The NMR solid-echo polycrystalline tris-guanidinium nonachlorodiantimonate (III) has been studied in a wide temperature range. The temperature dependences of a time position and an amplitude of solid-echo are characterized by minima at ca. 143 K and 273 K, which are assigned to the reorientation of two dynamically inequivalent guanidinium cations \( [\text{C(NH}_2\text{)}_3]^+ \). The motional parameters of the two types of guanidinium cations have been determined. — PACS: 64.70K; 76.60.E

Key words: Organic Crystals; Nuclear Magnetic Resonance.