AVANCE III HD

DigiLock and SmartVT

Stable sample temperature and B_0 field are crucial prerequisites for high quality NMR results. In automation, multi peak solvents require powerful lock capabilities. AVANCE III HD consoles offer state-of-the-art signal acquisition and digital signal processing for digital H_o field control and ultimate temperature stability.

B_0 Field Stability

With Bruker's 2nd generation (2G) digital lock incorporating latest electronics and advanced signal processing it is now possible to obtain unmatched results, even under challenging conditions (external field disturbances, low content of deuterated solvent, high B_0 drifts, etc.) as shown in figure 1.

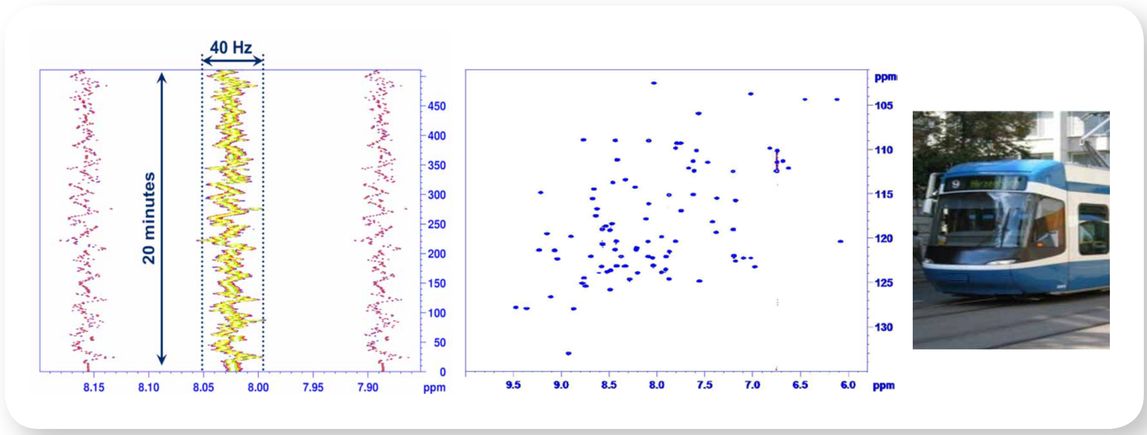

Fig. 1 Lineshape spectra as pseudo-2D (left), unlocked, showing field disturbances of +/- 20 Hz from nearby tramways. ^{1}H-^{15}N HSQC (right) of ^{15}N-ubiquitin locked on just 0.3% D_2O recorded under those conditions is virtually free of artifacts. Sample courtesy of Prof. L. Kay, University of Toronto.

Multi-Peak Solvent Locking

Bruker 2nd generation (2G) digital lock handles multi-peak solvents with ease allowing for automatic locking on one of the solvent peaks. Figure 2 shows 10 spectra of a sample in pyridine acquired in automation. The reliable lock operation yields spectra with perfect chemical shift match and no need for (external) calibration.

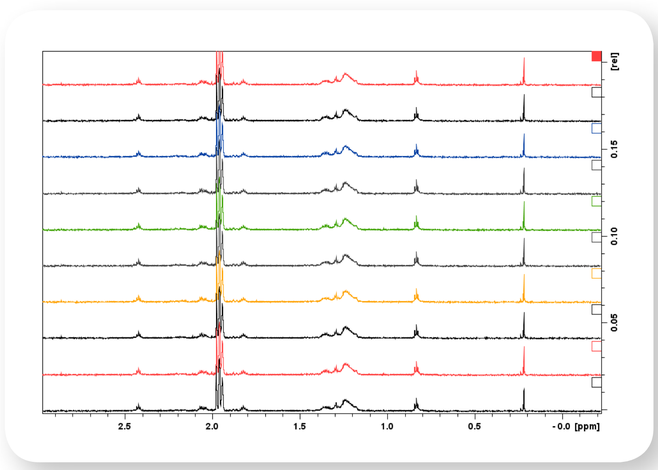

Fig. 2 Reliable & reproducible locking with perfect spectra match on pyridine sample: 10 spectra acquired after locking in automation. Spectra overlay without referencing shows perfect match for all 10 datasets.

VT Control: Precision and Stability

Bruker Smart Variable Temperature Control (BSVT) delivers optimal sample temperature control and stability. The mass-flow regulated VT-gas stream allows regulating and observing sample temperature changes as small as 0.01°C! Figure 3 shows a contour plot (left) of the DSS signal recorded in a pseudo-2D over 1 hour with 2 increments / minute. After 10 minutes, the temperature was increased by 0.01°C for a duration of 13 minutes before setting it back to the original value. In the center increments 10-20 & 30-40 are shown illustrating the difference in chemical shift of 0.09 Hz resulting by the 0.01°C temperature change at 850 MHz.

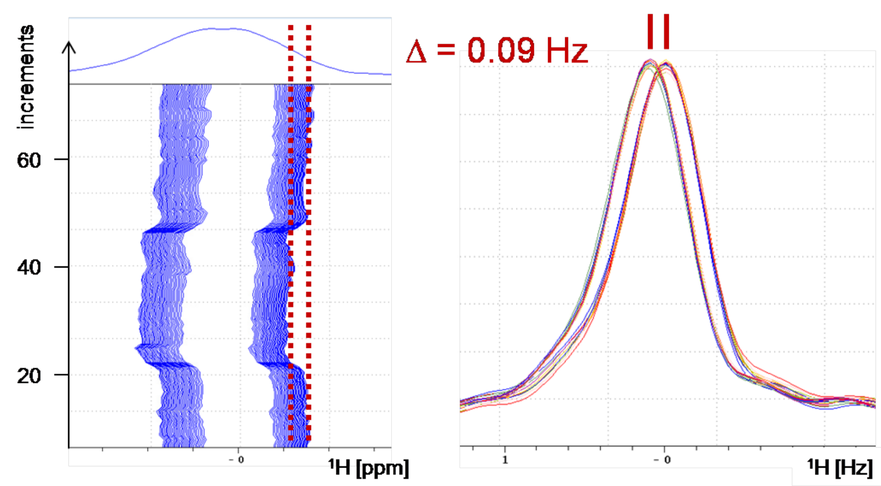

Fig. 3 DSS signal in H_2O:D_2O 90:10 at 850 MHz. 2 increments / minute. +0.01°C temperature step at increment 20, -0.01°C step at increment 47.

