Sample Temperature in a MAS probe

BRUKER

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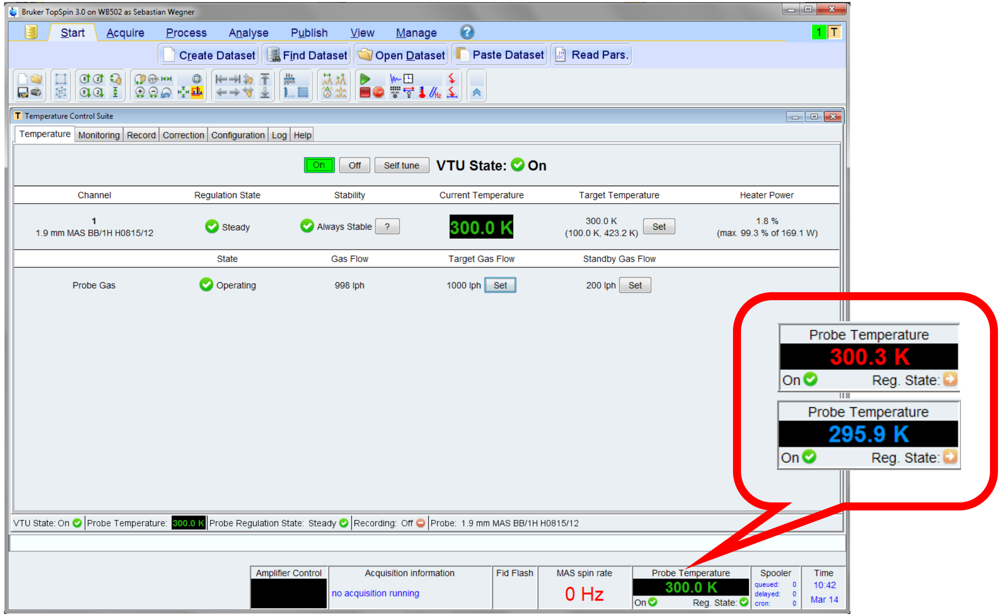
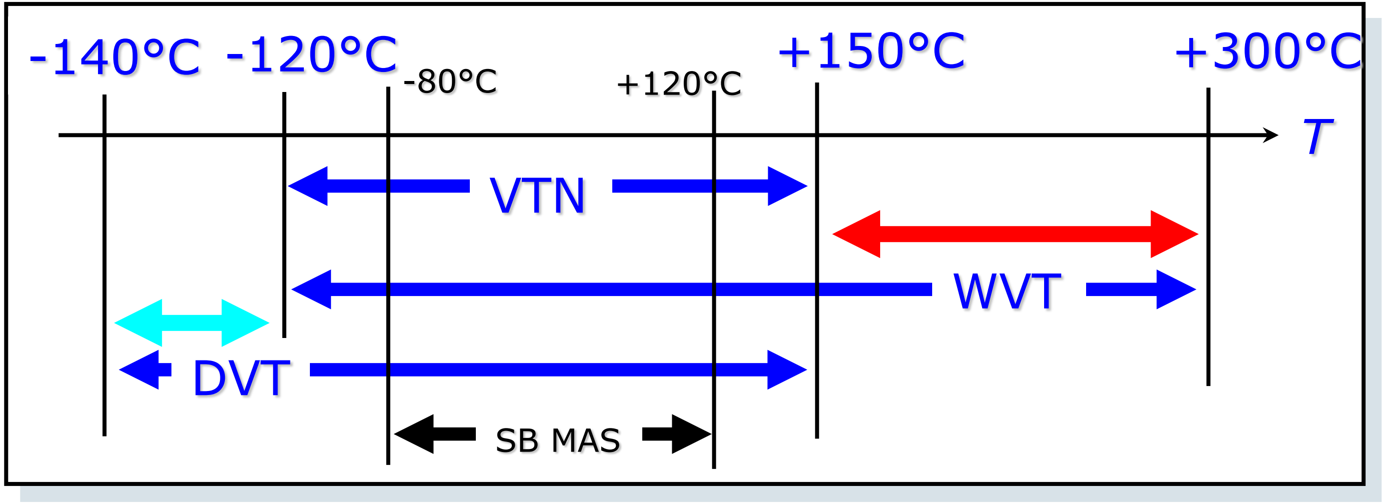
Bruker BioSpin Billerica, USA
Bruker Users Meeting China, July 23th – 26th 2019

