

## Identification and Characterization of Alkaline Uranyl(2+) Phosphates

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Received March 29, 1982

The spontaneous precipitation in the systems  $\text{UO}_2(\text{NO}_3)_2 - \text{MOH} - \text{H}_3\text{PO}_4 - \text{H}_2\text{O}$  and  $\text{UO}_2(\text{NO}_3)_2 - \text{MNO}_3 - \text{H}_3\text{PO}_4 - \text{H}_2\text{O}$  ( $\text{M} = \text{Li, Na, K, Rb, Cs}$ ) is examined. The formation of alkaline-uranyl(2+) phosphates polyhydrates is detected:  $\text{M}[\text{UO}_2\text{PO}_4] \cdot n \text{H}_2\text{O}$  ( $n = 4$  for  $\text{M} = \text{Li}$ ,  $n = 3$  for  $\text{M} = \text{Na, K, Rb}$  and  $n = 2.5$  for  $\text{M} = \text{Cs}$ ).

The X-ray powder patterns of these compounds are determined and compared with that of  $\text{H}_3\text{O}[\text{UO}_2\text{PO}_4] \cdot 3 \text{H}_2\text{O}$ . A close structural relation within this series is observed. The size of the alkaline ionic species in the particular compound affects the content of crystalline water in the unit cell.

### INTRODUCTION

In the precipitation system  $\text{UO}_2(\text{NO}_3)_2 - \text{H}_3\text{PO}_4 - \text{H}_2\text{O}$  the stable solid phases  $\text{HUO}_2\text{PO}_4 \cdot 4 \text{H}_2\text{O}^1$  (tetragonal polymorph type I according to Moroz et al.<sup>2</sup>) and  $(\text{UO}_2)_3(\text{PO}_4)_2 \cdot 8 \text{H}_2\text{O}^1$  were found.  $\text{K}[\text{UO}_2\text{PO}_4] \cdot 3 \text{H}_2\text{O}$  precipitates in the system  $\text{UO}_2(\text{NO}_3)_2 - \text{KOH} - \text{H}_3\text{PO}_4 - \text{H}_2\text{O}$  at high concentrations of  $\text{KOH}^3$ . The spontaneous precipitation and identification of other alkaline uranyl(2+) phosphates has not been systematically examined. The formation of compounds with general formula  $\text{M}[\text{UO}_2\text{PO}_4] \cdot n \text{H}_2\text{O}$  ( $\text{M} = \text{Li, Na, K, Rb, Cs}$ ) was described<sup>4-6</sup>. The number of crystalline water molecules for the particular compound has not been uniquely defined in the literature<sup>7-14</sup>.

The formation of alkaline-uranyl(2+) phosphate hydrates is described in this paper; compounds are identified by chemical and TG analyses, and X-ray powder patterns.

### EXPERIMENTAL

Stock solutions were prepared by dissolving the following p.a. chemicals in triply distilled water: uranyl(2+)nitrate hexahydrate, phosphoric acid, alkaline hydroxides and alkaline nitrates (Merck, Darmstadt). Standardization of solutions was performed according to classical analytical methods.

The precipitation systems were prepared by the mixing<sup>15</sup> of uranyl(2+)nitrate solution with an equal volume of alkaline-phosphate solution (the mixture of  $\text{H}_3\text{PO}_4$  with  $\text{MOH}$  or  $\text{MNO}_3$ ) and aged 1 day at a temperature of 298 K, pH was measured with the electrode GK 2302 C, using a Radiometer Mo 26 pH-meter. The morphology of the precipitates was examined in white, polarized and UV light under an Ortho-

plan microscope (Leitz, Wetzlar) and by electron microscopy (Elmiskop I, Siemens). Turbidity measurements were performed on the Zeiss tyndallometer in connection with Pulfrich photometer.

