Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

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To cite this Article Mhaske, Ashok and Dhadke, Purushottam(2001) 'LIQUID-LIQUID EXTRACTION AND SEPARATION OF RHODIUM(III) FROM OTHER PLATINUM GROUP METALS WITH CYANEX 925', Separation Science and Technology, 36: 14, 3253 — 3265

To link to this Article: DOI: 10.1081/SS-100107771

URL: http://dx.doi.org/10.1081/SS-100107771

SEPARATION SCIENCE AND TECHNOLOGY, 36(14), 3253–3265 (2001)

LIQUID-LIQUID EXTRACTION AND SEPARATION OF RHODIUM(III) FROM OTHER PLATINUM GROUP METALS WITH CYANEX 925

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ABSTRACT

Liquid-liquid extraction of platinum group metals Rh(III), Pt(IV), and Pd(II) was carried out from aqueous chloride media with Cyanex 925 in the absence and presence of tin (II) chloride. In the absence of tin (II) chloride, the order of extraction was found to be Pd $>$ Pt $>$ Rh. However, in the presence of tin (II) chloride, it changed to Pt $>$ Rh and is negligible in the case of palladium. The extraction of rhodium was studied under the influence of variables such as hydrochloric acid, reagent, and tin (II) chloride concentrations; equilibration time; and effect of diverse ions. The separation of Rhodium (III) from Pt(IV) and Pd(II) was enabled because of differences in their extraction and stripping abilities when Cyanex 925 is used.

Key Words: Extraction; Rh(III); Cyanex 925; Stripping; Separation

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INTRODUCTION

Precious metals, comprised of the platinum metals, gold, and silver, are in extensive use for catalysis reactions, electronic devices, and space materials. Rhodium is one of the most expensive rare metals and is indispensable for automobile catalytic converters. However, separation and purification of rhodium is a most difficult process because of its chemical behavior in chloride media (1). Therefore, the development of a more efficient recovery process for rhodium is essential.

Preliminary treatment of rhodium solution with tin (II) chloride is useful for making it labile and for accelerating its reactions (2–5). Benguerel, Demopoulos, and Harris carried out the extraction of rhodium (III) (6), but they have not reported the effect of tin (II) chloride on other platinum metals such as Pt and Pd. The selective reactive extraction and separation of rhodium, platinum, and palladium from spent autocatalysts was studied by Nowattny, Halwachs, and Schugerl (7). However, the process includes extraction, reextraction, and use of ion exchange columns. Alam carried out extraction and separation of rhodium from platinum and palladium using tin (II) chloride as a labilizing agent (8) and found that extraction of rhodium was almost quantitative (98%). However, the stripping was incomplete, reaching only a maximum of 70% with 1.7 mol/L H_2SO_4 and 12 mol/L HNO_3. Extraction and separation of rhodium from iridium with basic extractant 4-(non-5-yl) pyridine was reported by Kostanski (9) The maximum extraction for rhodium was found to be 95% when H_3PO_2 was used as the labilizer, but the method has the disadvantage of longer equilibration time (<15 hrs) than extraction with the pyridine. The extraction and recovery of rhodium from aqueous nitric acid and its recovery from fission products through the use of different organo phospine sulfides was reported by Longdon, Patel, and Thornback (10). However, the process requires a long equilibration period (8 hrs) and high temperature (<60°C). Moijski carried out extraction of rhodium in the presence of tin (II) chloride with a triphenyl phosphine solution in 1–2-dichloroethane, and the extraction was found to take place in less than 3 minutes. However, quantitative extraction was observed only after the aqueous phase was heated in a boiling water bath (11). Many other methods have been reported for extraction and separation of rhodium from other platinum group metals with different extractants (12–19).

Recently, a series of organophosphine compounds were marketed under the trade name Cyanex (272, 301, and 302) as potential acidic extractants for metal ions. Cyanex 921, 923, and 925 are other neutral extractants. Cyanex 925 is a liquid comprised of 19 components with 26.4% tri-(2,4,4-trimethyl pentyl) phosphine oxide and trioctyl phospine oxide (65.9%) as major components. The other component consists of sulfur compounds (20).

