

Sorption of ^{60}Co with Composite Sorbent Termoksid-35 in the Presence of Humic Acids

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Abstract—According to the data of static method of sorption of trace amounts of Co(II) ions by the Termoksid-35 (T-35) sorbent in a chloride-acetate solution, the cobalt distribution coefficient K_d was calculated as a function of pH, concentration of humic acids (HA), and temperature. The distribution of the complexes Co^{2+} , CoAc^+ , CoCl^+ , Co(OH)^+ , Co(OH)_2^0 , Co(OH)_3^- between the solution and the sorbent was modeled within the framework of the surface complexation concept. The found stability constants of the Co(OH)^+ and Co(OH)_2^0 complexes coincide with the thermodynamic values. In the presence of HA (10.0 mg/L), the best agreement between the model function of K_d -pH and the experimental data corresponds to the sorption of a complex with the composition Co(OH)A^0 , where A^- is the anion of humic acids. It was concluded that T-35 can be used as a sorbent for removal of Co(II) radionuclides from industrial process at $\text{pH} > 7$ and natural water in the presence of humic acids.

Keywords: Termoksid-35, Co(II), humic acids, statics, sorption, surface complexation, modeling

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Removal of cobalt radionuclides from aqueous media is one of the important problems of ensuring the nuclear fuel cycle enterprise safety because of the high radiotoxicity of ^{60}Co and ^{58}Co isotopes [1]. Decommissioning of NPP power units that have completed their cycle presupposes the designing effective technologies for the sorption decontamination of equipment and materials, in which the leading role will be played by multifunctional sorbents capable not only of ensuring the removal of long-lived radionuclides from the contaminated surfaces of dismantled equipment, but also performing in the future the role of chemically and radiation-resistant inert matrix for disposal of radioactive waste of high and medium specific activity [2, 3]. There are difficulties in concentrating Co(II,III) radionuclides from decontamination solutions associated with the formation of an ionic-colloidal state during the formation of complexes with ethylenediaminetetraacetic acid [4]. Therefore, promising methods of decontamination should take into account the factors of the physicochemical state of cobalt ions in the treated solutions of industrial process or natural origin. In the latter case, it is required to know how humic substances, ubiquitous in natural waters,

can affect the state and reactivity of Co(II) ions [5–7]. Potentially promising multifunctional sorbents include materials based on zirconium oxide [3], for example, sorbents of the Termoksid 3 (T-3) and Termoksid 35 (T-35) grades [8, 9]. ZrO₂-Based composite T-35 has sorption specificity to Cs radionuclides due to the presence of mixed nickel-potassium cyanoferrate, and zirconium oxide is characterized by high distribution coefficients of Eu(III), U(IV,VI), Np(IV,V), Pb(II), Co(II), Ni(II) [9–16]. Hydrated zirconium oxide exhibits amphoteric properties, and it is also used for the separation of anions in a column version, for the concentration and purification of organic peroxide compounds. The high selectivity of ZrO₂ to the anions of some metals [for example, Mo(VI), W(VI), Re(VII), Sb(V)] makes it possible to achieve chromatographic separation of anions in one sorption stage [17]. It is noted that the position of the point of zero charge (PZC) of zirconium oxides in the region $\text{pH} \sim 7$ promotes active interaction of the zirconium oxide surface with a gas atmosphere and chemisorption of carbon dioxide, organic compounds, and water vapor [17–19]. One of the features of amorphous zirconium oxides prepared by colloidal-chemical methods is their active interaction

