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Liquid Anion Exchange Chromatographic Extraction and Separation of Platinum (IV) with N-Octylaniline as an Metallurgical Reagent: Analysis of Real Samples

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Abstract

A simple and selective method was developed for the determination of platinum (IV) with n-octylaniline in toluene. In present study, the use of n-octylaniline in toluene for the extraction of platinum (IV) from ascorbate media was carried out. The effect of various parameters, such as pH, equilibrium time, extractant concentration and organic solvent on the extraction has been discussed. The back extraction of has been performed. On the basis of slope analysis, the composition of the extracted species was determined as $[RR'NH_2^+ \text{ Pt (Succinate)}_2^-]_{(org)}$. The interfering effects of various cations and anions were also studied, and use of suitable masking agents enhances the selectivity of the method. The proposed method is rapid, reproducible and successfully applied for the determination of platinum (IV) in binary and synthetic mixtures. The separation of platinum (IV) from other associated metals has been studied. Comparison of the results with those obtained using an atomic absorption spectrophotometer were also tested the validity of the method.

Keywords: Platinum (IV); Solvent extraction; n-octylaniline; Toluene

Introduction

The platinum group of metals (PGMs: Pt, Pd, Rh, Ir, Ru, and Os) are extremely scarce in comparison to the other precious metals due to not only to their low natural abundance but also to the complexity of the processes required for their extraction and refinement. In comparison with other precious metals (Au and Ag), these platinum group metals (PGMs) are extensively in the use as catalysts in the automobile, chemical and petroleum industries. In automobiles, Pt and Pd are used as catalytic converter to reduce harmful emissions. In the petroleum cracking process, the catalyst containing Pt and Pd helps to break oil molecules into smaller organic molecules [1]. Besides, PGMs are also used as conductors in the electrical and electronic industries, in extrusion devices, in dental and medical prostheses, and in jewellery [2]. Due to their special chemical and physical properties, the substitution of PGMs by other metals is difficult [3]. Since the need for PGMs are more, recovery of PGMs from spent catalysts is very important.

Economically, the precious metals have been historically important as currency, and remain important as investment commodities. Gold, silver, platinum and palladium are internationally recognized as forms of currency under ISO 4217 [4]. Technological and analytical platinum-containing solutions have diverse ratios of the concentrations of platinum and associate precious, base, and ferrous metals, acidities, and salt backgrounds. This diversity arises from the complex and variable composition of raw materials. Naturally, selective extraction and separation of platinum (as well as platinum-group metals and gold) from associate metals becomes a problem in subsequent hydrometallurgical processing of these complex solutions. Technological and analytical platinum-containing solutions have diverse ratios of the concentrations of platinum and associate precious, base, and ferrous metals, acidities, and salt backgrounds. This diversity arises from the complex and variable composition of raw materials. A major problem in developing extraction methods for the platinum group metal ions, especially for platinum (II) arises from slow extraction rates. Heating the solution [5-7] and/or the use of tin (II) chloride as a catalyst [8-10] have been commonly used to accelerate the extraction. Among the PGMs separations, Pd (II)/Pt (IV) mutual separation has been extensively investigated due to its difficulty based on their chemically similar properties. The large difference in the extraction rate between Pd (II) and Pt (IV) from aqueous chloride solutions, which is attributable to the extremely inert properties of Pt (IV) compared with Pd (II) is usually applied to their separation [11].

High molecular weight amine (HMWAs) finds many applications in the solvent extraction of platinum (IV) from weak organic acid and mineral acid medium. These are p-octylaniline [12], N-octyl-, N,N-dioctyl aniline and N,N,N-trioctyl anilinium O,O-di(iso-propyl) dithiophosphates [13], N-n-octylaniline [14], Tri-octylamine [15-20], alamine 336 [21-24], alamine 300 [25], quaternary amine base salts [25-32]. However, these amines shows emulsion formation, narrow extraction pH range. Extraction of platinum (II) by bis-acylated diethylenetriamine [33] from hydrochloric acid solution was studied. Platinum can be separated by this method from non-noble metals. Extraction of platinum (II) from acidic solution at pH 1-4 by using two hydrophobic analogs of N,N,N',N'-tetrakis [2-pyridyl-methyl]-1,2-ethyl-enediamine (TPEN).

