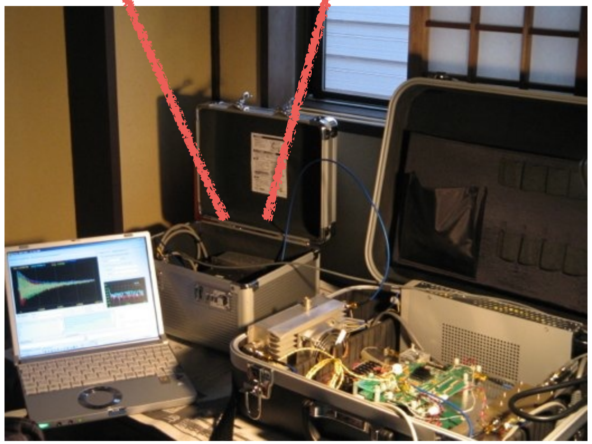
1 T magnet

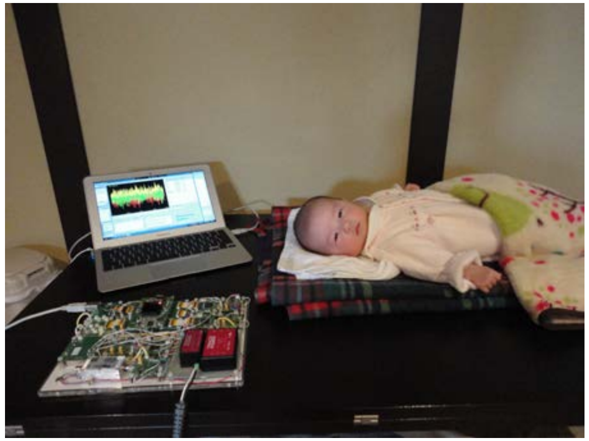




1 T magnet

NMR at home

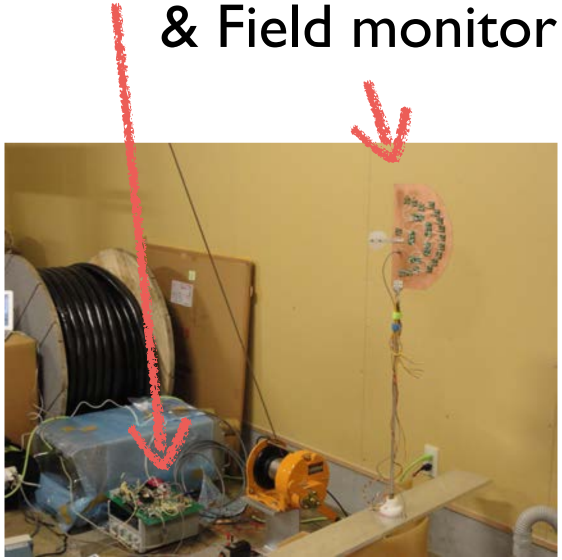




^{1}H FID in water

Application to MRI

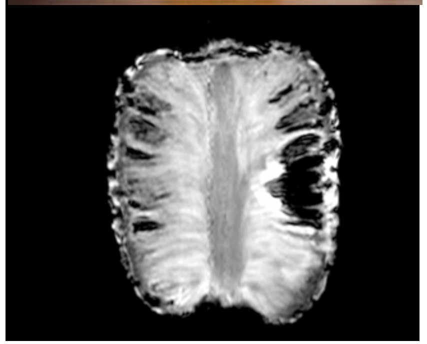
Spectrometer
& Field monitor



Birdcage coil

500 mm bore high-Tc SCM
(Bi-2223 tape conductor)

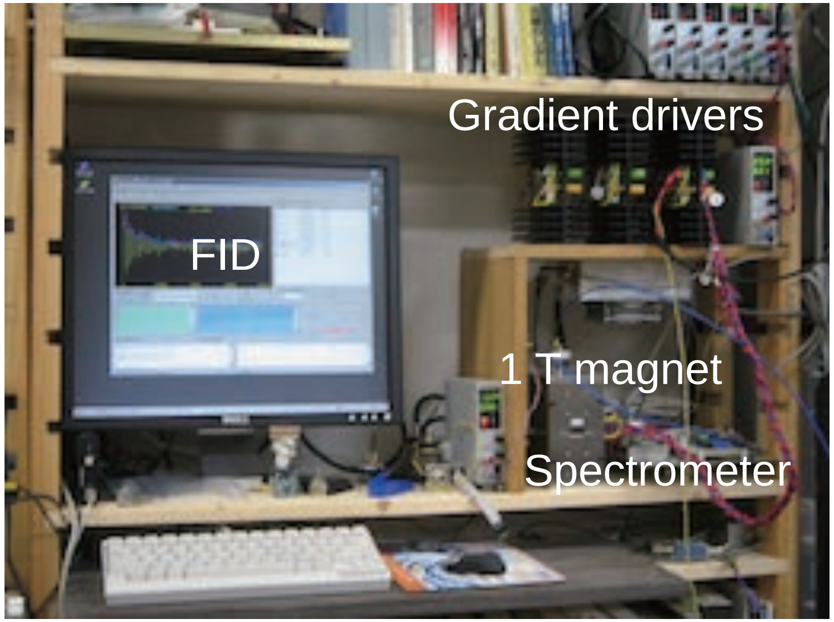
pineapple

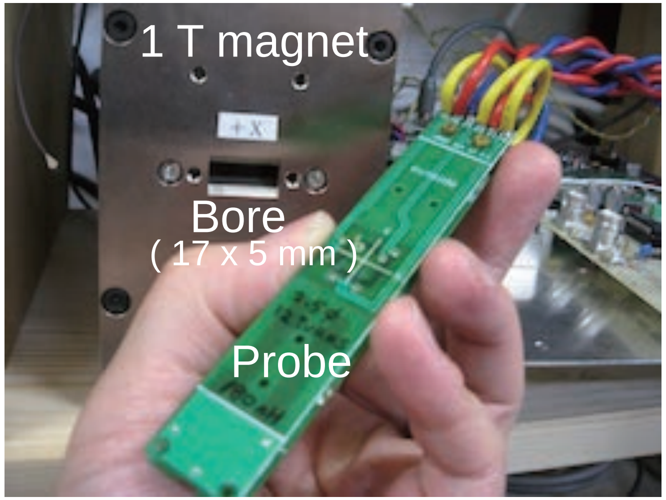


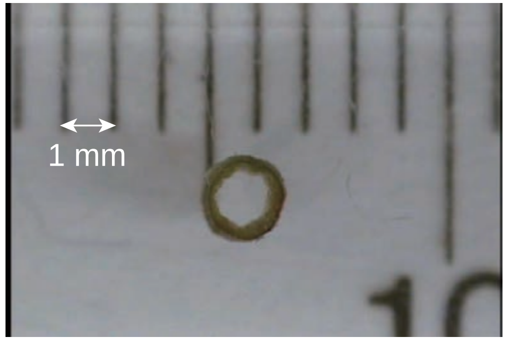
Macaca brain

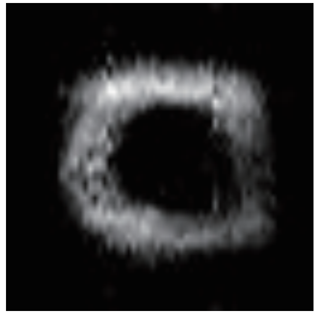
IEEE Transaction on Applied Superconductivity 23 (2013) 4400904

Bookshelf NMR / MRI

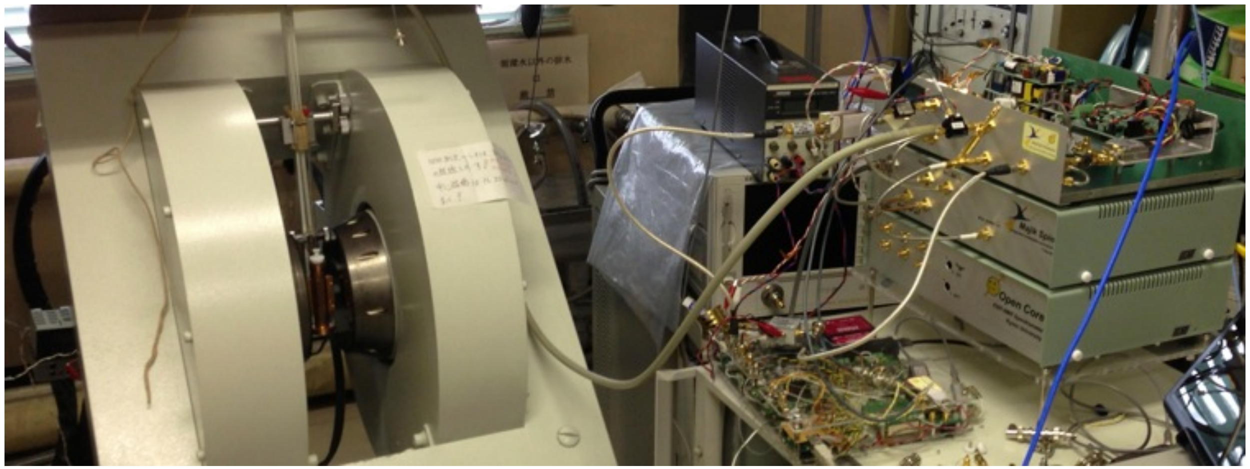


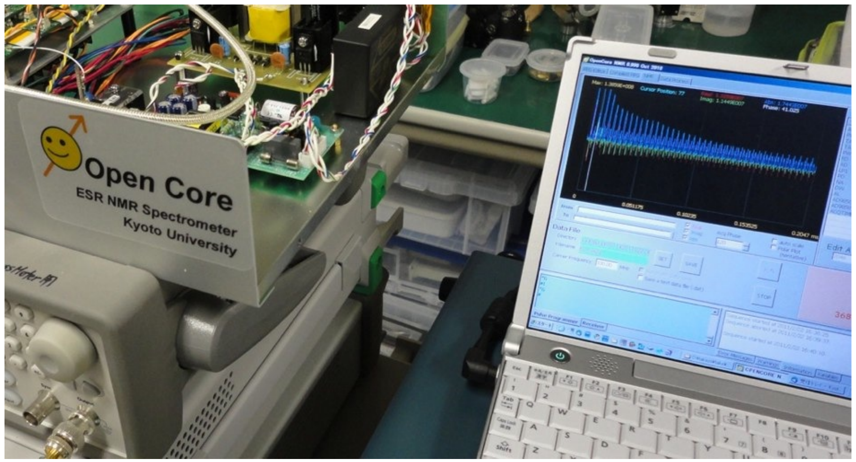






Extension to X-band ESR





In short…

We hack NMR to hack nuclear-spin dynamics.

Challenge in NMR

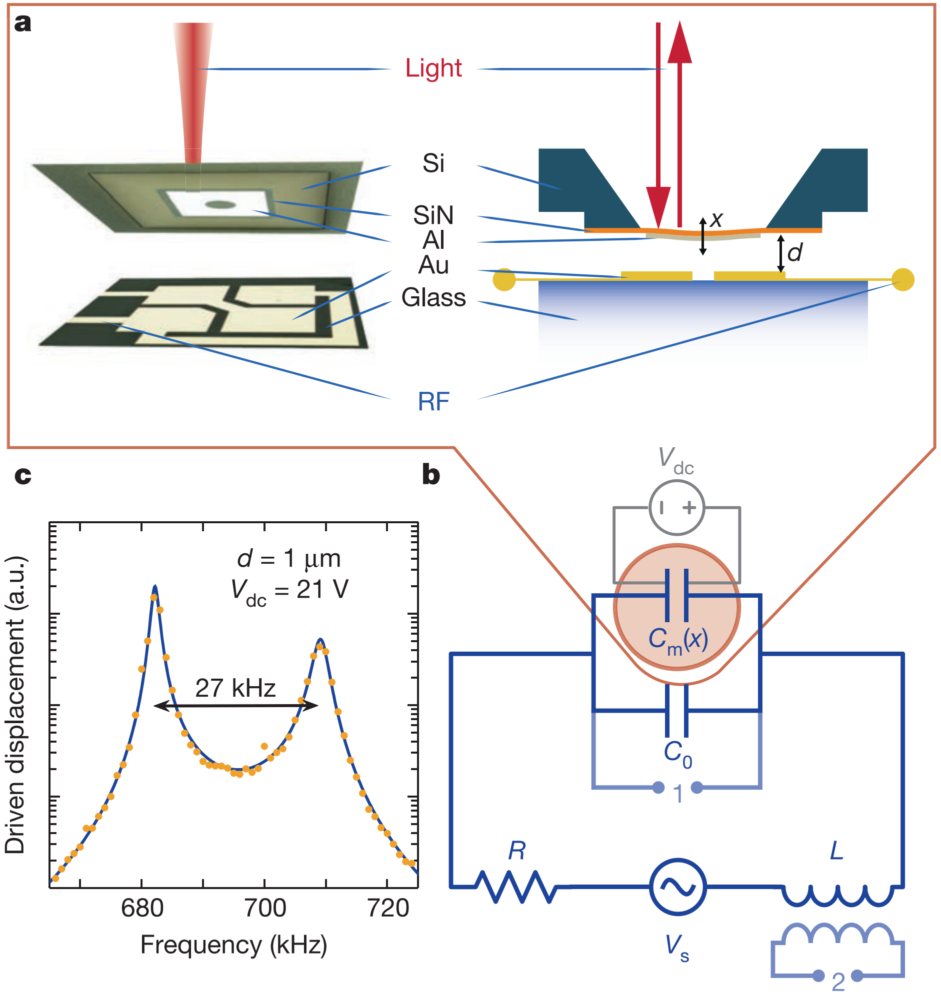
Low Sensitivity !

