m14.p02

(3+2)-dimensional superspace approach of the structure of lévyclaudite-(Sb), a member of the cylindrite-type minerals

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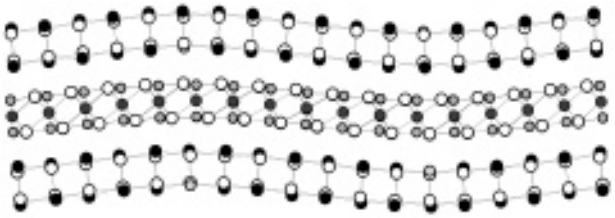
Lévyclaudite, ~ $Pb_8(Bi,Sb)_3Sn_7Cu_3S_{28}$, is a member of the cylindrite mineral group [1]. These complex sulfides are inorganic misfit layer compounds, composed of two interpenetrating, incommensurately modulated subsystems: *Q* pseudotetragonal *M*S (*M* = Pb, Sn, Bi, Sb ...) and *H* pseudohexagonal *M'*S_2 (*M'* = Sn, Fe, Cu). In lévyclaudite-(Sb), ~ $(Pb_{1.47}Sb_{0.53}S_2)_{0.678}(Sn_{0.85}Cu_{0.30}S_2)$, the partial substitution of Pb^{II} by Sb^{III} in the *Q* subsystem is balanced by that of Sn^{IV} by 2 Cu^{I} in the *H* subsystem. Its structure has been determined from a synthetic sample [2] by single-crystal X-ray diffraction on the basis of the (3+2)-dimensional space group *P*-1(*abg*), using a beta version of Jana2006 [3]. Since satellites up to the 5th order have been measured, occupation waves up to the 4th order (Pb/Sb) could be introduced in the refinement, along with displacive (6th: Pb/Sb; 4th: Sn; 3rd: Cu,S1,S2) and Debye-Waller (4th: Pb/Sb; 2nd: Sn; 1st: S2) waves. Refinement on the basis of 11986 reflections with $I > 3s(I)$ converged to $R = 0.071$ ($wR = 0.086$) for 188 parameters.



As opposed to the structure of the $(MX)_{1+\delta}(TX_2)_n$ inorganic misfit layer compounds, the lévyclaudite structure presents a very large corrugation of the layer subsystem (see figure above). The structure analysis shows that in the *Q* layer the Pb^{II} substitution by the Sb^{III} atoms occurs in the inner side of the curved layer whereas in the *H* layer the Sn^{IV} by 2 Cu^{I} exchange is fully disordered, with two copper atoms in triangular coordination on both sides of the curved layer, in replacement of one tin atom in octahedral coordination in the middle of the layer.

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m14.p03

The organic modulated structure of 3,4-diphenyl-2a,5a,6,7,8,8a,8b-heptahydro-furo[4,3,2-de]chromen-2-one

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Keywords: organic crystal structures, modulated crystals, single-crystal structure determination

The incommensurate structure of the title compound, with empirical formula $C_{22}H_{20}O_3$, is presented. Data collection was performed on a single crystal, at 173 K, on a Stoe II image plate diffractometer. The average structure is monoclinic, space group $P2_1/c$, with cell parameters a = 7.2526(9) Å, b = 24.952(3) Å, c = 9.896(1) Å, β = 106.646(8)°. Satellite reflections are visible up to second order.
The refinement was performed using some constraints in hydrogen atomic positions, taking into account geometrical considerations. Atomic displacement parameters (ADP) were also restrained. We used the program JANA2000 [1] to refine the average and the modulated structure. The average structure displayed huge ADP values (Fig. 1), which were considerably reduced by introducing a displacive modulation wave function up to third order. All ADP values became quasi-isotropic, and the main displacement of the molecule occurs along the long axis of the structure.

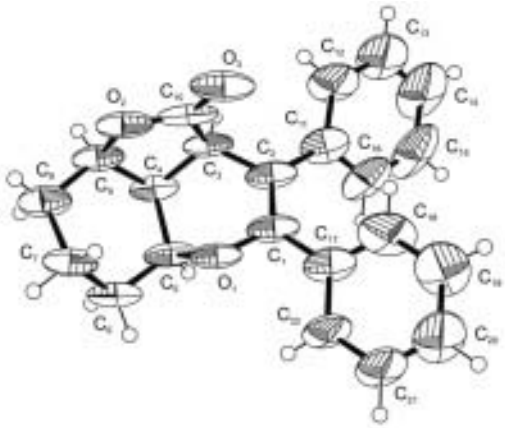

Fig. 1: The molecular structure, corresponding to the average structure, showing 50% probability displacement ellipsoïds and the crystallographic numbering scheme. H atoms are drawn as open circles for clarity.

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