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Pd(II) extraction from acid solutions by bis-(2-hydroxyl-5-alkylbenzyl)amine

It is known that a series of organic compounds contained in the molecule SH, NH groups have the ability to form the intracomplex compounds under certain conditions. These compounds allow to carry out the extraction in acidic medium and therefore prevent the hydrolysis process. They are not dissolved in water but are soluble in various solvents and form colored solutions and so may be used in extraction chemistry. The main purpose of this paper was to study the ability to extract palladium by bis-(2-hydroxyl-5-alkylbenzyl)amine, synthesized in the laboratory on the basis of industrial alkylphenols. Ammonium acetate with various pH was used as a buffer to extract palladium from PdCl₂·2H₂O 0.1 mg/ml solution. The main task for the use of inert organic compound in extraction is the selection of a reagent which dissolves it but does not form any compound. For this purpose the influence of different solvents on this reagent was researched. The experiments show that bis-(2-hydroxyl-5-alkylbenzyl)amine is dissolved well in organic solvents. Its solution, for example in kerosene, is light-resistant, does not hydrolyze in water, alkalis and acids. Thus bis-(2-hydroxyl-5-alkylbenzyl)amine may be recommended for palladium extraction.

Keywords: extraction, palladium, alkylbenzylamines, diluent, intracomplex compounds, acidic environment, hydrolysis, industrial alkylphenol.

Introduction

A consistent and thorough study of the search and synthesis of new extractants and their ability to extract important elements, in particular, platinum group metals, are important. Every work done in this area leads to the development of extraction chemistry of platinum group metals and accelerates the solution of practical issues.

From this point of view, a number of organic compounds have the ability to form inner complex combinations with metal ions under certain conditions. These organic compounds mainly include SH, NH groups in the molecule. Extraction became a modern technique to recover and separate noble metals, including palladium(II). Several reagents were proposed, including hydroxyoximes, alkyl derivatives of 8-hydroxyquinoline, hydrophobic amines and esters of pyridine carboxylic acids [1]. Some of them are used in the industry. However, the extraction occurs very slowly which is caused by the nature of hydrophobic extractant and symmetrical palladium complex.

The amine type extraction reagents are perspective for the separation of palladium from acidic nitrate solutions like waste solutions. Palladium is extracted by quaternary ammonium salts from the nitric acid solutions effectively, tertiary amines offer low distribution ratios. The effectiveness of extraction by these reagents increases considerably in the presence of small amounts of chloride ions [2].

The mechanism of palladium extraction from hydrochloric acid is well studied; the extraction from nitrate medium is less known. At low chloride concentrations, it has been observed that with an increase of palladium concentration the distribution ratio increases, and the mechanism of extraction is complex.

It was shown that palladium(II) is efficiently extracted with (RS)-1-[2-(2,4-dichlorophenyl)pentyl]-1H-1,2,4-triazole from 0.2–5.0 M HNO₃ solutions and can be selectively separated from Ni(II), Cu(II), Pb(II), Fe(III), Ag(I), as well as from lanthanides(III) in 2–4 M HNO₃ solutions [3]. The extraction of Pd(II) from

1 M HNO₃ solution proceeds via coordination mechanism; this is an endothermic process in the 10–30 °C temperature range.

It was shown by extraction methods and IR spectroscopy that thiacalixarenes 2 extract complex species [Pd_nL_mH_{4-2n}] (*m* = 1, *n* = 1 and 2) and [(PdA₂)_nL_mH₄] (*A* = NO₃⁻, *m* = 1, *n* = 1–4) from nitric acid solutions at pH 3 [4]. Extraction constants for these palladium species that describe experimental data were calculated.

A comparison [5] was made between the separate extraction and coextraction of neodymium and palladium from nitric acid solutions with bifunctional phosphorylated thiacalix[4]arene (TCPO) and model monofunctional extractants (phosphorylated calixarene (CPO), thiacalixarene (TCA), and their mixtures) for mutual influence evaluation of S- and PO-donor sites of the extractants. The results of the extraction with TCA–CPO mixtures are of interest for a simplified process (PUREX) and a process being developed (CARBEX) as applied to recovery of neodymium and palladium from nitric acid solutions and their separation from carbonate solutions after neodymium stripping.

