MS39 P01

A natural $C_{21}H_{26}O_6$ bicyclo-octenone disordered modulated structure. Nicolas Guiblin[a], Kurt Schenk[a], Lukáš Palatinus[a], Andrew Marston[b], Gervais Chapuis[a]. *aLaboratoire de Cristallographie, EPFL, CH-1015 Lausanne, Switzerland. bLaboratoire 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) Á, b = 12.049(7) Á, c = 12.638(7) Á, α = 107.10(5)°, β =105.25(5)° and γ = 89.96(6)°. Two independent molecules in the asymmetric unit were used.

The modulation, associated with the **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.

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MS39 P02

Single crystal x-ray study of $Ba_2Cu_2Te_4O_{11}Br_2$ and its incommensurate modulated superstructure companion $Ba_2Cu_2Te_4O_{11+\delta}Br_2$ Rie Takagi, Mats Johnsson and Sven Lidin
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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 $Ba_2Cu_2Te_4O_{11}Br_2$ (**I**) and $Ba_2Cu_2Te_4O_{11.237}Br_2$ (**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), α =89.662(4), β=106.895(4), γ=89.992(4). It is layered and built from [TeO_3E] tetrahedra, [$TeO_{3+1}E$] trigonal bipyramids (E being the lone pair of Te^{IV}), [CuO_4] squares and irregular [$BaO_{10}Br$] 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 q vector≈1/16**c***.

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MS39 P03

Incommensurate phases in ternary solid solutions P. J. Bereciartua[a], T. Breczewski[b], F. J. Zuñiga[a], *aDept. of Condensed Matter Physics, bDept. 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 In_2O_3:TiO_2:A_2O_3 and In_2O_3:TiO_2: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 InO_6 octahedral layers which alternate along $\vec{c}$ 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)Å, b=3.361(3)Å, c=6.355(5)Å and β=107.96(5)º, and the modulation wavevector is parallel to the monoclinic axis $\vec{q} = 0.3503(5)\vec{b}^*$. The systematic extinctions and the structure refinement point out that superspace group is $C2/m(0\sigma_2 0)s0$.

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 octahedrals 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 domanin of the O2 is discontinuous, described as in the modulated model. The equivalence between both models is shown.

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