Oral presentation

Building Bridges between X-Ray Diffraction and NMR Spectroscopy: Boron Electric Field Gradients in Chlorinated Carboranes

N. Graw[1], K. Rachuy[1], F. Rüttger[1], J. Probst, R. Herbst-Irmer[1], A. Krawczuk[1], D. Stalke[1], M. John[1]

[1]Georg-August-Universität Göttingen, Tammannstraße 4, 37077 Göttingen, Germany

ngraw@chemie.uni-goettingen.de

The MetalJet X-ray source provides In K_α radiation for in-house diffraction experiments.[1] It has been demonstrated that diffraction setups utilizing In K_α radiation are suitable to collect high quality high-resolution data for experimental electron density investigations.
[2] We have now used this setup to collect data on two isomers of dichloro *closo*-dicarbadodecaboranes. The data were modelled according to the Hansen-Coppens multipole formalism[3] and a topological analysis based on Bader's *Quantum Theory of Atoms in Molecules* (QTAIM)[4] was performed to obtain insights into the bonding situation of these compounds. Furthermore, the electron density distribution was used to derive the electric field gradient (EFG) tensor.

NMR spectroscopic experiments in anisotropic media allow for the measurement and analysis of residual quadrupolar couplings (RQCs) of quadrupolar nuclei. As they derive from the interaction of the nuclear quadrupole moment with the EFG tensor, which is highly dependent on the direct coordination sphere of a nucleus, RQCs can be used for structure determination and resonance assignment as has been shown for several species containing ^{7}Li and ^{11}B nuclei.[5] This method has been applied to the two chlorinated carboranes and experimental RQCs could be obtained. Values for the EFG tensor derived from the experimentally determined electron density distribution as well as values determined by DFT calculations on either isolated molecules or with periodic boundary conditions were used and outcomes for the interpretation of the NMR experiments were compared.

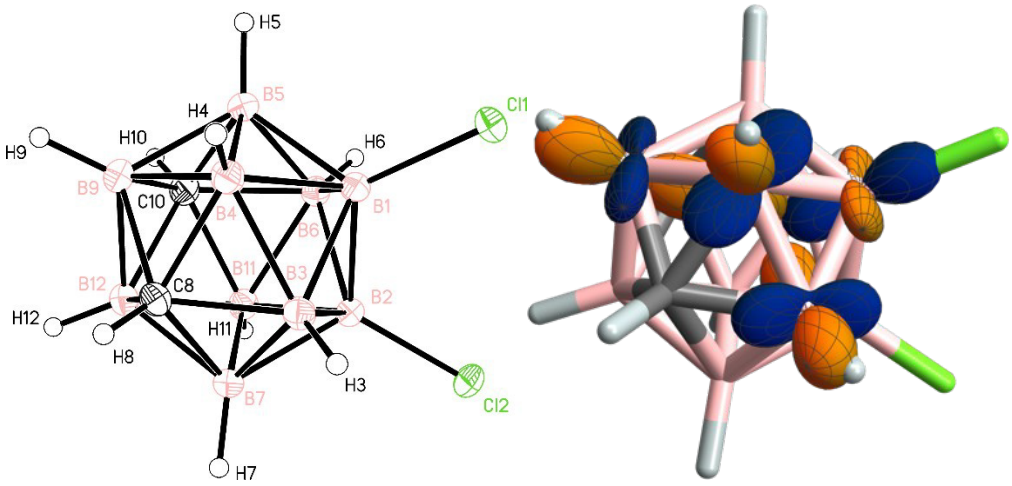

Figure 1. Left: Excerpt of the crystal structure of *m*-$B_{10}C_2H_{10}Cl_2$ at 110 K. ADPs are depicted at the 50% probability level. Right: Graphical representation of EFG tensors at the different boron positions derived from gas phase DFT calculations for *m*-$B_{10}C_2H_{10}Cl_2$. The orange and blue lobes represent the negative and positive values of the tensors, respectively. The tensor at the chlorinated boron atom was scaled up by a factor of five to roughly match the size of the EFG tensors at the protonated boron atoms.

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