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Study of the State of Uranyl Orthovanadate $(UO_2)_3(VO_4)_2·4H_2O$ in Aqueous Solutions

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Abstract—The state of uranyl orthovanadate $(UO_2)_3(VO_4)_2·4H_2O$ in aqueous solutions was studied by the methods of chemical analysis, X-ray diffraction, and IR spectroscopy. Uranyl vanadate is transformed into compounds of other composition and structure upon contact with aqueous phases of various acidity. Equilibrium constants of reactions occurring in heterogeneous systems $(UO_2)_3(VO_4)_2·4H_2O$–aqueous solution were calculated from the data on the solubility. Phase diagrams of bottom solid phases and of equilibrium aqueous solutions were constructed.

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The synthesis, structure, and some properties of uranyl orthovanadate $(UO_2)_3(VO_4)_2·4H_2O$ were reported earlier [1]. This compound is a structural analog of the known uranyl phosphates and arsenates $(UO_2)_3(VO_4)_2·4H_2O$ (B^V = P, As) [2, 3] and can be classified with the numerous set of uranium(VI) derivatives [4-6] of a layer structure. The structure of uranyl orthovanadate includes $[VUO_6]^{\delta-}_{2\infty}$ layers, in which chains of pentagonal bipyramids UO_7 bound with each other along a common rib are joined by VO_4 tetrahedra [1]. Opposite layers are chemically bound with each other by UO_2^{2+} groups and H_2O molecules to form a uniform crystal lattice. At present uranyl orthovanadate is the only known uranium(VI) compound in which vanadium exhibits a tetrahedral coordination. Because of a low solubility it can be used as a compound capable of chemical fixing uranium and radionuclides accompanying it in natural environment and various technological processes.

This work comprises the structural and physicochemical study of the state of uranyl orthovanadate $(UO_2)_3(VO_4)_2·4H_2O$ in aqueous solutions in a wide range of acidity. No such information is available in the literature.

Uranyl orthovanadate $(UO_2)_3(VO_4)_2·4H_2O$ was synthesized from uranyl divanadate $(UO_2)_2V_2O_7$ by reaction (1).

$$3(UO_2)_2V_2O_7 + 8H_2O \rightarrow 2(UO_2)_3(VO_4)_2·4H_2O + V_2O_5. \quad (1)$$

The X-ray data given in Table 1 are in full agreement with the data [1] for the compound $(UO_2)_3(VO_4)_2·4H_2O$.

Experimental values of uranium(VI) (c_U) and vanadium(V) (c_V) concentrations in heterogeneous water-salt systems are presented in Table 2. In the whole studied acidity range the saturated aqueous solutions are true solutions and do not contain colloidal particles. We have calculated the solubility product of uranyl orthovanadate from the data on the solubility of $(UO_2)_3(VO_4)_2·4H_2O$ in aqueous solutions. For this

Table 1. X-ray characteristics of $(UO_2)_3(VO_4)_2·4H_2O$

d, Å	*I*, %	*d*, Å	*I*, %
8.986(5)	73	3.064(0)	46
5.412(1)	93	2.987(3)	25
4.493(7)	100	2.900(4)	40
4.353(8)	42	2.801(2)	19
3.739(4)	21	2.706(6)	26
3.581(9)	45	2.588(6)	25
3.475(3)	38	2.494(3)	31
3.327(7)	50	1.797(7)	21
3.172(2)	31		

Table 2. Concentrations of U(VI) and V(V) in saturated aqueous solutions of $(UO_2)_3(VO_4)_2 \cdot 4H_2O$ (25°C)