Software - EDTE



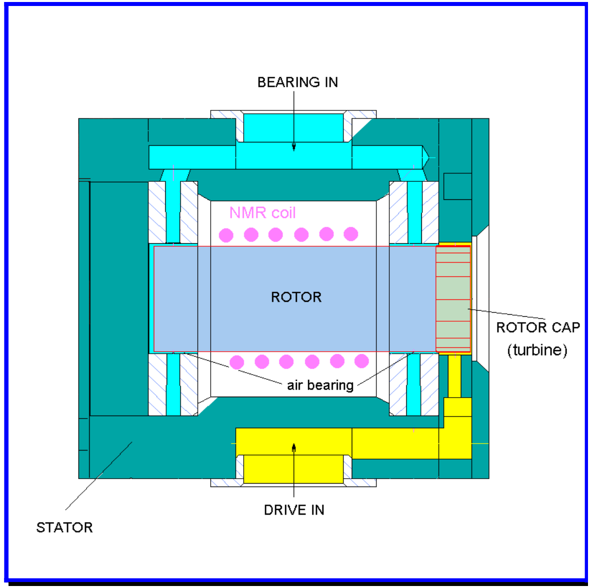
VT ranges of WB MAS probes

VTN : Variable Temperature Normal (BN-Stator)
WVT : Wide Variable Temperature (MgO-Stator)
DVT : Direct Variable Temperature (BN-Stator)

