EUROMAR 2022

Solid-State DNP (Dynamic Nuclear Polarization)

Making the invisible visible

DNP is microwave irradiation that transfers the 660x higher polarization of native or admixed electron spins to nuclear spins in the solvent matrix and sample. Bruker DNP systems enable solid-state NMR with unsurpassed sensitivity gains (>200x typical) excellent cold (<100K) and stability (up to 14-days continuously running experiment sets) performance in low-temperature (LT) MAS format that DNP typically utilizes. The full Bruker package allows exciting new applications in biological solids, materials science, and pharmaceuticals.

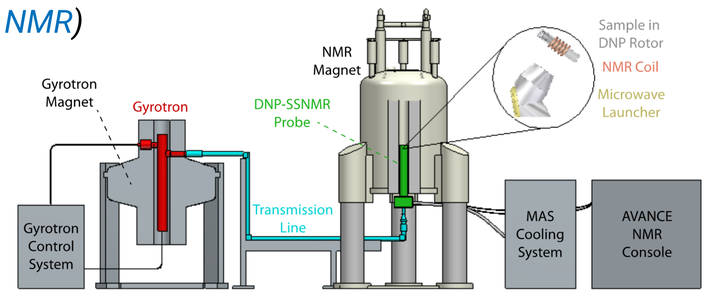
Gyrotron & Klystron Microwave Sources

Since 2008: 53 systems *installed or underway*

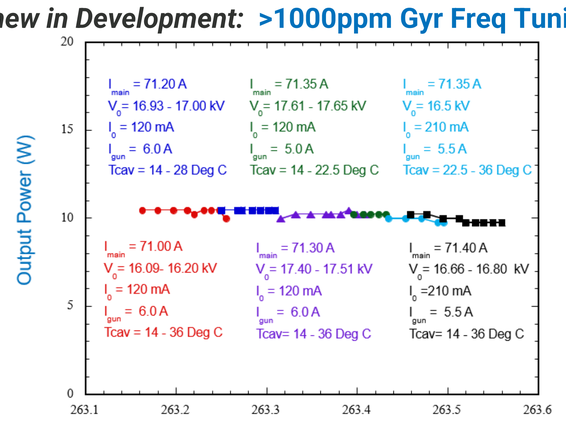
28x 400 MHz (263 GHz) *7x 800 MHz (527 GHz)*
17x 600 MHz (395 GHz) *1x 900 MHz (593 GHz)*

***Gyrotrons** (for 400 – 900 MHz NMR)*
with CPI Palo Alto

- ✓ *10 – 30 W output*
- ✓ *CF magnet*
- ✓ *stable operation*
- ✓ *up to 10 yr lifetime*

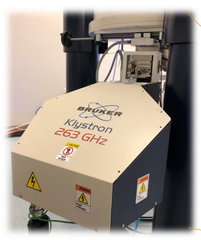


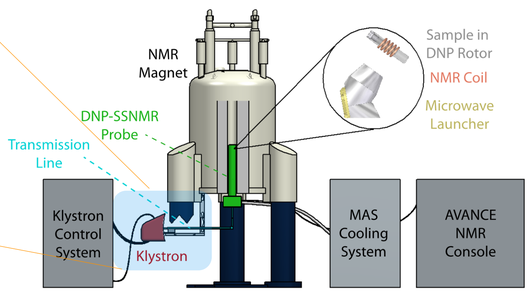
***new in Development:* >1000ppm Gyr Freq Tuning**



***Klystrons** (for 400 MHz only)* **80% DNP performance at ~70% cost !**
with CPI Canada

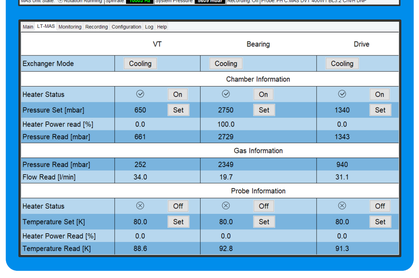
- ✓ *5 W output*
- ✓ *stable operation*
- ✓ *small footprint*
- ✓ *simple facility needs*
- ✓ *20k operational hours*
  (up to 5-10 years, dep. on usage)