The present investigation is concerned with extraction and separation of rhodium (III) from platinum (IV) and palladium (II) with Cyanex 925.

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EXPERIMENTAL

Apparatus and Reagent

A GBC 911A/UV visible spectrophotometer was used for absorption measurements. Cyanex 925, supplied by Cytec Canada Industries Inc (Canada), was used without further purification. The stock solutions of Rh(III), Pt(IV), and Pd(II) were prepared by dissolving analytical reagent grade $RhCl_3$, $K_2[PtCl_6]$, and $PdCl_2$ in a minimum quantity of dilute hydrochloric acid and standardizing them by known methods (21–22). The required concentrations of the solutions were prepared by further dilution with double-distilled water. The tin (II) chloride solution prepared in 2.0 mol/L HCl was used for the experiments. All other chemicals used were of analytical reagent grade.

General Extraction Procedure

An aliquot of solution containing 200 μg of Rh(III) was taken in the separating funnel and to it varying concentrations of hydrochloric acid or tin (II) chloride were added for extraction studies. This aqueous phase (10 cm^3) was equilibrated for 1 minute with an equal volume of 1×10^{-1} mol/L Cyanex 925 in toluene. The 2 phases were allowed to separate. The metal concentration in the aqueous phase was determined spectrophotometrically by the tin (II) chloride method at 475 nm (22). Rhodium in the metal-loaded organic phase was determined by mass balance of the metal before and after extraction.

RESULTS AND DISCUSSION

Influence of Hydrochloric Acid Concentration

Rh(III) was extracted in the acidity range of 1.0–10.0 mol/L HCl with 1×10^{-1} mol/L Cyanex 925 in toluene. However, not more than 40% of Rh(III) was extracted in this range (Fig. 1). The extraction behavior of Pt(IV) and Pd(II) was also studied under the same experimental conditions.

Influence of Tin (II) Chloride

The extraction of Pt(IV) was studied under the varying concentration range of $1 \times 10^{-3}-1.0$ mol/L tin (II) chloride at 1.0 mol/L fixed hydrochloric acid concentration and 1 minute equilibration time. In the presence of tin (II) chloride, the extraction of Rh(III) and Pt(IV) increased with an increase in tin (II) chloride con-

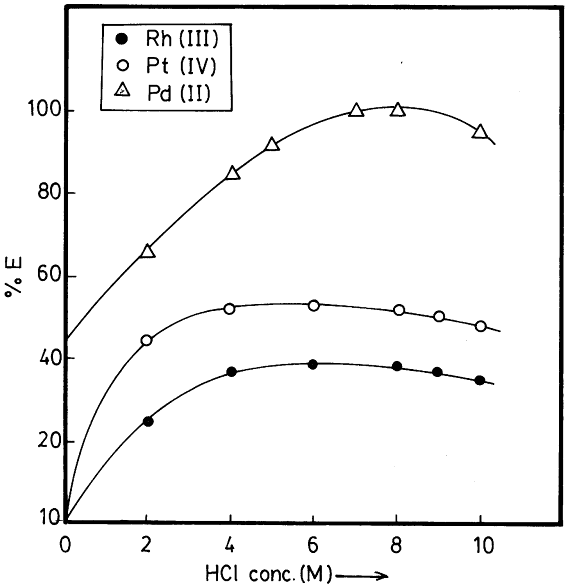

Figure 1. Effect of HCl concentration on percentage of extraction (E). Equilibration time = 5 minutes; organic phase = 1×10^{-1} mol/L.

centration, but it was negligible in the case of Pd(II) (Fig. 2). In the presence of tin (II) chloride, the Rh(IV) and Pt(IV) metal ions chloride complexes are activated by their reduction to Rh(I)- and Pt(II)-tin complexes and Sn(II) is simultaneously oxidized to Sn(IV). These complexes are more labile than other kinetically inert complexes due to the π acid nature of $SnCl_3$ (23–27). The decrease in extraction of Pd(II) by addition of tin (II) chloride, contrary to that of Rh(III) and Pt(IV), may be due to the coextraction of tin; that is, tin is also extracted with Cyanex 925 (Fig. 3).