Solvent	pH	c_U, M	c_V, M
1 M $HClO_4$	0.58	$(5.29\pm0.13)\times10^{-2}$	$(3.98\pm0.11)\times10^{-2}$
1×10^{-1} M $HClO_4$	1.05	$(9.04\pm0.20)\times10^{-2}$	$(1.31\pm0.29)\times10^{-2}$
5×10^{-2} M $HClO_4$	2.39	$(4.27\pm0.12)\times10^{-3}$	$(0.67\pm0.02)\times10^{-3}$
1×10^{-2} M $HClO_4$	2.84	$(4.89\pm0.21)\times10^{-4}$	$(2.64\pm0.11)\times10^{-4}$
5×10^{-3} M $HClO_4$	3.39	$(11.3\pm0.5)\times10^{-5}$	$(6.70\pm0.44)\times10^{-5}$
1×10^{-3} M $HClO_4$	3.51	$(7.33\pm0.42)\times10^{-5}$	$(4.50\pm0.30)\times10^{-5}$
5×10^{-4} M $HClO_4$	3.62	$(5.30\pm0.32)\times10^{-5}$	$(2.86\pm0.20)\times10^{-5}$
1×10^{-4} M $HClO_4$	3.78	$(3.84\pm0.19)\times10^{-5}$	$(1.86\pm0.11)\times10^{-5}$
H_2O	4.21	$(1.44\pm0.08)\times10^{-5}$	$(0.64\pm0.08)\times10^{-5}$
1×10^{-4} M NaOH	4.33	$(7.49\pm0.58)\times10^{-6}$	$(5.74\pm0.63)\times10^{-6}$
1×10^{-3} M NaOH	4.35	$(1.22\pm0.06)\times10^{-5}$	$(0.55\pm0.08)\times10^{-5}$
5×10^{-3} M NaOH	5.50	$(1.78\pm0.15)\times10^{-6}$	$(1.26\pm0.25)\times10^{-6}$
1×10^{-2} M NaOH	10.09	$(2.39\pm0.11)\times10^{-5}$	$(3.31\pm0.15)\times10^{-3}$
1×10^{-1} M NaOH	12.37	$(7.60\pm0.30)\times10^{-5}$	$(3.76\pm0.15)\times10^{-3}$
1 M NaOH	14.00[a]	$(1.84\pm0.06)\times10^{-4}$	$(3.76\pm0.13)\times10^{3}$

[a] Calculated value.

purpose we presented the transfer of $(UO_2)_3(VO_4)_2 \cdot 4H_2O$ in solution as reaction (2).

$$(UO_2)_3(VO_4)_2 \cdot 4H_2O \leftrightarrow 3UO_2^{2+} + 2VO_4^{3-} + nH_2O. \quad (2)$$

On condition that activities of the solid phase and water are equal the equilibrium constant of this reaction is the solubility product K_s [Eq. (3)].

$$K_s = a(UO_2^{2+})^3 \cdot a(VO_4^{3-})^2. \quad (3)$$

When calculating activities of ions in this expression we took into account the fact that alongside heterogeneous reaction (2) homogeneous equilibria take place in solutions, in which U(VI) and V(V) are present in the form of an assemblage of ionic and molecular species presented in Table 3 [7–10]. Activity coefficients of the ionic forms were calculated by the Debye-Höckel equation in view of the theory of specific ionic interaction [7]. Activity coefficients of molecular species were accepted as equal to unity. The average value of the solubility product index pK_s of uranyl orthovanadate calculated by the data of Table 2 for the range pH 2–7, in which $(UO_2)_3(VO_4)_2 \cdot 4H_2O$ retains its composition and structure, is equal to 54.8±0.3.

The calculated K_s value was used for the calculation of the Gibbs function of $(UO_2)_3(VO_4)_2 \cdot 4H_2O$ formation. The calculation was carried out by relations (4), (5).

$$\Delta G_r = -RT\ln K_s, \quad (4)$$

$$\Delta G_f^0[(UO_2)_3(VO_4)_2 \cdot 4H_2O(t)] = 3\Delta G_f^0(UO_2^{2+}) + 2\Delta G_f^0(VO_4^{3-}) + 4\Delta G_f^0[H_2O_{(l)}] - \Delta G_r \quad (5)$$

Here ΔG_f^0 are standard Gibbs functions of formation of UO_2^{2+} (–952.551), VO_4^{3-} (–891.12), and H_2O (–237.140) kJ mol^{-1} [8–11]; ΔG_r is the Gibbs function of reaction (2). Thus calculated ΔG_f^0 value for $[(UO_2)_3(VO_4)_2 \cdot 4H_2O]$ is –5899±20 J mol^{-1} K^{-1}.

