

04.2-5 DEVIATION FROM THE STOICHIOMETRY AND LATTICE FLEXIBILITY. By M. Farkas-Jahnke, M. Somogyi, P. Tekula-Buxbaum, L. Petrás, Research Institute for Technical Physics of the Hungarian Academy of Sciences, Budapest, Hungary.

Several compounds containing crystal water showed rather interesting behaviour during heat treatment. In temperature regions below phase transformations caused by partial or total loss of "structural" crystal water an anisotropic lattice deformation occurred /T. Sato, T. Nakamura, *Thermochemica Acta* /1982/ 53, 281/. In the case of GaOOH this phenomenon was proved to be connected with the departure of water being in excess to the "structural" water bounded within the GaOOH lattice, but nevertheless this excess water was fixed somehow inside the lattice, because the temperature region of its leaving was between 100-270 °C even when using very low heating rates /M. Farkas-Jahnke, Eight European Crystallographic Meeting, Liège /1983/ Abstract 241/. The amount of water was very large, about 20 w% of the "structural" water.

Similar phenomenon, i.e. the presence of excess "fixed" NH<sub>3</sub> was observed in ammonium paramolybdate tetrahydrate samples prepared at different pH values. The "apparent" structure of the material in all cases was alike, i.e. the site and intensity distribution of the reflections in room temperature X-ray patterns made in focusing Guinier-cameras were the same. The NH<sub>3</sub> content of samples, however, prepared at basic pH values was about 50 w% higher than the value corresponding to stoichiometry, while of those prepared at acidic pH values was approximately equal to the stoichiometric NH<sub>3</sub> content. These two kinds

of materials, looking thus alike according to the room temperature X-ray diffraction investigations differ significantly in the way of their thermal decomposition, investigated in a high temperature Guinier-Lenné type camera. When ageing the samples containing excess "fixed" NH<sub>3</sub> for several days at room tempera-

ture solid state reaction was observed between the "mother" ammonium paramolybdate tetrahydrate lattice and the excess NH<sub>3</sub> fixed in the lattice, resulting in an other molybdate compound having higher NH<sub>3</sub> content stoichiometrically. Not only the mentioned compounds, but also several other lattices may contain considerable amount of excess "fixed" species /water, ammonium etc./ which can influence the further chemical and physical behaviour of the material. The results of such investigations led us to the conclusion that it would be necessary to revise the present conception of the stoichiometry. Complex investigations would be required using beside the room temperature X-ray diffraction measurements high temperature X-ray techniques, DTA, DTG, mass spectrometry, NMR and many other methods to reveal the real stoichiometry of the material and its relation to the lattice stoichiometry, i.e. to the ratio of the building elements of the crystal lattice. Due to this recognized flexibility of lattices, being able to incorporate excess water, or other species by expanding themselves anisotropically great care has to be taken when characterizing the structure of such a compound by its room temperature unit cell parameters.

04.2-6 DISTORTED OCTAHEDRAL COPPER(II) COMPLEX OF CIMETIDINE. By A. Sancho, J. Borrás and L. Soto, Dept. Quím. Inorgánica, Fac. Farmacia, Valencia, Spain, and C. Esteban-Calderón, M. Martínez-Ripoll and S. García-Blanco, Dept. de Rayos X, Inst. Rocasolano CSIC, Serrano 119, Madrid-6, Spain.

Cimetidine, whose structure has been reported recently [Hädicke et al. (1978) *Chem. Ber.* III, 3222; Shibata et al. (1983) *Acta Cryst.* C39, 1255] can act as a bidentate ligand through the N atom of imidazole and the S atom of the chain. The title compound corresponds to a Cimetidine-Cu(II) perchlorate complex with formula Cu(CM)2 (ClO4)2. Crystal data are M=769.14, D<sub>x</sub>=3.04 Mg m<sup>-3</sup>, monoclinic, P2<sub>1</sub>/c, Z=4, a = 12.306(3), b = 8.576(3), c = 15.995(4) Å, β = 94.48(2)°, V = 1683(1) Å<sup>3</sup>, λ(MoKα) = 0.71069 Å, μ(MoKα) = 19.7 cm<sup>-1</sup>, R = 0.042 for 1265 observed reflexions. The figure shows a perspective view of the molecular structure without the perchlorate group. The metal atoms, lying on crystallographic symmetry centres and interconnecting cimetidine molecules, are six coordinated, with a distorted octahedral environment. The crystal packing is due to H-bonds of N-H...O type, which connect the perchlorate group with the organic rest. The IR spectrum of the complex shows the shifts of the -C≡N, C=N-C=C and C-S stretching vibrations, reflecting the involvement of these groups in the coordination. Other bands can be assigned to the stretching vibrations of M-S, M-N(nitrile) and M-N(imidazole). Polycrystalline EPR spectral data (g// ca. 2.20 and a<sub>1</sub> = 2.08) and magnetic data (μ<sub>eff</sub> = 2.11 MB) confirm a pseudooctahedral coordination around Cu(II).

