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# Separation of Te(IV) and Se(IV) by Extraction with Cyanex 925

Ashok A. Mhaske[a]; Purushottam M. Dhadke[a]
[a] Inorganic Chemistry Laboratory, Applied Chemistry Division, Department of Chemical Technology, University of Mumbai, Matunga, Mumbai, India

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# Separation of Te(IV) and Se(IV) by Extraction with Cyanex 925

**Ashok A. Mhaske and Purushottam M. Dhadke***

Inorganic Chemistry Laboratory, Applied Chemistry Division,
Department of Chemical Technology, University of Mumbai,
Matunga, Mumbai, India

## ABSTRACT

Liquid–liquid extraction of tellurium(IV) and selenium(IV) with Cyanex 925 in toluene from aqueous hydrochloric acid media was studied separately, under the influence of variables such as: hydrochloric acid concentration, reagent concentration, equilibration time, diverse ions, stripping agents, and temperature. The difference in extractability of these metal ions in hydrochloric acid media toward Cyanex 925 facilitates their separation. The temperature dependence of extraction equilibriums were examined by the temperature variation method to estimate apparent thermodynamic function, ΔH. From the temperature study, the extraction reaction for Te(IV) was found to be exothermic in nature, while that of Se(IV) endothermic.

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[*] Correspondence: Purushottam M. Dhadke, Inorganic Chemistry Laboratory, Applied Chemistry Division, Department of Chemical Technology, University of Mumbai, Matunga, Mumbai 400019, India; Fax: 91-22-4145614; E-mail: dvc_pmd@aplchem.udct.emet.in.

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*Key Words:* Extraction; Tellurium(IV); Selenium(IV); Separation; Temperature; Cyanex 925.

## INTRODUCTION

Tellurium and selenium are important elements with wide applications in electronic and alloy industries. Apart from sulfide ores, the major source of these elements is the anode slime residue. They are separated and recovered by pyrometallurgical process but their recovery is incomplete.[1] The extraction study of Te(IV) from Te(VI),[2] Te(IV) from Fe(III),[3–4] and Te(IV) from Al, Bi, Cr, Co, Ni, and Se[5] has been reported. The separation of Te(IV) from Fe(III) is possible by precipitation method,[6] but it is not suitable if the tellurium concentration is less than 0.1 mg/mL. The reported work on extraction and separation of Te(IV) and Se(IV) with TBP,[7–8] indicates that very high TBP concentrations are required for their extraction.

Recently, a series of organophosphine compounds have been marketed under the trade name of Cyanex, (272, 301, 302) as potential acidic extractants for metal ions, while Cyanex (921, 923, 925) are the other neutral extractants found to be superior extractants for the extraction of precious metals.[9–14] Cyanex 925 is a liquid mixture having 26.4% tri(2,4,4-tri methyl pentyl) phosphine oxide and 65.9% tri-alkyl phosphine oxide as major components. The other components are sulfur compounds.[15–16] The present work deals with the extraction and separation study of tellurium(IV) and selenium(IV) using Cyanex 925 in toluene.

## EXPERIMENTAL

### Apparatus and Reagents

A GBC 911A/UV visible spectrophotometer was used for absorption measurements. The extractant, Cyanex 925 supplied by Cytec Canada Inc., Canada, was used without further purification. The stock solutions of Te(IV) and Se(IV) were prepared by treating metallic tellurium/sellenium with aqua-regia, evaporating to dryness and finally treating with dilute hydrochloric acid. These solutions were standardized by known methods.[17] The working solutions were prepared by further dilution with double distilled water. All other chemicals used were of analytical reagent grade.