with electromagnetic radiation in the UV range, which affects the sorption and structural-morphological characteristics of zirconium oxyhydroxide gels [20]. The chemical introduction of the second component into the zirconium oxide solid phase stabilizes and significantly enhances the sorption affinity and selectivity of the resulting composites with respect to U(VI) ions [16]. Organo-inorganic composites based on a combination of polysiloxanes with functional groups of iminopropionic acids and aluminum, zirconium, titanium oxides are able to efficiently extract Cu(II) ions from aqueous solutions in the mixed diffusion mode [21]. Mixed cyanoferrate Ni–K in the form of its own phase and in composites based on silica gel and aluminosilicate exhibits selectivity for cesium ions and, at the same time, an increased affinity for cobalt ions in the region of the cation hydrolysis onset [22, 23]. Data on the HA effect on the Co(II) sorption by the T-35 sorbent or its components, ZrO_2 , $\text{K}_2\text{NiFe}(\text{CN})_6$, have not been found in the literature, but the formation of a humate complex of cobalt has been established [24]. Evaluation of Co(II) sorption affinity for T-35 will allow selecting the conditions for purification of technological and natural solutions from Co(II) radionuclides using T-35 sorbent. In this work we studied sorption equilibria between $^{60}\text{Co}(\text{II})$ ions and the T-35 sorbent, analyzed the dependence of the total distribution coefficient K_d of a set of Co(II) ion-molecular complexes between the solution and the sorbent on pH, temperature, and the content of humic acids in the solution based on the concept of surface complexation of Co(II) ions with ol-groups of the hydrated T-35 sorbent. The data obtained will make it possible to establish the possibility of using the T-35 sorbent for the joint extraction of Cs, Co radionuclides from natural and process solutions.

EXPERIMENTAL

The composition of the functional groups of T-35 samples was determined by vibrational spectroscopy and X-ray phase analysis in order to confirm the main characteristics of the manufacturer. IR spectra in the range of $4000\text{--}400\text{ cm}^{-1}$ were recorded on a Vertex 80 (Bruker) FT-IR spectrometer using an MVP-Pro (Harrick) ATR (attenuated total reflectance) accessory (prism material diamond). The Raman spectrum of the sample in the wavenumber range $4000\text{--}50\text{ cm}^{-1}$ was recorded at room temperature on an InVia Reflex RENISHAW Raman dispersive spectrometer ($\lambda =$

532 nm , $P = 10\text{ mW}$). The obtained IR spectra are presented in Supplementary Information (Fig. 1S). The elemental composition of the T-35 sorbent and the morphology of its individual particles were detected by scanning electron microscopy and local elemental analysis on a JEOL JED-2300 device. Since the samples of Termoksid-35 are highly electrified under the action of an electron beam, for obtaining high-quality images a thin layer of metallic gold was deposited on the samples using a deposition attachment to the microscope. The shooting modes, including the accelerating voltage of the electron beam, were selected to detect the distribution of both matrix elements of the material (Zr, O) and elements with a lower concentration (Ni, Fe, N, C).

X-ray photographs were taken on a STOE STADI-P diffractometer system combined with the Bragg–Brentano diffractometer and a secondary monochromator (CuK_α radiation). Polycrystalline silicon was used as an external standard.

The specific surface area of the materials and the pore size distribution were determined by the low temperature nitrogen adsorption with the T-35 sample on a Gemini VII 2390 V1.30t setup. We used T-35 samples with a shelf life of about 2.5 years. Before measuring the specific surface area, the sample in the test tube was placed in a Micromeritics VacPrep 061 preliminary degassing device and vacuumed to a residual pressure of 0.4 mbar. Then the sample was heated step by step to 150°C and kept for 2800 min at a constant temperature. At the end of the heat treatment, the pressure in the test tube dropped to 0.1–0.15 mbar.

The static sorption of Co(II) ions by the T-35 sorbent was studied using a $^{60}\text{CoCl}_2$ radioactive indicator with a carrier concentration of $2 \times 10^{-7}\text{ M}$. To detect the chemical composition a 0.5 M NaCl solution in an acetate buffer with a 0.28 M total acetate ion concentration (HAc + NaAc) and a 0.78 M ionic strength. The concentration of humic acids was set at the level of 10 mg/L ($6.6 \times 10^{-5}\text{ M}$) by diluting in the test solution an aliquot of natural water containing mainly humic acids with a concentration of 0.99 g/L.

