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EXTRACTION STUDIES OF PLATINUM GROUP METALS WITH CYANEX 925 IN TOLUENE—ROLE OF TIN(II) CHLORIDE IN THEIR SEPARATION

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ABSTRACT

Extraction of platinum group metals Os(VIII), Ru(IV), and Ir(III) was carried out from aqueous chloride media with Cyanex 925 in the absence and in the presence of tin(II) chloride. In the presence of hydrochloric acid (HCl) alone, only Os(VIII) and Ru(IV) get extracted quantitatively, while extraction of Ir(III) was incomplete. Further, Os(VIII) could be extracted at lower concentrations of HCl (1×10^{-2}–$5 \times 10^{-2}\,M$) and Ru(III) at higher concentrations (3.5–5.0 M). In the presence of tin(II) chloride, the extraction of Ir(III) increases and becomes quantitative. However, it decreases to some extent in the case of Os(VIII) and Ru(IV). The extraction conditions for all the metal ions were optimized under influence of variables such as HCl concentration, reagent concentration, tin(II)chloride concentration, equilibration time, stripping agents, and effect of diverse ions. The separation of individual metal ions Os(VIII), Ru(IV), and Ir(III) was carried out

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by taking advantage of differences in their extraction and stripping conditions towards Cyanex 925. The methods developed were applied to the recovery of these metal ions from some real catalysts samples.

Key Words: Extraction; Os(VIII); Ru(IV); Ir(III) Cyanex 925; Stripping; Separation

INTRODUCTION

Platinum metals are used in areas such as manufacturing of automobile catalysts, cancer therapy, and homogeneous phase catalysis. However, the scarcity of these metals has led to mining from low-grade ores where these metals are present in trace levels. Since platinum metal materials retain a large proportion of their initial value, resulting in the occurrence of many diverse scrap materials that are sources of recoverable platinum metals (1). Therefore, the recovery and recycling of these metals have become important.

The extraction study of platinum metals with organophosphorus extractants has been reported by different authors (2–5). There are many other extraction methods reported for extraction and separation of platinum metals (6–11). In a previous paper, the authors have studied the extraction behavior of Rh(III), Pt(IV), and Pd(II) with neutral organophosphine oxide, Cyanex 925 (12). The present investigation is concerned with the extraction separation study of Os(VIII), Ru(IV), and Ir(III) with Cyanex 925 in toluene.

EXPERIMENTAL

Apparatus and Reagent

The GBC 911A/UV visible spectrophotometer was used for absorption measurements. Cyanex 925 is a liquid mixture containing 19 components with 26.4% tri (2,4,4-trimethyl pentyl) phosphine oxide and tri-octyl phosphine oxide (65.9%) as major components, while the other components consist of sulfur compounds (13). The extractant supplied by Cytec Canada Industries Inc., Canada, was used without further purification. The stock solutions of Os(VIII) and Ir(III) were prepared by dissolving analytical reagent grade OsO_4 and $IrCl_3$ in minimum quantity of dilute hydrochloric acid (HCl) ($0.1\,M$). However, Ru(IV) solution was made by dissolving $RuCl_3$ using chlorine water (14). These solutions were standardized by known methods (15,16). The required concentration of their solution was prepared by further dilution with double distilled water. The tin(II) chloride solution prepared in 2.0 M HCl was used for experimental study. All other chemicals used were of analytical reagent grade.

General Extraction Procedure

An aliquot of solution containing 100 μg of each platinum metal ion [Os(VIII)/Ru(IV)/Ir(III)] was taken in a separating funnel, and varying concentrations of HCl or tin(II)chloride were added to it for extraction studies. Ten cubic centimeters of this aqueous phase was equilibrated for 10 min (and 1 min in presence of tin(II) chloride) with an equal volume of 1×10^{-1} M Cyanex 925 in toluene. In the presence of tin(II) chloride, these metal-ion solutions were also maintained at a temperature of 60°C for 15 min before extraction. The two phases were allowed to separate. The Os(VIII) and Ru(IV) concentrations in the aqueous phase were determined spectrophotometrically by thiourea method at 480 and 620 nm, respectively, while Ir(III) concentration was determined by *p*-nitroso dimethyl aniline method at 530 nm (16). The metal-ion concentration in the metal-loaded organic phases was determined by mass balance of the metal before and after extraction.

