

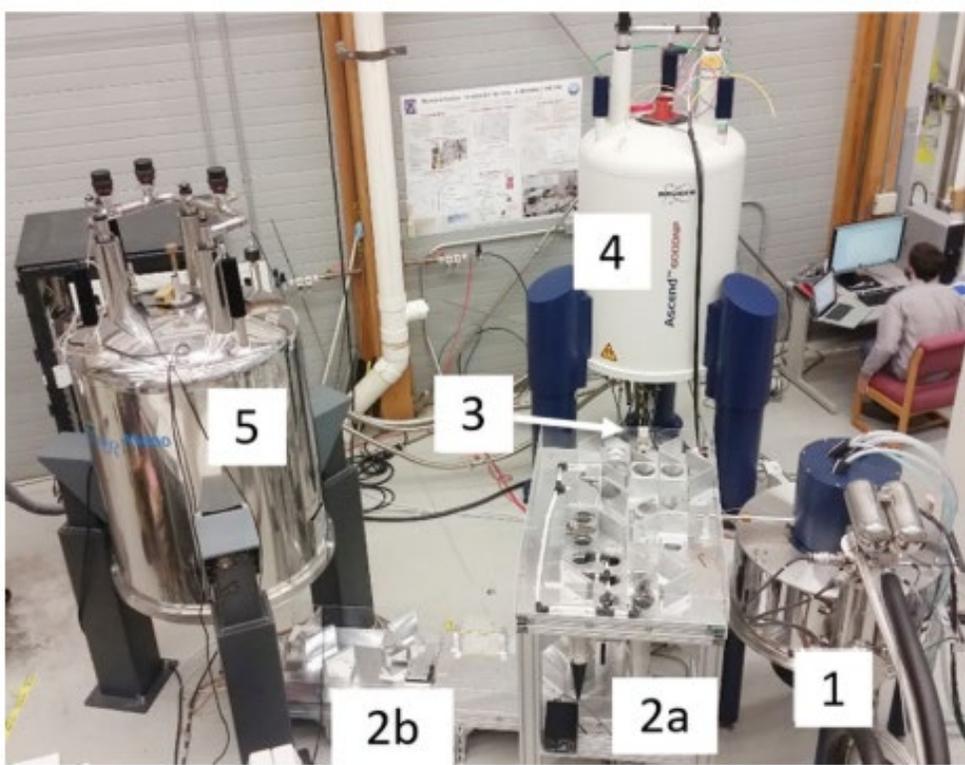


Gyrotrons, probes and
other equipment for MAS
DNP

Faith Scott

8/26/2025

DNP components

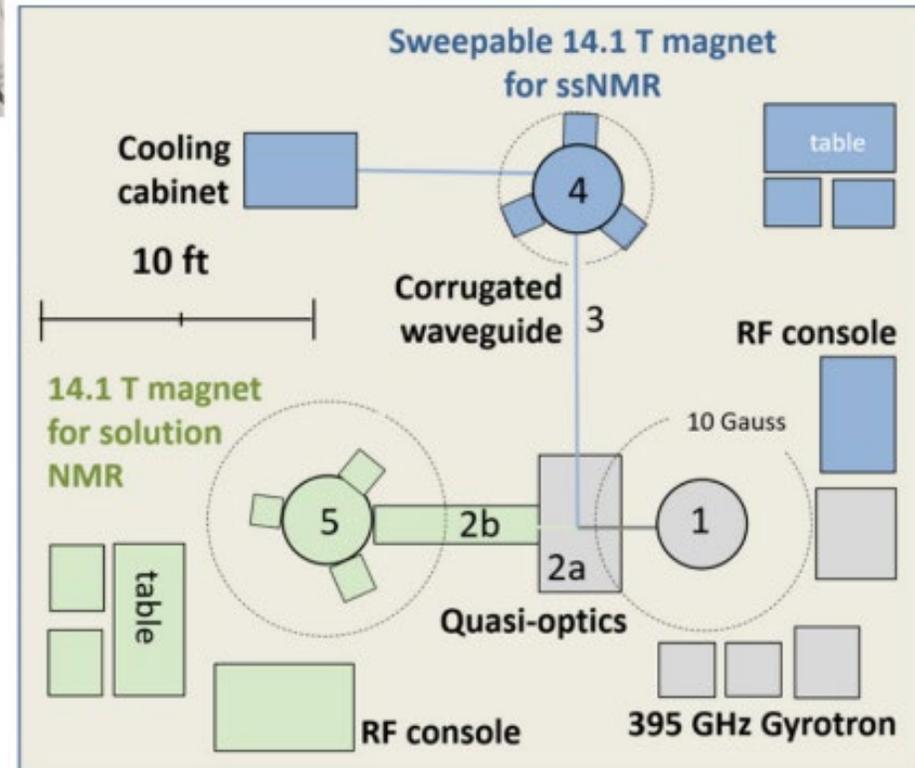


- RF Console
- Gyrotron (1)
- Microwave transmission (2,3)
- Cooling cabinet
- Magnet and probe (4)

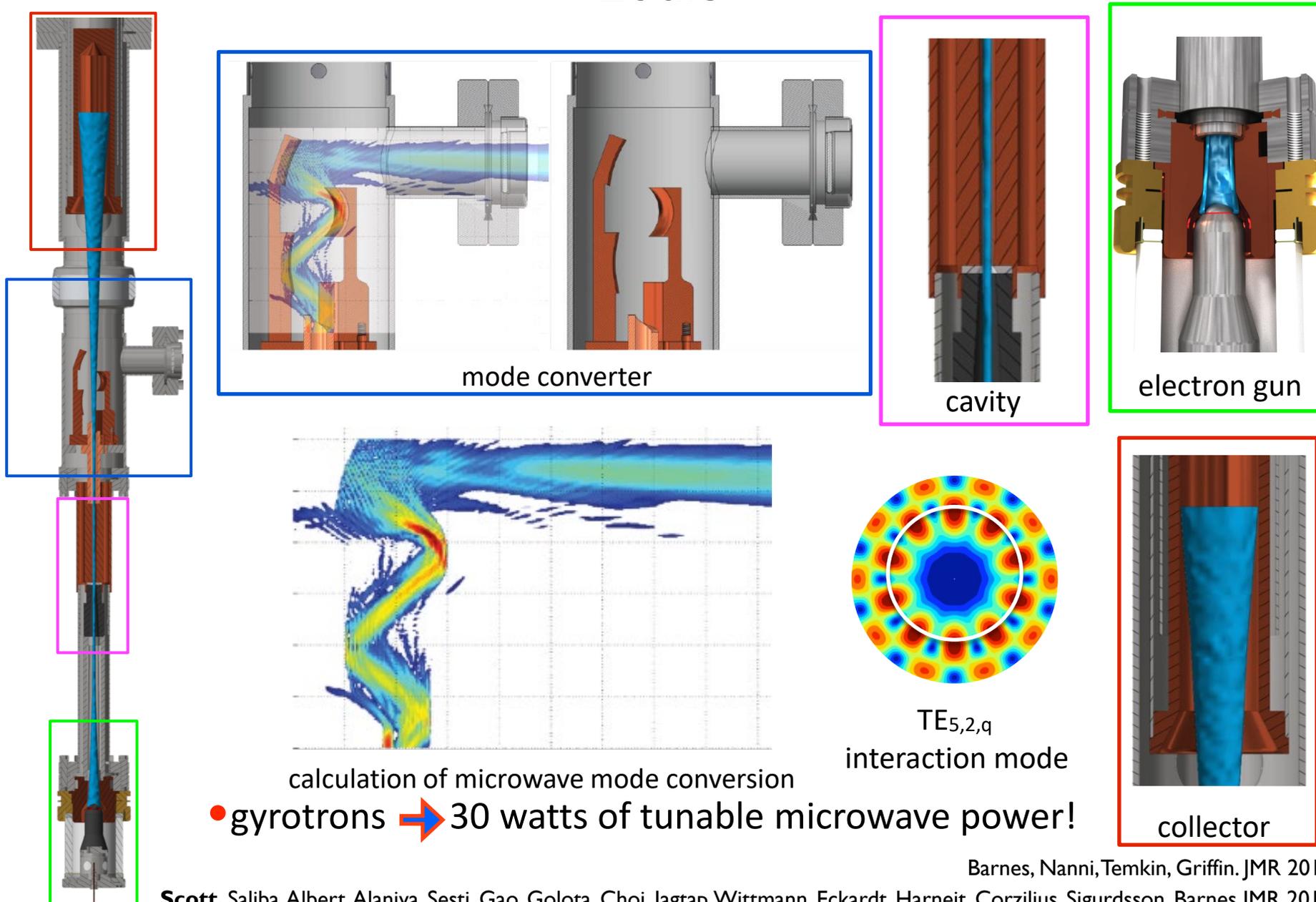
- Cooling requires significant insulation and liquid nitrogen

Probes:

- Bruker DNP 3.2 mm
- Custom NHMFL 3.2 mm- Complete July 2022
- Custom NHMFL 1.3 mm- Complete July 2025!
- Custom NHFML 1.9 mm- Complete late next year