### General Extraction Procedure

A 10 cm$^3$ of aqueous phase containing 100 μg of Te(IV)/Se(IV) ion was shaken for 10 minutes with equal volume of organic phase containing Cyanex 925 in toluene using a mechanical shaker immersed in a water bath maintained at 303 ± 1°K (except for temperature study). After extraction, the two phases were allowed to separate. The Te(IV) concentration in the aqueous phase was determined spectrophotometrically by thiourea method at 366 nm,[17] while Se(IV) was determined by using 3,3′-diaminobenzidine at 420 nm.[18] The metal content in the loaded organic phase was determined by the mass balance of the metal before and after extraction. In some experiments, the ionic strength of the aqueous phase was adjusted using LiCl/HCl and the hydrogen ion concentration was determined potentiometricaly using 10 cm$^3$ of the same solution separately as standard.

## RESULTS AND DISCUSSION

### Effect of Hydrochloric Acid Concentration

The dependence of the extraction of Te(IV) and Se(IV) on the hydrochloric acid concentration (Fig. 1) exhibits that the extraction increases with an increase in hydrochloric acid and becomes quantitative in the range of 3.0 to 4.0 M and 7.0 to 8.0 M HCl, respectively. The decrease in their extraction at a high hydrochloric acid concentration is due to the mass action effect of the chloride ion.[9–11]

### Effect of Reagent Concentration

Extraction of Te(IV) and Se(IV) was carried out by varying the Cyanex 925 concentration from $1 \times 10^{-3}$ M to $1.5 \times 10^{-1}$ M in toluene. The optimum concentration required for the quantitative extraction of Te(IV) was found to be $1 \times 10^{-1}$ M at 3.5-M HCl. For Se(IV), it was with $5 \times 10^{-2}$ M at 7.5-M HCl (Fig. 2).

### Loading Isotherm and Loading Capacity

The extraction of Te(IV) and Se(IV) by varying the metal concentration in the aqueous phase was studied individually at a constant temperature of 303°K

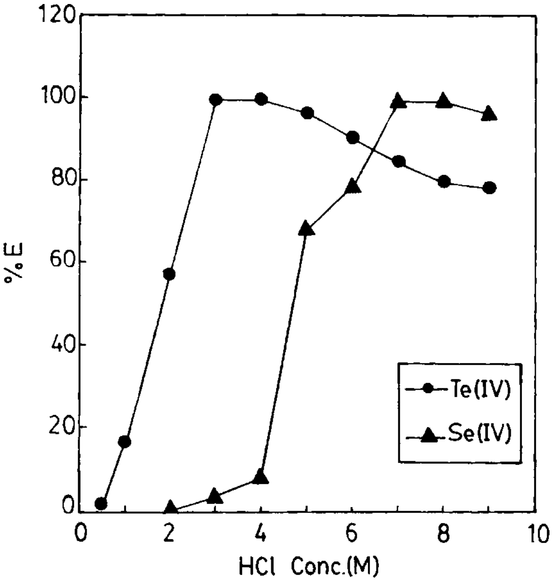


*Figure 1.* Effect of hydrochloric acid concentration on percentage extraction (% E). (Org. phase: $1 \times 10^{-1}$ M, equilibration time: 10 min.)

under the optimum conditions required for their extraction. The slope ratio analysis suggests that the extracted species are monomeric in nature (Fig. 3). The maximum loading capacity for Te(IV) was found to be 750 μg per 10 $cm^3$ and 1125 μg per 10 $cm^3$ for Se(IV), respectively from 3.5-M and 7.5-M HCl.

## Effect of Equilibration Period

The equilibration period for Te(IV) and Se(IV) extraction was studied separately at different hydrochloric acid concentrations, 3.5 M and 7.5 M, respectively. A minimum equilibration period of 5 minutes was found to be optimum for the quantitative extraction of Te(IV) with 3.5-M HCl, while it was a maximum 97.4% at 7.5-M HCl. Increasing the period up to 30 minutes had no adverse effect on extraction. In case of Se(IV), quantitative extraction was found from 5 to 30 minutes with 7.5-M HCl. However, with 3.5-M HCl, there was no extraction of Se(IV) up to 5 minutes. The extraction was 4.6% with a period of 10 to 30 minutes. These results show that Te(IV) can be easily separated at 3.5-M HCl with a 5-minute equilibration period (Table 1).