Using the assumed physicochemical description and calculated K_s values, we have simulated the state of the system $(UO_2)_3(VO_4)_2 \cdot 4H_2O$-aqueous solution in a wide range of acidity. In this simulation we took into account the fact that secondary bottom phases of various origins can be formed in the heterogeneous

Table 3. Equilibrium constants of uranium(VI) and vanadium(V) reactions in aqueous solutions

Reaction no.[a]	Reaction equation	Equilibrium constant
1	$UO_2^{2+} + H_2O \rightleftarrows UO_2OH^+ + H^+$	5.62×10^{-6}
2	$UO_2^{2+} + 2H_2O \rightleftarrows UO_2(OH)_2^0 + 2H^+$	7.08×10^{-13}
3	$UO_2^{2+} + 3H_2O \rightleftarrows UO_2(OH)_3^- + 3H^+$	5.62×10^{-21}
4	$UO_2^{2+} + 4H_2O \rightleftarrows UO_2(OH)_4^{2-} + 4H^+$	3.98×10^{-33}
5	$2UO_2^{2+} + 2H_2O \rightleftarrows (UO_2)_2OH^{3+} + H^+$	2.00×10^{-3}
6	$2UO_2^{2+} + 2H_2O \rightleftarrows (UO_2)_2(OH)_2^{2+} + 2H^+$	2.40×10^{-6}
7	$3UO_2^{2+} + 5H_2O \rightleftarrows (UO_2)_3(OH)_5^+ + 5H^+$	2.82×10^{-16}
8	$3UO_2^{2+} + 7H_2O \rightleftarrows (UO_2)_3(OH)_7^- + 7H^+$	6.31×10^{-33}
9	$4UO_2^{2+} + 7H_2O \rightleftarrows (UO_2)_4(OH)_7^+ + 7H^+$	1.26×10^{-22}
10	$VO_2^+ + 2H_2O \rightleftarrows H_3VO_4^0 + H^+$	2.04×10^{-3}
11	$VO_2^+ + 2H_2O \rightleftarrows H_2VO_4^- + 2H^+$	1.61×10^{-7}
12	$VO_2^+ + 2H_2O \rightleftarrows HVO_4^{2-} + 3H^+$	2.79×10^{-16}
13	$VO_2^+ + 2H_2O \rightleftarrows VO_4^{3-} + 4H^+$	1.54×10^{-30}
14	$2VO_2^+ + 3H_2O \rightleftarrows H_2V_2O_7^{2-} + 4H^+$	4.97×10^{-12}
15	$2VO_2^+ + 3H_2O \rightleftarrows HV_2O_7^{3-} + 5H^+$	1.24×10^{-21}
16	$2VO_2^+ + 3H_2O \rightleftarrows V_2O_7^{4-} + 6H^+$	6.48×10^{-33}
17	$4VO_2^+ + 4H_2O \rightleftarrows V_4O_{12}^{4-} + 8H^+$	2.85×10^{-20}
18	$10VO_2^+ + 8H_2O \rightleftarrows H_2V_{10}O_{28}^{4-} + 14H^+$	1.47×10^{-10}
19	$10VO_2^+ + 8H_2O \rightleftarrows HV_{10}O_{28}^{5-} + 15H^+$	9.18×10^{-15}
20	$UO_3\cdot2H_2O(t) \rightleftarrows UO_2^{2+} + 2OH^- + H_2O$	1.91×10^{-22}
21	$NaVUO_6\cdot2H_2O(q) \rightleftarrows Na^+ + UO_2^{2+} + VO_4^{3-} + 2H_2O$	3.02×10^{-31}
22	$Na_2U_2O_7(q) + 3H_2O \rightleftarrows 2Na^+ + 2UO_2^{2+} + 6OH^-$	8.55×10^{-56}

[a] nos. 1–9 – calculated by the data of [7, 8], nos. 10–19 – by the data of [9, 10], nos. 20–22 – by the data of [10] and our data.

systems under study. For this purpose we have set up a system of Eqs. (6)–(14).

