

NMR analysis in solid state

The application of solid-state NMR techniques usually arises due to specific interest in the physics of solid state, including packing effects and polymorphic structures. Solid-state NMR can be also used to study chemical shielding anisotropy. Sometimes, the application is motivated by an inability to dissolve the material of interest. 1D CP MAS spectra of ^{13}C , ^{15}N , ^{27}Al , ^{29}Si , ^{31}P , ^{11}B and some other elements can be measured.

Examples of solid-state NMR spectra

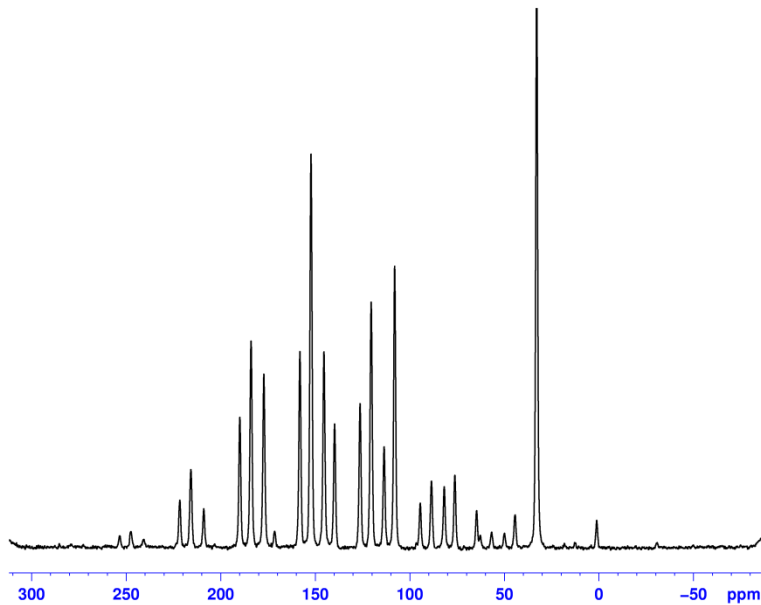


Fig. 1. ^{13}C spectrum of 3-methylxanthine with main peaks and sidebands, spinning rate 4 kHz. Spectrum can be used for the CSA evaluation. The principal values of the chemical shielding tensor can be obtained from this type of spectra.

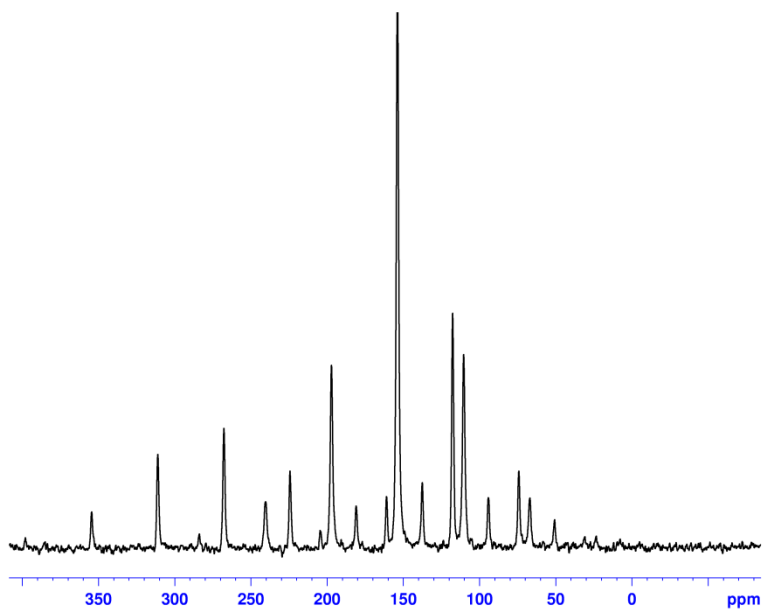


Fig. 2. ^{15}N spectrum of 3-methylxanthine with main peaks and sidebands, spinning rate 2.2 kHz. Spectrum can be used for the CSA evaluation. The principal values of the chemical shielding tensor can be obtained from this type of spectra.