The optimum concentration of tin (II) chloride required for quantitative extraction of Rh(III) and Pt(IV) was found to be 4×10^{-1} mol/L and 1×10^{-2} mol/L, respectively. Furthermore, in the presence of tin (II) chloride the order of extraction was found to be Pt(IV) > Rh(III).

Influence of Reagent Concentration

Rh(III) was extracted with varying concentrations of Cyanex 925 (1×10^{-5}–1×10^{-1} mol/L) in toluene, and quantitative extraction occurred above 1×10^{-1} mol/L Cyanex 925. From the slope ratio method (Fig. 4), the metal-Cyanex 925 ratio was found to be 1:3.

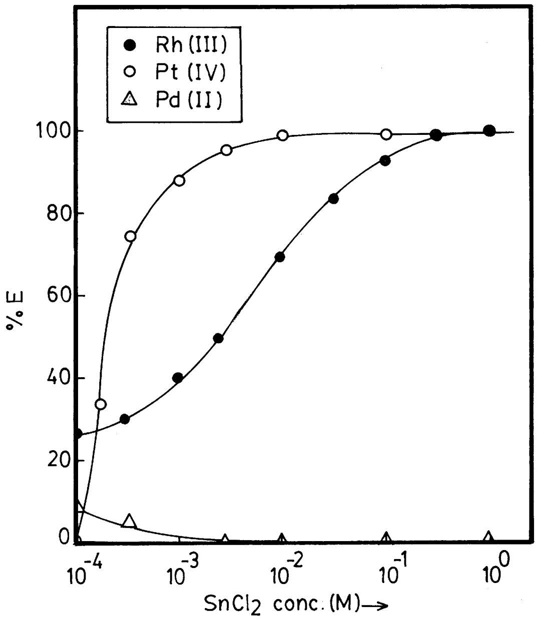

Figure 2. Effect of $SnCl_2$ concentration on percentage of extraction (E). Equilibration time = 1 minute; organic phase = 1×10^{-1} mol/L.

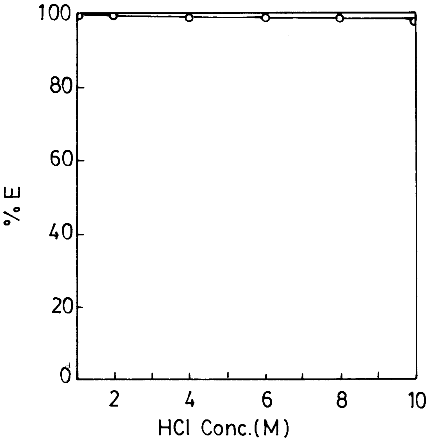

Figure 3. Extraction of tin (II) with Cyanex 925 in toluene. Equilibration time = 5 minutes; organic phase = 1×10^{-1} mol/L; tin (II) = 100 μg.