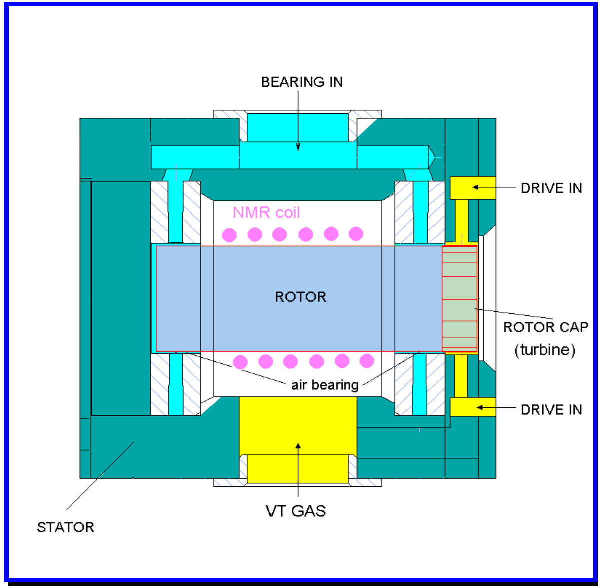


Exception: 2.5 mm probes: -30°C ↔ +70°C

VT – probe design

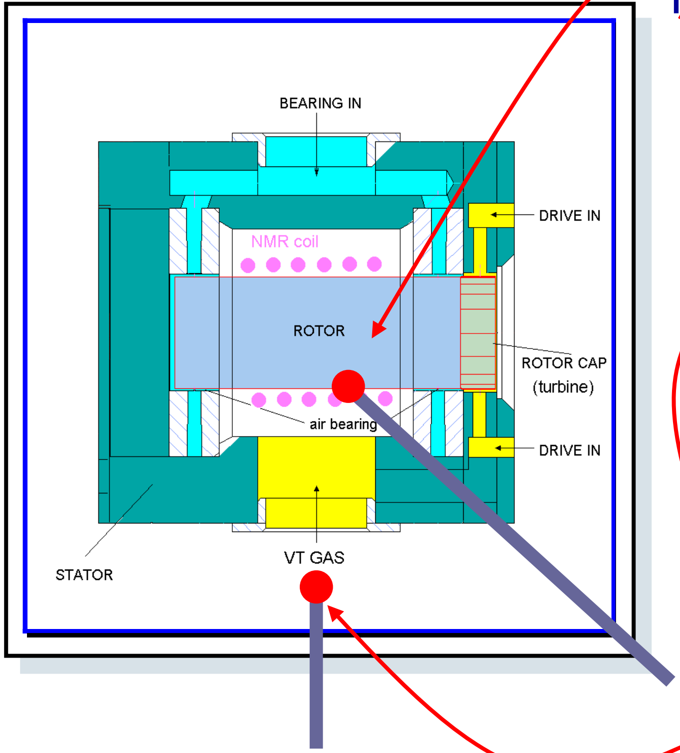


VTN / WVT
bearing gas for temperature regulation

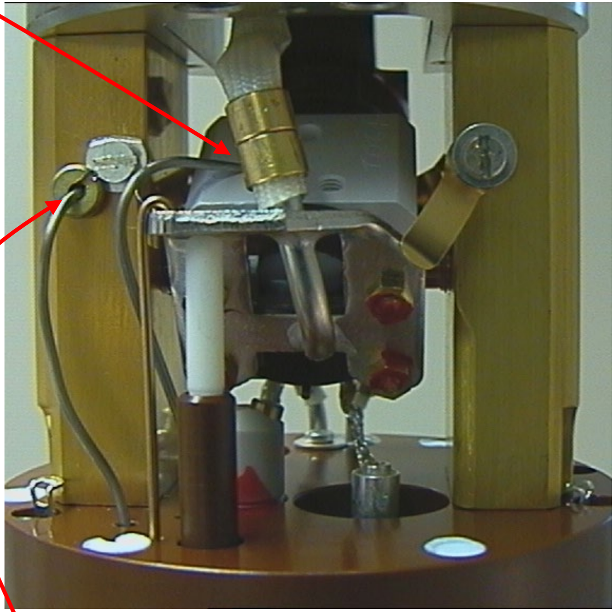


DVT
separate VT gas line

VT – DVT design



TC 1: control (read)



PH MAS DVT 600 WB BL4 (H8724)

TC 2: regulation (regul)

Sample Temperature in a MAS probe

NMR Thermometer:

Lead Nitrate $Pb(NO_3)$ $T = \square \cdot 0.753\frac{ppm}{K}$

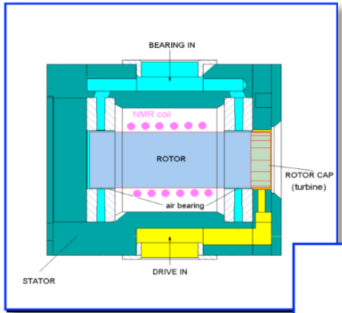
Bielecki, A. et al, J. Magn. Reson. 116, 215 – 220, 1995

Sample Temperature in a MAS probe

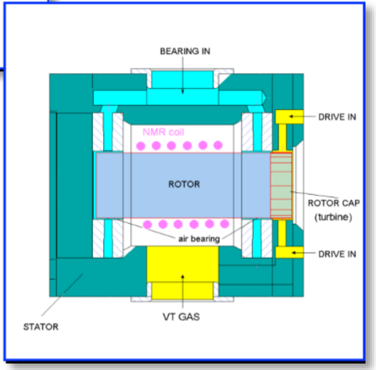
–VT design: Stator Designs

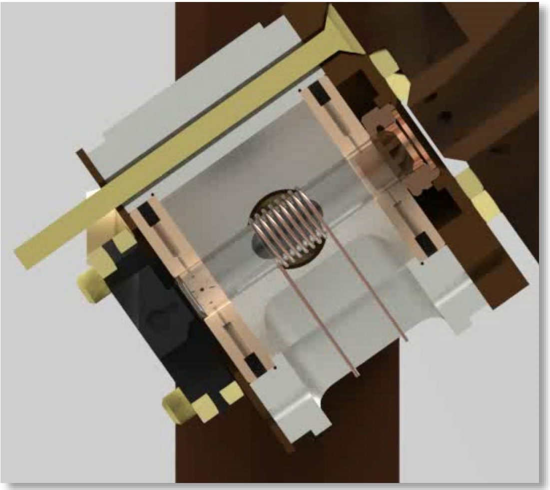
- Thermocouple locations remote from rotor