The work [6] studied the extraction of chloropalladium complexes with solutions of trioctylmethylammonium di(2,4,4-trimethylpentyl)dithiophosphinate in toluene over a wide range of aqueous acidities. Distribution factors and spectroscopic studies of extraction products showed that (R₄N)[Pd₂Cl₄A] complexes are formed in the organic phase. As the concentration of the dialkyldithiophosphinic acid increases, palladium di(2,4,4-trimethylpentyl)dithiophosphinate is formed in the organic phase.

Thus, various derivatives of organic acids have been studied as extractants for the recovery and separation of palladium from acid solutions [7–10].

Successful solution of the problem of protecting the biosphere, reducing the negative impact of industrialization on the state of the environment is directly related to the development of effective analysis methods. And the above extractants for the selective determination of palladium do not always satisfy the requirements for the lower boundaries of the determined contents. These extractants are often unstable reagents that can be gradually oxidized by air. Therefore, it was interesting to study the distribution of palladium over effective extractant solutions based on bis-(2-hydroxy-5-alkylbenzyl)amine, which is highly soluble in organic solvents. Our task was to effectively determine the composition of the extracted compounds.

Experimental

Ammonium acetate buffer was used to obtain a PdCl₂·2H₂O salt solution with a density of 0.1 mg/ml. The solutions were concentrated using concentrated HCl in a water bath before the experiment. Then the solutions were diluted to achieve the required concentrations (*C*) of metal and acids. The fixed values of chloride ion concentration and ionic strength in aqueous solutions were adjusted using lithium chloride so that *C*_{LiCl} + *C*_{HCl} = 3 mol/l.

The bis-(2-hydroxyl-5-alkylbenzyl)amine 0.1 M kerosene solution was synthesized on the basis of the industrial alkylphenols (employing Mannich reaction) in the laboratory. The structure of the obtained bis-(2-hydroxy-5-alkylbenzyl)amine was confirmed by spectroscopic methods (Shimadzu (Japan) SF-2000 SKB Spectr, PromEcoLab PE-5300V, UV-1700 and UV-2450 spectrophotometers) [11–12].

The palladium-containing solutions used for extraction were prepared according to the procedure described in [13–17]. Separate solutions of bis-(2-hydroxyl-5-alkylbenzyl)amine in kerosene were used as extractants, followed by stirring an organic solution with an equal volume of 1 M NaOH, and then with water.

Extraction was carried out in test tubes at equal volumes of aqueous and organic phases at 20 °C (±1 °C). The concentration of palladium in solutions was determined by the photometric method, and in organic phases by the difference between the concentrations of palladium in the initial solution and in the aqueous phase after extraction. The optical density of the solutions was determined by IR spectrometer. To assess the degree of extraction the following quantitative values were used in the organic and aqueous phases:

$$R = \frac{q_{org}}{q_w^o} = \frac{C_{Pd,org} \cdot V_{org}}{C_{Pd,org} \cdot V_{org} + C_{Pd,w} \cdot V_w} \quad (1)$$

The proportion of the substance remaining in the aqueous phase was determined by the formula:

$$\infty_w = \frac{q_w}{q_w^o} = \frac{C_{Pd,w} \cdot V_w}{C_{Pd,org} \cdot V_{org} + C_{Pd,w} \cdot V_w} \quad (2)$$

To determine the solubility of the synthesized bis-(2-hydroxyl-5-alkylbenzyl)amine in organic solvents an inert solvent was experimentally chosen, which does not form a compound upon dissolution of the extractant. For this, the effect of different grades of solvents was tested (Table 1).

Selection of organic solvents for palladium — bis-(2-hydroxyl-5-alkylbenzyl)amine system at 20 °C

Organic solvents	The result of reaction	The color of the extract	Optical density, (A)	Notes
Benzene	extracted	weak yellow	0.10–0.41	The water phase is blurred
CCl ₄	–"–	weak yellow	0.42–0.47	The phase is blurred
Kerosene	–"–	yellow	0.83–0.84	Transparent to both phases
Octane	–"–	colorless	–"–	–"–
Hexane	–"–	colorless	–"–	–"–
Isobutyl alcohol	–"–	weak yellow	0.50–0.52	Phases are clean
<i>n</i> -Butyl alcohol	–"–	weak yellow	0.55–0.56	The phase are blurred

Measurements of optical density were carried out according to the standard method by SF-2000 SKB "Spectrum" and PromEcoLab PE-5300V spectrophotometers in the visible and ultraviolet regions [18].