AWG Driven Frequency Agile Gyrotron- @WashU in St Louis



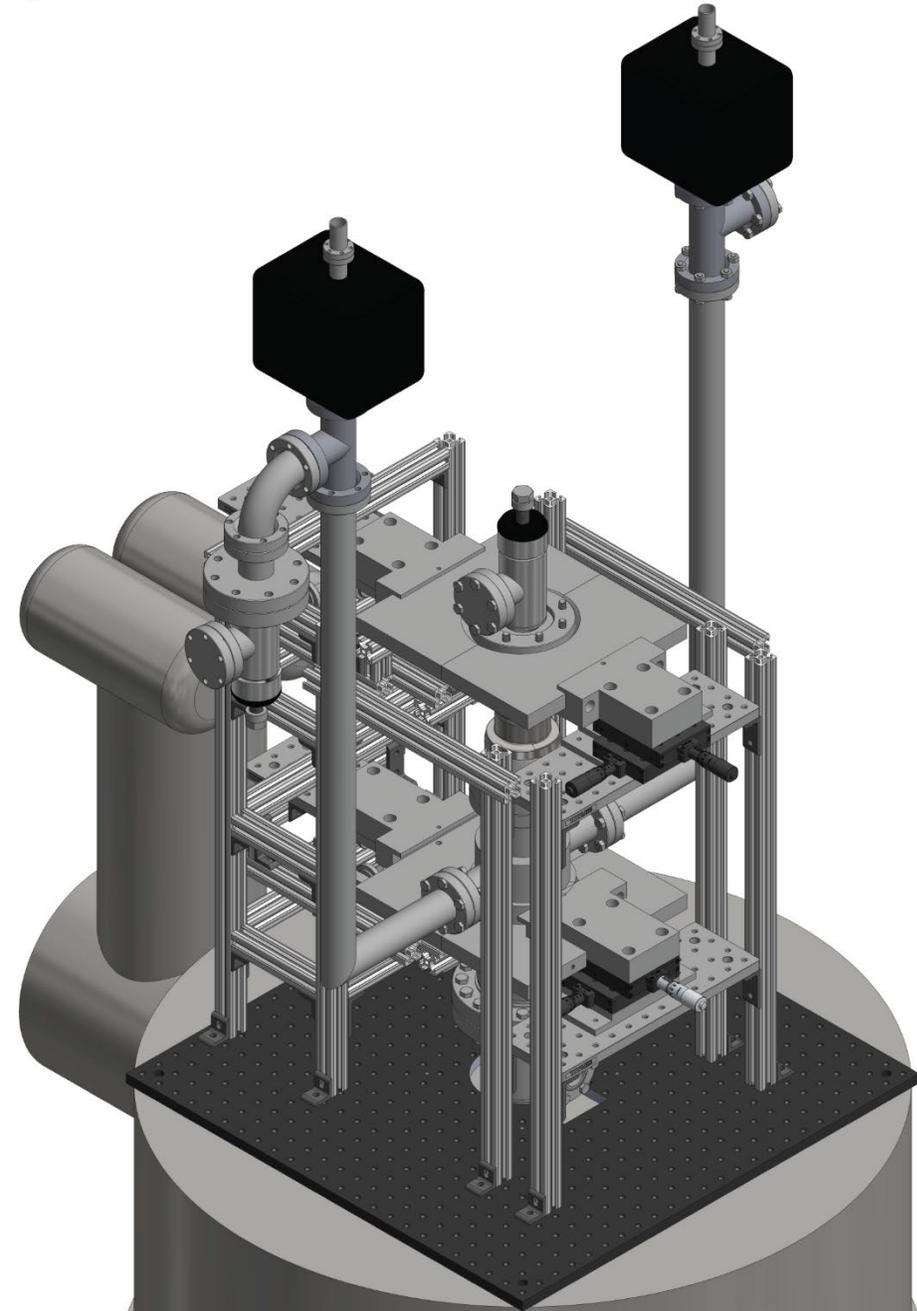
Barnes, Nanni, Temkin, Griffin. JMR 2012.

Scott, Saliba, Albert, Alaniva, Sesti, Gao, Golota, Choi, Jagtap, Wittmann, Eckardt, Harneit, Corzilius, Sigurdsson, Barnes JMR 2017

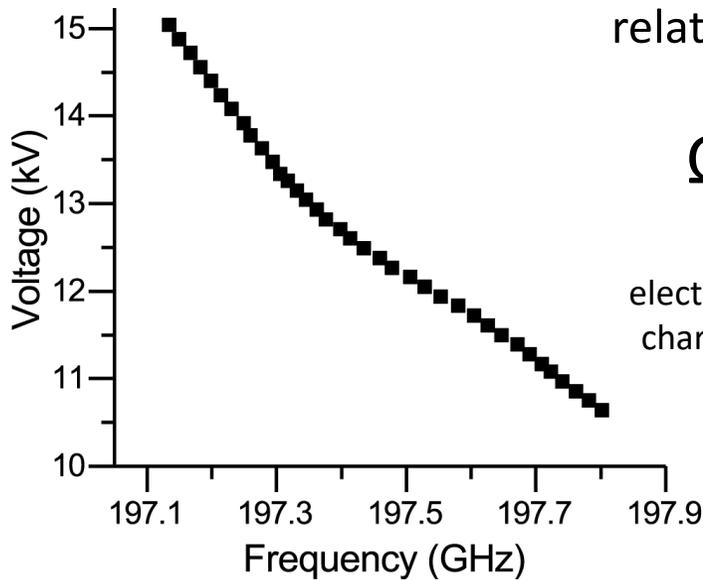
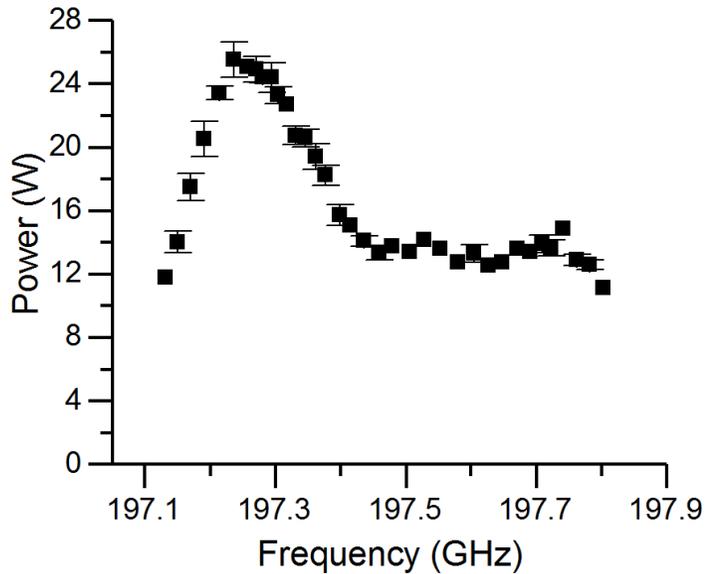
Assembly, Bakeout, and Installation



Assembly, Bakeout, and Installation



Voltage Tuning of Gyrotrons

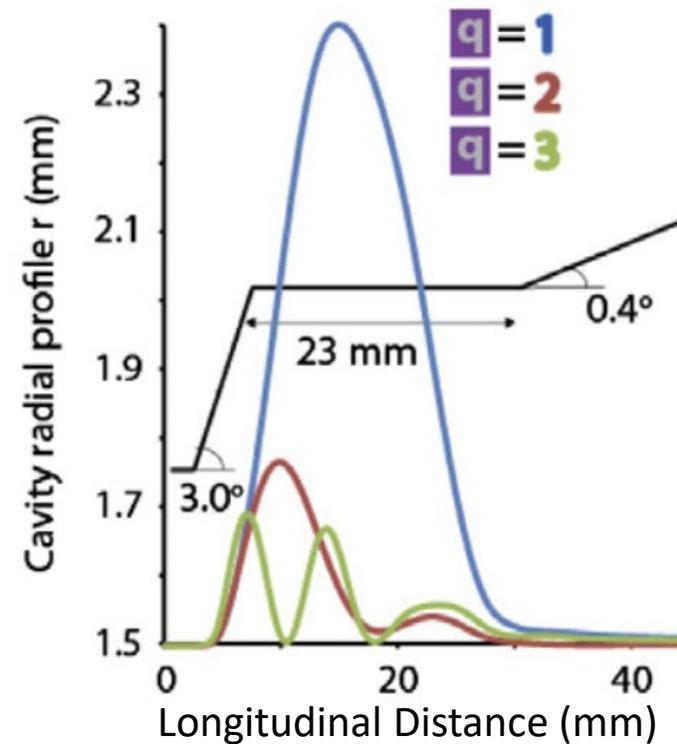


relativistic cyclotron frequency:

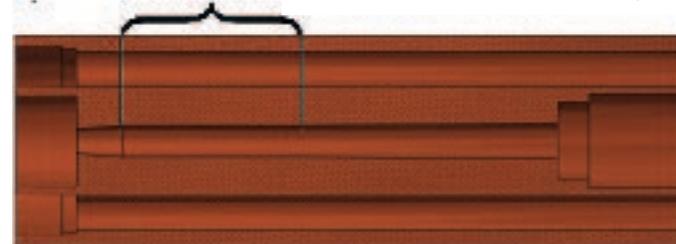
$$\Omega_z = eB_0/m$$

electron charge relativistic electron mass

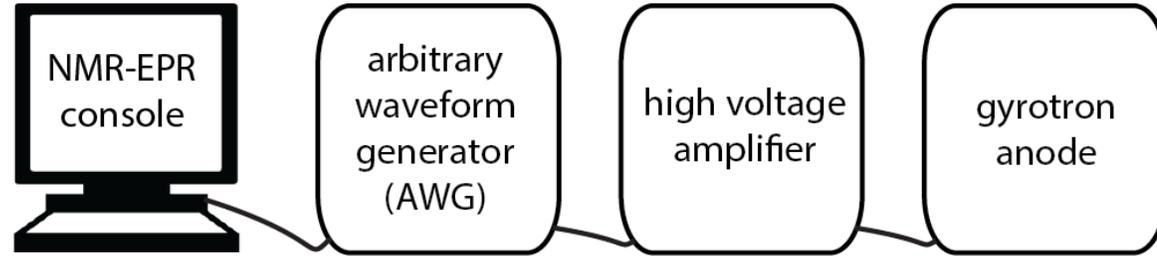
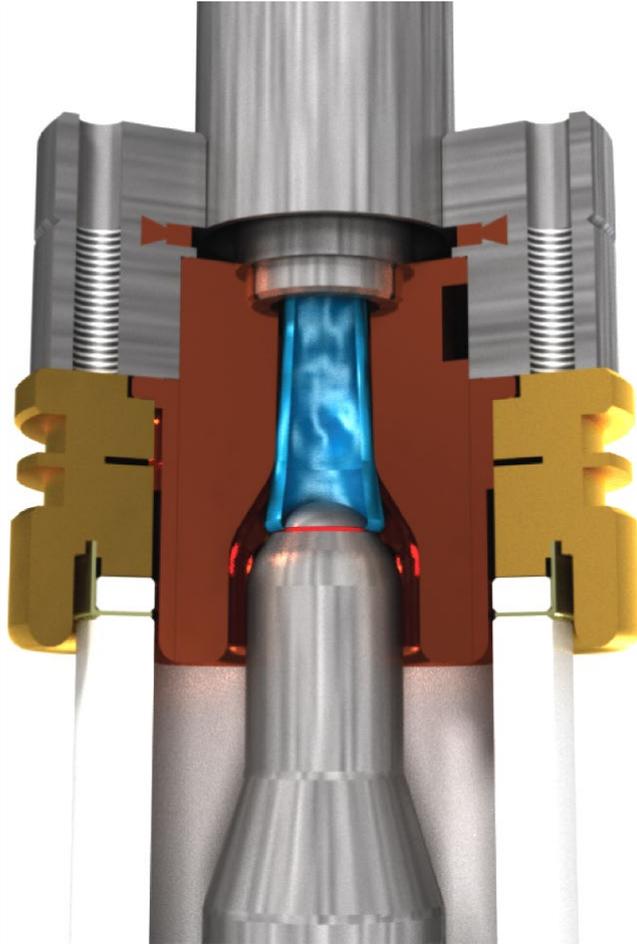
axial modes (q): TE_{5,2,q}