The equilibrium sorption S was measured by the count rates of the initial (I_{in}) and equilibrium samples (I_{eq}) after a week phase contact on an MKS-AT1315 γ -spectrometer (the Atomtech company) with a NaI (TI) $62 \times 62\text{ mm}$ scintillation detector in a lead shield in a Denta vessel:

$$S = \left(1 - \frac{I_{\text{eq}}}{I_{\text{in}}} \right). \quad (1)$$

The count rates were registered in the region of ^{60}Co radionuclide photopeaks in the range of 1150–1500 keV. Cobalt distribution coefficient K_d was determined by the formula

$$K_d = \frac{\left(\frac{S}{1-S} \right)}{[m]}, \quad (2)$$

where S is equilibrium sorption, $[m]$ is the ratio of the sorbent weight (g) to the solution volume (mL).

RESULTS AND DISCUSSION

The IR spectra of the studied T-35 samples contain bands of stretching $\nu(\text{H}_2\text{O})$ and bending $\delta(\text{H}_2\text{O})$ vibrations of water molecules. The presence of nickel cyanoferrate manifests itself in the IR spectra in the form of an intense narrow absorption band $\nu(\text{C}\equiv\text{N})$ at 2088 cm^{-1} . The bands at 2099 and 2137 cm^{-1} correspond to symmetric stretching vibrations $\nu(\text{C}\equiv\text{N})$, at 580 cm^{-1} , to vibrations $\delta(\text{FeC})$ of the cyanide complex $[\text{Fe}(\text{CN})_6]_4^-$ [1]. The Raman spectra of T-35 also contain a line at 245 cm^{-1} of the main phase of ZrO_2 of the tetragonal modification and a symmetric stretching vibration $\nu(\text{C}\equiv\text{N})$, as well as weak bands corresponding to normal vibrations ν_1 , ν_3 , ν_4 of the CO_3^{2-} anion [25] due to contact with air [13]. According to the data of scanning electron microscopy and local elemental analysis recorded on a JEOL JED-2300 device, the spherical particles of the sorbent have a smooth outer surface. Its micro-relief is an alternation of rounded cavities and micron-sized hills. The spectrum of the characteristic excited X-ray radiation of the sample surface shows the presence of Zr, O, Fe, Ni, K atoms, as well as traces of Na and C. The distribution of these elements over the surface layer of the sample of T-35 particles shows that zirconium in the form of ZrO_2 (tetragonal modification) is the main matrix element. The local distribution of Ni–K cyanoferrate atoms on the size-scale of an individual sorbent granule is characteristic of 1–5 μm particles uniformly distributed over the entire granule volume. The morphological features of the sorbent particles are described in more detail in Supplementary Information.

According to X-ray phase analysis, the T-35 sample is a phase blend of mixed cyanoferrate $\text{K}_2\text{NiFe}(\text{CN})_6$, space group $F\bar{4}3m$, coherent scattering region 8.5 nm,

and weakly crystallized ZrO_2 , presumably space groups $P4_2/n$, $P2_1/m$, $P2/c$ (Figs. 2S, 3S). These data are consistent with the results obtained by vibrational spectroscopy and electron microscopy.

Data of low-temperature nitrogen adsorption by the T-35 sample (see Supplementary Information) show that the total specific surface area of the sorbent particles is $188.5\text{ m}^2/\text{g}$ (according to the BET method), the total volume of pores with a size of less than 462 \AA is $0.106\text{ cm}^3/\text{g}$, the average pore size by adsorption is 24.0 \AA , and that by desorption is 28.7 \AA . T-35 is mainly a microporous and transitional-porous sorbent.

The combination of physicochemical analysis methods demonstrates the presence of the main components of the T-35 composite: the tetragonal modification hydrated ZrO_2 matrix and $\text{K}_2\text{NiFe}(\text{CN})_6$ phase particles.

Humic acids were of natural origin from the Salekhard area water [26]. According to the data of chemical analysis, humic acids compose more than 98 wt % of organic carbon of this natural water (Supplementary Information) [3, 26]. In all experiments, the sorbent weight m in the system corresponded to the ratio of the solution volume to the sorbent weight $V/m = 100\text{ mL/g}$; the size of the sorbent granules was 0.4–1.0 mm, the capacity by Cs(I) ions was 0.4 mmol/g . The static composite capacity by zirconium oxide protons (4.4 mmol/g [9]) corresponded to the maximum ratio of cobalt ions to proton ol groups, $5 \times 10^{-6}\text{ mol/mol}$ (Henry's law region). The temperature in all experiments was maintained using a liquid thermostat.

