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SORPTION AND ION-EXCHANGE PROCESSES

Platinum Complexation at Sorption of Hexachloroplatinate(IV) Ion with Fibrous N,S-Containing Sorbents Based on Polyacrylonitrile (of TIOPAN Type)

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Abstract—Sorption properties of fibrous nitrogen-containing sorbents based on polyacrylonitrile containing diethyldithiocarbamate (TIOPAN-2) and 8-mercaptoquinoline groups (TIOPAN-6) with respect to the platinum(IV) chloride complex were studied. The effects of the temperature, platinum concentration, and acidity on the metal recovery were determined. The kinetics of sorption and sorption capacity of sorbents in hydrochloric acid and chloride solutions were established. The most probable mechanism of sorption and the composition of compounds formed in the fiber phase were proposed.

Sorbents with N,S-containing functional groups are promising materials for recovery of platinum metals from solutions [1–4]. The mechanism of sorption is of special interest. It was discussed in [5–16]. A review on sorption recovery of platinum with N,S-containing materials was made in [5].

Materials containing thioamide groups are widely used for recovery of platinum metals [6–12]. MTILON-T fiber, which is a copolymer of polyacrylonitrile (PAN) and cellulose treated with hydrogen sulfide, recovers 0.5 mmol g^{-1} of K_2PtCl_6 from 1 M HCl at 100°C. In the presence of Fe(III) and Cu(II) salts, sorption decreases by 50–70% [6]. The mechanism of platinum recovery with MTILON-T fiber was not considered.

The modified copolymer of polyvinyl alcohol with PAN (MSPVS fiber) containing thioamide groups quantitatively and selectively recovers platinum(II) and (IV) chloro complexes from HCl and NaCl solutions of various concentrations [7–12]. In 2 M HCl, the sorption capacity of the MSPVS fiber with respect to K_2PtCl_4 is 0.48 and 1.50, and with respect to K_2PtCl_6, 0.16 and 0.42 mmol g^{-1} at 20 and 98°C, respectively. It was found that the sorption in both cases proceeds with formation of a $[Pt^{2+}\{R\text{-}C(NH_2)S\}Cl_3]$ type complex, in which thioamide groups are coordinated via sulfur atoms, with the successive hydrolytic conversion of thioamide complexes into sulfides strongly retained in the fiber matrix [7]. To recover Pt(IV) complexes, Ni Cai-Hia and Xu Yu-Wu [13] proposed a chelating ion exchanger containing groups of the $CH_2–CH_2–SH–CH_2–N(C_2H_5)_2$ type, which was prepared by treating polyoxyethylene with diethylaminomethylthiirane. The composition of the sorbed complexes was not determined. Chelating resins containing dithiocarbamate groups quantitatively and very selectively recover precious metals, including Pt(IV), from solutions. The platinum species were not studied [14].

Fibrous sorbent TIOPAN-13 based on PAN with mercaptobenzothiazole functional groups very rapidly recovers Pt(IV) complexes from solutions at pH 0.5–2.0. Large amounts of heavy metals do not interfere with the recovery [15]. The mechanism of the sorption recovery was not studied.

Monivex and Srafion resins, which are styrene–divinylbenzene copolymers containing isothiourea, can recover platinum group metals, including platinum [16]. The chelating resin based on macronetwork styrene–divinylbenzene copolymer with thiosemicarbazide as a functional group quantitatively recovers platinum group metals, including Pt(IV), from hydrochloric acid solutions. The maximal sorption capaccity with respect to Pt(IV) (0.71 mmol g^{-1}) is observed in 1 M HCl, in which platinum group metals form stable complexes. It was found that the metal–thiosemicarbazide ratio in the complex formed in the sorbent phase is close to 1 : 1 [17].

Earlier we studied the sorption properties of

Table 1. Conditions of preparation of TIOPAN-type fibers and their properties [20]

Sorbent	Modifying reagent, wt %	τ, min, at 100°C	C_S, %	SSC_{Ag}, mmol g^{-1}
TIOPAN-1	Thiosemicarbazide, 2	15	10.0	2.04
TIOPAN-2	Sodium diethyldithiocarbamate, 8	30	14.3	1.48
TIOPAN-3	Thioacetamide, 8	60	15.5	2.59
TIOPAN-4	Thiourea, 8	40	8.7	1.48
TIOPAN-5	Ammonium thiocyanate, 4	60	7.8	1.48
TIOPAN-6	Sodium 8-mercaptoquinolinate, 3	20	4.0	1.11