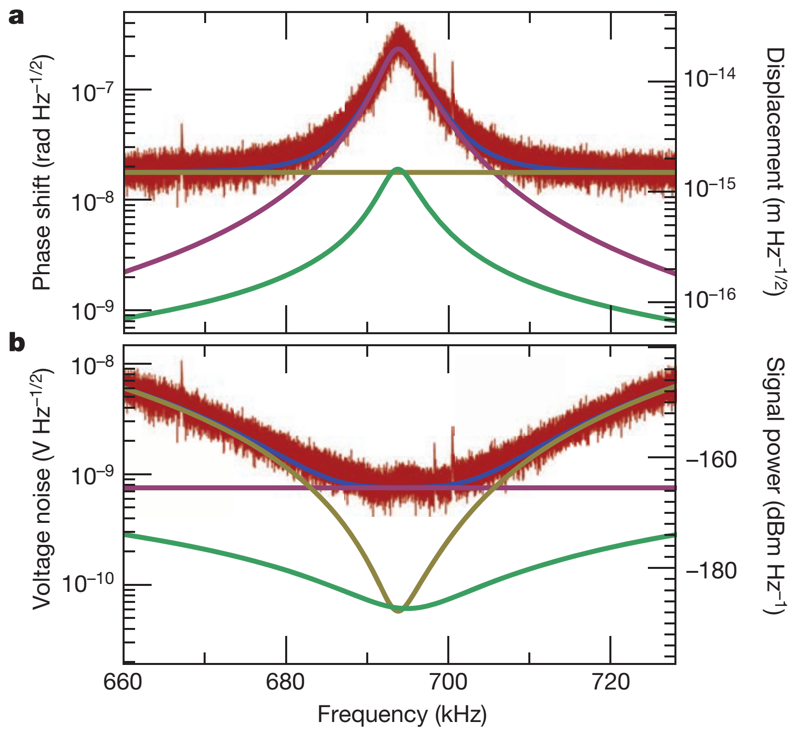
Innovations toward better sensitivity are welcome.

- **Nuclear Hyperpolarization**
- **Ultra high-field**
- **Cryo-probe**
- **…**

RF-to-light up-conversion via SiN membrane

T. Bagci et al., Nature **507**, 81 (2014)

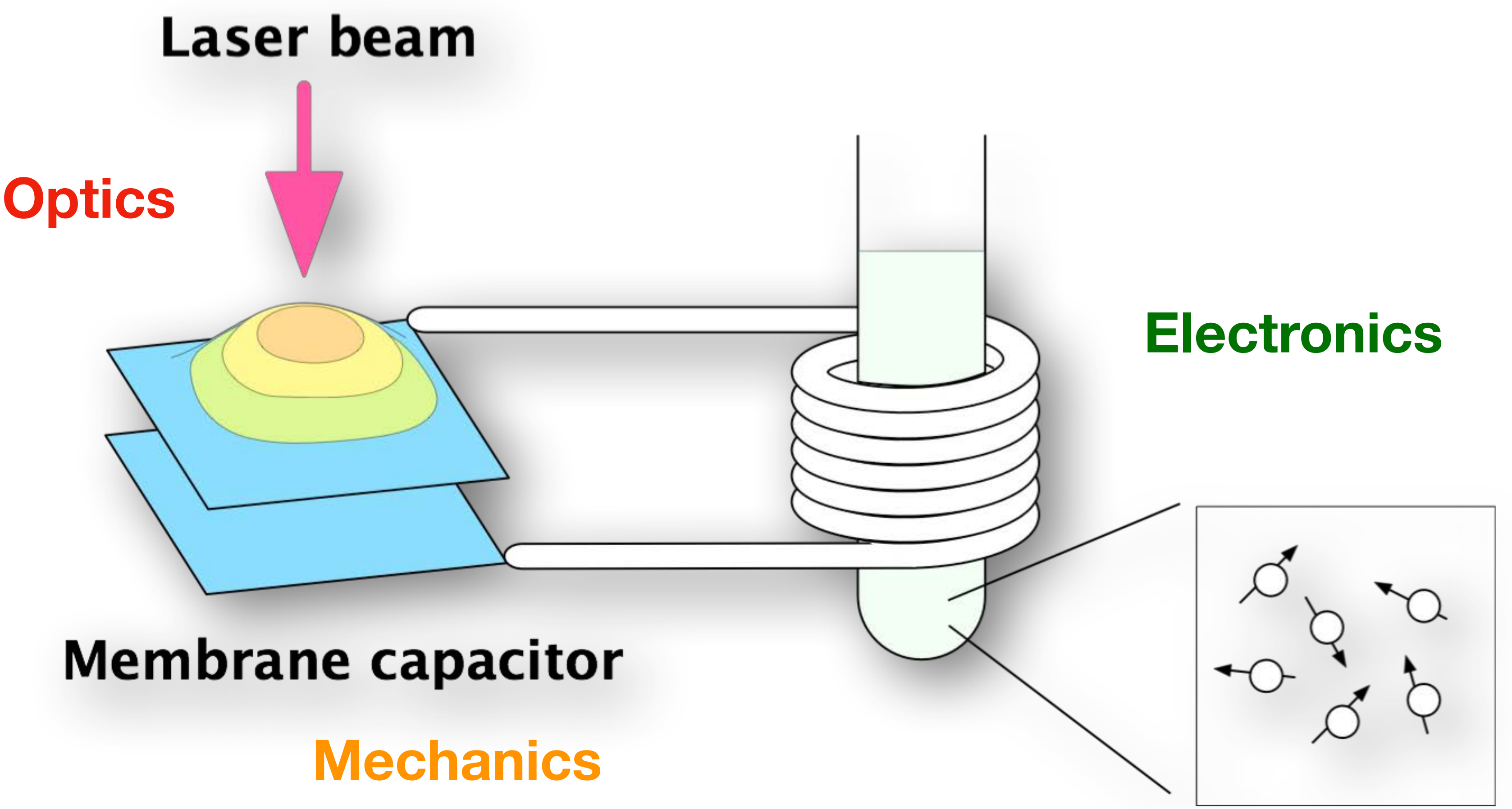




Potential applications to:

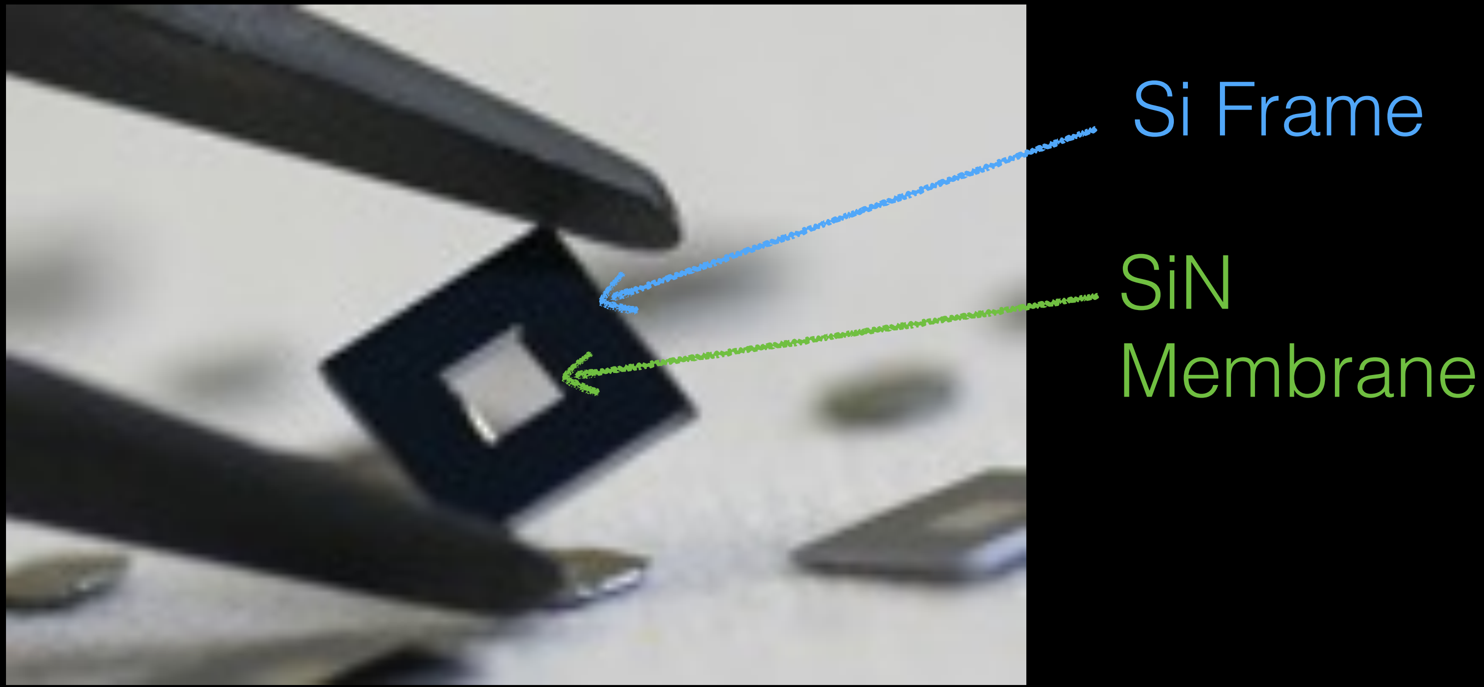
- radio astronomy
- magnetic resonance!

Toward application to NMR…



➪ ***Electro-Mechano-Optical (EMO) NMR***

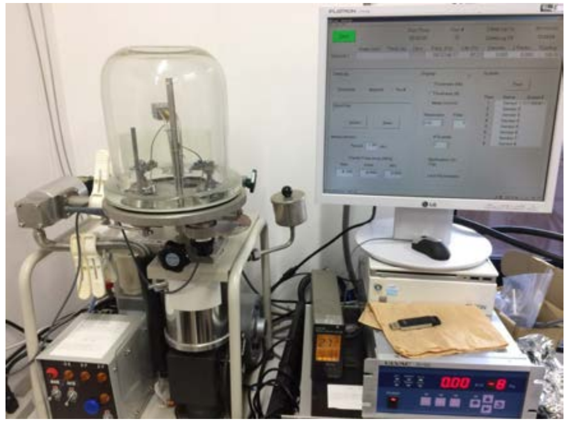
😅 Development of the EMO NMR system



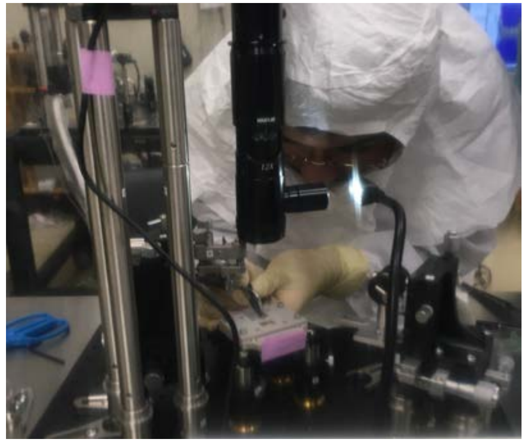
SiN membranes *are* available,
but membrane capacitors are *not*.

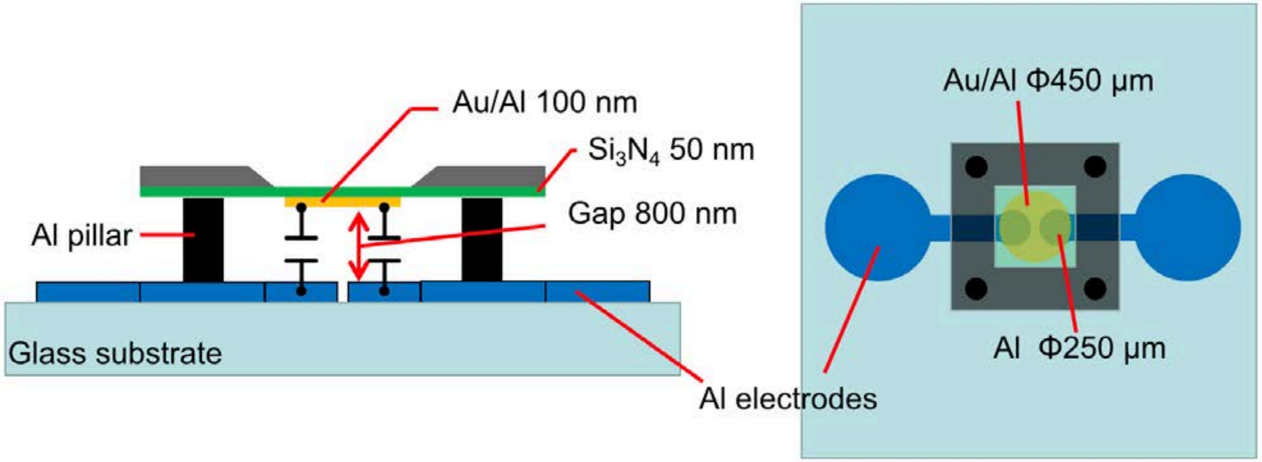
Design and fabriaction of membrane capacitors

vacuum deposition



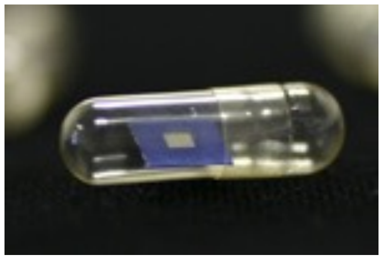
assembly

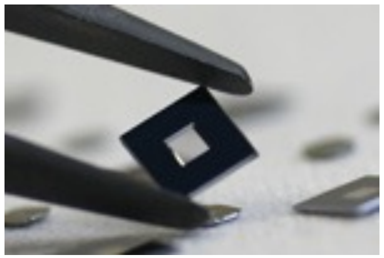




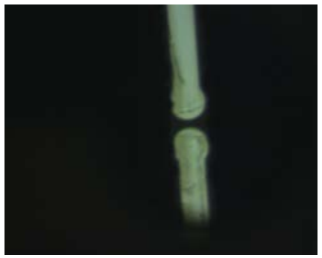
C ~ 0.1 pF

Stoichiometric SiN membrane (Norcada inc.)





