

THERMAL TRANSFORMATIONS OF SOLID NICKEL(II) AQUA AMMINE MONODIPHOSPHATE**N.M. Prokopchuk***National University of Life and Environmental Sciences of Ukraine*

Synthesis and thermal studies of new inorganic phosphate materials are becoming more actual, because they are widely used in modern technology and agriculture.

The aim of this research was to study the thermal transformations of Nickel(II) aqua ammine monodiphosphate.

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.

The X-ray phase analysis was carried out on a DRON-UM1 diffractometer (CuK α radiation). A single crystal of graphite placed in a diffracted beam was used as monochromator. The diffraction patterns were taken by the method of step scanning in the range of angles $2\Theta = 4...80^\circ$. The scanning step was 0,05°, and the expose time in a was 3...9 s. The measured diffraction maxima were approximated by the pseudo-Voigt function, with the K α_1 component being separated out.

The IR spectra were taken on a Specord 75-IR spectrophotometer. Samples of compounds to be analyzed were prepared in the form of KBr pellets in which the concentration of a substance under study was 0.2-0.3 wt %.

Anions components in phosphates were estimated by using data of qualitative and quantitative paper chromatography.

The nickel content of the samples was determined gravimetrically with dimethylglyoxime; the phosphorus content, gravimetrically by the quinolone-molybdenum method; the ammonia content, by distillation in a vacuum on Seren'ev's apparatus.

Ni_{2,5}(PO₄)_{1,0}(P₂O₇)_{0,5}·3,4NH₃·6,0H₂O was prepared by salting out from aqueous ammoniac solution. As starting reagents we used mechanical mixture consisting of Ni₃(PO₄)₂·8H₂O and Ni₂P₂O₇·6H₂O with a specified molar ratio of the PO₄³⁻ i P₂O₇⁴⁻; water ammonia (23÷25% mas) and acetone.

Thermal analysis of aqua ammine monodiphosphate was studied in the temperature range 292...1118 K. Thermolysis of this compound is accompanied by several weak endothermic and exothermic effects which superimpose to each other in the DTA and DTG curves. The most visible effects were with minima at 351, 369, 526 K. At range 858...936 K some exoeffects were observed, they were poorly separated at DTA and DTG curves.

There are four distinctly visible steps of the sample weight loss at 335...480; 486...573, 580...800 i 818...1041 K in thermo gravimetric curve.

It was found by X-ray analysis that starting Nickel(II) aqua ammine monodiphosphate was amorphous; final products of heating are crystalline and biphasic. One phase is isostructural to $\text{Ni}_3(\text{PO}_4)_2$, second – to $\text{Ni}_2\text{P}_2\text{O}_7$.