

Sample Temperature in a MAS probe



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



Software - EDTE



Temperature Control Suite

Temperature Monitoring Record Correction Configuration Log Help

VTU State: On

Channel	Regulation State	Stability	Current Temperature	Target Temperature	Heater Power
1 1.9 mm MAS BB/1H H0815/12	<input checked="" type="checkbox"/> Steady	<input checked="" type="checkbox"/> Always Stable	300.0 K	300.0 K (100.0 K, 423.2 K) <input type="text" value="Set..."/>	1.8 % (max. 99.3 % of 169.1 W)

State	Gas Flow	Target Gas Flow	Standby Gas Flow
Probe Gas <input checked="" type="checkbox"/> Operating	998 lph	1000 lph <input type="text" value="Set..."/>	200 lph <input type="text" value="Set..."/>

VTU State: On | Probe Temperature: 300.0 K | Probe Regulation State: Steady | Recording: Off | Probe: 1.9 mm MAS BB/1H H0815/12

Amplifier Control: Acquisition information: no acquisition running | Fid Flash: MAS spin rate: 0 Hz | Probe Temperature: 300.0 K Reg. State: | Spooler: Time: 10:42 Mar 14

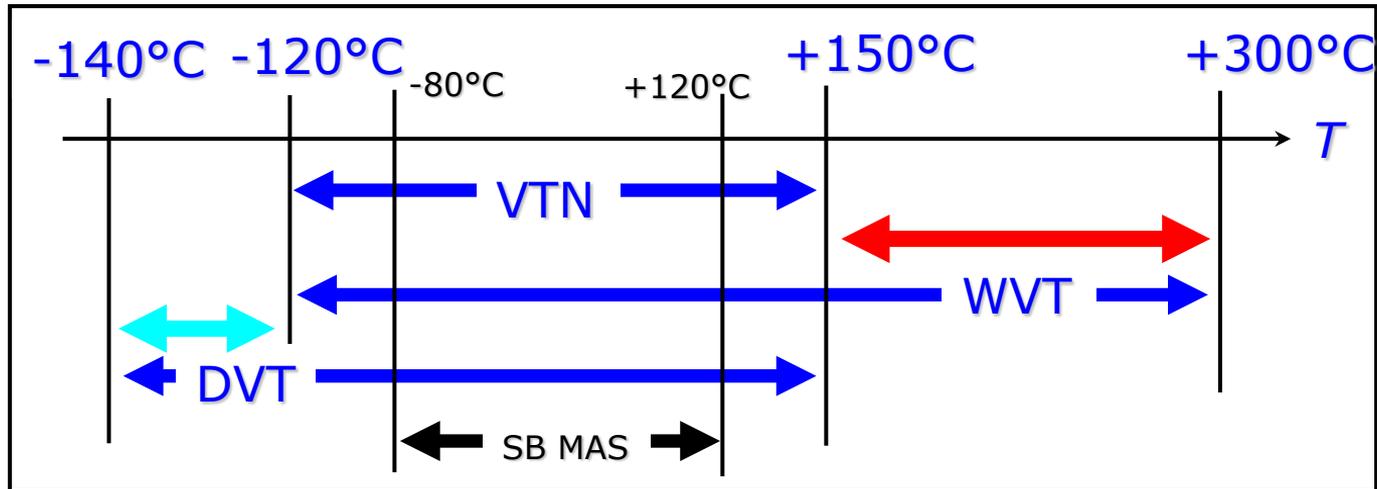
Probe Temperature
300.3 K
On Reg. State:

Probe Temperature
295.9 K
On Reg. State:

VT ranges of WB MAS probes

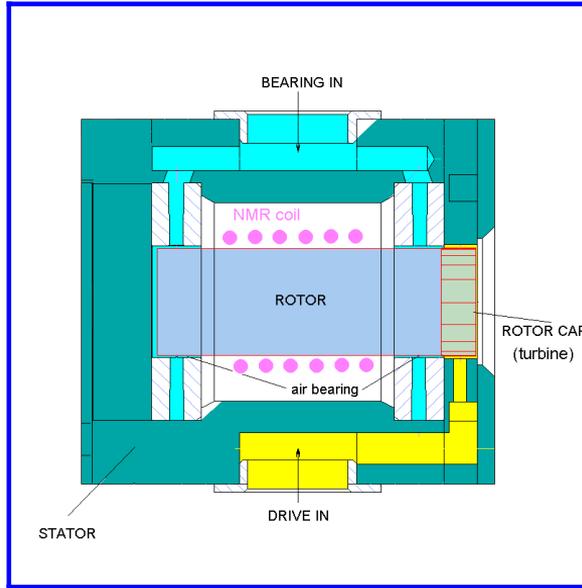


- VTN** : Variable Temperature Normal (BN-Stator)
- WVT** : Wide Variable Temperature (MgO-Stator)
- DVT** : Direct Variable Temperature (BN-Stator)

