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TECHNICAL NOTE

COMPARATIVE STUDY FOR EXTRACTION OF Hg(II) WITH CYANEX-923 FROM CHLORIDE AND BROMIDE MEDIA AND ITS SEPARATION FROM Cd(II) AND Zn(II)

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ABSTRACT

Extraction of mercury (II) was carried out from chloride and bromide media with Cyanex-923 in toluene, to find out the effect of media on extraction. The quantitative extraction was observed in the range of 0.01–0.10 mol dm^{-3} HCl with 0.10 mol dm^{-3} Cyanex-923 and in the range of 0.30–0.70 mol dm^{-3} HBr with 0.08 mol dm^{-3} Cyanex-923. The recovery of Hg(II) extracted into organic phases from HCl and HBr media were observed with the mixture of 5.0 mol dm^{-3} $HNO_3 + 4.0$ mol dm^{-3} HCl and 2.0 mol dm^{-3} HNO_3, respectively. The stoichiometric ratio of Hg(II): Cyanex-923 was 1:2 for both the media.

On the basis of extraction and stripping study, the separation of Hg(II) from Cd(II) and Zn(II) was carried out. These methods were successfully applied for the analysis of Hg(II) in real samples.

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Key Words: Extraction; Mercury(II); Cyanex-923; Hydrochloric acid; Hydrobromic acid; Stripping; Separation

INTRODUCTION

Mercury is known from ancient times. It is used in thermometers, high pressure gauges, electrical bulb, explosives, and amalgamation processes. It is toxic and hazardous in nature having a tolerance limit of 0.5 ppm (1). The environmental contamination of mercury is known to have serious problems. The removal of mercury is a challenging problem, so in the present study, solvent extraction method is employed for removal of mercury form wastewater.

Mercury in +2 oxidation state is more hazardous than other states. The extraction of Hg(II) was tried with different types of extractants like, trioctyl phosphine oxide (TOPO) (2), tributyl phosphate (TBP) (3), trioctyl amine (TOA) (4), and tricaprylammonium chloride (Aliquate-336) (5,6), however, these reagents were found to have poor selectivity for Hg(II). Few selective reagents were used by Mikhailov et al. (7) and Sato et al. (8) having S as a donor atom, similarly, Baba et al. used triisobutyl phosphine sulfide (Cyanex-471X), and 1-2,bis(hexylthio)ethane (9) having two S atoms for extraction of Hg(II), but all these suffer from stripping problem (10).

Singh et al. reported study of the chromosorb 102 (11) impregnated with triisobutyl phosphine sulfide for chromatographic extraction of Hg(II) from HNO_3 media. Some sulfur containing novel reagents like 2-ethyl-2-(isobutylthiomethyl)butanethiol, 3,3-diethylthietane, and 3,3-di-butylthietane (12) were studied by Inoue et al. for extraction of Hg(II) from chloride media. Baba et al. studied the 2-butyl-thiododecanoic acid (BTDA) (13), while Lobana et al. tried tertiary phosphine chalcogenides (14) for Hg(II) extraction from chloride media. Later, separation of Zn(II) from Hg(II) and Cd(II) was studied by Raman and Shinde with triphenyl phosphine oxide (TPPO) (15). Handley et al. showed that the distribution coefficient of Hg(II)) was highest in acetic acid with trialkyl thiophosphate (16) than other media. The synergic effect of 1-phenyl-3-methyl-4-trifluoroacetyl-2-pyrazolin-5-one (HPMTFP) and TPPO (17) on extraction of Hg(II) was studied by Cheema et al.

Recently, Cytec Industries Inc., Canada, have manufactured a series of organo phosphine compounds that are marketed under the trade name of "Cyanex", like trialkylphosphine oxide (Cyanex-923) with "O" as donor atom having a strong affinity to class (hard) metals. Besides, O atom has also the ability to extract class b (soft) metal ions. Au(I) and Au(III) were extracted with Cyanex-923 from cyanide and chloride media by Alguacil et al. (18), and Martinez et al. (19), respectively. Thus the extractant Cyanex-923 is useful for extraction of class b (soft) metal ions by forming neutral species.