RESULTS AND DISCUSSION

Influence of Hydrochloric Acid Concentration

Os(VIII), Ru(IV), and Ir(III) were extracted in the acidity range of $5 \times 10^{-3} - 8 \times 10^{0}$ M HCl, with 1×10^{-1} M Cyanex 925 in toluene. Os(VIII) and Ru(IV) were quantitatively extracted in presence of HCl, while not more than 50% of Ir(III) was extracted under the studied HCl range (Fig. 1). Further, the quantitative extraction of Os(VIII) takes place at a lower range of HCl $1 \times 10^{-2} - 5 \times 10^{-2}$ M, while for Ru(IV), it takes place at a higher concentration $3.5 - 5.0$ M.

Ru(IV) is more labile towards aquation, whereas osmium is inert like iridium; therefore, the extraction rate for Ru(IV) increases with addition of chloride ions and decreases in the case of osmium and iridium (17). However, at very high HCl concentrations, the extraction of all these platinum metals decreases due to the mass action effect of chloride ion (18).

Influence of Tin(II) Chloride

The extraction of Os(VIII), Ru(IV), and Ir(III) was studied with the varying concentration range $1 \times 10^{-3} - 1.0$ M tin(II) chloride at 1.0 M fixed HCl

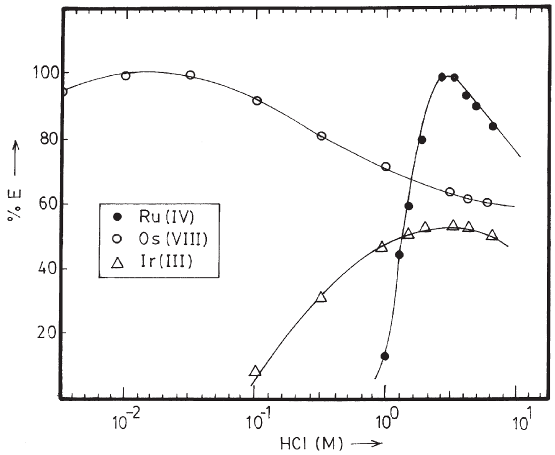


Figure 1. Effect of HCl concentration on *% E*. (Eq. time = 10 min; organic phase = $1 \times 10^{-1}\,M$).

concentration and 1 min equilibration time, with and without heating these metal-ion solutions before extraction at 60°C for 15 min. In presence of tin(II) chloride, the extraction of Ir(III) was found to increase with tin(II) chloride concentration and it was maximum (~80%) with $1 \times 10^{-1}\,M$ tin(II) chloride (Fig. 2). However, the extraction was complete only if it was maintained at 60°C for 15 min before extraction, and it was incomplete for Os(VIII) and Ru(IV) (Fig. 3).

In the presence of tin(II) chloride, the Ir(III) metal ion chloride complexes are activated by their reduction to Ir(I)–tin complexes, with simultaneous oxidation of Sn(II) to Sn(IV). Similar to Rh(III), these complexes are more labile than the other kinetically inert complexes due to the π acid nature of $SnCl_3^-$ (19–21). The chloride complexes of both rhodium and iridium react very slowly to form extractable anionic complexes. The use of labalising agent (22,23) and heating before extraction (5) increases their extraction rate. However, both Rh(III) and Ir(III) have differences in their reaction rate such that more extractable Ir(III) complexes are formed only by heating before extraction (24). The decrease in extraction of Os(VIII) and Ru(IV) to some extent by addition of tin(II) chloride is contradictory to Ir(III) due to the co-extraction of tin with Cyanex 925 as reported earlier (12). In the presence of tin(II) chloride, Ru(IV) was reduced to less-extractable Ru(III) (25). While, as suggested by Mojsky (5), Os(VIII) is also reduced to a less-stable oxidation state lower than the tetravalent. Further, the optimized concentration of tin(II) chloride required for quantitative extraction of Ir(III) was found to be $1 \times 10^{-1}\,M$.