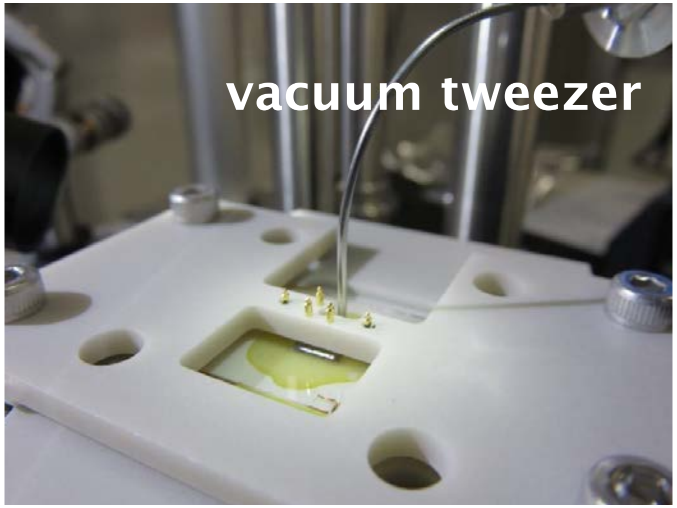
electrodes



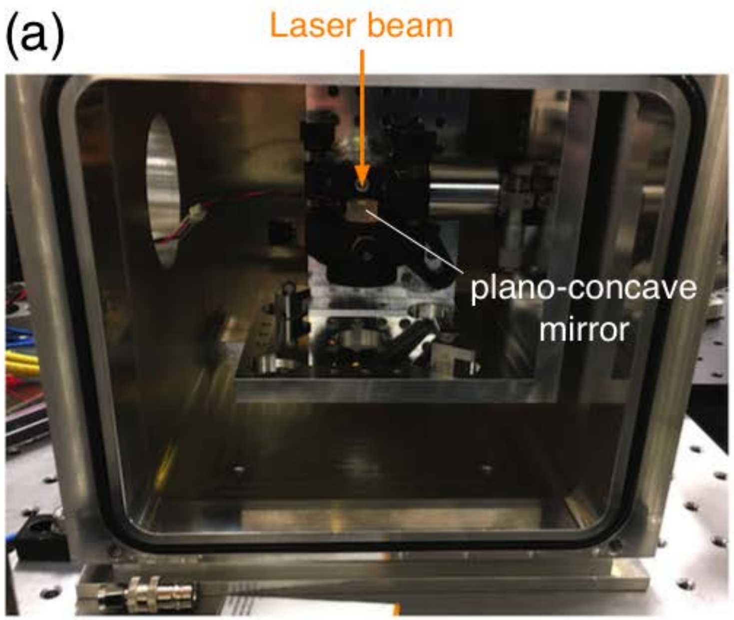
$$\omega_m/2\pi \approx 180 \sim 440 \text{ kHz}$$

Assembly in clean environment

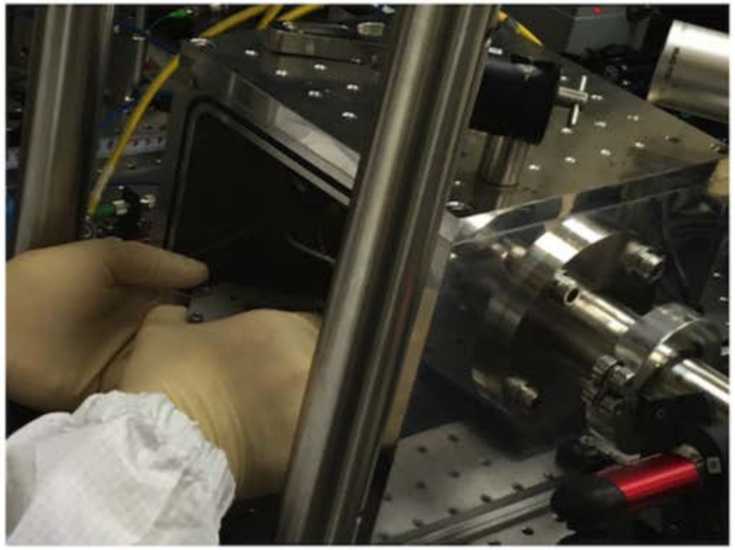
Membrane insertion



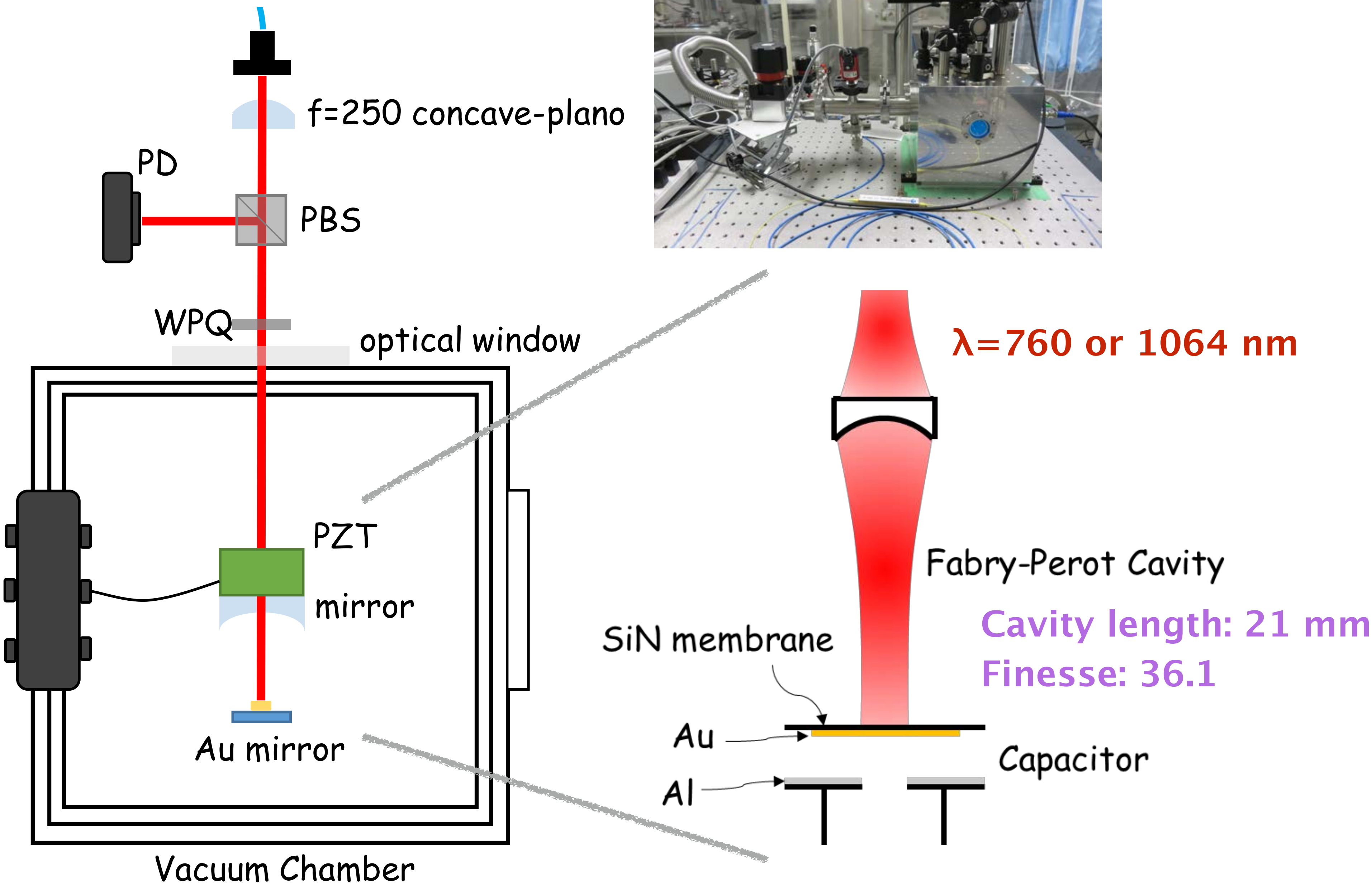
(a)



(b)

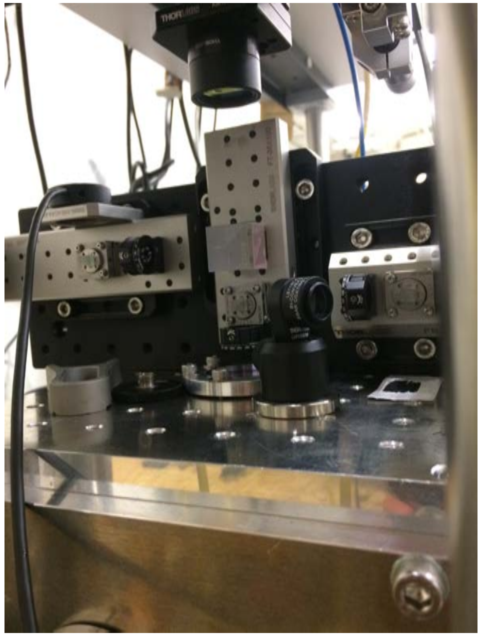


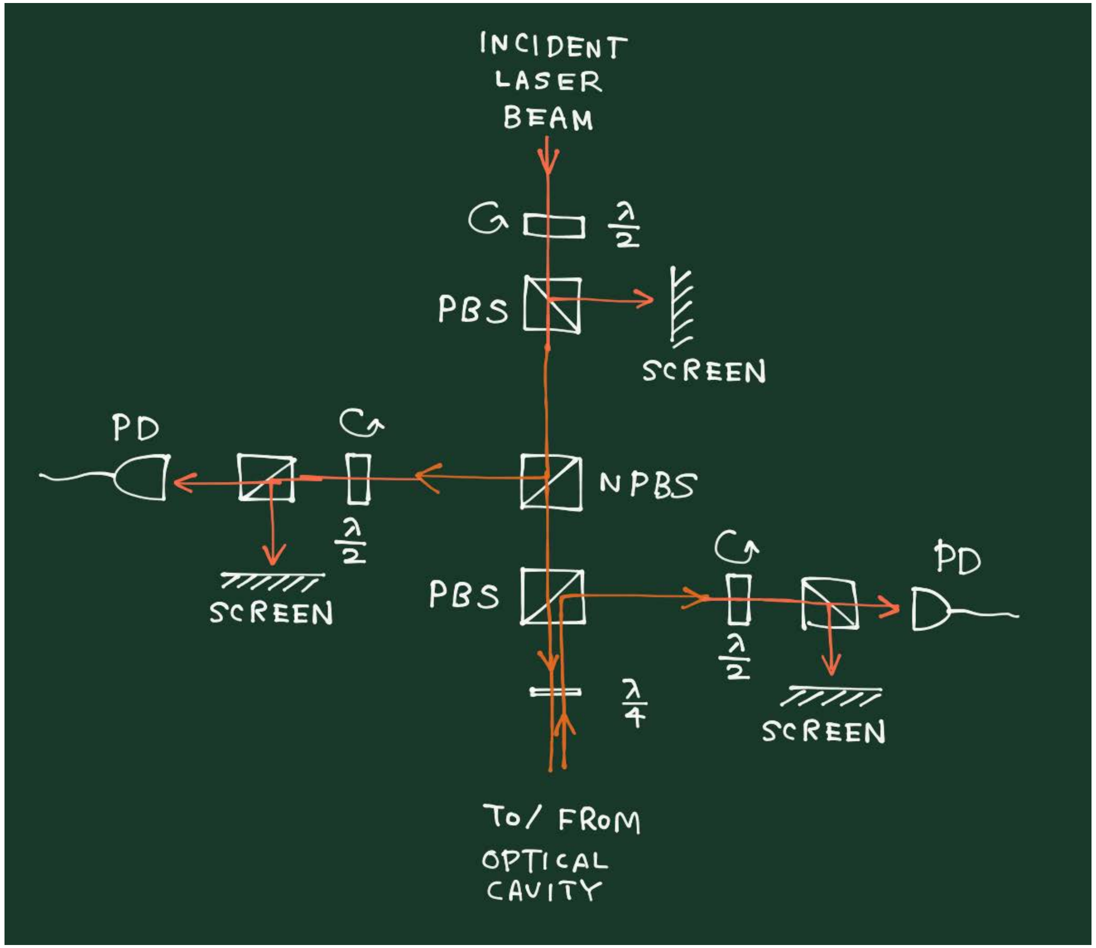
Optical Setup



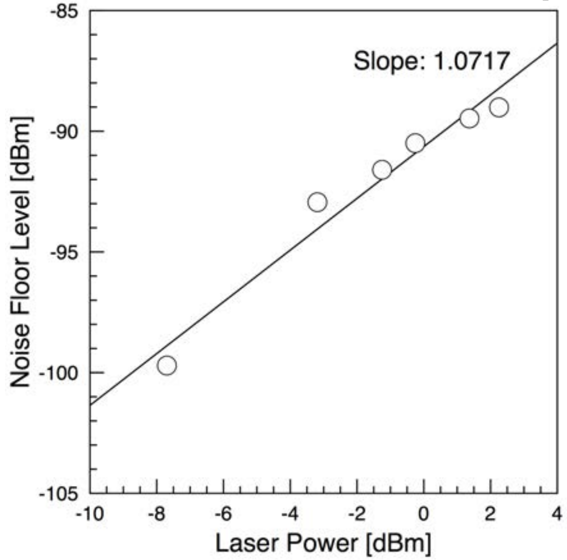
(to avoid air-damping)