GLIPAN-1 fiber (which is a fibrous sorbent based on PAN modified with thiosemicarbazide) with respect to Pt(IV) chloride complexes and showed that this fiber quantitatively recovers K_2PtCl_6 from HCl and NaCl solutions (C_{Pt} = 1–10 mM) with similar rates [18]. The maximal sorption capacity (1.35 mmol g^{-1}) is observed in 0.5 M HCl. Sorption of Pt(IV) chloride complexes with GLIPAN-1 in all cases is caused by metal complexation with sorbent as a polymeric ligand and metal reduction in the fiber phase. The complex [R–NH–C(NH–NH_2)=SPt(II)Cl_2], in which the thiosemicarbazide group is a bidentate ligand, is the final sorbed species of platinum [18].

For recovery of platinum(II) chloride complexes, we proposed fibrous nitrogen-containing sorbents of the TIOPAN type (copolymers of PAN with grafted polyglycidyl methacrylate PAN–PGMA) modified with various N,S-containing reagents (Table 1) [19–22]. High Pt(II) sorption is observed with TIOPAN-6 at room temperature and with TIOPAN-2, TIOPAN-5, and TIOPAN-6 at elevated temperatures. The static sorption capacity (SSC) of these fibers in 1 M HCl decreases in the order TIOPAN-6 > TIOPAN-5 > TIOPAN-2 at 20°C and TIOPAN-2 > TIOPAN-5 > TIOPAN-6 at 98°C (Table 2). We concluded that in all cases the Pt(II) sorption is caused by complexation with the sorbents as polymeric ligands. In the fiber phase, TIOPAN-2 forms complexes [Pt(SR_2)Cl_2], TIOPAN-5 gives [Pt$(SHR)_2$$Cl_2$] in which polymeric ligands coordinate via sulfur atoms, and TIOPAN-6 forms *cis*-[Pt$(SQuinR)_2$$Cl_2$] in which 8-mercaptoquinoline group is a monodentate ligand bonded to Pt(II) via nitrogen atom [19].

Table 2. Recovery of platinum from 1 M HCl with TIOPAN-type sorbents

Sorbent	Recovery, %		
	Pt(IV) at 98°C	Pt(II) at indicated temperature, °C [19]	
		20	98
TIOPAN-1	1	29	47
TIOPAN-2	82	15	100
TIOPAN-3	0	3	8
TIOPAN-4	19	9	25
TIOPAN-5	2	43	82
TIOPAN-6	97	100	96

In this work, we studied the sorption properties of TIOPAN-type N,S-containing fibers with respect to Pt(IV) chloride complexes and the composition of the species formed in the sorbent phase.

EXPERIMENTAL

Sorption was studied under static conditions at the continuous agitation and at 18 and 98°C. The starting Pt(IV) compound, K_2PtCl_6, was synthesized by the method described in [23]. Sorption was performed from freshly prepared 1–20 mM solutions of K_2PtCl_6 in 0.1–4.0 M HCl or 0.1–2 M NaCl. In all cases, the volume of solutions taken was 20 ml, and the sorbent sample was 0.1 g. The platinum content in solutions was determined spectrophotometrically with tin(II) chloride [24]. The amount of the metal sorbed was calculated as the difference between its content in the solution before and after sorption. The IR spectra of fibers within the 400–4000 cm^{-1} range were measured on an IKS-29 spectrophotometer, and within the 140–400 cm^{-1} range, on a Hitachi FIS-3 spectrophotometer. Samples were prepared as in [5]. X-ray photoelectron spectra (XPES) were recorded on a Perkin–Elmer PH 15400 spectrometer with excitation by Mg X-ray radiation. Spectra were treated (quantitative analysis and line separation) using standard programs. The spectra were standardized against C1*s* electron binding energy of 285.0 eV. The binding energy was determined with an error of 0.2 eV. Thermograms of sorbents were recorded on an MOM (Hungary) derivatograph within the 20–800°C range at a heating rate of 10 deg min^{-1}. Isothermal heating of sorbents was performed on a special device equipped with a system for a qualitative analysis of the liberated gaseous products. Thermal conversion of samples (temperature of the decomposition onset) was visually controlled on a Koeffler stage.