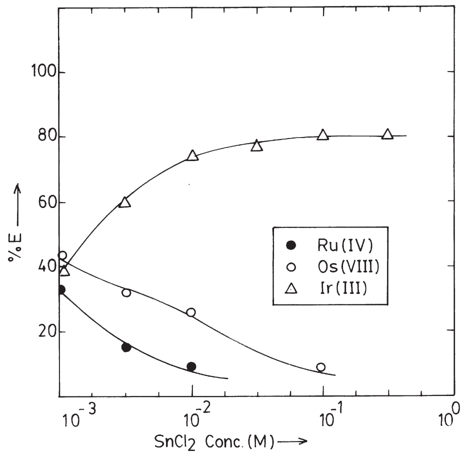


Figure 2. Effect of $SnCl_2$ concentration on % E of Os(VIII), Ru(IV), and Ir(III)—without heating the solution before extraction. (Eq. organic phase = $1 \times 10^{-1} M$).

Influence of Reagent Concentration

The platinum metal ions were extracted with varying concentrations of Cyanex 925 (1×10^{-5}–$1 \times 10^{-1} M$) in toluene. The quantitative extraction for Os(VIII) and Ru(IV) was found to take place above 1×10^{-2} and $1 \times 10^{-1} M$

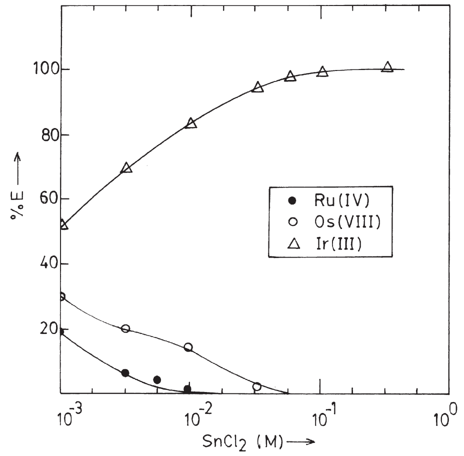


Figure 3. Effect of $SnCl_2$ concentration on % E of Os(VIII), Ru(IV), and Ir(III)—on heating the solution before extraction. (Eq. time = 1 min; organic phase = $1 \times 10^{-1} M$).

Cyanex 925 in the presence of 0.025 and $4.0\,M$ HCl, respectively. However, Ir(III) extraction was quantitative with $5 \times 10^{-2}\,M$ Cyanex 925 in the presence of $1 \times 10^{-1}\,M$ tin(II) chloride. From the slope ratio method (Fig. 4), the metal to Cyanex 925 ratio was found to be 1:2 for Ru(IV) and Ir(III), while it was 1:1 in the case of Os(VIII). Thus, in the absence of tin(II) chloride, the probable composition of Os(VIII) and Ru(IV) in the extracted complex can be given as OsO_4·Cyanex 925 and $RuCl_4$·2Cyanex 925, respectively. Yamakawa et al. (20) have reported the presence of Ir(III) species in tin(II) chloride containing HCl as $[IrH(SnCl_3)_5]^{3-}$, therefore the probable species of Ir(III) with Cyanex 925 may be $[IrH(SnCl_3)_5]^{3-} \cdot 3H^+ \cdot$2Cyanex 925.

Influence of Equilibration Time

The aqueous phase containing Os(VIII)/Ru(IV)/Ir(III) was equilibrated from 1–30 min with $1 \times 10^{-1}\,M$ Cyanex 925 in toluene. The quantitative extraction for Os(VIII) was observed to take place with a minimum equilibration period of 6 min and Ru(IV) in 5 min. In case of Ir(III), a 1 min equilibration period was found to be sufficient for its quantitative extraction. The extraction remains quantitative up to 30 min for all these metal ions.

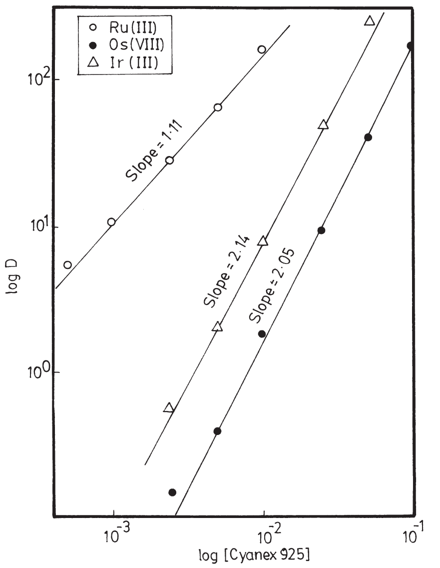


Figure 4. Effect of Cyanex 925 concentration on distribution ratio (D). [(○) Os(VIII), Slope = 1.11; (●) Ru(IV), Slope = 2.05; (Δ) Ir(III), Slope = 2.14].

