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STUDY OF SYNTHESIS AND THERMAL TRANSFORMATIONS OF DOUBL NICKEL(II)-CADMIUM AQUA AMMINE MONOPHOSPHATES

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Studying phosphates of divalent metals, which are used in industry as phosphors, phosphate cement, phosphate optical glass, refractory compounds, fertilizers, ceramics, pigments, catalysts, active substances of corrosion-protection materials, is of considerable practical importance and is promising for obtaining new materials with valuable properties. Not only individual phosphates, but their solid solutions, in which can be changed the ratio between the cations or anions, or both cations and anions, are important.

The aim of this research was to study the thermal transformations of Nickel(II)-Cadmium aqua ammine monophosphates, general formula of compounds was $Ni_xCd_{3-x}(PO_4)_2 \cdot nNH_3 \cdot mH_2O$. Aforementioned compounds were prepared with salting out from aqueous ammoniac solution by acetone. The search for solid solutions based on such phosphates is of considerable practical importance for determining ranges of stability and properties of substances, which can be used as high-temperature materials, and for determining temperature ranges of formation new anhydrous substances, that can not be synthesized in another way.

Thermal transformations of compounds were studied by complex thermal analysis using a Q-1500D derivatograph. The samples were heated in air. In experiments with a constant heating rate of 5°C/min, the samples (0.5 g) were placed in covered cylindrical platinum crucibles with lids. IR spectra were measured on a Specord 75-IR spectrophotometer, using samples prepared as pellets with KBr where the analyze concentration was 0.2-0.3 wt %. For the analysis of synthesized aqua ammino phosphates and products of their thermolysis were used IR-spectrophotometry (Specord 75-IR spectrophotometer), qualitative and quantitative paper chromatography, X-ray phase analysis (DRON-UM1 diffractometer, CuK_{α} radiation).

Were identified and studied patterns of thermal transformations of synthesized: $Ni_{2,0}Cd_{1,0}(PO_4)_2$ ·4,8NH₃·6,2H₂O; $Ni_{1,5}Cd_{1,5}(PO_4)_2$ ·2,2NH₃·5,3H₂O; $Ni_{1,0}Cd_{2,0}$ (PO₄)₂·1,5NH₃·5,9H₂O. It was found, that they are crystalline and type of crystalline lattices is rhombic.

Was found, that thermolysis of $Ni_{1,5}Cd_{1,5}(PO_4)_2 \cdot 2,2NH_3 \cdot 5,3H_2O$ occurs in the range 291 K-1083 K and is accompanied by several endothermic effects in the DTA curve with minima at 396, 483, 553 i 573 K, and by several exothermic effects. It should be noted that, formation of diphosphate group stated by

polycondensation of monophosphate ion. It was found by X-ray analysis that at 506 K the substance becomes amorphous.

Formation of new crystalline phase - $Ni_{1,5}Cd_{1,5}(PO_4)_2$ started at 798 K and accompanied by exothermic effects at 943, 1033 K. Water and ammonia removed completely. Products of thermolysis are crystalline and isostructural to each other (type of crystalline lattices - monoclinic).