In recent years, scientists are more interested in studying the effect of media on metal extraction, which is seen from the use of different media like bromide (20) that was tried by Rakhman'ko et al., iodide (21) by Pillia et al., and thiocyanide (22) by Oleschuk et al. rather than the routine chloride media. The extraction of Pd(II) was found more favorable in HBr media than HCl media with Di-*n*-octyl sulfide (23) (DOS) as an extractant in cyclohexane. Holbrook et al. reported that the extraction of Au(III) (24) from HBr media was more effective than HCl media, while Morris et al. and Moriya et al. have similar observation for Zn(II) (25,26). In a novel separation scheme, Dreher et al. converted chloro complexes to the bromo form, where the bromo complexes undergo aquation to a lesser extent, and hence are more easily extractable than chloro$^-$ complexes. Thus the use of bromide (27) media has opened a new path for the extraction and separation of metal ions. Shetker et al. employed the bromide media (28) for quantitative extraction of Au(III) with triphenyl phosphine sulfide. In our previous study (29), it was observed that the distribution coefficient (D) of Ir(III) varies considerably by employing HBr media instead of HCl media.

The present work deals with the extraction and separation of Hg(II) from Cd(II) and Zn(II) from HCl and HBr media with Cyanex-923 as an extractant in toluene.

EXPERIMENTAL

Apparatus and Reagents

G.B.C.-911A UV/Visible spectrophotometer (GBC Scientific Equipments Pvt Ltd., Australia, Model 6BC911A) was used for absorbance measurements. The reagent Cyanex-923 kindly supplied by Cytec (30,31) Industries Inc. Canada was used without further purification. According to the product manual, Cyanex-923, (Cytec Industries, Inc. Canada) is a mixture of four trialkylphosphine oxides with the general formula $R_3P(O)$ (14%), $R_2R'P(O)$ (42%), $RR'_2P(O)$ (31%), and $R'_3P(O)$ (8%), where R = *n*-hexyl and R′ = *n*-octyl. Its average molecule mass is 348 and density at 20°C is 880 kg m^{-3}. The stock solution of Hg(II), Cd(II) and Zn(II) was prepared by dissolving $HgCl_2$, $CdCl_2$ and $ZnCl_2$ in HCl as well as mercury oxide, zinc oxide and $CdBr_2$ in HBr and standardized (32) by a known method. The required concentration of their solution was prepared by further dilution with double distilled water.

PROCEDURE

Extraction/stripping experiments were performed by shaking the appropriate organic and aqueous solutions at an O/A phase ratio of 1 for 10 min. All extraction studies were carried out at 25°C. The initial concentration of Hg(II) was maintained at $1.0\,mg\,L^{-1}$ throughout the experiments (unless otherwise stated) and the metal content in the equilibrated aqueous phase was determined by Zincon (33) method at 600 nm spectrophotometrically. The metal ions Cd(II) and Zn(II) were determined spectrophotometrically (34,35) from aqueous phase, where as the metal content in the organic phase was calculated by mass balance. The distribution coefficient D was calculated as the ratio of the equilibrium concentration of Hg(II) in organic phase to aqueous phase.

All experiments were repeated three times and the metal ion concentration in the organic phase was calculated by mass balance as well as checked by complete stripping of the loaded organic phase. An average accuracy of 98%, between calculated and analytical result was observed.

RESULTS AND DISCUSSION

Hydrobromic Acid Concentration Dependence

The bromide complexes are generally weaker or less stable (25) than chloride complexes in aqueous solution, so they are easily extracted. This seems to be due to their large molecular size and high aquophobic tendency (26). Therefore, the effect of media on the extraction of Hg(II) with Cyanex-923 was undertaken in chloride and bromide media.

