Analysis Wizard offers three methods for initial structure determination: a) Direct method, b) Direct space method and c) Charge flipping method.

[1] *The Rigaku Journal* (English version), **2010** *26(1),* 23-27. [2] *The Rigaku Journal* (English version) **2010**, *26(2),* 10-14.

Keywords: Ab-initio crystal structure analysis, charge flipping method, rietveld method

MS96.P01

Acta Cryst. (2011) A**67,** C800

Recent Progress in joint charge and spin densities refinement

Maxime Deutsch,[a] Nicolas Claiser,[a] Yurii Chumakov,[a] Mohamed Souhassou,[a] Claude Lecomte,[a] Sébastien Pillet,[a] Béatrice Gillon,[b] Jean-Michel Gillet [c] *[a]Laboratoire CRM², (UMR UHP-CNRS 7036), Institut Jean Barriol, Université Nancy I, Vandoeuvre-lès-Nancy (France). [b]Laboratoire Léon Brillouin (CEA-CNRS), Centre d'Etudes de Saclay (France). [c]Laboratoire SPMS, Ecole centrale de Paris, grande voie des vignes, Chatenay malabry (France).* E-mail: Maxime.deutsch@crm2.uhp-nancy.fr

Electron density, in all its representations, plays a key role in the understanding of the nature of interatomic interactions and chemical bonds. In case of molecular magnetic materials, as the experimental electron densities rely on high resolution X-ray diffraction experiments, it is necessary to go further and enrich the electronic model by the use of complementary techniques such as unpolarized and polarized neutron (PND) diffraction or Compton scattering. This implies to be able to include all these different experimental contributions into a unique electronic model, and to carry out a joint refinement. In this respect we are developing a new program to model the charge, spin and momentum densities (ANR CEDA project [*]).

We will report on the first charge and spin densities joint refinement applied to the molecular magnetic compound MnCuPba, (Fig.1 [1],[2]). The results are consistent with the previous separated studies of spin and charge densities. This method can bring new insights in the nature of interactions in the solid.

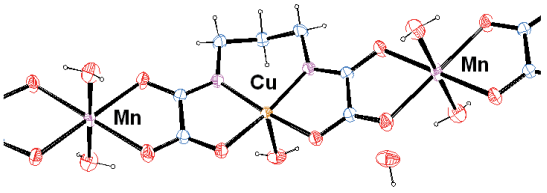

Fig.1. ORTEP view of the MnCuPba complex chain structure at 100K

[1] S. Pillet, M. Souhassou, C. Mathonière, C. Lecomte, *J. Am. Chem Soc*. **2004** *126,* 1219–1228. [2] V. Baron, B. Gillon, A. Cousson, C. Mathoniere, O. Kahn, A. Grand, L. Ohrstrom, B. Delley, M. Bonnet, J.-X. Boucherle, *J. Am. Chem. Soc.* **1997**, *119*, 3500-3506.

Keywords: charge, spin, densities

MS96.P02

Acta Cryst. (2011) A**67,** C800

Structure and magnetic order in CrOCl at low temperatures

Andreas Schönleber,[a] Joachim Angelkort,[a] Jian Zhang,[a] Sander van Smaalen,[a] Reinhard K. Kremer,[b] Anatoliy Senyshyn,[c] *[a]Laboratory of Crystallography, University of Bayreuth, Bayreuth (Germany). [b]MPI for Solid State Research, Stuttgart (Germany). [c]Forschungs-Neutronenquelle Heinz Maier-Leibnitz, FRM-II, Garching (Germany).* E-mail: andreas.schoenleber@uni-bayreuth.de

The metal(III) oxyhalide *MOX* structures with *M* = Ti, V, Cr and *X* = Cl, Br are isostructural with FeOCl at room temperature [1-7] (they are built by slabs consisting of M_2O_2 bilayers enclosed by layers of *X* atoms; the interaction between the slabs is of the van der Waals type; the symmetry is orthorhombic, space group *Pmmn*), but they show different magnetic order at low temperatures [8-15]. In CrOCl one phase transition was observed towards antiferromagnetic ordering [13].