N,N,N',N'-tetrakis[4-(2-butyloxy)-2-pyridyl-methyl]-1,2-ethylenediamine (TBPEN) and N,N,N',N'-tetrakis (2-quinolinylmethyl)-1,2-ethylenediamine (TQEN) [34] have shown enhanced solvent

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extraction performance in more acidic media than TPEN. The platinum ion was extracted in the deactivated catalyst for the hydrochlorination reaction of ethyne by acidic thiourea [35] solution. N'N'- dihexyl and phenyl and N'-hexyl and phenyl derivatives of N-benzoyl thiourea [36] are very good reagents for pH selective extractions, especially for platinum. Important interfering elements in the extraction of platinum as Cu, Fe, Ni. Solvent extraction of platinum (IV) by N,N-dihexyl-N'-benzoyl-thiourea [37] in toluene is substantially accelerated in the presence of stannous chloride. The extraction behavior of platinum (II) is not affected by the treatment of stannous chloride. Liquid- liquid extraction of the platinum (IV) with pure synthesized N,N-diethyl-N'-benzoyl-thiourea (DEBT) [38] was carried out by optimizing the concentration of acid, mole ratio of metal to chelating agent, temperature and extraction time. The extraction behavior of platinum (II) with 1,3-dimethyl-2-thiourea (DMTU) [39] from a chloride solution was studied. Bromocresol green ion as a counter anion and 1,2-dichloroethane as an extraction solvent were used. Platinum (II) was quantitatively extracted into 1,2-dichloroethane within 15 min. The interferences of Mn (II), Cu (II), Zn (II), Pd (II), Ag (I) and Cd (II) was removed by adding suitable masking agents. The extractant nonylthiourea (NTH) [40, 41] in chloroform has been investigated for the extraction of platinum (IV) from chloride solution at ionic strength 4.0 M. It has been found that chloride concentration has a negative effect on the extraction while proton concentration has no effect. N-Phenyl-N'-[o-(2-ethylhexylthio)phenyl] thiourea (PEPT) [42] which contained donor atoms of sulfur and nitrogen, was synthesized to develop a selective extractant for platinum (IV) from base metals from acidic chloride media at 303 K.

In present study, the use of n-octylaniline in toluene for the extraction of platinum (IV) from ascorbate media was carried out. The extraction system is studied as a function of pH, extractant concentration, diluents, and equilibrium period. The conventional slope analysis method was employed for analysis of species formed in organic phase. The method is free from large number of interferences due to various foreign ions.

The review of literature for comparison of liquid-liquid extractive separation of platinum (IV) is given in table 6.

Experimental

Instruments

An Elico digital spectrophotometer model 12 Chemito 215D with 1 cm quartz cells was used for absorbance measurements and pH measurements were carried out using an Elico digital pH-meter model LI-127. All weighing operations were carried out by using Tapson's analytical single pan balance model 200 T having 0.001 g accuracy.

Chemicals and solutions

Standard solution of platinum (IV) was prepared by dissolving 1 g of hydrogen hexachloro palatinate (IV) hydrate, $H_2PtCl_6.H_2O$ (Johnson and Matthey, U.K.) in 1 M hydrochloric acid and was standardized gravimetrically [43]. A working solution (100 µg/mL) was made there from by appropriate dilution. All chemicals used were of AR grade. Double distilled water was used throughout.

n-octylaniline, 0.1 mol L^{-1}: The extractant n-octylaniline was prepared by the method of Pohlandt's [44] and its 0.1 M solution was prepared in toluene. All other solutions were prepared from A. R. grade reagents and aqueous solutions were prepared using water.

Standard solution of diverse ions was prepared by dissolving AR grade reagents in water or dil HCl. All the organic solvents were used after double distillation. All chemicals used were of AR graded.