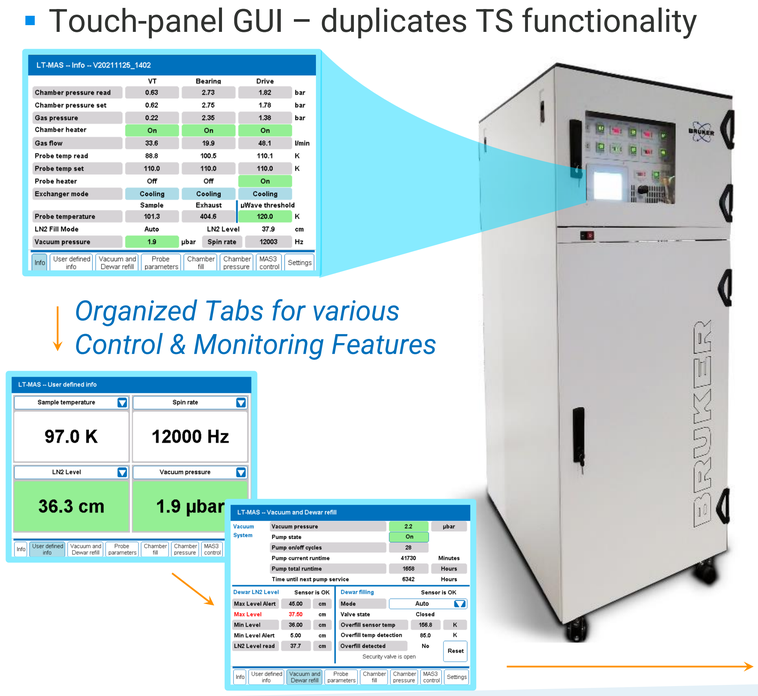




3rd-generation LT-MAS Cooling Cabinet

- Updated main control unit
  - ✓ integrated *Topspin* control (≥4.1.3, ≥3.6.5) →→
  - ✓ improved regulation (pressure, flow and fill)
  - ✓ integrated venturi for fast probes (≤1.9 mm)
- MAS-3 controller integration
  - ✓ automated cold insert/eject & spin up/down
  - ✓ best-in-field spin regulation
- Improved cryogenic efficiency
- Touch-panel GUI – duplicates TS functionality



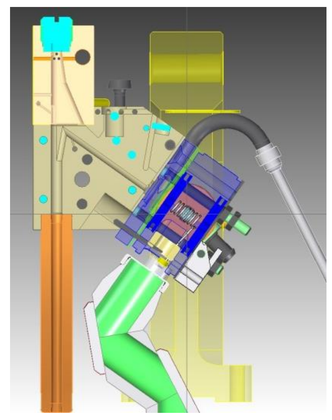


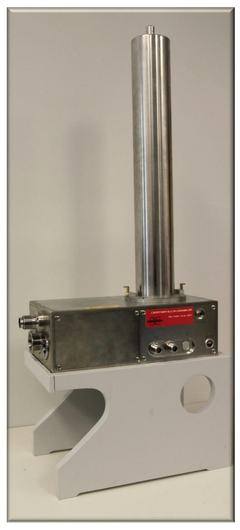
Organized Tabs for various Control & Monitoring Features

LT-MAS DNP probes

A large DNP probes portfolio covers Classic & Emerging Apps.

- High-efficiency microwave coupling to the sample is a key for best enhancements.
- Varieties for many applications
  - ✓ H/X/Y (*various X/Y combos on one probe*)
  - ✓ Low-gamma probes
- Static probes (*e.g., a key for battery apps*)
- High-performance LT-MAS
  - ✓ 3.2 mm (15 kHz MAS @ 100 K)
  - ✓ 1.9 mm (24 kHz MAS @ 100 K)
  - ✓ 1.3 mm (40 kHz MAS @ 100 K)
  - ✓ 0.7 mm (65 kHz MAS @ 100 K)
- All with cold insert/eject





FAST LT-MAS DNP probes (1.3 & 0.7 mm, for 40 & 65 kHz at 100K)

Fast spinning provides state-of-the-art spectral performance in LT-MAS DNP, via averaging of anisotropic interactions (resolution), longer coherence lifetimes, and larger RF field strengths. Fast MAS plus DNP provides the ultimate in sensitivity & resolution to enable new applications.

