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Solubility of $A^IVUO_6 \cdot nH_2O$ (A^I = H, Na, K, Rb, Cs, Tl) Uranovanadates

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Abstract—The solubility of uranovanadates of the series $A^IVUO_6 \cdot nH_2O$ (A^I = H, Na, K, Rb, Cs, Tl) in water and aqueous solutions of acids under various conditions were determined. On this basis standard Gibbs functions of formation were calculated, which made it possible to consider the state of these compounds under conditions not covered by the experiment.

Earlier we determined by calorimetry the thermodynamic functions of alkali metal uranovanadates and made an attempt to estimate on this basis the solubility of these compounds in water [1–5]. Similar studies with potassium uranovanadate have been performed in [6–8]. The interest in such compounds is explained by the fact that many of them are minerals and can form in the environment from uranium-containing radioactive wastes. In the present communication we determined experimentally the solubility of compounds $A^IVUO_6 \cdot nH_2O$ (A^I = H, Na, K, Rb, Cs, Tl) in aqueous solutions at 25°C, calculated the standard Gibbs functions of their formation, and examined, by methods of equilibrium thermodynamics of solutions, the effects of various factors on the behavior of these compounds in aqueous media.

It was found that the solution–precipitate equilibrium in the system uranovanadates–water (aqueous solutions of acids) is attained within 35–40 days at intermittent stirring and within 7–10 days at constant stirring. However, in the latter case experimental difficulties arose, because the solution contained colloid particles. Therefore, intermittent stirring was applied.

Table 1 shows that in the pH range 1–7 the solubility of uranovanadates generally decreases with increasing pH. In acidic media, the solubility is near ~10^{-3} m (hereinafter, m is molal concentration), and in weakly acidic and neutral media, depending on the nature of A^I, it varies in the range 10^{-6}–10^{-8} m.

Furthermore, our attention was attracted by the fact that anhydrous (A^I = K, Rb, Cs, Tl) and hydrated phases (A^I = H, Na) differently behave in aqueous media. In the first case, the vanadium and uranium concentrations in aqueous solutions (C_V and C_U) practically coincide throughout the entire pH range. This is confirmed both by the agreement in the vanadium

Table 1. Concentrations of uranium(VI) and vanadium(V) (m) in saturated solutions of $A^IVUO_6 \cdot H_2O$

C_{HCl}, m	Parameter	$HVUO_6 \cdot 2H_2O$	$NaVUO_6 \cdot 2H_2O$	$KVUO_6$	$RbVUO_6$	$CsVUO_6$	$TlVUO_6$
1×10^{-1}	pH	1.4	1.5	1.4	1.25	1.3	1.3
	$C_V \times 10^3$	6.0 ± 0.3	6.5 ± 0.7	8.5 ± 0.7	3.6 ± 0.4	1.2 ± 0.1	0.67 ± 0.04
	$C_U \times 10^3$	6.2 ± 0.7	12.6 ± 0.2	9.0 ± 0.1	4.0 ± 0.4	1.1 ± 0.1	0.71 ± 0.07
1×10^{-2}	pH	2.9	2.75	2.25	2.2	2.2	2.2
	$C_V \times 10^4$	3.3 ± 0.2	1.21 ± 0.05	5.7 ± 0.2	2.2 ± 0.1	1.2 ± 0.6	0.44 ± 0.07
	$C_U \times 10^4$	13.1 ± 1	16.2 ± 0.3	6.4 ± 0.6	2.5 ± 0.1	1.1 ± 0.3	0.80 ± 0.06
1×10^{-3}	pH				3.6	3.95	
	$C_U \times 10^6$				4.1 ± 0.2	0.21 ± 0.01	
1×10^{-4}	pH	–	7.4	4.8	5.2	4.6	4.7
	$C_U \times 10^7$	–	14 ± 1	39 ± 5	0.78 ± 0.07	0.8 ± 0.2	0.80 ± 0.07
0	pH	4.6	7.6	7.2	6.9	6.8	6.1
(water)	$C_U \times 10^7$	190 ± 10	32 ± 2	6.0 ± 0.8	0.80 ± 0.07	1.1 ± 0.2	0.86 ± 0.07