Solvent extraction of Hg(II) was carried out for 10 min by varying the concentration of HCl and HBr in the aqueous phase from 0.01 to $8.0\,mol\,dm^{-3}$ with $0.10\,mol\,dm^{-3}$ Cyanex-923 in toluene (for HCl media) and with $0.08\,mol\,dm^{-3}$ Cyanex-923 in toluene (for HBr media) (Fig. 1). The quantitative extraction was observed in the range of $0.01–0.10\,mol\,dm^{-3}$ HCl and $0.30–0.50\,mol\,dm^{-3}$ HBr. However, with the increase in the acid concentration, the extraction was found to decrease. This seems to be due to the competition between Hg(II) and HCl or HBr to associate with the phosphine oxide. This behavior is similar to the earlier reported extraction of Au(III) from HCl with Cyanex-923 by Martinez et al. (19).

For further study, extraction of Hg(II) was carried out from $0.01\,mol\,dm^{-3}$ HCl and $0.3\,mol\,dm^{-3}$ HBr.

Reagent Concentration Dependence

The $\log D$ vs. $\log\,[R]$ plot for the extraction of Hg(II) with Cyanex-923 in toluene was studied. The results obtained showed that there is an increase in the extraction with the increase in the reagent concentration. The nature of the

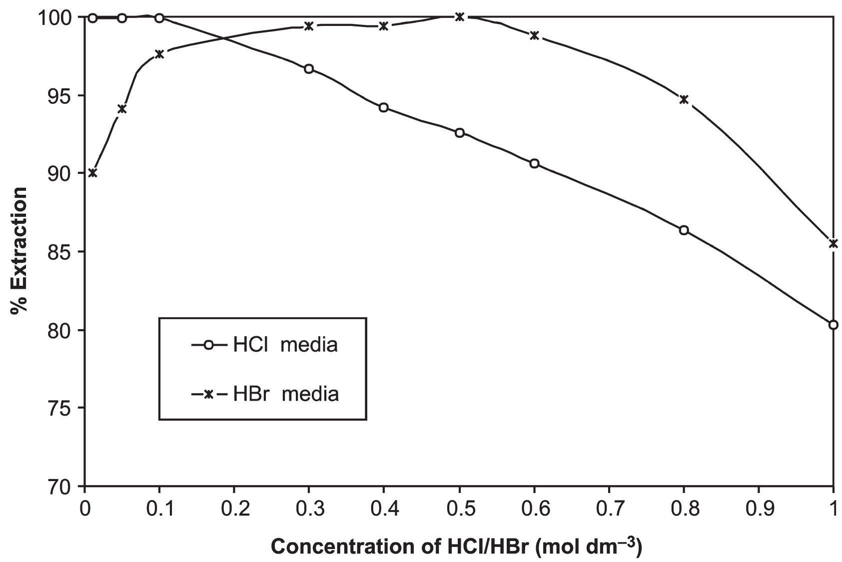


Figure 1. Extraction of Hg(II) ($1.0\,mg\,L^{-1}$) from HCl and HBr media with 0.1 and $0.08\,mol\,dm^{-3}$ Cyanex-923 in toluene, respectively.

extracted species was ascertained from the plot of log D vs. log [Cyanex-923] for chloride and bromide media (Fig. 2).

The Cyanex-923 is a neutral extractant and extracts the neutral species of metal ion by solvation mechanism. The slopes of graphs observed 2.11 for chloride media and 2.06 for bromide media, indicated that in complex species formed Hg(II) is solvated by two molecules of Cyanex-923 in toluene in both the media and the stoichiometric ratio 1:2.

The Cl^- and Br^- species of Hg(II) (36) are responsible for the metal extraction. The extraction is usually better in the order $Br^- > Cl^-$. However, extraction becomes poorer at high acid concentrations, because of the formation of increased anionic complexes such as HgX_3^- and HgX_4^{-2}, which are having lower extractability than the neutral complexes HgX_2. Therefore, the extraction may be expressed as follows,

$$Hg^{+2} + 2Cl^-(aq) + \overline{2R(org)} \rightleftarrows \overline{HgCl_2 \cdot 2R(org)} \quad (1)$$

$$Hg^{+2} + 2Br^-(aq) + \overline{2R(org)} \rightleftarrows \overline{HgBr_2 \cdot 2R(org)} \quad (2)$$

where R = Cyanex-923 in toluene which is similar to the species reported earlier for TOPO (2).