For the chosen systems the initial concentrations of reactants were:  $[\text{UO}_2(\text{NO}_3)_2] = 1 \cdot 10^{-2} \text{ mol dm}^{-3}$ ,  $[\text{H}_3\text{PO}_4] = 8 \cdot 10^{-3} \text{ mol dm}^{-3}$ ; the solution of alkaline nitrate was added to the phosphoric acid solution. The concentrations of alkaline nitrates were:  $[\text{LiNO}_3] = 3.0 \text{ mol dm}^{-3}$ ;  $[\text{NaNO}_3] = 1.4 \text{ mol dm}^{-3}$ ;  $[\text{MNO}_3] = 0.2 \text{ mol dm}^{-3}$  ( $M = \text{K}, \text{Rb}, \text{Cs}$ ).

After the mixing, the precipitation systems were kept in a water bath at 298 K for 24 hours. The precipitates were separated, washed with triply distilled water, and dried at 293 K for 24 hours. Selected precipitates were characterized by means of chemical and thermogravimetric analyses, X-ray powder diffraction patterns and IR spectra. After dissolution in nitric acid solid phase was chemically analysed for uranium, phosphorus and alkaline metal. Uranium was precipitated with  $(\text{NH}_4)_2\text{HPO}_4$ , heated at 1373 K and weighed as  $\text{U}_2\text{O}_5\text{P}_2\text{O}_7$ <sup>16</sup>. Phosphorus was determined gravimetrically by precipitation with ammonium molybdate<sup>17</sup> and spectrophotometrically as a phosphovanadatomolybdate complex<sup>17</sup>. Alkaline metals were determined by atomic absorption spectroscopy (Li, Rb), flame photometry (Na, K) and gravimetrically as chloroplatinates (Rb, Cs)<sup>18</sup>. Water was determined by thermogravimetric analysis (Cahn RG recording electromicrobalance, sensitivity  $10^{-6} \text{ g}$ , heating rate 4 K in minute, heating to 1050 K).

X-ray diffraction patterns were recorded on a Philips X-ray diffractometer with proportional counter, using graphite monochromated  $\text{CuK}\alpha$  radiation. X-ray patterns were calibrated with an internal standard, graphite, with a unit cell  $a = 2.463 \text{ \AA}$ ,  $c = 6.714 \text{ \AA}$  ( $\lambda = 1.54178 \text{ \AA}$ )<sup>19</sup>. Relative intensities,  $I_{\text{rel}}$ , are given as a peak height. IR spectra were carried out on Mo-221 spectrophotometer (Perkin-Elmer) by the standard KBr platelets technique.

## RESULTS

The precipitation of alkaline-uranyl(2+) phosphates at  $1 \cdot 10^{-3} < [\text{UO}_2(\text{NO}_3)_2] = [\text{H}_3\text{PO}_4] < 1 \cdot 10^{-2} \text{ mol dm}^{-3}$  and  $1 \cdot 10^{-5} < [\text{MOH}] < 1 \cdot 10^{-1} \text{ mol dm}^{-3}$  was examined. Figure 1 shows dependence of turbidity and pH ( $[\text{UO}_2(\text{NO}_3)_2] = [\text{H}_3\text{PO}_4] = 1 \cdot 10^{-3} \text{ mol dm}^{-3}$ ) on the concentration of MOH; the presented curves for all alkaline hydroxides are of the same shape. In the region of low MOH concentration precipitates  $(\text{UO}_2)_3(\text{PO}_4)_2 \cdot 8 \text{ H}_2\text{O}$  which does not exhibit fluorescence, pH keeps the constant value. At higher concentrations of CsOH, RbOH and KOH (different values for particular hydroxide — Figure 1) the fluorescent precipitates of  $M[\text{UO}_2\text{PO}_4] \cdot n \text{ H}_2\text{O}$  ( $M = \text{Cs}, \text{Rb}, \text{K}$ ) are formed in the very narrow range. Increasing the concentration of MOH successively, a significant increase of pH and the formation of stable colloids were observed. Similar properties of particles, morphology and fluorescence, prevent an identification of pure solid phase of  $M[\text{UO}_2\text{PO}_4] \cdot n \text{ H}_2\text{O}$  or its mixture with  $\text{HUO}_2\text{PO}_4 \cdot 4 \text{ H}_2\text{O}$ . Uranates precipitate in all systems at  $\text{pH} > 9.5$ . In the concentration range with stable colloidal particles the turbidity does not exhibit a significant dependence on time.