General extraction and determination procedure for platinum (IV)

An aliquot of 150 µg platinum (IV) solution was mixed with a sufficient quantity of ascorbic acid to make its concentration 0.01 M in a total volume of 25 mL of the solution. The pH of the aqueous solution was adjusted to 0.50 by dilute sodium hydroxide and hydrochloric acid solution. The solution was then transferred to a 125 mL separating funnel and shaken with 10 mL of 0.1 M n-octylaniline in toluene for 3 min. After separating the two phases, the aqueous phase was discarded and the organic phase was stripped with two 10 mL portions of water solution. The stripped aqueous phase was evaporated to moist dryness and extracted into water.

A percentage extraction (% E) and metal distribution ratio (D) were calculated according to Eqs. (1) and (2), respectively.(1)

$$\%E=\frac{[M]_{org}}{[M]_{aq.,init}}\times 100 \tag{1}$$

$$D=\frac{[M]_{org}}{[M]_{aq}} \tag{2}$$

Where, $[M]_{aq., init.}$ represents the initial concentration of metal ion in the aqueous phase. $[M]_{aq.}$ and $[M]_{org.}$ are the total concentrations of metal ion in the aqueous and organic phases after equilibrium, respectively.

Estimation procedure for platinum (IV): The resulting aqueous phase was mixed with 5 mL of concentrated hydrochloric acid in 25 mL volumetric flask and 10 mL of 25% (W/V) stannous chloride in concentrated hydrochloric acid and the solution was diluted to mark with water. The absorbance of the resulted solution was measured at 405 nm [45]. The concentration of platinum (IV) was found from calibration curve.

Results and Discussion

Extraction as a function of pH

The extraction study of platinum (IV) was performed at fixed concentration of 0.01 M ascorbic acid and between pH 0.10-5.0 with a 0.1 M solution of n-octylaniline in toluene. The pH range observed for the quantitative extraction was 0.50-1.0 with n-octylaniline. Hence the extraction of platinum (IV) was carried out at pH 0.50 for all extraction experiments (Figure 1).

Effect of weak organic acid concentration

The extraction of platinum (IV) was examined at pH 0.50 with 0.1 M n-octylaniline in toluene in presence of varying concentrations from 0.001-0.1 M of ascorbic acid. The extraction of ion-pair complex of platinum (IV) was found to be quantitative in the range of 0.007-0.02 M ascorbic acid. Hence, 0.01 M concentration of ascorbic acid was used for further studies while zero extraction of platinum (IV) was found to be in sodium salicylate, malonate, succinate (Figure 2).

Effect of n-octylaniline concentration

Extraction of platinum (IV) was carried out with various concentrations of n-octylaniline in toluene. To optimize the extraction condition, other parameters like pH, period of equilibration and diluent

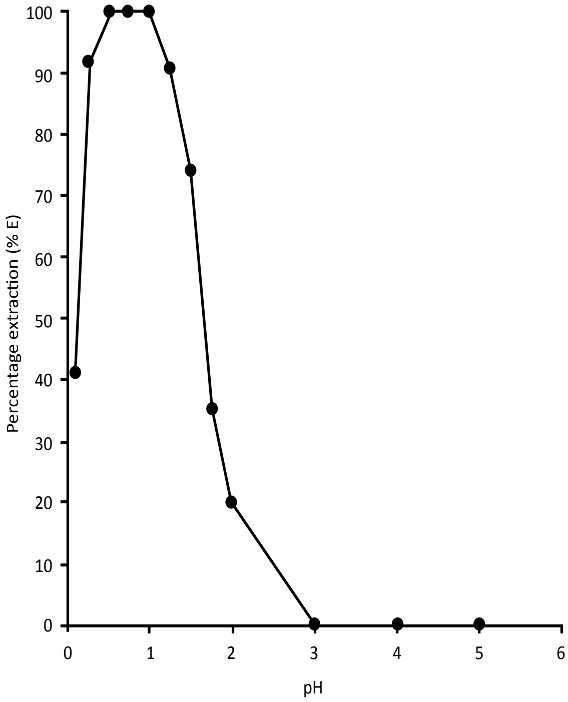

Figure 1: Plot of pH versus percentage extraction of platinum (IV) (150 µg/mL) from Ascorbic acid medium (0.01 M) by using n-octylaniline (0.1 M) as an extractant in toluene with 3 min shaking time.