Initially, the sorption power of TIOPAN fibers with respect to Pt(IV) at sorption from solutions of K_2PtCl_6 in 1 M HCl was studied at 98°C (Table 2). The sorption duration was 1 h, the platinum concentration in the initial solution was 1 mM. As seen, Pt(IV) is efficiently recovered with TIOPAN-2 and TIOPAN-6, and Pt(II), also with TIOPAN-5. There is no correlation between the sulfur content in the fiber and the recovery of both Pt(II) and Pt(IV) (Tables 1, 2). TIOPAN-1, TIOPAN-3, TIOPAN-4, and TIOPAN-5 fibers recover Pt(IV) poorly.

TIOPAN-2 and TIOPAN-6 fibers containing diethyldithiocarbamate and 8-mercaptoquinoline groups, respectively, were studied in detail. It was found that the time of attaining the apparent equilibrium τ_{ap} in distribution of Pt(IV) between the fiber and 1 M HCl solution is 2 h at 18°C and 1 h at 98°C for TIOPAN-6 and 2 h at 98°C for TIOPAN-2. The half-sorption time $\tau_{1/2}$ for TIOPAN-6 is 2 and 4 min at 98 and 18°C, respectively. The low rate of Pt(IV) sorption with TIOPAN-2 should be noted: $\tau_{1/2}$ is 25 min even at 98°C (Fig. 1a, Table 3).

For sorption from 0.5 M NaCl, the apparent equilibrium of Pt(IV) between the solution and sorbent in all cases is attained in 2 h, and $\tau_{1/2}$ both at 98 and 18°C is 4 min for TIOPAN-6 and ~23 min for TIOPAN-2 (Fig. 1a, Table 3). At room temperature, TIOPAN-2 recovers Pt(IV) chloride complexes neither from HCl nor from NaCl solutions.

The type of the diffusion kinetics of sorption is commonly determined using the known dependence of the exchange degree F ($F = Q_\tau/Q_\infty$, where Q_τ is sorption in time τ and Q_∞ is the equilibrium sorption) on sorption time τ [25] and the dependence on sorption time of B_τ and $-\ln(1 - F)$, where B_τ is the kinetic coefficient of sorption [$B_\tau = (F/1.08)^2$] [26]. The analysis of the kinetic curves of sorption (Fig. 1) shows mixed diffusion character of the kinetics of Pt(IV) sorption with TIOPAN-6 fiber from acid solutions. The linear sections of $-\ln(1 - F)$–τ curves indicate that the "film diffusion" is the major factor affecting the kinetics of Pt(IV) sorption with TIOPAN-2 fiber [26] (Fig. 1c).

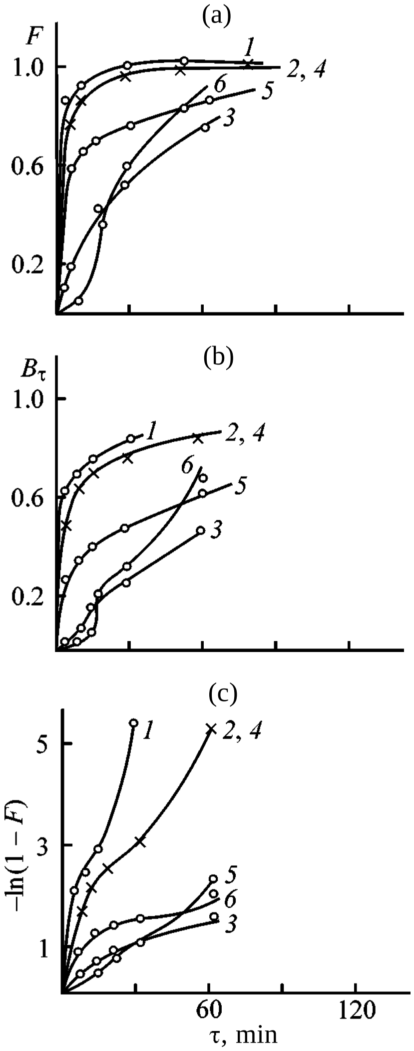

Fig. 1. Dependences of (a) F, (b) B_τ, and (c) $-\ln(1 - F)$ on the time of sorption τ of K_2PtCl_6 from 1 M HCl with (*1, 2*) TIOPAN-6 and (*3*) TIOPAN-2 and from 0.5 M NaCl with (*4, 5*) TIOPAN-6 and (*6*) TIOPAN-2. (F) Exchange degree; $F = Q_\tau/Q_\infty$, where Q_τ is Pt sorption in time τ and Q_∞ is equilibrium sorption; (B_τ) kinetic coefficient of sorption, $B_\tau = (F/1.08)^2$. Temperature, °C: (*1*) 3, (*6*) 98, and (*4, 5*) 18.