It is obvious that  $\text{UO}_2(\text{NO}_3)_2 - \text{H}_3\text{PO}_4 - \text{MOH} - \text{H}_2\text{O}$  system was not suitable for the isolation of alkaline-uranyl(2+) phosphates. However, the system with  $[\text{UO}_2(\text{NO}_3)_2] = [\text{H}_3\text{PO}_4]$  and with  $\text{MNO}_3$  (instead of MOH) is suitable, giving crystalline precipitates. At low concentrations of  $\text{MNO}_3$  (and MOH — Figure 1)

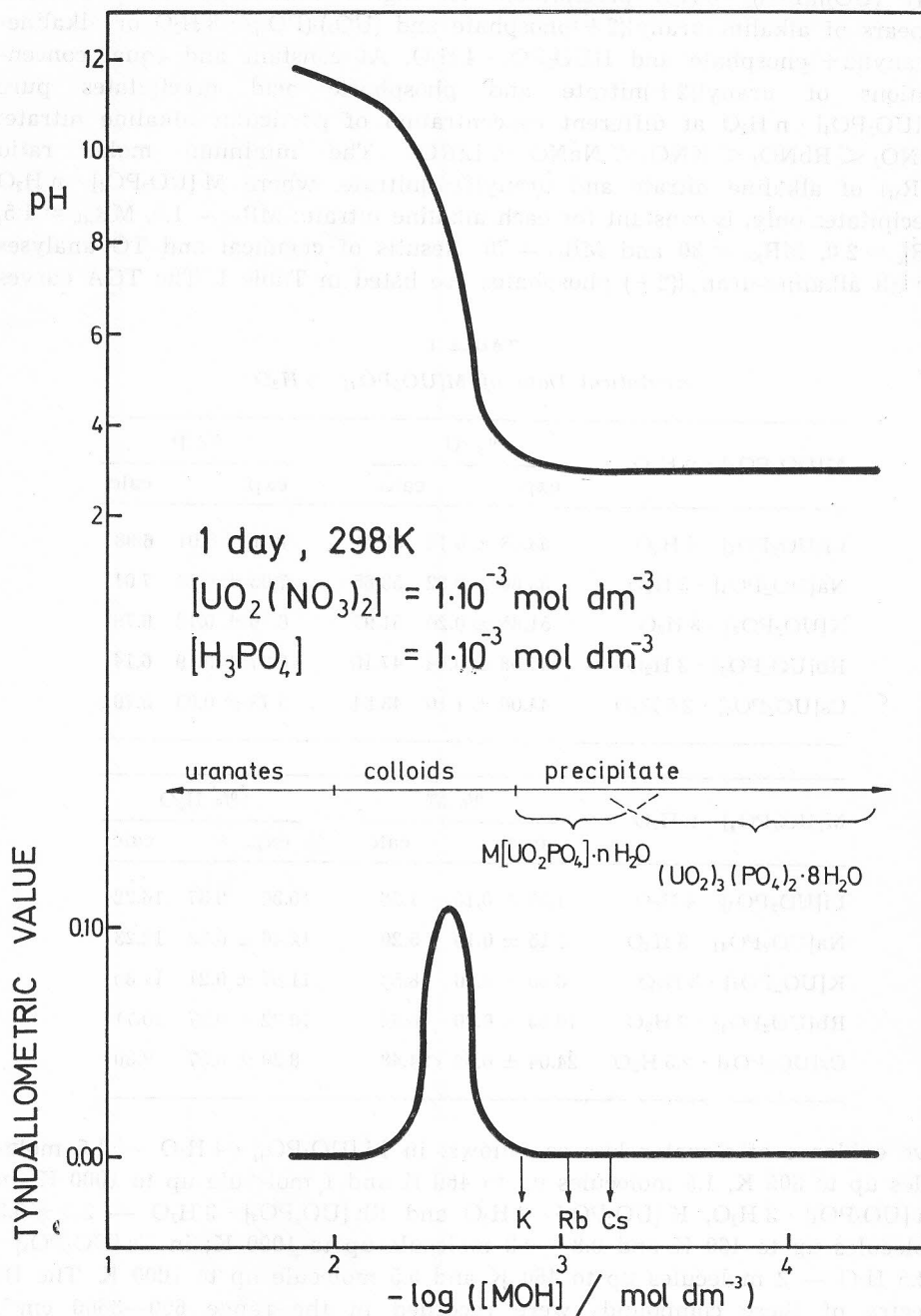


Figure 1. Turbidity and pH versus  $[MOH]$  ( $M = Li, Na, K, Rb, Cs$ ). Arrows show concentrations of CsOH, RbOH and KOH where precipitate their alkaline-uranyl(2+)phosphate.

only  $(\text{UO}_2)_3(\text{PO}_4)_2 \cdot 8 \text{H}_2\text{O}$  precipitates. At higher concentrations, a mixture appears of alkaline-uranyl(2+)phosphate and  $(\text{UO}_2)_3(\text{PO}_4)_2 \cdot 8 \text{H}_2\text{O}$  or alkaline-uranyl(2+)phosphate and  $\text{HUO}_2\text{PO}_4 \cdot 4 \text{H}_2\text{O}$ . At constant and equal concentrations of uranyl(2+)nitrate and phosphoric acid precipitates pure  $\text{M}[\text{UO}_2\text{PO}_4] \cdot n \text{H}_2\text{O}$  at different concentration of particular alkaline nitrate:  $\text{CsNO}_3 < \text{RbNO}_3 < \text{KNO}_3 < \text{NaNO}_3 \ll \text{LiNO}_3$ . The minimum molar ratio ( $\text{MR}_M$ ) of alkaline nitrate and uranyl(2+)nitrate, where  $\text{M}[\text{UO}_2\text{PO}_4] \cdot n \text{H}_2\text{O}$  precipitates only, is constant for each alkaline nitrate:  $\text{MR}_{\text{Cs}} = 1.0$ ,  $\text{MR}_{\text{Rb}} = 1.5$ ,  $\text{MR}_{\text{K}} = 2.0$ ,  $\text{MR}_{\text{Na}} = 30$  and  $\text{MR}_{\text{Li}} = 70$ . Results of chemical and TG analyses for all alkaline-uranyl(2+) phosphates are listed in Table I. The TGA curves

TABLE I  
Analytical Data of  $\text{M}[\text{UO}_2\text{PO}_4] \cdot n \text{H}_2\text{O}$

$\text{M}[\text{UO}_2\text{PO}_4] \cdot n \text{H}_2\text{O}$	% U		% P	
	exp.	calc.	exp.	calc.
$\text{Li}[\text{UO}_2\text{PO}_4] \cdot 4 \text{H}_2\text{O}$	$53.83 \pm 0.13$	53.61	$7.01 \pm 0.01$	6.98
$\text{Na}[\text{UO}_2\text{PO}_4] \cdot 3 \text{H}_2\text{O}$	$53.60 \pm 0.32$	53.85	$7.05 \pm 0.11$	7.01
$\text{K}[\text{UO}_2\text{PO}_4] \cdot 3 \text{H}_2\text{O}$	$51.85 \pm 0.20$	51.95	$6.76 \pm 0.12$	6.76
$\text{Rb}[\text{UO}_2\text{PO}_4] \cdot 3 \text{H}_2\text{O}$	$47.08 \pm 0.44$	47.18	$6.27 \pm 0.19$	6.14
$\text{Cs}[\text{UO}_2\text{PO}_4] \cdot 2.5 \text{H}_2\text{O}$	$44.00 \pm 1.10$	43.84	$5.73 \pm 0.03$	5.70