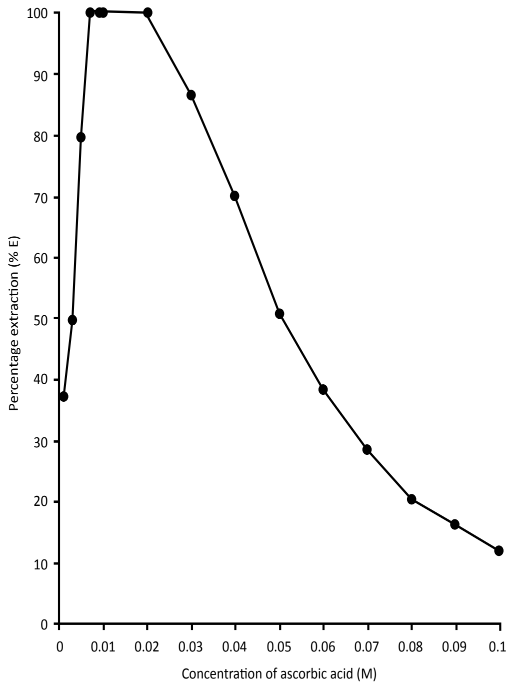

Figure 2: Extraction of platinum (IV) (150 µg) with 0.1 M n-octylaniline at pH 0.50 as a function of weak acid concentration.

were kept constant. The extraction was found to be increased with increasing reagent concentration. The extraction of platinum (IV) was quantitative in the range 0.09 M to 0.4 M of n-octylaniline in toluene. However, 10 mL of 0.1 M n-octylaniline in toluene was recommended for general extraction procedure (Figure 3).

Effect of diluents

The studies were then performed to find out the most suitable solvent for the extraction of the ion-pair complex of platinum (IV). It was found that a 0.1 M solution of n-octylaniline in benzene, toluene, xylene provides quantitative extraction of platinum (IV). The extraction of platinum (IV) was incomplete if n-octylaniline is dissolved in chloroform (23.8%), methyl isobutyl ketone (60.8%) while no extraction in amyl alcohol, 1,2-dichloroethane, n-butyl alcohol, amyl acetate (Table 1). On safety ground, toluene was preferred to other solvents.

Effect of equilibration time

The extraction of platinum (IV) was studied for various time

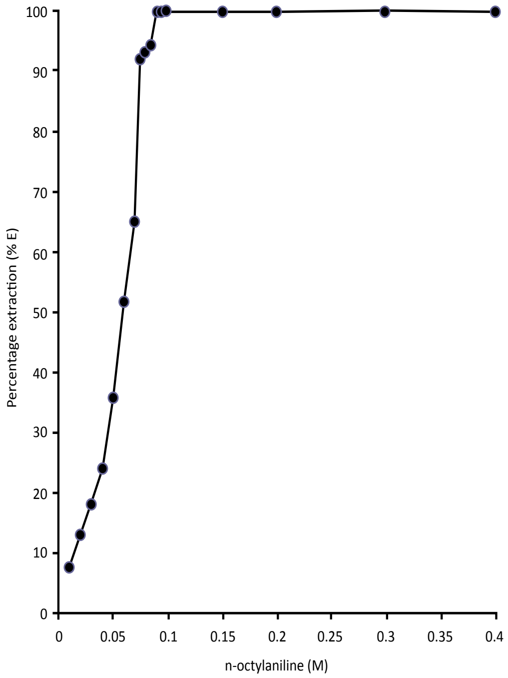

Figure 3: Extraction of platinum (IV) (150 µg/mL) at pH 0.50 from 0.01 M Ascorbic acid medium as a function of n-octylaniline concentration.