The transmission (T) of the sample placed between the light source and the detector was determined at a given UV/visible wavelength. Transmission is the ratio of the intensity of the light transmitted through the sample to the intensity of the incident light, which is determined by the formula:

$$T = I/I_0,$$

where I is the intensity of the transmitted radiation; I_0 is the intensity of the radiation incident on the sample.

Optical density was determined as the decimal logarithm of the reciprocal of the transmission for monochromatic radiation. It is a dimensionless quantity, which is determined by the formula:

$$A = \log(1/T) = \log(I_0/I).$$

Results and discussion

Extraction was performed using bis-(2-hydroxyl-5-alkylbenzyl)amine by studying the distribution of palladium between two immiscible solvents (water and an organic solvent). In this case, the extraction obeys the Gibbs phase rule:

$$N + F = K + 2,$$

where N is the number of phases; F is the number of degrees of freedom; K is the number of components.

Therefore, the studied system was monovariant ($F = 1$) at constant temperature and pressure. In this case, if the concentration of dissolved palladium in one phase is constant, then its concentration in the other phase is also constant.

It was found that the bis-(2-hydroxyl-5-alkylbenzyl)amine synthesized by us, on the one hand, allows extraction in an acidic medium, thereby preventing hydrolysis. On the other hand, the extractant increases the selectivity of the palladium extraction process. The reagent gives soluble colored solutions, which is important for their use, in particular in extraction chemistry.

The third light filter was used during all measurements. As can be seen from the Table 1, the largest optical density was obtained for palladium extraction by bis-(2-hydroxyl-5-alkylbenzyl)amine compound with kerosene. Therefore, kerosene is taken as an organic solvent useful in subsequent experiments. One of the main conditions is to study the dependence on the duration of stirring 5 ml of 1N H₂SO₄, 0.5 ml of reagent (0.1 mol), 5 ml of kerosene. The optical density was measured by separating the organic phase. The results are given in Figure 1.

It is sufficient to intensively stir the mixture for three minutes to form palladium bis-(2-hydroxyl-5-alkylbenzyl)amine and complete its transition to the organic phase. On this basis, in all subsequent experiments the mixture was stirred for three minutes.

The effect of temperature (20–60 °C) on the distribution of Pd(II) was investigated (Fig. 2). The results show that there is a decrease in percent extraction with the increasing temperature. It is indicating the process to be exothermic.

To study the durability of the obtained bis-(2-hydroxyl-5-alkylbenzyl)amine palladium compound, 0.5 ml (0.1 M) of a solution containing palladium and 0.5 ml (0.1 M) kerosene solution were mixed for three minutes. Then the optical density of the solution was measured by separating the organic phase.

It was found that the dependence of the optical density of palladium formed by bis-(2-hydroxyl-5-alkylbenzyl)amine at various concentrations ($[Pd^{2+}] = 2\text{--}4 \cdot 10^{-4}$ M) on time (1–24 h) remains constant. In other

words, the synthesized reagent does not change its optical density for 24 hours, regardless of the palladium concentration. This indicates that it creates a stable complex with palladium.

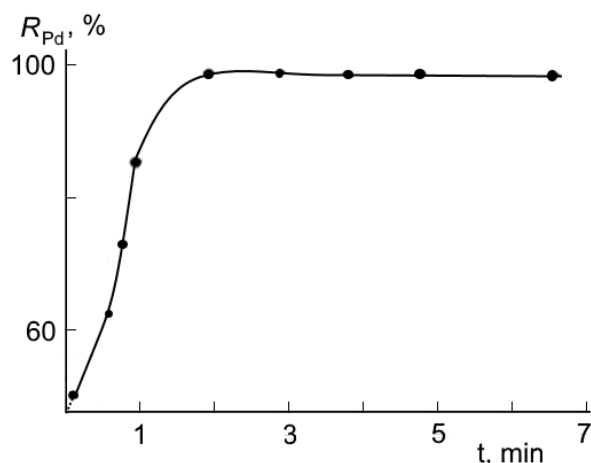


Figure 1. The dependence of palladium extraction degree on stirring time at 20 °C