$\text{M}[\text{UO}_2\text{PO}_4] \cdot n \text{H}_2\text{O}$	% M		% $\text{H}_2\text{O}$	
	exp.	calc.	exp.	calc.
$\text{Li}[\text{UO}_2\text{PO}_4] \cdot 4 \text{H}_2\text{O}$	$1.55 \pm 0.10$	1.56	$16.36 \pm 0.37$	16.22
$\text{Na}[\text{UO}_2\text{PO}_4] \cdot 3 \text{H}_2\text{O}$	$5.15 \pm 0.10$	5.20	$12.46 \pm 0.33$	12.23
$\text{K}[\text{UO}_2\text{PO}_4] \cdot 3 \text{H}_2\text{O}$	$8.50 \pm 0.10$	8.53	$11.67 \pm 0.21$	11.80
$\text{Rb}[\text{UO}_2\text{PO}_4] \cdot 3 \text{H}_2\text{O}$	$16.65 \pm 0.20$	16.94	$10.82 \pm 0.27$	10.71
$\text{Cs}[\text{UO}_2\text{PO}_4] \cdot 2.5 \text{H}_2\text{O}$	$24.04 \pm 0.62$	24.48	$8.29 \pm 0.57$	8.30

give evidence of a water loss as follows: in  $\text{Li}[\text{UO}_2\text{PO}_4] \cdot 4 \text{H}_2\text{O}$  — 1.5 molecules up to 305 K, 1.5 molecules up to 460 K and 1 molecule up to 1000 K; in  $\text{Na}[\text{UO}_2\text{PO}_4] \cdot 3 \text{H}_2\text{O}$ ,  $\text{K}[\text{UO}_2\text{PO}_4] \cdot 3 \text{H}_2\text{O}$  and  $\text{Rb}[\text{UO}_2\text{PO}_4] \cdot 3 \text{H}_2\text{O}$  —  $2.2 \pm 0.3$  molecules up to 460 K and  $0.8 \pm 0.3$  molecule up to 1000 K; in  $\text{Cs}[\text{UO}_2\text{PO}_4] \cdot 2.5 \text{H}_2\text{O}$  — 2 molecules up to 460 K and 0.5 molecule up to 1000 K. The IR spectra of these compounds were recorded in the range 600–3600  $\text{cm}^{-1}$ , characteristic vibrations of uranyl(2+) and phosphate groups are in accord with literature<sup>13</sup>. The X-ray powder patterns are separated in two groups: A (Table II) and B (Table III).

TABLE II  
X-ray Powder Patterns for Compounds of Group A.

$\text{H}_3\text{O}[\text{UO}_2\text{PO}_4] \cdot 3 \text{H}_2\text{O}$		$\text{Na}[\text{UO}_2\text{PO}_4] \cdot 3 \text{H}_2\text{O}$		$\text{K}[\text{UO}_2\text{PO}_4] \cdot 3 \text{H}_2\text{O}$		$\text{Cs}[\text{UO}_2\text{PO}_4] \cdot 2.5 \text{H}_2\text{O}$	
$d/\text{\AA}$	$I_{\text{rel}}$	$d/\text{\AA}$	$I_{\text{rel}}$	$d/\text{\AA}$	$I_{\text{rel}}$	$d/\text{\AA}$	$I_{\text{rel}}$
8.76	100	8.85	100	8.95	100	8.90	100
5.47	26	5.48	44	5.52	20	5.55	26
4.95	18	4.95	36	4.95	18	4.99	20
		4.46	18	4.46	25	4.82	4
4.37	15	4.39	23			4.45	25
4.31	18	4.33	42	4.33	18	4.36	27
						3.98	8
3.71	59	3.72	80	3.77	55	3.76	100
3.51	24	3.51	50	3.51	26	3.53	32
3.25	30	3.26	55	3.26	28	3.27	53
				3.15	5	3.16	21
3.09	2	3.08	3	3.09	4	3.12	5
2.95	15	2.94	30	2.95	15	2.98	20
						2.76	9
2.73	3	2.73	9	2.74	20	2.73	17
2.69	20	2.69	39				
2.55	8	2.54	20	2.56	8	2.57	5
2.51	3	2.52	6			2.54	5
2.48	6	2.47	14	2.48	7	2.48	5
2.38	9	2.37	18	2.39	9	2.40	9
2.25	4	2.26	12	2.26	6	2.27	9
				2.23	15	2.23	11
2.21	10	2.21	22	2.21	11	2.21	21
2.19	10	2.19	12				
2.15	10			2.16	14	2.16	25
2.13	12			2.13	10	2.11	11
2.09	7	2.09	12				
2.06	8	2.06	17	2.07	9	2.07	9
2.01	8	2.00	14	2.03	8	2.02	8
1.896	3	1.895	8	1.896	5	1.900	3
1.856	3	1.859	6			1.871	3
1.823	6	1.822	11	1.837	6	1.834	7
1.794	3	1.795	8				
				1.782	10	1.785	7
1.775	7	1.775	14				
1.752	7	1.749	10	1.752	5	1.760	7
1.717	3	1.715	8	1.717	4	1.720	5
1.676	2	1.665	4	1.679	11		
1.651	10	1.652	13			1.665	8
1.646	6						
				1.627	6	1.630	6
1.617	6	1.615	13				
1.596	2	1.600	4			1.595	5
1.584	3	1.582	7	1.589	8	1.577	10
1.566	8	1.564	13	1.568	6	1.570	8
1.562	3						
1.556	6						
1.542	3						
1.468	2	1.465	6			1.476	5
		1.450	3			1.444	4