Differential optical measurement



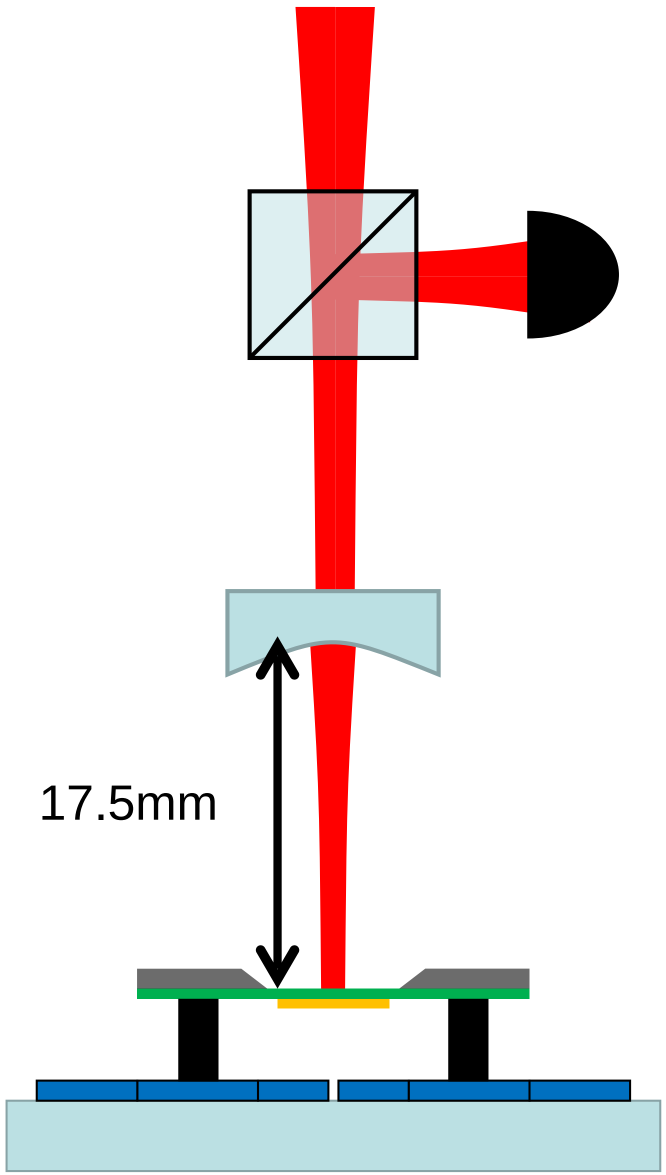


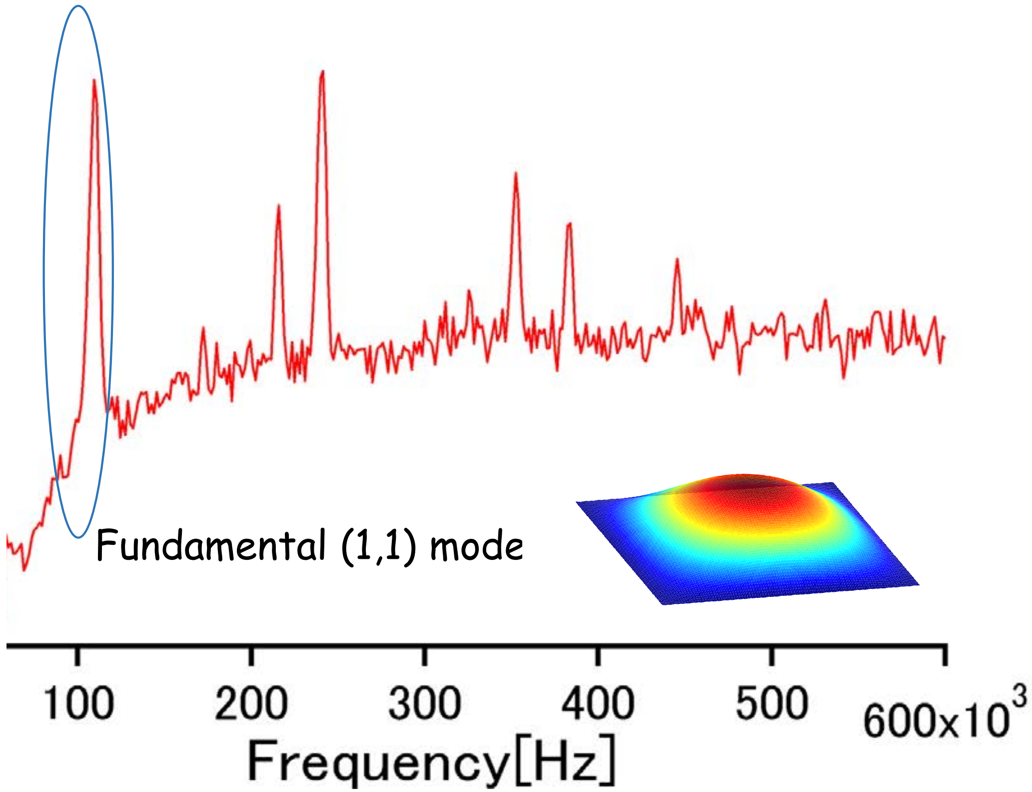
Noise foor level vs laser power

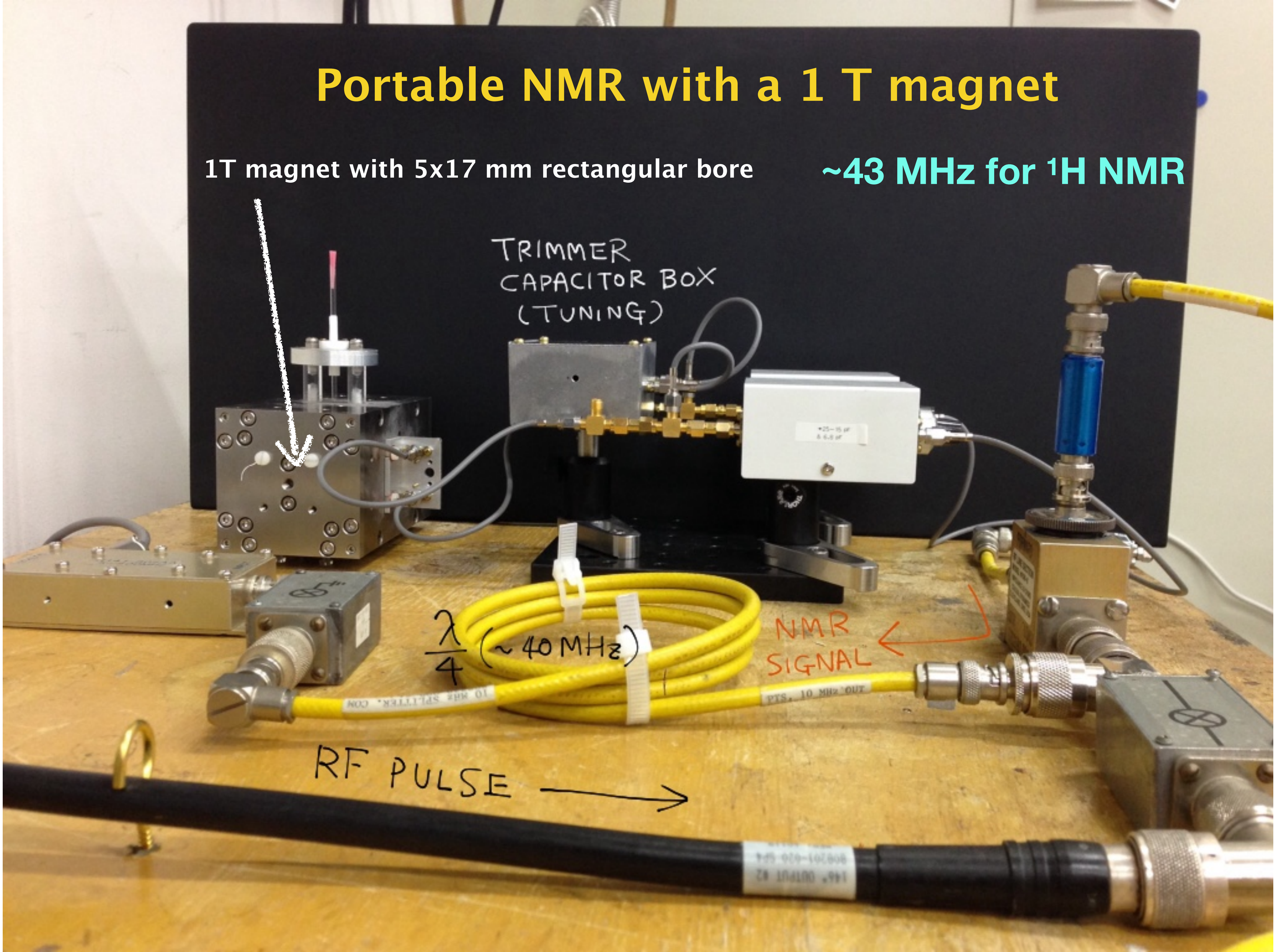


Slope 1 corresponds to shot-noise-limited measurement

Optically-detected thermal membrane oscillation

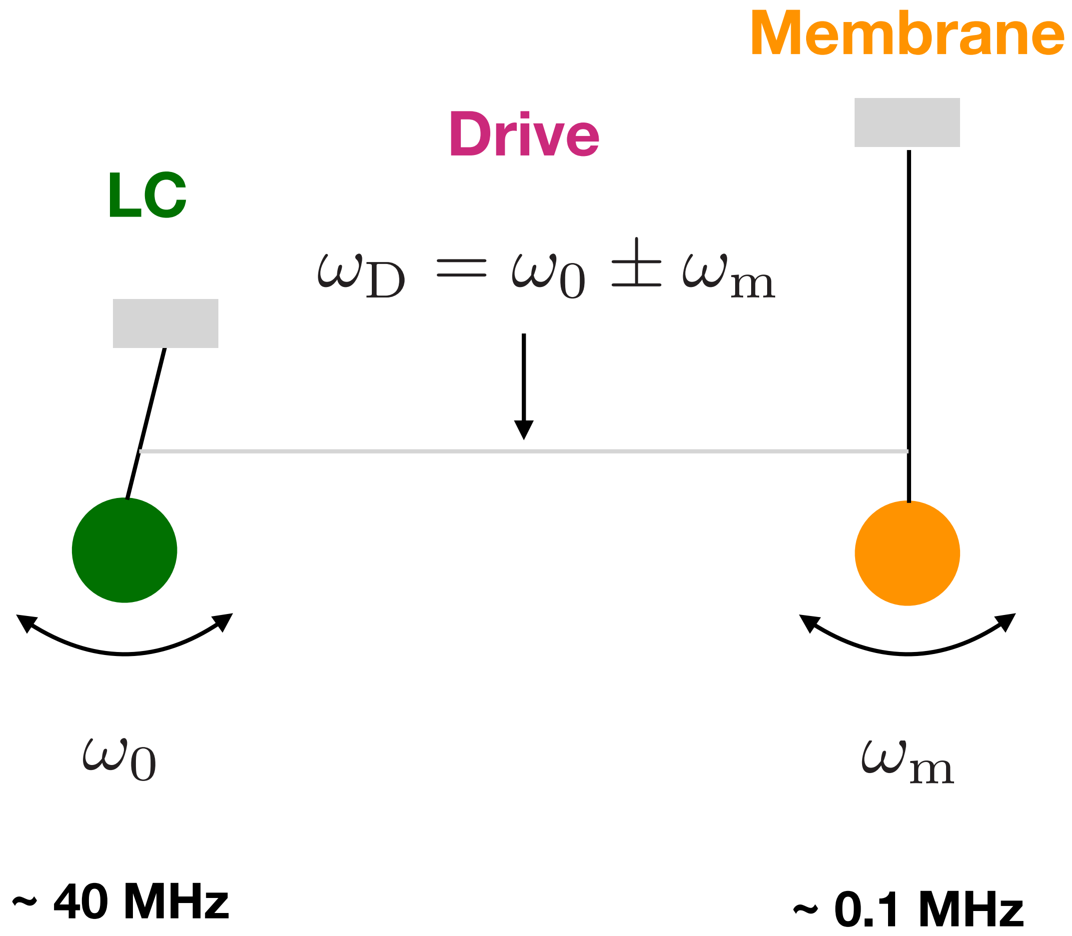






Portable NMR with a 1 T magnet
1T magnet with 5x17 mm rectangular bore
~43 MHz for 1H NMR
TRIMMER CAPACITOR BOX (TUNING)
λ/4 (~40 MHz)
NMR SIGNAL
RF PULSE

