

MS39 P01

A natural C₂₁H₂₆O₆ bicyclo-octenone disordered modulated structure. Nicolas Guiblin^a, Kurt Schenk^a, Lukáš Palatinus^a, Andrew Marston^b, Gervais Chapuis^a. ^a*Laboratoire de Cristallographie, EPFL, CH-1015 Lausanne, Switzerland.* ^b*Laboratoire de pharmacognosie et phytochimie, Université de Genève, CH-1211 Geneva, Switzerland.* E-mail: nicolas.guiblin@epfl.ch

Keywords: organic materials obtained from plants, modulated crystals, single-crystal structure determination

The modulated structure of the natural compound, 6-benzo[1,3]dioxol-5-yl-4-hydroxy-3,5-dimethoxy-7-methyl-1-propyl-bicyclo[3.2.1]oct-2-en-8-one, is presented.

Data collection was performed on a single crystal, at room temperature, on a Stoe I image plate diffractometer.

The average structure is triclinic, space group P1, with cell parameters $a = 6.718(4) \text{ \AA}$, $b = 12.049(7) \text{ \AA}$, $c = 12.638(7) \text{ \AA}$, $\alpha = 107.10(5)^\circ$, $\beta = 105.25(5)^\circ$ and $\gamma = 89.96(6)^\circ$. Two independent molecules in the asymmetric unit were used.

The modulation, associated with the \mathbf{q} -vector (0.4993(8), 0.500(1), 0.250(1)), was found using the charge flipping method [1].

We used the program JANA2000 [2] to refine the average and the modulated structure.

The refinement was performed using a riding model for hydrogen atoms. Atomic displacement parameters (ADP) were also restrained, with a value of 1.2 times the neighbour atom. All the atoms present a displacive modulation. The propyl group attached to the bicycle presents two alternative positions, which is observed in the average structure by huge ADP values. This was refined using occupational modulation.

[1] Oszlanyi G., Sütő A. (2004): Ab initio structure solution by charge flipping, *Acta Cryst.* A60, 134-141; Oszlanyi G., Sütő A. (2005): Ab initio structure solution by charge flipping. II. Use of weak reflections, *Acta Cryst.* A61, 147-152.

[2] Petříček, V., Dušek, M., Palatinus, L. JANA2000. The crystallographic computing system (2003). Institute of Physics, Praha, Czech Republic.

MS39 P02

Single crystal x-ray study of Ba₂Cu₂Te₄O₁₁Br₂ and its incommensurate modulated superstructure companion Ba₂Cu₂Te₄O_{11+δ}Br₂ Rie Takagi, Mats Johnsson and Sven Lidin

Inorganic Chemistry, Stockholm University, SE-106 91 Stockholm, Sweden. E-mail: takagi@inorg.su.se

Keywords: incommensurate, superstructure, modulation

Compounds containing lone pair elements, such as Te, are very interesting from the structural point of view, as the lone pair non-bonding regions create low dimensional geometrical arrangements. We synthesized the two new compounds Ba₂Cu₂Te₄O₁₁Br₂ (**I**) and Ba₂Cu₂Te₄O_{11.237}Br₂ (**II**) as members of the AE-Cu-Te-O-X (AE=alkaline earth, X=halide) family of compounds *via* transport reactions. Preliminary single crystal X-ray analysis indicates that compound **I** crystallizes in the orthorhombic system, but attempts at refinement proved unsatisfactory. A closer inspection of the reciprocal lattice reveals systematic, non-crystallographic absences that indicate twinning. The structure is in fact triclinic (S.G. C-1,

equivalent to P-1), with the unit cell parameters (at 120K) $a=10.9023(6)$, $b=15.0824(8)$, $c=9.3810(5)$, $\alpha=89.662(4)$, $\beta=106.895(4)$, $\gamma=89.992(4)$. It is layered and built from [TeO₃E] tetrahedra, [TeO₃₊₁E] trigonal bipyramids (E being the lone pair of Te^{IV}), [CuO₄] squares and irregular [BaO₁₀Br] polyhedra. The crystal structure of **II** shows the same basic structure as **I**, but with partial occupancy on one O site. The presence of satellites reveals that ordering of these O partial vacancies creates an incommensurate modulation for Br and Te. The modulated structure of **II** was solved in the triclinic super space group X-1($\alpha\beta\gamma$)0, with the \mathbf{q} vector $\approx 1/16\mathbf{c}^*$.

[1] C.R. Feger and J. W. Kolis, *Inorg. Chem.*, 37 (1998) 4046-4051.

[2] J. Galy, G. Meunier, S. Andersson, and A. Åström, *J. Solid State Chem.*, 13 (1975) 142.

MS39 P03

Incommensurate phases in ternary solid solutions P. J. Bereciartua^a, T. Breczewski^b, F. J. Zuñiga^a, ^a*Dept. of Condensed Matter Physics,* ^b*Dept. of Applied Physics II, University of the Basque Country, Bilbao, Spain.* E-mail: wmbbepep@ehu.es

Keywords: incommensurate structures, solid solutions, modulated crystal structures

In the ternary system In₂O₃:TiO₂:A₂O₃ and In₂O₃:TiO₂:BO, with A: Al, Cr, Mn, Fe, Ga and B: Mg, Mn, Co, Ni, Cu, Zn, there exist some solid solutions within certain range. They are monoclinic or orthorhombic incommensurate phases depending on the temperature and the composition [1]. The present work has studied the structure of the monoclinic one in the system InAl_{1-x}Ti_xO_{3+x/2} ($x=2/3$). It consists on InO₆ octahedral layers which alternate along \bar{c} with M-O planes (M=Al, Ti). These M atoms share the same position with complementary occupation, whereas the sites of O atoms of this plane (O2) are partially occupied.

The parameters of the unit cell are $a=5.857(4)\text{\AA}$, $b=3.361(3)\text{\AA}$, $c=6.355(5)\text{\AA}$ and $\beta=107.96(5)^\circ$, and the modulation wavevector is parallel to the monoclinic axis $\bar{q}=0.3503(5)\bar{b}^*$. The systematic extinctions and the structure refinement point out that superspace group is $C2/m(0\sigma_2)0s0$.

A modulated structure model has been proposed in which the atoms of the octahedral layers modulate their positions and the M atoms modulate also their occupation complementary. Besides, the atomic domain of O2 can be represented by a sawtooth function combined with several orthogonal harmonic functions.

On the other hand, a composite model has been checked. It includes two subsystems. One of them is formed by the octahedral layers and the M atoms, while the O2 atoms make up the other one, as in the model proposed for the InCr_{1-x}Ti_xO_{3+x/2} ($x=2/3$) system [2]. However, unlike in this model, the atomic domain of the O2 is discontinuous, described as in the modulated model. The equivalence between both models is shown.

[1] Brown F., Kimizuka N., et al., *J. Solid State Chem.*, 1999, 147, 438.

[2] Michiue Y., Onoda M., et al., *J. Solid State Chem.*, 2004, 177, 2644.