$$c_U = \Sigma f\,[a(UO_2^{2+})], \quad (6)$$

$$c_V = \Sigma f\,[a(VO_4^{3-})], \quad (7)$$

$$K_s[(UO_2)_3(VO_4)_2\cdot4H_2O] = a(UO_2^{2+})^3\cdot a(VO_4^{3-})^2, \quad (8)$$

$$K_s(V_2O_5\cdot H_2O) = a(VO_4^{3-})^2\cdot a(H^+)^6, \quad (9)$$

$$K_s(UO_3\cdot2H_2O) = a(UO_2^{2+})^2\cdot a(OH^-)^2, \quad (10)$$

$$K_s(NaVUO_6\cdot2H_2O) = a(Na^+)\cdot a(UO_2^{2+})\cdot a(VO_4^{3-}), \quad (11)$$

$$K_s(Na_2U_2O_7) = a(Na^+)^2\cdot a(UO_2^{2+})^2\cdot K_w^6\cdot a(H^+)^{-6}, \quad (12)$$

$$m[(UO_2)_3(VO_4)_2\cdot4H_2O]/M[(UO_2)_3(VO_4)_2\cdot4H_2O] = c_U\cdot V + \Sigma[m_L\cdot\omega_{U,L}/M(U)], \quad (13)$$

$$m[(UO_2)_3(VO_4)_2\cdot4H_2O]/M[(UO_2)_3(VO_4)_2\cdot4H_2O] = c_V\cdot V + \Sigma[m_L\cdot\omega_{V,L}/M(V)]. \quad (14)$$

Here $m[(UO_2)_3(VO_4)_2\cdot4H_2O]$ is a weight of the primary phase of uranyl orthovanadate; V is a volume of the $HClO_4$ or NaOH initial solution or H_2O, M is a molar weight; m_L is a weight of formed component L of a solid phase, L is V_2O_5, $UO_3\cdot2H_2O$, $NaVUO_6\cdot2H_2O$, or $Na_2U_2O_7$; $\omega_{U,L}$, $\omega_{V,L}$ is a weight fraction of uranium(VI) and vanadium(V) in compound L.

Set of Eqs. (6)–(14) allows us accounting for the presence of primary uranyl orthovanadate in the bottom phase and for the formation of poorly soluble compounds of the secondary origin: V_2O_5, $UO_3\cdot2H_2O$, $NaVUO_6\cdot2H_2O$, $Na_2U_2O_7$, and some others. Eqs. (6), (7) in the above-mentioned set consider homogeneous equilibria involving various ionic-molecular forms of uranium(VI) and vanadium (V). Equations (8)–(12) represent equilibrium constants of heterogeneous reactions between the primary and secondary compounds in the solid phase and the solution, and Eqs. (13), (14) correspond to the total amount of U(VI) and V(IV) in the solid phase and in the solution. The

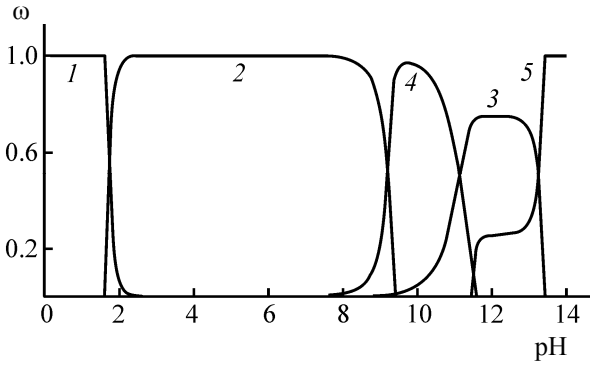

Fig. 1. Calculated phase diagrams of the solid phase $(UO_2)_3(VO_4)_2·4H_2O$ in aqueous solution and of products of $(UO_2)_3(VO_4)_2·4H_2O$ conversion: (*1*) $V_2O_5·H_2O$, (*2*) $(UO_2)_3·(VO_4)_2·4H_2O$, (*3*) $NaVUO_6·2H_2O$, (*4*) $UO_3·2H_2O$, and (*5*) $Na_2U_2O_7$.