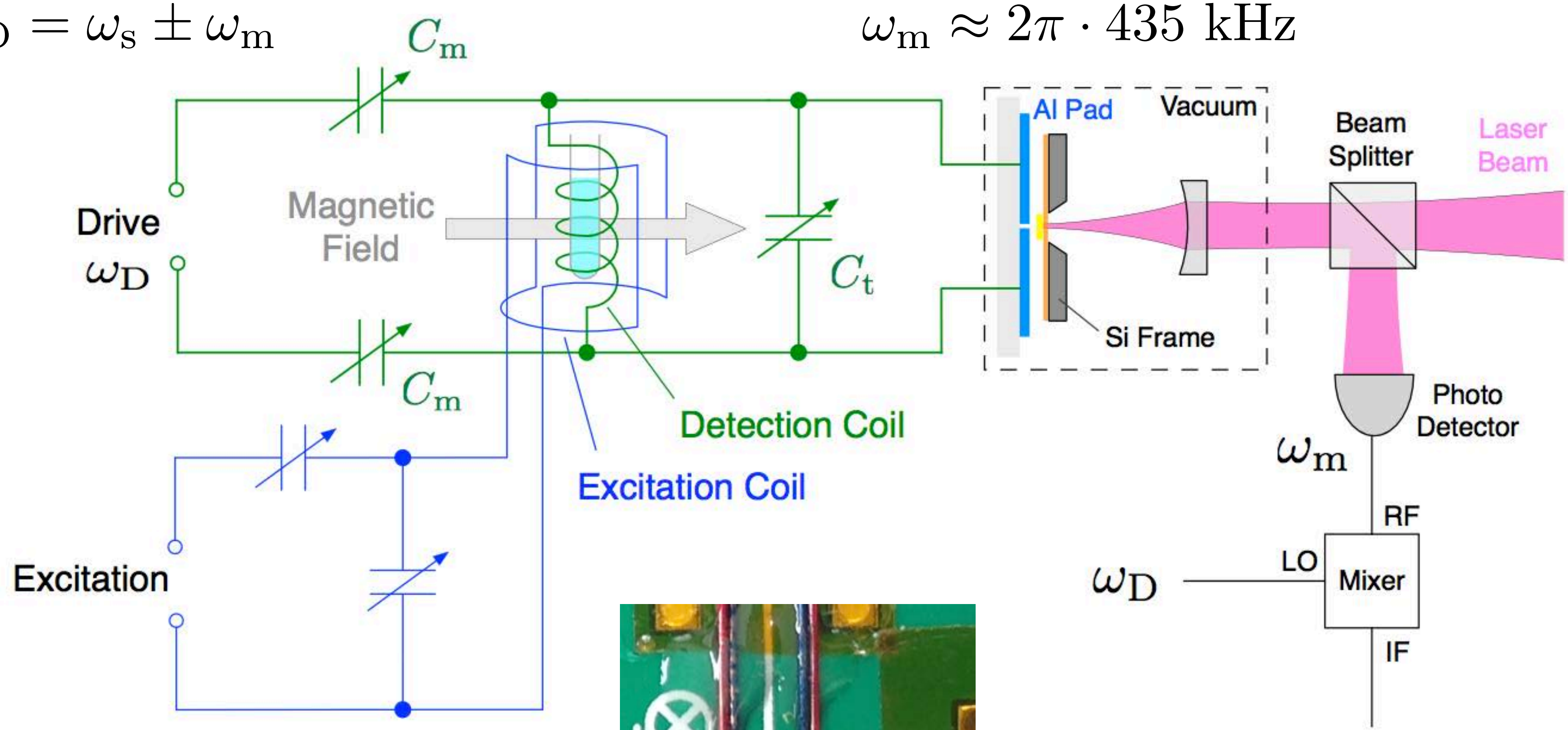
We need a Drive signal



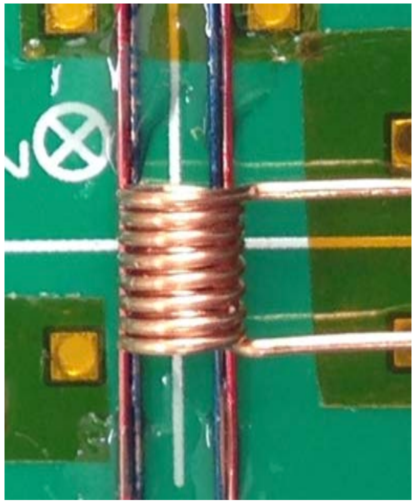
Circuit diagram

$$\omega_s = \omega_{LC} \approx 2\pi \cdot 42.8 \text{ MHz}$$

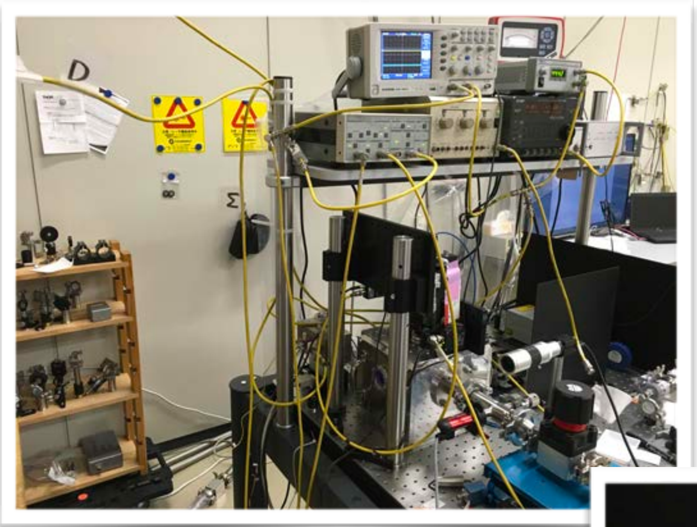
$$\omega_D = \omega_s \pm \omega_m \qquad \omega_m \approx 2\pi \cdot 435 \text{ kHz}$$