It was found that at platinum sorption with TIOPAN-6 at 18 and 98°C (C_{Pt} = 1 mM) the Pt(IV) recovery decreases with increasing concentration of hydrochloric acid: platinum is practically completely recovered from 0.1–0.5 M HCl and to only 75–80% from 4 M HCl. The effect of HCl concentration on platinum(IV) sorption with TIOPAN-2 is even stronger. At 98°C, platinum recovery decreases from 100 to 45% in going from 0.1 to 4 M HCl.

The dependence of SSE with respect to Pt(IV) on equilibrium metal concentration is shown in Fig. 2. Sharp slope of the isotherms of the Pt(IV) sorption from HCl solutions allows TIOPAN-2 and TIOPAN-6 to be recommended for quantitative recovery of platinum from both dilute and concentrated solutions, the

Table 3. Recovery of platinum(IV) chloride complexes with TIOPAN-2 and TIOPAN-6 fibers

T, °C	Medium, M	SSC, mmol g^{-1}	$\tau_{1/2}$	τ_{app}	Pt(IV) recovery, %
			min		
		TIOPAN-2			
98	HCl, 1	0.75	25	120	82
98	NaCl, 0.5	0.54	23	120	79
		TIOPAN-6			
18	HCl, 1	0.57	4	120	91
18	NaCl, 0.5	0.16	4	120	21
98	HCl, 1	0.80	2	60	97
98	NaCl, 0.05	0.53	4	120	35

former sorbent at heating and the latter, both at room temperature and heating. The data on SSC of fibers are listed in Table 3. The platinum(IV) recovery from NaCl solutions is weaker (Table 3, Fig. 2).

Sorption of Pt(IV) with TIOPAN-2 and TIOPAN-6 is practically irreversible: only 0.6 and 1.0% of Pt is desorbed with 6 M HCl and 22 and 26% of Pt is desorbed with thiourea solution in 10% HCl from TIOPAN-2 and TIOPAN-6, respectively.

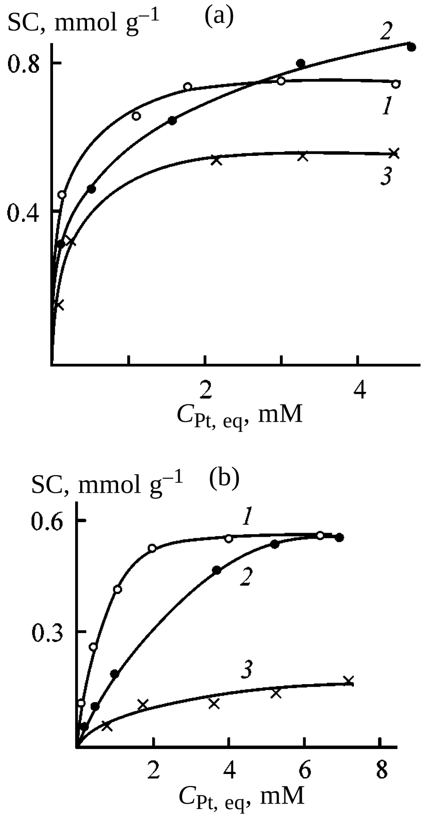

Fig. 2. Isotherms of K_2PtCl_6 sorption from (a) 1 M HCl and (b) 0.5 M NaCl at (*1, 2*) 98 and (*3*) 18°C. (SC) Sorption capacity; ($C_{Pt, eq}$) equilibrium concentration. Sorbent: (*1*) TIOPAN-2 and (*2, 3*) TIOPAN-6.

The comparison of the kinetic and capacity characteristics of the fibers shows that in all cases platinum(II) is recovered faster and with greater SSC as compared to Pt(IV). This may be due to two factors: (1) greater charge density on $PtCl_4^{2-}$ as compared to $PtCl_6^{2-}$, which increases electrostatic attraction of the complex anion to the protonated nitrogen atom of the fiber, and (2) greater steric hindrance in interaction of the octahedral $PtCl_6^{2-}$ complex as compared to the square-planar $PtCl_4^{2-}$ ion.

It should be noted that TIOPAN-5 containing thiol groups practically completely recovers K_2PtCl_4 [19] but sorbs K_2PtCl_6 poorly (Table 2).