The composition of the test solution, which did not contain humic acid, included aqua ion and Co(II) complex compounds. Figure 1 shows the regions of predominance of Co(II) complex compounds vs. pH. The stability constants of hydroxo, acetate, and chloride ion complexes for 23°C were used to build the diagram: $\text{Co}(\text{OH})^+ \log \beta_1 = 4.35$, $\text{Co}(\text{OH})_2^0 \log \beta_2 = 8.40$, $\text{Co}(\text{OH})_3^- \log \beta_3 = 9.70$ [27]; $\text{CoAc}^+ \log \beta_{\text{Ac}} = 1.46$ [2]; $\text{CoCl}^+ \log \beta_{\text{Cl}} = 2.27$ [28]. The main cations in the studied pH range are $\text{Co}(\text{OH})^+$, $\text{Co}(\text{OH})_2^0$, $\text{Co}(\text{OH})_3^-$ at $\text{pH} < 9.5$ and hydroxo complexes at $\text{pH} > 9.5$ (Fig. 1). The choice of a chloride-acetate buffer sorbate solution as a medium was determined by the fact that sorption equilibria with the Termoksid-35 sorbent are accompanied by a change in the solution pH, especially in the ZCP region of zirconium oxide (matrix). The electrolytic background

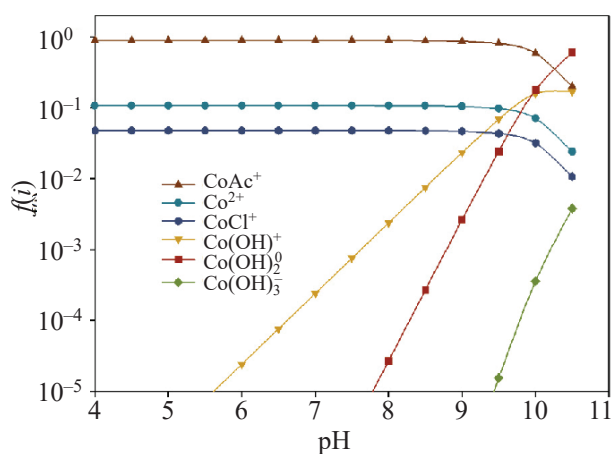


Fig. 1. (Color online) Fractional composition $f(i)$ of Co(II) ions in a water–chloride–acetate solution with an ionic strength (M) of 0.78 (acetate ions 0.28, chloride ions 0.50), calculated at 22°C.

solution we used was a buffer solution that stabilized the pH during the sorption experiment. The sorption affinity of proton groups on the surface of hydrated zirconium oxide for Co(II) cations has a different character in comparison with nickel–potassium cyanoferrate (Fig. 5S) [13, 23, 29–33]. The phases of zirconium oxide and nickel–potassium cyanoferrate weakly adsorb Co^{2+} and CoCl^+ aqua ions [15]. Taking this fact into account, the model of equilibrium sorption discussed below considers the interaction of the sorbate with the proton groups of the main phase of zirconium oxide by the surface complexation mechanism [13, 32]. The leading role of this mechanism is indirectly confirmed by the enthalpy of sorption (-21 ± 11 kJ/mol), found by the function of the distribution coefficient vs. temperature in the pH range 7.0–7.7 (Fig. 6S). Its value significantly exceeds by orders of magnitude the thermal effects characteristic of ion-exchange sorption of cations by organic resins [34].

Figure 2 shows for comparison the experimental $\log K_d$ –pH curves of the Co(II) sorption for a solution of the NaCl–NaAc/HAc (I) and NaCl–NaAc/HAc–HA compositions, where HA is protonated humic acids. Separate experiments have demonstrated that an average absolute error for the reproducibility of the $\log K_d$ under the studied conditions is 0.50–0.60 log units.

