

A CONTRIBUTION TO THE INVESTIGATION OF $\text{CaCO}_2/\text{PO}_4/2$ *
CRYSTALLIZATION

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In synthesizing the compound we have used those substances that react on increase in temperature by releasing by-products in gaseous state those being :

$\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, CaCO_3 and $(\text{NH}_4)_2\text{HPO}_4$. All the substances were of p.a. purity.

The synthesis can be expressed in the following manner: $2\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O} + \text{CaCO}_3 + 2(\text{NH}_4)_2\text{HPO}_4 = \text{CaCO}_2(\text{PO}_4)_2 + \text{CO}_2 + 4\text{NO}_2 + \text{O}_2 + 4\text{NH}_3 + 15\text{H}_2\text{O}$.

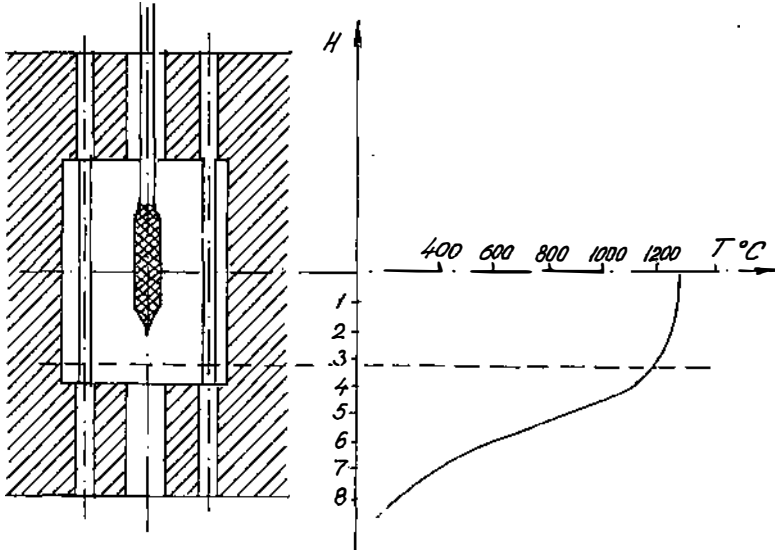
The substances used were ground in the achate vessel, measured with 0.1 mg accuracy in the stoichiometric ratio and intimately mixed. The substance prepared in such a manner, was heated in the china cup for two hours at the temperature of 700°C . The substance was ground again, then heated for 24 hours at the temperature of 900°C , and finally again powdered in the achate vessel. In that manner the substance was prepared for melting.

The melting and crystallization (by use of Bridgman method) was performed in an open and appropriately shaped quartz vessel of 15 cm^3 in volume. The furnace with a high temperature gradient was used. The temperature gradient along the furnace vertical axes in the crystallization zone was $65^\circ\text{C}/\text{cm}$. The constant temperature was maintained by the stabilization of the furnace power by means of a magnetic voltage stabilizer. During the operation lasting for several tens of hours the temperature variation did not exceed 2°C .

The vessel with the sample was placed into the central zone of thermally stabilized furnace where the temperature was about 150°C above the sample melting point.

The merged mass was kept at this temperature for two hours and then the mechanism for sample lowering toward the crystallization zone (1050°C) was put into operation. The lowering was performed at a constant rate.

This procedure was repeated several times and we have tried to vary only the substance speed through the crystallization zone, while the rest of the conditions were kept constant.



This series of crystallizations was started with the minimum shift speed $V_{\min}=2$ mm/h, and then this speed was increased so as to reach $V_{\max}=25$ mm/h.

The following has been observed during the experiment:

At low crystallization speeds, the obtained substance was in the form of tiny crystal grains which are of irregular shape and orientation, porous and full of bubbles.

At greater crystallization speeds the more homogeneous substance of larger crystal grains is obtained.

The tendency toward a certain orientation can be observed, and the number of holes in the substance is found to be smaller. These characteristics are especially noticeable at $V_{\max}=25$ mm/h. Then the compact substance of clearly oriented crystal faces is obtained.

* The characteristics of this compound are described in the following papers:

1. M.Lj.Napijalo, Lj.Novaković, J.Dojčilović, B.Žižić, M.Rodić and R.Ristić, The Investigation of some Physical Characteristics of Calcium-Cobalt Phosphate
2. M.Lj.Napijalo, J.Dojčilović, B.Žižić, M.Rodić and A.Žikić, The Investigation of Dielectrical Characteristics of Mixed Phosphates of Natural Alkali and Transitional (3d) Metals.
Contr.papers on V Yugoslav Symposium on Physics of Condensed Matter.