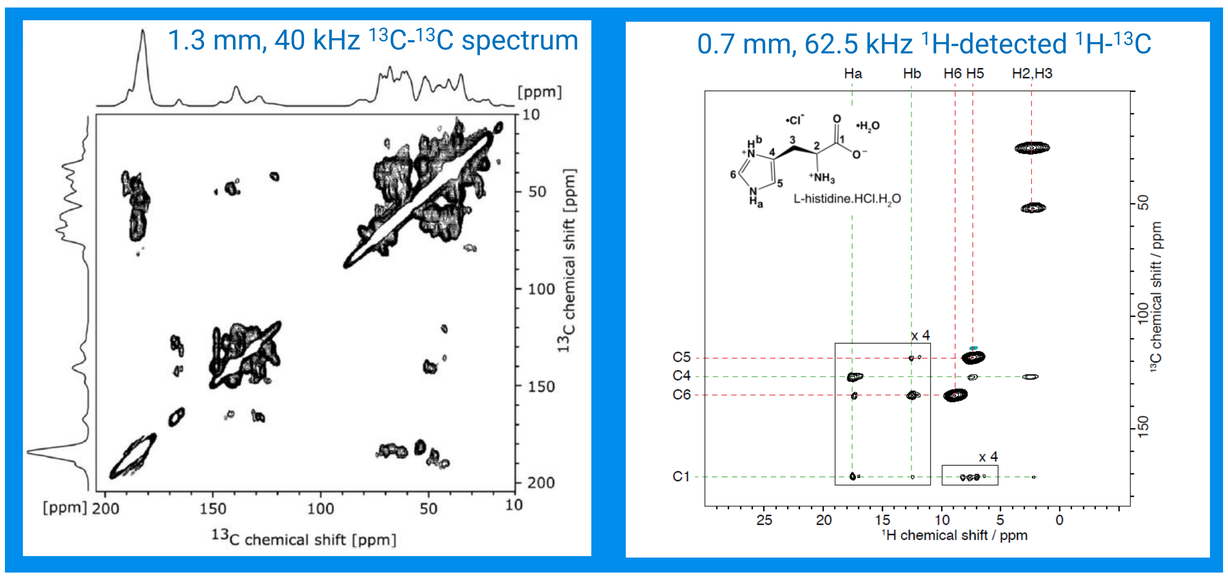


Fig.1 DNP-enhanced 2D SQ-SQ ^{13}C-^{13}C correlation spectrum using DARR with U-^{13}C,^{15}N LecA protein impregnated with 32 mM of the radical cAsymPol-POK in glycerol-d_8/D_2O (60/40), 1.3 mm rotor at 40 kHz MAS, 400 MHz ^1H (9.4 T) and *T* = 106 K.

Data courtesy of G. de Paëpe, S. Hediger (CEA Grenoble, France)
See also, Angew. Chem. (2022)

Fig.2 DNP enhanced, ^1H detected ^1H-^{13}C HETCOR of [U-^{13}C,^{15}N] histidine·HCl·H_2O sample impregnated with 32 mM of the radical HyTEK2 in TCE solvent, 0.7 mm rotor at 62.5 kHz MAS, and 900 MHz ^1H (21.1 T) and *T* = 105 K.

Data courtesy of P. Berruyer, L. Emsley (EPFL Lausanne, Switzerland)
See also, JPC Lett. (2020).

Conclusion

- Bruker offers turn-key solutions for DNP-enhanced solids NMR from 400 – 900 MHz with strong track records for reliability & cutting-edge developments.
- High-power Gyrotron microwave sources meet all needs across bio, materials and pharma Apps.
- Klystrons provide a cost- and user-friendly package for near-max DNP performance at 400 MHz
- Low-temperature (100 K) MAS probes optimize DNP enhancement combined with best-in-field spinning.
- High-performance, automated, user-friendly LT-MAS control makes DNP more accessible than ever.

TECHNOLOGY & APPLICATIONS