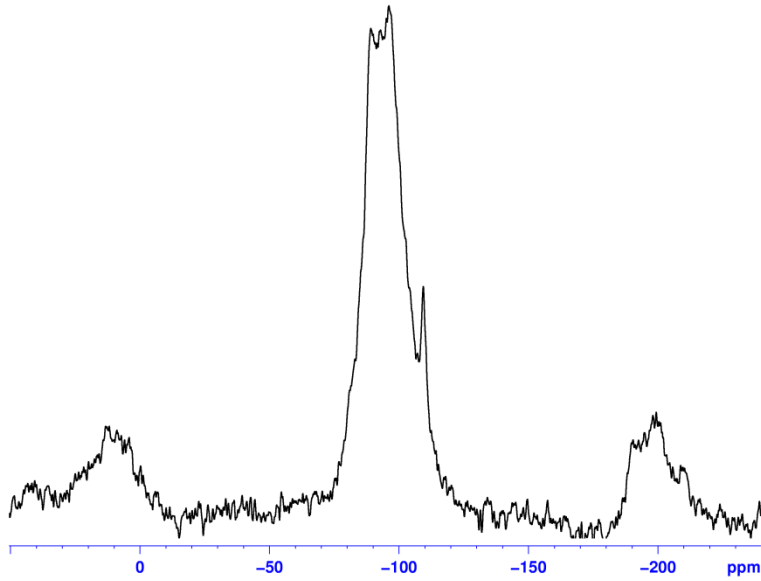


Fig. 3. ^{29}Si spectrum of geopolymer, spinning rate 10 kHz. Spectrum can be used to determine Si connectivity. ^{29}Si MAS NMR spectroscopy can differentiate between various geopolymeric building units such as $\text{SiQ}_4(4\text{Al})$, $\text{SiQ}_4(3\text{Al})$, $\text{SiQ}_4(2\text{Al})$ and $\text{SiQ}_4(1\text{Al})$ [1,2].

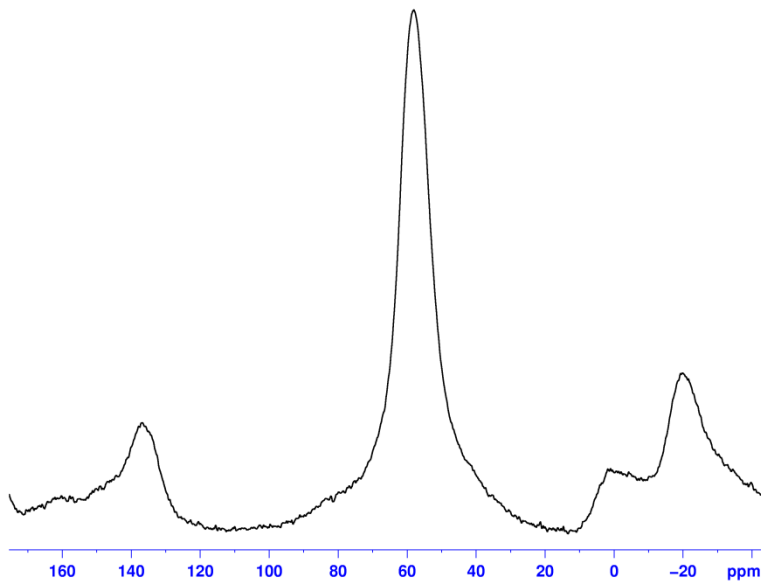


Fig. 4. ^{27}Al spectrum of geopolymer, spinning rate 10 kHz. ^{27}Al MAS NMR spectroscopy is able to recognize coordination number of Al in silico-aluminates. Four coordinated aluminium resonates at approximately 50 ppm while six coordinate aluminium resonates at about 0 ppm.

[1] G. Taveri, J. Toušek, E. Bernardo, N. Toniolo, A. R. Boccaccini, I. Dlouhý, *Mater. Lett.*, 2017, **200**, 105-108.

[2] G. Taveri, S. Grasso, F. Gucci, J. Toušek, I. Dlouhý, *Adv. Funct. Mater.*, 2018, 1805794.