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Ion Exchange Recovery of Platinum(IV) from Hydrochloric Acid Solutions in the Presence of Silver(I)

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Abstract—The ion exchange sorption of the platinum(IV) chloride complexes in the presence of silver(I) chloro complexes from 2 and 4 M aqueous solutions of hydrochloric acid is studied on Purolite anion exchangers with different functional groups. The high selectivity of the investigated sorbents with respect to platinum during its recovery from individual hydrochloric acid solutions and in the presence of silver chloride complexes is determined. Since the latter are in this case virtually not sorbed in the presence of platinum(IV), they can be separated at the stage of sorption. The excellent kinetic properties of the investigated anion exchangers are revealed, allowing platinum to be eluted after its separation from silver chloro complexes in a dynamic mode.

Keywords: platinum, silver, hydrochloric solutions, anion exchangers, sorption **DOI:** 10.1134/S003602442004007X

INTRODUCTION

The consumption of precious metals is steadily growing. Raw material resources can be replenished through both the discovery and development of new deposits, and the wider use of different secondary raw materials and waste [1, 2].

In view of the above, newly discovered deposits of platinum group metals containing a considerable amount of related metals (particularly silver) on an industrial scale, electronic waste scrap, and the great volume of spent refining and washing solutions require processing for the extraction and separation of recoverable components [1-7].

Numerous problems in the field of recovering precious metals from different technological solutions create a many opportunities for applying sorption techniques that are simple, selective, environmentally friendly, and can be used to process great volumes of solutions without changing their composition [1, 2, 4, 6-9]. However, it is still important to seek high-efficiency sorbents for these purposes.

It is well known [1, 4, 7, 10] that commercial solutions of platinum metals are mainly chemically inert chloride complexes of different compositions that are characterized by a diversity of oxidation states and component forms. The use of selective ion exchangers for the concentration of precious metals is therefore of great importance, since such sorbents can recover valuable components even in trace amounts and in the presence of related elements, the concentrations of which sometimes exceed those of precious metals by several orders of magnitude [1, 2, 4, 6, 9, 11-15].

It should be noted that the ionic state of platinum in hydrochloric acid and chloride media was thoroughly investigated in [1, 10, 16–19]. However, there is a lack of literature data on the sorption concentration of platinum from hydrochloric acid solutions in the presence of silver(I), in which the latter takes the form of chloride complexes. The only study of platinum and palladium sorption from nitric acid solutions of silver so far is [20]. In [21, 22], we investigated the sorption of silver(I) from hydrochloric acid media on anion exchangers and the recovery of palladium(II) in the presence of silver(I) from these solutions. We established the anomalous character of ion exchange equilibria in the systems' anion exchanger-silver(I)-HCl complexes, which affects the sorption of palladium(II). The aim of this work was to investigate the ion exchange recovery of platinum(IV) and silver(I) chloride complexes present simultaneously in hydrochloric acid solutions.

EXPERIMENTAL

Our objects of study were Purolite ion exchangers with different functional groups. The physicochemical properties of these media are given in Table 1. After

Ion exchanger	Туре	Physical structure	Functional groups	Q _{Cl} , mmol/g	Н, %	A, %
Purolite A500	Type-I strongly basic anion exchanger	MP	QAB, type I	1.2	57-63	20
Purolite A300	Type-I strongly basic anion exchanger	G	QAB, type II	1.4	40-45	10
Purogold TM S992	Complexing anion exchanger	MP	MA	4.4	47-55	—
Purolite S985	PA-DVB-based complexing anion exchanger	MP	PAG	2.3	52-57	20
Purolite A110	Weakly basic anion exchanger	MP	Primary AG	2.0	60-66	50
Purogold [™] A193	Mixed-type anion exchanger	MP	QAB, type I; TAG	3.8	46-56	22

 Table 1. Physicochemical characteristics of the investigated St–DVB-based ion exchangers

Styrene St, divinylbenzene DVB, microporous MP, gel G, quaternary ammonium base QAB, mixed amines MA, polyacrylate PA, polyamine groups PG, amine groups AG, tertiary amine group TAG, chloride ion exchange capacitance Q_{Cl} , hygroscopicity H, and swelling capacity A.

appropriate preparation by conventional techniques, the sorbents were converted to chloride form.

The initial platinum(IV) solution with a concentration of 10.76 mmol/L was prepared by dissolving an exact weight of metallic platinum in a small amount of aqua regia. It was then evaporated to wet salts by alternately adding concentrated HCl and distilled water for three times. The residue was dissolved in 6 M HCl, transferred to a 50-mL graduated flask, and adjusted to the mark with the same acid [16, 17]. Freshly prepared platinum(IV) solutions with concentrations of 0.0025–0.25 mmol/L in 2 and 4 M HCl were used.