20 λ = 30 mm Interaction Cavity



AWG Driven Frequency Agile Gyrotron



relativistic cyclotron
frequency:

$$\Omega_z = eB_0/m$$

electron
charge

relativistic
electron mass

spectrometer control

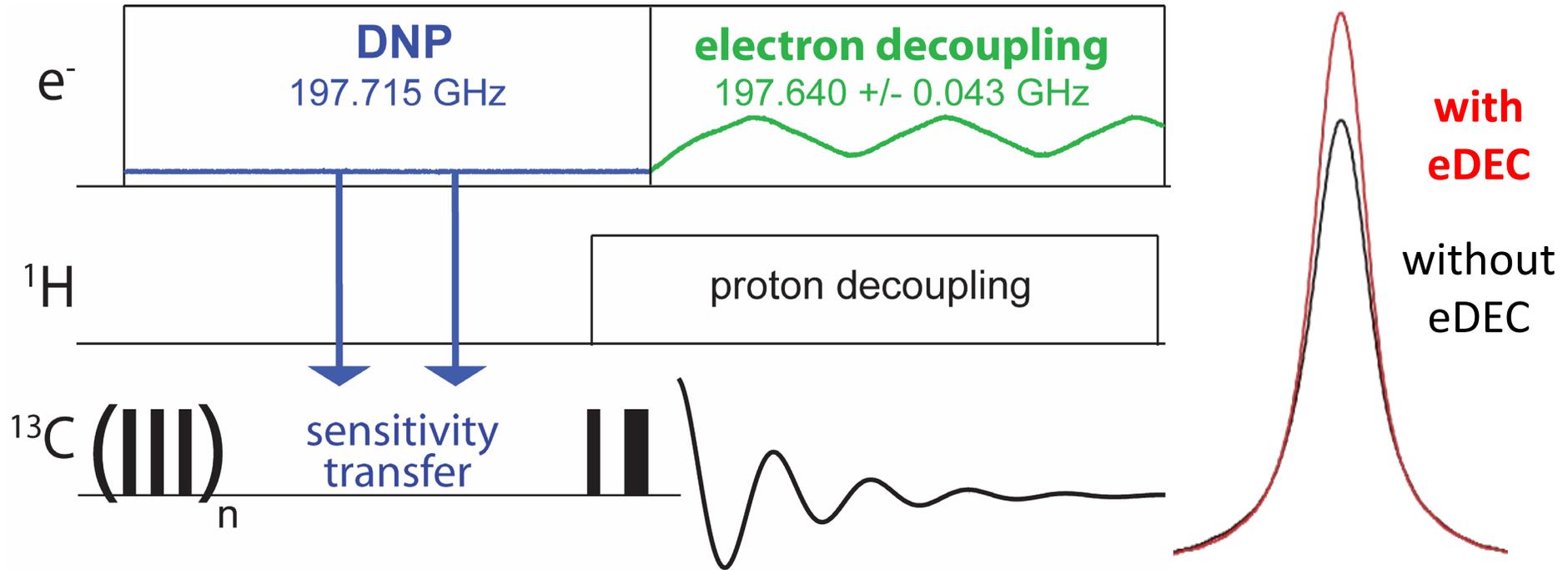


microwave frequency

Hoff, Albert, Saliba, **Scott**, Choi, Mardini, Barnes SSNMR 2015

Scott, Saliba, Albert, Alaniva, Sesti, Gao, Golota, Choi, Jagtap, Wittmann, Eckardt, Harneit, Corzilius, Sigurdsson, Barnes JMR 2017

Special experiments with agile gyrottron: Electron Decoupling with DNP in Rotating Solids



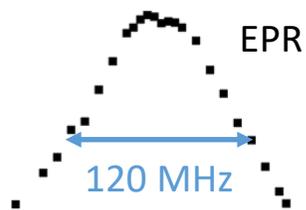
Decoupling of ^{13}C -electron Interactions

- ➡ increases intensity
- ➡ narrows linewidths
- ➡ extends homogenous transverse relaxation time
- ➡ attenuates hyperfine shift

Precedence

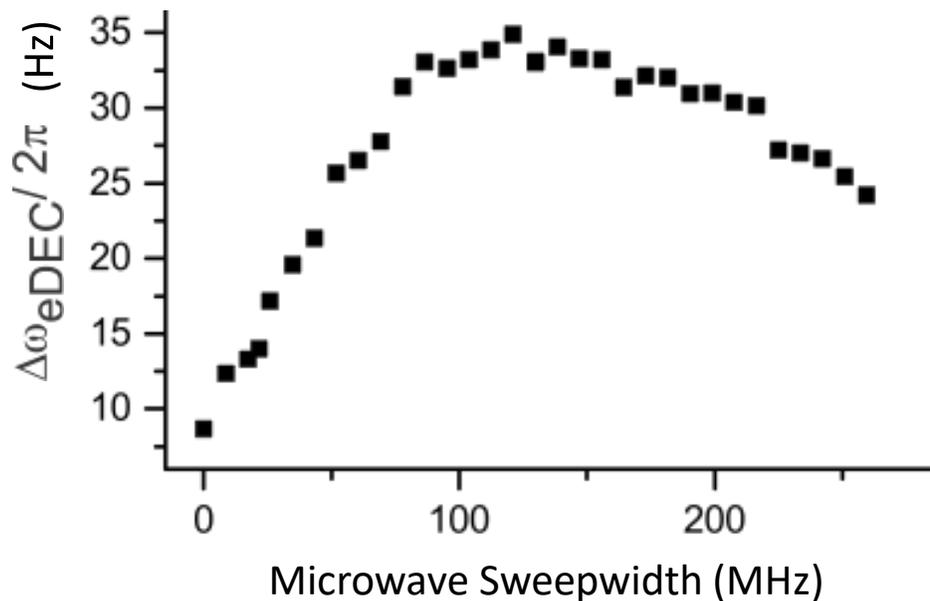
Jeschke et al. *JCP* 1997, Griffin, Corzilius, Andreas, Smith in *JMR* 2014, Han, Suter, Du, Wachtrup, many others

Special Expts: Chirped (Time Domain) Electron Decoupling

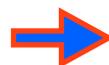


Electron Decoupling Dependence on Microwave Sweep Width

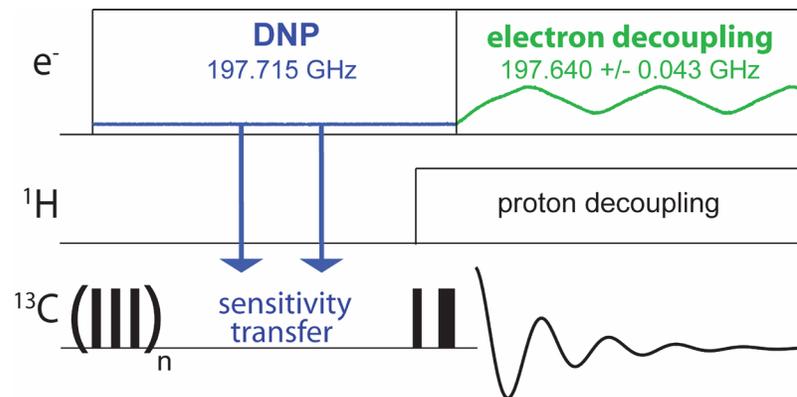
(7 second polarization time)



