

P.14.02.1*Acta Cryst.* (2005). A61, C421**Advances in Charge Density and Electrostatic Interaction Analyses**Christian Jelsch, Angélique Lagoutte, Benoit Guillot, Virginie Pichon-Pesme, Claude Lecomte, LCM3B CNRS UHP Faculté des Sciences. *Vandoeuvre les Nancy*. E-mail: christian.jelsch@lcm3b.uhp-nancy.fr

The number of high resolution biological macromolecules X-ray structures increases regularly. Feasibility of multipolar refinement of protein structures has been proved [1, 2], assuming subatomic resolution and sufficiently low thermal motion. The high resolution crystallographic refinement program MoPro employs a multipolar representation of the electron density, the latest functionalities will be described [3,4].

Electrostatic properties are of major importance in numerous biological and molecular recognition. Three methods are employed in the VMOPro software to calculate the accurate electrostatic potential and the interaction energy:

- * numerical grid integration
- * Buckingham summation of charges & multipoles interaction
- * coulombic energy using real and virtual spherical atoms.

The electron density parameters are obtained either from a crystallographic refinement or from a database transfer [5]. The applications go from small compounds crystals to protein-ligand complexes.

[1] Muzet N., Guillot B., Jelsch C., Howard E., Lecomte C., *PNAS*, 2003, **100**, 8742. [2] Jelsch C., Teeter M.M., Lamzin V., Pichon-Pesme V., Blessing R.H., Lecomte C., *PNAS*, 2000, **97**, 3171-3176. [3] Jelsch C., Guillot B., Lagoutte A., Lecomte C., *J. Appl. Cryst.* A, 2005, **38**, 38-54. [4] Guillot B., Viry L., Guillot R., Lecomte C., Jelsch C., *J. Appl. Cryst.*, 2001, **34**, 214. [5] Pichon-Pesme V., Jelsch C., Guillot B., Lecomte C., *Acta Cryst.*, 2004, **A60**, 204-208.

Keywords: charge density, protein, database**P.14.03.1***Acta Cryst.* (2005). A61, C421**Bond-dependent Crystal Response on Electric Field: Synchrotron Diffraction Study**Ulrich Pietsch^a, Semen Gorfman^{a,b}, Vladimir Tsirelson^{a,b}, ^a*Institute of Physics, Potsdam University, Potsdam, Germany*. ^b*Quantum Chemistry Department, Mendeleev University of Chemical Technology, Moscow, Russia*. E-mail: upietsch@gad.iphysik.uni-potsdam.de

The interaction of a crystal with the permanent external electric field resulting in dielectric polarization and converse piezoelectric effect is well described on the macroscopic level. In last years the microscopic origin of these phenomena is actively studied by means of synchrotron diffraction technique. In this work, we present our recent investigations of the structural response of single crystals to an external electric field, and analyse the results in terms of the chemical bond in different structural units of the crystal.

Special attention will be paid to the study of bond specific structural response in α -GaPO₄. The experiment was carried out by modulation-demodulation technique at D3 beamline at HASYLAB. The measured diffraction intensities of 42 independent reflections were fitted to a structural model, validated on the basics of the developed theory of X-ray diffraction by a crystal in the external electric field [1]. The difference of field-induced deformations of GaO₄ and PO₄ tetrahedra is explained in terms of the different character of chemical bonds and by charge density features. Support by DFG and A.v.Humboldt-Foundation (V.T.) is kindly acknowledged.

[1] Gorfman S., Tsirelson V., Pietsch U., *Acta Cryst.*, 2005, **A**, submitted.

