ISSN 0036-0244, Russian Journal of Physical Chemistry A, 2018, Vol. 92, No. 10, pp. 2053–2059. © Pleiades Publishing, Ltd., 2018.
Original Russian Text © O.N. Kononova, E.V. Duba, D.V. Medovikov, A.S. Krylov, 2018, published in Zhurnal Fizicheskoi Khimii, 2018, Vol. 92, No. 10, pp. 1641–1647.

PHYSICAL CHEMISTRY OF SEPARATION PROCESSES. CHROMATOGRAPHY

Ion-Exchange Sorption of Palladium(II) from Hydrochloric Acid Solutions in the Presence of Silver(I)

O. N. Kononova[a,*], E. V. Duba[a], D. V. Medovikov[a], and A. S. Krylov[b]

[a] *Institute of Nonferrous Metals and Materials Science, Siberian Federal University, Krasnoyarsk, 660025 Russia*
[b] *Kirensky Institute of Physics, Russian Academy of Sciences, Siberian Branch, Krasnoyarsk, 660036 Russia*
**e-mail: cm2@bk.ru*

Received October 12, 2017

Abstract—The sorptive extraction of palladium(II) chloride complexes in the presence of silver(I) chloride complexes from 2 and 4 M aqueous solutions of HCl with a series of Purolite anionites with different functional groups is studied. An anion exchange mechanism of sorption is identified from Raman spectroscopy data. Kinetic properties that are inherent to the investigated ionites and allow the elution of palladium(II) and silver(I) after their extraction in a dynamic mode are revealed.

Keywords: palladium, silver, hydrochloric acid solutions, sorption, sorptive extraction

DOI: 10.1134/S0036024418100138

INTRODUCTION

Due to the annual increase in the volume of processing poor or refractory ores containing noble metals, there is a need for the active use of such secondary sources of raw materials as spent catalysts (from the automobile and chemical industries), radioelectronic and capacitor scrap, and other technogenic wastes [1–3]. Palladium and silver are found together in such raw materials; they are present in small amounts, so the methodes involving sorption and providing selectivity, simplicity and environmental safety are used for their efficient extraction [1, 2, 4].

Industrial solutions containing noble metals are generally solutions of chloride complexes that differ in composition and chemical inertness [1, 2, 4–7]. It should also be noted that these are multicomponent solutions; i.e., along with complexes of noble metals, they contain iron(III) ions of and a number of nonferrous metals (nickel, cobalt, copper, and zinc). Accompanying ions are present in amounts considerably greater than those of noble metals, and industrial solutions themselves differ in the complexity of their composition and degrees of oxidation, along with the species of components they contain [1, 2, 6, 7].

Since sorption allows us to extract valuable components even in trace amounts and with a number of accompanying elements, it is understandable that many researchers are interested in these techniques. At the same time, a number of authors have focused on selective complexation-type ionites [5, 8–13]. However, the use of strongly basic anionites to concentrate noble metals remains relevant [11, 14–16].

It should be noted that both the ionic state of palladium in chloride and hydrochloric acid solutions and their sorptive extraction from these solutions have been studied in great detail [1, 2, 4–6, 8–19]. However, there are no published data concerning the sorptive extraction of palladium in the presence of silver chloride complexes, or on the sorption of these complexes, although their formation and thermodynamic properties were studied relatively long ago [20, 21]. In [22], we studied the sorption of silver chloride complexes from hydrochloric acid media onto different types of anionites (strongly and weakly basic, and complexation-type), and the anomalous character of ion-exchange equilibria was revealed in the studied systems. The aim of work was to study the ion-exchange extraction of palladium(II) and silver(I) chloride complexes under conditions of their simultaneous presence in solutions of hydrochloric acid.

