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Single Crystal Solid-State NMR of Magnetically Oriented Powder

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Solid-state NMR spectroscopy is one of the most widely used methods for investigating crystal structures, along with the X-ray and neutron diffraction methods. Solid-state NMR can provide structural information including isotropic chemical shift, dipolar and quadrupolar couplings, spin diffusion, and chemical shift tensor. Among these, the chemical shift tensor is of particular significance because the electronic environment around a nucleus is directly reflected on the chemical shift tensor. However, full information of the chemical shift tensor, including principal values and axes, is difficult to obtain experimentally because a large single crystal is required for the measurement.

On the other hand, we have proposed the use of a magnetically oriented microcrystal array (MOMA) as an alternative to a single crystal.[1,2] A MOMA is a composite in which microcrystals are aligned three-dimensionally, prepared by using a time-dependent magnetic field. We recently demonstrated that the ^{13}C chemical shift tensors of L-alanine crystal can be completely determined by application of the standard procedure in the single-crystal rotation method to a MOMA of L-alanine microcrystals,[3] as shown in Figure 1. The L-alanine MOMA produces sharp resonance peaks without resolution enhancement by magic angle spinning (MAS). In addition, we observed that the positions of the ^{13}C resonance peaks vary systematically as a function of the angle ψ that is the sample-rotation angle about the axis inclined by the magic angle with respect to the NMR magnetic field. From the ψ-dependence of the chemical shifts, ^{13}C chemical shift tensor was completely determined. We confirmed that the combination of MOMA with the single-crystal rotation method can be applied to other nuclei such as ^{31}P and ^{15}N. These results clearly show that the MOMA method is a powerful tool for obtaining full information of the chemical shift tensor from a microcrystalline powder without MAS.

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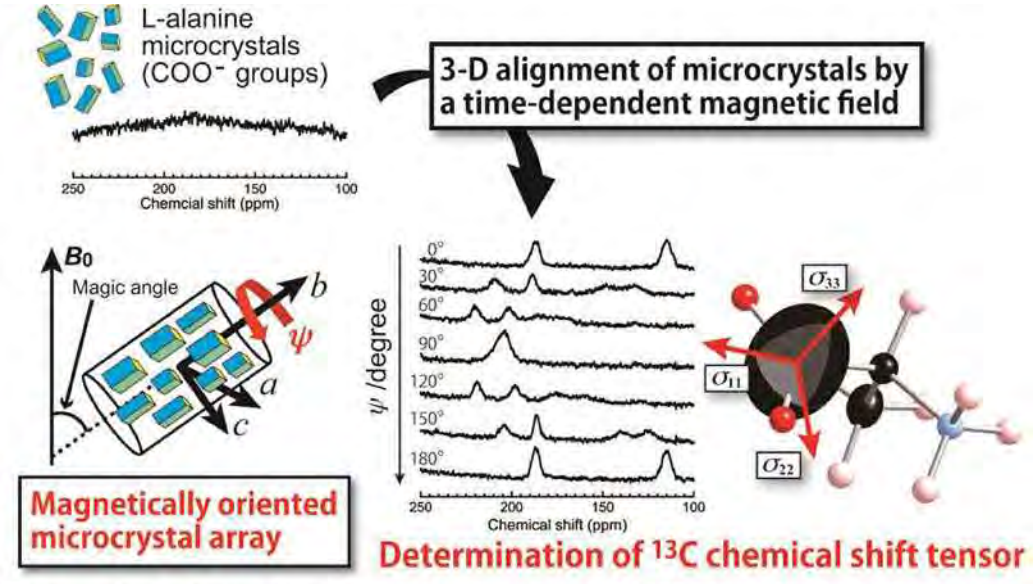

Figure 1 Schematic for determination of ^{13}C chemical shift tensor by using a magnetically oriented microcrystal array (without MAS).

Keywords: solid-state NMR, chemical shift tensor, magnetically oriented microcrystal array