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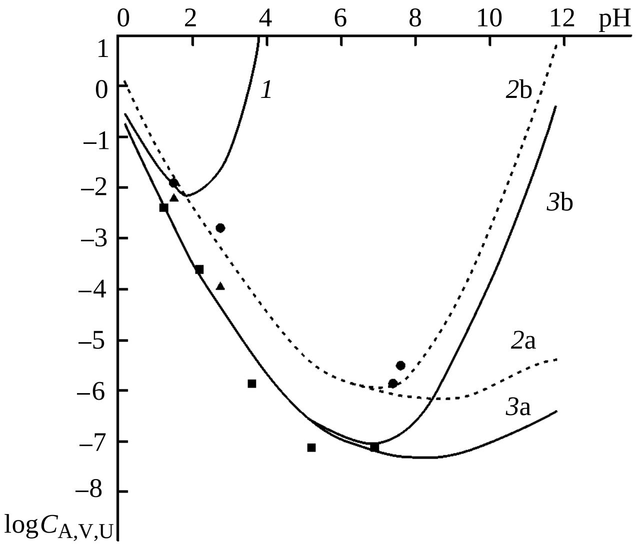

Calculated dependences of the solubility of (*1*) $V_2O_5 \cdot H_2O$, (*2*a, *2*b) $NaVUO_6 \cdot 2H_2O$, and (*3*a, *3*b) $RbVUO_6$ on pH (a) in the absence and (b) in the presence of carbonate ions ($S = C_A = C_V = C_U$, m). Experimental solubilities of $NaVUO_6 \cdot 2H_2O$ [(triangles) C_V, (circles) C_U], and $RbVUO_6$ [(squares) $C_V = C_U$].

concentrations determined by independent methods (photometry and titration) and by their closeness to the total concentrations of solutes, found by evaporation of saturated solutions to dryness [$(5.70 \pm 0.02) \times 10^{-4}$ m for $KVUO_6$, $(2.49 \pm 0.03) \times 10^{-4}$ m for $RbVUO_6$, and $(1.17 \pm 0.02) \times 10^{-4}$ m for $CsVUO_6$ dissolved in 0.01 m HCl, cf. Table 1]. As shown by X-ray phase analysis, the products remaining after evaporation of solutions contacted with precipitates are compounds A^IVUO_6. Thus, in the case of potassium, rubidium, cesium, and thallium derivatives we can accept the metal A^I concentration (C_A) in the solution equal to the half-sum of C_V and C_U.

In the case of $HVUO_6 \cdot 2H_2O$ and $NaVUO_6 \cdot 2H_2O$, C_U is higher than C_V by approximately an order of magnitude (pH 1.4–3.3). According to the X-ray phase analysis, evaporation of a solution equilibrium with sodium uranovanadate, yields the phase $Na_2U_2O_7$, i.e., again, the concentration of vanadium in the solution is much lower than that of uranium, and $C_A \approx C_U$. Structurally, we can suggest that the peculiar behavior of hydrogen and sodium derivatives derives from a lower stability of their crystal structure. This is also proved by the fact that these derivatives form crystal hydrates, where water molecules play a stabilizing role, by releaving the strain in chemical bonds, characteristic of the anhydrous compounds. From the physicochemical viewpoint, the decrease in vanadium concentration is explained by the possibility of formation of one more solid phase in the solution–precipitate system. Such a phase is hydrated vanadium(V) oxide whose solubility in acidic medium, as will be shown below, is comparable with the solubilities of $HVUO_6 \cdot 2H_2O$ and $NaVUO_6 \cdot 2H_2O$ (see figure).

The solubility of $A^IVUO_6 \cdot nH_2O$ at close pH values decreases in the order A^+ = Na > K > Rb > Cs > Tl, i.e. with increasing size of A^+. The observation of such a tendency for structurally related compounds points to increase in the strength of their crystal structure (lattice energy) in the above order, which is in complete agreement with our previous results. For example, as shown in [9], decrease in the ionic potential of A^+ results in increasing melting (decomposition) point of uranovanadate and in decreasing algebraic value of its thermodynamic functions, such as Gibbs functions of formation $\Delta_f G^0(298)$ and enthalpy of formation $\Delta_f H^0(298)$, which are proportional to the energy of chemical bonds.

