Supporting Information

On the Nature of the B4 Banana Phase: Crystal or not a Crystal?

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Details on the derivation of $\Delta\omega_{dipolar}$

To estimate the magnitude of the homonuclear dipolar coupling, the second-moment of the ^{1}H NMR signal of B4 at room temperature was first determined under non-spinning (static) sample conditions (**Figure 1**). For homogeneously broadened lines at intermediate magic-angle-spinning frequencies, the spinning-sideband appearance vs. spinning-frequency is nearly impossible to calculate explicitly. However, by performing variable spinning-speed MAS ^{1}H NMR experiments at room temperature (**Figure 1**), the spinning sideband pattern of the homogeneously broadened line provides a semi-quantitative determination of the appearance of $\Delta\upsilon$(dipolar) vs υ(MAS).[1] By performing variable temperature MAS ^{1}H NMR of the sample at constant spinning speed (**Figure 2)**, the spinning-sideband pattern (including relative linewidths) was matched to the room-temperature variable-spinning experiments, thereby yielding an estimate of $\Delta\upsilon$(dipolar)/υ(MAS). For example, at 153 °C the line shape in **Figure 2** resembles the one that is obtained at 18.5 kHz in **Figure 1**. Therefore, the $\Delta\omega_{dipolar}/\omega_{rotor}$ ratio is 2.2. The spinning rate at the variable temperatures was ~10 kHz.

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[1] See *Solid-State NMR Spectroscopy, Principles and Applications*, ed. Melinda J. Duer, Blackwell Science Ltd., **2002** 84-85.

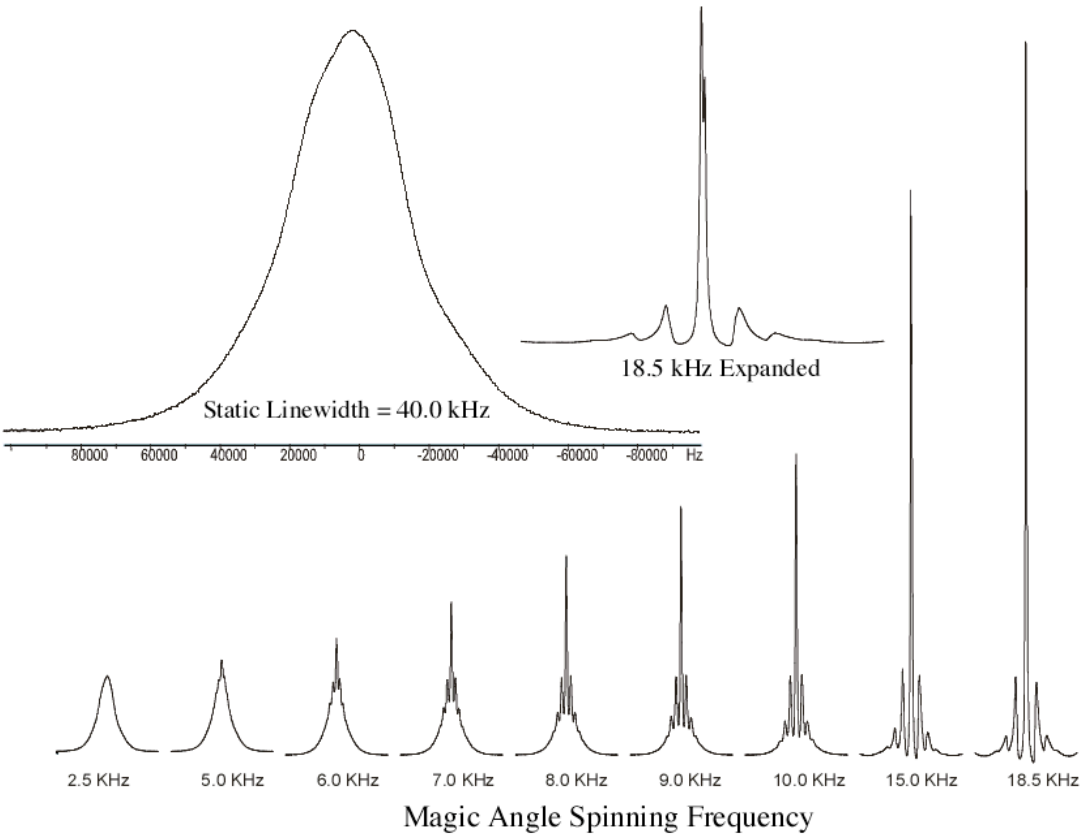

Figure 1. Variable-frequency, ^{1}H MAS NMR of B4 at 20°C.