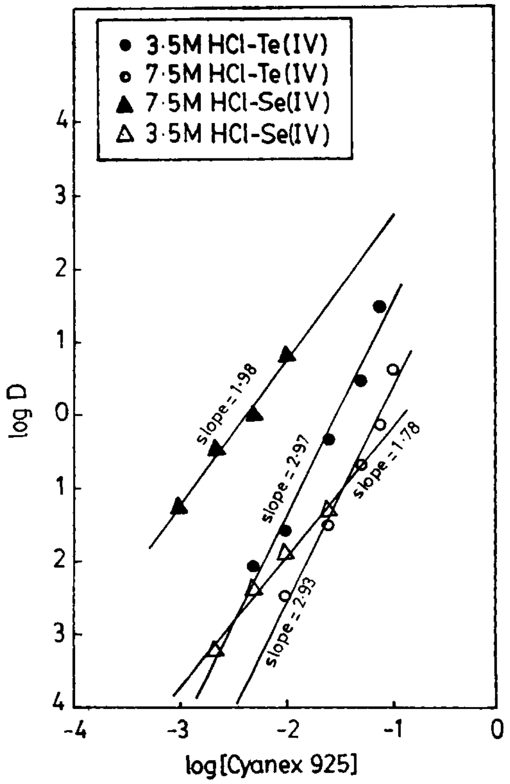


***Figure 2.*** Effect of extractant concentration on distribution ratio (D). (Equilibrium time: 10 min.)

From the rate of reaction study at different acid concentration, different species of Se(IV) were found to be present.

## Extraction Mechanism

An attempt was made to ascertain the nature of the extracted species using logD–logC plots. The plots of logD vs log[Cyanex 925] exhibited slopes of $\approx 3$ and $\approx 2$ for Te(IV) and Se(IV), respectively, at 3.5-M and 7.5-M HCl (see Fig. 2). This indicates that the metal to Cyanex 925 ratio is 1:3 for Te(IV) and 1:2 for Se(IV). Similarly, the plot of logD versus log[HCl] for Te(IV) gives slopes of 3.91 and $-3.78$ in the range of 0.5 to 3.0 M HCl and 5.0 to

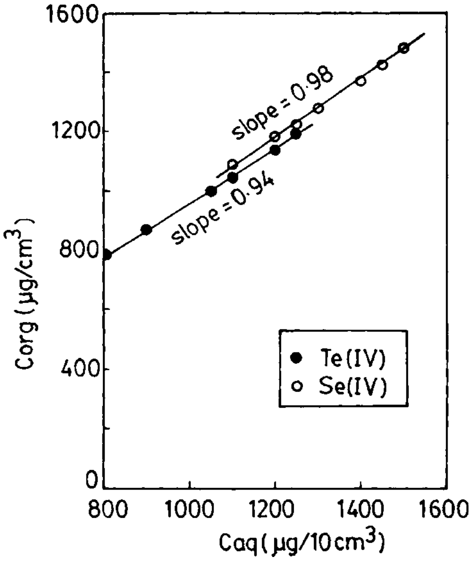


***Figure 3.*** Loading isotherm for Te(IV) and Se(IV). (Org. phase: $1 \times 10^{-1}$ M; equilibrium time: 10 min.)

***Table 1.*** Effect of equilibration time on percentage extraction of Te(IV) and Se(IV).

| Equilibration time (min) | Percentage extraction (% E) | | | |
|---|---|---|---|---|
| | 3.5-M HCl | | 7.5-M HCl | |
| | Te(IV) | Se (IV) | Te(IV) | Se (IV) |
| 1 | 58.1 | 0.0 | 36.3 | 54.3 |
| 2 | 85.6 | 0.0 | 65.5 | 81.6 |
| 5 | 99.4 | 0.32 | 97.4 | 99.1 |
| 7 | 99.4 | 4.6 | 97.4 | 99.1 |
| 10 | 99.4 | 4.6 | 97.4 | 99.1 |
| 15 | 99.4 | 4.6 | 97.4 | 99.1 |
| 30 | 99.4 | 4.6 | 97.4 | 99.1 |