The initial solutions of the silver(I) chloride complexes were prepared by dissolving accurate amounts of AgNO₃ (analytical grade) in 2 or 4 M HCl. The procedures for preparing these complexes were described in detail in [21, 22]. In this work, we used solutions of chloride complexes with a silver concentration of 0.90-1.0 mmol/L.

The Pt(IV) and Ag(I) concentrations in the contacting hydrochloric acid solutions were determined spectrophotometrically using an $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ solution and bromopyrogallol red, respectively [16, 17, 23]. The ionic state of the precious metals in the investigated solutions was controlled by recording the absorption spectra in the range of 190–800 nm on a Carl Zeiss Jena-Analytic Specol 1300 spectrophotometer.

The initial platinum and silver concentrations and the medium acidity were chosen by approximating the experimental conditions to the production conditions of recovery of precious metals from primary or secondary sources [1, 2, 4].

The sorption of Pt(IV) and Ag(I) ions from hydrochloric acid media was conducted under static conditions at a ratio of 1 : 100 between the solid and liquid phases (the ion exchanger weights were 0.1-0.2 g and the contacting solution volume was 10.0-20.0 mL) and a temperature of $(20 \pm 1)^{\circ}$ C. The equilibrium determined in a separate experiment was established in 24 h.

The sorption capacity of the investigated ion exchangers was determined using exchange capacity EC, degree R of extraction of precious metals (%), distribution coefficients D, and separation coefficients S:

$$EC = \frac{(C_0 - C_p)V}{g},\tag{1}$$

$$R = \frac{(C_0 - C_p) \times 100\%}{C_0},$$
 (2)

$$D = \frac{EC}{C_p},\tag{3}$$

$$S = \frac{D_{\rm Pt}}{D_{\rm Ag}},\tag{4}$$

where C_0 is the platinum or silver concentration in the initial solution (mmol/L), C_p is the same in the equilibrium solution, V is the solution volume (L), g is the ion exchanger weight (g), and D_{Pt} and D_{Ag} are the platinum(IV) and silver(I) distribution coefficients, respectively.

The sorption isotherms were built by varying the molar ratios between ion exchangers and the amount of Pt(IV) or Ag(I) ions in the contacting solution [24,

25]. Using the obtained isotherms, coefficients \tilde{K} of equilibrium were calculated according to the law of mass action [26, 27], with which the anion exchange equilibrium can be written as

$$z_B \overline{A}^{z_{\overline{A}}} + z_A B^{z_{\overline{B}}} \leftrightarrow z_B A^{z_{\overline{A}}} + z_A \overline{B}^{z_{\overline{B}}}.$$
 (5)

The coefficient of equilibrium then takes the form

$$\tilde{\tilde{K}} = \frac{\overline{C_B}^{z_A} C_A^{z_B}}{\overline{C_A}^{z_B} C_B^{z_A}},\tag{6}$$

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 quantities in the ion exchanger, and z_A and z_B are the ion charges.

Raman spectra of the investigated sorbents were recorded on a Bruker RFS 100/S Raman Fourier spectrometer. The spectra were obtained with 300 scans in the range of 100-3500 cm⁻¹. The kinetic properties of ion exchangers were studied using the limited volume technique [24, 25, 28]. The 0.1-g ion exchanger weights were mixed with 10.0 mL of the platinum solution (individual solutions or in the presence of silver) at $(20 \pm 1)^{\circ}$ C in periods of 1 min to 24 h. The periods of saturation were 1, 2, 3, 5, 15, 30, and 45 min; 1, 2, 3, 6, and 24 h. The suspensions were vigorously stirred at 800 rpm. After a certain contact time, the ion exchangers and the solution were quickly separated on a porous glass filter and the platinum (silver) concentration in the solutions was determined spectrophotometrically. Using the results, the degree of exchange F was calculated as

$$F = \frac{Q_t}{Q_{\infty}},\tag{7}$$

where Q_t and Q_{∞} are the amounts of platinum and silver sorbed in time *t* and after reaching equilibrium (mmol), respectively.

Kinetic curves F = f(t) were built and used to determine exchange half-time $t_{1/2}$ (s) at F = 0.5. Diffusivity D_s (cm²/s) was calculated as

$$D_s = \frac{r^2}{4\pi^2 t_{1/2}},$$
 (8)

where *r* is the ionite grain radius (cm).