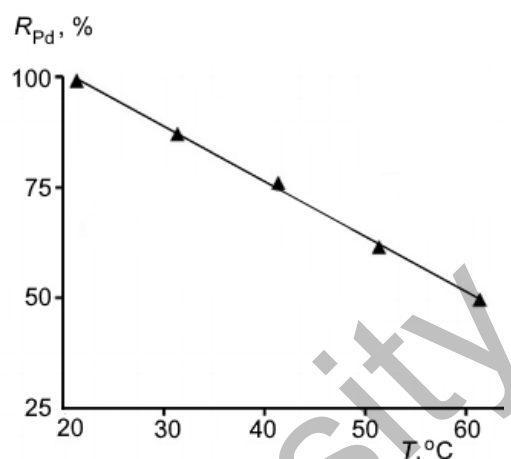


Figure 2. Effect of temperature on the extraction of Pd(II) from H₂SO₄. Conditions: [metal ion] = 5 × 10⁻⁴ mol L⁻¹, [bis-(2-hydroxyl-5-alkylbenzyl)amine] = 0.10 mol L⁻¹, [H₂SO₄] = 1.0 mol L⁻¹ (for Pd(II))

Ammonium acetate was used as a buffer to create pH medium. To obtain optimal conditions for the formation of a palladium complex with bis-(2-hydroxyl-5-alkylbenzyl)amine, the dependence of the degree of palladium recovery on the pH was studied. With an increase in pH from 1 to 5.0, the main concentration of palladium (100–95 %) is transferred into the organic phase. An increase in the concentration of [OH]⁻ ions in solution leads to a decrease in the percentage of palladium recovery.

Analysis has revealed that palladium bis-(2-hydroxyl-5-alkylbenzyl)amine is a compound that has an intensive color in the acid medium. Thus, the dependence of the optical density of the resulting compound on various concentrations of chloride, sulfate and phosphate acids has been studied. The results are given in Table 2.

Table 2

Dependence of the optical density of the compound formed by the bis-(2-hydroxyl-5-alkylbenzyl)amine with palladium on mineral concentrations; [Pd²⁺] = 9.4 · 10⁻⁴M, [HR] = 0.1M, t = 3 min, 20 °C

The concentration of acids, N	Optical density, (A)	Pd ²⁺ , C _{org} , mg/ml	Pd ²⁺ , C _w , mg/ml	Degree of extraction, R _{Pd} , %	
HCl	0.01	0.85	0.008	n.f.*	100.0
	0.1	0.84	0.008	n.f.*	100.0
	1.0	0.84	0.008	n.f.*	100.0
	2.0	0.72	0.0076	0.0004	95.0
	3.0	0.72	0.0076	0.0004	95.0
H ₂ SO ₄	5.0	0.32	0.0030	0.00150	37.7
	0.01	0.87	0.0080	n.f.*	100.0
	6.0	0.87	0.0080	n.f.*	100.0
	7.0	0.74	0.0076	0.0004	95.0
	8.0	0.67	0.0070	0.001	88.0
H ₃ PO ₄	9.0	0.60	0.0062	0.0018	70.0
	10.0	0.60	0.0062	0.0018	70.0
	0.01	0.85	0.0080	n.f.*	100.0
	1.0	0.86	0.0080	n.f.*	100.0
	7.0	0.71	0.0076	0.0004	95.0
	8.0	0.71	0.0076	0.0004	95.0
	9.0	0.70	0.0076	0.0004	95.0
	10.0	0.70	0.0076	0.0004	95.0

Note. n.f.* – not found.

It was found that palladium(II) passes into the organic phase during the extraction in solutions from 0.01 N to 1 N. The percentage of extracts decreases starting from 2 N. For example, the extraction percentage in 5 N chlorous acid is 37.7%.

In sulfuric acid medium from 0.01 N to 6 N, palladium(II) passes into the organic phase. Starting with a concentration of 7 N sulfuric acid, the percentage recovery is gradually reduced. At 7 N, this indicator was 95 %, and at 10 N ~70%.

Depending on the concentration of phosphoric acid from 0.1 N to 6 N, the degree of palladium(II) extraction with bis-(2-hydroxyl-5-alkylbenzyl)amine remains constant. Palladium passes into the organic phase in this interval. The extraction at 7 N is 95 %, and this indicator remains constant until the concentration of phosphoric acid is up to 10 N.

Conclusion

The present investigation highlights that bis-(2-hydroxyl-5-alkylbenzyl)amine is a useful extractant for Pd(II) as well as for its separation from the most commonly associated metal ions. The separations do not require the addition of any modifier or synergist and can be accomplished at room temperature. The stripping agents used for separations are simple and convenient for further processing of solutions. The developed conditions of separations have been successfully extended to recover Pd(II) from synthetic mixture. The hydrolytic stability and the recycling capacity of the extractant are good and the phase separation is fast.

