ISSN 0036-0236, Russian Journal of Inorganic Chemistry, 2022, Vol. 67, No. 9, pp. 1386–1392. © Pleiades Publishing, Ltd., 2022.
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SYNTHESIS AND PROPERTIES OF INORGANIC COMPOUNDS

Study of Strontium Sorption by Amorphous Calcium Silicate

S. B. Yarusova[a, b, *], P. S. Gordienko[a], O. O. Shichalin[c], E. K. Papynov[c], E. A. Nekhliudova[a, b], A. V. Perfilev[b], S. Yu. Budnitskiy[d], N. V. Zarubina[d], Yu. A. Parotkina[a], A. N. Drankov[a], S. B. Bulanova[a], and N. V. Ivanenko[b]

[a] *Institute of Chemistry, Far Eastern Branch, Russian Academy of Sciences, Vladivostok, 690022 Russia*
[b] *Vladivostok State University of Economics and Service, Vladivostok, 690014 Russia*
[c] *Far Eastern Federal University, Russky Island, Vladivostok, 690922 Russia*
[d] *Far East Geological Institute, Far Eastern Branch, Russian Academy of Sciences, Vladivostok, 690922 Russia*
**e-mail: yarusova_10@mail.ru*

Received March 6, 2022; revised March 28, 2022; accepted March 30, 2022

Abstract—Data on the sorption characteristics of synthetic X-ray amorphous calcium silicate obtained in the $CaCl_2$–Na_2SiO_3–H_2O model multicomponent system are presented. The isotherms of sorption of Sr^{2+} ions from aqueous solutions without supporting salt at Sr^{2+} concentrations of 0.016 to 1.22 mmol/L at temperatures of 20, 40 and 60°C and the solid to liquid phase ratio S : L = 1 : 400 are presented. The maximum sorption capacities of synthetic X-ray amorphous calcium silicate and the recoveries of Sr^{2+} ions were determined for various S : L ratios and for solutions containing no supporting salt and solutions mimicking the specific composition of fresh water. The Sr^{2+} sorption kinetics at various temperatures was studied for the first time; and the activation energy of sorption was determined. The results can be used to develop practical recommendations for the production of this material and application in the processes of strontium sorption and immobilization.

Keywords: calcium hydrosilicate, model system, sorption, strontium, isotherms, kinetics, activation energy

DOI: 10.1134/S0036023622090194

INTRODUCTION

Among the radioactive strontium isotopes, the greatest contribution to the environmental pollution is made by ^{89}Sr and ^{90}Sr. The short-lived ^{89}Sr isotope ($\tau \sim$ 50.5 days) can be traced in the environment at early stages of technogenic accidents [1, 2]. ^{90}Sr is a β-emitter with a high specific activity and long half-life ($\tau \sim$ 28 years), i.e., it is one of the main components of radioactive contamination in the first decades after an accident; therefore, it is hazardous for natural ecosystems and biosphere as a whole. Upon bioaccumulation, radionuclides get into the ends of food chains, thus being dangerous for health and inducing changes in the species populations. The osteotropic ^{90}Sr isotope is strongly held mainly in the human and animal skeleton bones (especially when there is deficiency of calcium). The biological action of radioactive strontium in radioactive contamination areas is diverse, depending on the chemical speciation, route of entry of radionuclides into the body, distribution in the body, and the formed doses: they may induce acute and chronic radiation injuries and the concomitant hematological disorders, leukemia, and bone tumors in higher animals and humans [3–5].

Quite a few publications are devoted to isolation and immobilization of strontium. Among the diverse inorganic sorbents proposed for the recovery of strontium from aqueous media, of certain scientific and practical interest are natural and synthetic silicates and composite materials based on them [6–10]. A publication of Russian scientists gives a good review and describes the authors' own studies on the materials developed for the extraction of strontium from natural media, in particular from sea water [11].

In the studies of strontium adsorption, alkali metal silicates and aluminosilicates are considered as a promising class of sorbents [12–18].

A number of Russian and foreign publications address the sorption properties of calcium silicates towards Sr^{2+} ions [16–19]. Calcium silicates studied for this purpose are 11 Å tobermorite, tobermorites with various contents of Al^{3+}, xonotlite, and wollastonite. The maximum sorption capacity varies in the range of 0.017–1.8 mmol/g depending on the sorption conditions (temperature, ratio of solid and liquid phases, pH, and supporting salt).