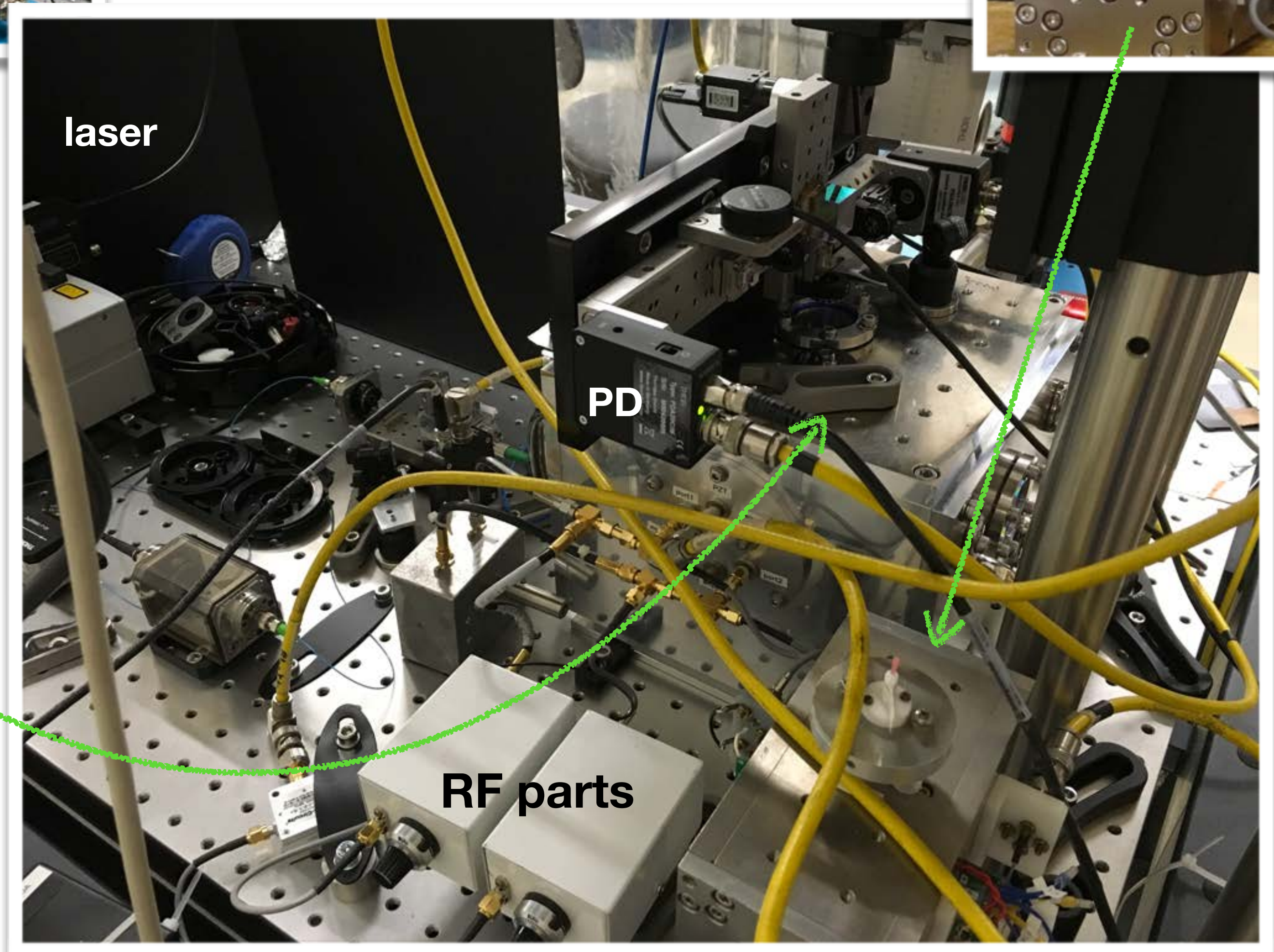
Q ~ 20



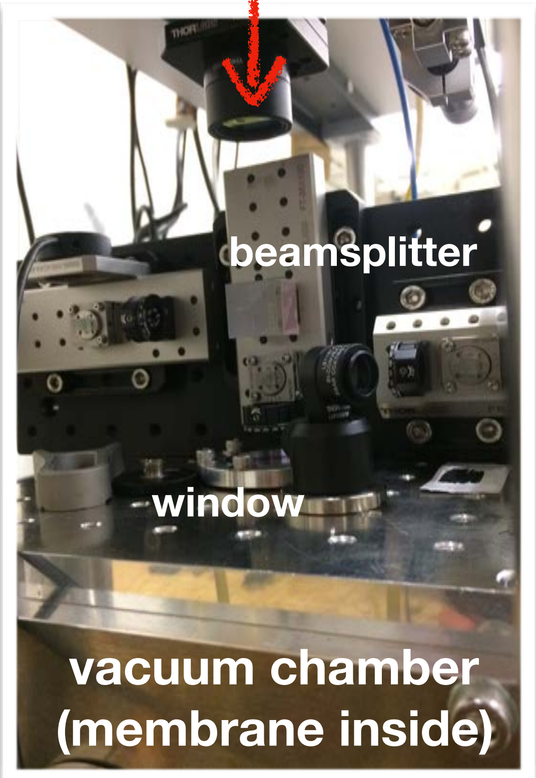
EMO NMR System



1 T magnet

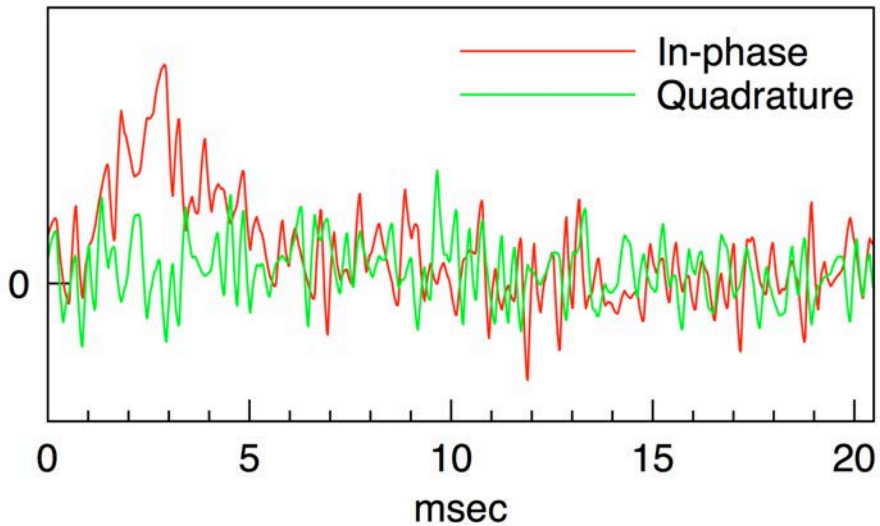


incident laser beam

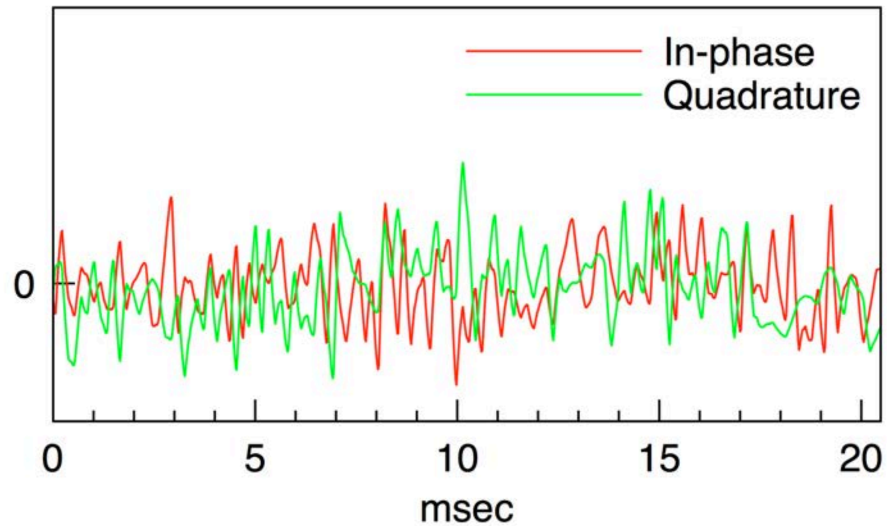


The First Signal (^{1}H spin echo in water)

on-resonance



2.5 kHz off-resonance



Happy birthday, EMO NMR! (28 Oct 2016)

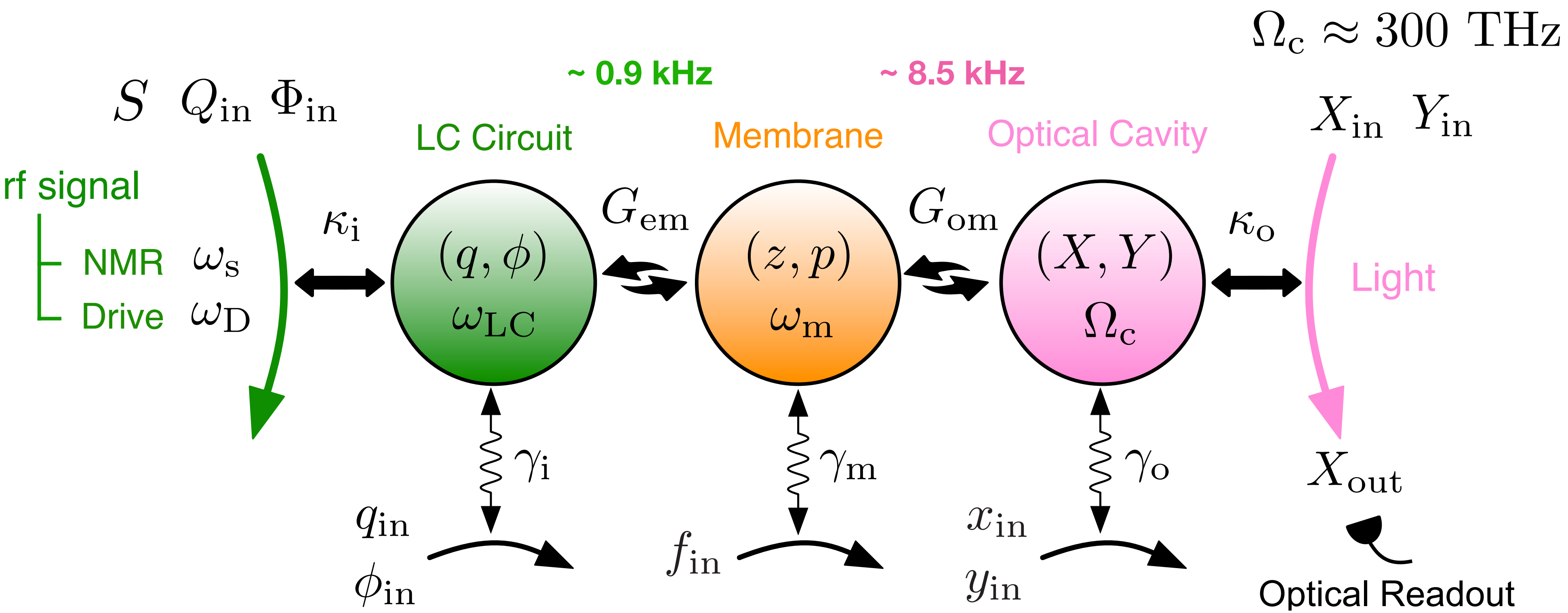
🎉👏😆🍻🎊

Bloch (1946)

And happy 70th birthday, NMR!

(in codensed matter)

Electro-Mechano-Optical (EMO) signal transduction

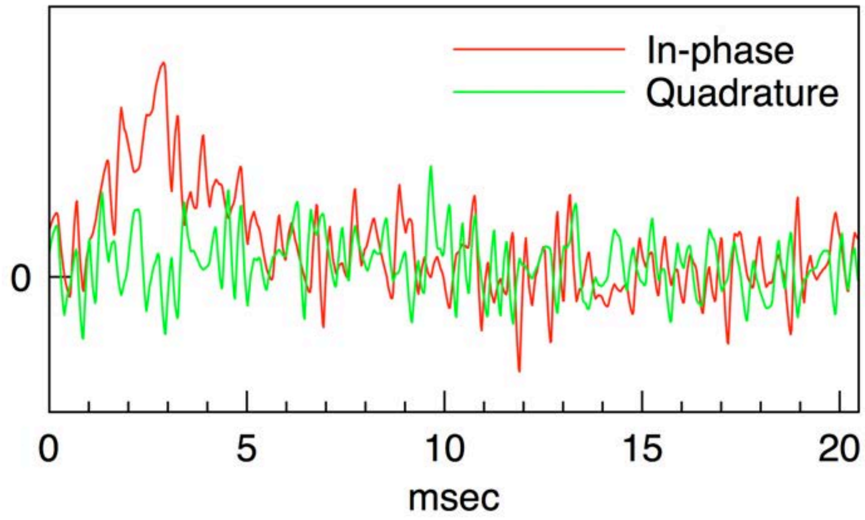


Effective Hamiltonian

$$H = -\frac{\Delta_i}{2}\left(q^2+\phi^2\right) - \frac{\Delta_o}{2}\left(X^2+Y^2\right) + \frac{\omega_m}{2}\left(z^2+p^2\right) + G_{em}qz + G_{om}Xz.$$

LC **Optical cavity** **Membrane oscillator** **electro-mechanical coupling** **opto-mechanical coupling**

For 5000 times accum., SNR was ~ 5 ⟶ Single-shot SNR ~ 0.1



Single-shot SNR

$$\frac{S}{N} = \sqrt{\frac{S^2 \frac{T_2^*}{2}\left(\gamma_{\mathrm{m}} \frac{T_2^*}{2}\right)}{\frac{\kappa_{\mathrm{iT}}}{\kappa_{\mathrm{i}}} \frac{S_{XX}(\omega_{\mathrm{m}})}{C_{\mathrm{om}} \frac{\kappa_{\mathrm{o}}}{\kappa_{\mathrm{oT}}} C_{\mathrm{em}}(\omega_{\mathrm{m}})} + \frac{\kappa_{\mathrm{iT}}}{\kappa_{\mathrm{i}}} \frac{S_{FF}(\omega_{\mathrm{m}})}{C_{\mathrm{em}}(\omega_{\mathrm{m}})} + \frac{\kappa_{\mathrm{iT}}}{\kappa_{\mathrm{i}}} S_{qq}(\omega_{\mathrm{m}}) + \eta_{\mathrm{p}} \frac{P_{\mathrm{D}}}{\hbar \omega_{\mathrm{D}} \gamma_{\mathrm{m}}}}} \approx 0.12$$

Optical shot noise ⟶ **0**

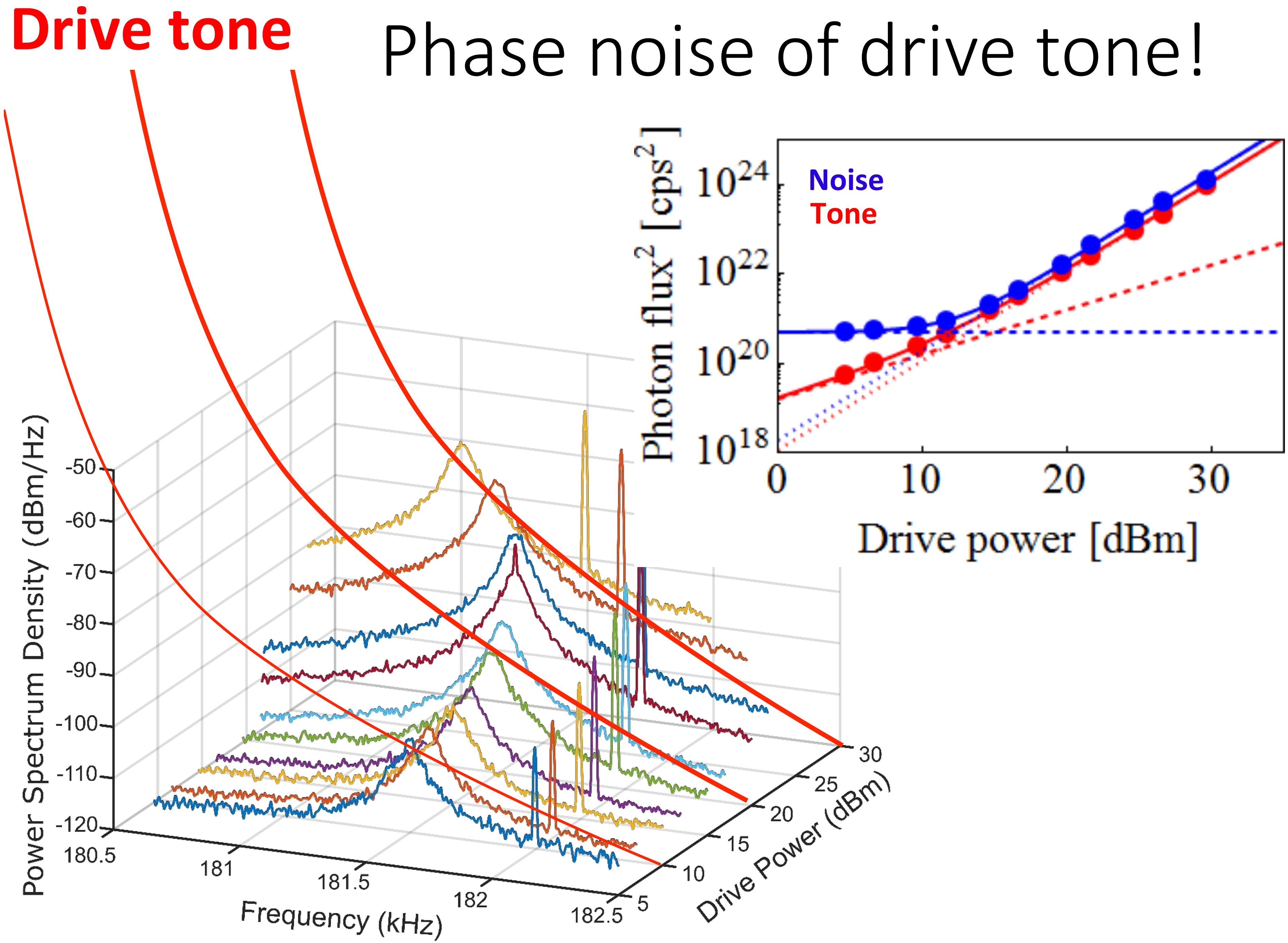
Membrane fluctuation

Johnson noise

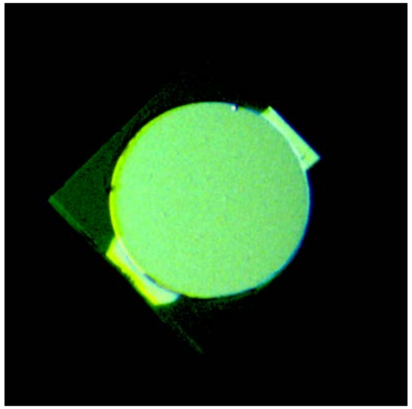
Drive phase noise

Takeda, Nagasaka, Noguchi, Yamazaki, Nakamura, Iwase, Taylor, Usami, *Opica*, 5 (2018) 152

Phase noise of drive tone!

