Solid-state NMR characterization of quadrupolar nuclei in metallocenes, phthalocyanines and mesoporous solids

Robert W. Schurko, Hiyam Hamaed, Aaron Rossini, and Luke O'Dell

Department of Chemistry and Biochemistry, University of Windsor, Windsor, Ontario

rschurko@uwindsor.ca

Ultrahigh-field NMR was integral in five published projects, including assignments of ^{15}N resonances in layered silver-containing solids, characterization of pharmaceutical polymorphs via ^{35}Cl NMR, development of ^{209}Bi SSNMR as a structural characterization method, obtaining ^{71}Ga NMR spectra for work on microcoils, characterization of metallocenes with ^{35}Cl NMR [1-5]. We also received assistance in the form of CASTEP calculations for several projects. We are continuing work on pharmaceuticals (^{35}Cl, ^{23}Na, ^{14}N), nanoparticle samples (multinuclear), metallocenes (^{91}Zr and ^{35}Cl).

We have amassed a large amount of ^{35}Cl, $^{47/49}Ti$ and ^{91}Zr NMR data for a series of titanocenes, zirconocenes and hafnocenes. These metallocenes are important in homogeneous and heterogeneous catalysis for polyethylene production; however, little is known about the precise mechanism of these catalytic processes. Probing the metal centers of these metallocenes may provide rich insight into initiation, polymerization and termination processes. The chemical shift and quadrupolar parameters extracted from ^{35}Cl, $^{47/49}Ti$ and ^{91}Zr NMR spectra are very sensitive to slight structural modifications, ligand substitution and variation in substituents on the cyclopentadienyl rings. Highfield NMR spectra of these insensitive nuclei can be acquired quite rapidly, suggesting that the 900 MHz spectrometer will be instrumental in conducting studies on metallocenes loaded onto micro- and mesoporous support materials.

While the ultrahigh magnetic field does not narrow the spin-1 powder patterns, it certainly will be useful for boosting S/N in ^{14}N solid-state NMR spectra and allow increasingly efficient spectral acquisitions. Our new WURST-QCPMG techniques have been implemented successfully on the 900 MHz NMR spectrometer. We plan to run a series of pharmaceutical polymorphs at the 900 MHz spectrometer over the coming year.

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