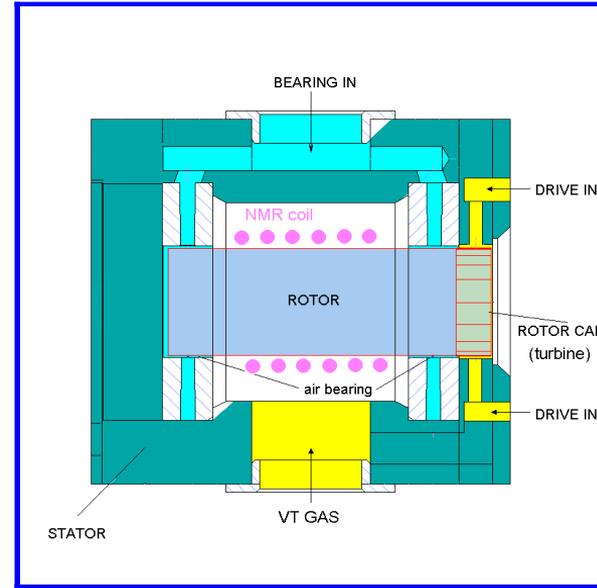


Exception: 2.5 mm probes: -30°C ↔ +70°C

VT – probe design

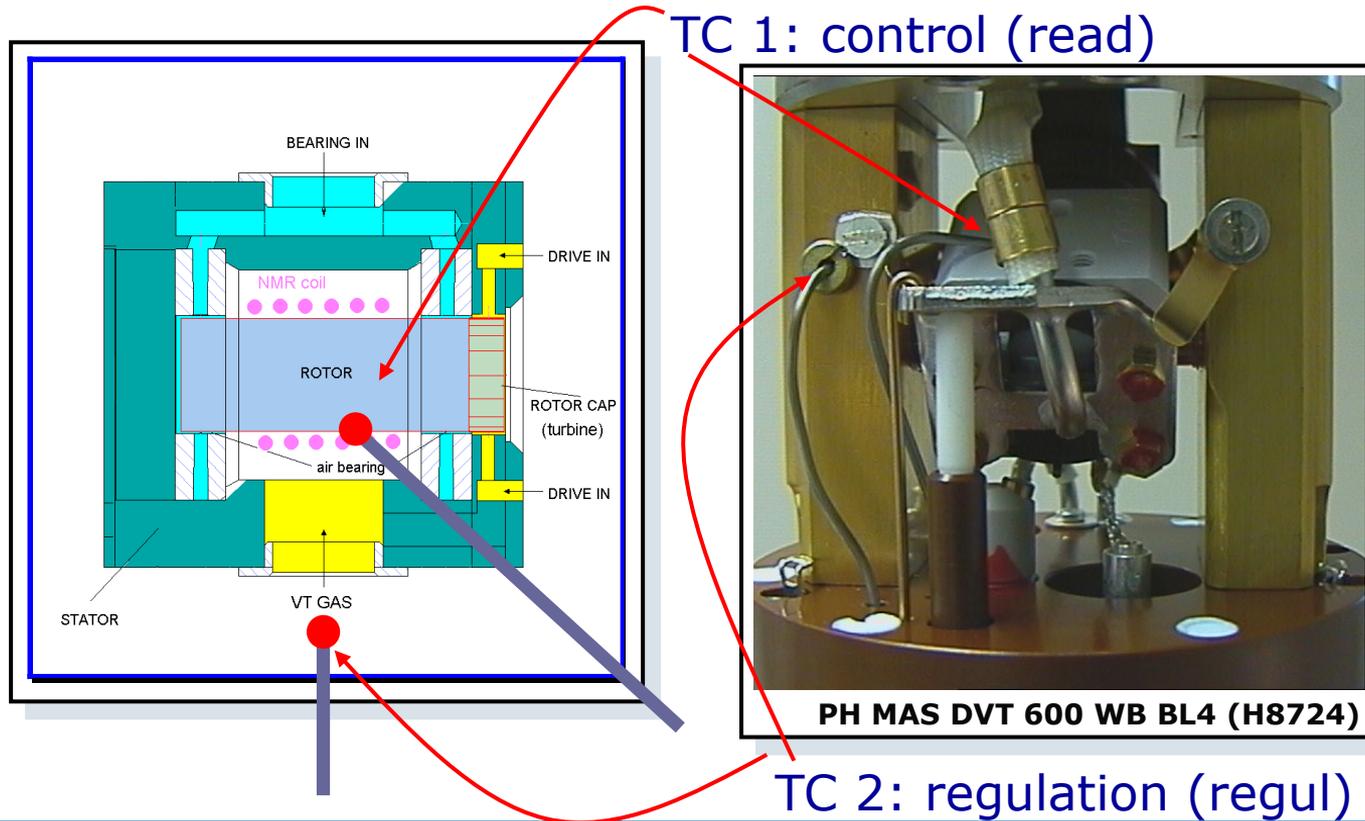


VTN / WVT
bearing gas for temperature regulation



DVT
separate VT gas line

VT – DVT design



Sample Temperature in a MAS probe



NMR Thermometer:

$$\text{Lead Nitrate Pb(NO}_3\text{)} \quad T = \square \cdot 0.753 \frac{\text{ppm}}{\text{K}}$$