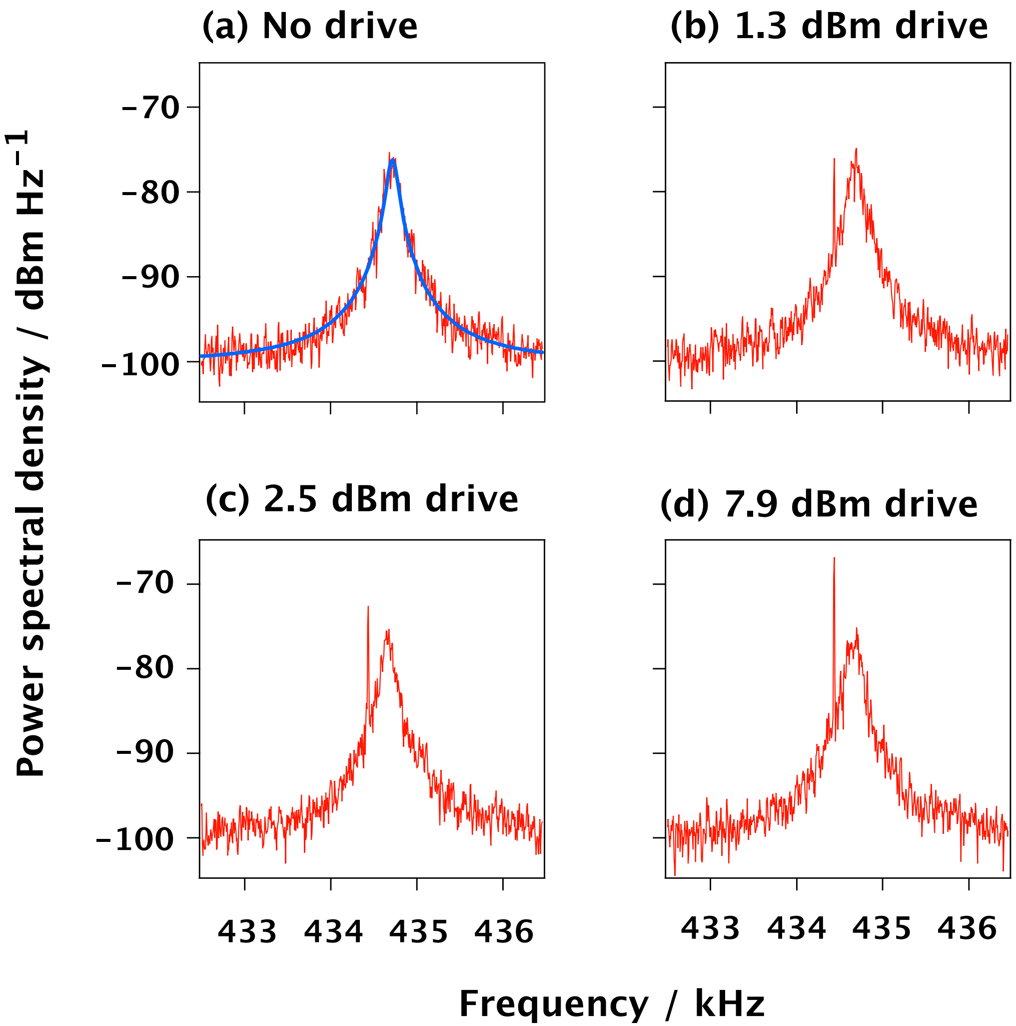


Phase-noise-free transduction



Au → **Al**

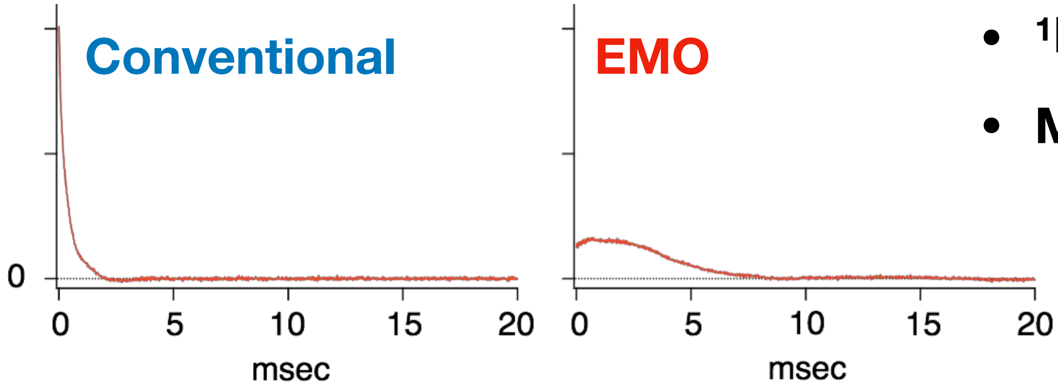
180 kHz → **430 kHz**



Tominaga, Nagasaka, Usami, Takeda, Under Review.

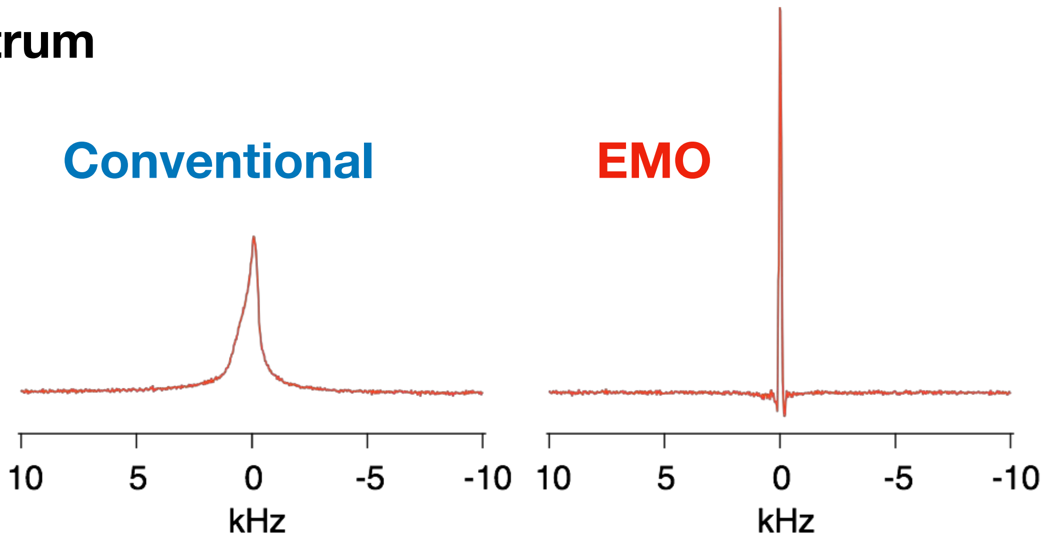
Phase-noise-free EMO NMR

- ^{1}H Free-induction decay



- $0.1M\ CuSO_4$ aq., ~3 mm^3
- ^{1}H NMR Freq.: 42.74 MHz
- Membrane Freq.: 435 kHz

- ^{1}H Spectrum

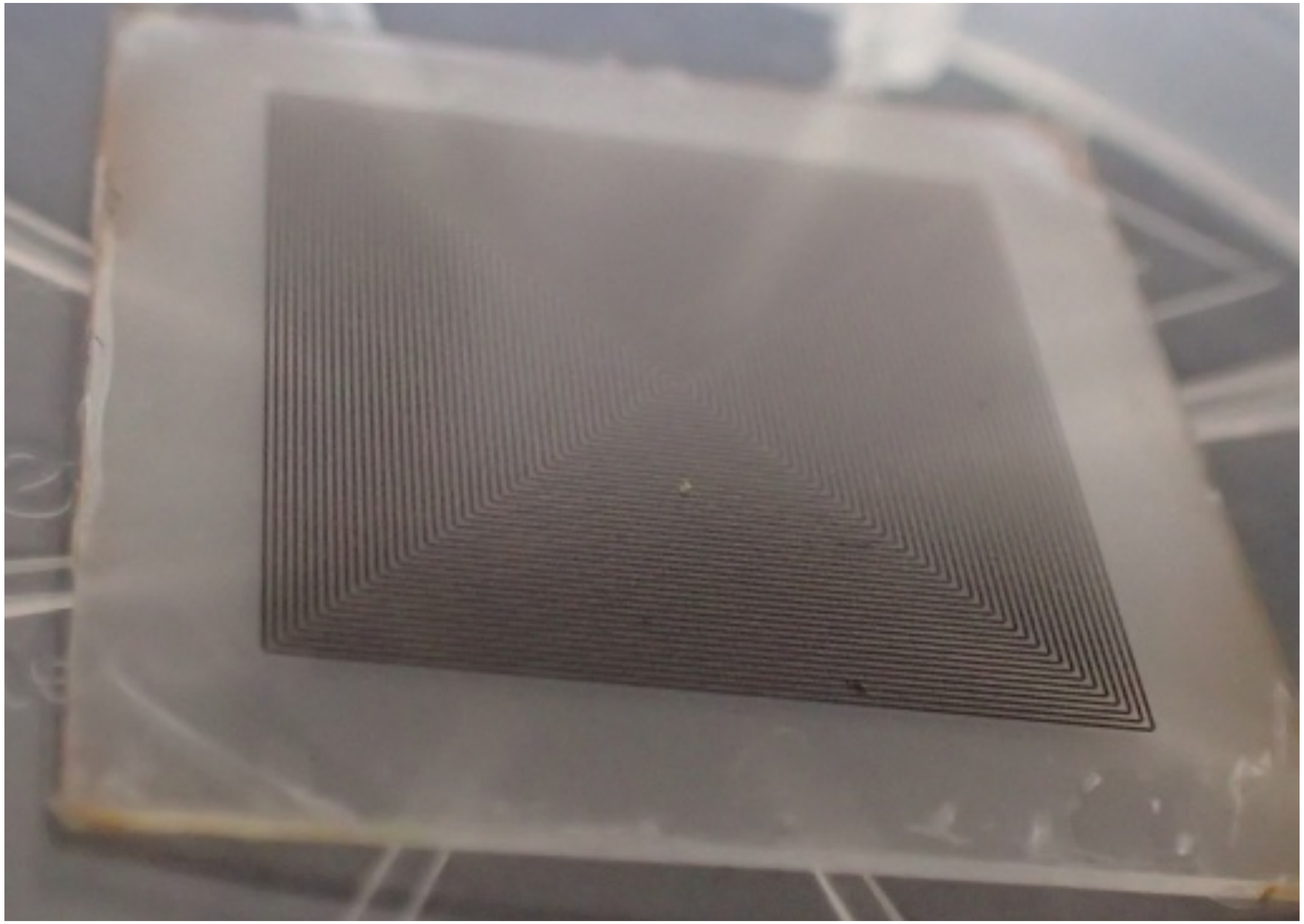


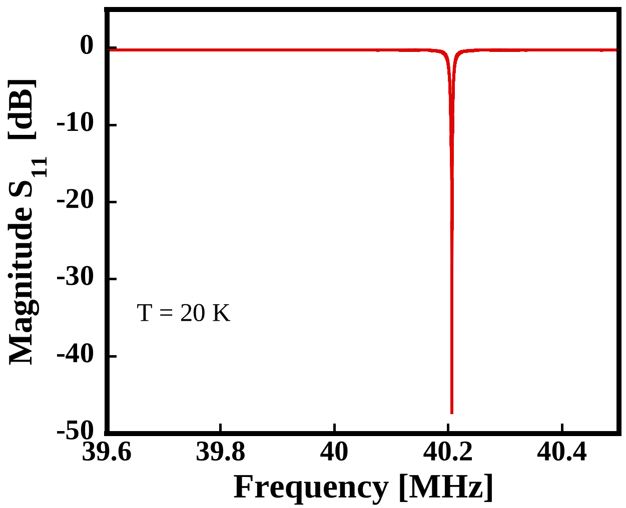
Tominaga, Nagasaka, Usami, Takeda, Under Review.

In-progress…

YBCO HTS coil

Courtesy: M. Takahashi (RIKEN), A. Saito (Yamagata Univ.)





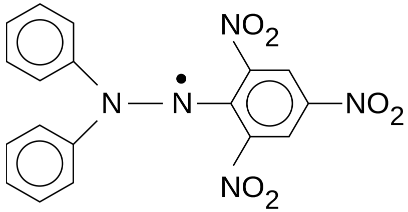
At 20 K, Q = 9098 (40 MHz) !

~ 100 times Q of copper coil with the same geometry

(Nuclear) Spin-cavity coupling may be explored.