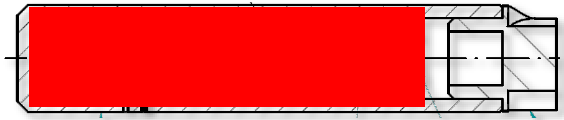
VTN

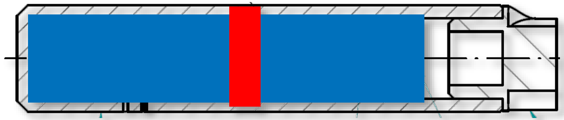


DVT

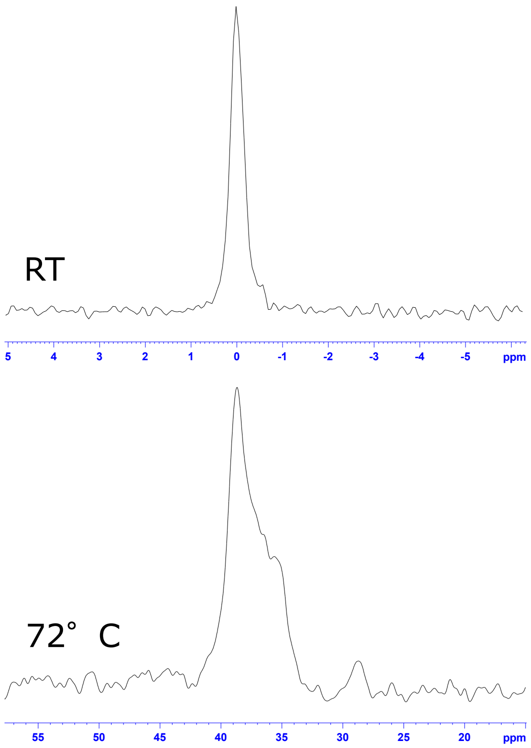








Thermal Gradient: Center Packed Rotor



- **Leadnitrate at room temperature:**

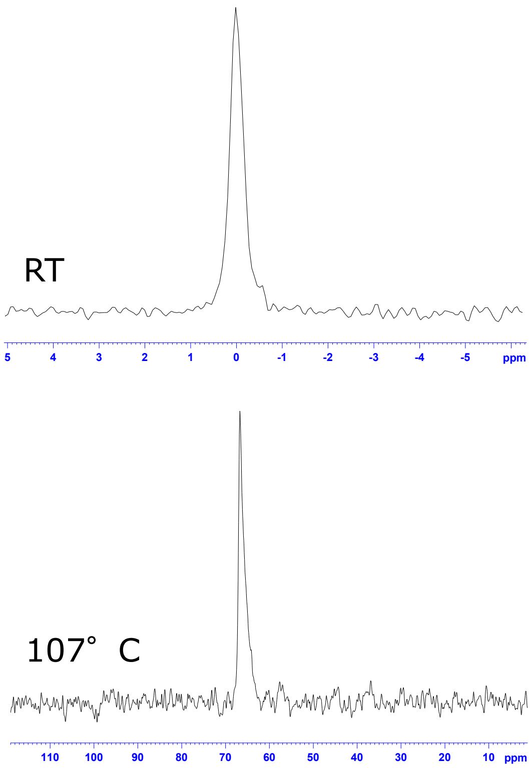
  FWHM: 0.34ppm / <1K

- **Leadnitrate at 72° C:**

  FWHM: app. 5ppm / 6K

- **Flow Rate: 500l/h**

Thermal Gradient Center Packed Spinner



- **Leadnitrate at room temperature:**

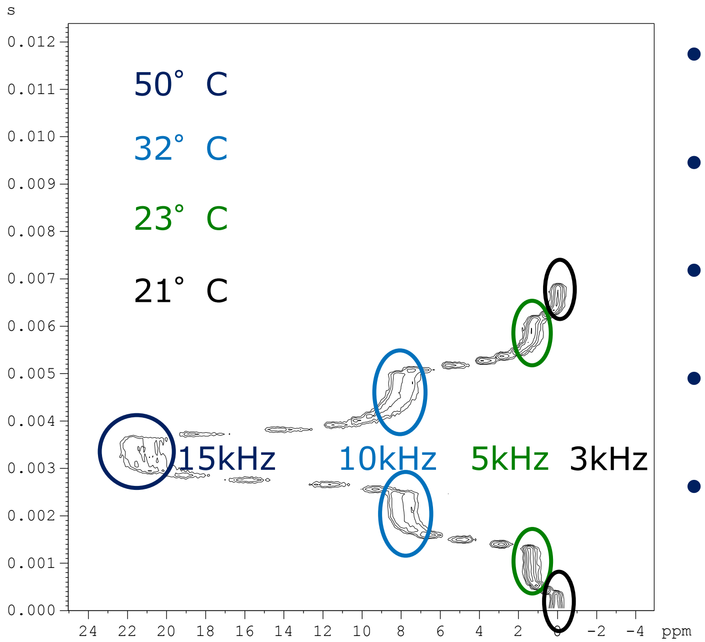
