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THERMAL AND X-RAY STUDY OF $FeH_3(PO_4)_2 \cdot 2,5\ H_2O$

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ABSTRACT

The monocrystals synthesized at hydrothermal crystallization in Fe_2O_3-P_2O_5-H_2O system have been investigated by the thermal X-ray structure methods in vacuum. It have been considered the products of decomposition in vacuum at the temperatures 20°-250°C and above these temperatures. The corresponding interplane distances d_{hke} are given.

INTRODUCTION

The determination of crystal structure of $GaH_3(PO_4)_2 \cdot 2,5\ H_2O$ showed that the compounds such as $MH_3(PO_4)_2 \cdot 2,5\ H_2O$ (where M are Ga, Al, Fe) having a framework structure with quite large caves which consist of eight-membered rings, can be used as a high selective adsorbents I .

METHOD AND MATERIALS.

As subject of present investigations there were monocrystals $FeH_3(PO_4)_2 \cdot 2,5\ H_2O$ obtained, at hydrathermal crystallization in Fe_2O_3-P_2O_5-H_2O system at 120°C as transparent colourless well shaped hexagonal prisms. Preliminary hexagonal cells constans were obtained by photomethod and then refined at automated diffractometer "Syntex $P2_1$" (MoK_α - radiation, graphite monochromator The final parameters are:

a=9,078 Å, c=16,773 Å.

Thermo-X-ray investigations have been carried out invacuum (UTDD-2000, $1 \cdot 10^{-4}$mm, DRON-2,0) at heating from 20° to 1000°C.

RESULTS AND DISCUSSION

The analysis of diffractograms obtained (table 1) shows that in vacuum at room temperature the intensity of some lines decrease while lines fully completeling disappear. This intensity decrease also accomplished decrease elementary cell constans

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Table 1.

X-ray powder data for $FeH_3(PO_4)_2 \cdot 2{,}5\ H_2O$ in different conditions.

	I		II		III		IV	
hKL	J	d(Å)	J	d	J	d	J	d
002	10	8.393						
100	18	7.900			15	7,803		
101	100	7.176	100	7,047	100	7,059	100	7,196
102	45	5,776	35	5,644	40	5,703		
110	50	4.543	45	4,497	40	4,529		
111	24	4.353	20	4,291	35	4,320	20	4,366
112	80	4.026						
200	16	3.945					15	3,969
104	12	3.717						
202	25	3.581	15	3,562	18	3,542	30	3,596
113	15	3.417						
203	45	3.232	35	3,187	30	3,216	15	3,262
114(105)	90	3.094	45	3,058	60	3,064	15	3,153
210	8	2.992			10	2,978		
211	20	2.946			10	2,931		
204	30	2.879			20	2,866		
006(212)	30	2.809	30	2,780	35	2.797		
106	12	2.650			15	2.623	10	2.667
301	15	2.601	15	2.565				
205	5	2.550						
214	10	2.442						
116(007)	6	2.392						
303	5	2.376					15	2.386
206(107)	8	2.284			10	2.265		
215	6	2.243	5	2.215	10	2.213		
310	6	2.178			5	2.151		
262(117)	5	2.116						
008	6	2.094					20	2.107
216	12	2.043						
108	8	2.014						
224	8	1.998						
401	6	1.946						
118	5	1.895						
225	8	1.875						
403	5	1.859						
820	10	1.806						
404	5	1.778						
307	15	1.769						

I - initial, II - vacuum $1 \cdot 10^{-4}$(20°) in during 2h.

III - after disturbance of vacuum the sample is under air at 20 h.

IV - in vacuum at 260°C.

(20°C under vacuum). The cell constant decreasing appears corresponds to remove process of weak bonded water molecules in eight-membered space caves. The repeated diffractogram obtained without vacuum in during 20 hours unaffected that indicates on the reversible nature of the process.

Table 2.

Cell constants	I	II	III	IV
a (Å)	9,08	9,01	9,06	9,17
b (Å)	16,77	16,69	16,76	16,86

In spite of considerable increase of the cell constants at the further heating the product conserves its chemical individuality up to 400°C, after that there is observed the remove process of the strong bonded hydroxil groups which are coordinated to phosphor-oxygen tetrahedrons, in accompaniment of the amorphization process.

The further heating up to 1000°C do not influences to the nature of the product and do not changes amorphity.

CONCLUSIONS

1. It is established that under vacuum the cell constants are unreversably decreased, the crystal structure is unaffected.
2. The cell constants of the thermal decomposition products of $FeH_3(PO_4)_2 \cdot 2,5\ H_2O$ had been determined.

REFERENCES

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