EXPERIMENTAL

In our experiments, we used ionites with different functional groups synthesized by Purolite Ltd. Their physicochemical characteristics are described in Table 1. Before each experiment, the ionites were prepared according to conventional procedures and brought to their chloride form. The initial solution of palladium(II) with a concentration of 5.55 mmol/L was prepared by dissolving a precisely weighed amount of analytical grade $PdCl_2 \cdot 2H_2O$ in a small volume of concentrated HCl, followed by dilution with distilled water to 50 mL in a volumetric flask [17, 18]. Palla-

Table 1. Physicochemical characteristics of the investigated ionites based on styrene (St) and divinylbenzene (DVB)

Ionite	Type	Structure	FG	EC, mmol/g	H, %	α, %
Purolite A 500	Strongly basic anionite, type I	MP	QAB, type I	1.2	57–63	20
Purolite A 300	Strongly basic anionite, type II	G	QAB, type II	1.4	40–45	10
Purogold™ S 992	Complexation anionite	MP	MA	4.4	47–55	–
Purolite S 985	Complexation anionite based on PA–DVB	MP	PAG	2.3	52–57	20
Purolite A 110	Weakly basic anionite	MP	Primary AG	2.0	60–66	50
Purogold™ A 193	Mixed type anionite	MP	QAB, type I; TAG	3.8	46–56	22

FG is functional groups; EC is the exchange capacity of chloride ions; H is hygroscopicity; α is swelling capacity; MP is macroporous; G is gel; QAB is quaternary ammonium base; MA is mixed amines; PA is polyacrylate; PAG is polyamine groups; AG is amino groups; TAG is tertiary amino groups.

dium(II) solutions with concentrations of 0.025 and 0.25 mmol/L in 2 and 4 M HCl were used.

Stock solutions of silver(I) chloride complexes were prepared by dissolving precisely weighed samples of analytical grade $AgNO_3$ in 2 or 4 M HCl. After dissolution, we verified the completeness of complex formation by taking a small individual portion of the solution to conduct a staining reaction with potassium hydrogen orthophosphate (yellow coloring was observed for incomplete formation of the complex) [23]. The concentration of silver concentration in the initial solution (7.1 mmol/L) was determined by means of potentiometric titration with a silver indicator electrode and a silver chloride reference electrode [24]. We used solutions of chloride complexes with concentrations of silver ranging from 0.90 to 1.0 mmol/L.

Our choice of palladium and silver concentrations was based on our experimental conditions being close to those of industrial production during the extraction of noble metals from primary and secondary sources. Under these conditions, the initial concentrations of palladium are minimal, compared to those of silver and other competing ions [1, 2].

The concentrations of Pd(II) and Ag(I) in the contacting hydrochloric acid solutions were determined via spectrophotometry with Nitroso-R–Salt and Bromopyrogallol Red indicators, respectively [17, 18, 24]. Freshly prepared solutions of noble metals were used, and the ionic state of each was controlled by registering their absorption spectra in the 190 to 700 nm range of wavelengths using a Carl Zeiss Jena-Analytic Specol 1300 spectrophotometer.

The sorption of Pd(II) and Ag(I) ions from 2 and 4 M HCl solutions was conducted under static conditions at a 1 : 100 ratio of the solid and liquid phases (the weighed samples of ionites ranged from 0.1 to 0.2 g, while the volume of the contacting solution lay in the range of 10.0 to 20.0 mL) at a temperature of 20 ± 1°C. The time required to reach equilibrium was 24 h (as determined in a special experiment).

The sorptive capacity (SC) of the studied ionites was estimated from the exchange capacity, the level of palladium or silver extraction (R, %), and coefficients D of distribution and S of separation:

$$S = D_{Pd}/D_{Ag}, \quad (1)$$

where D_{Pd} and D_{Ag} are the coefficients of distribution for the sorption of palladium(II) and silver(I), respectively.

Sorption isotherms were built by varying the molar ratios between the amount of ionites and the content of Pd(II) and Ag(I) ions in the contacting solution [25, 26] of the resulting isotherms were used to calculate equilibrium coefficients ($\tilde{\tilde{K}}$) according to the law of acting masses [27, 28], in accordance with which the anion-exchange equilibrium can be written as

$$z_B\overline{A}^{z_{\overline{A}}} + z_A B^{z_{\overline{B}}} \leftrightarrow z_B A^{z_{\overline{A}}} + z_A \overline{B}^{z_{\overline{B}}}. \quad (2)$$

The coefficient of this equilibrium then takes the form

$$\tilde{\tilde{K}} = \frac{\overline{C}_B^{\,z_A} C_A^{\,z_B}}{\overline{C}_A^{\,z_B} C_B^{\,z_A}}, \quad (3)$$

where C_A and C_B are the molar concentrations of ions in the solution; $\overline{C}_A$ and $\overline{C}_B$ are the same values in the ionite; and z_A and z_B are the charges of the ions.