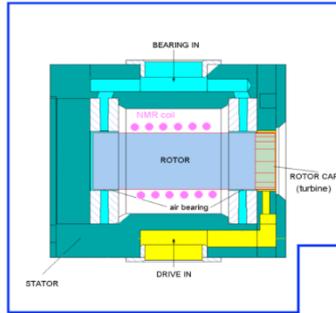
Bielecki, A. et al, J. Magn. Reson. 116, 215 – 220, 1995

Sample Temperature in a MAS probe

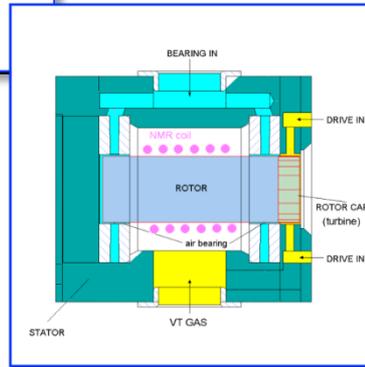


-VT design: Stator Designs

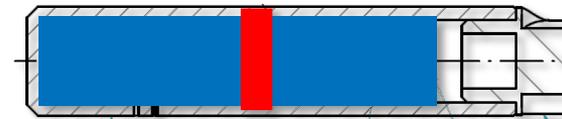
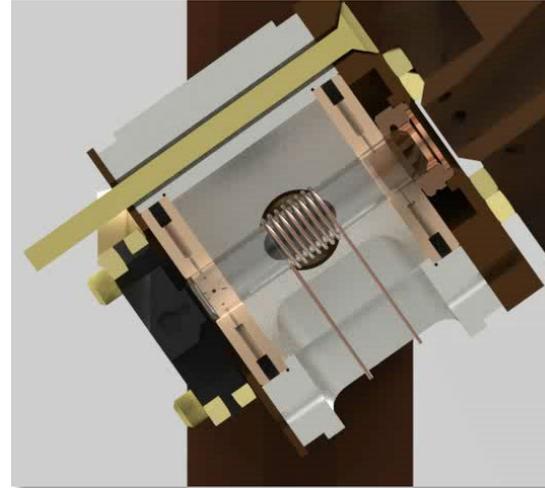
- Thermocouple locations remote from rotor



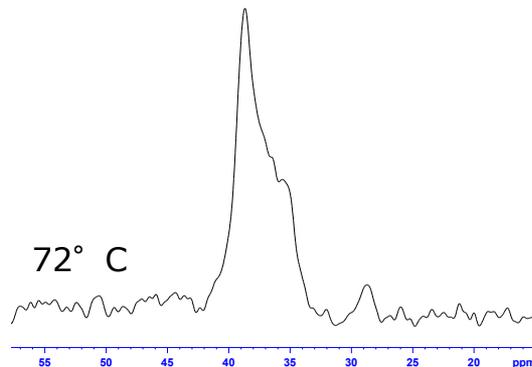
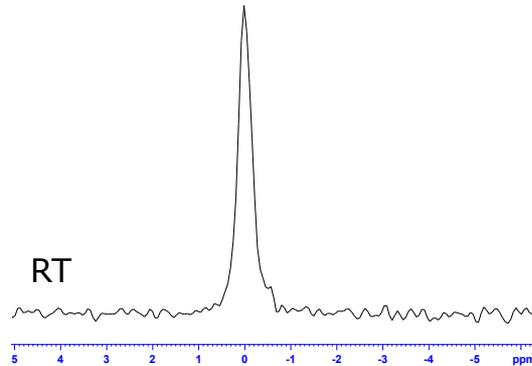
VTN



