Crystal Structure and Phase Transition of 4-Aminopyridinium Tetrabromoantimonate(III) as Studied by Bromine and Antimony NQR, Proton NMR, and Single Crystal X-Ray Diffraction

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The crystal structure of the room temperature phase (RTP) of the title compound was determined at 297 K (monoclinic, space group C2/c, a = 1384.2(2), b = 1377.8(3), c = 755.5(2) pm, β = 121.58(1)°). A complicated disorder was found for the cation. A phase transition from the low-temperature phase (LTP) to the RTP was found at (224 ±1) K (T_c). The ^{1}H NMR spectra showed a sharp motional narrowing at ca. $T = T_c$, indicating the occurrence of a reorientational motion of the cation in the RTP in accord with the disorder. It was found that another reorientational motion is excited in the LTP. Four ^{81}Br NQR lines (132.71, 115.38, 61.54 and 59.31 MHz at 77 K) and two Sb NQR lines (53.78 and 33.76 MHz at 77 K) were found in the LTP, while a single ^{81}Br NQR line was observed at $T > 276$ K (ca. 121.80 MHz at 300 K). Crystal dynamics are discussed on the basis of the temperature dependence of the NQR, ^{1}H NMR line width, and ^{1}H NMR T_1.

Key words: 4-NH_2PyHSbBr_4; Crystal Structure; Phase Transition; NQR; ^{1}H NMR.