To trace the effect of the anionic background on the behavior of uranovanadates in aqueous media, we studied the solubility of the potassium compound in the presence of anions able to complex formation with vanadium(V) and uranium(VI) (sulfate, chloride, and nitrate) and also of anions unable to complex formation with these elements (perchlorate). The experiments were carried out in 0.01 m solutions of acids. The solubility of potassium uranovanadate in chloric, nitric, and hydrochloric acids ranges within $(0.4–0.6) \times 10^{-3}$ m, and in sulfuric acid it is higher (1.4×10^{-3} m). These data agree with the stability constants of uranium(VI) complexes with the corresponding ions {for example, $K(UO_2Cl^+$, s.) 2.7 and $K(UO_2SO_4$, s.) 2.2×10^3, calculated with data of [10]}.

Our data on the solubility of uranovanadates in hydrochloric acid (Table 1, C_V and C_U) at pH 1–3 allowed us to calculate equilibrium constants for reactions with their participation and, on their basis, $\Delta_f G^0(298)$ values.

In doing so we took into consideration the fact that in the uranovanadate–solution system under consideration there is a combination of equilibria involving vanadium and uranium in several ionic forms. To calculate $\Delta_f G^0(298)$ values for the compounds, we found expedient to select a scheme corresponding to equilibrium (1). It involves vanadium and uranium species having the highest concentrations in the solution in the experimental pH range (~2.5).

$$A^IVUO_6 \cdot nH_2O(\text{cr.}) + 4H^+(\text{s.}) \rightleftarrows A^+(\text{s.})$$
$$+ UO_2^{2+}(\text{s.}) + VO_2^+(\text{s.}) + (n + 2)\ H_2O(\text{l.}).$$

Table 2. Gibbs functions of formation of $A^IVUO_6 \cdot nH_2O$

Compound	$-\Delta_f G^0(298)$, kJ/mol	
	our data	data of calorimetry
$HVUO_6 \cdot 2H_2O$	2489 ± 7	2477 ± 6 [12]
$NaVUO_6 \cdot 2H_2O$	2749 ± 13	2758 ± 7 [5]
$KVUO_6$	2305 ± 6	2289 ± 9 [1]
$RbVUO_6$	2314 ± 9	2298 ± 14 [1]
$CsVUO_6$	2329 ± 17	2317 ± 14 [1]
$TlVUO_6$	2073 ± 18	–

This process is described by Eqs. (1)–(3).

$$K_S = [a(A^+)][a(UO_2^{2+})][a(VO_2^+)][a(H_2O)]^{(n+2)}[a(H^+)]^{-4}, \quad (1)$$

$$\Delta_r G^0(298) = -RT\ln K_S, \quad (2)$$

$$\Delta_f G^0(298,\ A^IVUO_6 \cdot nH_2O,\ \text{cr.}) = \Delta_f G^0(298,\ A^+,\ \text{s.}) + \Delta_f G^0(298,\ UO_2^{2+},\ \text{s.}) + \Delta_f G^0(298,\ VO_2^+,\ \text{s.}) + (n+2)\Delta_f G^0(298,\ H_2O,\ \text{l.}) - \Delta_r G^0(298). \quad (3)$$

Here K_S is the equilibrium constant, a(L) is the activity of ion (water), R is the universal gas constant, T is temperature (298 K), and $\Delta_s G^0(298)$ is the Gibbs function of solution.