DVT

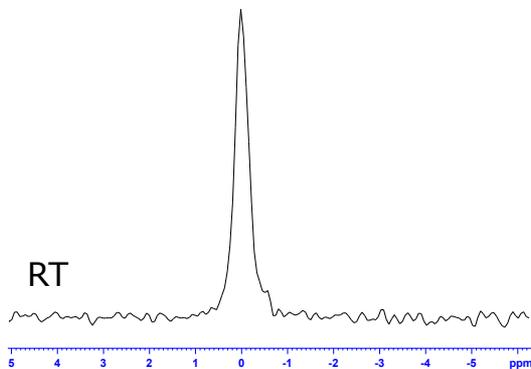


Thermal Gradient: Center Packed Rotor



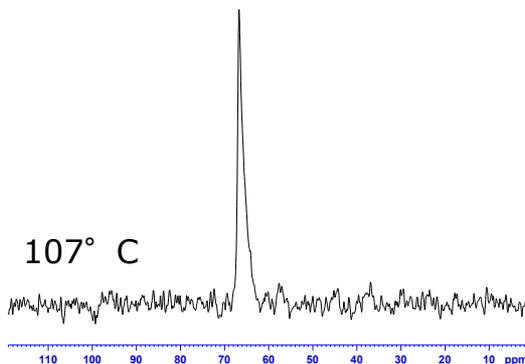
- **Leadnitrate at room temperature:**
FWHM: 0.34ppm / <1K
- **Leadnitrate at 72° C:**
FWHM: app. 5ppm / 6K
- **Flow Rate: 500l/h**