The analysis of the kinetics and capacity parameters of TIOPAN-2 with respect to K_2PtCl_6 suggests that the platinum recovery from both hydrochloric acid and NaCl solutions proceeds with formation of a Pt(II) compound of the $[Pt(SR)_2Cl_2]$ type in the sorbent phase. The diethyldithiocarbamate group of the polymeric ligand coordinates to Pt(II) via sulfur atom as a monodentate ligand. The low rate of sorption, sharp temperature dependence of sorption, and comparison of the IR and far-IR spectra of fibers after sorption of K_2PtCl_6 (Figs. 3, 4) and K_2PtCl_4 [19] indicate complex formation. In both cases the spectra are similar. A strong change of the IR spectrum in the ν(CS) and ν(CN) range (1200–1600 cm^{-1}) (Fig. 3) shows formation of a covalent bond of platinum with the fiber. XPES indicates reduction of Pt(IV) to Pt(II) during K_2PtCl_6 sorption. The binding energy of the Pt4$f_{7/2}$ electron is 72.7 eV, which agrees with reference data for Pt(II) [27].

For comparison, we performed a reaction of K_2PtCl_4 with the monomeric ligand, sodium diethyldithiocarbamate. To a hot solution of K_2PtCl_4, a hot solution of sodium diethyldithiocarbamate was added gradually with stirring. The resulting pale yellow precipitate was filtered off and washed with ether. As shown by elemental analysis and far-IR and IR spectra, the compound corresponds to the previously described complex $[PtS_2N(C_2H_5)_2]$ [28] in which diethyldithiocarbamate ion coordinates to platinum atom via sulfur atom. In the IR spectra of this compound and of TIOPAN-2 fiber after K_2PtCl_6 sorption the same bands assigned to stretching vibrations of the coordinated diethyldithiocarbamate group are observed in the 1200–1600 cm^{-1} range (Fig. 3). However, their far-IR spectra differ substantially: the spectrum of the fiber after sorption of K_2PtCl_6 from 1 M HCl contains two bands at 310 and 328 cm^{-1} assigned to the stretching vibrations ν(Pt–Cl) of asymmetrical *cis*-$[Pt(SR)_2Cl_2]$. TIOPAN-2 fiber after sorption of K_2PtCl_6 from 0.5 M NaCl has the similar spectrum. In the far-IR spectrum of the chelate with the monomeric ligand such bands are absent [28]. The difference in reaction of K_2PtCl_6 with the monomeric ligand and the fibrous sorbent, both containing diethyldithiocarbamate group, may be due to two factors: (1) one of two sulfur atoms of the polymeric ligand (TIOPAN-2) is bound with the fiber matrix PAN–PGMA and cannot participate in formation of the chelate bond with platinum and (2) steric hindrances arise in interaction of the platinum atom with the bulky diethyldithiocarbamate group bonded with the polymeric matrix of the fiber.

The high rate of recovery of the platinum(IV) chloride complexes with TIOPAN-6 fiber from both acid and neutral media and weak dependence on temperature suggest that in the first stage of sorption the initial complex reacts with protonated fiber

$$2RQuinS\cdot HCl + K_2PtCl_6 \rightarrow (RQuinSH)_2PtCl_6 + 2KCl,$$

$$2RQuinS\cdot H_2O + K_2PtCl_6 \rightarrow (RQuinSH)_2PtCl_6 + 2KOH.$$

The next stage is the partial reduction of the onium platinum(IV) complex to the analogous platinum(II) complex $[(RQuinSH)_2PtCl_4]$ with 8-mercaptoquinoline groups that do not participate in sorption. The XPES data indicate the prevalence of platinum(II) complexes on the fiber. The binding energy of the $Pt4f_{7/2}$ electron of 73.1 eV is in a good agreement with the reference data for $Pt4f_{7/2}$, e.g., in K_2PtCl_4 [27]. We can assume that sorption from NaCl solutions involves in the second stage the Anderson rearrangement with formation of *cis*-$(RQuinSH)_2PtCl_4$ coordinated via the nitrogen atom:

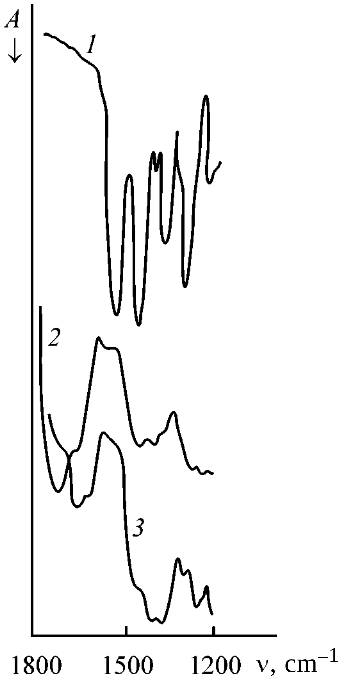

Fig. 3. IR spectra of (*1*) $[Pt(S_2N(C_2H_5)_2]$, (*2*) TIOPAN-2 fiber after K_2PtCl_6 sorption from 1 M HCl, and (*3*) sodium diethyldithiocarbamate. (*A*) Absorption and (ν) wave number; the same for Fig. 4.

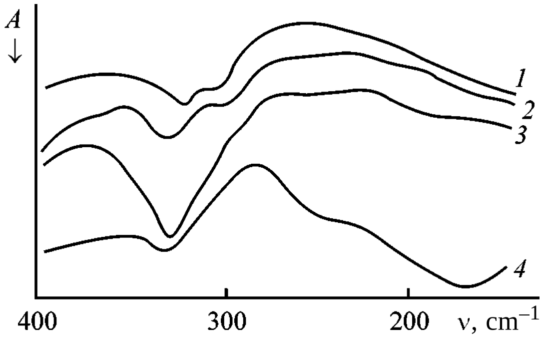

Fig. 4. Far-IR spectra of fibers after K_2PtCl_6 sorption. Sorption temperature 98°C, C_{Pt} = 8 mM. Sorption from (*1–3*) 1 M HCl and (*4*) 0.5 M NaCl. (*2*) Fiber heated at 180°C for 0.5 h. Sorbent: (*1*) TIOPAN-2 and (*2–4*) TIOPAN-6.

$$(RQuinSH)_2PtCl_4 \rightarrow cis\text{-}[Pt(RQuinS)_2Cl_2] + 2HCl.$$

Heating promotes such transformation. To simulate this reaction, we estimated the rearrangement temperature from the termogravimetric data for the fiber. In the far-IR spectrum of TIOPAN-6 fiber after sorption of K_2PtCl_4 from 1 M HCl, subjected to isothermal heating at 180°C for 0.5 h, two bands are observed at 305 and 329 cm^{-1}, belonging to Pt–Cl stretching vibrations of the asymmetrical *cis*-dichlorodiammine complex. Formation of H bonds between protonated nitrogen of the fiber mercaptoquinoline groups and chloride ions in the inner coordination sphere of the metal complex facilitates such transformation.

It is known that in reactions of platinum complexes with 8-mercaptoquinoline and its derivatives yield the Pt(II) chelates [29]. For instance, reaction of 8-mercaptoquinoline with both K_2PtCl_4 and K_2PtCl_6 produces the compound $[Pt(SNC_9H_6)_2]$ in which the organic ligand coordinates to platinum via N and S atoms [29]. Since, when PAN–PGMA is modified with sodium 8-mercaptoquinolinate [20], the modifying agent is incorporated into the fiber matrix via the sulfur atom. The platinum atom is probably bound with the fiber via the nitrogen atom.

CONCLUSIONS

(1) Sorbents TIOPAN-2 and TIOPAN-6 (copolymers of polyacrylonitrile with grafted polyglycidyl methacrylate modified with sodium diethyldithiocarbamate and 8-mercaptoquinoline, respectively) can quantitatively recover platinum(IV) chloride complexes from acid and neutral solutions. TIOPAN-6 with high rate and quantitatively recovers Pt(IV) at 18 and 98°C, while TIOPAN-2 efficiently sorbs Pt(IV) only at heating.

(2) In all cases, Pt(IV) sorption is caused by complexation with sorbents as polymeric ligands. The platinum(IV) chloride complex reacts with TIOPAN-2 fiber with formation of the $[Pt(SR)_2Cl_2]$-type compound, in which the polymeric ligand coordinates via sulfur atom. At Pt(IV) sorption with TIOPAN-6, the onium compound $(RQuinSH)_2PtCl_4$ is formed. At heating, this compound converts into *cis*-$[Pt \cdot (RQuinS)_2Cl_2]$ in which 8-mercaptoquinoline group coordinates via nitrogen atom.

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