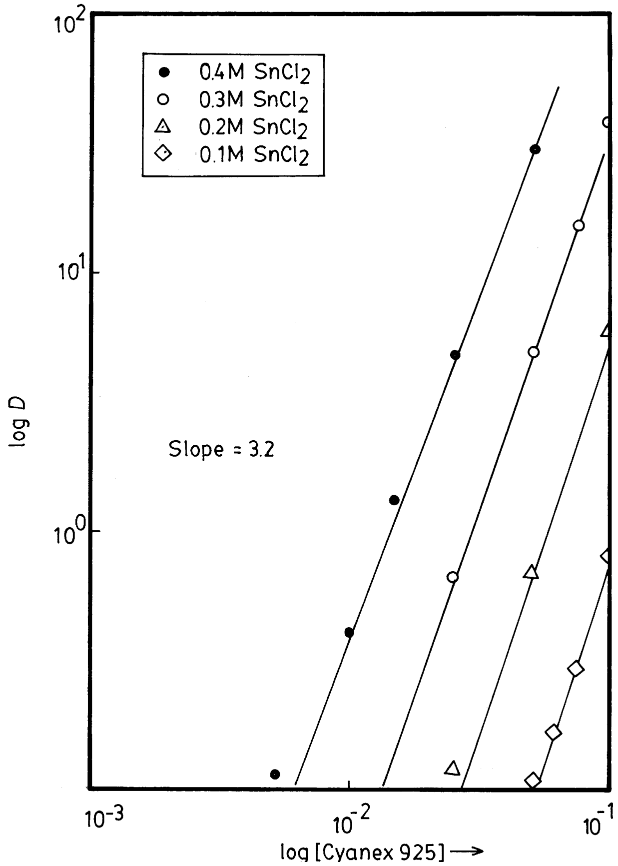

Figure 4. Effect of Cyanex 925 concentration on distribution ratio (*D*). Slope = 3.2.

Influence of Various Diluents

The extraction of Rh(III) was carried out with 1×10^{-1} mol/L Cyanex 925 in different solvents. The extraction was quantitative with toluene (99.6%), xylene (99.6%), *n*-hexane (99.6%), and cyclohexane (99.0%), while benzene (96.0%), carbon tetrachloride (92.6%), and chloroform (91.4 %) used as diluents did not favor quantitative extraction.

Influence of Equilibration Time

The aqueous phase containing Rh(III) was equilibrated from 1–30 minutes with 1×10^{-1} mol/L Cyanex 925 in toluene. The quantitative extraction started after 1 minute and remained quantitative for up to 30 minutes.

Table 1. Effect of Diverse Ions on Percentage of Rh(III) Extraction

Metal Ions (μg)	Tolerance Limits
Na^+, K^+, Cs^+, Rb^+, Mg^{2+}, Ca^{2+}, Ba^{2+}, Sr^{2+}	1:20
V^{5+}, Mn^{2+}, Co^{2+}, Cu^{2+}, Cd^{2+}	1:15
Cr^{3+}, Fe^{3+}, Zn^{2+}, Al^{3+}, Bi^{3+}	1:12
Pd^{2+}, Os^{8+}, Ru^{3+}	1:6
Pt^{2+}	1:4
Cl^-, Br^-, I^-, SO_3^{2-}, Citrate, Oxalate	1:20
thiocyanate, thiourea	1:0

Rh(III) = 200 μg.

Influence of Diverse Ion on Percentage Extraction of Rh(III)

Rh(III) was extracted in the presence of a large number of elements. The tolerance limit was determined to be a foreign ion causing interference of ±2% of Rh(III) extraction yields. Alkali and alkaline metals were tolerated up to the ratio of 1:20. Elements of the first transition series and Al^{3+}, Hg^{2+}, Pb^{2+}, Bi^{3+} were tolerated at the ratio of 1:15 (Table 1). Anions like chloride, bromide, nitrate, sulfite, citrate, and oxalate were tolerated up to a 20-fold excess of added Rh(III). Thiocyanate and thiourea were found to strongly interfere with extraction of Rh(III).

Influence of Various Stripping Agents

The metal-loaded organic phase containing Rh(III) was stripped with various stripping agents, such as HCl, H_2SO_4, HNO_3, H_2O_2, and an HNO_3 and H_2O_2 mixture (Table 2). The stripping was observed only with HNO_3, H_2O_2, and the

Table 2. Effect of Stripping Agent on Percentage Recovery of Rh(III)

Stripping Agents	% Recovery			
Strength (mol/L)	1 mol/L	2 mol/L	4 mol/L	6.0 mol/L
HCl	0.00	0.00	0.00	0.00
HNO_3	32.40	54.10	68.15	68.15
2nd strip with HNO_3	51.48	65.20	95.40	95.40
H_2SO_4	0.00	0.00	—	—
H_2O_2 (% vol/vol)	28.15 (0.1%)	41.55 (0.2%)	52.61 (0.4%)	61.10 (1.0 %)
H_2O_2 + HNO_3 (0.4% H_2O_2 + X mol/L HNO_3)	45.46, 1 mol/L HNO_3	58.35, 2 mol/L HNO_3	72.20, 4 mol/L HNO_3	73.11, 6 mol/L HNO_3

—Data unavailable.