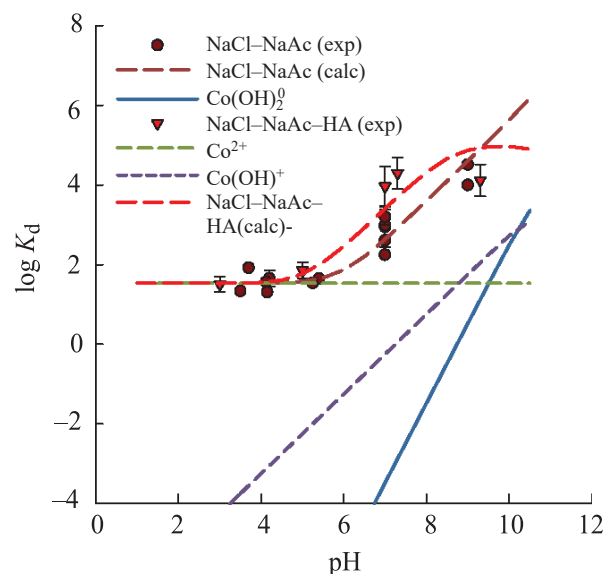
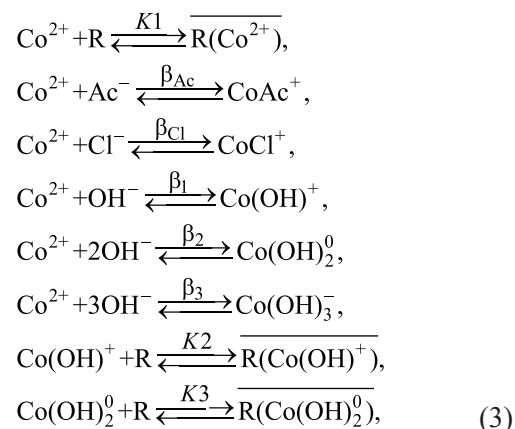


Fig. 2. (Color online) Experimental curves of the Co(II) sorption by Thermoxide 35 in the test solution vs. pH. $K_d(\text{model})$ curve obtained as a result of modeling according to Eq. (4) taking into account equilibria of Eqs. (3), (6). (Colored dots) Co(II) sorption vs. pH in solution in the presence of humic acids, 20°C.

The resulting dependence in system (I) was analyzed based on the surface complexation model in view of the following homogeneous and heterogeneous reactions (3):



where R is a functional proton containing group, the line designates attributing to the sorbent surface, ZrO_2 . In the system of Eqs. (3), the stability constants β_{Ac} , β_{Cl} , β_{1-3} are defined above. K_1 – K_3 are heterogeneous constants of surface complexation of ol-groups of a hydrated oxide surface with positively charged and neutral particles Co^{2+} , $\text{Co}(\text{OH})^+$, $\text{Co}(\text{OH})_2^0$, and the products of their concentrations and the concentration [R] of surface functional groups (OH_2^+ –OH) of the T-35 composite

give the grade distribution coefficients K_{di} [5, 31, 32, 35]. The sorption curve corresponding to equilibria of

Eqs. (3) for the total distribution coefficient of cobalt ions K_d has the form

$$K_d = \frac{K_{11} + K_{12}[\text{OH}^-] + K_{13}[\text{OH}^-]^2}{1 + \beta_A c[\text{Ac}^-] + \beta_C I[\text{Cl}^-] + \beta_1[\text{OH}^-] + \beta_2[\text{OH}^-]^2 + \beta_3[\text{OH}^-]^3}$$

$$= K_{d1}f(1) + K_{d2}f(2) + K_{d3}f(3),$$

$$K_{11} = K_1[R] = K_{d1}, K_{12} = K_2[R]\beta_1 = K_{d2}\beta_1, K_{13} = K_3[R]\beta_2 = K_{d3}\beta_2, \quad (4)$$

where $f(1)$ – $f(3)$ are the equilibrium mole fraction of particles Co^{2+} , $\text{Co}(\text{OH})^+$, $\text{Co}(\text{OH})_2^0$ taken in the model as sorbed.