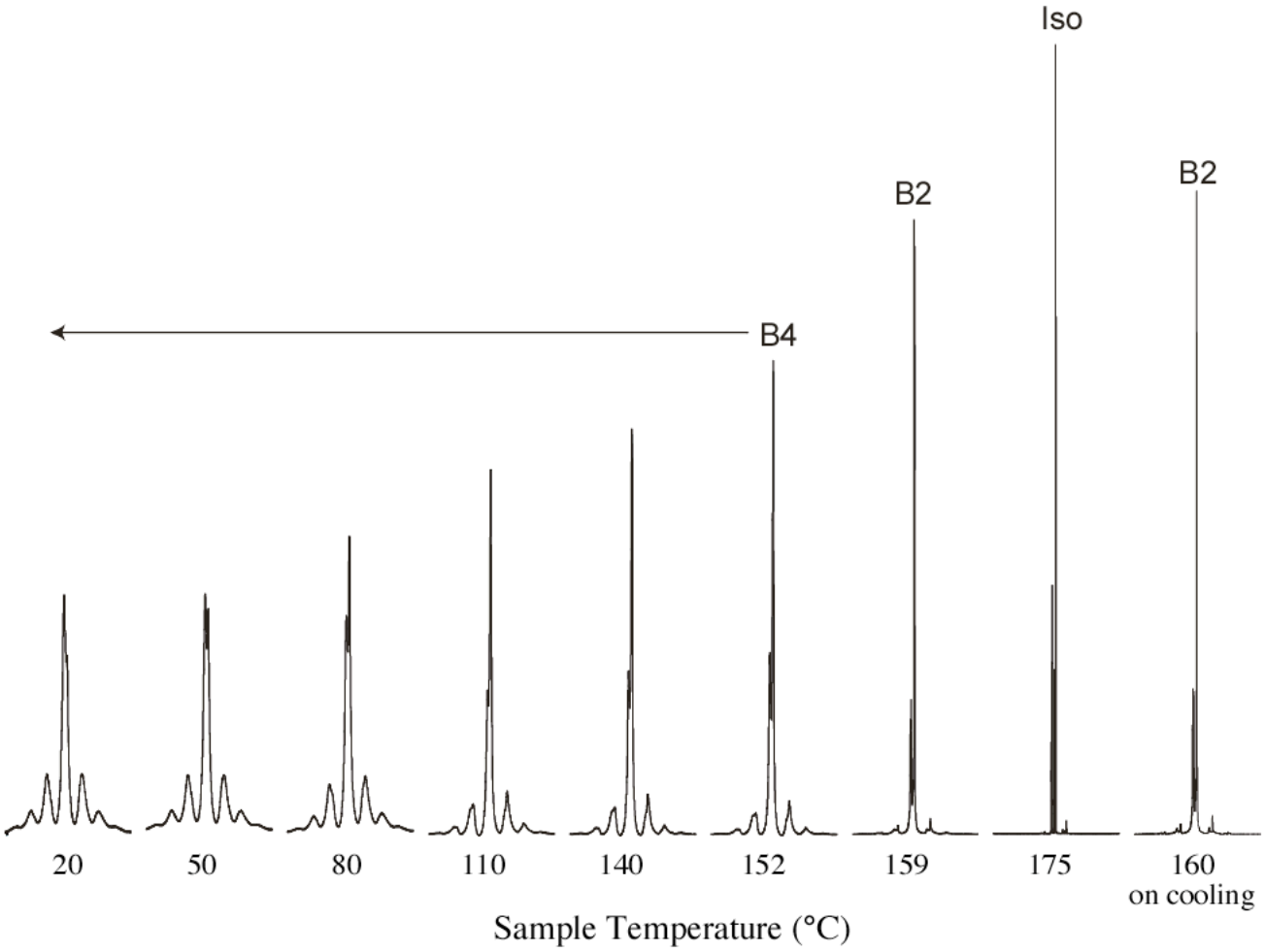

Figure 2. Broad range VT ^{1}H MAS single pulse spectra showing the spinning side bands. Spinning rate ~ 10 kHz.