Solvent	Dielectric constant, (ε)	Percentage extraction, (% E)	Distribution ratio, (D)
Benzene	2.27	100	∞
Xylene	2.30	100	∞
Toluene*	2.38	100	∞
Chloroform	4.80	23.8	0.78
Methyl isobutyl ketone	13.10	60.8	3.87
n-Butanol	17.80	No extraction	-
Amyl alcohol	13.90	No extraction	-
Amyl aceate	13.90	No extraction	-
1, 2-Dichloroethane	10.50	No extraction	-

Pt(IV)=150 µg Aq: Org=2.5: 1
pH=0.50 Ascorbic acid=0.01 M
Equilibrium time=3 min Strippant=Water (2×10 mL)* Recommended for general extraction procedure

Table 1: Extraction behaviour of platinum (IV) as a function of diluents.

intervals in the range of 1-20 min with 0.1 M n-octylaniline. It was observed that, under the optimized experimental conditions a minimum 2 min time interval was required for attaining equilibrium in the sense to extract platinum (IV) quantitatively. But with prolonged shaking over 10 min there was decrease in the percentage extraction of platinum (IV) due to the dissociation of ion-pair complex. Hence, in all further studies both the phases were equilibrated for 3 min.

Effect of stripping agent

Platinum (IV) from organic phase was stripped with the two 10 mL portions of various stripping agents at different concentrations of mineral acids, buffer solutions and some bases. Platinum (IV) was quantitatively stripped with water (Table 2). However, percentage recovery of platinum (IV) from organic phase was found to be incomplete with strippants ammonia, hydrochloric acid, nitric acid, sulphuric acid, sodium chloride and hydrobromic acid and no stripping in ammonia buffer (pH 10). In recommended procedure, two 10 mL portions of water were used for the complete stripping of loaded organic phase [46].

Effect of aqueous to organic volume ratio

The extraction of platinum (IV) was carried out in different aqueous volumes in the range 10-100 mL from 0.01 M ascorbic acid medium with 10 mL 0.1 M n-octylaniline in toluene (Table 3). There was observed quantitative extraction of platinum (IV) when phase ratio A/O maintained from 1:1 to 4:1. Therefore, in the recommended procedure the phase ratio 2.5:1 was maintained through the all experimental study.

Strippant	mol/L/pH	Percentage extraction, (%E)
Ammonia	1-10	53.4
HCl	1-3	16.7
H_2SO_4	1-3	20.7
HNO_3	1-3	31.9
HBr	1-3	12.3
Water*	-	100
NaCl	1-5%	65.2
Ammonia buffer	pH-10	No stripping

*Recommended for general extraction procedure

Pt(IV)=150 µg, Aq: Org=2.5: 1
pH=0.50, Ascorbic acid=0.01 M
Equilibrium time=3 min, n-octylaniline=0.1 M in toluene

Table 2: Extraction behavior of platinum (IV) as a function of stripping agents.

Aqueous to organic volume ratio	Percentage extraction (% E)	Distribution ratio (D)
10:10	100	∞
20:10	100	∞
25:10*	100	∞
30:10	100	∞
35:10	100	∞
40:10	100	∞
50:10	94.6	43.79
75:10	74.1	7.15
100:10	59.4	3.65

*Recommended for general extraction procedure

Pt(IV)=150 µg, Aq: Org = 2.5: 1
pH=0.50, Ascorbic acid=0.01 M
Equilibrium time=3 min, n-octylaniline=0.1 M in toluene

Table 3: Extraction of platinum (IV) as a function of aqueous to organic volume ratio.

Metal loading capacity of extractant

The influence of the initial platinum (IV) concentration 50-2500 µg on the extraction by 0.1 M n-octylaniline in toluene was studied. It was observed that, varying the initial platinum (IV) concentration in the range of 50-1500 µg has no significant influence on platinum (IV) extraction with the 10 mL of 0.1 M extractant (Table 4). The maximum loading capacity of 10 mL 0.1M solution of n-octylaniline in toluene was found to be 1500 µg platinum (IV).