NMR Thermometer

By combining both, 2G DigiLock and SmartVT, the unique Bruker NMR Thermometer controls the sample temperature via the temperature dependent ^{2}H chemical shift of the NMR signals inside the sample.

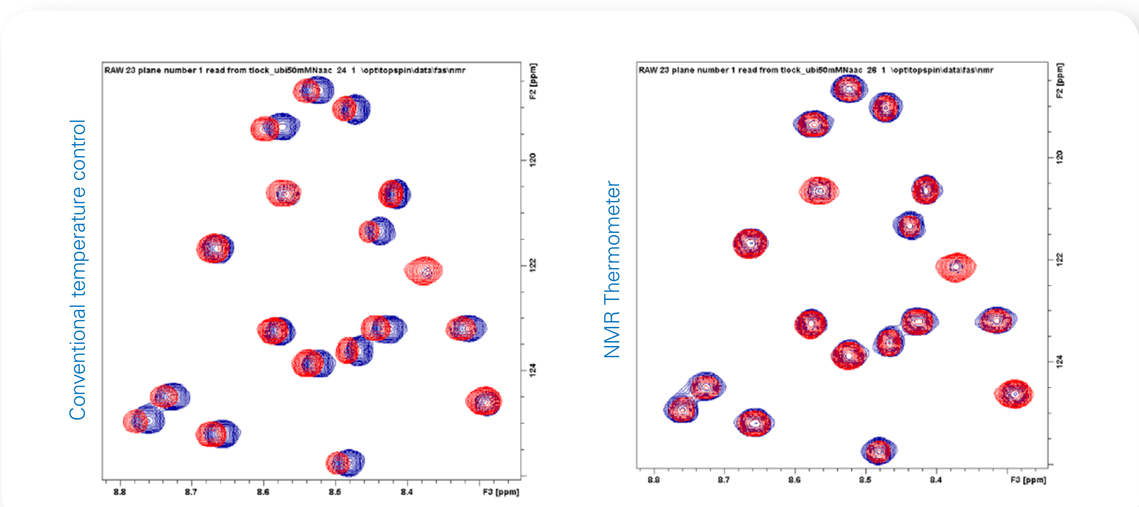

Fig. 4 Overlay of NOESY-HSQC (blue) and TOCSY-HSQC (red) experiments. Conventional temperature control results in peak shifts (left) induced by RF heating, NMR Thermometer control (right) yields perfect chemical shift match between the spectra.

Under experimental conditions, where the solvent signal is symmetrically split into two signals (e.g. liquid crystals) the 2G DigiLock is able to lock on a "virtual" signal defined by the center of two real signals (red arrow). This allows maintaining the chemical shift even when changing the quadrupolar splitting distance by changing the temperature as shown in figure 5.

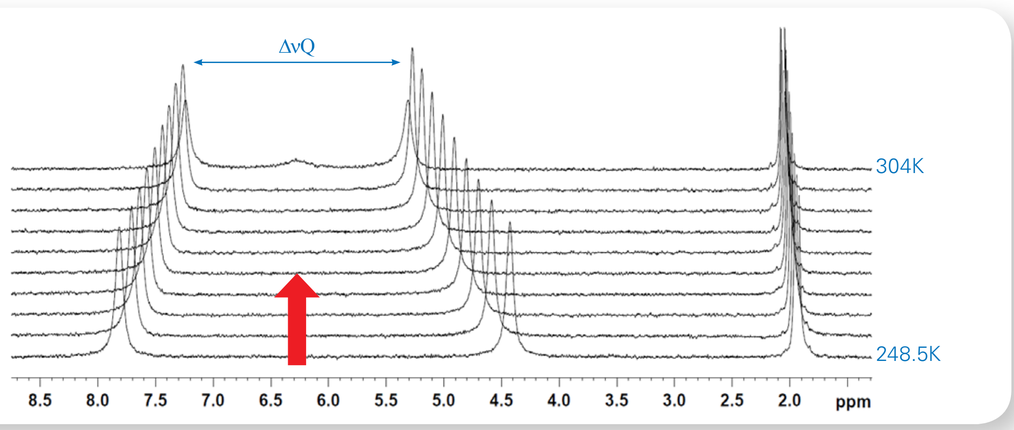

Fig. 5 ^{2}H spectra showing quadrupolar splitting of $CDCl_3$ as a function of temperature in a liquid crystalline PBLG phase, with additional acetone-d_6 in a capillary. Sample locked on the center frequency of the split $CDCl_3$ resonances. Courtesy: Christina Thiele, Volker Schmidts, TU Darmstadt, Germany; Roberto R Gil: Carnegie-Mellon University, United States.

Summary

- Ultimate B0 field and sample temperature stability with modern digital signal processing, RF electronics and VT control.
- Easy and reliable locking on multi peak solvents.
- Combined operation of Lock and VT control.