The kinetic data were interpreted according to the classical Boyd model [24, 28] using the formal kinetic coefficient

$$B = \frac{(1.08)^2 F^2}{t}.$$
 (9)

If the process is controlled by the helium diffusion, function Bt = f(t) should be linear.

In addition to the static conditions, we studied the sorption of Pt(IV) and Ag(I) chloride complexes and then their elution using glass columns ~ 1 cm in diameter in the dynamic mode. Ion exchangers pre-swollen in HCl with the desired concentration were placed in the column to a layer height of 1.5 cm and 100.0 mL of the platinum and silver solutions were passed at a rate of 1 mL/min. The layer height and the rate of solution transmission optimal under the specified conditions were established in separate experiments. The effluent filtrate was collected in portions of 5.0–10.0 mL and the content of precious metal ions in each one was determined.

After saturating the ion exchangers with sorbed ions, platinum ions were eluted with a hydrochloric acid solution of thiourea (1 M solution in 0.5 M HCl) and silver ions, with a 1% ammonia solution. The eluates were collected in 10.0-mL fractions and the concentrations of the extracted ions in each one were determined spectrophotometrically: the concentration of platinum was determined with thiourea solution from the intrinsic light absorption of the complexes at a wavelength of 309 nm [29]; the concentration of silver, with rubyenoic acid [23, 29].

All results were processed statistically. The average error for three parallel results was no larger than 6%.

RESULTS AND DISCUSSION

It is well known that the ionic state of platinum metals in hydrochloric acid solutions depends on the acidity of the medium, the concentration of chloride ions, and the temperature [1, 16-19]. In solutions with HCl concentrations of 3 mol/L and more, the $[PtCl_6]^{2-}$ form dominates. At $0.1 < C_{HCl} < 3$ M, there is $[Pt(H_2O)Cl_5]^-$ complex along with hexachloroplatinate(IV). Both forms are highly stable and chemically inert [1, 18, 19]. The constant of the cumulative stability of the $[PtCl_6]^{2-}$ ions was estimated to be $\log \beta_6 = 33.9$ [1, 17]. The stepwise stability constants of the $[Pt(H_2O)Cl_5]^-$ complex at a temperature of 25°C and a solution ionic force of 0.5–0.8 are $\log K_5 = 3.70$ – 5.60 [1, 19]. With silver ions, it most likely existed in the form of $[AgCl_2]^-$ complex in the investigated solutions of 2–4 M HCl [30, 31]. The constant of the cumulative stability of this complex is estimated to be $\log \beta_2 = 5.04$ [32]. In the absorption spectra of the initial hydrochloric acid solutions of Pt(IV) and Ag(I), the absorption maxima correspond to the given complexes; i.e., they are consistent with the literature data.

We earlier studied the sorption properties of the investigated ion exchangers at the recovery of platinum(IV) and silver(I) from individual solutions. It was found that anion exchangers exhibit excellent sorption characteristics in the recovery of platinum (the degree of recovery is more than 95%), regardless of the initial HCl concentration. Under the same conditions, the recovery of silver chloride complexes proceeds at a level of 80–90%. It depends somewhat on the acid concentration in the contacting solution, and grows in 2 M HCl. This is explained by the weakening of the competitive effect of chloride ions.

The sorption properties of the investigated ion exchangers were studied by extracting platinum(IV) in the presence of silver(I) chloride complexes. The results are presented in Table 2. The data show that anion exchangers virtually retain their sorption characteristics relative to platinum, since the degree of its recovery remains at a level of 95%. No more than 4% of the silver is in this case recovered in the presence of platinum, which corresponds to the error of spectrophotometric analysis [23]. This behavior of ion

Ion exchanger	C ₀ (HCl), mol/L	Platinum(IV)		Silver(I)		S
		$\log D$	<i>R</i> , %	$\log D$	<i>R</i> , %	6
Purolite A500	4	2.44 ± 0.15	97 ± 3	-0.40	4.1	690
	2	2.59 ± 0.16	98 ± 2	-0.40	4.2	983
Purolite A300	4	2.55 ± 0.15	97 ± 3	-0.39	4.3	903
	2	3.33 ± 0.21	99 ± 1	-0.38	4.3	5383
Purogold [™] S992	4	2.31 ± 0.14	95 ± 5	-0.38	4.3	184
	2	2.84 ± 0.17	98 ± 2	-0.39	4.3	1738
Purolite S985	4	2.32 ± 0.14	95 ± 5	-0.38	4.2	180
	2	2.58 ± 0.16	97 ± 3	-0.38	4.3	940
Purolite A110	4	2.36 ± 0.14	96 ± 4	-0.39	4.3	650
	2	2.48 ± 0.15	97 ± 3	-0.39	4.3	752
Purogold [™] A193	4	2.33 ± 0.14	96 ± 4	-0.39	4.3	530
	2	2.51 ± 0.15	97 ± 3	-0.39	4.2	805