The Raman spectra of the investigated ionites were registered on a Bruker RFS 100/S Raman Fourier transform spectrometer. The spectra were obtained as a result of 300 scans in the 100 to 3500 cm^{-1} range of wave numbers. The kinetic properties of ionites were studied using the limited volume technique [25, 26, 29], and the kinetic parameters (average process speed, half-exchange time and diffusion coefficients) were calculated from the results.

In addition to static conditions, the combined sorption of Pd(II) and Ag(I) chloride complexes was studied with their subsequent elution in the dynamic mode using glass columns with diameters of ~1 cm. Preliminarily swollen in 4 M HCl, the ionites were placed in a column with a layer height of 1.5 cm;

Table 2. Sorptive extraction of simultaneously present palladium(II) and silver(I) from hydrochloric acid media (C_0(Pd) = 0.025 mmol/L; C_0(Ag) = 0.90 and 0.95 mmol/L in 2 and 4 M HCl, respectively)

Ionite	C_0(HCl), mol/L	Pd(II)		Ag(I)		S
		log D	R, %	log D	R, %	
Purolite A 500	4	1.23 ± 0.074	63 ± 4	1.36 ± 0.082	70 ± 4	0.74
	2	1.20 ± 0.072	61 ± 4	1.66 ± 0.099	82 ± 5	0.35
Purolite A 300	4	0.85 ± 0.051	40 ± 2	1.11 ± 0.067	56 ± 3	0.50
	2	2.42 ± 0.15	96 ± 4	1.38 ± 0.082	71 ± 4	11
Purogold™ S 992	4	0.78 ± 0.047	39 ± 2	1.36 ± 0.082	70 ± 4	0.31
	2	2.18 ± 0.11	94 ± 5	1.11 ± 0.067	56 ± 4	12
Purolite S 985	4	0.70 ± 0.042	33 ± 2	1.51 ± 0.091	76 ± 5	0.20
	2	2.29 ± 0.12	95 ± 5	2.03 ± 0.12	91 ± 6	1.8
Purolite A 110	4	1.11 ± 0.067	56 ± 4	1.18 ± 0.071	60 ± 4	0.87
	2	1.18 ± 0.071	60 ± 4	1.11 ± 0.067	56 ± 3	1.2
Purogold™ A 193	4	0.90 ± 0.054	44 ± 3	1.34 ± 0.081	69 ± 4	0.36
	2	1.20 ± 0.072	61 ± 4	1.53 ± 0.092	77 ± 5	0.47

100.0 mL of palladium and silver solutions were then passed through at a rate of 1 mL/min. The height of the layer and the rate of the solution passing through were chosen on the basis of special experiments. The outflowing filtrate was collected in 5.0–10.0 mL portions, each of which was used to determine the content of noble metal ions.

After the ionites were saturated with absorbed ions, palladium ions were eluted with a solution of thiourea in hydrochloric acid (80 g/L of thiourea in 0.5 M HCl), while silver was eluted using an 1% ammonia solution. The eluates were collected in 10.0 mL fractions, each of which was used to determine the concentrations of the extracted ions by means of spectrophotometry. The content of palladium was determined in a thiourea solution according to the intrinsic absorption of the complexes at a wavelength of 331 nm [30], while that of silver was determined using rubeanic acid (dithiooxamide) [24, 30].

All of our results were treated statistically. The average error for three parallel measurements did not exceed 6%.