  FWHM: 0.34ppm / <1K

- **Leadnitrate at 107° C:**

  FWHM: app. 2ppm / 2K

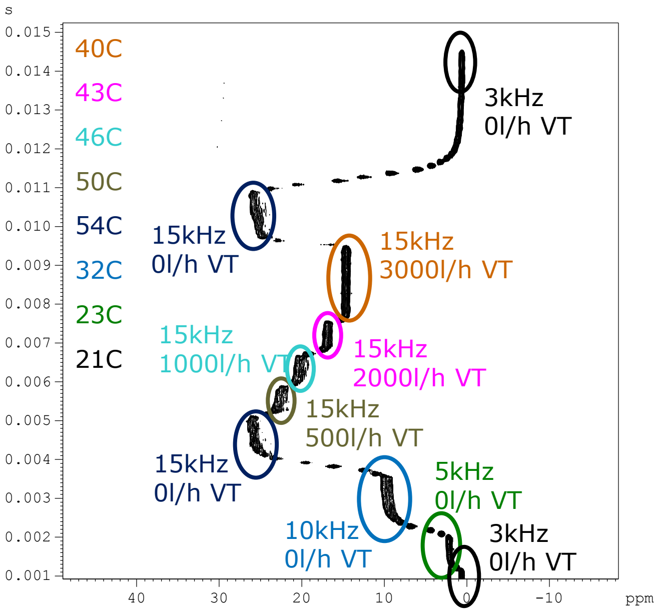
- **Flow Rate: 3000l/h**

Influence of magic-angle spinning on the temperature



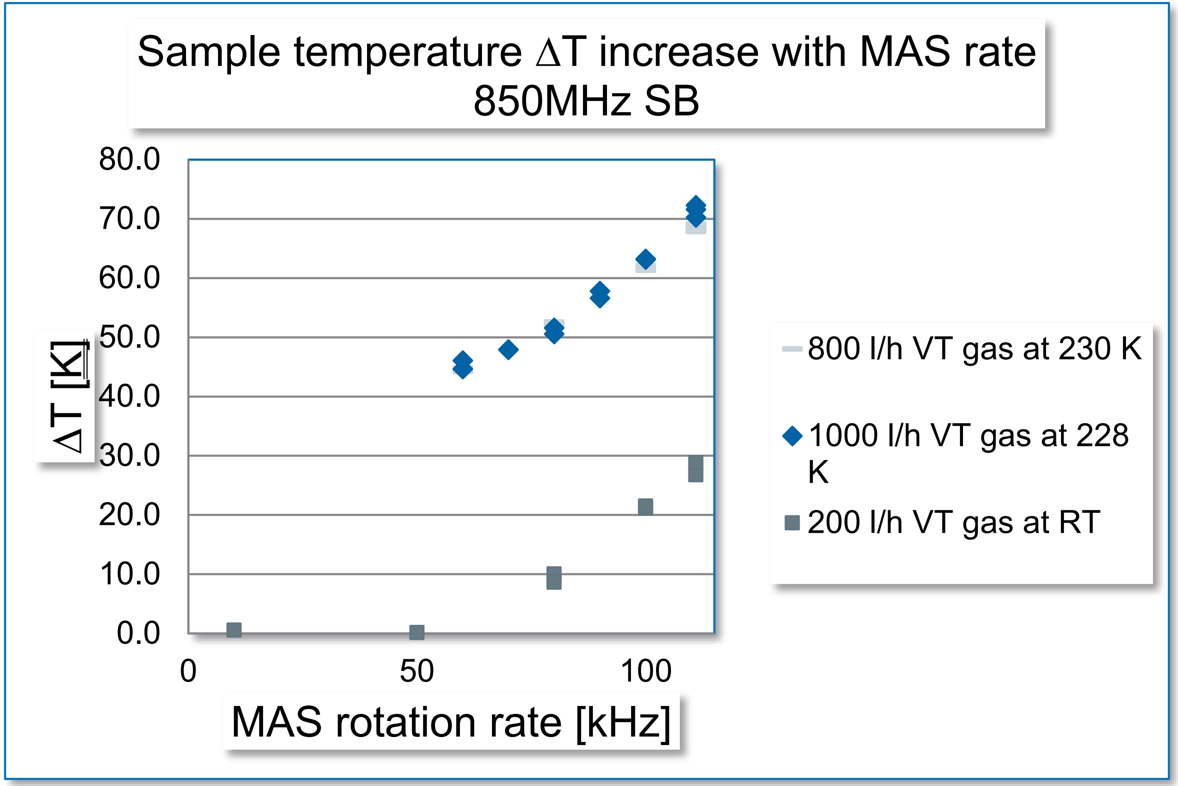
- Center packed sample (4 mm, ZrO_2 powder)
- 21° C at 3 kHz (room temperature, referencing)
- Lead nitrate for temperature calibration, 0.753 ppm/K
- Spin rate was changed within running experiment
- Maximum temperature difference about 30 K above RT