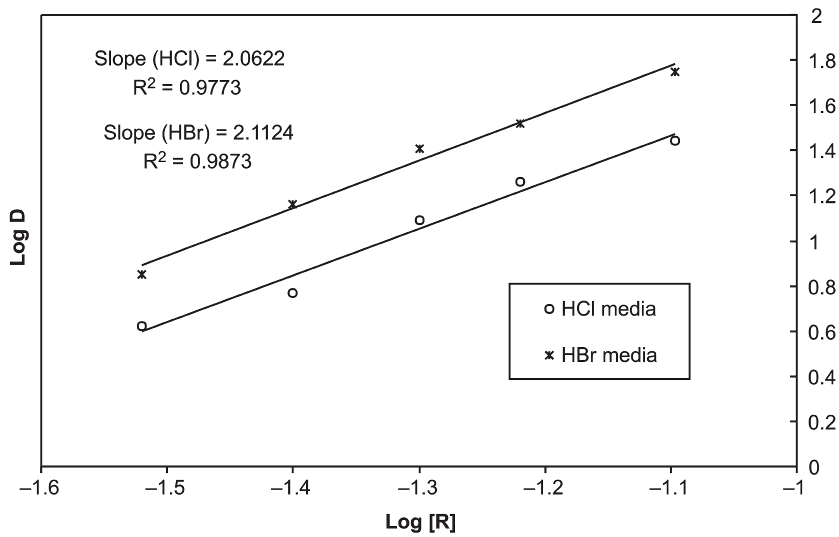


Figure 2. Stoichiometric ratio of Hg(II) (1.0 mg L^{-1}) with 0.1 and 0.08 mol dm^{-3} of Cyanex-923 in toluene from 0.01 mol dm^{-3} HCl and 0.3 mol dm^{-3} HBr media, respectively.

Loading Capacity of Extractants

In order to find out the loading capacity of a reagent, the extraction was carried in the range of 0.05–50 mg L^{-1} of Hg(II) in aqueous phase. The extraction was quantitative (99.6%) up to the metal ion concentration of 25 mg L^{-1} Hg(II) from HCl media and 32.5 mg $^{L-1}$ Hg(II) from HBr media with Cyanex-923 in toluene. Thus the loading capacity of reagent Cyanex-923 is 25 and 32.5 mg L^{-1} from HCl and HBr media, respectively.

Influence of Diluents

The extraction was found to be quantitative with 0.1 mol dm^{-3} Cyanex-923 in toluene and xylene, while *n*-hexane, cyclohexane, carbon tetrachloride, and chloroform do not favor quantitative extraction from HCl. Similarly, quantitative extraction was observed with 0.08 mol dm^{-3} Cyanex-923 in toluene and xylene, while *n*-hexane, cyclohexane, carbon tetrachloride, and chloroform do not favor quantitative extraction from HBr media (Table 1).

Table 1. Effect of Diluents on Percentage Extraction of Hg(II) with 0.1 and 0.08 mol dm^{-3} Cyanex-923 from 0.01 mol dm^{-3} HCl and 0.3 mol dm^{-3} HBr Media, Respectively. Hg(II) = 1.0 mg L^{-1}

	Percentage Extraction of Hg(II)	
Diluents	HCl	HBr
Toluene	99.6	99.9
Xylene	99.4	100.0
n-Hexane	87.55	97.3
Cyclohexane	93.2	97.6
Carbon tetrachloride	8.5	96.2
Chloroform	80.4	97.2

Influence of Diverse Ions on Percentage Extraction of Hg(II)