This study deals with the sorption properties of a material based on calcium hydrosilicate formed in the $CaCl_2$–Na_2SiO_3–H_2O model multicomponent sys-

tem towards Sr^{2+} ions, in particular, when they occur in complex salt solutions.

EXPERIMENTAL

Synthesis of the sorbent. Calcium hydrosilicate was synthesized in the $CaCl_2$–Na_2SiO_3–H_2O model system using reagent grade sodium silicate with a silicon content of 22.4% (silicate modulus $SiO_2/Na_2O = 1$) and calcium chloride dihydrate $CaCl_2{\cdot}2H_2O$ (TU 2152-069-00206457-2003; $CaCl_2{\cdot}2H_2O$ content of not less than 98.3%). The components were mixed in water in open vessels according to the guidelines reported in [20]. The obtained white-colored voluminous precipitate was thoroughly washed, filtered through a "blue ribbon" filter, and dried at 85°C.

Sorption experiments. The sorption experiments were carried out under static conditions at the solid to liquid phase ratio of 1 : 400 and temperatures of 20, 40, and 60°C using aqueous solutions of strontium chloride ($SrCl_2{\cdot}6H_2O$) without a supporting salt, with the starting concentration of Sr^{2+} ions varying from 0.016 to 1.22 mmol/L. The mixture was stirred for 3 h using an RT 15 power magnetic stirrer (Ika Werke, Germany). In order to measure the kinetic curves of sorption under similar conditions, weighed portions of the sorbent were placed into a series of test tubes, an aqueous solution of $SrCl_2{\cdot}6H_2O$ with a starting Sr^{2+} concentration of 1.22 mmol/L was added, and the tubes were shaken for 1 to 180 min.

Experiments of sorption of Sr^{2+} ions from fresh water mimicking solutions used in [21] (below referred to as FWM), mg/L : Sr^{2+}, 10–12; Ca^{2+}, 100; Mg^{2+}, 75; Na^+, 132; K^+, 15; Cl^-, 82; SO_4^{2-}, 650 (solution pH 7.6). The ratio of solid and liquid phases, S : L, was 1 : 40, 1 : 100, 1 : 400, 1 : 1000, or 1 : 2000.

The FWM solution was prepared using special purity grade potassium sulfate K_2SO_4 and sodium sulfate Na_2SO_4; analytical grade magnesium sulfate $MgSO_4{\cdot}7H_2O$; and reagent grade calcium chloride $CaCl_2$ and strontium chloride $SrCl_2{\cdot}6H_2O$.

Methods of analysis. X-ray diffraction patterns of the precipitates were measured on a D8 Advance automated diffractometer (Germany) with sample rotation in CuK_α radiation. Powder X-ray diffraction analysis was carried out using the EVA search software and PDF-2 powder database.

The specific surface area of samples was determined by low-temperature nitrogen adsorption using the Sorbtometr-M instrument (Russia).

Solution pH was measured on a Multitest IPL-102 pH-meter/ionometer with an ESK-10601/7 glass electrode (Russia) standardized against buffer solutions.

The contents of Sr^{2+} ions in the initial solutions and in the filtrates after sorption were determined by atomic absorption spectrometry (AAS) on a SOLAAR M6 two-beam spectrometer (Thermo Scientific, USA) using the 460.7 nm analytical lines. The limits of detection of strontium and calcium ions in aqueous solutions were 0.002 μg/mL and 0.0005 μg/mL, respectively. The error of determination of strontium in solutions in the concentration range of 0.001–10 mg/L was 20%.

Some of the samples were analyzed for Sr^{2+} content by inductively coupled plasma mass spectrometry on an Agilent 8800 spectrometer (Agilent Techn., USA).

The sorption capacity (A_c, mmol/g) of the sorbent samples was calculated by the relation

$$A_c = \frac{(C_{init} - C_{eq})}{m}V, \quad (1)$$

where C_{init} is the initial concentration of Sr^{2+} ions in the solution, mmol/L; C_{eq} is the equilibrium concentration of Sr^{2+} ions in the solution, mmol/L; V is the solution volume, L; m is the sorbent weight, g.

The recovery of Sr^{2+} ions (α, %) was calculated by the relation

$$\alpha = \frac{(C_{init} - C_{eq})}{C_{init}} \times 100\%. \quad (2)$$

RESULTS AND DISCUSSION

Sorbent Characteristics

Table 1 summarizes data on the phase composition and the specific surface area of the precipitate formed in this multicomponent system. More detailed data on the composition, morphology, and thermal behavior of the product in the given system were reported in [22].