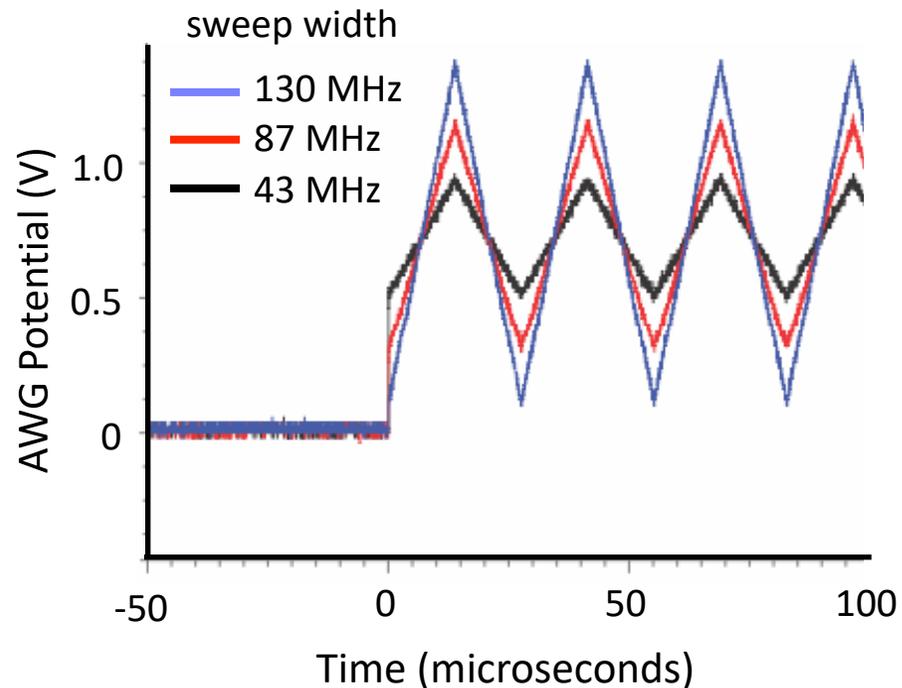
• **chirped** microwave pulses



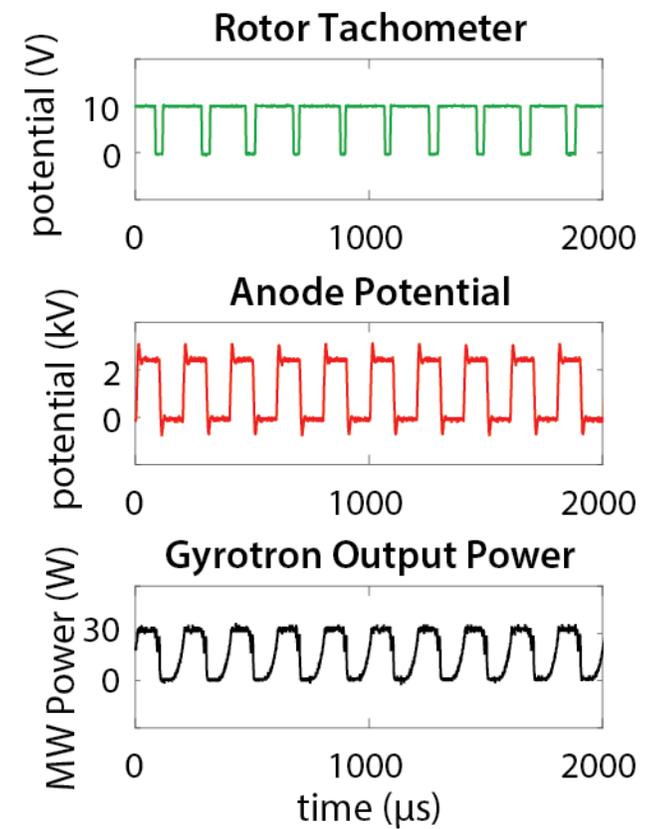
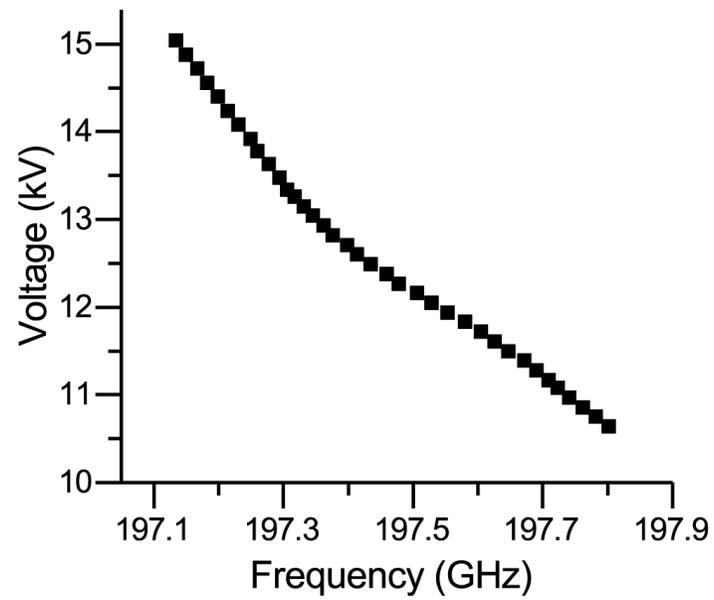
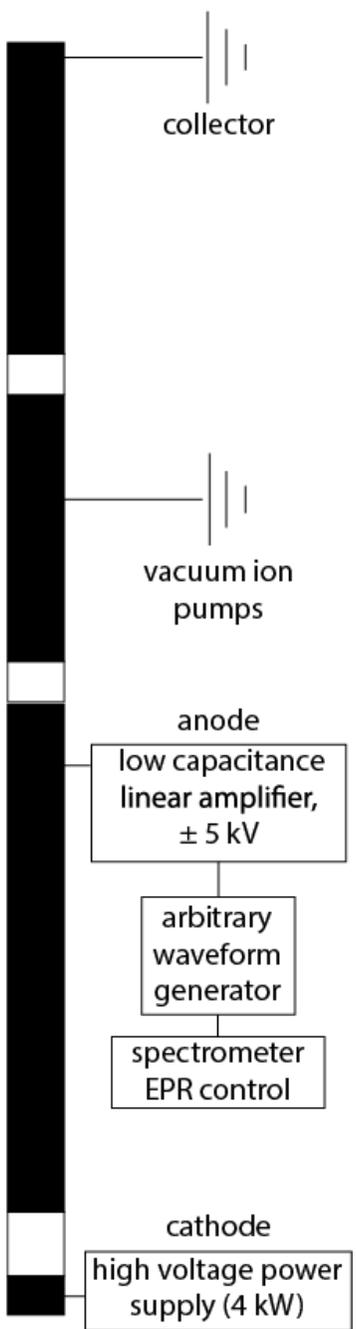
improved electron decoupling



Driving Potential (Frequency Control)



Duty Cycling of Frequency Agile Gyrotron @WashU in St Louis

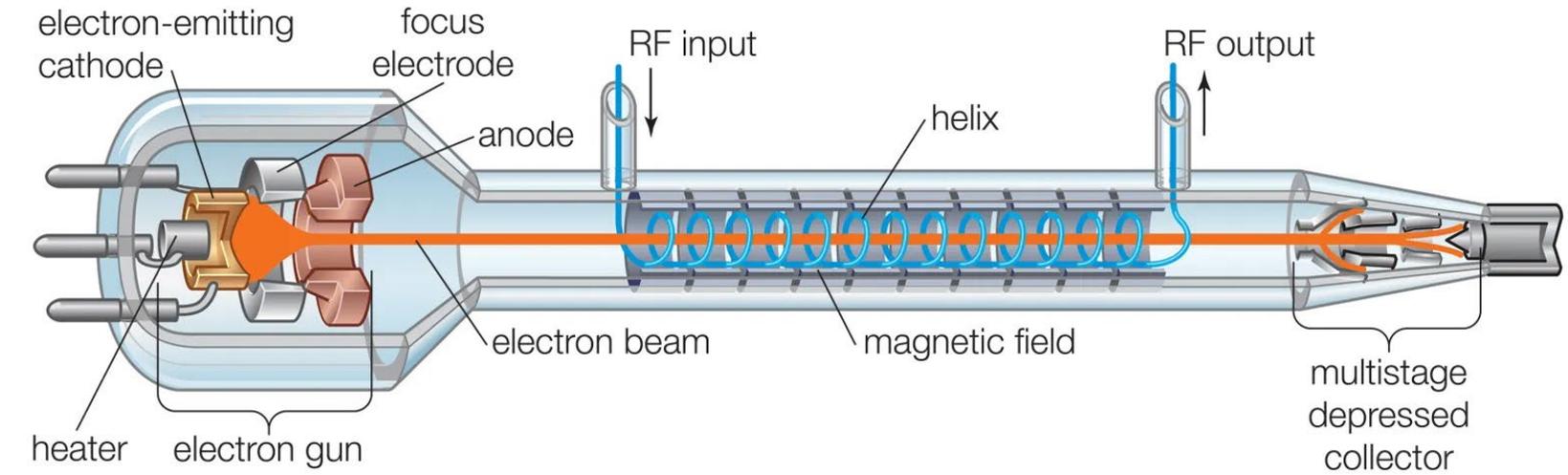
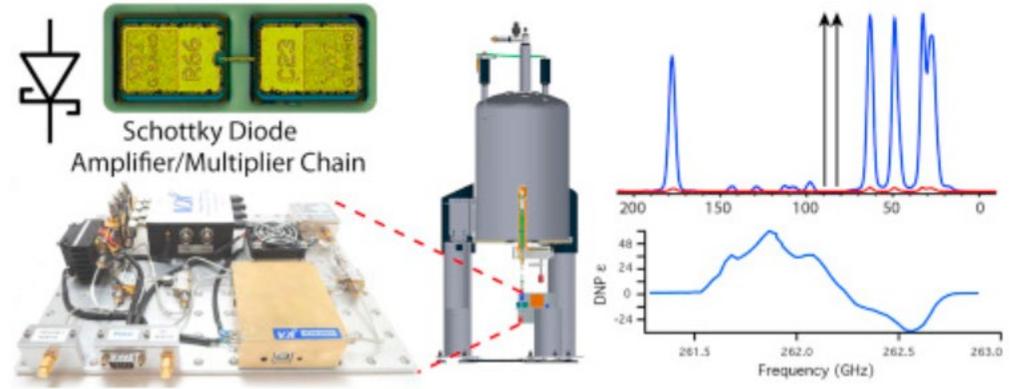


!! Not available with standard Bruker gyrotron, like we have here.

Fundamental mode gyrotron. Here, we duty cycle with a microwave shutter!