When calculating the required activities of A^+, UO_2^{2+}, and VO_2^+, we took into account that along with the uranyl ions UO_2^{2+} the system contains their chloride UO_2Cl^+ and UO_2Cl_2, as well as hydroxide complexes UO_2OH^+, $UO_2(OH)_2^0$, $(UO_2)_2(OH)_2^{2+}$, $(UO_2)_2(OH)^{3+}$, and $(UO_2)_3(OH)_5^+$. Vanadium(V) exists in the solution as the chloride complex VO_2Cl and also as the acids H_3VO_4, HVO_3, $H_4V_2O_7$, and $H_6V_{10}O_{28}$, and their dissociates; the concentrations of K, Rb, Cs, or Tl are equal to the half-sum of C_V and C_U; and $C_{Na} = C_U$. Taking the above into account, we can write down: $C_V = [a(VO_2^+)]/\gamma_1 + \Sigma k_i a_i/\gamma_{n\pm}$ and $C_U = [a(UO_2^{2+})]/\gamma_2 + \Sigma k_j a_j/\gamma_{n\pm}$. Here a_i and a_j are the activities of vanadium- and uranium-containing ionic and molecular species, $\gamma_{n\pm}$ are the activity coefficients of the corresponding ionic species with the charge $n\pm$, and k_i and k_j are the numbers of vanadium and uranium atoms in their unit formulas.

The a_i and a_j values were expressed in terms of the activities of VO_2^+, UO_2^{2+}, H^+, and Cl^- ions and water molecules, and of the equilibrium constants (K_i and K_j) of reactions between them, corresponding ions, or neutral vanadium and uranium complexes, as shown below in a general form.

$$VO_2^+ + Cl^- \rightleftarrows VO_2Cl,$$

$$a(VO_2Cl) = K_i[a(VO_2^+)][a(Cl^-)],$$

$$kVO_2^+ + (m - 2k)\ H_2O \rightleftarrows H_pV_kO_m^{n\pm} + (2m - 4k - p)\ H^+,$$

$$a(H_pV_kO_m^{n\pm}) = K_i[a(VO_2^+)]^k[a(H_2O)]^{(m-2k)}[a(H^+)]^{[p-2m+4k]},$$

$$n\pm = p + 5k - 2m,$$

$$UO_2^{2+} + x\ Cl^- \rightleftarrows UO_2Cl_x^{n\pm},$$

$$a(UO_2Cl_x^{n\pm}) = K_j[a(UO_2^{2+})][a(Cl^-)]^x,$$

$$n\pm = 2 - x,\ x = 1,\ 2,$$

$$yUO_2^{2+} + zH_2O \rightleftarrows [(UO_2)_y(OH)_z]^{n\pm} + zH^+,$$

$$a([(UO_2)_y(OH)_z]^{n\pm}) = K_j[a(UO_2^{2+})^y[a(H_2O)]^z[a(H^+)]^{-z},$$

$$n\pm = 2y - z.$$

These ion-molecular equilibrium constants, in their turn, were calculated using the $\Delta_f G^0(298)$ values for water, ions, and complexes, given in [10]. In view of the low ionic strengths of the solutions under study, the activity coefficients of the species were calculated by the Debye–Huckel formula. The activity of water was calculated by the equations given in [11]. As a result, the following equations were obtained.

$$C_V = [a(VO_2^+)][\gamma_1]^{-1} + K_i[a(VO_2^+)][a(Cl^-)] + \Sigma k_iK_i[a(VO_2^+)]^k[a(H_2O)]^{(m-2k)}[a(H^+)]^{[p-2m+4k]}[\gamma_{n\pm}]^{-1},$$

$$C_U = [a(UO_2^{2+})][\gamma_2]^{-1} + \Sigma K_j[a(UO_2^{2+})][a(Cl^-)^x[\gamma_{n\pm}]^{-1} + \Sigma k_jK_j[a(UO_2^{2+})]^y[a(H_2O)]^z[a(H^+)]^{-z}[\gamma_{n\pm}]^{-1}.$$

The sets of solutions of these equations contained one real positive root $[a(VO_2^+),\ a(UO_2^{2+})]$ each, which were substituted into Eq. (1). Further, by Eqs. (2) and (3), with account for the $\Delta_f G^0(298)$ values for VO_2^+, UO_2^{2+}, and water [10], we calculated the mean K_S, $\Delta_r G^0$, and $\Delta_f G^0(298,\ A^IVUO_6 – n\ H_2O,\ \text{cr.})$ values listed in Table 2.