Table 2. Sorption recovery of platinum(IV) and silver(I) at the simultaneous presence of hydrochloric acid media ($C_0(Pt) = 0.25 \text{ mmol/L}$; $C_0(Ag) = 0.90$ and 0.95 mmol/L in 2 and 4 M HCl, respectively)

exchangers is clearly related to the pronounced selectivity toward platinum chloride complexes, which can be explained by the substantial differences between the stability of the $[PtCl_6]^{2-}$, $[Pt(H_2O)Cl_5]^-$, and $[AgCl_2]^$ chloride complexes. As is known from the theory of selectivity, anion exchangers have higher absorption capacities for more stable complexes [33, 34]. The acidity of the contacting solution in this case has virtually no affect the sorption ability of ion exchangers. The coefficients of separation of platinum and silver in the HCl solutions (Table 2) have such high values (far above unity), suggesting that the separation of platinum(IV) chloride complexes from silver(I) ones is complete at the stage of sorption. The sorption isotherms of the Pt(IV) chloride complexes from individual solutions and in the presence of Ag(I) on the investigated anion exchangers were obtained next. It follows from the isotherms presented in Fig. 1 using the example of a PurogoldTM A193 mixed-type anion exchanger that they have the form of convex curves, which remains virtually unchanged in the presence of silver(I) chloro complexes.

Using the obtained sorption isotherms, we calculated the equilibrium coefficients given in Table 3. According to the general theory of ion exchange equilibrium [24, 25, 33, 34], these coefficients are quantitative characteristics of the selectivity of ion exchang-



Fig. 1. (Color online) Isotherms of sorption of platinum(IV) on a PurogoldTM A193 anion exchanger (a) from individual solutions and (b) in the presence of silver(I) at HCl concentrations of (1) 2 and (2) 4 mol/L.

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Ion exchanger	Individual Pt	(IV) solutions	Pt(IV) in the presence of $Ag(I)$		
	2 M HCl	4 M HCl	2 M HCl	4 M HCl	
Purolite A500	0.42 ± 0.025	0.96 ± 0.058	1.23 ± 0.074	2.15 ± 0.13	
Purolite A300	0.18 ± 0.011	0.47 ± 0.028	0.27 ± 0.018	0.58 ± 0.035	
Purogold [™] S992	0.21 ± 0.013	0.45 ± 0.027	0.16 ± 0.011	0.41 ± 0.025	
Purolite S985	0.37 ± 0.022	0.84 ± 0.051	0.28 ± 0.017	0.30 ± 0.018	
Purolite A110	0.62 ± 0.037	0.74 ± 0.044	0.76 ± 0.046	0.91 ± 0.055	
Purogold [™] A193	0.31 ± 0.014	0.63 ± 0.032	0.40 ± 0.024	0.43 ± 0.028	

Table 3. Equilibrium coefficients during sorption recovery of platinum(IV) from individual hydrochloric acid solutions and in the presence of silver(I)

ers. For binary systems, the equilibrium coefficients are less than unity [24, 34], as is the case when platinum is recovered from individual hydrochloric acid solutions. It should be noted that the presence of silver(I) chloride complexes in the investigated systems has no great effect on the selectivity of platinum(IV) sorption; i.e., the equilibrium coefficients remain less than unity. The only exception is the strongly basic Purolite A500 anion exchanger, which has equilibrium coefficients that are more than unity. As follows from Table 2, however, this ion exchanger also exhibits the high selectivity during the recovery of platinum (R = 97-98%), sorbing silver chloro complexes at the level of the experimental error.

The ion exchange processes occurring at the sorption of Pt(IV) and Ag(I) chloride complexes were studied via Raman spectroscopy. Raman spectra for the complexing Purolite S985 sorbent and the strongly basic Purolite A500 anion exchanger are presented in Fig. 2.

It is interesting that in the Raman spectra of both ion exchangers after their saturation with platinum, two peaks emerge at 317 and 342 cm⁻¹, respectively. These correspond to the vibrations of the $[PtCl_6]^{2-}$ and $[PtCl_4]^{2-}$ ions in the ion exchanger phase absorbed as a result of ion exchange [35]. Platinum thus exists in the form of Pt(IV) and Pt(II) chloride complexes in the phase of the investigated ion exchangers. The role of functional groups and matrices of sorbents in this process is a subject for separate study.