Influence of magic-angle spinning and VT gas on the temperature



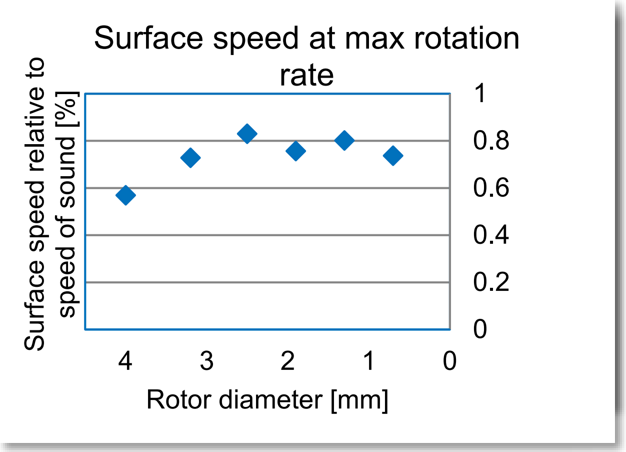
- Center packed sample (4 mm, ZrO_2 powder)
- 21° C at 3 kHz (room temperature, referencing)
- Lead nitrate for temperature calibration, 0.753 ppm/K
- Spin rate was changed within running experiment and VT gas flow was adjusted
- Maximum temperature difference about 34 K above RT