Thermal Gradient Center Packed Spinner



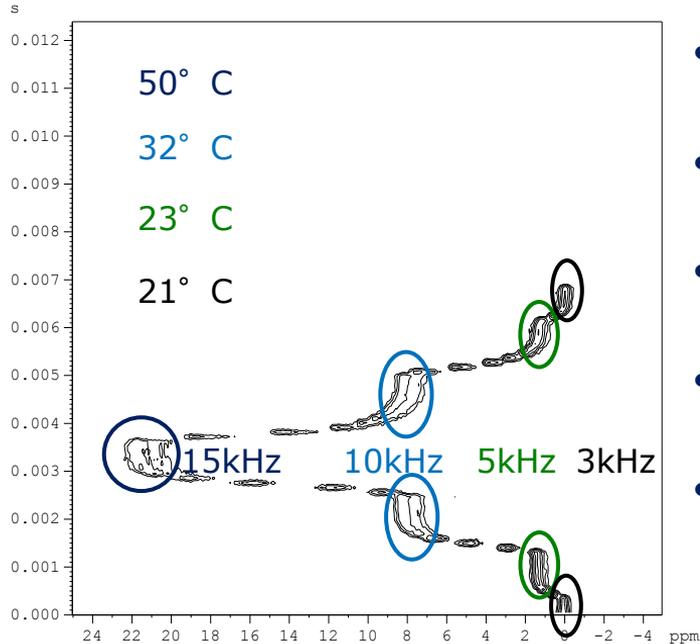
- **Leadnitrate at room temperature:**

FWHM: 0.34ppm / <1K



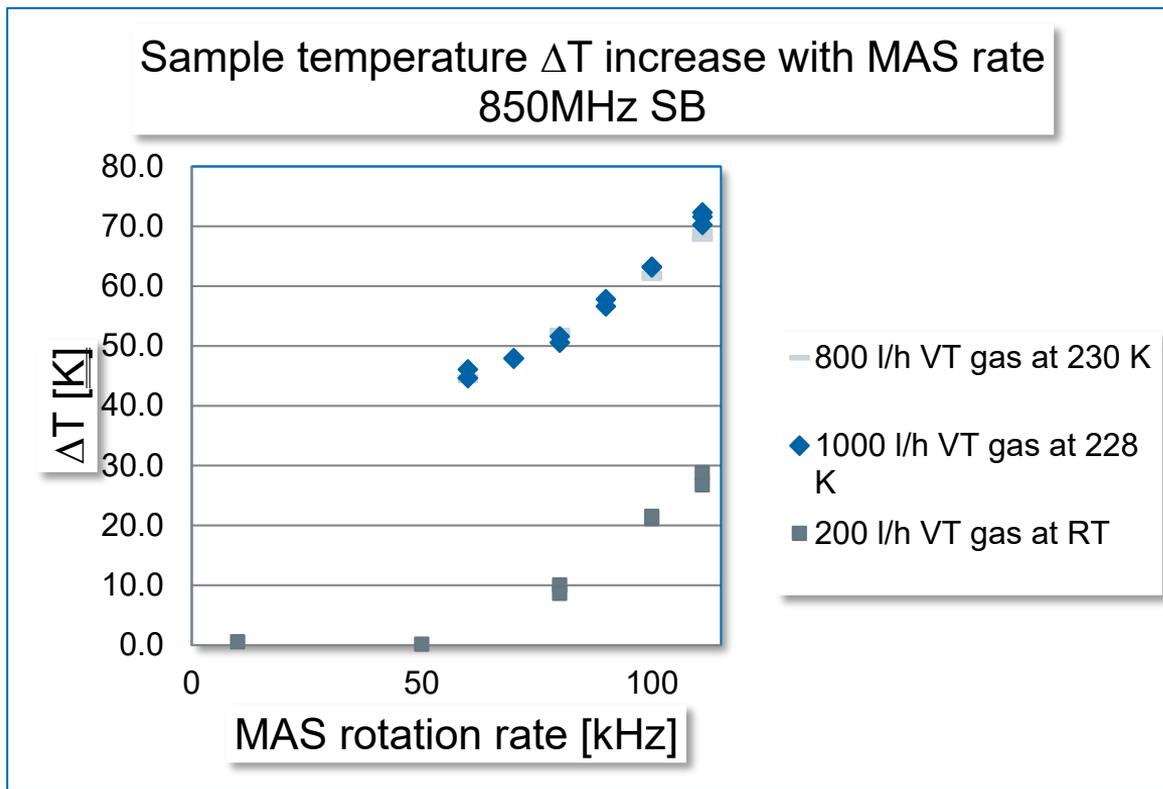
- **Leadnitrate at 107° C:**
FWHM: app. 2ppm / 2K
- **Flow Rate: 3000l/h**

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

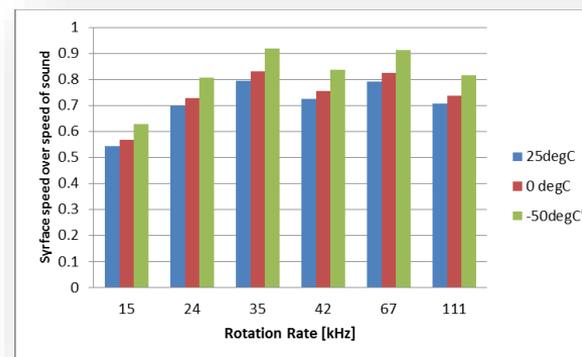
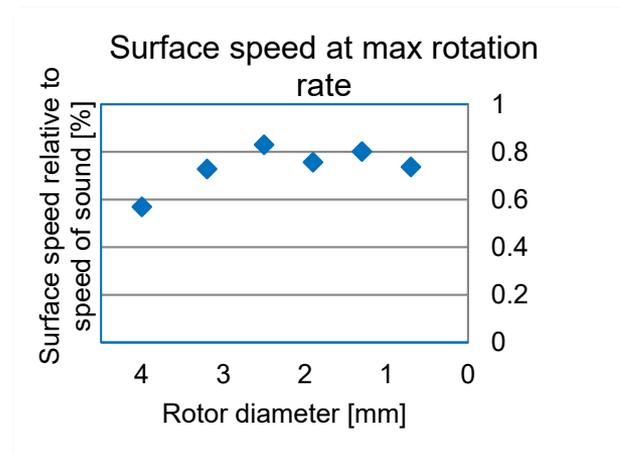
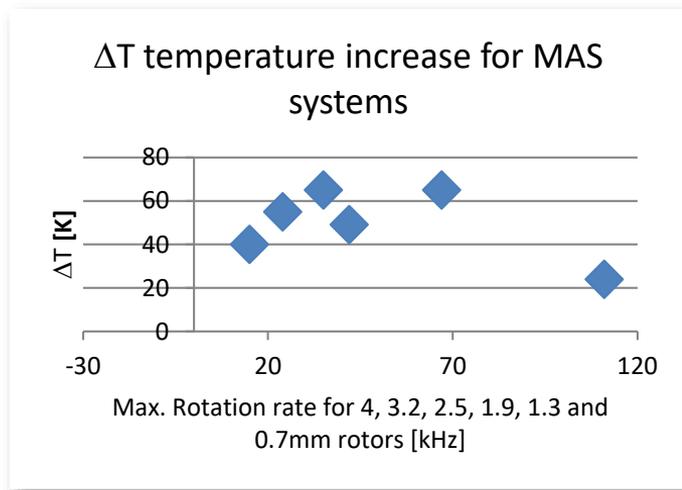
The DVT Problem



