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NMR Solid-Echo Study of Guanidinium Cation Reorientations in $[C(NH_2)_3]_3Sb_2Cl_9$

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In guanidinium salts the planar or nearly planar guanidinium cation $[C(NH_2)_3]^+$ undergoes a simple reorientation around its C_3 symmetry axis. X-ray analysis of the tris-guanidinium nonachlorodiantimonate, $[C(NH_2)_3]_3Sb_2Cl_9$, has revealed in the compound two types of crystallographically inequivalent guanidinium cations: the first one – situated between polyanionic $(Sb_2Cl_9^{3-})_n$ layers, the second one – inside cavities formed by polyanions [1]. From temperature dependences of the proton NMR second moment and spin-lattice relaxation time performed for the compound it follows that different crystalline environments of the guanidinium cations lead to an unusual large dynamical inequivalence of two types of the cations [2].

The aim of the present work is to study the dynamics of the guanidinium cations in $[C(NH_2)_3]_3Sb_2Cl_9$ by using the temperature

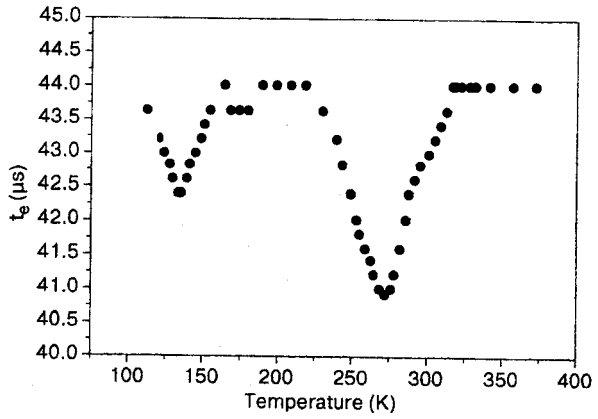

Fig. 1. The temperature dependence of the time position t_e.

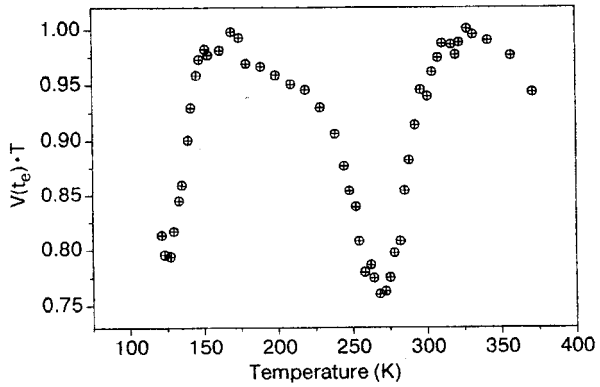

Fig. 2. The temperature dependence of amplitude $V(t_e) \cdot T$ of echo signal.

dependences of the time position and amplitude of solid-echo signals observed after the pulse sequence $(2n + 1)$–90^0_Y–90^0_X–t $(n = 1, 2...)$ [3]. As it was shown in our earlier papers [3, 4] the time position and amplitude of the solid-echo maximum can yield valuable information about dynamical processes in solids. The experimental temperature dependences of the time position t_e (μs) and amplitude $V(t_e) \cdot T$ of solid-echo signal for pulse sequence 630^0_Y–90^0_X–t, obtained for the compound studied are shown in Figs. 1 and 2. The both plots show two distinctly separated minima of different depths: one – at 130 K and another one – at 275 K. In order to explain the experimental data the results presented in [3, 4] have been applied. A comparison of theoretical and experimental data well confirms the model proposed in [2], that is an existence of two dynamically different guanidinium cations.

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