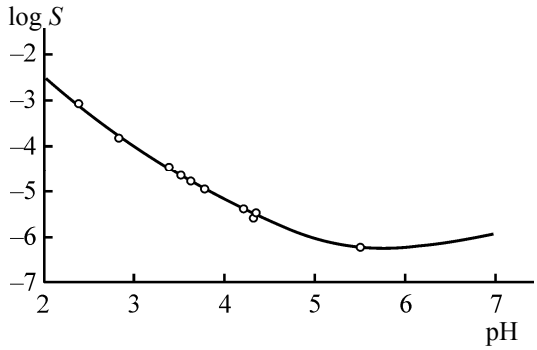

Fig. 2. Calculated (solid line) and experimental (points) values of $(UO_2)_3(VO_4)_2·4H_2O$ solubility.

assumed set of Eqs. (6)–(14) allows calculating various parameters of the heterogeneous systems $(UO_2)_3(VO_4)_2·4H_2O$(s)-aqueous solution under study in a wide range of the acidity of the medium. We have calculated activities of ionic species (UO_2^{2+}), (VO_4^{3-}), total concentrations c_U, c_V in solution, and also weights of solid-phase components of the primary and secondary origin: $m[(UO_2)_3(VO_4)_2·4H_2O]$, $m(V_2O_5)$, $m(UO_3·2H_2O)$, $m(NaVUO_6·2H_2O)$, and $m(Na_2U_2O_7)$ for preset pH values of the equilibrium solution, its volume V, and weight of primary uranyl orthovanadate in a bottom phase $m[(UO_2)_3(VO_4)_2·4H_2O]$. Using these results, we have constructed phase diagrams of the solid phase and have found acid-base ranges of existence of its separate components (Fig. 1). Calculated concentrations of uranium(VI) and vanadium(V) were used for the calculation of the uranyl orthovanadate solubility (15) and for plotting the solubility curve (Fig. 2).

$$S = 0.5(c_U/3 + c_V/2). \quad (15)$$

Activities of ions in aqueous solutions were applied to the calculation of the phase diagrams of U(VI) and V(V) in saturated aqueous solutions (Fig. 3).

Experimental study and simulation of the state of the heterogeneous system $(UO_2)_3(VO_4)_2·4H_2O$-aqueous solution has shown that uranyl orthovanadate conservs its composition and structure on the contact with aqueous solutions of various acidity in the pH range from 2 up to 7. It follows from the acid-base boundaries of transformations of equilibrum bottom phases in the system $(UO_2)_3(VO_4)_2·4H_2O$(s)-aqueous solution shown in Fig. 1 that at pH < 2 uranyl orthovanadate decomposes to release hydrated vanadium(V) oxide into a bottom phase by reaction (16), and uranium(VI) completely passes into solution in the form of UO_2^{2+}.

$$(UO_2)_3(VO_4)_2·4H_2O(s) + 6H^+ \rightarrow V_2O_5(s) + 3UO_2^{2+} + 7H_2O. \quad (16)$$

In alkaline media at pH > 7 uranyl orthovanadate is converted by reaction (17) into a mixture of amorphous hydrated uranium(VI) oxide $UO_3·2H_2O$ and $NaVUO_6·2H_2O$.

$$(UO_2)_3(VO_4)_2·4H_2O(s) + 2NaOH + 2H_2O \rightarrow UO_3·2H_2O(s) + 2NaVUO_6·2H_2O(s). \quad (17)$$

In strongly alkaline media at pH > 12 the structure of uranyl vanadate under study is transformed to sodium diuranate [reaction (18)].

$$2(UO_2)_3(VO_4)_2·4H_2O(s) + 18NaOH \rightarrow 3Na_2U_2O_7(s) + 4Na_3VO_4 + 17H_2O. \quad (18)$$

We have confirmed the appearance of secondary compounds by the method of X-ray phase analysis of bottom-phase samples calcined up to the crystalline state. The identification of all phases is based on the complete coincidence of the X-ray data with the set of X-ray reflection maxima of known compounds [11–14].