As can be seen from Table 1, the composition of the precipitate indicates that the following reactions should be considered for this system:

$$SrCl_2 + CaSiO_3 + CO_2 \rightarrow SrCO_3 + SiO_2 + CaCl_2, \quad (3)$$

$$SrCl_2 + CaCO_3 \rightarrow SrCO_3 + CaCl_2, \quad (4)$$

$$2CaSiO_3 + 2SrCl_2 = Sr_2SiO_4 + 2CaCl_2 + SiO_2, \quad (5)$$

$$CaSiO_3 + CaCO_3 + 2SrCl_2 = Sr_2SiO_4 + 2CaCl_2 + CO_2, \quad (6)$$

$$CaSiO_3 + CaCO_3 + 2SrCl_2 + 2H_2O = 2Sr(OH)_2 + 2CaCl_2 + SiO_2 + CO_2, \quad (7)$$

$$SrCl_2 + CaSiO_3 \rightarrow SrSiO_3 + CaCl_2. \quad (8)$$

The Ca^{2+} and Sr^{2+} cation exchange can take place by equations (3) and (7); $\Delta G^\circ_{x.p}$ is –63.3 and –86.7 kJ, respectively. The $\Delta G^\circ_{x.p}$ value for reaction (3), possible under these conditions, was confirmed in our previous

Table 1. Phase composition of the products of synthesis in the $CaCl_2$–Na_2SiO_3–H_2O system

Sample	Phase composition	Specific surface area, m^2/g
After drying at 85°C	Amorphous phase, calcite $CaCO_3$	105.9
After annealing at 900°C	Wollastonite $CaSiO_3$, calcium oxide CaO	8.2

study by powder X-ray diffraction, X-ray photoelectron spectroscopy, and Raman spectroscopy [18].

However, the solubility products (reference and calculated data) known for compounds participating in reactions in this multicomponent system suggest the possible formation of strontium metasilicate and orthosilicate (Table 2).

Figure 1 shows the adsorption isotherms of Sr^{2+} ions from strontium chloride solution without a supporting salt at temperatures of 20, 40, and 60°C. It can be seen that the sorption capacity of this sorbent increases with increasing temperature. The most pronounced increase in the sorption capacity takes place at 60°C. The isotherms for 20 and 40°C barely differ, being within the error of determination of Sr^{2+} ions by atomic absorption spectroscopy.

The sorption properties were evaluated by analyzing the isotherms in the coordinates of the Langmuir equation and the empirical Freundlich equation.

Langmuir equation:

$$\frac{C_{eq}}{A_c} = \frac{1}{A_m k} + \frac{C_{eq}}{A_m}, \quad (9)$$

where C_{eq} is the equilibrium concentration of Sr^{2+} ions in solution, A_m is the maximum sorption capacity, k is the Langmuir constant.

The equation constants were calculated from the slope and intersection of straight lines in the plot of C_{eq}/A_c vs. C_{eq}.

The logarithmic form of the Freundlich equation was used to plot the linear dependence $\ln A_c$–$\ln C_{eq}$ and to determine K_F and n:

$$\ln a = \ln K_F + \frac{1}{n}\ln C_e, \quad (10)$$

where K_F is the equilibrium constant of the Freundlich equation related to the sorption capacity; $1/n$ is the parameter reflecting the sorbent–sorbate interaction intensity.

The parameters of the Langmuir and Freundlich equations found by the graphical method are summarized in Table 3.

It can be seen that sorption of Sr^{2+} ions with calcium silicate is described most adequately by the Freundlich equation, as evidenced by the correlation coefficients. The maximum sorption capacity A_m calculated by the Langmuir equation increases from 0.28 to 0.41 mmol/g as the temperature is raised.

Figure 2 shows the kinetic curves for the sorption of Sr^{2+} ions by calcium silicate at temperatures of 20, 40, and 60°C.

As can be seen in the dependences for various temperatures shown in Fig. 2, there are no significant changes in the kinetics of Sr^{2+} extraction; therefore, the kinetic curves are separated into two plots.

Figure 3 shows the experimental data on the sorption kinetics as logarithmic dependences according to the Kolmogorov–Erofeev equation after twice taking the logarithm:

$$\ln(-\ln(1 - \alpha)) = \ln k + n\ln\tau. \quad (11)$$

The linear dependences observed in the logarithmic coordinates attest to invariability of n in the given time intervals. The kinetic data processed in terms of the generalized topochemical equation are summarized in Table 4.