Bring voltage outside the cavity bandwidth to gate gyrotron power

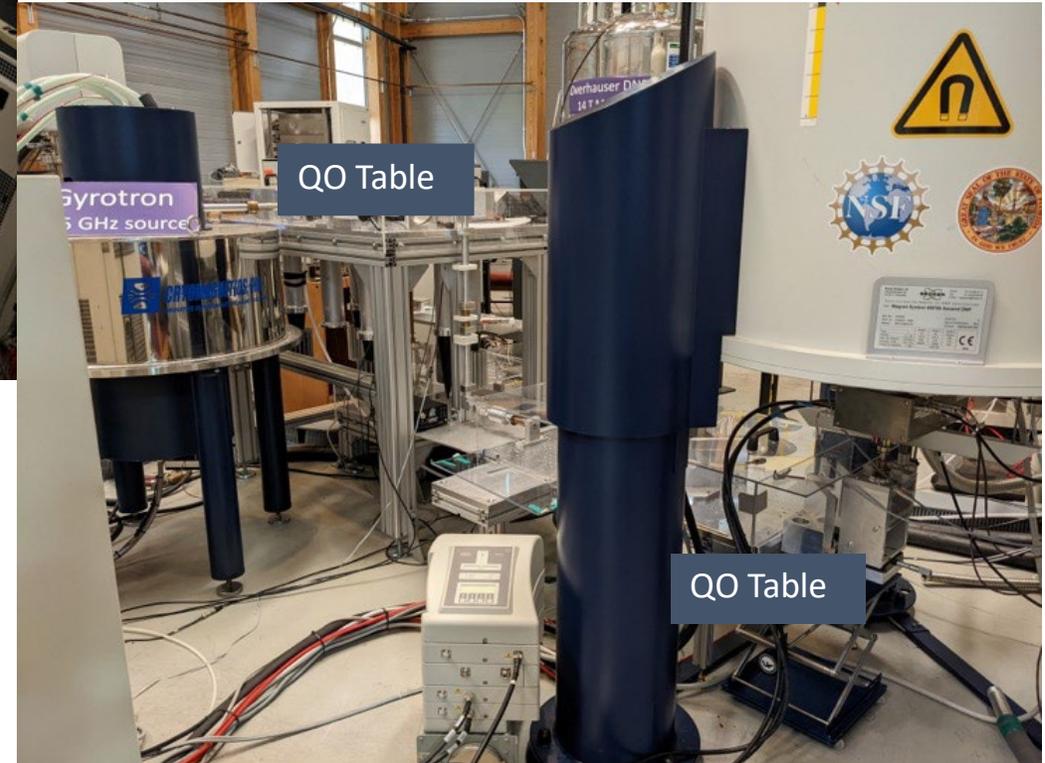
Sergeyev...Rosay SSNMR 2019



© 2010 Encyclopædia Britannica, Inc.

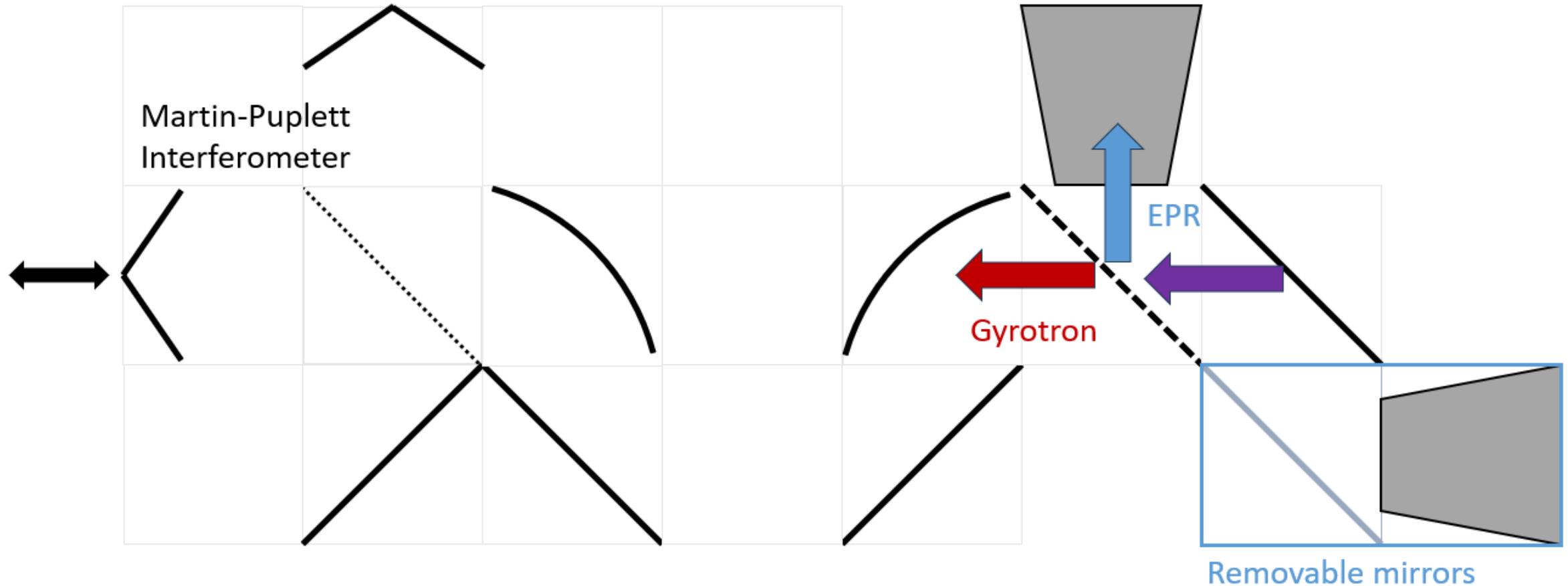
Travelling Wave Tube- Griffin Lab

QO microwave management

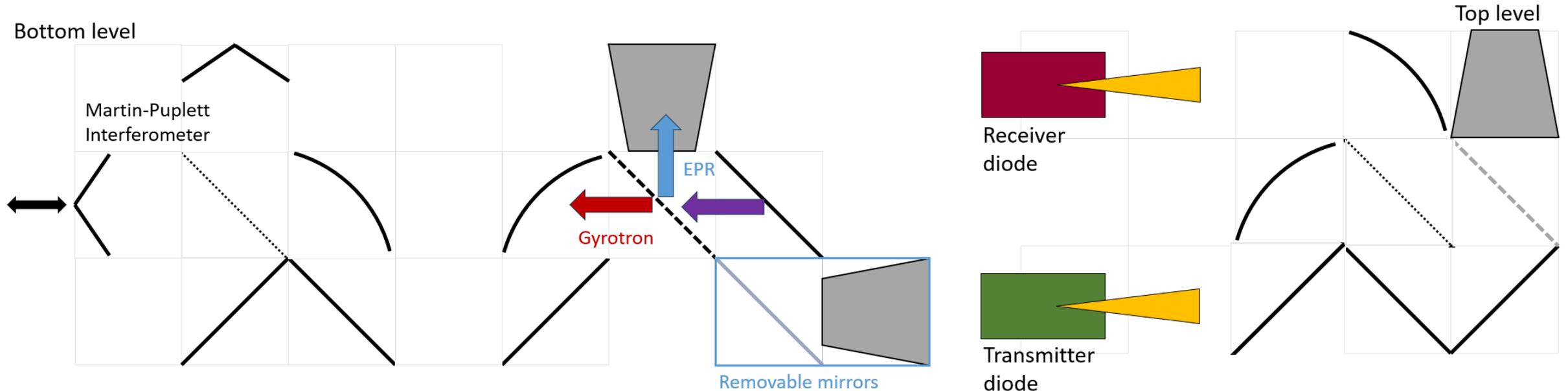


QO table and EPR capability

Bottom level



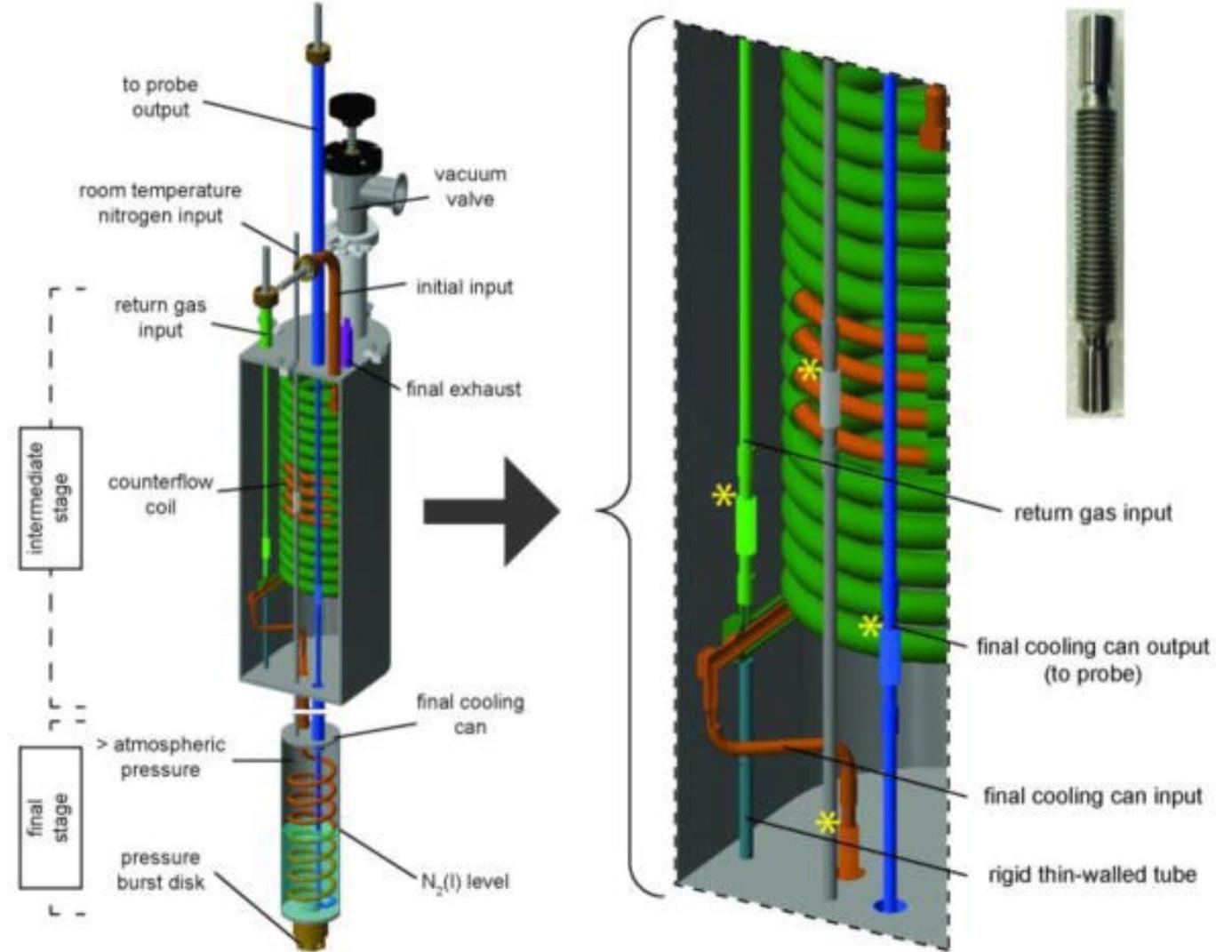
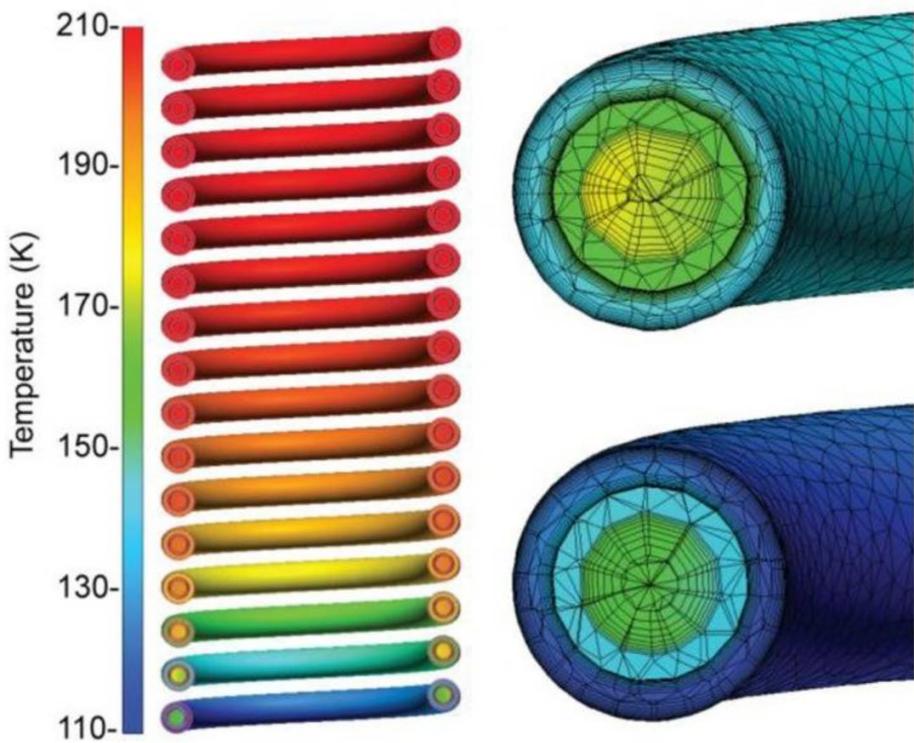
QO table and EPR capability