RESULTS AND DISCUSSION

As noted above, the complexation of palladium in chloride and hydrochloric acid media was studied in detail by the authors of [1, 17–19]. Complexes predominated in solutions with concentrations of HCl ≥ 1 mol/L, $[PdCl_4]^{2-}$, since they were very stable. Their overall stability constant was estimated at $\log\beta_4$ = 11.12–12.24 [1, 17]. With regard to silver complexes, silver in the form of $[AgCl_2]^-$ was most likely present in our 2 and 4 M HCl solutions, according to the data presented in [20, 21, 31, 32]. In the absorption spectra of the initial Pd(II) and Ag(I) solutions in hydrochloric acid, the absorption maxima corresponded to reduced complex forms; i.e., they were consistent with the literature data.

We preliminarily studied the sorption properties of the investigated ionites for the extraction of Pd(II) and Ag(I) from individual hydrochloric acid solutions. It was found that the level of palladium(II) extraction from 2 and 4 M HCl solutions ranged from 81 to 99%, while that of silver(I) under the same conditions ranged from 57 to 99%. At the same time, a change in the acidity of the medium had virtually no effect on the sorption results for most of the ionites. It was therefore of interest to study the extraction of noble metals when they were present together in hydrochloric acid solutions. The results are given in Table 2.

As can be seen from the presented data, the level of palladium(II) extraction in the presence of silver(I) for most of the investigated ionites depended on the acidity of the medium (it rose as the concentration of HCl fell). Such differences were displayed especially sharply by complex-forming Purolite S 985 and Purogold™ S 992 anionites, and by the strongly basic II-type Purolite A 300 anionite. The dependence on the HCl concentration was not as pronounced as for other sorbents. This behavior of ionites could be explained by a reduction in the competing effect of chloride ions in 2 M HCl, compared to a 4 M solution. With regard to the sorptive concentration of silver(I) chloride complexes in the presence of palladium(II), their levels of extraction generally remained the same as for sorption from individual solutions, though the acidity of the contacting solutions was more pronounced. It should be noted that the mutual influence of Pd(II) and Ag(I) chloride complexes was exhibited in the form of decreasing sorption parameters in extracting palladium(II) from 4 M HCl solutions.

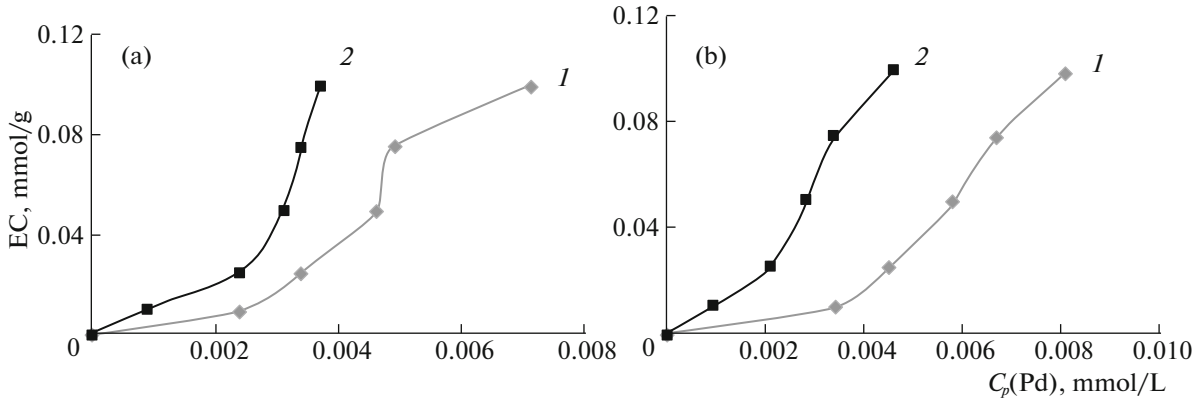

Fig. 1. Sorption isotherms for the absorption of palladium(II) chloride complexes by anionites in the presence of silver(I) chloride complexes: (a) Purogold™ A 193 and (b) Purolite A 500 at concentrations of HCl (*1*) 2 and (*2*) 4 mol/L.

Table 2 shows the coefficients of separation for palladium and silver, calculated for their joint sorption, the values of which also confirm the higher sorptive capacity of the investigated ionites with respect to silver(I) chloride complexes in 4 M HCl ($S < 1$). This can apparently be explained by the competing influence of chloride ions, and by the anomalous sorption of silver(I) chloride complexes, which we revealed in [22]. When the concentration of HCl was reduced to 2 mol/L, the sorptive capacity for most of the studied anionites with respect to chloride complexes of palladium showed a rise that was apparent from an increase in the distribution coefficients.