As seen from Table 2, the $\Delta_f G^0(298)$ values calculated from solubility data differ from those determined by calorimetry by no more than 16 kJ/mol (< 1%), i.e., the model in use describes the crystalline uranovanadate–solution equilibrium with a reasonable accuracy. This fact allows us, first, to recommend a value of $\Delta_f G^0(298)$ for $TlVUO_6$ which was not studied by calorimetry and, second, to solve a number of problems connected to simulation of the above equilibria over a wide range of characteristics of aqueous medium. Thus, in view of the importance of the problem of uranium migration in the environment, we considered the effects of the pH of the solution and its dissolved carbon dioxide (carbonate ions) content and anionic composition on the solubility of the compounds.

Table 3. Coefficients of the approximating equation $\log S = a_5(\mathrm{pH})^5 + a_4(\mathrm{pH})^4 + a_3(\mathrm{pH})^3 + a_2(\mathrm{pH})^2 + a_1(\mathrm{pH})^1 + a_0$

Compound	$a_5 \times 10^4$	$a_4 \times 10^3$	$a_3 \times 10^2$	$a_2 \times 10$	a_1	a_0
$HVUO_6 \cdot 2H_2O$	3.0	–9.4	11.7	–5.5	–0.07	–0.67
$HVUO_6 \cdot 2H_2O$ (CO_2)	–2.0	5.8	–4.1	1.5	–1.4	0.17
$NaVUO_6 \cdot 2H_2O$	0.7	–2.6	3.1	–0.6	–1.3	0.24
$NaVUO_6 \cdot 2H_2O$ (CO_2)	–3.0	9.3	–7.9	3.5	–1.9	0.43
$KVUO_6$	0.7	–2.4	2.8	–0.2	–1.5	–0.035
$KVUO_6$ (CO_2)	–3.0	7.9	–6.5	3.1	–1.9	0.11
$RbVUO_6$	0.2	–0.9	1.2	0.5	–1.6	–0.43
$RbVUO_6$ (CO_2)	–3.0	7.5	–6.3	3.1	–2.0	–0.32
$CsVUO_6$	0.3	–1.2	1.5	0.5	–1.7	–0.74
$CsVUO_6$ (CO_2)	–3.0	7.3	–6.0	3.1	–2.0	–0.63
$TlVUO_6$	0.4	–1.5	1.7	0.4	–1.7	–0.99
$TlVUO_6$ (CO_2)	–3.0	7.7	–6.4	3.3	–2.0	–0.87
$V_2O_5 \cdot H_2O$			12.3	–0.2	–1.4	–0.24

To simplify calculations, we accepted that, irrespective of the nature of A^+, the C_A, C_V and C_U in a saturated solution are equal to each other and can be labeled by a common character (S). Clearly, as pointed out earlier, in the pH range, where the solubility of $V_2O_5 \cdot nH_2O$, $HVUO_6 \cdot 2H_2O$, and $NaVUO_6 \cdot 2H_2O$ is comparable, calculated equilibrium concentrations of vanadium and uranium would be somewhat higher and lower, respectively, than experimental values. Furthermore, the necessity of this assumption is dictated by the impossibility of strict determination of $\Delta_f G^0(T)$ for $V_2O_5 \cdot nH_2O$ in view of its variable composition and amorphous structure. In this connection we calculated the solubility of hydrated vanadium(V) oxide as a function of solution pH (see fugure) for the monohydrate $V_2O_5 \cdot H_2O$ whose $\Delta_f G^0(298)$ is given in [10]. Taking account of the aforesaid, we set up the following set of three equations.