TABLE III  
X-ray Powder Patterns for Compounds of Group B.

Li[ $\text{UO}_2\text{PO}_4$ ] · 4 H <sub>2</sub> O		Rb[ $\text{UO}_2\text{PO}_4$ ] · 3 H <sub>2</sub> O	
d/Å	I <sub>rel</sub>	d/Å	I <sub>rel</sub>
9.25	100	9.25	100
5.61	13	5.58	15
5.00	8	4.96	6
4.58	83	4.60	60
4.40	12	4.36	13
3.83	83	3.84	90
3.53	12	3.50	12
3.29	17	3.26	15
3.11	1		
3.04	19		
2.98	10	2.95	8
2.80	31	2.80	60
2.59	7	2.59	6
		2.57	8
2.50	1	2.47	3
2.41	7		
		2.38	7
2.28	92	2.30	38
2.23	7	2.26	1
		2.20	7
2.18	12	2.18	37
2.17	22	2.15	7
2.09	7	2.08	23
2.02	1		
1.910	3	1.911	6
		1.890	1
1.860	3		
1.842	5	1.845	12
1.821	19	1.831	18
1.794	5	1.780	2
1.760	5		
1.730	1	1.741	1
1.710	14	1.715	26
1.705	14		
1.643	2		
1.635	2	1.630	7
1.617	3	1.620	13
1.613	2		
1.591	2	1.588	8
1.575	1		
1.516	7	1.525	6
1.481	8	1.490	6
1.470	4		
1.458	12		

## DISCUSSION

A number of crystalline water molecules established for Li and Na compounds are in agreement with the literature<sup>7-11</sup>. K compound is defined with 2, 3 and 3.5 molecules of water. The formation of various hydrates can be caused by different conditions under which the samples were kept. Our analytical data correspond to three water molecules<sup>3,12,9</sup>. The appearance of Rb compound monohydrate is described by Muraveva et al.<sup>9</sup>, whereas our analytical data correspond to trihydrate. However, dehydration of Na, K,

Rb and Cs uranyl(2+) phosphate hydrates starts at room temperature, these low hydrates were detected by TG and chemical analyses.

The X-ray powder patterns of the series with general formula  $M [UO_2PO_4] \cdot n H_2O$  ( $M = H_3O, Li, Na, K, Rb$  and  $Cs$ ) are compared. They can be classified in two groups; the patterns of  $H_3O, Na, K,$  and  $Cs$  compounds form one group (Table II group — A) whereas those of  $Li$  and  $Rb$  form another group (Table III — group B).