Nature of extracted species

Attempts were made to ascertain the nature of extracted species of platinum (IV) with the extractant using conventional slope analysis method. The distribution ratio of platinum (IV) evaluated at different concentration in molar of ascorbic acid was used at fixed n-octylaniline concentration at pH 1.25 and pH 1.50. A graph of log $D_{[Pt(IV)]}$ versus log $C_{[ascorbate]}$ gave a slope of 2.8 and 3.0 respectively (Figure 4). Similarly,

Pt(IV), (µg)	Percentage extraction	Distribution ratio
50	100	∞
100	100	∞
150*	100	∞
200	100	∞
300	100	∞
600	100	∞
800	94.6	∞
1000	74.1	∞
1500	59.4	∞
2000	81.5	11.01
2500	39.5	1.63

*Recommended for general extraction procedure

pH=0.50, Aq: Org=2.5: 1
Ascorbic acid=0.01 M, Equilibrium time=3 min
n-octylaniline=0.1 M in toluene

Table 4: Metal loading capacity of n-octylaniline.

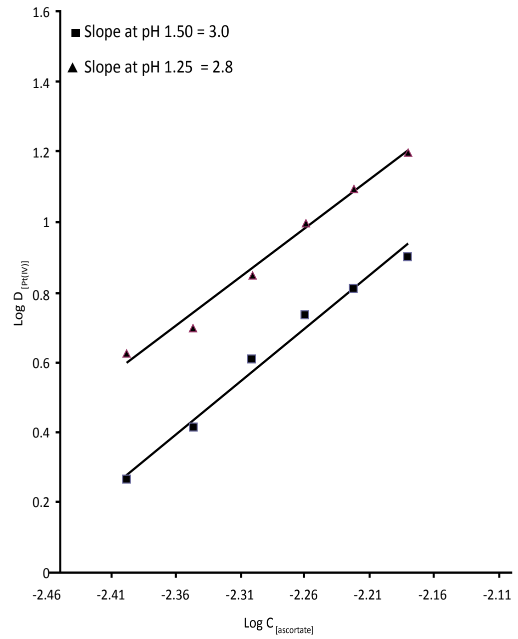

Figure 4: Log-log plot of distribution ratio $D_{[Pt(IV)]}$ versus Log $C_{[ascorbate]}$ at fixed n-octylaniline concentration.

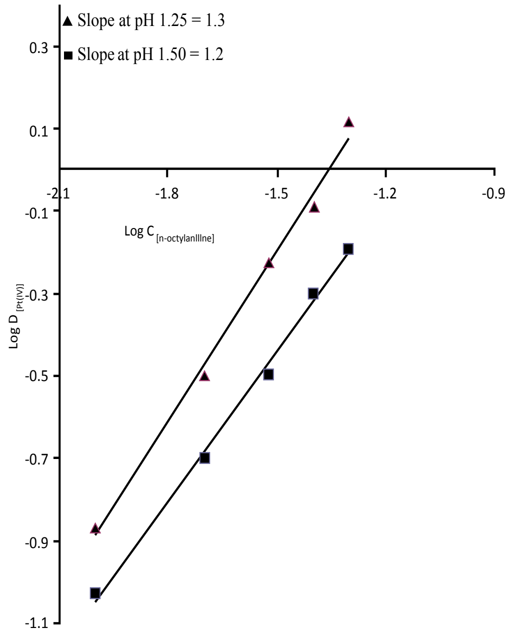

Figure 5: Log-log plot of distribution ratio $D_{[Pt(IV)]}$ versus Log $C_{[n\text{-octylaniline}]}$ at fixed ascorbate concentration.

Tolerance limit, mg	Foreign ion added
25	Tartrate, Mg(II)
15	Fe(III), Zn(II), Mo(VI), Se(IV), Ba(II), Ce(IV), oxalate, citrate, bromide
10	Ni(II), Co(II), U(VI), Hg(II)
5	Cu(II), Pb(II), Cr(VI), Cr(III), Bi(III), Te(IV), Cd(II), EDTA, nitrate
2	Tl(I), Ca(II), Al(III), Cr(III), Cd(II)
1	Ag(I), Os(VIII), Ru(III)
0.5	Rh(III)[a], Pd(II)[a], Ir(III), Au(III)

[a]masked with 20 mg tartrate

Pt(IV)=150 µg, Aq: Org=2.5: 1
pH=0.50, Ascorbic acid=0.01 M
Equilibrium time=3 min, n-octylaniline=0.1 M in toluene

