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Cesium and Its Analogs, Rubidium and Potassium, in Rhombohedral [$NaZr_2(PO_4)_3$ Type] and Cubic (Langbeinite Type) Phosphates: 2. Properties: Behavior on Heating, in Aqueous Solutions, and in Salt Melts

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Abstract—The properties (behavior on heating, in aqueous solutions, and in salt melts) of orthophosphates $A_2RM(PO_4)_3$, $A_2B_{0.5}Zr_{1.5}(PO_4)_3$, and $ABR_2(PO_4)_3$ [A = K, Rb, Cs; B = Mg, Sr, Ba; R = Ga, Fe, Cr, Ln (Ce–Lu)] crystallizing in the structure of langbeinite mineral (cubic system, space group $P2_13$, $Z = 4$) were studied and compared with those of NZP-type phosphates. The thermal transformations of the structure and the influence of temperature on the distortion of the framework-forming polyhedra were examined. The volatilization of cesium, in particular, from the solid phase in the course of its formation, was evaluated. The rates of cesium and barium leaching at 90 and 95°C were determined.

Diversified isomorphism in rhombohedral {of $NaZr_2(PO_4)_3$ type [1]} and cubic (of langbeinite type [2]) phosphates is characteristic of many phosphate minerals. It provides a basis for substantiated formulation of single-phase ceramic materials for various purposes, including matrix materials for concentrating wastes from nuclear and other industries, with the aim to firmly isolate these wastes from the environment.

The successful application of such materials depends on their properties that determine the safety of long-term storage: resistance to heat, high pressure, and radiation; behavior in water, aqueous solutions, and in the presence of other chemical agents.

Our goal was to study the thermal behavior of new cesium, rubidium, and potassium phosphates described in [2], to evaluate their stability under hydrothermal conditions and in alkali metal chloride melts, and to summarize the relevant data concerning NZP- and langbeinite-type phosphates.

The properties of cesium, rubidium, and potassium phosphates of the rhombohedral NZP-type modification are better studied compared to those of the cubic langbeinite phosphates.

NZP-type phosphates of cesium and other alkali metals of the formula $AM_2(PO_4)_3$ are readily formed by precipitation from aqueous solutions followed by thermal treatment of the precipitates, from molten salts (alkali metal chlorides or fluorides) on adding appropriate precipitates, by solid-phase synthesis, and in ion-exchange processes [3–6]. Ceramic samples based on cesium phosphates were prepared [7]. The phosphates $KZr_2(PO_4)_3$, $RbZr_2(PO_4)_3$, $CsZr_2(PO_4)_3$, and their titanium and hafnium analogs exist in a wide temperature range and retain the chemical and phase composition when heated to 1200°C. Their thermal expansion is negligible. The mean coefficients of thermal expansion of $KZr_2(PO_4)_3$, $RbZr_2(PO_4)_3$, $CsZr_2 \cdot (PO_4)_3$, $KHf_2(PO_4)_3$, $RbHf_2(PO_4)_3$, and $CsHf_2(PO_4)_3$ are close to zero, and the coefficients along particular axes are as follows: $\alpha_a = (-7.3\text{–}0.0) \times 10^{-6}$ and $\alpha_c = (1.0\text{–}22.3) \times 10^{-6}$ deg^{-1} [8].

Irradiation of $CsTi_2(PO_4)_3$ and $CsZr_2(PO_4)_3$ with a ^{60}Co γ-ray source at a dose rate D of up to 10 Gy s^{-1} to doses of 1×10^6–5×10^8 Gy caused no changes in the phase and chemical compositions, as judged by the IR and X-ray diffraction data [9, 10]. Cesium zirconium phosphate underwent no noticeable changes under bombardment with accelerated Ar nuclei [11].

