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Keywords: charge density, spin-crossover, hemeprotein

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Cation order and magnetic structure of sbvo$_4$ catalyst

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$SbVO_4$ series of compounds could be used as a catalyst for the production of 20% cheaper acrylonitrile by the ammoxidation of propane compared to current method producing 8×10^6ton/year. In the non-stoichiometric series described as $Sb_{0.9}V_{0.9+x}\square_{0.2-x}O_4$ ($0\leq x\leq0.2$), cation vacancies ($\square$) are introduced in the basic rutile type-structure following the mechanism: $4V^{3+} \longrightarrow 3V^{4+} + \square$, while antimony remains as Sb^{5+}. The reduced phase richest in V^{3+} has been reported to be $Sb_{0.9}V_{1.1}O_4$, which shows by electron diffraction superlattice reflections characteristic of a 2-fold rutile superstructure along ***c***. However the X-ray powder diffraction pattern showed only basic rutile reflections (a_r = 4.6085(1), c_r = 3.0867(1) Å, S.G. $P4_2/mnm$). The careful reciprocal lattice study by electron diffraction has revealed the following unit cell for the rutile superstructure: $a=\sqrt{2}a_r$, $b = \sqrt{2}b_r$, $c = 2c_r$. (subindex *r* refers to the basic rutile unit cell). Its space group, $I4_1md$, was determined by means of CBED. A structural model based on alternating Sb and V cations ordering along ***c*** in the chains of edge-sharing octahedra was proposed. No cation vacancies have been observed for this reduced phase, while for the compounds synthesized in oxidizing conditions the presence of vacancies has been confirmed. In fact, electron diffraction experiments have shown that vacancies order in the basic rutile structure giving rise to a modulated structure on the other end member of the series, namely $Sb_{0.9}V_{0.9}O_4$. On the other hand, our magnetic susceptibility studies indicate for the first time possible magnetic ordering. Thus for the magnetic structure determination we performed the study of 3 different samples of the series $Sb_{0.9}V_{0.9+x}\square_{0.2-x}O_4$ by using neutron diffraction: **1)** a reduced phase rich on V^{3+} which shows the nuclear superstructure by Sb-V ordering. **2)** an oxidized phase rich on V^{4+} which shows the vacancies ordering. **3)** an intermediate phase which shows Sb-V disorder. In this way, we try to determine the magnetic structure of reduced $SbVO_4$ coming from the ordering of vanadium magnetic moments, which certainly takes place at T_N ~50K, and to study how the substitution of V^{3+} (S=1) by V^{4+} (S = ½), while the synthesis conditions become more oxidizing, affects the spin arrangement in $Sb_{0.9}V_{0.9+x}\square_{0.2-x}O_4$. This order seems to be destroyed by the appearance of vacancies and the chemical disorder in the cations positions. To follow the evolution of the order parameter we carried out neutron diffraction experiments ranging at temperatures from room (RT) to 1.5K. Another aim is to confirm the model of nuclear superstructure that we proposed based on the electron diffraction and HRTEM data, as far as it is not possible to measure the intensity in an absolute scale with the mentioned technique, and due to the higher contrast for the cations involved which is achievable with neutron diffraction compared with X-rays. The fact that V coherent scattering length for neutrons is rather small give us an obvious advantage to determine the superstructure due to alternating Sb-V occupation of two sites split from the substructure. To measure the samples at RT we used high resolution neutron powder diffraction. Due to the unique property of the neutrons to interact with ordered magnetic moments, the magnetic scattering coming from the V spins could be constrained to its crystallographic position, giving more information than at first glance could be expected by its almost null nuclear scattering. Weak reflections could be associated to the vacancies modulation in the oxidized sample.

Keywords: catalyst structure; neutron, X-ray, electron diffraction

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H-bonding in clathrates bis-p-toluidine gossypol with DMFA and 1,4-dioxane

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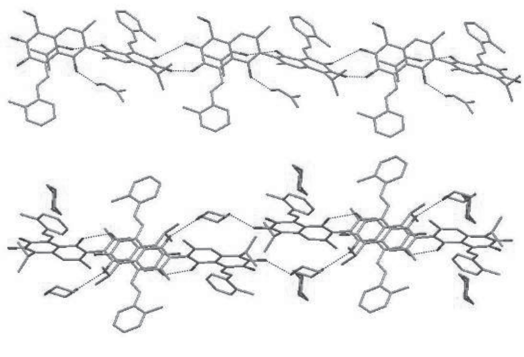
Gossypol and its products of condensation with some amines (Schiff's base type derivative) are versatile hosts, capable to trap within the crystal structure the numerous types of guest species [1]. The studied clathrates of bis-p-toluedinegossypol with DMFA (1) and dioxane (2) were prepared by reaction of condensation between gossypol and p-toluedine in the corresponding solvents. The crystal of (1) – brown plate, Sp.gr.$P2_1/n$, cell parameters: a=15.8157(6)Å, b=15.9752(9)Å, c=18.8890(9)Å, β=102.940(4)°, V= 4651.3(4) Å^3 , the host/guest ratio is 1:2. Host and guest molecules through hydrogen bonding are formed 1D supramolecular associate in the *a*-axis direction. The crystal of (2) – brown prism, Sp. gr.P-1, a=9.3027(3)Å, b=12.6333(4)Å, c=20.5659(5)Å, α=93.249(2)°, β=96.086(2)°, γ=109.954(3)°, V=247.90(10)Å^3. Host/guest ratio is 1:2. In this structure host molecules form centrosymmetric dimers *via* H-bonds and together with 1,4-dioxane molecule of the first type form host-guest matrix, which running as 1D chain in the *c*-axis direction. Second type of 1,4-dioxane molecules are located in channels of the crystal structure without any H-bonding. Thermal stability of the obtained clathtates has been studied by TG-DSC method.



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New molecular conductors with iron bis(dicarbollide) anion - synthesis, crystal structure and electrical conductivity

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