Table 5: Effect of foreign ions on the extraction of platinum (IV).

a plot of log $D_{[Pt(IV)]}$ versus log $C_{[n\text{-octylaniline}]}$ concentrations at a fixed pH 1.25 and pH 1.50 with 0.1 M ascorbate gave slope of 1.3 and 1.2 respectively (Figure 5). This indicates a mole ratio of platinum (IV) to ascorbic acid as 1:3 and that of n-octyaniline as 1:1. Thus, the extracted species was calculated to be an ion association complex with the probable composition 1:3:1 (metal:acid:extractant).

In the extraction of platinum (IV) with ascorbate medium, first platinum (IV) was reduced to platinum (II) [47] then it was converted into platinum(II) ascorbate as an anion and interacted with $[CH_3(CH_2)_7C_6H_4NH_3]+_{(org)}$. Hence the probable extracted species in toluene is $[CH_3(CH_2)_7C_6H_4NH_3.Pt(C_6H_7O_6)_3^-]_{(org)}$.

Effect of various diverse ions on percentage extraction

The effect of various diverse ions was tested, when platinum (IV) was extracted with n-octylaniline in toluene. The tolerance limit was set as the amount of foreign ion causing a change ± 2% error in the recovery of platinum (IV). It was observed that the method is free from interference from a large number of cations and anions (Table 5). Species like iodide, thiocyanate, thiourea, palladium (II) and rhodium (III) interfere in the extraction of platinum (IV). Howee4r, the interference of Pd (II) and Rh (III) was eliminated by masking with tartrate.

Applications

Separation and determination of platinum (IV) from binary mixture

The separation of Pt (IV) from some commonly associated metal ions like Ru (III), Au (III), Os (VIII), Se (IV), Te (IV), Fe (III), Co (II), Ni (II) and Cu (II) using n-octylaniline was achieved by taking advantage of the difference in the extraction conditions of metal such as pH of the aqueous phase, reagent concentration and use of masking agent (Table 6). All the added metal ions were remained quantitatively in aqueous phase from which they are determined spectrophotometrically by standard methods [45,47-52].

Rh (III) and Pd (II) interfere in the extraction of Pt (IV). Rh (III) and Pd (II) were separated from Pt (IV) by masking with 20 mg tartrate.

Analysis of platinum (IV) in catalysts sample

Platinum (IV) activated alumina (0.1 g) was dissolved in 20 mL aqua regia. The solution was evaporated to moist dryness. Two 5 mL portions of hydrochloric acid were added and evaporated till all the nitric acid was removed. The residue was extracted in 1 M hydrochloric acid. The solution was filtered and the filtrate was diluted to 100 mL. An aliquot of this diluted solution was analyzed for platinum (IV) content by the proposed method. It was found that there is a good agreement with the certified value (Table 7).

Analysis of platinum (IV) from real samples

Cytoplatin (cisplatin injection): The method permits the

Amount of metal ion, (µg)	Mass taken,/µg	Average (%) Recovery*	Chromogenic ligand
Pt(IV)	150	99.5	
Ir(III)	100	99.4	stannous chloridehydrobromic acid
Pt(IV)	150	99.7	
Rh(III)[a]	300	99.2	stannous chloride-KI
Pt(IV)	150	99.8	
Pd(II)[a]	200	99.4	4'-ChloroPTPT
Pt(IV)	150	99.2	
Ru(III)	200	98.9	4'-ChloroPTPT
Pt(IV)	150	99.8	
Au(III)	200	99.1	$SnCl_2$
Pt(IV)	150	99.7	
Se(IV)	200	99.0	4'-ChloroPTPT
Pt(IV)	150	99.8	
Te(IV)	200	98.9	4'-ChloroPTPT
Pt(IV)	150	99.4	
Os(VIII)	200	99.0	Thiourea
Pt(IV)	150	99.4	
Fe(III)	500	98.7	Thiocynate
Pt(IV)	150	99.0	
Cu(II)	1000	98.6	4'-ChloroPTPT
Pt(IV)	150	99.0	
Ni(II)	1000	97.9	DMG
Pt(IV)	150	99.9	
Co(II)	500	97.8	Thiocynate

[a]masked with 20 mg tartrate

Table 6: Separation of platinum (IV) from binary mixtures.