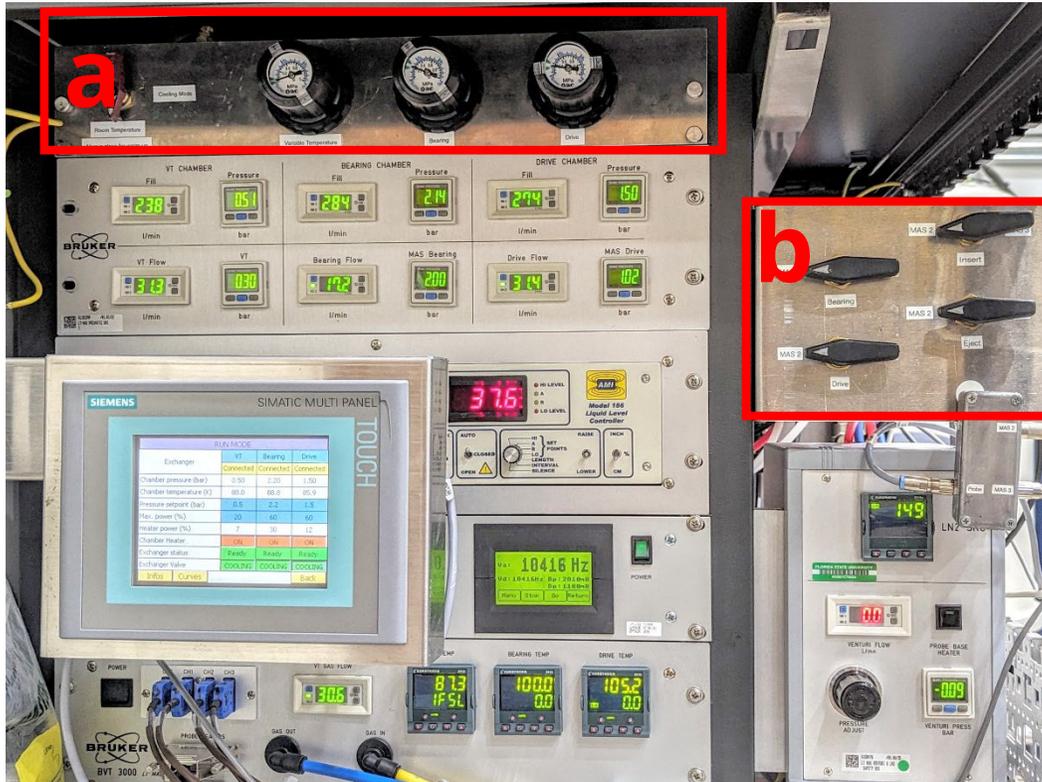
EPR will also require an additional coil at the top of the probe to provide a field sweep, while our MW source stays static.

Cooling for DNP @ WashU

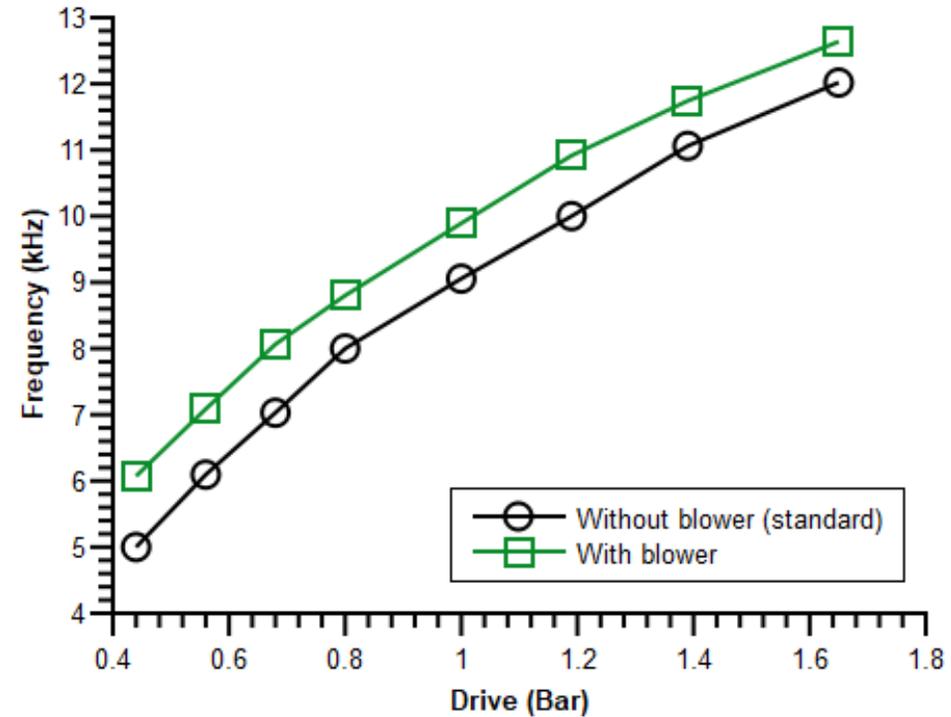
Counterflow coils pre-cool the gas in three chambers (Bearing, Drive, and VT) before the final cooling stage in contact with liquid nitrogen



Cooling cabinet- Cooling and spinning



a) Backup for cooling chamber heaters,
b) Quick switch between MAS 2 and MAS 3 spinning control.



A regenerative blower pulling on the exhaust can help increase spinning speed by reducing the backpressure of the exhaust gases



Regenerative blower used?	Drive gas temperature	Gas	Max spinning frequency
No	90	Helium	18 kHz
No	100	Nitrogen	12 kHz
Yes	100	Nitrogen	12.6 kHz
No	298	Nitrogen	12.6 kHz
Yes	298	Nitrogen	13.3 kHz

3.2 mm 600 MHz / 395 GHz, funded by NIH

Collaboration with Bruker

Microwave

- In-house design → cheap design is possible
- Very high efficiency in transmission
- EM simulated lens design → simple yet more efficient irradiation

Ergonomics/Cooling

- Faster cooling time
- Better magic angle adjustment:
 - Works both ways at low temp
 - Distinct from tuning knobs and lockable

Radio frequency

- Tune cards
 - Easy and repeatable tuning
- RF performance improved
 - Much better ^1H efficiency → fast MAS