The X-ray powder pattern of tetragonal polymorph of  $HUO_2PO_4 \cdot 4 H_2O$  with space group  $P 4/n c c$  (type I according to Moroz et al.<sup>2</sup>) was previously reported<sup>1</sup>. The X-ray structure analysis of this polymorph<sup>20</sup> revealed its correct structural formula —  $H_3O [UO_2PO_4] \cdot 3 H_2O$ . Its X-ray powder pattern is similar to those of alkaline-uranyl(2+) phosphates with an analogous formula;  $d$  — values and relative intensities are listed in Table II for comparison. The crystal structure of  $H_3O [UO_2PO_4] \cdot 3 H_2O$  explains by itself relations between the X-ray powder patterns of these compounds.  $UO_2^{2+}$  is coordinated with four oxygen atoms from four  $PO_4$  tetrahedra. None of the four water molecules is involved in coordination; they form a continuously hydrogen bonded network containing  $H_3O^+$  species<sup>2,21</sup>. Thus the structure is composed of  $[UO_2PO_4]^-$  and water layers containing  $H_3O^+$ . In the network of four water molecules there is enough space to accommodate the  $Li$  ion ( $r_i = 0.68 \text{ \AA}$ )<sup>22</sup>. Thus lithium uranyl(2+)phosphate crystallized as a tetrahydrate. In the holes of this water layer there is no space available for  $Na$  ( $r_i = 0.97 \text{ \AA}$ )<sup>22</sup> and  $K$  ( $r_i = 1.33 \text{ \AA}$ )<sup>22</sup>. However, three molecules of crystalline water leave enough space to accommodate these ions. In the lattice of the  $Cs$  compound ( $r_i = 1.67 \text{ \AA}$ )<sup>22</sup> the water content is reduced to 2.5 molecules. The unit cell is slightly decreased. Therefore, the  $d$  — values of the  $Cs$  compound are lower than those of  $Na$  and  $K$  compounds (Table II). The  $Rb$  compound ( $r_i = 1.47 \text{ \AA}$ )<sup>22</sup> with three crystalline water molecules exhibits the X-ray powder pattern which is very close to one of the  $Li$  compound with four water molecules (Table III).

The octahedral coordination around uranium is maintained in all the structures but the size of the alkaline ionic species in particular compound affects the content of crystalline water in the unit cell. An accommodation of the particular ion in the water layer has been already explained. These structural relations give an explanation for similar X-ray powder patterns of all the mentioned compounds in both groups, A and B (Tables II and III);  $d$  — values show a better agreement within each group.

*Acknowledgement.* — The authors wish to thank Professor M. Branica for valuable suggestions at the initial stage of this work.

Support of this research by the Self-Management Council for Scientific Research of S. R. Croatia is gratefully acknowledged.

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## SAŽETAK

### Identifikacija i karakterizacija alkalijskih uranil(2+) fosfata

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Spontani taložni procesi ispitani su u sistemima  $\text{UO}_2(\text{NO}_3)_2\text{—MOH—H}_3\text{PO}_4\text{—H}_2\text{O}$  i  $\text{UO}_2(\text{NO}_3)_2\text{—H}_3\text{PO}_4\text{—MNO}_3\text{—H}_2\text{O}$  ( $M = \text{Li, Na, K, Rb, Cs}$ ). Utvrđeno je nastajanje alkalijskih uranil(2+) fosfata polihidrata, opće formule  $M[\text{UO}_2\text{PO}_4] \cdot n\text{H}_2\text{O}$  ( $n = 4$  za  $M = \text{Li}$ ,  $n = 3$  za  $M = \text{Na, K, Rb}$  i  $n = 2.5$  za  $M = \text{Cs}$ ).

Navedeni su međuplošni razmaci i intenziteti svih alkalijskih uranil(2+) fosfata dobiveni rendgenskom difrakcijom na praškastim uzorcima, te su uspoređeni s pripadnim vrijednostima dobivenim za  $\text{H}_3\text{O}[\text{UO}_2\text{PO}_4] \cdot 3\text{H}_2\text{O}$ . Utvrđena je izrazita sličnost između njihovih struktura. Ovisno o veličinama iona različitih alkalijskih metala mijenja se broj kristalnih voda u jediničnoj ćeliji pojedinog spoja.