One can see that for the temperature range that is considered, the order of reaction n for the sorption of Sr^{2+} ions is less than unity, which is typical of reactions accompanied by diffusion processes.

The activation energy for the sorption of Sr^{2+} ions was determined graphically according to the Arrhenius equation using the $\ln k$ values found from the temperature dependences

$$\ln k = \ln A - Q/RT, \quad (12)$$

where Q is the activation energy; R is the gas constant (8.3145 J/(mol K)); T is temperature.

The activation energy of the process calculated from experimental data was 0.44 kJ/mol. This is rather low; therefore, the rate of the cation exchange reaction

Table 2. Solubility products of the starting compounds and reaction products in the $CaSiO_3$–$CaCO_3$–SiO_2–$SrCl_2$ system

Compound	Solubility product
Starting compounds	
$CaCO_3$	3.8×10^{-9}
$CaSiO_3$	7.4×10^{-11}
Reaction products	
$SrCO_3$	1.1×10^{-10}
Sr_2SiO_4	5.1×10^{-7}
$SrSiO_3$	1.0×10^{-13}
$Sr(OH)_2$	3.2×10^{-4}

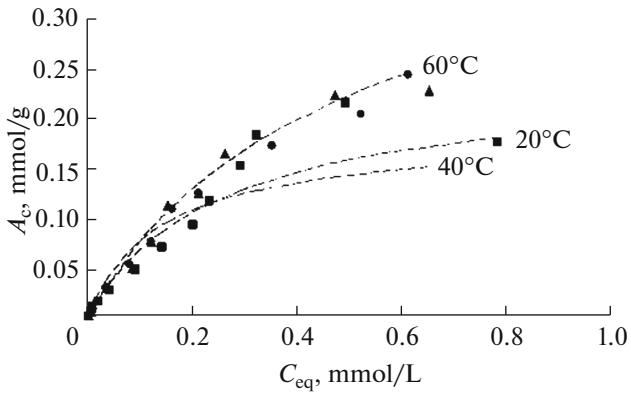

Fig. 1. Isotherms of Sr^{2+} sorption by calcium silicate.

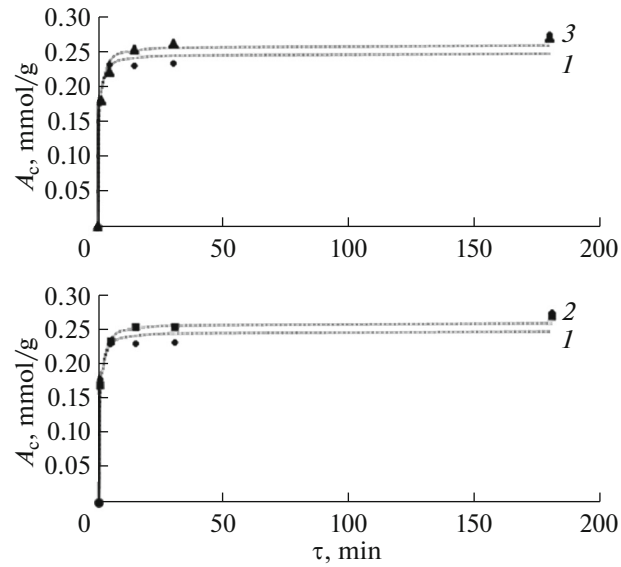

Fig. 2. Kinetic dependences of the sorption of Sr^{2+} ions by calcium silicate at various temperatures: (*1*) 20, (*2*) 40, (*3*) 60°C.

changes only slightly with temperature, which was confirmed experimentally.

The obtained kinetic data were analyzed according to the kinetic equation proposed in [23]:

$$\alpha_\tau = \alpha_m K\tau\left[\frac{1}{(1+k\tau)}\right], \quad (13)$$

where k is a constant (time^{-1}), α_τ is progress of the reaction at time point τ, α_m is the maximum progress of the reaction, τ is the reaction time.

The applied kinetic equation for topochemical reactions describes, with minor deviations, the sorption of metal ions by sorbents that act via ion exchange mechanism, the kinetics of formation of calcium hydrosilicates, and kinetics of sorption by microorganisms [24, 25].