Reproducing spin-cavity coupling

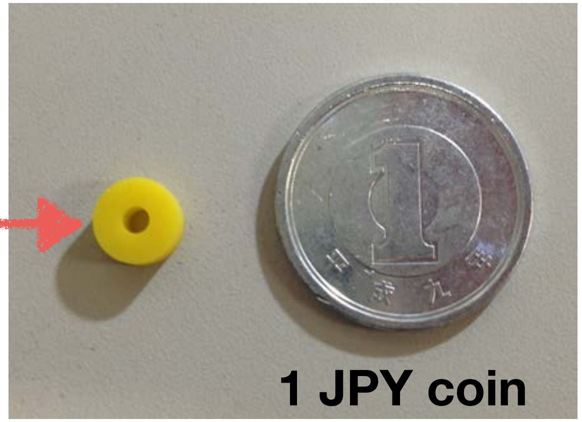
Abe et al., Appl. Phys. Lett. 98 (2011) 251108



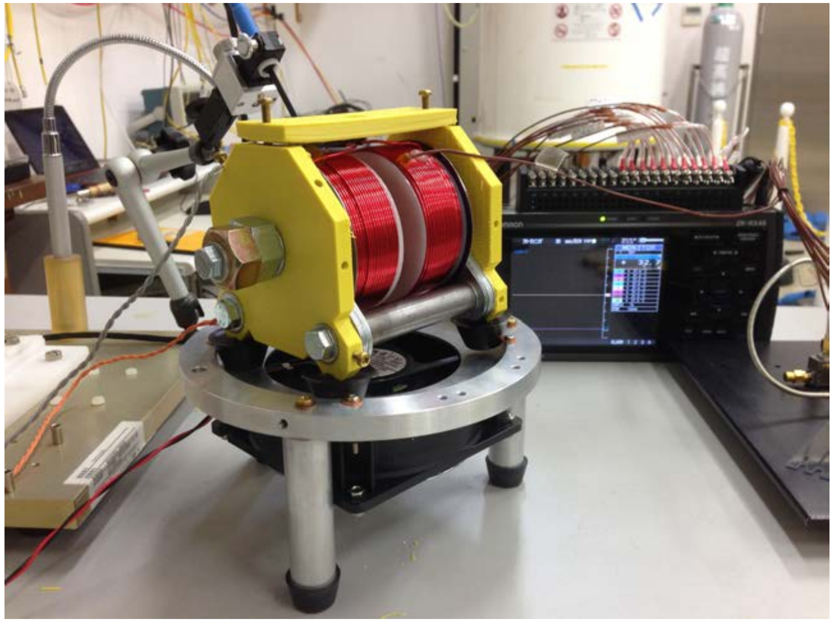
DPPH was packed tightly

(~60 mg)

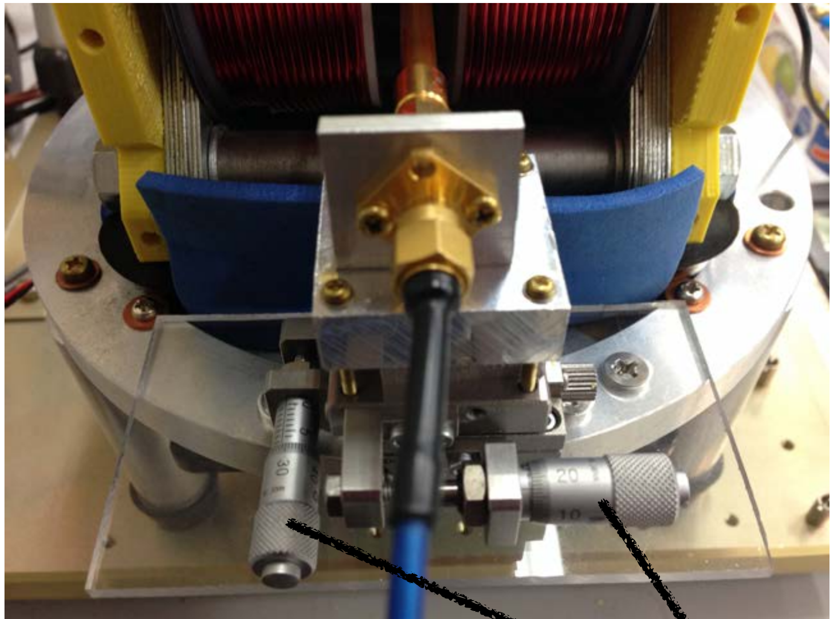
Dielectric resonator



- **dielectric const.: 37.4**
- **unloaded Q: >6000**
- **outer diameter: 5.98 mm**
- **inner diameter: 2.0 mm**
- **height: 2.7 mm**

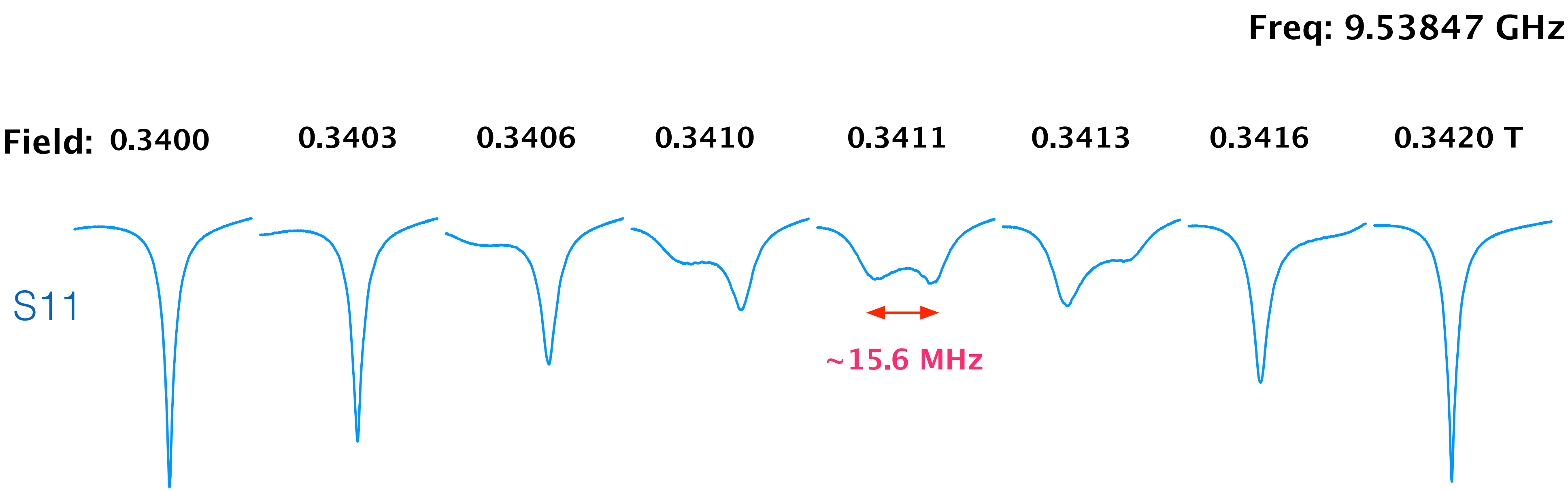


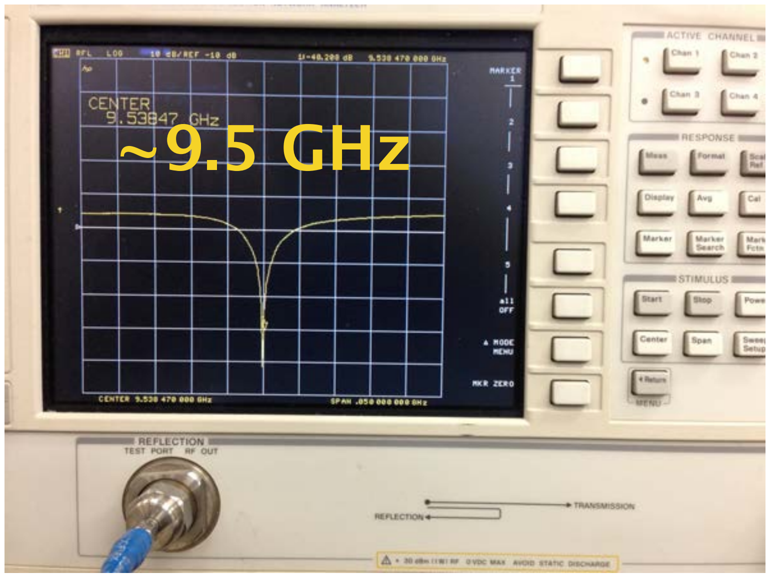
Partially 3D-printed electromagnet

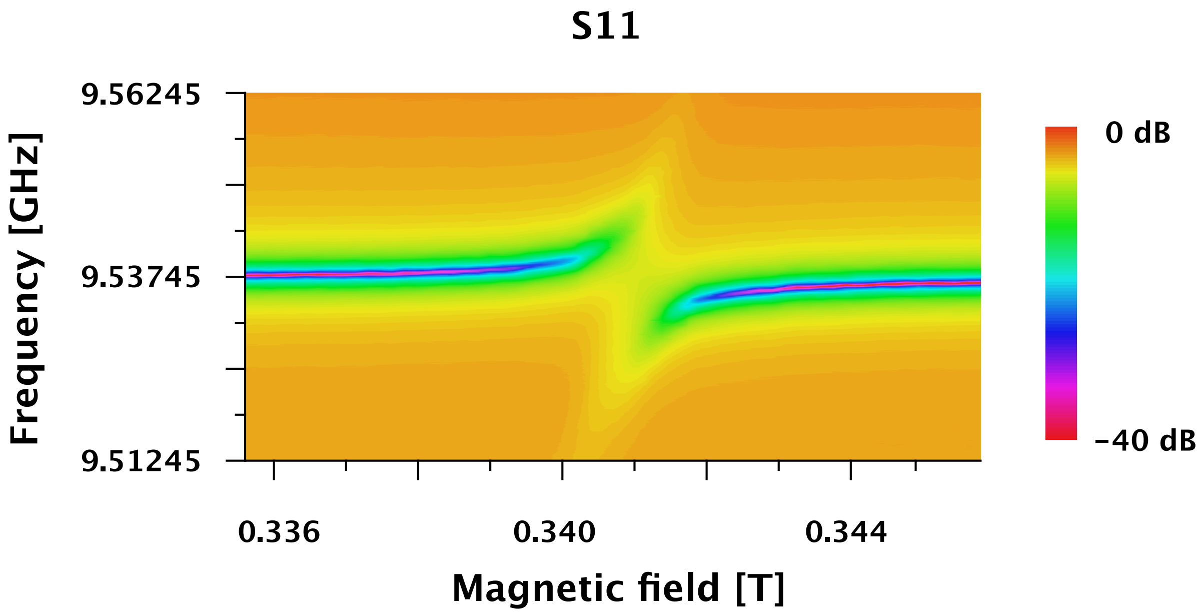


matching adjustment

Strong coupling between spin ensemble and DR



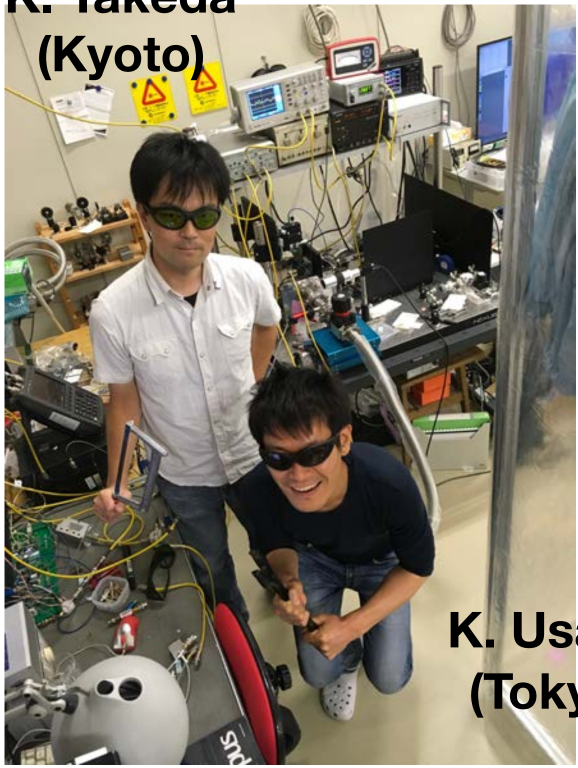




Field-Gradient Coils for MRI, and hopefully EMO-MRI

😎😎😎 Acknowledgment 😎😎😎

K. Takeda
(Kyoto)



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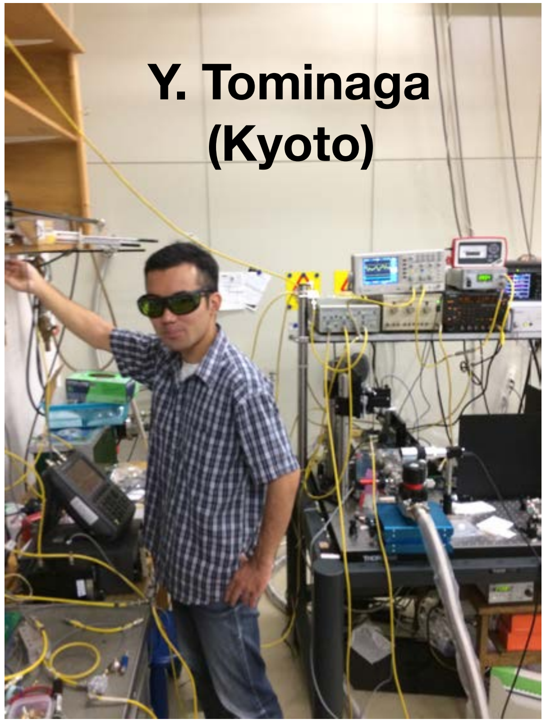
(Tokyo)

K. Nagasaka
(Tokyo)

Y. Tominaga
(Kyoto)



E. Iwase

R. Yamazaki

Y. Nakamura

A. Noguchi

J. M. Taylor

K. Yamada, M. Takahashi

💴 JST SENTAN

Thank you!