Depending on the acidity of the medium not only the composition and structure of a bottom phase in the system $(UO_2)_3(VO_4)_2·4H_2O$(s)-aqueous solution varies, but also both composition and properties of saturated aqueous solutions. Experimental values of uranium(VI) and vanadium (V) concentrations in heterogeneous water-salt systems presented in Table 2 agree well with the results of studying bottom phases. In the acid-base range of $(UO_2)_3(VO_4)_2·4H_2O$ existence the ratio of

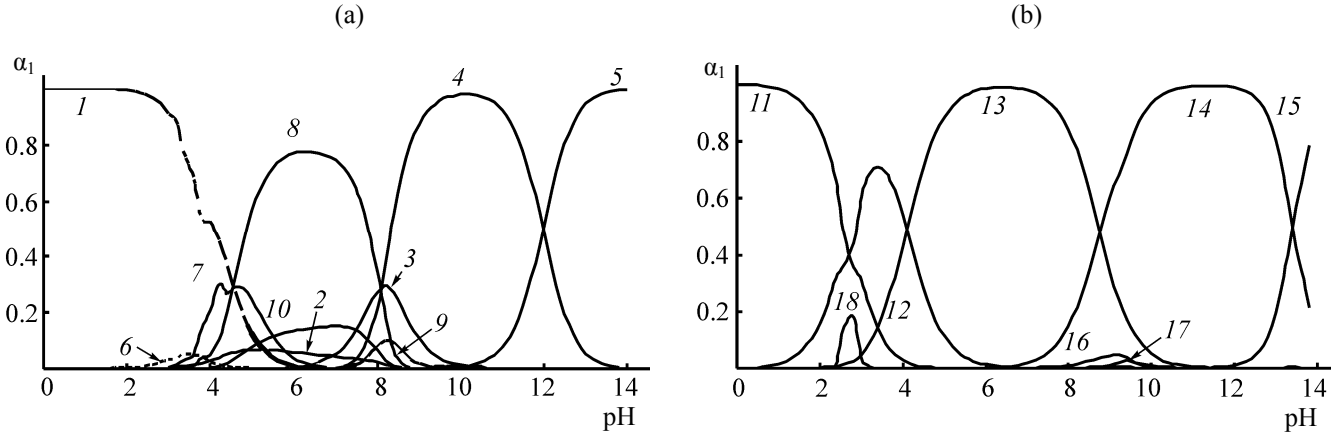

Fig. 3. Calculated phase diagrams of (a) U(VI) and (b) V(V) in aqueous solutions of $(UO_2)_3(VO_4)_2{\cdot}4H_2O$. (*1*) UO_2^{2+}, (*2*) UO_2OH^+, (*3*) $UO_2(OH)_2^0$, (*4*) $UO_2(OH)_3^-$, (*5*) $UO_2(OH)_4^{2-}$, (*6*) $(UO_2)_2OH^{3+}$, (*7*) $(UO_2)_2(OH)_2^{2+}$, (*8*) $(UO_2)_3(OH)_5^+$, (*9*) $(UO_2)_3(OH)_7^-$, (*10*) $(UO_2)_4(OH)_7^+$, (*11*) VO_2^+, (*12*) $H_3VO_4^0$, (*13*) $H_2VO_4^-$, (*14*) HVO_4^{2-}, (*15*) VO_4^{3-}, (*16*) $H_2V_2O_7^{2-}$, (*17*) $HV_2O_7^{3-}$, (*18*) $HV_{10}O_{28}^{5-}$.

concentrations c_U/c_V in equilibrium solution corresponds to the stoichiometry of equilibrium bottom phases c_U/c_V 1.5 within the limits of error. Values of the uranyl vanadate solubility in the region of its existence calculated from relation (15) are presented in Table 4 and are plotted as points on the theoretical curve (Fig. 2).

The solubility of $(UO_2)_3(VO_4)_2{\cdot}4H_2O$ depending on pH varies by several orders of magnitude from 10^{-7} M in weakly acidic and neutral media up to 10^{-5}–10^{-4} M in acid solutions. At pH ≤ 2 as a result of reaction (16) uranium(VI) concentration in the saturated aqueous solutions increases, and the ratio c_U/c_V becomes greater than 1.5. The conversion of compounds in alkaline media [equation (18)] also results in a variation of the stoichiometric ratio of uranium and vanadium in the solution, and the ratio c_U/c_V becomes less than 1.5. Equilibrium aqueous solutions have rather complicated compositions changing as a function of pH values in saturated aqueous solution and corresponding to the composition of the solid bottom phase.