Sample Temperature in a MAS probe



- Frictional heating
 - Surface speed and speed of sound



Sample Temperature in a MAS probe



-NMR Thermometers

- Lead Nitrate $\text{Pb}(\text{NO}_3)_2$ $T = \square \cdot 0.753 \frac{\text{ppm}}{\text{K}}$

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

- Stannates, $\text{Sm}_2\text{Sn}_2\text{O}_7 + \text{SnO}_2$ $T = \frac{8.87 \cdot 10^4}{204 - \delta_{CS}}$ approx: $T \cong \square \cdot 1 \frac{\text{ppm}}{\text{K}} + \delta T$

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

1993.

- *KBr*

- Chemical shift $T = \square \cdot 0.024 \frac{\text{ppm}}{\text{K}}$

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

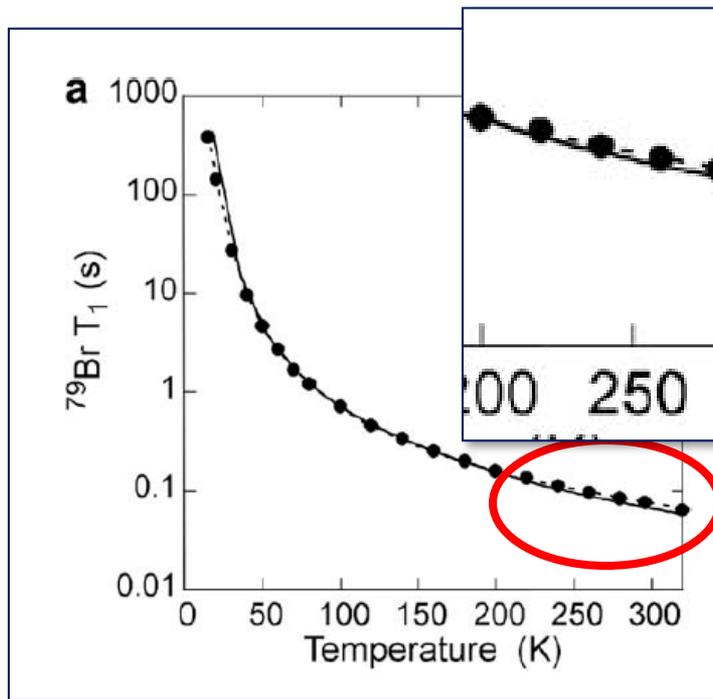
Thurber, K.R., et al., J. Magn. Reson. 196, 84–87, 2009.

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

Sample Temperature in a MAS probe



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



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

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

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

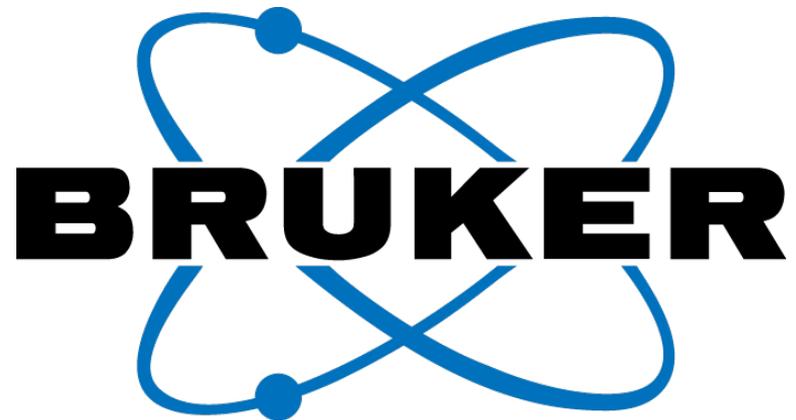
Thank you!



谢谢

结束





Innovation with Integrity