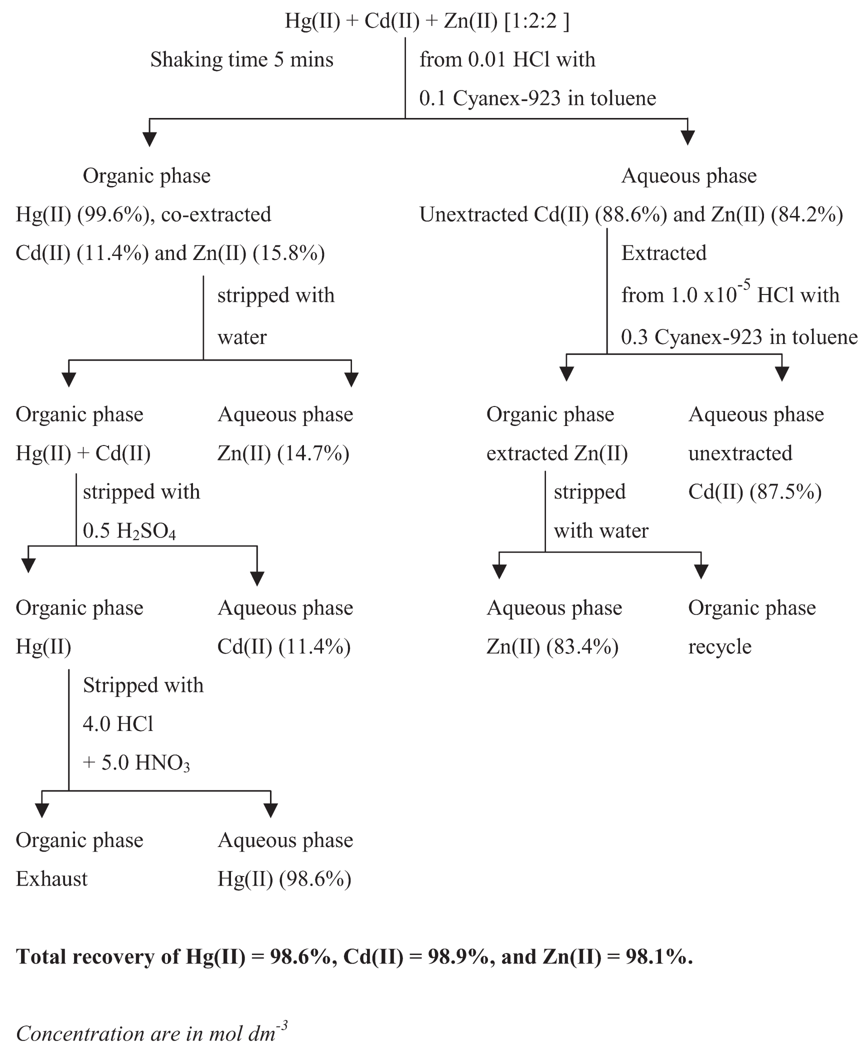
Hg(II) was extracted in the presence of a large number of foreign ions with Cyanex-923 in toluene from HCl and HBr media. The tolerance limit was set such that the foreign ions caused an interference of ±2% on the extraction of Hg(II) with various metal ions (Table 2). The transition metal ions, alkali, and alkaline earth metal ions are tolerated for > 1:4 to < 1:10 ratio, while noble metal ions interfere during extraction.

Table 2. Effect of Diverse Ions on Percentage Extraction of Hg(II) with 0.10 and 0.08 mol dm^{-3} Cyanex-923 in Toluene from 0.01 mol dm^{-3} chloride and 0.3 mol dm^{-3} Bromide Media, Respectively. Hg(II) = 1.0 mg L^{-1}