Thus, it was found that bis-(2-hydroxyl-5-alkylbenzyl)amine has the ability to form complexes with palladium(II) under certain conditions. This reagent allows extraction in acidic medium and, therefore, prevents hydrolysis. Being soluble in various solvents, it forms colored solutions and can be used in extraction chemistry.

Solution of bis-(2-hydroxyl-5-alkylbenzyl)amine in kerosene is light-resistant, is not hydrolyzed in water, alkalis and acids. Palladium extraction by this reagent depends on the aqueous and organic phases. This makes it possible to vary the extraction characteristics in palladium-containing systems. Reagent may be recommended for palladium extraction.

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С.Р. Маммадова

Қышқыл ерітінділерден бис-(2-гидроксил-5-алкилбензил)аминмен Pd(II) экстракциясы

Молекуласында SH, NH-топтары бар бірқатар органикалық қосылыстар белгілі бір жағдайда ішкікомплексті қосылыстар түзуге қабілетті. Бұл қосылыстар экстракцияны қышқылды ортада жүргізуге мүмкіндік береді, гидролиз процесін болдырмайды. Олар суда ерімейді және әртүрлі еріткіштерде ерітін болғандықтан түрлі-түсті ерітінділер түзеді, сондықтан оларды экстракциялық химияда қолдануға болады. Зерттеудің басты мақсаты зертханада өндірістік бис-(2-гидрокси-5-алкилбензил)амин алкилфенолдары негізінде синтезделген реактивтің палладийге қатысты экстракция қабілетін зерттеу. Палладийдің рН ерітінділерінің 0,1 мкг/мл алу үшін PdCl₂·2H₂O бастап буфер ретінде аммоний ацетаты қолданылды. Экстракция кезінде инертті органикалық қосылысты пайдаланудағы басты мақсат — оны ерітетін, бірақ басқа қосылыстар түзбейтін реагентті таңдау. Осы мақсатта әртүрлі еріткіштердің осы реактивке әсері зерттелді. Зерттеулер көрсеткендей, бис-(2-гидрокси-5-алкилбензил)амин органикалық еріткіштерде оңай ериді. Оның ерітіндісі мысалы, керосинде, жарыққа төзімді, суда, сілтілерде және қышқылдарда гидролизденбейді. Осылайша, палладийді экстракциялау үшін бис-(2-гидрокси-5-алкилбензил) аминді ұсынуға болады.

Кілт сөздер: экстракция, палладий, алкилбензиламин, еріткіш, ішкікомплексті қосылыстар, қышқыл орта, гидролиз, өндірістік алкилфенол.

С.Р. Маммадова

Экстракция Pd(II) бис-(2-гидрокси-5-алкилбензил)амином из кислых растворов

Ряд органических соединений, содержащих в своей молекуле SH, NH-группы, способны образовывать при определенных условиях внутрикомплексные соединения. Данные соединения позволяют проводить экстракцию в кислой среде, что предотвращает процесс гидролиза. Они не растворяются в воде и, будучи растворимы в различных растворителях, образуют окрашенные растворы, по этой причине могут быть использованы в экстракционной химии. Основная цель данного исследования — изучение экстракционной способности по отношению к палладию реагента бис-(2-гидрокси-5-алкилбензил)амин, синтезированного в лаборатории на основе промышленных алкилфенолов. Ацетат аммония с различными значениями рН был применен в качестве буфера при экстракции палладия из 0,1 мкг/мл раствора PdCl₂·2H₂O. Главным в использовании инертного органического соединения в экстракции является выбор реагента, который растворяет его, но не образует с ним других соединений. С этой целью изучалось влияние различных растворителей на этот реагент. Исследование показало, что бис-(2-гидрокси-5-алкилбензил)амин хорошо растворяется в органических растворителях. Его раствор, например в керосине, устойчив к свету, не гидролизует в воде, щелочах и кислотах. Таким образом, бис-(2-гидрокси-5-алкилбензил)амин может быть рекомендован для экстракции палладия.

Ключевые слова: экстракция, палладий, алкилбензиламин, растворитель, внутрикомплексные соединения, кислая среда, гидролиз, промышленный алкилфенол.

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