$$\begin{cases} K_S = S[a(UO_2^{2+})][a(VO_2^+)]\,[a(H_2O)]^{(n+2)}[a(H^+)]^{-4}, \\ S = [a(VO_2^+)][\gamma_1]^{-1} + K_i[a(VO_2^+)[a(Cl^-)) \\ \quad + \Sigma k_i K_i[a(VO_2^+)]^k[a(H_2O)]^{(m-2k)}[a(H^+)]^{[p-2m+4k]}\,[\gamma_{n\pm}]^{-1}, \\ S = [a(UO_2^{2+})][\gamma_2]^{-1} + \Sigma\, K_j[a(UO_2^{2+})[a(Cl^-)]^x[\gamma_{n\pm}]^{-1} \\ \quad + \Sigma k_j K_j[a(UO_2^{2+})]^y[a(H_2O)]^z\,[a(H^+)]^{-2z}[\gamma_{n\pm}]^{-1}. \end{cases}$$

By solving this system with respect to S, $a(VO_2^+)$, and $a(UO_2^{2+})$ for a series of pH values in the range from 1 to 12 (step 0.1), we obtained $S = f(\mathrm{pH})$ dependences. The corresponding plots for sodium and rubidium uranovanadates are shown in the figure. Both with these metals and with the other elements (A^I = H, K, Cs, Tl), calculated values fit experimental ones throughout the entire pH range under study. This is also valid for pH values close to 7, where the equilibrium concentration was determined only for uranium. The dependences for all the compounds were approximated by a fifth-power polynomial whose coefficients given in Table 3.

The resulting data show that uranovanadates have a maximum solubility in the ultra-acidic range. As the acidity decreases, the solubility of the compounds sharply decreases, and in the neutral and weakly alkaline ranges it varies only slightly. Chloride ions hardly affect the solubility of uranovanadates. For example, at pH 7, as the concentration of chloride ions is raised from 0 to 0.1 m, the solubility changes from 2.1×10^{-7} to 2.4×10^{-7} m; in the case of sulfate ions, this change is more essential, up to 3.2×10^{-7} m.

To estimate the effect of atmospheric carbon dioxide, we calculated the solubility at its partial pressure $P(CO_2)$ of 6.3×10^{-4} atm. It is evident from the figure that both solution of CO_2 and formation of uranyl carbonate complexes increase the solubility at pH > 5–6, and the solubility is most strongly affected the strongly alkaline region. The corresponding dependences for all the A^I derivatives are approximated by a fifth-power polynomial whose coefficients given in Table 3.

Thus, on the basis of our experimental data we proposed a model describing the behavior of $A^IVUO_6 \cdot nH_2O$ compounds in aqueous solutions over a wide range of characteristics of the medium.

EXPERIMENTAL

The samples of compounds $A^IVUO_6 \cdot nH_2O$ were synthesized by the procedures described in [9, 13].

All reagents in use were of chemically pure grade. For solubility measurements in water and weak acids, a corresponding solution (0.2–1.5 l) was added to a weighed sample (0.2–1.0 g) of a compound, placed in a polyethylene vessel. The mixture was stirred at 25°C for several weeks. The solution was periodically separated from the precipitate by centrifugation and analyzed for vanadium(V) and uranium(VI). Vanadium(V) was determined by two methods: by photometry using the reaction with xylenol orange (λ_{max} 575 nm, pH 4) and by titration with a Mohr's salt solution [14]. Uranium(VI) was determined by photometry using the reaction with arsenazo III (λ_{max} 650 nm, pH 3) [15]. When the uranium concentration was $\sim 10^{-7}$–10^{-8} m, preliminary concentration by coprecipitation with aluminum phosphate was applied [16]. In this work we used a KFK-3 photocolorimeter and a pH-121 pH-meter–millivoltmeter. The total concentrations of solutes were determined by weighing the dry residues after evaporation of saturated solutions and calcination to constant weight at 500 K.

The vanadium(V) solution (10^{-2} m) was prepared and standardized by the procedure described in [14]. The uranium(VI) solution (10^{-2} m) was prepared by solution of uranium(VI) oxide in 3.5 m sulfuric acid and standardized by titration with potassium dichromate after reduction of uranium(VI) to uranium(IV) with a bismuth reducer [15]. Solutions with concentration lower than 10^{-4} m were prepared by dilution directly before use. All solutions were prepared using twice-distilled water (specific electrical conductivity 0.478 mS/m).

Equations and their systems were solved using the Mathcad 8.0 program.

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