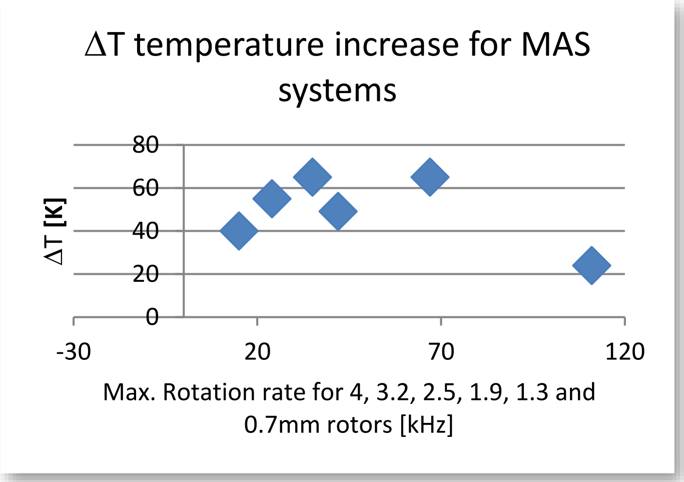
The DVT Problem

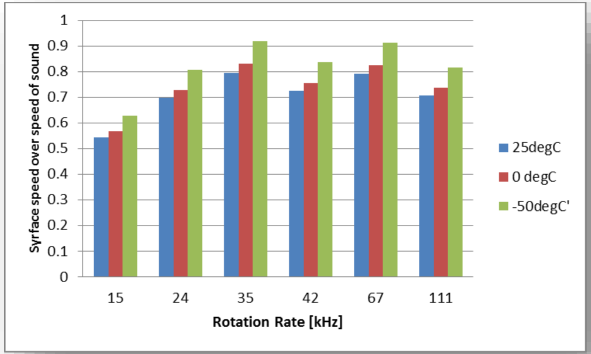


Sample Temperature in a MAS probe

- Frictional heating
  - Surface speed and speed of sound







Sample Temperature in a MAS probe

–NMR Thermometers

- Lead Nitrate $Pb(NO_3)$ $T = \square \cdot 0.753 \frac{ppm}{K}$

  Bielecki, A. et al, J. Magn. Reson. 116, 215 – 220, 1995

- Stannates, $Sm_2Sn_2O_7$ + SnO_2 $T = \frac{8.87 \cdot 10^4}{204 - \delta cs}$ approx: $T \cong \square \cdot 1 \frac{ppm}{K}$ $+\delta T$

  Grey, C.P., et al, J. Magn. Reson. A. 101, 299 – 306, 1993.

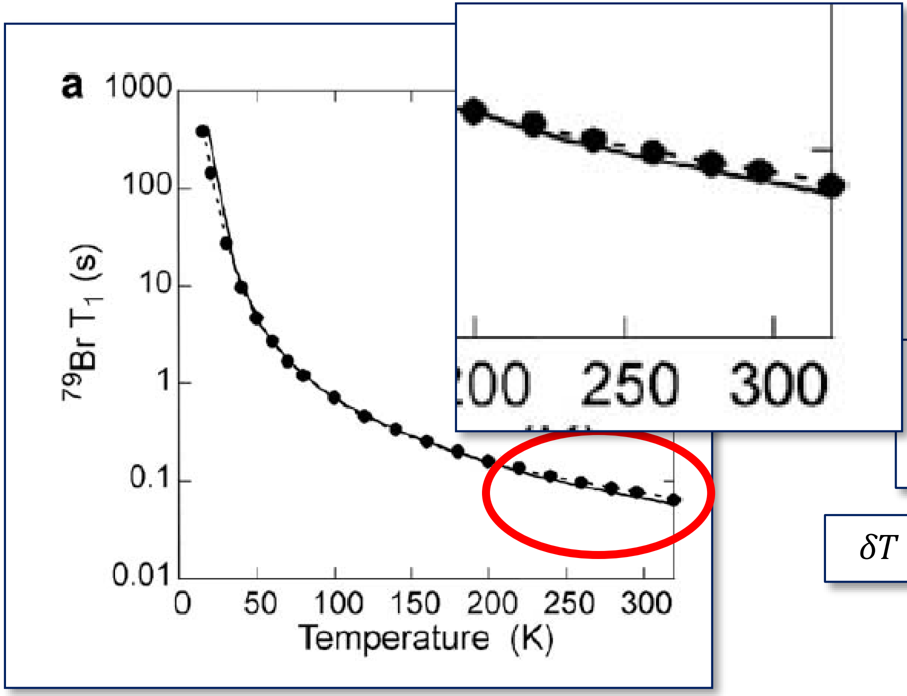
- *KBr*
  - Chemical shift $T = \square \cdot 0.024 \frac{ppm}{K}$
  - T_1-experiment $T = \sqrt{\frac{5330}{T_1 - 0.0145}} T = \sqrt{\frac{5330}{T_1 - 0.0145}} + \delta T$

    Thurber, K.R., et al., J. Magn. Reson. 196, 84–87, 2009.

    Sarkar, R., et al., J. Magn. Reson. 212, 460-462, 2011

Sample Temperature in a MAS probe

$$T_1 = 0.0145 + 5330T^{-2} + (1.42 * 10^7)T^{-4} + (2.48 * 10^9)T^{-6}$$



$$T_{T_1} = \sqrt{\frac{5330}{T_1 - 0.0145}}$$

$$T = \sqrt{\frac{5330}{T_1 - 0.0145}} + \delta T$$

$$\delta T = T_{read} - T_{T_1}$$

Thank you!

谢谢

结束

Innovation with Integrity