HNO_3 and H_2O_2 mixture. However, H_2O_2 and the HNO_3 and H_2O_2 mixture cannot be used as stripping agents because reagents are disintegrated in their presence.

Separation of Rh(III) from Pt(IV) and Pd(II)

Separation of Rh(III) from Pt(IV) and Pd(II) was carried out through the use of different extraction and stripping conditions of these metal ions toward Cyanex 925.

Separation of Rh(III) from Pt(IV) and Pd(II) can be completed when Rh(III) is restrained in the aqueous phase. For this procedure, the aqueous solution containing a mixture of 200 μg Rh(III), 50 μg Pt(IV), and 25 μg Pd(II) at 7.0–8.0 mol/L HCl in the absence of tin (II) chloride was equilibrated for 5 minutes with 7.5×10^{-2} mol/L Cyanex 925, and Pd(II) was quantitatively extracted while Rh(III) and Pt(IV) remained in the aqueous phase. The remaining mixture was then again equilibrated for 1 minute in the presence of 1.0 mol/L HCl and 1×10^{-2} mol/L $SnCl_2$ with 1×10^{-2} mol/L Cyanex 925, and Pt(IV) was completely extracted and Rh(III) remained in the aqueous phase. The metal-loaded organic phase containing Pt(IV) was then back-extracted with 5.0 mol/L HCl. However, this is the case only for Rh(III) extraction and separation from Pd(II) and Pt(IV). In cases in which Rh(III) extraction and separation from other platinum and heavy metals is necessary or in which Rh(III) extraction and separation is applied to fission products or spent catalyst recovery, a stripping process is needed to extract and recover Rh(III).

Separation of Rh(III) from Pt(IV)

Separation of Rh(III) from Pt(IV) was carried out through the use of different reagent concentrations as required for extraction. The aqueous phase containing 200 μg Rh(III) and 50 μg Pt(IV) in a mixture of 1.0 mol/L HCl and 1×10^{-2} mol/L $SnCl_2$ was equilibrated first with 1×10^{-2} mol/L Cyanex 925. Pt(IV) was extracted at this concentration, while Rh(III) was not extracted. The metal-loaded organic phase was then back-extracted with 5.0 mol/L HCl.

Separation of Rh(III) from Pd(II)

For separation of Rh(III) from Pd(II), an aqueous solution containing 200 μg Rh(III) and 25 μg Pd(II) in a mixture of 1.0 mol/L HCl and 0.4 mol/L $SnCl_2$ was equilibrated for 1 minute with 1×10^{-1} mol/L Cyanex 925. Under this condition, Rh(III) was extracted in the organic phase, while Pd(II) remained unextracted. The metal-loaded organic phase containing Rh(III) was then back-extracted twice with 4.0 mol/L HNO_3.

Separation of Rh(III) from Pt(IV) and Pd(II)

The separation of rhodium (III) from platinum (IV) and palladium (II) was carried out by taking advantage of differences in their optimum extraction and stripping conditions. The aqueous solution containing a mixture of 200 μg Rh(III), 50 μg Pt(IV), and 25 μg Pd(II) in 1.0 mol/L HCl and 0.4 mol/L $SnCl_2$ was equilibrated for 1 minute with 1×10^{-1} mol/L Cyanex 925. While Rh(III) and Pt(IV) were extracted in the organic phase, Pd(II) remained as unextracted. The mixture of Rh(III) and Pt(IV) was resolved by taking advantage of differences in their stripping properties. The metal-loaded organic phase containing Rh(III) and Pt(IV) was first stripped with 5.0 mol/L HCl and Pt(IV) was completely stripped. The unstripped Rh(III) was later stripped twice with 4.0 mol/L HNO_3 (Fig. 5).