Rhombohedral CsTi, CsZr, and CsHf phosphates

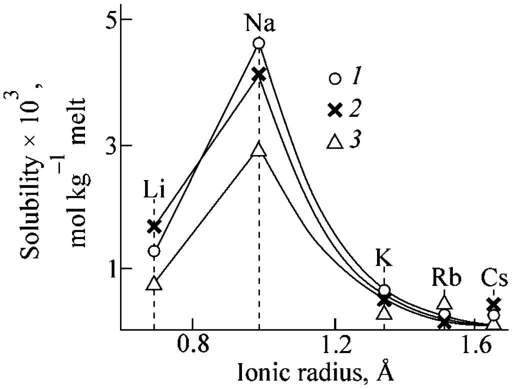

Fig. 1. Solubility of $AM_2(PO_4)_3$ in ACl melts as a function of the ionic radius of A (Li–Cs): (*1*) $ATi_2(PO_4)_3$, (*2*) $AZr_2 \cdot (PO_4)_3$, and (*3*) $AHf_2(PO_4)_3$.

were also subjected to hydrothermal tests, which were performed in the dynamic and static modes at room temperature and at 95 and 200°C in distilled water and, in some experiments, in seawater.

In some experiments, the testing time reached two years (at $T = 200°C$ and pressure of up to 600 atm). The chemical degradation was never observed; the minimal attained rates of cesium leaching did not exceed 1×10^{-5} g cm^{-2} day^{-1}.

Preliminary irradiation (absorbed dose 3×10^8 Gy) had virtually no effect on the leaching rates [12].

The phosphate $KZr_2(PO_4)_3$ is difficultly soluble in water and dilute mineral acids ($HClO_4$, HCl, HNO_3, H_2SO_4). Its solubility (equilibration time 120–150 h) depends on pH and anionic composition of the solution. As the pH is decreased from 6.5 to 0.3, the solubility product (molar scale) changes from 2×10^{-26} to 7×10^{-22} in $HClO_4$, from 2×10^{-23} to 1×10^{-21} in HCl, from 7×10^{-22} to 4×10^{-20} in HNO_3, and from 1×10^{-21} to 10×10^{-19} in H_2SO_4. The effect of the anion on the solubility is due to zirconium complexation and correlates with the complexing power of the anion, decreasing in the order $H_2SO_4 > HNO_3 > HCl > HClO_4$. These data were obtained using ^{95}Zr and ^{32}P tracers introduced into $KZr_2(PO_4)_3$ in the course of its synthesis.

The solubility of the phosphates $AM_2(PO_4)_3$, where A = K, Rb, Cs and M = Ti, Zr, Hf, was studied in molten salts ACl: KCl at 800–850°C and CsCl at 650–750°C. It depended on particular cations A and M, and also on temperature (Fig. 1). Data for A = Li and Na are also shown in Fig. 1 for comparison. The equilibration time was 2.5–3 h. The compositions and structures of the initial phases remained unchanged after keeping in molten chlorides ACl at the above-indicated temperatures and in molten nitrates ANO_3 at 300–350°C for 10–12 h [13].

All the above data on the thermal, radiation, and chemical stability of framework phosphates of Cs and other alkali elements {framework unit $[T_2(PO_4)_3]^{n-}$} concern phosphates of the $NaZr_2(PO_4)_3$ structural type. We found no such data on langbeinite-type phosphates.

The experiments made in this study were aimed to evaluate the stability of langbeinite-type phosphates of cesium and other alkali elements. Their synthesis and crystal chemistry were described previously [2].

In addition to the methods described in [2], we used in this study atomic absorption, X-ray fluorescence, atomic emission, and photometric methods. Elemental analysis was performed with a Perkin–Elmer 603 atomic absorption spectrophotometer (solutions to be analyzed were atomized in the acetylene–air flame of a slit burner), an ERA-03 energy-dispersive X-ray fluorescence analyzer (energy resolution ≤300 eV, identification of elements with *Z* from 21 to 92), an SAE-01 inductively coupled plasma atomic emission spectrometer (wavelength range 240–800 nm), and a KFK-3 photoelectric photometer equipped with a microprocessor, with computer data treatment (wavelength range 400–950 nm, range of optical densities measured from 0 to 3.0).

