Synthese, Kristallstruktur und Eigenschaften von Triguanidinium-tri-μ-imidocyclotriphosphat-Monohydrat und Tetraguanidinium-tetra-μ-imidocyclotetraphosphat-Tetrahydrat, $[C(NH_2)_3]_3(PO_2NH)_3 \cdot H_2O$ und $[C(NH_2)_3]_4(PO_2NH)_4 \cdot 4\ H_2O$

Synthesis, Crystal Structure, and Properties of Triguanidinium Tri-μ-imidocyclotriphosphate Monohydrate and Tetraguanidinium Tetra-μ-imidocyclotetraphosphate Tetrahydrate, $[C(NH_2)_3]_3(PO_2NH)_3 \cdot H_2O$ and $[C(NH_2)_3]_4(PO_2NH)_4 \cdot 4\ H_2O$

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Z. Naturforsch. **53 b,** 1115–1126 (1998); eingegangen am 16. April 1998

Trimetaphosphimate, Tetrametaphosphimate, Crystal Structure, Thermal Properties, IR Data

Coarsely crystalline $[C(NH_2)_3]_3(PO_2NH)_3 \cdot H_2O$ (**1**) and $[C(NH_2)_3]_4(PO_2NH)_4 \cdot 4\ H_2O$ (**2**) have been obtained by addition of $[C(NH_2)_3]_2CO_3$ to a freshly prepared solution of $H_3(PO_2NH)_3$ or to a suspension of $H_4(PO_2NH)_4 \cdot 2\ H_2O$, respectively, followed by diffusion controlled addition of acetone. The crystal structures of **1** and **2** have been determined by single crystal X-ray methods ($[C(NH_2)_3]_3(PO_2NH)_3 \cdot H_2O$: Pbca; a = 1565.6(2), b = 1068.31(6), c = 2091.8(2) pm, Z = 8; $[C(NH_2)_3]_4(PO_2NH)_4 \cdot 4\ H_2O$: Pbcn; a = 1739.64(10), b = 1084.19(6), c = 1334.96(11) pm, Z = 4). The P_3N_3 and P_4N_4 rings of the anions exhibit the chair conformation. In **1** two cyclic anions are interconnected into pairs via hydrogen bonds. In **2** columns along [001] are formed by hydrogen bonds between the anions. Hydrogen bonding through water molecules and $C(NH_2)_3^+$ ions results in a three-dimensional network in **2**. Complete dehydration is achieved below 160 or 180 °C for **1** or **2**, respectively. Heating to 700 °C causes condensation reactions with evolution of NH_3 and H_2O yielding X-ray amorphous products.

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