Sorption isotherms of palladium(II) chloride complexes in the presence of silver(I) were then obtained for the investigated ionites. These curves are presented in Fig. 1 for mixed-type Purogold™ A 193 anionite and strongly basic Purolite A 500 anionite. It is known [25, 26, 29] that the shape of sorption isotherms for binary systems can be used to judge the selectivity of ionites. If the ion-exchanger preferentially absorbs the ions of the contacting solution, the curves have a convex shape. In the case of ionite selectivity with respect to ions initially contained in the phase of ionite, the isotherms are concave. Linear isotherms are observed when there is equal selectivity. In our case, the investigated system contained palladium and silver complex chlorides, along with chloride ions. Since the concentration of the last was much higher than that of noble metal complexes, the system appeared as if there were only these ions at the beginning of the sorption process. This corresponded to the linear region of the isotherms shown in Fig. 2 [25].

As the process proceeded, however, the presence of other ions undoubtedly affected the exchange equilibrium of palladium(II) chloride complexes. This influence can be apparent from such factors as a change in the Donnan potential and the swelling pressure, the direct impact of ion-exchange participants, different degrees of polyfunctional ionite dissociation, and electrical selectivity [25, 33] that alters the shape of sorption isotherms from the one characteristic of binary systems. It should be noted that ion-exchange equilibria in multicomponent systems remain poorly studied.

Based on the obtained sorption isotherms, we calculated the equilibrium coefficients presented in Table 3. It can be seen from these data that the studied ionites have a different degree of affinity with respect to palladium(II). It is known that for binary systems, the selectivity of ionites with respect to the ions being extracted results from the values of the equilibrium coefficients being less than unity [25, 33]. The values of $\tilde{\tilde{K}}$ we calculated for the investigated ionites during Pd(II) extraction from individual solutions ranged from 0.37 to 0.68 for 2 M HCl and from 0.13 to 0.94 for 4 M HCl. As can be seen from Table 3, the values of the equilibrium coefficients in the ternary system differ greatly from those for the binary system. However, since the concentrations of competing chloride ions and silver(I) chloride complexes far exceed that of palladium(II), the ionites nevertheless exhibit an affinity for palladium, as can be judged from data presented in Table 2.

The mechanism of ion-exchange processes that occur during the sorption of Pd(II) and Ag(I) chloride complexes was also studied by means of Raman spectroscopy. Raman spectra for the strongly basic Purolite A 500 anionite, and for the complexation Purolite S 985 sorbent, are shown in Fig. 2.

The Raman spectra of Purolite A 500 anionite exhibit a band in the range of 275–303 cm^{-1} (Fig. 2a, spectrum *2*) and a peak at 268 cm^{-1} (spectrum *3*)—as opposed to the spectrum of the ionite in the initial form (spectrum *1*)—correspond to the vibrations of Me–Cl bonds of Pd(II) and Ag(I) chloride complexes in the phase of the sorbent, indicating anion exchange between the counterions of anionite and the chloride complexes of noble metals [34, 35].