THERMAL BEHAVIOR

The phosphates with the langbeinite framework motif, prepared and characterized in [2], crystallize at $T = 800$–1000°C. They undergo no chemical degradation or phase transitions when kept at 1000°C for 24 h. Some samples were heat-treated at 1300°C for 24 h. In so doing, $K_2Mg_{0.5}Zr_{1.5}(PO_4)_3$ underwent no phase or chemical transformations, according to X-ray phase analysis. $K_2ErZr(PO_4)_3$ decomposed under the same conditions; its diffraction pattern contained the reflections of zirconium oxide and simple erbium orthophosphate.

To reveal changes occurring in the structure under heat treatment, we examined the phosphate $Cs_2Mg_{0.5} \cdot Zr_{1.5}(PO_4)_3$ [14] by high-temperature neutron diffraction using the method of full-profile analysis (Rietveld method). The measurement temperature was 15, 150, 300, 450, and 600°C. The results are listed in Table 1.

Variation of the selected interatomic distances and bond angles is illustrated by Table 2. The structure (Fig. 2) is formed by discrete MgO_6, ZrO_6, and PO_4 polyhedra combined in the $[Mg_{0.5}Zr_{1.5}(PO_4)_3]$ frame-

Table 1. Results of refinement of the crystal structure of $Cs_2Mg_{0.5}Zr_{1.5}(PO_4)_3$*

Characteristic	15°C	150°C	300°C	450°C	600°C
a, Å	10.2624(5)	10.2657(9)	10.2735(9)	10.2835(9)	10.2886(9)
V, Å^3	1080.804	1081.847	1084.315	1087.484	1089.103
Number of reflections	303	322	315	323	317
R_{exp}	1.69	2.62	2.58	2.56	2.37
R_{wp}	3.75	4.89	4.52	4.45	4.39
R_p	4.17	5.65	5.38	5.21	4.89
χ^2	6.94	5.78	4.02	3.58	5.03

* Cubic system, space group $P2_13$, Z 4, white powder, range of d 0.73–3.53 Å, 39 refined parameters.

work with the random distribution of the magnesium and zirconium cations over two nonequivalent crystallographic positions, and it somewhat deforms on heating.

The distortion of the framework-forming polyhedra, evaluated by the maximal scattering Δ in the bond lengths, varies with temperature. In particular, at 15°C the phosphorus tetrahedra are deformed insignificantly: Δ = 0.05 Å, and the O–P–O bond angles are close to the ideal tetrahedral angle, 109.5°. With increasing temperature, the bond lengths change, and at 600°C Δ in the phosphorus tetrahedron reaches 0.21 Å. The distortion of the MgO_6 and ZrO_6 octahedra depends on temperature to a lesser extent: Δ = 0.23 Å at 15°C and 0.35 Å at 600°C. On heating, the polyhedra turn relative to each other, as shown in Fig. 3.

The temperature dependences of the unit cell pa-

Table 2. Selected interatomic distances (Å) and bond angles (deg) in the structure of $Cs_2Mg_{0.5}Zr_{1.5}(PO_4)_3$ at T = 15 and 600°C

Bond, angle	15°C	600°C	Bond, angle	15°C	600°C
Cs^1O_9 polyhedron			PO_4 tetrahedron		
Cs^1–O^2	3.16	3.18	P–O^1	1.48	1.63
Cs^1–O^3	3.06	3.08	P–O^2	1.45	1.42
Cs^1–O^4	2.83	3.36	P–O^3	1.50	1.48
Cs^2O_9 polyhedron			P–O^4	1.50	1.56
			O^1–P–O^2	113.9	110.3
Cs^2–O^1	3.11	3.11	O^1–P–O^3	107.7	107.0
Cs^2–O^2	3.19	3.11	O^1–P–O^4	115.9	112.2
Cs^2–O^4	2.83	2.79	O^2–P–O^3	107.8	113.7
MO_6 octahedron			O^2–P–O^4	102.8	104.7
			O^3–P–O^4	108.3	109.0
Zr–O^1	1.95	1.89			
Zr–O^2	2.18	2.19			
Mg/Zr–O^3	2.05	1.84			
Mg/Zr–O^4	2.07	2.17			