In use since July 2022

1.3 mm commissioned, entering user program soon

Sets the groundwork for 1.3 and 1.9 mm probes



Waveguide design

Waveguide sections

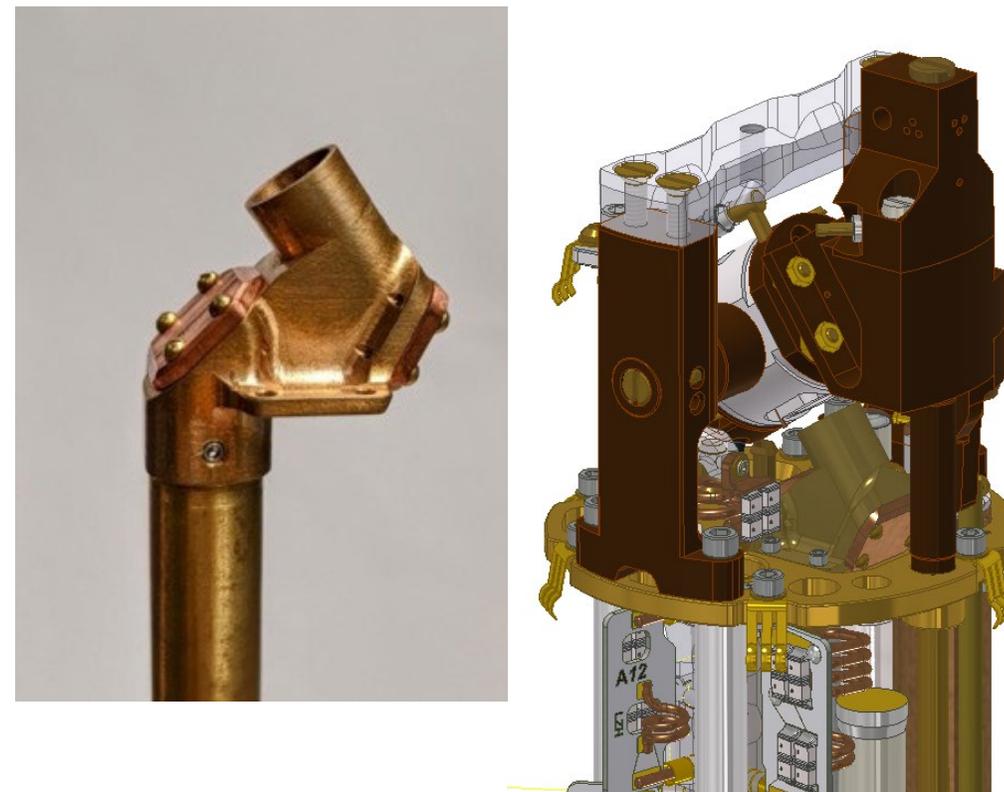
- Brass, gun drilled in sections of 30 cm (12 in)
- Corrugation made in-house (tap)
- Losses and beam shape were characterized on our QO bench

WG bend bolted to top deck

- No change in orientation
- Controlled distance with stator
- Bend machined as a single part → reduced degrees of freedom

Lens trials

- tested on 10 mM AMUPol, 250 mM labelled proline in d8-glycerol/D₂O/H₂O (15/75/10 vol%)
- Bruker original probe requires 12.5 W, $\epsilon = 130-140$, then sample melting
- Type A : optimal DNP for ~3-4 W power, but only 80-90 ϵ and sample melting
- Type B: optimal power 8.5 W, $\epsilon = 130-140$, with sample softening
- Type C: Lens focuses along rotor profile while diverging along rotor axis for full coverage



	Bruker standard probe	Custom NHMFL probe	Difference
MW power needed for same enhancement	12 W	8.5 W	-30%

Ease of use and ergonomics

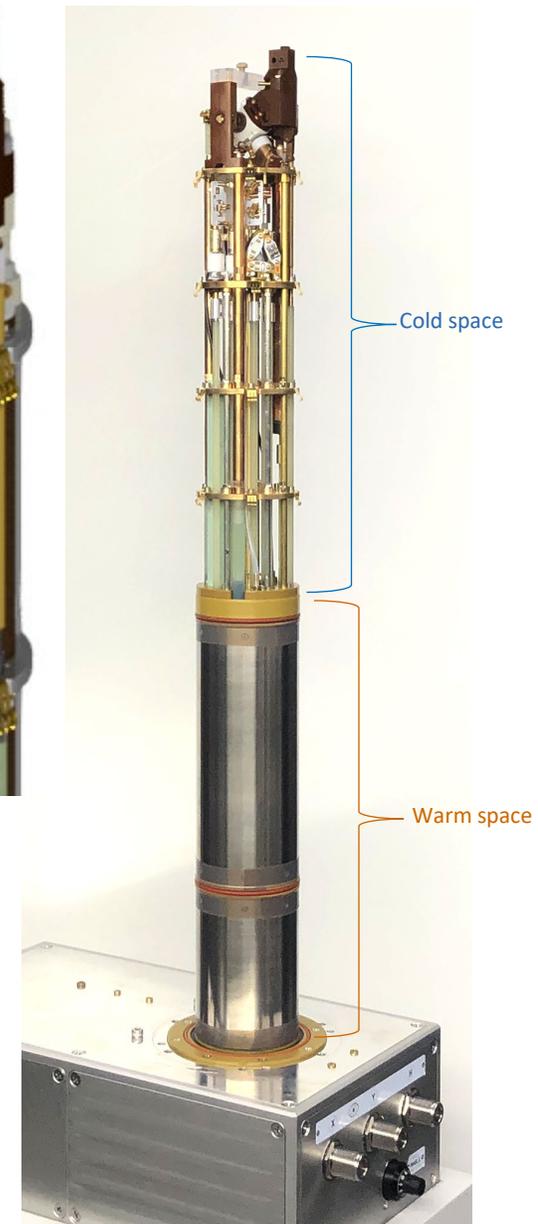
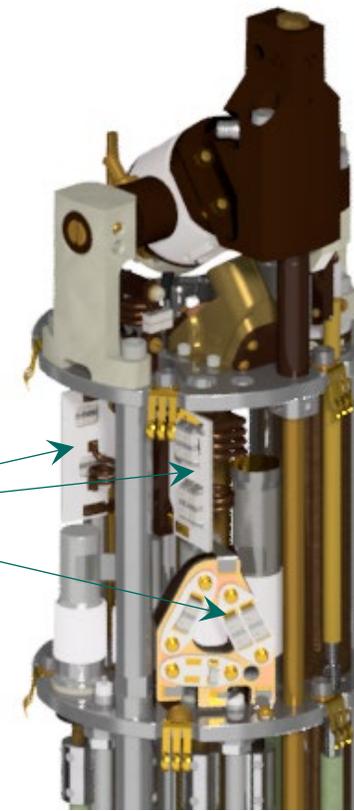
- Internal cold space reduced – 2X faster cooldown
- Colder with lower gas flows
- Evacuated exhaust - probe base is warmer
- Box weight reduced - probe ~ 1/3 lighter
- Magic angle adjustment w/ lockable knob
- Convenient connector placement
- RF tuning with knobs (not a screwdriver!)
- Compatible with Bruker LT MAS cooling cabinet



MA pivot
with
differential
screw

Works both
ways at 100 K

X/Y channels tune
cards



1.3 mm probe with new redesigned internal transfer lines

1.3 spinning speed = 40 kHz

- 1.9 mm probe coming in 2025 (24 kHz spinning)

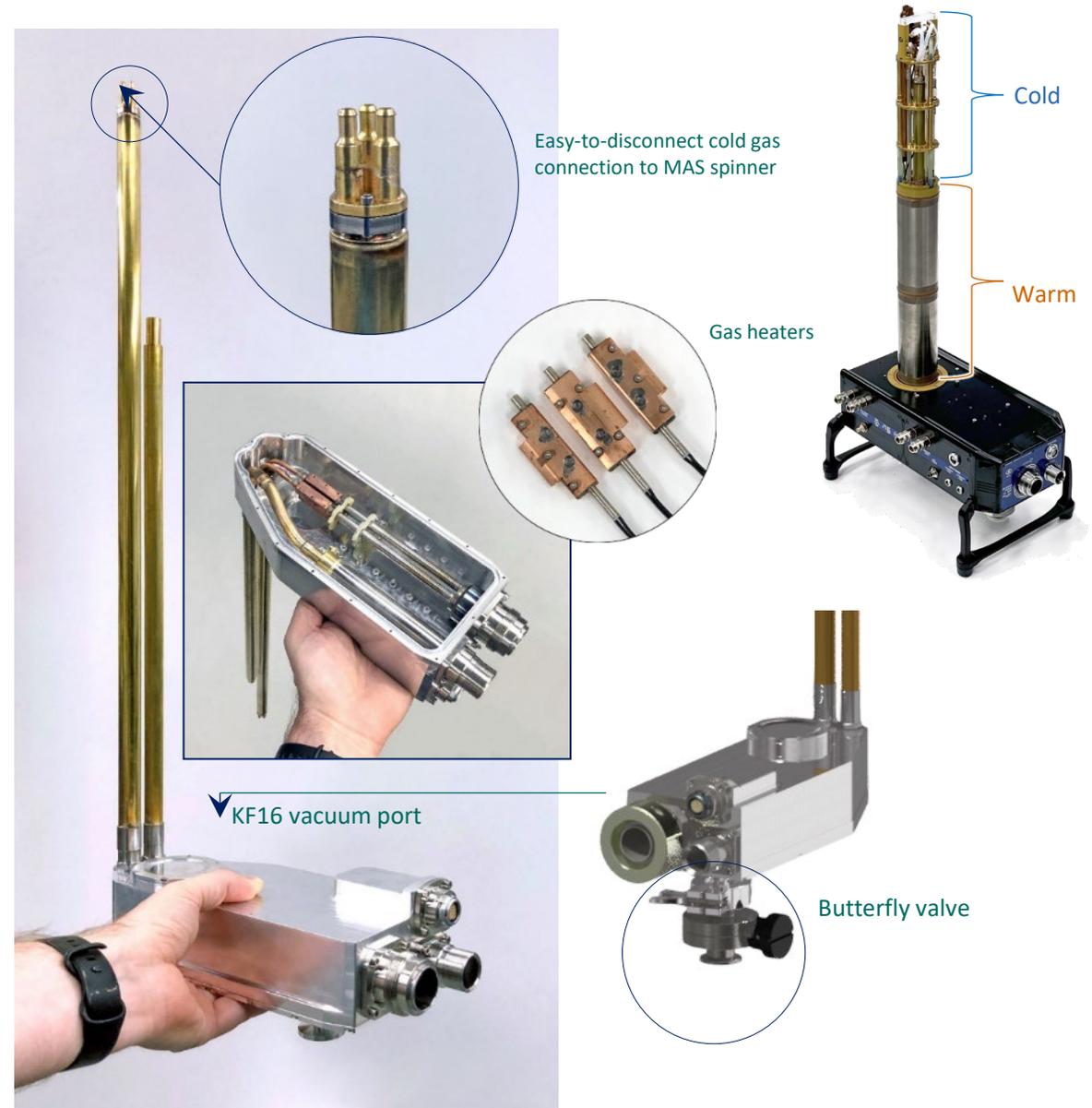
Custom internal transfer line set:

- Fully evacuated exhaust path for cold gases
- Exhaust and input gas dewars share vacuum space
- Large KF16 vacuum connector for more robust vacuum
- Butterfly vacuum valve to ease disconnection
- Gas heaters:
 - Serviceable screw-on design
 - No added back-pressure in bearing and drive channels

Includes features first implemented in the 3.2 probe:

- RF circuit
- Smaller cold space (Even smaller now)
- Custom waveguide and lens

Manuscript in prep, Scott et al

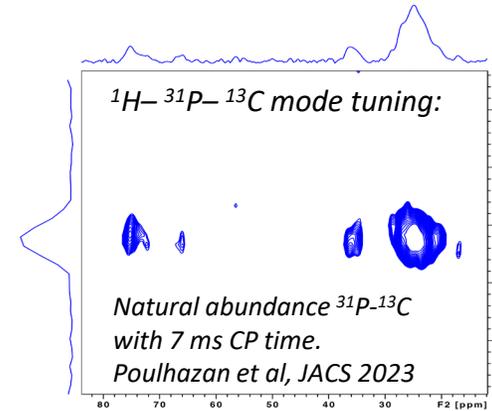
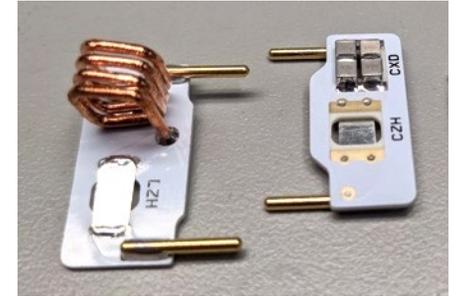


RF circuit



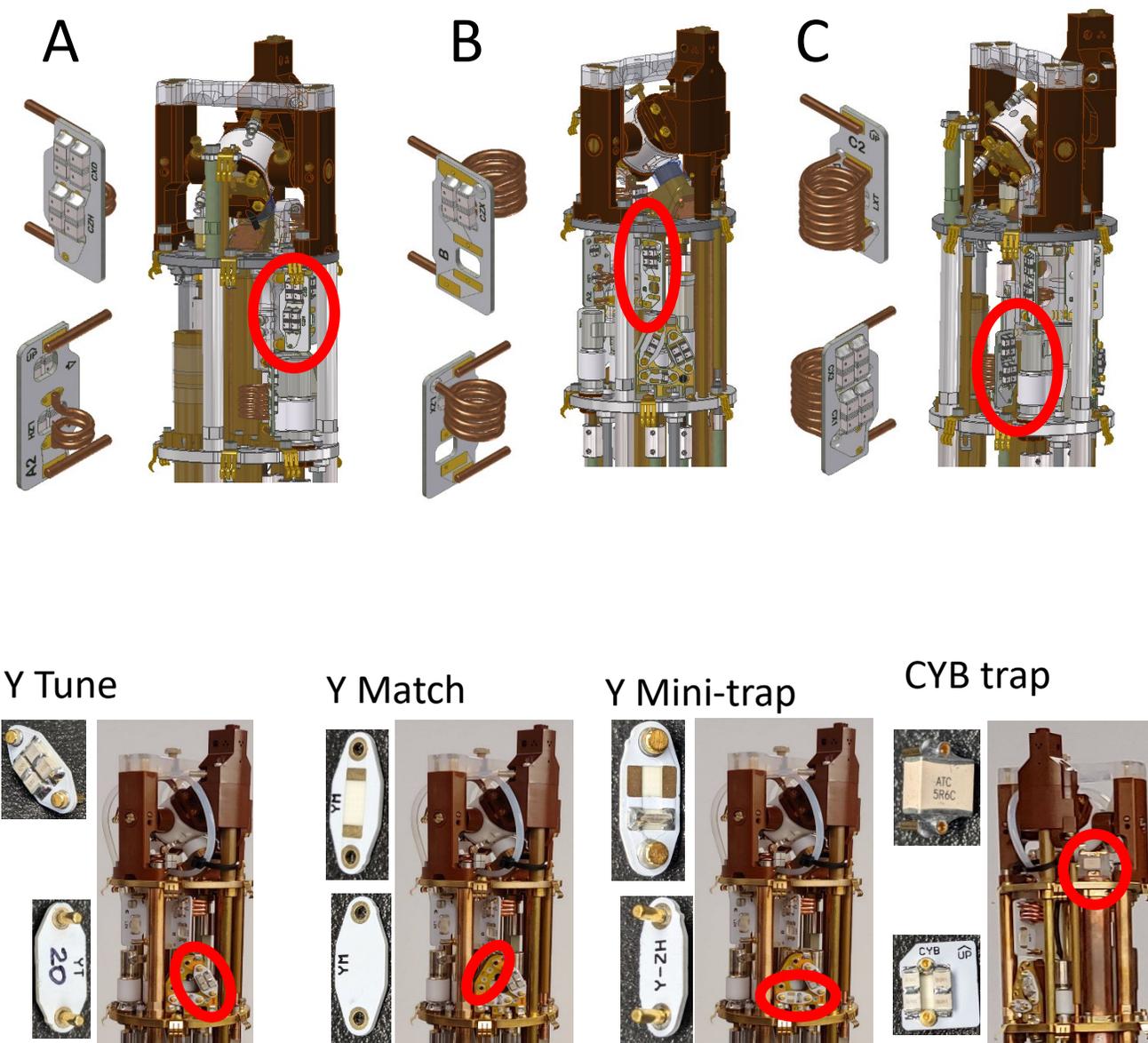
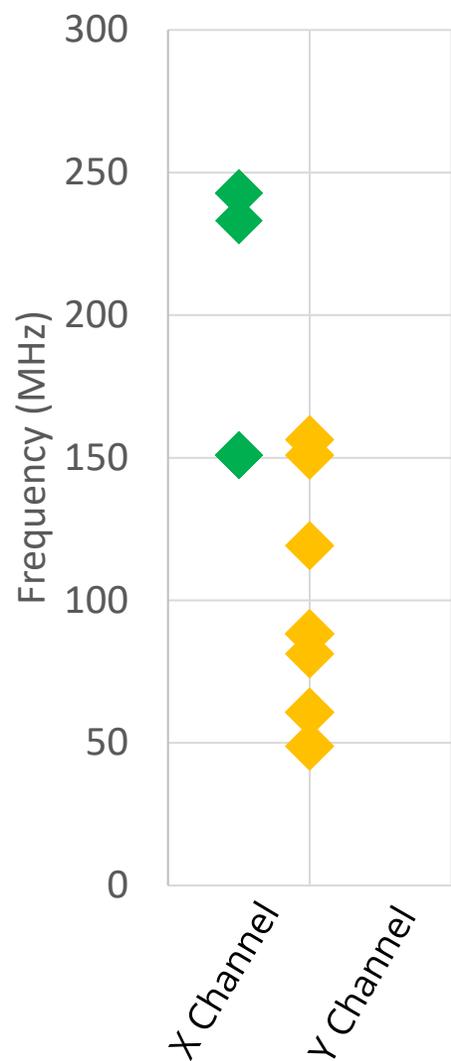
Cold RF came with challenges → variable capacitors seizing

- Tune cards for fast tuning reconfiguration
- Tune cards = consistent power levels (no playing with $\lambda/4$ and leads orientation)
- If arcing → replacement parts available on site.
- Tunable to many X-Y isotopes by swapping tune cards:
 - $^{13}\text{C}/^{15}\text{N}$, $^{13}\text{C}/^{17}\text{O}$, $^{13}\text{C}/^2\text{H}$, $^{31}\text{P}/^{13}\text{C}$, $^{31}\text{P}/^{15}\text{N}$, $^{13}\text{C}/^6\text{Li}$, $^{31}\text{P}/^{27}\text{Al}$, $^{13}\text{C}/^{29}\text{Si}$, metalloproteins, etc.
- Can switch to 2 resonance: **X** \approx [$^{14}\text{N} \dots ^{31}\text{P}$]
- ~ 2X better efficiency of proton channel- eventually will apply to fast MAS with 1.3 and 1.9 mm stators
- Fluorine detection instead of proton- exchanged trap
- Fluorine and Proton at the same time--- Not trivial!
Bruker has some probe examples of this



	Bruker standard probe	Custom NHMFL probe	Difference
100 kHz ^1H nutation	72 W	42 W	-40%
^{13}C CP condition with 50 kHz ^1H nutation	65 W	53 W	-20%
^{15}N CP condition with 50 kHz ^1H nutation	300 W	210 W	-40%

X	X Frequency (MHz)	Y	Y Frequency (MHz)
¹³ C	150.9	³⁷ Cl	48.9
¹³ C	150.9	¹⁵ N	60.8
¹³ C	150.9	¹⁷ O	81.3
¹³ C	150.9	⁶ Li	88.3
¹³ C	150.9	²⁹ Si	119.2
³¹ P	242.9	¹⁵ N	60.8
³¹ P	242.9	¹³ C	150.9
³¹ P	242.9	²⁷ Al	156.4
³¹ P	242.9	² H	92.1
³¹ P	242.9	⁶ Li	88.3
⁷ Li	233.2	⁶ Li	88.3



Acknowledgements



Frederic Mentink-Vigier

Peter Gor'kov

Thierry DuBroca

Rob Schurko

Joanna Long

Jason Kitchen

Wenping Mao

Steve Hill



RM1-GM148766



DMR 2128556



Washington
University in St. Louis

Alexander Barnes

Nicholas Alaniva Seong Ho Pahng

Brice Albert Peter Rand

Erika Sesti Eric Choi

Edward Saliba

Natalie Golota

Lauren Price

Pinhui Chen

Chukun Gao



FSU Provost's
Postdoctoral Fellowship