Influence of Diverse Ions on Percentage Extraction

The extraction of 100 μg each of Os(VIII), Ru(IV), or Ir(III) was carried out in presence of a large number of metal ions. The tolerance limit was set so that the foreign ion causing interference was not more than ± 2% in the extraction of Os(VIII)/Ru(IV)/Ir(III). The relative tolerance limits of each of these metal ions with respect to the diverse ions are reported in Table 1.

Influence of Various Stripping Agents

The metal-loaded organic phase containing Os(VIII)/Ru(IV)/Ir(III) was stripped with various stripping agents such as HCl, H_2SO_4, HNO_3, and thiourea (Table 2).While, in case of Ru(IV), the stripping was carried out using HCl in 1% ascorbic acid. The complete stripping for Os(VIII) was observed only with 2.0–4.0 *M* thiourea and Ru(IV) with 0.5–1.0 *M* HCl in 1% ascorbic acid. While in case of Ir(III), it was found to be complete with 8.0–10.0 *M* HCl or 4.0 *M* HNO_3.

Separation of Os(VIII), Ru(III), and Ir(III)

Separation of Os(VIII), Ru(IV), and Ir(III) metal ions from their mixture was carried out by taking advantage of differences in their extraction and stripping conditions towards Cyanex 925. For their separation, the aqueous solution containing a mixture of 100 μg of each of the metal ions

Table 1. Effect of Diverse Ions on % Extraction of Os(VIII), Ru(IV), and Ir(III) Aqueous Phase: 100 μg Each Os(VIII)/Ru(IV)/Ir(III)

	Tolerence Limit		
Diverse Ions (μg)	Os(VIII)	Ru(IV)	Ir(III)
Na^+, K^+, Cs^+, Rb^+, Mg^{+2}, Ca^{+2}, Ba^{+2}, Sr^{+2}	1:22	1:15	1:25
V^{+5}, Mn^{+2}, Co^{+2}, Cu^{+2}, Cd^{+2}	1:16	1:10	1:18
Cr^{+3}, Zn^{+2}	1:14	1:12	1:20
Fe^{+3}	1:12	1:0	1:20
Rh^{+3}, Pt^{+2}	1:25	1:15	1:5
Al^{+3}, Bi^{3+}, In^{3+}, Ga^{3+}, Th^{3+}, Pd^{+2}, $Au + ^{3}$	1:8	1:12	1:20
Cl^-, Br^-, I^-, SO_3^{-2}, Citrate, Oxalate	1:20	1:15	1:25
Thiocyanate, thiourea	1:0	1:0	1:0

Table 2. Effect of Stripping Agent on % Recovery of Os(VIII), Ru(IV), and Ir(III). Loaded Organic Phase: 100 μg Each Os(VIII)/Ru(IV)/Ir(III)

Stripping Agents, [Concentration (*M*)]	Platinum Metal Ions		
	Os(VIII)	Ru(IV)	Ir(III)
HCl			
0.1	7.60	36.25[a]	5.80
0.5	7.60	99.60[a]	16.60
1.0	9.15	99.60[a]	31.25
2.0	18.86	78.15[a]	65.60
4.0	28.65	58.65[a]	78.60
6.0	28.10	48.20[a]	84.85
8.0	24.25	35.15[a]	99.10
10.0	18.40	28.14[a]	99.10
HNO_3			
1.0	11.65	11.60	68.15
2.0	16.40	10.85	79.10
4.0	31.10	7.50	98.85
6.0	30.85	5.45	97.60
H_2SO_4			
1.0	13.65	16.50	—
2.0	21.40	22.10	—
4.0	36.50	22.10	—
$HClO_4$			
0.5	36.10	61.50	28.25
1.0	53.25	78.80	58.50
2.0	53.60	78.15	78.65
4.0	50.40	77.20	78.00
Thiourea			
1.0	88.10	28.10	—
2.0	99.90	36.65	—
4.0	99.60	36.28	—

[a] Stripping in presence of 1% ascorbic acid.