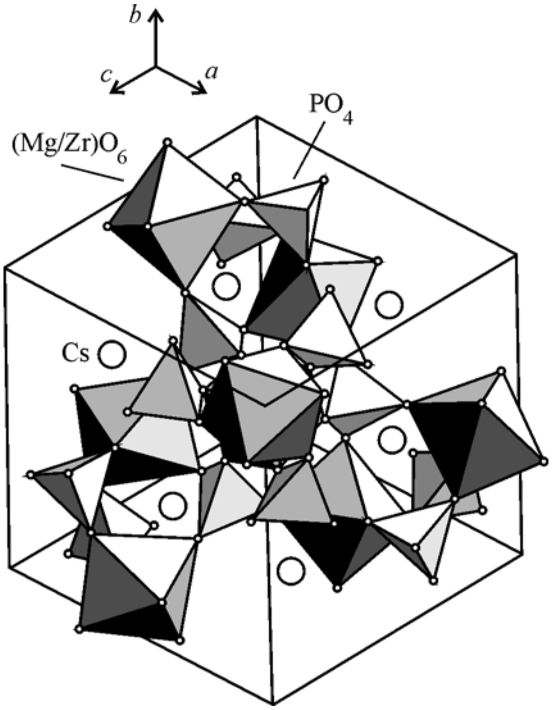

Fig. 2. Fragment of the $Cs_2Mg_{0.5}Zr_{1.5}(PO_4)_3$ structure.

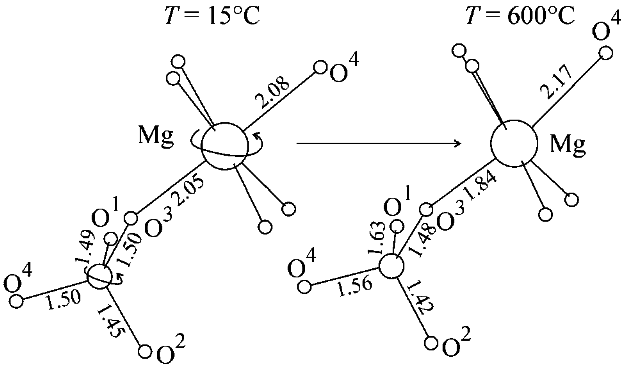

Fig. 3. Distortions of the framework-forming polyhedra on heating.

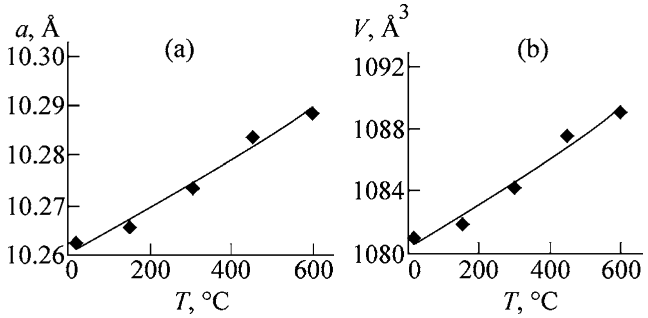

Fig. 4. Temperature dependences of the (a) unit cell parameter a and (b) unit cell volume V. Regression equations: (a) $a = 1 \times 10^{-8}T^2 + 4 \times 10^{-5}T + 10.261$ and (b) $V = 4 \times 10^{-6}T^2 + 1 \times 10^{-2}T + 1080.3$.