The experiments were carried out in the pH range of ZrO_2 PZR (pH ~ 7 [13]). Therefore, the change in the composite surface charge was not taken into account in the model. The study was performed in the pH range in which the cations Co^{2+} , CoAc^+ , CoCl^+ , and the hydroxo complexes $\text{Co}(\text{OH})^+$, $\text{Co}(\text{OH})_2^0$ are present. Analysis of the experimental data $K_d(\text{experiment})$ –pH according to the model (4) using the approximation software (TableCurve) allows calculating the numerical values of all coefficients in the equilibrium system [Eqs. (3)], and comparing the found values of the hydrolysis constants $\log \beta_{1,2}$ with the tabulated values for zero ionic strength: $\log K_{11} = \log K_{d1} = 1.50$; $\log K_{12} = 9.70$; $\log K_{d2} = 5.30$; $\log K_{13} = 10.70$; $\log K_{d3} = 2.20$; $\log \beta_1 = 4.40$ (tabulated value 4.35 [27]); $\log \beta_2 = 8.50$ (tabulated value 8.40 [27]).

The agreement between the calculated and tabulated data is, in our opinion, evidence of the adequacy of

the considered model to the correct choice of the ion-molecular composition of the particles, which is responsible for the equilibrium sorption of cobalt ions by the hydrated surface of the T-35 sorbent oxide matrix. In an equilibrium system consisting of three ($I = 1$ – 3) fractions (f_i) of sorbed cobalt particles, Co^{2+} , $\text{Co}(\text{OH})^+$, $\text{Co}(\text{OH})_2^0$, the following relation

$$K_d = \sum_i K_{di} f_i \quad (5)$$

holds between the total distribution coefficient K_d and the grade distribution coefficients K_{di} of each type of particles (i) resulting from Eq. (4) [31]. Figure 2 shows the calculated curves characterizing the contribution of individual sorbed cobalt particles to the total distribution coefficient according to Eqs. (3), (4) (see data above). It can be seen that in the range of pH < 6 , sorption is determined by Co^{2+} cations and the presence of CoAc^+ and, in part, CoCl^+ . In the pH range 6–8, the largest contribution to the sorption of cobalt ions is made by $\text{Co}(\text{OH})^+$ cations and, to a lesser extent, $\text{Co}(\text{OH})_2^0$. Note

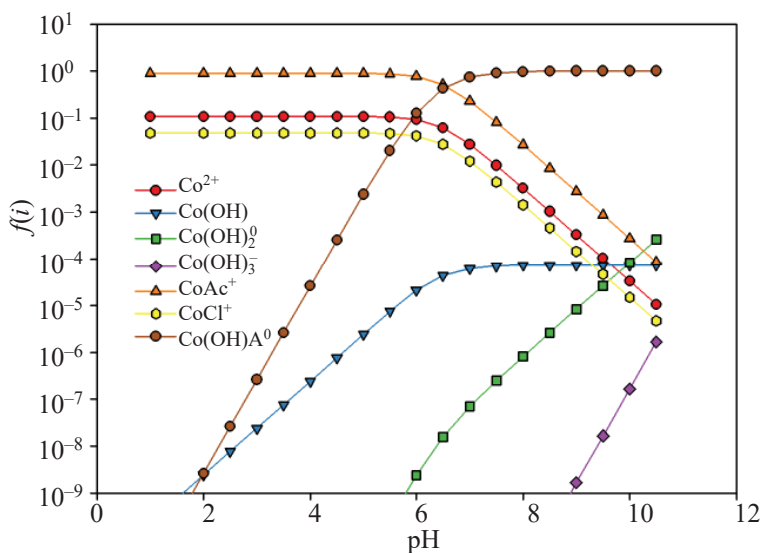


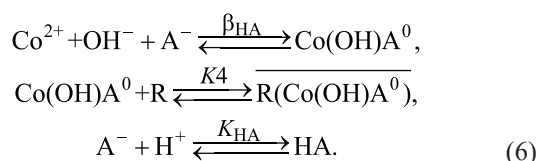
Fig. 3. (Color online) The fields of Co(II) ionic-molecular particles predominance in solution (II) according to the data in Tables 1, 2 for 20–23°C.

