

Dehydration, polymorphism and ionic conductivity of sodium hypodiphosphates

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A series of 12 compounds: $\text{Na}(\text{H}_3\text{P}_2\text{O}_6)$ (**1** and **2**), $\text{Na}(\text{H}_3\text{P}_2\text{O}_6)\cdot\text{H}_2\text{O}$ (**3**), $\text{Na}_2(\text{H}_2\text{P}_2\text{O}_6)$ (**4** and **5**), $\text{Na}_2(\text{H}_2\text{P}_2\text{O}_6)\cdot 2\text{H}_2\text{O}$ (**6**), $\text{Na}_2(\text{H}_2\text{P}_2\text{O}_6)\cdot 6\text{H}_2\text{O}$ (**7** and **8**), $\text{Na}_5(\text{H}_2\text{P}_2\text{O}_6)(\text{HP}_2\text{O}_6)\cdot 21\text{H}_2\text{O}$ (**9**), $\text{Na}_3(\text{HP}_2\text{O}_6)$ (**10** and **11**) and $\text{Na}_3(\text{HP}_2\text{O}_6)\cdot 9\text{H}_2\text{O}$ (**12**) has been characterized by variable-temperature optical microscopy, thermogravimetry, single-crystal and powder X-ray diffraction. Crystalline salts **1–4**, **6–10** and **12** have been obtained in typical solution crystallization processes. Dehydrations of $\text{Na}(\text{H}_3\text{P}_2\text{O}_6)\cdot\text{H}_2\text{O}$ (**3**), $\text{Na}_2(\text{H}_2\text{P}_2\text{O}_6)\cdot 6\text{H}_2\text{O}$ (both **7** and **8**) and $\text{Na}_3(\text{HP}_2\text{O}_6)\cdot 9\text{H}_2\text{O}$ (**12**) were monitored by TGA, variable-temperature microscopy and powder or microsample powder X-ray diffraction, and turned out to be one-step, destructive transformations, resulting in almost pure triclinic form of $\text{Na}(\text{H}_3\text{P}_2\text{O}_6)$ (**1**), $\alpha\text{-Na}_2(\text{H}_2\text{P}_2\text{O}_6)$ (**4**) and $\alpha\text{-Na}_3(\text{HP}_2\text{O}_6)$ (**10**), respectively. Sodium ionic conductivity was calculated using the bond valence sum method for all obtained anhydrous substances and measured for the best candidate, compound **4** in the temperature range 300–420 K, reaching 10^{-4} S m^{-1} .

Słowa kluczowe

crystal structure, sodium salts, hypodiphosphoric acid, polymorphism, dehydration, powder X-ray diffraction, thermal analysis, ionic conductivity

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