Table 4. Solubility of $(UO_2)_3(VO_4)_2{\cdot}4H_2O$ in aqueous solutions of $HClO_4$

pH	*S*, M
2.39	$(8.78\pm1.26)\times10^{-4}$
2.84	$(1.48\pm0.24)\times10^{-4}$
3.39	$(3.56\pm0.66)\times10^{-5}$
3.51	$(2.35\pm0.52)\times10^{-5}$
3.62	$(1.60\pm0.38)\times10^{-5}$
3.78	$(1.10\pm0.22)\times10^{-5}$
4.21	$(4.00\pm1.08)\times10^{-6}$
4.33	$(2.68\pm0.86)\times10^{-6}$
4.35	$(3.41\pm0.90)\times10^{-6}$
5.50	$(6.11\pm2.92)\times10^{-7}$

Thus, uranyl vanadate $(UO_2)_3(VO_4)_2{\cdot}4H_2O$ has strictly determined acid-base boundaries of existence, outside which it is converted to compounds of other composition and structure. The physicochemical description of the equilibrium in the system $(UO_2)_3(VO_4)_2{\cdot}4H_2O$-aqueous solution given in this work was used for a quantitative estimate of solubility and other characteristics of uranyl vanadate in a wide range of acidity of a medium and composition of equilibrium solutions.

EXPERIMENTAL

Phase individuality of compounds was determined using a Shimadzu XRD-6000 X-ray diffractometer (CuK_α radiation). Photometric measurements were carried out on a Shimadzu UV-1650 spectrophotometer.

Uranyl orthovanadate $(UO_2)_3(VO_4)_2{\cdot}4H_2O$ was synthesized under hydrothermal conditions in an aqueous solution (pH 7) within 48 h at 200°C. The temperature of 200°C limited a possibility of inclusion of weakly-bound molecular H_2O into the structure of uranyl orthovanadate. Admixture of free V_2O_5 in uranyl orthovanadate was removed by washing up a bottom phase by an ammonia aqueous solution heated to 50°C. After that the bottom phase was washed out by distilled water and dried in air.

To study the state of uranyl orthovanadate in heterogeneous water-salt systems, a sample of $(UO_2)_3(VO_4)_2 \cdot 4H_2O$ weighing from 70 to 350 mg was brought into aqueous solutions (from 10 up to 150 ml) of various acidity created by $HClO_4$ or NaOH. To exclude contact of solutions with atmospheric CO_2, the experiments were carried out in hermetic plastic containers with a minimal free space. In all the experiments we used bidistilled water and other necessary reagents free from CO_2 admixtures [16, 17]. Solutions with precipitates were held within 180 days at 25°C up to reaching an equilibrium, periodically stirred, and their pH was controlled with an Akvilon 410 instrument with an ESK-10601/7 glass electrode. After reaching a constant pH value the precipitate was separated from the solution by centrifuging, washed by distilled water, and dried in air. Air-dry bottom phases were studied by the X-ray diffraction and IR spectroscopy methods. Saturated aqueous solutions were analyzed by the photometry method. The concentration of vanadium(V) was determined from the light absorption of its complex with xylenol orange (λ_{max} 475 nm, pH 4.5) [17]. The standard solution of V(V) was prepared by dissolution of NH_4VO_3 in a 1×10^{-3} M solution of sulfuric acid [18]. The concentration of U(VI) in solutions was determined by the reaction with arsenazo III (λ_{max} 650 nm, pH 3) [19]. The standard solution of U(VI) was prepared by dissolution of $UO_2(NO_3)_2 \cdot 6H_2O$ in water [18].

Aqueous solutions were also investigated by the methods of nephelometry and turbidimetry with the purpose of detection suspended and colloidal particles. The intensity of radiation scattered by saturated aqueous solutions was measured using an NFM nephelometer (Russia). Turbidity of solutions was measured on a UV-1650 spectrophotometer (Shimadzu). Statistical treatment of the results was carried out by means of the Mathcad 8.0 program.

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