Table 1. OLS-Estimates of the coefficients of Eqs. (3, 4) for the solution (I)–T-35 system, obtained by the least squares method

Value	K_{11}/K_{d1}	K_{12}/K_{d2}	K_{13}/K_{d3}	β_1	β_2
log(value)	1.50/1.50	9.70/5.30	10.70/2.20	4.40	8.50
log($\beta_{1,term}$), [27]	–	–	–	4.35	8.40

that the buffer solution affects the chemical composition of the sorbate up to pH 9, but in the presence of the T-35 sorbent this effect decreases and is limited to a pH range of less than 5, since Co(II) hydroxo complexes become the main sorbed complexes in the presence of T-35.

As can be seen from Fig. 3, the presence of milligram amounts of humic acids in the test solution changes the nature of the function of the distribution coefficient K_d vs. pH. To reconcile the observed changes in the course of the K_d (pH) function, it is sufficient to introduce the following additional equilibria into the model [Eqs. (3), (4)]:



Equations (6) assume the thermodynamic possibility of a mixed molecular complex formation of the $\text{Co}(\text{OH})\text{A}^0$ composition by Co(II) ions in a 6.6×10^{-5} M humic acid solution. Sorption of this complex by T-35 is described by the heterogeneous constant of surface complexation K_4 . The system of Eqs. (6) also takes into account the change in the concentration of humic acid anions as a result of their protonation at pH < 6 with the conditional protonation constant K_{HA} [30]. The OLS estimates of the coefficients of model of Eqs. (3), (4), (6) for the solution (II)–T-35 system are given below: $\log K_{14} = 12.70$, $\log K_{d4} = 4.20$, $-\log K_{\text{HA}} = 6.40$ (according to the data of [36], 6.6 ± 0.8), $\log \beta_{\text{HA}} = 8.50$.

The estimation of the coefficients K_{14} , K_{d4} , K_{HA} , and β_{HA} according to the model of Eqs. (3), (4), (6) was carried out using the values of parameters previously found (Table 1). Their relative error, taking into account the accumulation of the error of indirect measurements, did not exceed 0.45–0.55. As seen from a comparison of the data obtained taking into account and without Eqs. (6) the found value of the stability constant of the hypothetical mixed-ligand humate complex $\text{Co}(\text{OH})\text{A}^0$ is by about 2 log units higher than the stability constant

Table 2. OLS estimates of the model coefficients of Eqs. (6, 3, 4) for the solution (II)–T-35 system

Value	K_{14}/K_{d4}	K_{HA}	β_{HA}
log(value)	12.70/4.20	6.40	8.50
$-\log(K_{\text{HA}})$, [36]	–	6.6 ± 0.80	–

of the first hydroxocomplex (β_1), and the sorption affinity (K_{d4}) of the $\text{Co}(\text{OH})\text{A}^0$ complex for T-35 sorbent is lower than that of the $\text{Co}(\text{OH})^+$ complex. The diagram of the predominance of ionic molecular forms of Co(II) in solution (II) vs. pH (Fig. 3) shows that in the presence of 10 mg/L natural HA the $\text{Co}(\text{OH})\text{A}^0$ complex prevails at pH > 6–7, where no effect of HA protonation occurs.

In general, a dataset on the equilibrium sorption of microquantities of Co(II) ions in a chloride-acetate solution by the sorbent depending on pH shows that Termoksid-35 exhibits a high sorption affinity not only for cesium ions, but also to hydrolysable Co(II) cations in the pH range where the main contribution to the ion state is made by the $\text{Co}(\text{OH})^+$ and $\text{Co}(\text{OH})_2^0$ complexes. In the presence of humic acid anions, the humate complex $\text{Co}(\text{OH})\text{A}^0$ becomes the dominant particle at pH above 6.5. Its distribution coefficient is close to that of the $\text{Co}(\text{OH})^+$ and $\text{Co}(\text{OH})_2^0$ complexes. The data obtained show that the T-35 sorbent can be used for the joint sorption of microquantities of cesium and Co(II) ions from process solutions with pH > 7 and natural water in the presence of humic acids.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

SUPPLEMENTARY INFORMATION

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