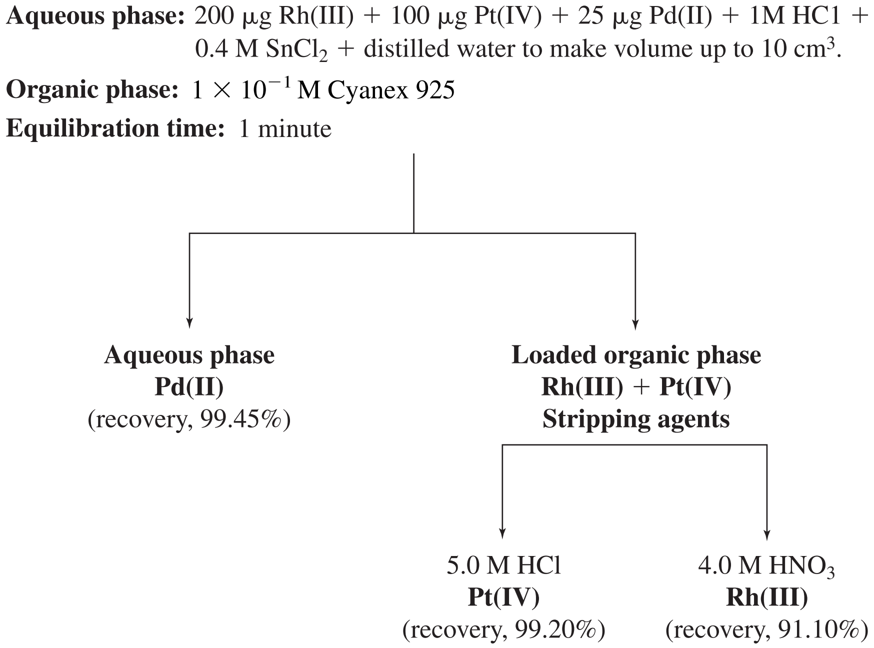

Figure 5. Flowchart rendering of process for Rh(III) separation from Pt(IV) and Pd(II).

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Multicomponent Separation of Rh(III) from Some Commonly Associated Metals

Separation of Rh(III) from some commonly associated metal ions, such as Ru(III), Os(VIII), Au(III), and Co(II), was also studied (Table 3). In the binary metal ion separation, the aqueous solution of Rh(III) that contained 1.0 mol/L HCl and 0.4 mol/L $SnCl_2$ was equilibrated for 1 minute with 1×10^{-1} mol/L Cyanex 925 in the presence of these associated metal ions. All these metal ions remained unextracted under this experimental condition. Perhaps in the presence of tin (II) chloride, Au(III) and Os(VIII) are reduced to lower oxidation states and are present in the unextracted aqueous phase as Au(I) and Os(VI). The metal-loaded organic phase containing Rh(III) was back-extracted twice with 4.0 mol/L HNO_3.

The ternary mixture containing Rh(III), Au(III), and Os(VIII) was resolved by first equilibrating at pH = 1.5 for 6 minutes. Os(VIII) was extracted under this condition, while Rh(III) and Ru(III) remained unextracted. Os(VIII) from the metal-loaded organic phase was back-extracted with 2.0 mol/L thiourea. The aqueous phase containing unextracted Rh(III) and Au(III) was equilibrated for 1 minute with 1×10^{-1} mol/L Cyanex 925 in a mixture of 1.0 mol HCl and 0.4

Table 3. Multicomponent Separation of Rh(III) from Some Other Associated Metal Ions