We found that anion exchange is the main mechanism of platinum sorption on the investigated ion exchangers:

$$mRCl + [MeCl_n]^{m-} \leftrightarrow R_m[MeCl_n] + mCl^{-}$$

where Me = Pt(II), Pt(IV), n = 4 (for Pt(II)), and 6 (for Pt(IV)), m = 2.

To solve practical problems, we must know the kinetic properties of ion exchangers. We therefore studied the kinetics of platinum sorption from individual solutions in the presence of silver chloride complexes and established the intradiffusion type of kinet-



Fig. 2. (Color online) Fragments of Raman spectra of (a) Purolite S985 and (b) Purolite A500 anion exchangers (1) in the initial chloride form and (2) saturation with the platinum(IV) solution; $C_0(\text{HCl}) = 4 \text{ mol/L}$; $C_0(\text{Pt}) = 0.5 \text{ mmol/L}$.



Fig. 3. (Color online) Dependences of the formal kinetic criterion on the sorption time at the recovery of platinum(IV) (1-3) from individual solutions and (4-6) in the presence of silver(I) on (1, 4) Purolite A500, (3, 5) Purolite S985, and PurogoldTM A193 (2, 6) ion exchangers.

ics according to the classical Boyd model [24, 25, 28]. Confirmation of the process kinetics for the investigated systems of this model follows from the linear time dependence of the formal kinetic criterion (Fig. 3). The calculated kinetic parameters are given in Table 4. The presented data also testify to the excellent kinetic characteristics of the investigated anion exchangers during platinum recovery, which do not diminish in the presence of competing chloride ions.

The sorption extraction of platinum under dynamic conditions was examined next. The output curves are shown in Fig. 4. It can be seen that the platinum chloride complexes were recovered at a level of $\sim 100\%$, due to both the high selectivity of the investigated sorbents and the more complete use of their exchange capacity in dynamics. Silver chloride com-



Fig. 4. (Color online) Fragments of the output curves at sorption of platinum(IV) and silver(I): (1) individual Pt(IV) solutions, (2) Pt(IV) in the presence of Ag(I), (3) Ag(I) in the presence of Pt(IV), $C_0(Pt) = 0.25 \text{ mmol/L}$, $C_0(Ag) = 0.95 \text{ mmol/L}$, and $C_0(HCl) = 4 \text{ mol/L}$.

plexes in the presence of platinum are in this case still not sorbed (their recovery remains at a level of the spectrophotometric determination error), as was observed under static conditions.

We can thus separate the platinum and silver chloride complexes at the stage of sorption. The next stage was therefore the elution of platinum adsorbed on anion exchangers. As was mentioned above, for this we used a 1 M solution of thiourea in 0.5 M HCl, which forms cationic complexes with the recovered platinum ions [4, 6]. As a result, platinum was eluted at a level of 83–85% in one sorption–desorption cycle. This was quite satisfactory, considering the high selectivity of the investigated anion exchangers toward platinum.

Ion exchanger	Individual Pt(IV) solutions			Pt(IV) in the presence of Ag (I)			
	$D_S \times 10^5$, cm ² /s	<i>t</i> _{1/2} , s	$\overline{v} \times 10^4$, mmol/(g s)	$D_s \times 10^5$, cm ² /s	<i>t</i> _{1/2} , s	$\overline{v} \times 10^4$, mmol/(g s)	
Purolite A500	0.91	3.2	5.6	1.1	5.9	6.8	
Purolite A300	1.2	7.6	5.4	1.5	6.0	6.8	
Purogold [™] S992	15	7.3	5.2	19	5.8	6.7	
Purolite S985	0.92	7.4	5.5	1.1	5.9	6.7	
Purolite A110	7.6	7.3	5.5	9.5	6.0	6.1	
Purogold [™] A193	14	7.6	5.3	1.9	5.9	6.7	

Table 4. Kinetic characteristics of the investigated ion exchangers during sorption of platinum(IV) from individual solutions and in the presence of silver(I) in 4 M HCl medium ($C_0(Pt) = 0.25 \text{ mmol/L}$, $C_0(Ag) = 0.95 \text{ mmol/L}$)

 D_S is the internal (gel) diffusivity, $t_{1/2}$ is the exchange half-time, and \overline{v} is the average process rate.

CONCLUSIONS

The investigated sorbents can be recommended for use in technological schemes for the joint recovery of platinum and silver from hydrochloric acid solutions.

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