Sr. No.	Alloy	Platinum (IV) taken, µg	Platinum (IV) *Found by method in µg	Average, % recovery	Average, % recovery
1.	Pt-Pd[a] -Rh[a] catalyst on alumina	150	149.2	99.4	0.6
2	Pt-Rh[a] catalyst on alumina	150	149.5	99.6	0.4
3	Pt-Pd[a] catalyst on alumina	150	149.8	99.8	0.2
4	Pt catalyst on alumina	300	299.5	99.8	0.2

[a] masked with 20 mg tartrate.

*Average of five determinations

Composition of synthetic mixtures in percentage
1. Pt, 0.03-0.25; Rh, 0.005-0.03
2. Pt, 0.03-0.15; Pd, 0.02-0.12
3. Pt, 0.03-0.20; Pd, 0.03-0.150; Rh, 0.015-0.05
4. Pt, 0.3-0.8

Table 7: Determination of platinum (IV) from catalysts.

Sample	Manufacturer	Amount taken	Amount of platinum(IV) found	RSD, %
Cytoplatin (cisplatin injection)	Cipla, Goa	200 µg/ mL	198.6 µg/mL	0.7
Platinumrhodium[a] thermocouple wire (Pt, 87; Rh,13)	Ruia Resistance Wire Pvt. Ltd., Mumbai	87%	86.4%	0.6

[a] masked with 20 mg tartrate

Table 8: Analysis of platinum (IV) from real samples.

separation and determination of platinum (IV) from drug sample, cytoplatin (cisplatin injection) and platinum-rhodium thermocouple wire. Cytoplatin (cisplatin injection) in an antineoplastic agent with the biochemical properties similar to those of bifunctional alkylating agents. Although, not yet clearly established, its cytotoxic actions and antitumor activity is consistent with the hypothesis that major cytotoxic target of cisplatin in DNA. The drug covalently binds to DNA bases and disrupts DNA functions. Cytoplatin appears to enter cells by diffusion.

Cytoplatin is a sterile solution of cisplatin I.P. 1.0 mg/mL, sodium chloride I.P. 9 mg/mL in water for injection I.P. Cytoplatin containing cisplatin which is a heavy metal platinum coordination complex containing a central atom of platinum surrounded by two entities each of chloride ions and two ammonia molecules in cis geometry. It has a melting point of 207°C.

The empirical formula of the active compound is $PtCl_2H_6N_2$ with a molecular weight of 300.1.

A known volume (10 mL) of cisplatin solution was digested in perchloric acid /nitric acid (10:1) and evaporated to dryness until organic matter was removed. The obtained residue was dissolved in concentrated hydrochloric acid and diluted with water to 10 mL in a standard volumetric flask.

An aliquot of the sample solution was taken and platinum (IV) was determined using the recommended procedure (Table 7).

Thermocouple wire: A known weight (0.100) of thermocouple wire was preliminary fused with zinc powder and the melt was cooled and dissolved in hydrochloric acid. The black powder remained was treated with 5 mL aqua regia. After the reaction was over the whole solution was heated with two 5 mL portions of concentrated hydrochloric acid until complete removal of oxides of nitrogen and diluted with distilled water to 10 mL in standard volumetric flask.

An aliquot of sample solution was taken and platinum (IV) was determined using the procedure described earlier. The results of the analysis are given in table 8.

Conclusion

i. Quantitative extraction of platinum (IV) was achieved in 3 min with 0.1 M n-octyaniline in toluene at pH 0.50.

ii. Extraction reaction occurred through anion-exchange mechanism.

iii. Developed method is efficient for quantitative separation of platinum (IV) in presence of various interfering cations and anions.

iv. The proposed extractive separation method is simple, rapid, selective reproducible and suitable for separation and determination of platinum (IV) from associated metal ions, real samples and synthetic mixtures.

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