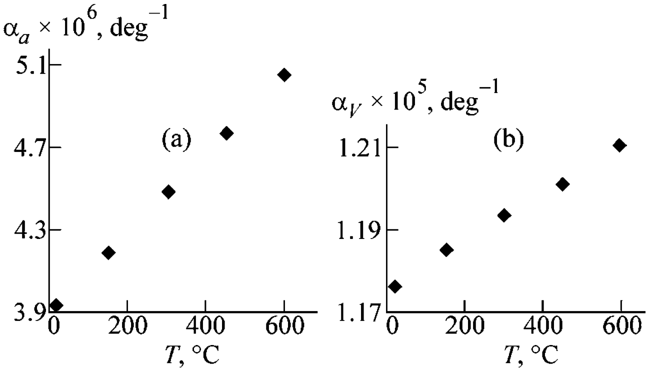

Fig. 5. Temperature dependences of the thermal expansion coefficients (a) α_a and (b) α_V.

rameters a and V (Fig. 4) are described by quadratic polynomials. The polynomial parameters of the thermal expansion were calculated by Eqs. (1) and (2):

$$p = p_2T^2 + p_1T + p_0, \tag{1}$$

$$\alpha = (p_1 + 2p_2T)/p, \tag{2}$$

where α is the thermal expansion coefficient; $\alpha = (1/p)\mathrm{d}p/\mathrm{d}T$.

The thermal expansion coefficients α_a and α_V are given in Table 3.

Table 3. Thermal expansion coefficients of $Cs_2Mg_{0.5}Zr_{1.5} \cdot (PO_4)_3$

T, °C	$\alpha_a \times 10^6$, deg^{-1}	$\alpha_V \times 10^6$, deg^{-1}
15	3.93	11.76
150	4.18	11.85
300	4.48	11.93
450	4.76	11.20
600	5.05	12.10

Figure 5 shows the temperature dependence of the thermal expansion coefficients. As seen, α_a and α_V increase with temperature, reaching at T = 600°C 5.05×10^{-6} and 12.10×10^{-6} deg^{-1}, respectively.

As seen, the langbeinite-type and rhombohedral phosphates differ in the characteristics of thermal expansion. The expansion of the former compounds is stronger (for an NZP-type phosphate $CsZr_2(PO_4)_3$, $\alpha_a \le -1 \times 10^{-6}$ and $\alpha_c \le 1 \times 10^{-6}$ deg^{-1}) and isotropic (the thermal expansion of NZP phosphates is anisotropic, although for the CsZr and CsHf phosphates the anisotropy is close to zero).

Although the chemical composition of the samples does not noticeably change under prolonged heat treatments, volatilization of small amounts of cesium cannot be ruled out. Therefore, we made special experiments to estimate the volatilization of Cs from the phosphates. For this purpose, a sample of $Cs_2Fe \cdot Zr(PO_4)_3$ (final synthesis temperature 800°C) was heated at T = 800 and 1000°C for 2 h in each step, and the cesium content in the gas phase was determined. The volatilization of Cs from the solid phase in the course of its formation was also determined for the phosphate $Cs_2Mg_{0.5}Zr_{1.5}(PO_4)_3$. Simultaneously with cesium, we monitored the volatilization of other components.

The experimental procedure was as follows. A crucible with a powdered sample was placed in a quartz beaker. The gas phase was passed through a thermally insulated quartz tube, a U-shaped air-cooled steel condenser, and a bubbler, using a water-jet pump. The water from the bubbler and the material washed out from the walls of the unit with a dilute acid were analyzed. The analysis was performed after each step of heating: 2 h at 800°C and 2 h at 1000°C for $Cs_2FeZr(PO_4)_3$; 24 h at 80°C, 24 h at 600°C, 24 h at 800°C, and 24 h at 1000°C in the experiment on synthesis of $Cs_2Mg_{0.5}Zr_{1.5}(PO_4)_3$, in accordance with [2].

