VIII-I The Effects of the 2D Spin-Echo NMR Experiment on a Solid-State Homonuclear Sipn-1/2 Pair

The dipolar interaction for a solid-state homonuclear sipn-1/2 pair is averaged out by magic-angle sample spinning (MAS). The 2D spin-echo NMR experiment can reintroduce the influence of the homonuclear dipolar interaction into MAS powder signals.

VIII-I-1 Real Figure of Two-Dimensional Spin-Echo NMR Spectra for a Homonuclear Two-Spin System in Rotating Solids

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The 2D spin-echo NMR experiments were recently carried out on polycrystalline [2, 3-$^{13}C_2$] L-alanine under magic-angle sample spinning (MAS) conditions, so that two unusual resonance lines emerged along the F_1 axis (*Chem. Phys. Lett.* **305**, 35 (1999)). To examine a spectral structure observed in the F_1 direction more closely, we executed the 2D NMR experiment using a sufficiently small t_1 increment. As a result, we found many more resonance lines on a spectrum sliced off along the F_1 axis. The line distribution had a very unique and interesting structure. To elucidate the line positions theoretically, we calculated analytically the signals for the 2D spin-echo experiment performed with any t_1 increment on a homonuclear spin-1/2 pair undergoing MAS. We discovered that virtually six resonance lines (exactly twelve resonance lines) occurred on a spectrum sliced off along the F_1 axis. In addition, it was proved that the intensities of some resonance lines were largely dependent on the dipolar interaction.

VIII-J The Applications of Double-Rotation NMR Method

Double-Rotation NMR method (DOR) was applied to rare spins surrounded by abundant homonuclear spins. The application of the method to solid-state quadrupolar nuclei having $I = 1$ was investigated.

VIII-J-1 The Observation of REDOR Phenomenon for CH_x ($x \geq 2$) Spin Systems under DOR

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Double-Rotation NMR method (DOR) was developed originally to remove second-order line broadenings for solid-state quadrupolar nuclei having half-integer spins. We applied the method to rare spins surrounded by abundant homonuclear spins (*i.e.* protons). DOR averaged out the homonuclear dipolar interactions, so that a CH_x system could be taken as an ensemble of independent CH systems. We could, therefore, detect heteronuclear dipolar interactions for CH_x ($x \geq 2$) spin systems without homonuclear decoupling techniques. In addition, we showed that DOR is also applicable to a solid-state ^{13}C-^{14}N spin pair in order to recover the heteronuclear dipolar interaction that was removed by sample spinning.