Series No.	Metal Ions	Concentration (μg)	Extraction Conditions	Stripping Conditions	% Recovery
1.	Rh(III)	100	1.0 mol/L HCl +	4.0 mol/L HNO_3	94.60
	Au(III)	150	0.4 mol/L $SnCl_2$	unextracted	98.00
2.	Rh(III)	200	1.0 mol/L HCl +	4.0 mol/L HNO_3	93.60
	Ru(III)	100	0.4 mol/L $SnCl_2$	unextracted	98.20
3.	Rh(III)	200	1.0 mol/L HCl +	4.0 mol/L HNO_3	94.25
	Os(VIII)	150	0.4 mol/L $SnCl_2$	unextracted	99.40
4.	Rh(III)	200	1.0 mol/L HCl +	4.0 mol/L HNO_3	94.25
	Co(II)	100	0.4 mol/L $SnCl_2$	unextracted	98.80
5.	Os(VIII)	150	pH 1.5	2.0 mol/L thiourea	98.15
	Au(III)	150	4.0 mol/L HCl	water	97.68
	Rh(III)	200	1.0 mol/L HCl + 0.4 mol/L $SnCl_2$	4.0 mol/L HNO_3	91.20
6.	Co(II)	100	0.4 mol/L NaAc.	0.5 M HCl	98.80
	Rh(III)	200	1.0 M HCl + 0.4 M $SnCl_2$	4.0 mol/L HNO_3	94.25
	Ru(III)	100	n.a.	unextracted	98.00

Organic phase: 1×10^{-1} mol/L Cyanex 925. Equilibration time was 1 minute.
n.a. Not applicable.

mol/L $SnCl_2$. Au(III) remained unextracted while extracted Rh(III) from the organic phase was stripped twice with 4.0 mol/L HNO_3 (Table 3). Ternary separation of Rh(III), Ru(III), and Co(II) was carried out first through the equilibration of the aqueous phase containing 0.4 mol/L sodium acetate with 1×10^{-1} mol/L Cyanex 925. Co(II), stripped with 0.5 mol/L HCl, was the only metal extracted that was stripped with 0.5 mol/L HCl. The unextracted Rh(III) and Ru(III) solution was again equilibrated with 1×10^{-1} mol/L Cyanex 925 for 1 minute in the presence of 1.0 mol/L HCl and 0.4 mol/L $SnCl_2$, and Rh(III) was extracted while Ru(III) remained unextracted. The extracted Rh(III) from the organic phase was stripped twice with 4.0 mol/L HNO_3.

CONCLUSION

The extraction of Rh(III), Pt(IV), and Pd(II) from the aqueous chloride media was carried out with Cyanex 925 in toluene in the absence and presence of tin (II) chloride. The following conclusions are drawn from this study:

1. In hydrochloric acid, the order of extraction was found to be Pd(II) $>$ Pt(IV) $>$ Rh(III).
2. In the presence of tin (II) chloride the order of extraction was Pt(IV) $>$ Rh(III) and extraction was negligible in the case of Pd(II). Furthermore, the quantitative extraction of Rh(IV) takes place at an optimum concentration of 4×10^{-1} mol/L tin (II) chloride, which is higher than that for Pt(IV) (1×10^{-2} mol/L).
3. Separation of Rh(III) from Pt(IV) and Pd(II) was carried out based on differences in their extraction and stripping properties toward Cyanex 925.
4. The stripping of Rh(III) was carried out in 2 steps with 4.0 mol/L HNO_3.

The proposed method was found to be simple for extraction-separation of Rh(III) from Pt(IV) and Pd(II) in the presence of associated metal ions and a large number of diverse ions. Also, the time period required for extraction and separation of rhodium from other platinum metals is relatively small. Although the recovery for rhodium from the metal-loaded organic phase is incomplete, its recovery with Cyanex 925 offers more advantages than does its recovery with Kelex 100 (8).

ACKNOWLEDGMENT

The authors are thankful to Cytec Canada Inc, Canada, for supplying the Cyanex 925 gift sample.

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Received May 2000
Revised August 2000

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