Neue Tetraaminophosphonium-Salze durch Anionenaustausch in flüssigem Ammoniak

Novel Tetraaminophosphonium Salts by Anion Exchange in Liquid Ammonia

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$[P(NH_2)_4]Br$ and $[P(NH_2)_4][NO_3]$ have been prepared by anion exchange in liquid ammonia. Single crystals of $[P(NH_2)_4]Br$ were obtained from an acetonitrile solution in a temperature gradient between 60 °C and room temperature while attempts to grow single crystals of $[P(NH_2)_4][NO_3]$ yielded $[P(NH_2)_4][NO_3](OP(NH_2)_3)$. Both crystal structures were determined by single crystal X-ray methods at room temperature ($[P(NH_2)_4]Br$: P4/nbm, a = 809.2(1), c = 468.1(1) pm, Z = 2, $R1$ = 0.042, $wR2$ = 0.077; $[P(NH_2)_4][NO_3](OP(NH_2)_3)$: $Pna2_1$, Z = 4, a = 1023.4(1), b = 1704.7(1), c = 618.0(1) pm, $R1$ = 0.025, $wR2$ = 0.067. In the solid $[P(NH_2)_4]Br$ forms a tetragonally distorted variant of the CsCl type of structure. $[P(NH_2)_4][NO_3](OP(NH_2)_3)$ consists of $[P(NH_2)_4]^+$ cations, $[NO_3]^-$ anions, and $OP(NH_2)_3$ molecules which are interconnected by a complex system of hydrogen bonds.