Table 5 summarizes the parameters determined graphically upon plotting of experimental kinetics as the reciprocal of the reaction progress (in this case, the recovery of strontium) versus reciprocal of the reaction time. It can be seen that the proposed equation is suitable for describing the kinetics of these processes, as evidenced by the corresponding correlation coefficients.

For determination of the activation energy, the experimental ln$k$ values were presented as dependences on the reciprocal of the absolute temperature ($1/T$) (Fig. 4), which are described by the linear equation $y = 0.9x + 2.0267$ with the correlation coefficient $R^2 = 0.9959$.

The activation energy of sorption calculated from experimental data using the proposed equation was 7.5 kJ/mol. This value is more reliable, since the correlation coefficients found by analysis of experimental data are higher than those found using the Kolmogorov–Erofeev equation.

Table 6 gives the values for strontium recovery upon sorption from a solution without a supporting salt (distilled water) and from a FWM solution at different S : L phase ratios.

As can be seen from Table 6, the degrees of purification of aqueous solutions based on distilled water for S : L = 1 : 40, 1 : 100, and 1 : 400, were 96.8, 90.6, and 86.0%, respectively, which attests to efficiency of the used silicate sorbent under these conditions. The degree of purification of FWM solution was lower than that for solution based on distilled water. When the ratios of the solid and liquid phases were 1 : 40, 1 : 100, and 1 : 400, the degrees of purification were 70.4, 48.1,

Table 3. Parameters of the Langmuir and Freundlich equations for sorption of Sr^{2+} ions by calcium silicate

Temperature, °C	Langmuir equation parameters			Freundlich equation parameters		
	A_m, mmol/g	k, L/mmol	R^2	K_F, mmol/g (L/mmol)$^{1/n}$	$1/n$	R^2
20	0.28	3.07	0.8123	2.997	0.78	0.973
40	0.37	2.71	0.8591	2.48	0.7759	0.9902
60	0.41	2.1	0.9589	2.43	0.8214	0.9933

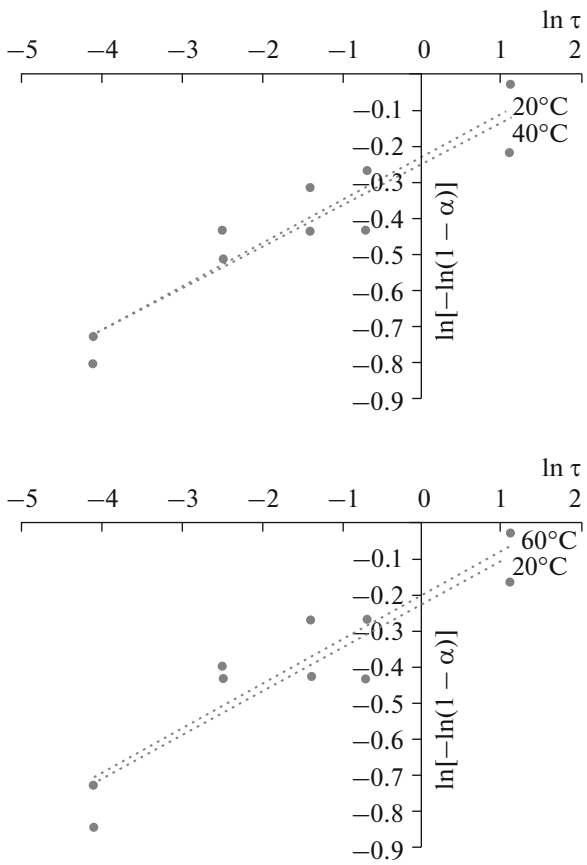

Fig. 3. Logarithmic dependences ln[−ln(1 − α)] vs. lnτ at various temperatures.

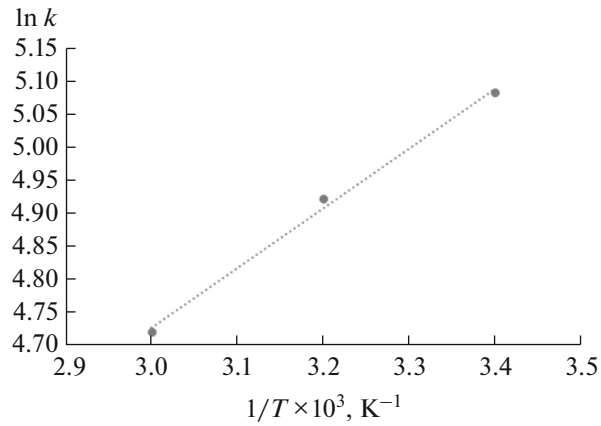

Fig. 4. Temperature dependence of the logarithm of the reaction rate constant ln*k* on the reciprocal of the absolute temperature for the sorption of Sr^{2+} ions.

and 55.6%, respectively, thus indicating that the supporting salt affects the process of sorption.