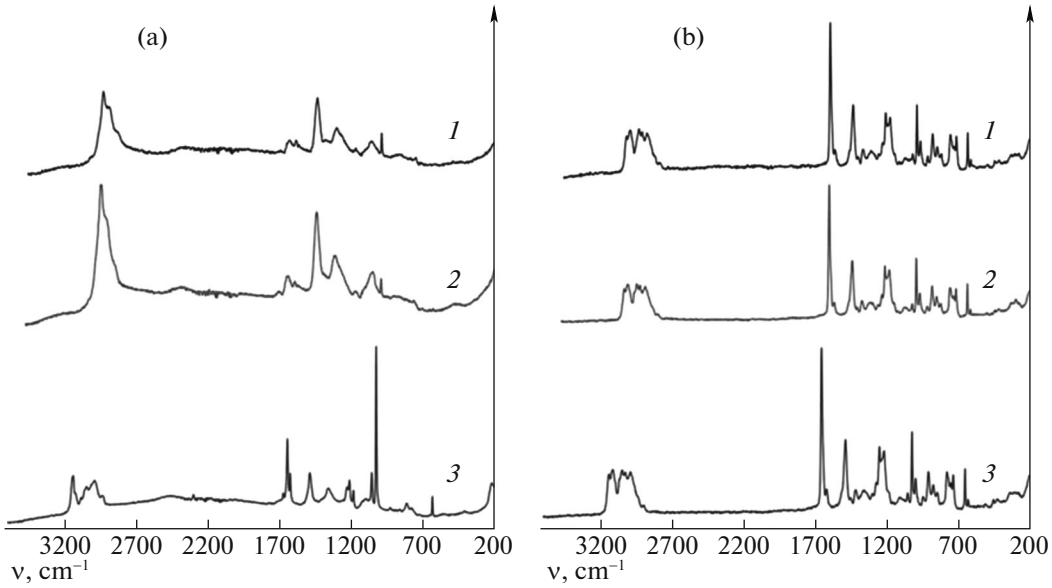

Fig. 2. Fragments of Raman spectra for anionites: (a) Purolite S 985 and (b) Purolite A 500; (*1*) in the initial Cl^- form; (*2*) saturated with Pd(II) solution; (*3*) saturated with Ag(I) solution; C_0(HCl) = 4 mol/L; C_0(Pd) = 0.25 mmol/L; C_0(Ag) = 0.95 mmol/L.

Similar changes were also observed in the Raman spectra of Purolite S 985 anionite (Fig. 2b). It should be noted, however, that when the spectrum of the ionite saturated with silver chloride complexes is compared (Fig. 2b, spectrum *3*) to the spectrum of the initial sample (spectrum *1*), we observe changes in the intensity of peaks and new bands that appear in the ranges of 900–1000, 2845–2870, 2900–2915, 2860–2885, and 2950–2975 cm^{-1} that characterize changes in the alkyl groups of the polymeric chain [36]. This indicates that the ionite matrix of Purolite S 985 undergoes deformation as a result of the anomalous sorption of silver chloride complexes [22].

In the investigated systems, the sorptive extraction of Pd(II) and Ag(I) chloride complexes thus proceeds according to the anion-exchange mechanism

$$m\overline{RCl} + [MeCl_n]^{m-} \leftrightarrow \overline{R_m[MeCl_n]} + mCl^-,$$

where Me = Pd; Ag; $n = 4$ (for Pd) and 2 (for Ag); m = 2 (for Pd) and 1 (for Ag).

We also studied the kinetic properties of the investigated anionites. An intradiffusion type of kinetics in agreement with the classical Boyd model [25, 26, 29] was preliminarily observed using the interruption technique, after which we calculated kinetic parameters presented in Table 4. It can be seen from these data that the ionites have excellent kinetic properties with respect to palladium(II) even in the presence of competing chloride ions and silver(I) chloride complexes.

Because industrial sorption processes proceed mainly under dynamic conditions, it is of interest to investigate the sorption properties of anionites under dynamic conditions. Strongly basic Purolite A 500 anionite and complexation-type Purolite S 985 sorbent were chosen for this, due to their superior kinetic properties (Table 4). For dynamic sorption, the concentration of HCl was set at 4 mol/L for the experimental conditions to ensure the best match to the conditions of industrial production.

The output curves for the example of Purolite A 500 anionite are shown in Fig. 3; we can see that the presence of silver(I) chloride complexes has virtually no effect on the sorptive concentration of palladium(II) chloride complexes under dynamic conditions. The output curves have a similar shape for Purolite S 985 anionite; i.e., the shape does not depend on

Table 3. Equilibrium coefficients ($\tilde{\tilde{K}}$) for the sorptive extraction of palladium(II) in the presence of silver(I) from hydrochloric acid media