Os(VIII)/Ru(IV)/Ir(III) in 2.5×10^{-2} *M* HCl was first equilibrated for 6 min with 1×10^{-2} *M* Cyanex 925. It was found that only Os(VIII) gets extracted in the organic phase, while Ru(IV) and Ir(III) remained unextracted. The extracted Os(VIII) from the organic phase was back-extracted with 2.0 *M* thiourea. While the mixture of unextracted Ru(IV) and Ir(III) was again equilibrated for 1 min at an aqueous acidity 1.0 *M* HCl + 1×10^{-1} *M* $SnCl_2$ (by maintaining the solution at 60°C for 15 min before extraction) using $5 \times 10^{-2}\,M$ Cyanex 925, when only Ir(III) got extracted and Ru(IV) remained unextracted. The extracted Ir(III) from the organic phase was back-extracted with $8.0\,M$ HCl (Sch. 1).

Multicomponent Separation of Os(VIII), Ru(IV), and Ir(III)

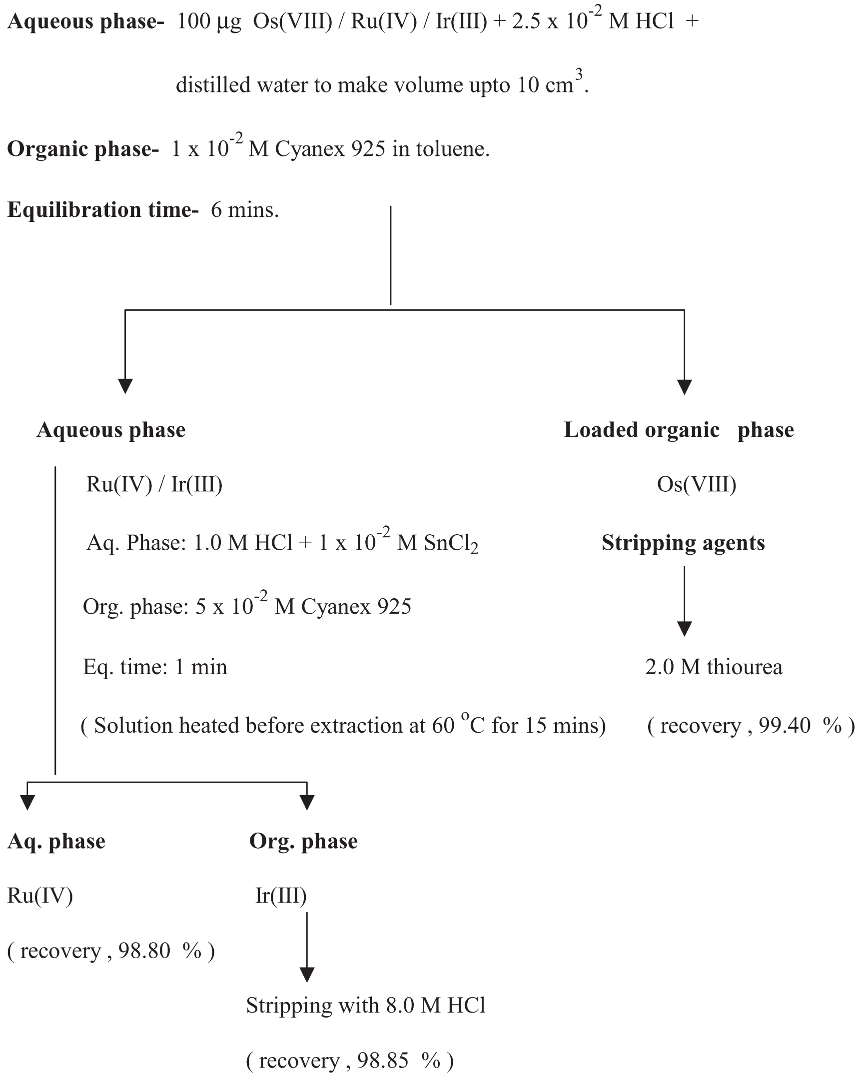
The binary and ternary separations of these metal ions from their mixture containing other platinum metals or some commonly associated transition metal ions [Fe(III), Co(II), and Ni(II)] were carried out by taking advantage of differences in their extraction and stripping conditions towards Cyanex 925.