In all the samples taken, the content of cesium and zirconium was below the detection limit. Therefore, the fraction of Cs and Zr that volatilized from the solid sample into the gas phase can be estimated at ≤0.0005 and ≤0.00003, respectively. The volatilization of Mg and Fe is somewhat larger, compared to the stoichiometric ratio. Apparently, the magnesium and iron compounds formed by thermal degradation of the phosphate sample have higher vapor pressure under the experimental conditions than the zirconium and cesium compounds.

The volatilization of cesium from $Cs_2FeZr(PO_4)_3$ and $Cs_2Mg_{0.5}Zr_{1.5}(PO_4)_3$ is considerably smaller

Table 4. Volatilization of cesium from solid matrices

Matrix	Process	Temperature, °C	Volatilization of Cs, %	References
Aluminophosphate glass	Evaporation of cesium-containing solutions with glass-forming additives	100–120	0.4	[15]
	Glass founding	1100	0.4	[16]
Borosilicate glass	Evaporation of cesium-containing solutions in the presence of silica gel	100–120	0.9	[17]
	Glass founding	800–1000	1–2	[18]
Synroc-C	Calcination	700	0.1	[19]
	Melting of charge from metal oxides and nitrates using IMCC*	1300–1600	3–18	[20]
Phosphate ceramic of langbeinite structure	Synthesis of cesium-containing sample	80–1000	<0.05	This work
	Calcination	800–1000	<0.05	

* Induction melting in a cold crucible.

compared to the known Cs-containing solid matrices of other compositions and structures (Table 4).

On the whole, phosphates of both langbeinite and NZP structures are resistant to high temperatures (up to 1200–1300°C), and the arising thermal deformations are insignificant.

HYDROTHERMAL STUDIES

Hydrothermal tests were performed with $Cs_2Mg_{0.5}\cdot Zr_{1.5}(PO_4)_3$, $CsBaFe_2(PO_4)_3$, $K_2Mg_{0.5}Zr_{1.5}(PO_4)_3$, and $K_2FeZr(PO_4)_3$. The experiments were performed in static [$K_2Mg_{0.5}Zr_{1.5}(PO_4)_3$, $Cs_2Mg_{0.5}Zr_{1.5}(PO_4)_3$] and dynamic [$CsBaFe_2(PO_4)_3$, $K_2FeZr(PO_4)_3$] modes. Samples of phosphates in the form of ceramics were prepared by pressing the powders prepared by the sol–gel method (final synthesis temperature 800°C), followed by annealing at 1000–1100°C for 24 h.

Ceramic discs of $Cs_2Mg_{0.5}Zr_{1.5}(PO_4)_3$ and $K_2\cdot Mg_{0.5}Zr_{1.5}(PO_4)_3$ were placed in Teflon test tubes 80% filled with distilled water; the tube were placed in hermetically sealed steel containers. The experiments were performed in the static mode at $T = 90°C$ for 28 days; samples were taken on the 3rd, 7th, 14th, and 28th days.

Samples of $CsBaFe_2(PO_4)_3$ and $K_2FeZr(PO_4)_3$, prepared as ceramics, were placed in the glass extractor of a Soxhlet apparatus. Tests were performed in the dynamic mode at $T = 90°C$ for 28 days. Samples were taken at regular intervals, and the content of elements was determined.

The leaching rate was calculated by the formula

$$R = m/(c_{sp}S_{surf}\Delta t),$$

where m is the weight (g) of an element released from the sample in time Δt (days); c_{sp}, specific concentration of an element in the solid phase (g g^{-1} solid phase); and s_{surf}, sample surface area (cm^2).

The kinetic curves and the dependences of the leaching rates on time for the phosphates $Cs_2Mg_{0.5}\cdot Zr_{1.5}(PO_4)_3$ and $CsBaFe_2(PO_4)_3$ are shown in Fig. 6. The alkali metal leaching rate was the highest in the first day, after which it rapidly decreased with time.

