
Characterization of Silanol Groups in Protonated Magadiite by ^1H and ^2H Solid-State Nuclear Magnetic Resonance

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Abstract: Silanol groups in protonated magadiite (H-magadiite) were characterized by ^1H and ^2H solid-state nuclear magnetic resonance (NMR). H-magadiite and deuterated (D) magadiite were synthesized by the treatment of Na-rich magadiite with 0.2 N HCl and 0.2 N DCl, respectively. In the ^1H NMR spectrum measured at room temperature, silanol groups of H-magadiite showed two signals at 3.75 and 5.70 ppm, indicating that two types of silanol groups were present. The ratio of silanol groups associated with strong hydrogen bonding (5.70 ppm) to those with weaker hydrogen bonding (3.75 ppm) was 2 to 1. The ^2H NMR spectra of deuterated magadiite were measured in the temperature range from 150 to 440 K. In the spectra measured at temperatures below 294 K, silanol groups showed Pake doublet patterns. These patterns were composed of two components corresponding to the two types of silanol groups shown in the ^1H NMR analysis. Both silanol groups produced wobbling motions with increasing temperature. Above 294 K, the profile of the Pake doublet pattern was transformed gradually to a near triangular pattern, indicating that the silanol groups underwent other motions also, such as a two-site jump.

Key Words: ^1H NMR • ^2H NMR • Hydrogen Bonding • Magadiite • Silanol Group

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