Only the extraction of Os(VIII) was found to take place in presence of each of these metal ions at aqueous acidity $2.5 \times 10^{-2}\,M$ HCl at a 6 min equilibration time with $1 \times 10^{-2}\,M$ Cyanex 925, which facilitates the binary separation of Os(VIII) from all these metal ions. When the binary separation of Ru(IV) from Rh(III)/Pt(IV)/Ir(III) was carried out at optimum extraction condition of Rh(III)/Pt(IV)/Ir(III), it was found that Ru(IV) remained as unextracted in the aqueous phase. The extracted Rh(III)/Pt(IV)/Ir(III) from the organic phase were back-extracted with $4.0\,M$ HNO_3 (twice), $5.0\,M$ HCl, or $8.0\,M$ HCl, respectively (Table 3). For the binary separation of Ru(IV) and Pd(II), the aqueous mixture containing 100 μg each of the Ru(IV) and Pd(II) was equilibrated for 6 min at aqueous acidity $4.0\,M$ HCl using $1 \times 10^{-1}\,M$ Cyanex 925, when Ru(IV) was quantitatively extracted along with co-extraction of Pd(II). The extracted Ru(IV) from the metal-loaded organic phase was back-extracted with $1.0\,M$ HCl in 1% ascorbic acid when only Ru(IV) was stripped out, while the unstripped Pd(II) was back-extracted using 1:1 $HCl-HClO_4$ mixture. Similarly, separations of other binary mixture were carried out. Separation of some ternary mixtures carried out are given in Table 4.

Application to Recovery from Catalysts Samples

The suitability of the present methods was examined by applying it for extraction and recovery of the metal ions from organometallic (cyclopentadien complexes) catalysts samples $Os(cp)_2$, $Ru(cp)_2$, and $Ir(cp)_2$. These samples were first treated with concentrated HNO_3 and evaporated. The sample of $Ru(cp)_2$ was then treated with chlorine water to oxidize Ru(III) to Ru(IV) and adjusted its molarity to $4.0\,M$ with HCl. Similarly, while preparing a solution of $Os(cp)_2$ its higher oxidation state Os(VIII) was maintained by using chlorine water and final dilution to $0.025\,M$ HCl. In case of $Ir(cp)_2$ the molarity was adjusted at $1.0\,M$ HCl in the presence of $1 \times 10^{-1}\,M$ tin(II) chloride. Thereafter, these metal ions from their solutions were extracted and recovered by using the proposed method as shown in Table 5.

Flow-sheet 1- Separation of Os(VIII) / Ru(IV) / Ir(III)



Scheme 1.

Table 3. Separation of Individual Platinum Metal Ions

			Extraction Conditions			
Serial No.	Metal Ions	(μg)	Aqeous Phase	Organic Phase	Stripping Conditions	% Recovery
1.	Ru(IV)	100	4.0 *M* HCl	$1 \times 10^{-1} M$	1.0 *M* HCl[a]	98.80
	Pd(II)	100			1:1 HCl–$HClO_4$	99.45
2.	Fe(III)	200	4.0 *M* HCl	$1.5 \times 10^{-1} M$	Water	98.10
	Ru(IV)	100			1.0 *M* HCl[a]	99.15
3.	Ir(III)[b]	100	1.0 *M* HCl + $1 \times 10^{-1} M$ $SnCl_2$	$5 \times 10^{-2} M$	8.0 *M* HCl	98.40
	Pd(II)	100			Unextracted	99.45
4.	Pt(IV)	200	1.0 *M* HCl + $1 \times 10^{-1} M$ $SnCl_2$	$1 \times 10^{-2} M$	5.0 *M* HCl	99.20
	Ir(III)[b]	100			Unextracted	98.15
5.	Ir(III)[b]	100	1.0 *M* HCl + $4 \times 10^{-1} M$ $SnCl_2$	$1 \times 10^{-1} M$	8.0 *M* HCl	98.15
	Rh(III)	200			4.0 *M* HNO_3[c]	94.25

[a] In presence of % ascorbic acid.