Ionite	2 M HCl	4 M HCl	Ionite	2 M HCl	4 M HCl
Purolite A 500	16 ± 1	21 ± 1	Purolite S 985	8.4 ± 0.5	7.8 ± 0.4
Purolite A 300	14 ± 1	17 ± 1	Purolite A 110	6.2 ± 0.4	16 ± 1
Purogold™ S 992	14 ± 1	5.1 ± 0.3	Purogold™ A 193	5.4 ± 0.3	7.8 ± 0.4

Table 4. Kinetic characteristics of the studied anionites during palladium(II) sorption in the presence of silver(I) in hydrochloric acid (C_0(Pd) = 0.025 mmol/L; C_0(Ag) = 0.90 and 0.95 mmol/L in 2 and 4 M HCl, respectively)

Ionite	2 M HCl			4 M HCl		
	$D_S \times 10^6$, cm^2/s	$t_{1/2}$, s	$\bar{v} \times 10^5$, mmol/(g s)	$D_S \times 10^6$, cm^2/s	$t_{1/2}$, s	$\bar{v} \times 10^5$, mmol/(g s)
Purolite A 500	2.3	7.0	17.3	2.4	6.6	17.7
Purolite A 300	3.8	6.0	17.7	1.6	14.3	7.9
Purogold™ S 992	51	5.4	18.0	33	8.5	4.3
Purolite S 985	2.9	5.4	17.3	1.8	8.6	13.7
Purogold™ A 193	42	6.6	18.6	40	7.0	16.4

D_S is the coefficient of internal (gel) diffusion; $t_{1/2}$ is the half-exchange time; $\bar{v}$ is the average rate of the process.

the type of functional groups characteristic of the sorbents. This is apparently due to the considerable competing effect of the chloride ions. The level of extraction for palladium(II) chloride complexes in a sorption cycle under dynamic conditions is 69 and 82% for Purolite A 500 and Purolite S 985, respectively, which considerably exceeds the data obtained under static conditions (Table 2).

It is known [36] that the more selective an ionite is with respect to extracted ions, the more difficult it is to desorb these ions from this ionite. This rule was confirmed by our data on the elution of precious metals. The desorption of silver thus proceeds at levels of 95 and 99% for Purolite S 985 and Purolite A 500, respectively, while palladium can be extracted at levels of 73 and 31%, respectively, with the same ionites. However, despite the seemingly low degree of palladium desorption from Purolite A 500 anionite, this level of extraction may be considered acceptable for a single sorption–desorption cycle. For this sorbent to be used under the conditions of industrial production, it would seem several cycles should be performed; this will be the subject of a special study.

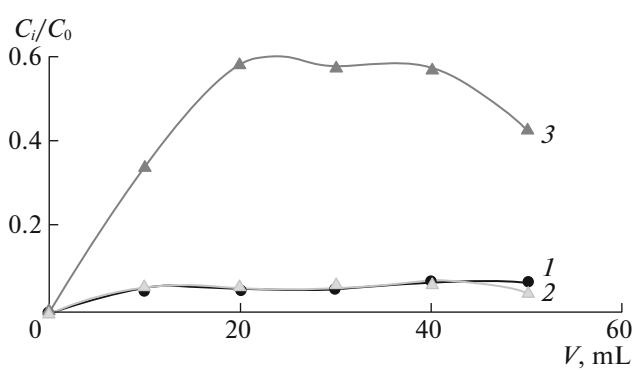

Fig. 3. Fragments of output curves for the sorption of noble metal chloride complexes by Purolite A 500 anionite from solutions with an HCl concentration of 4 mol/L: (*1*) sorption of Pd(II) from individual solutions; (*2*) sorption of Pd(II) in the presence of Ag(I); (*3*) sorption of Ag(I) in the presence of Pd(II); C_0(Pd) = 0.025 mmol/L; C_0(Ag) = 0.95 mmol/L.

CONCLUSIONS

Based on our results, such complex-forming anionites as Purolite S 985 may be recommended for use in technological schemes with the simultaneous extraction of Pd(II) and Ag(I) from hydrochloric acid solutions.

ACKNOWLEDGMENTS

The authors are grateful to the Moscow representative office of Purolite Ltd., and to M.A. Mikhailenko personally, for supplying the ionite samples for our investigations.

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Translated by O. Polyakov