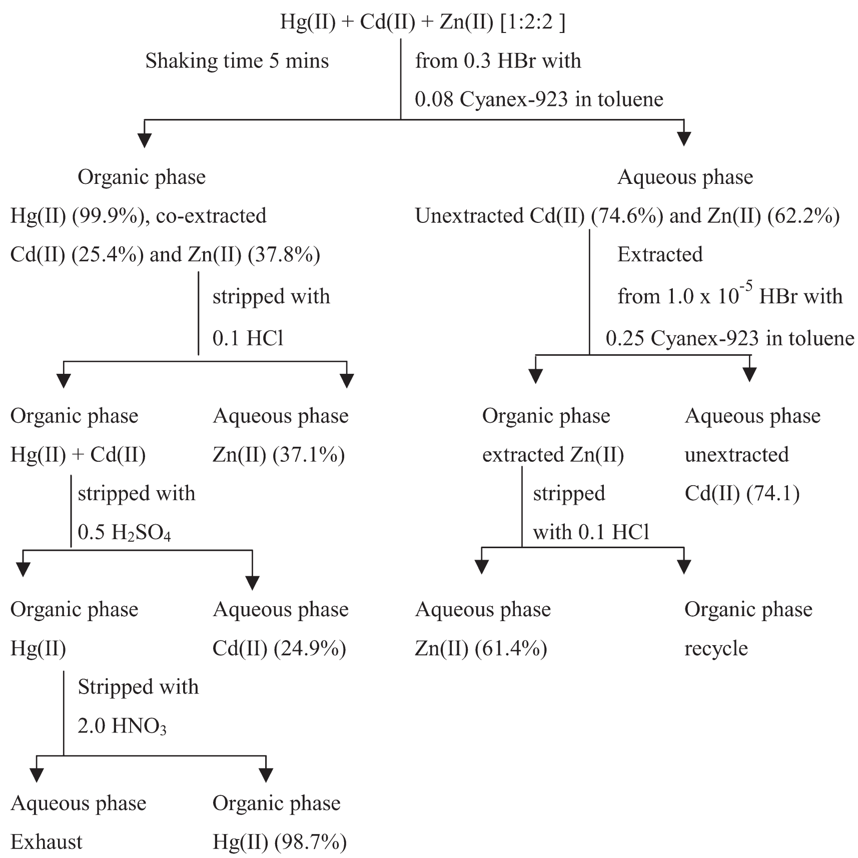
	Ratio of Hg(II) with Metal Ions in	
Metal Ions	Chloride Media	Bromide Media
Na^{+}, K^{+}, Cs^{+}, Rb^{+}, Mg^{+2}, Ca^{+2}, Ba^{+2}, Sr^{+2}	1:10	1:9
V^{+5}, Mn^{+2}, Co^{+2}, Cu^{+2}	1:6	1:4
Cr^{+3}, Fe^{+3}, Zn^{+2}, Al^{+3}, Bi^{+3}	1:4	1:3
Ru^{+3}	1:0	1:1
Pt^{+4}, Pd^{+2}, Au^{+3}, Os^{+8}, Rh^{+3}	1:0	1:0
Cl^{-}, I^{-}, SO_3^{-2}, citrate, oxalate	1:20	1:20

Table 3. Effect of Stripping Agents on Percentage Extraction of Hg(II) with 0.10 and 0.08 mol dm^{-3} Cyanex-923 in Toluene from 0.01 mol dm^{-3} Chloride and 0.3 mol dm^{-3} Bromide Media, Respectively. Hg(II) = 1.0 mg L^{-1}

	Percentage Recovery from									
	Chloride Media (mol dm^{-3})					Bromide Media (mol dm^{-3})				
Stripping agents	1.0	2.0	4.0	5.0	6.0	1.0	2.0	4.0	5.0	6.0
HBr	6.3	11.2	21.6	—	—	20.7	32.5	46.7	—	—
HCl	13.9	21.5	37.8	41.1	45.2	9.8	12.7	13.5	16.8	16.8
HNO_3	13.9	32.8	53.8	78.9	80.2	89.7	99.6	99.6	—	—
H_2SO_4	19.62	27.4	42.6	44.6	44.6	54.6	73.8	81.3	83.9	85.2
$HClO_4$	29.1	38.6	40.5	50.6	58.2	87.6	93.4	96.7	97.2	97.9
5.0 HNO_3 + 4.0 HCl	99.5	—	—	—	—	—	—	—	—	—



Flow Chart 1. Separation of Hg(II) from Cd(II) and Zn(II) with Cyanex-923 in toluene from HCl media. Total recovery of Hg(II) = 98.6%, Cd(II) = 98.9%, and Zn(II) = 98.1%. Concentrations are in mol dm^{-3}.