We have performed low-temperature single-crystal X-ray diffraction experiments at the synchrotron Hasylab/DESY (Hamburg, Germany) to explore the nuclear structure and the development of nuclear superstructure peaks (*h* , *k*+½ , *l)* below T_N = 13.5 K. We found the phase transition at T_N to be associated upon cooling with a loss of symmetry from orthorhombic towards monoclinic [16]. We also have observed by magnetic susceptibility measurements a second phase transition at T_{c2} = 27.8 K.

Our low temperature powder neutron diffraction experiments at the neutron reactor FRM-II (Garching, Germany) indicate the low temperature phase below T_N as a monoclinic fourfold magnetic superstructure (**a** × 4**b** × **c**) with respect to the room temperature phase and the intermediate phase ($T_N \leq T \leq T_{c2}$) as an incommensurately modulated magnetic superstructure. Both magnetic phases are analyzed with Rietveld refinements against the powder neutron diffraction data. For the structure of the low temperature phase the extended magnetic superspace group symmetry $P\,2_1/m\,1'\,(0\sigma_2\sigma_3)\,0ss$ (monoclinic, **a**-unique) is applied, the extended magnetic superspace group symmetry of the intermediate phase is $P\,mmn\,1'\,(0\sigma_2 0)\,s0ss$ (orthorhombic). The additional symmetry operator (1', *s*) in the extended symbol stands for the time inversion operator combined with an additional shift of ½ of the modulation function to describe antiferromagnetic order [17].

[1] H. Schäfer, F. Wartenpfuhl, E. Weise, *Z. Anorg. Allg. Chemie* **1958**, *295*, 268-280. [2] H.G. von Schnering, M. Collin, M. Hassheider, *Z. Anorg. Allg. Chemie* **1972**, *387*, 137-141. [3] P. Ehrlich, H.-J. Seifert, *Z. Anorg. Allg. Chemie* **1959**, *301*, 282-287. [4] A. Haase, G. Brauer, *Acta Crystallogr.* **1975**, *B31*, 2521-2522. [5] H. Schäfer, F. Wartenpfuhl, *Z. Anorg. Allg. Chemie* **1961**, *308*, 282-289. [6] S. Goldsztaub, C.R. Hebd*, Seances Acad. Sci.* **1934**, *198*, 667-669. [7] M.D. Lind, *Acta Crystallogr.* **1970**, *B26,* 1058-1062. [8] A. Seidel, C.A. Marianetti, F.C. Chou, G. Ceder, P.A. Lee, *Phys. Rev. B* **2003**, *67*, 020405. [9] A. Schönleber, S. van Smaalen, L. Palatinus, *Phys. Rev. B* **2006**, *73*, 214410. [10] S. van Smaalen, L. Palatinus, A. Schönleber, *Phys. Rev. B* **2005**, *72*, 020105(R). [11] A. Wiedenmann, J.P. Vénien, P. Palvadeau, J. Rossat-Mignod, *J. Phys. C: Solid State Phys.* **1983**, *16*, 5339-5350. [12] A. Schönleber, J. Angelkort, S. van Smaalen, L. Palatinus, A. Senyshyn, W. Morgenroth, *Phys. Rev. B* **2009**, *80*, 064426. [13] A. Nørlund Christensen, T. Johansson, S. Quézel, *Acta Chem. Scand A* **1975,** *28*, 1171-1174. [14] R.W. Grant, *J. Appl. Phys.* **1971**, *42*, 1619-1620. [15] A. Adam, G. Buisson, *Phys. Stat. Sol. A* **1975**, *30*, 323-329. [16] J. Angelkort, A. Wölfel, A. Schönleber, S. van Smaalen, R.K. Kremer, *Phys. Rev. B* **2009**, *80*, 144416. [17] V. Petříček, J. Fuksa, M. Dušek, *Acta Crystallogr.* **2010**, A*66*, 649-655.

Keywords: phase transition, magnetic order, superstructure

MS96.P03

Acta Cryst. (2011) A**67,** C800-C801

Probing the local sense of the Dzyaloshinskii-Moriya vector: neutrons vs x-rays

Vladimir E. Dmitrienko,[a] Elena N. Ovchinnikova,[b] Jun Kokubun,[c] Kohtaro Ishida,[c] Vyacheslav A. Chizhikov,[a] Steve Collins,[d] *[a]Institute of Crystallography, Moscow, 119333 (Russia). [b]Faculty of Physics,*