[b] Heated before extraction at 60°C for 15 min.

[c] Twice stripping.

Table 4. Ternary Separation of Os(VIII), Ru(IV), and Ir(III)

			Extraction Conditions			
Serial No.	Metal Ions	(μg)	Aqeous Phase	Organic phase	Stripping Conditions	% Recovery
1.	Os(VIII)	100	$2.5\times10^{-2}\,M$ HCl	$1\times10^{-2}\,M$	$2.0\,M$ thiourea	99.40
	Pt(IV)	200	$1.0\,M$ HCl $+1\times10^{-2}\,M$ $SnCl_2$	$1\times10^{-2}\,M$	$5.0\,M$ HCl	99.25
	Rh(III)	200			Unextracted	98.65
2.	Os(VIII)	100	$2.5\times10^{-2}\,M$ HCl	$1\times10^{-2}\,M$	$2.0\,M$ Thiourea	99.40
	Ir(III)[a]	150	$1.0\,M$ HCl $+1\times10^{-1}\,M$ $SnCl_2$	$5\times10^{-2}\,M$	$8.0\,M$ HCl	98.85
	Pd(II)	100			Unextracted	98.60
3.	Os(VIII)	100	$2.5\times10^{-2}\,M$ HCl	$1\times10^{-2}\,M$	$2.0\,M$ thiourea	99.40
	Ir(III)[a]	150	$1.0\,M$ HCl $+1\times10^{-1}\,M$ $SnCl_2$	$5\times10^{-2}\,M$	$8.0\,M$ HCl	98.85
	Ru(IV)	100			Unextracted	98.80
4.	Pt(IV)	150	$1.0\,M$ HCl $+1\times10^{-2}\,M$ $SnCl_2$	$1\times10^{-2}\,M$	$5.0\,M$ HCl	99.20
	Ir(III)[a]	200	$1.0\,M$ HCl $+ 4$ x $10^{-1}\,M$ $SnCl_2$	$1\times10^{-1}\,M$	$8.0\,M$ HCl	98.20
	Rh(III)	200			$4.0\,M$ HNO_3[b]	93.10
5.	Os(VIII)	100	$2.5\times10^{-2}\,M$ HCl	$1\times10^{-2}\,M$	$2.0\,M$ thiourea	99.40
	Co(II)	200	$4\times10^{-1}\,M$ Na − Ac	$1\times10^{-1}\,M$	$0.5\,M$ HCl	98.20
	Ni(II)	200			Unextracted	99.10
6.	Ru(IV)	100	$4.0\,M$ HCl	$1\times10^{-1}\,M$	$1.0\,M$ HCl[c]	94.25
	Co(II)	200	$4\times10^{-1}\,M$ Na-Ac	$1\times10^{-1}\,M$	$0.5\,M$ HCl	98.20
	Ni(II)	200			Unextracted	98.65
7.	Ir(III)[a]	150	$1.0\,M$ HCl $+ 1\times10^{-1}$ $SnCl_2$	$5\times10^{-2}\,M$	$8.0\,M$ HCl	98.40
	Co(II)	200	$4\times10^{-1}\,M$ Na-Ac	$1\times10^{-1}\,M$	$0.5\,M$ HCl	98.80
	NI(II)	200			Unextracted	99.10

[a] Heated before extraction at 60°C.
[b] Twice stripping.
[c] In presence of 1% ascorbic acid.

Table 5. Recovery of Platinum Metals from Catalysts Samples

Samples	Amount Present (%)	Amount Found (%)	Percentage Recovery (%R)	σ^a
$Os(cp)_2$	7.40	7.28	98.37	1.62
$Ru(cp)_2$	14.45	14.27	98.75	1.24
$Ir(cp)_2$	7.45	7.34	98.52	1.47

[a] σ Standard deviation for recovery based on average of three determinations.

CONCLUSIONS

The methods developed are found to be simple for extraction of Os(VIII), Ru(IV), and Ir(III) in the presence of large number of foreign ions and separation from their mixtures. The separation of Os(VIII), Ru(IV), and Ir(III) was found to be possible by using single extractant in one step, which is remarkable. Similarly, these metal ions can be smoothly recovered from real samples.

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