Structural polymorphism in new organic-inorganic hybrid:pyrazolium bromoantimonates(III) $[C_3N_2H_5]_6Sb_4Br_{18}\cdot 2H_2O$ (tetragonal and triclinic forms). Thermal, dielectric and proton magnetic resonance (^{1}H NMR) studies on the tetragonal form.

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Streszczenie

Two polymorphic forms of pyrazolium bromoantimonates(III) - $[C_3N_2H_5]_6Sb_4Br_{18}\cdot 2H_2O$: α modification - tetragonal form and β modification - triclinic form, have been synthesized and structurally characterized at 100 K. The α-polymorph consists of a unique three-dimensional (3D) anionic substructure built up of cyclic tetramers and discrete chains linked to each other, three nonequivalent pyrazolium cations and water molecules. The β-polymorph is characterized by discrete cyclic anionic tetramers $[Sb_4Br_{18}]^{-6}$, three nonequivalent cations and water molecules. The physical properties have been studied for the $[C_3N_2H_5]_6Sb_4Br_{18}\cdot 2H_2O$ (α form). The differential scanning calorimetry (DSC) revealed a complex sequence of phase transitions above room temperature. The low-frequency dielectric relaxation process was found to appear at low temperatures, which was assigned to the dynamics of dipolar pyrazolium cations. The molecular motions of the pyrazolium cations in the α-polymorph were studied in a wide temperature range by means of a proton magnetic resonance (^{1}H NMR) technique. Two minima of the spin-lattice relaxation time (T_1) are disclosed: at low temperatures a wide one attributed to a small-angle-libration of cations and motion of water molecules, whereas the high temperature one assigned to the reorientation of cation about its pseudo-fivefold axis.

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