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## Phosphorus, Sulfur, and Silicon and the Related Elements

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## High-Temperature Synthesis of the $\text{Mg}_{2-x}\text{Ca}_x\text{P}_4\text{O}_{12}$ and $\text{Cd}_{2-x}\text{Ca}_x\text{P}_4\text{O}_{12}$

M. Trojan<sup>a</sup>; D. Brandova<sup>a</sup>; P. Mazan<sup>a</sup>

<sup>a</sup> Institute of Chemical Technology, Pardubice, Czechoslovakia

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# HIGH-TEMPERATURE SYNTHESIS OF THE $\text{Mg}_{2-x}\text{Ca}_x\text{P}_4\text{O}_{12}$ AND $\text{Cd}_{2-x}\text{Ca}_x\text{P}_4\text{O}_{12}$

M.TROJAN, D.BRANDOVÁ, and P.MAZAN  
 Institute of Chemical Technology, Lenin sqr. 565,  
 Pardubice, 532 10 Czechoslovakia

The cyclo-tetraphosphates of the  $\text{M}^{\text{II}}_{2-x}\text{Ca}_x\text{P}_4\text{O}_{12}$  type ( $\text{M}^{\text{II}} = \text{Mg}, \text{Cd}$ ) have been synthesized as new binary compounds, and their existence for  $x \in (0; 1 > (\text{Mg}))$  or for  $x \in (0; 0.7 > (\text{Cd}))$  has been proved. The synthesis is based on a two-step thermal process. The first step starts from pure cyclo-tetraphosphates of the two divalent metals and  $\text{Ca}(\text{PO}_3)_2$  which are melted in normal atmosphere and then abruptly cooled to give a vitreous amorphous product composed of higher linear phosphates of the summary formula  $(\text{M}^{\text{II}}_{2-x}\text{Ca}_x)_n/4 \text{H}_2\text{P}_n\text{O}_{3n+1}$ . In the second step, this product is repeatedly heated to a suitable temperature and recrystallized to give microcrystalline product  $\text{M}^{\text{II}}_{2-x}\text{Ca}_x\text{P}_4\text{O}_{12}$ . The colourless (white) products crystallize in the monoclinic system, C2c group. Their structural parameters have the values for  $\text{Mg}_{2-x}\text{Ca}_x\text{P}_4\text{O}_{12}$ :  $a = 11.749(5)$  to  $12.063(4)$  Å,  $b = 8.278(4)$  to  $8.635(4)$  Å,  $c = 9.905(4)$  to  $9.875(3)$  Å and  $\beta = 118.92(2)^\circ$  to  $118.03(2)^\circ$ ; or for  $\text{Cd}_{2-x}\text{Ca}_x\text{P}_4\text{O}_{12}$ :  $a = 12.328(4)$  to  $12.457(5)$  Å,  $b = 8.639(3)$  to  $8.732(4)$  Å,  $c = 10.388(3)$  to  $10.443(4)$  Å and  $\beta = 119.33(2)^\circ$  to  $119.45(2)^\circ$ .