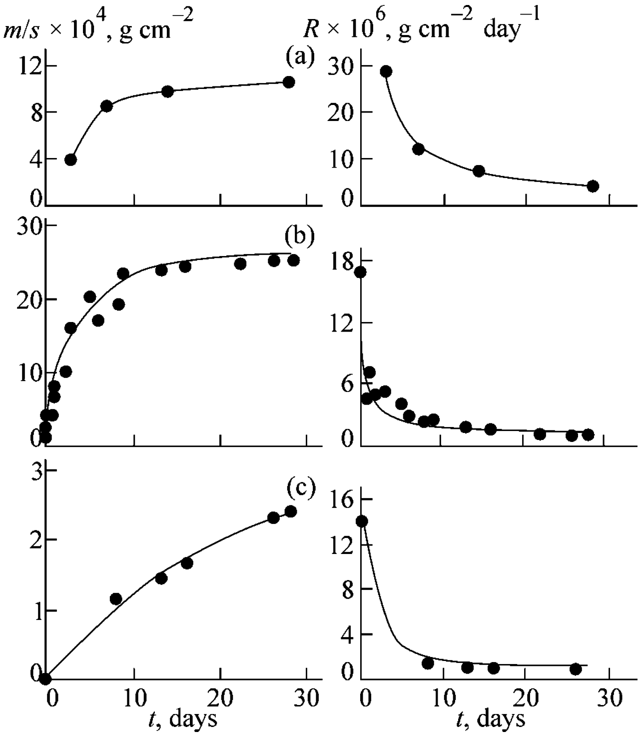

Fig. 6. Kinetic curves of leaching and variation with time of the leaching rates: (a) Cs from $Cs_2Mg_{0.5}Zr_{1.5}(PO_4)_3$ (static mode), (b) Cs from $CsBaFe_2(PO_4)_3$ (dynamic mode), and (c) Ba from $CsBaFe_2(PO_4)_3$ (dynamic mode).

The minimal leaching rate of K from $K_2FeZr(PO_4)_3$ and $K_2Mg_{0.5}Zr_{1.5}(PO_4)_3$ was $(1–2) \times 10^{-6}$ g cm^{-2} day^{-1}; that of Cs from $Cs_2Mg_{0.5}Zr_{1.5}(PO_4)_3$ and $CsBaFe_2 \cdot (PO_4)_3$, $(2–5) \times 10^{-6}$ g cm^{-2} day^{-1}; and that of Ba from $CsBaFe_2(PO_4)_3$, 8×10^{-6} g cm^{-2} day^{-1}.

STABILITY IN MELTS

Tests for stability in alkali metal chloride melts were performed with phosphates crystallizing in the langbeinite structure: $K_2FeZr(PO_4)_3$ and $K_2ErZr \cdot (PO_4)_3$. The experimental procedure was as follows. An intimate mixture of a phosphate sample (~0.5 g) and an alkali metal chloride (molar ratio 1 : 10) was placed in a platinum crucible and heated for several days at 830°C. Then the melt was cooled, and the soluble fraction was washed off with hot water acidified with HNO_3. The insoluble residue was dried at 100°C and analyzed by X-ray diffraction.

The phosphates tested underwent no changes on contact with a KCl melt. The crystallinity of the samples did not change either.

To sum up the results obtained in this study and reported in [2], we can note that, among framework phosphates of cesium, potassium, and rubidium, compounds of the langbeinite structure, along with phosphates of the $NaZr_2(PO_4)_3$ type, deserve attention as matrices for concentrating large alkali metal (primarily cesium) ions. In some parameters, langbeinite-type phosphates are even preferable. In particular, they incorporate more alkali metals: Cs up to 38, Rb up to 28, and K up to 15 wt %. Compounds with the langbeinite structural motif are prepared by relatively simple procedures and are stable thermally and chemically. The structural features of langbeinite and the revealed crystal-chemical relationships allow incorporation into these matrices of *d* elements, lanthanides, and, apparently, also actinides. Phosphates with NZP and langbeinite frameworks show isomorphism with many cations and in systems of different compositions and complexities. Such crystalline matrices are undoubtedly promising for immobilization of radioactive waste.

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