Keywords: X-ray diffraction, dielectric properties, chemical bonding**P.14.03.2***Acta Cryst.* (2005). A61, C421**Electron Density Analysis of Borocarbides**Tilmann Leisegang^a, Dirk C. Meyer^a, Dimitri Souptel^b, Günther

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Quaternary rare earth nickel borocarbides, space group (139) *I4/mmm*, exhibit different low temperature properties [1,2]. Only superconductivity at temperatures of about 8 K is observed for $R = Y, Lu$. For $R = Ho$ additional magnetic ordering occurs in the range of 4 to 6 K whereas superconductivity is destroyed. Further cooling leads to superconductivity again which is the so called re-entrant behaviour. This depends strongly on the crystal composition within the small homogeneity range and the thermal treatment after crystal growth.

Single crystals of compositions mentioned above were investigated by means of single crystal diffraction methods using both X-ray tubes and high energy synchrotron radiation sources under ambient conditions. Structure refinement and Fourier Difference Analysis of the experimental data were performed to determine small influences of chemical and structural disorder.

Experimentally determined electron densities are compared with theoretical calculations.

[1] Nagarajan R., Mazumdar C., Hossain Z., Dhar S.K., Gopalakrishnan K.V., Gupta L.C., Godard C., Padalia B.D., Vijayaraghavan R., *Phys. Rev. Lett.*, 1994, **72**, 274. [2] Siegrist T., Cava R.J., Krajewski J.J., Peck W.F., *J. Alloys Compd.*, 1994, **216**, 135.

Keywords: rare-earth, superconductivity, electron density**P.14.03.3***Acta Cryst.* (2005). A61, C421**Chemical Bonding and Electronic Configuration of Nitrosyl Iron Complexes**I-Jui Hsu^a, Tze-Yuan Wang^a, Jey-Jau Lee^a, Chung-Hung Hsieh^b, Gene-Hsiang Lee^a, Wen-Feng Liaw^b, Yu Wang^a, ^a*National Taiwan University, Taipei, Taiwan*. ^b*National Tsing Hua University, Hsinchu, Taiwan*. E-mail: ijuihsu@yahoo.com

The nitrosyl non-heme iron complexes with sulfur ligands are extensively synthesized to study the interaction of NO and iron-sulfur protein. In this report, the electron density distribution of mono- and di-nitrosyl iron sulfur compounds are studied with multipole model based on X-ray diffraction data. In the dinitrosyl iron complexes (DNIC), the coordination sphere of Fe is a tetrahedral $Fe(NO)_2(S)_2$ core such as $[S_3Fe(NO)_2]^-$ and $[Fe(NO)_2(SR)_2]^{1-0}$. In five-coordinated complexes, it turns out to be a tetragonal pyramidal $Fe(NO)(S)_4$ geometry for mononitrosyl complexes such as $[(NO)Fe(S, S-C_6H_4)_2]^{1-2}$ and $Fe_3(NO)_3(S, S-C_6H_4)_3$. To illustrate the bonding character, the topological properties associated with the bond critical points (BCP), Laplacian of the electron density as well as electron density at the BCP of each chemical bond will be presented. The comparison between experiment and theory will be made. Because of the ambiguity of the traditional way to discriminate the oxidation states of NO , NO^+ and NO^- group by bond angles and vibrational frequency, the X-ray absorption spectroscopy of Fe K-, L-edge and N/O/S K-edge are used to investigate the oxidation state of metal, NO groups and sulfur ligands. Moreover, the magnetic susceptibility and electron paramagnetic resonance results will be included to illustrate the electronic configuration among Fe and NO groups. Based on the Enemark-Feltham notation, all results toward the conclusion that the best way to describe the present dinitrosyl and mononitrosyl cases are $\{Fe^{+1}(\bullet NO)_2\}^9$ and $\{Fe^{+1}(NO^+)_2\}^7$.

Keywords: nitrosyl, BCP, X-ray absorption spectroscopy**P.14.03.4***Acta Cryst.* (2005). A61, C421-C422**Pump and Probe the Structure and Electronic Configuration of LIESST State**Yu Wang^a, C.F. Sheu^a, I-J. Hsu^a, Y-C. Lin^a, B.N. Wang^a, K. Toriumi^b, Y. Ozawa^b, ^a*Department of Chemistry, National Taiwan University,*