CONCLUSION

The study is concerned with the sorption properties of a material based on calcium hydrosilicate containing calcium carbonate with a surface area of 105.9 m^2/g, obtained in the $CaCl_2$–Na_2SiO_3–H_2O multicomponent model system, towards Sr^{2+} ions, in particular present in complex salt solutions.

It was found that the sorption capacity of the material increases with increasing temperature. The greatest increase in the sorption capacity is observed at 60°C.

Table 4. Order of the reaction (*n*) and rate constant (*k*) determined by analysis of the kinetic data in terms of the Kolmogorov–Erofeev equation

Temperature, °C	Equation parameters		
	n	ln*k*	R^2
20	0.1204	0.2282	0.8828
40	0.1143	0.2494	0.8689
60	0.1231	0.2042	0.817

It was shown that sorption of Sr^{2+} ions with calcium silicate is best described by the Freundlich equation, as evidenced by the correlation coefficients. The maximum sorption capacity A_m calculated using the Langmuir equation increases from 0.28 to 0.41 mmol/g with increasing temperature.

No significant changes in the Sr^{2+} sorption kinetics take place on temperature rise.

The activation energy of sorption calculated from experimental data is rather low (up to 7.5 kJ/mol), which implies a slight change in the cation exchange rate with temperature; this is also confirmed by experimental data.

The degrees of purification of aqueous solutions based on distilled water for solid to liquid phase ratios of 1 : 40, 1 : 100, and 1 : 400, were 96.8, 90.6, and 86.0%, respectively. The degree of purification of FWM solution was lower than that for solutions based on distilled water and did not exceed 70.4%.

ACKNOWLEDGMENTS

The analysis of solutions for the content of Sr^{2+} ions was performed on the equipment of the center for collective use "Far Eastern Center for Structural Research" of the Institute of Chemistry, Far Eastern Branch, Russian Academy of Sciences, and partly using the equipment of the center for collective use "Primorsky Center for Local, Elemental, and Isotopic Analysis" of the Far East Geological Institute, Far Eastern Branch, Russian Academy of Sciences.

FUNDING

This study was performed within the framework of the state assignment of the Institute of Chemistry, Far Eastern

Table 5. Data on the kinetics of sorption of Sr^{2+} ions by calcium silicate

Temperature, °C	Equation and its parameters		
	$y = ax + b$ (R^2)	k, min^{-n}	A_m, mmol/g
20	$y = 0.0252x + 4.0677$ (0.8875)	161.42	0.25
40	$y = 0.0283x + 3.8775$ (0.9373)	137.01	0.26
60	$y = 0.0341x + 3.817$ (0.9919)	111.9	0.262

Table 6. Recovery of strontium from solutions without a supporting salt (distilled water) and from a FWM solution

S : L	Solution without a supporting salt		FWM solution	
	Sr^{2+}, μg/mL	recovery of Sr^{2+} ions, (α), %	Sr^{2+}, μg/mL	recovery of Sr^{2+} ions, (α), %
Initial solution	10.86	–	10.8	–
1 : 40	0.35	96.8	3.2	70.4
1 : 100	1.02	90.6	5.6	48.1
1 : 400	1.52	86.0	4.8	55.6
1 : 1000	2.9	73.3	6.9	36.1
1 : 2000	3.5	67.8	7.55	30.1

Branch, Russian Academy of Sciences, no. FWFN(0205)-2022-0002, subject 2, section 3.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

AUTHOR CONTRIBUTIONS

S.B. Yarusova and P.S. Gordienko invented and designed the experiment; E.A. Nekhliudova, A.V. Perfilev, and A.N. Drankov synthesized the samples; O.O. Shichalin and E.K. Papynov participated in data processing; S.Yu. Budnitskii, N.V. Zarubina, and Yu.A. Parotkina carried out studies by methods of mass spectrometry with inductively coupled plasma and atomic absorption spectrometry. S.B. Bulanova carried out theoretical calculations; N.V. Ivanenko wrote the paper. All authors discussed the results and commented on the manuscript.

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Translated by Z. Svitanko