Total recovery of Hg(II) = 98.7%, Cd(II) = 99.0%, and Zn(II) = 98.5%.

Concentration are in mol dm^{-3}

Flow Chart 2. Separation of Hg(II) from Cd(II) and Zn(II) with Cyanex-923 in toluene from HBr media. Total recovery of Hg(II) = 98.7%, Cd(II) = 99.0%, and Zn(II) = 98.5%. Concentrations are in mol dm^{-3}.

Effect of Stripping Agents

The Hg(II) loaded in the organic phase was stripped with varying concentrations of different acids, HCl, HNO_3, H_2SO_4, and $HClO_4$ as well as bases, NaOH, NH_4OH, and $Na_2S_2O_3$ (Table 3). It was found that NaOH, NH_4OH, and $Na_2S_2O_3$ form emulsions in the organic phase and hence are not useful as stripping agents.

The extracted species of Hg(II) from HCl and HBr media were stripped out completely with a mixture of $5.0\,\mathrm{mol\,dm^{-3}}$ $HNO_3 + 4.0\,\mathrm{mol\,dm^{-3}}$ HCl and $2.0\,\mathrm{mol\,dm^{-3}}$ HNO_3, respectively.

Separation of Hg(II) from Cd(II) and Zn(II)

Separation of Hg(II) from Cd(II) and Zn(II) was carried out from both the media by taking advantage of the difference in their extraction and stripping conditions, similar to that reported earlier by Tandon et al. (37,38), which are shown in Flow Charts 1 and 2.

Validity of Methods

In order to study the applicability of the proposed methods in real samples, the proposed methods were employed for the analysis of mercury in Zandu parad tablet and Sutshekhar rasa sada tablet (manufactured by Zandu Pharmaceuticals India Ltd. Mumbai, Bombay), which are Ayurvedic medicines useful for controlling hyperacidity. The experimental results were found to be in good agreement with the reported values (Table 4). Similarly, the mercury present in creek water sample was also recovered by proposed methods (Table 5).

Table 4. Analysis of Mercury in Ayurvedic Drug Samples with 0.10 and 0.08 $\mathrm{mol\,dm^{-3}}$ Cyanex-923 in Toluene from Chloride and Bromide Media, Respectively

Medium	Sample	Present (mg)	Found (mg)	% Recovery	Relative Standard Deviation (RSD) (%)
HCl	Zandu parad tablet	60.0	58.6	99.35[a]	0.47
	Sutshekhar rasa sada tablet	10.8	10.5	98.50[a]	0.64
HBr	Zandu parad tablet	60.0	58.6	98.80[a]	0.86
	Sutshekhar rasa sada tablet	10.8	10.5	99.1[a]	0.59

[a] Mean of triplicate value.

Table 5. Analysis of Mercury in Creek Water Samples with 0.10 and 0.08 mol dm^{-3} Cyanex-923 in Toluene from Chloride and Bromide Media, Respectively

Sample	Found (mg L^{-1})	Media	% Recovery	Relative Standard Deviation (RSD) (%)
Creek water sample	0.232	HCl	98.4[a]	0.75
		HBr	98.7[a]	0.68

[a] Mean of triplicate value.

CONCLUSION

The results obtained show that commercially available phosphine oxide, i.e., Cyanex-923 in toluene can be used effectively for quantitative extraction of Hg(II) from chloride as well as bromide media.

The use of bromide media is advantageous than chloride because it requires less reagent concentration and its easy stripping.

Proposed methods for extraction of Hg(II) from bromide media are simple, rapid and can be successfully used for separation of Hg(II) from Cd(II) and Zn(II). Similarly, they were also successfully applied for the recovery of Hg(II) from real samples.

NOMENCLATURE

[ ] concentration [mg L^{-1} and mol dm^{-3}]
D distribution ratio
[R] reagent concentration
% E percentage extraction
% R percentage recovery

Subscripts
aq. aqueous phase
org. organic phase

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