8.0 M HCl, respectively. However, in case of Se(IV), the different slopes 1.74 (below 5.0 M HCl) and 3.86 (above 5.0-M HCl) observed indicate that different types of chlorocomplexes of Se(IV) are formed at different acid concentrations (Fig. 4). The extraction of Se(IV) and Te(IV) was carried out from LiCl solution by varying the hydrogen ion concentration, while keeping chloride ion concentration constant, i.e., $Li^+(Cl^-, H^+) = 2.5$ M, 4.5 M, and 7.5 M for Te(IV), and 4.5 M and 6.0 M in case of Se(IV), with varying hydrogen ion concentrations. The observed slopes of $\log[H^+]$ vs logD for Te(IV) were found to be 0.09, 0.14, and −0.04, respectively for 2.5 M, 4.5 M, and 7.5 M as total the strength of chloride. This means that the extraction was independent of hydrogen ion concentration under the studied acid range (Fig. 5). On the other hand, in case of Se(IV), the slopes obtained were 0.11 and 1.87, respectively for 4.5 M and 6.0 M as total ionic strength, suggesting that at lower acid concentration range (below 5.0 M), the extraction of Se(IV) as independent of the hydrogen ion concentration, while that at a higher range acid concentration (above 5.0 M), it shows a second-order dependency on the hydrogen ion.

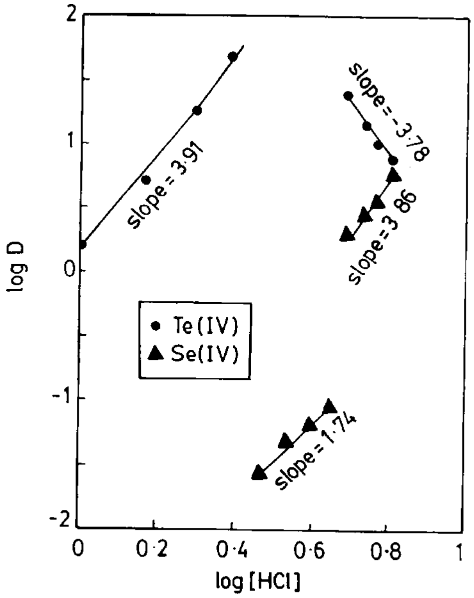


*Figure 4.* Effect of hydrochloric acid concentration on distribution ratio (D). (Org. phase: $1 \times 10^{-1}$ M; equilibration time: 10 min.)

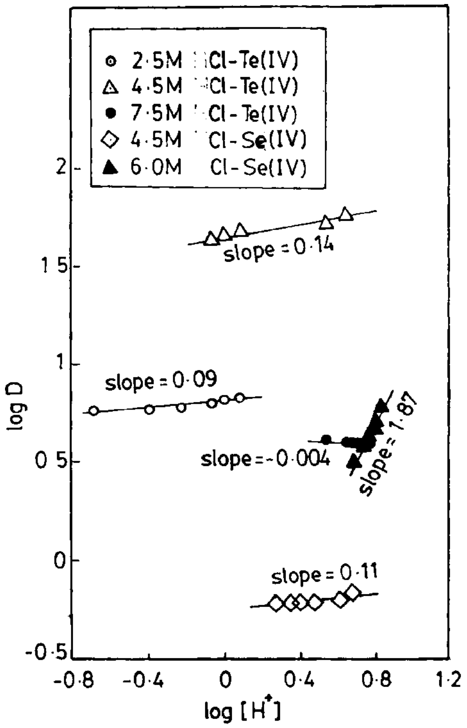


***Figure 5.*** Effect of hydrogen ion concentration on distribution ratio (D) of Te(IV) and Se(IV). (Org. phase: $1 \times 10^{-1}$ M; equilibration time: 10 min.)

Thus, the extraction mechanism for the Te(IV) and Se(IV) extraction with Cyanex 925 can be given as

$$\mathrm{Te^{4+} + 4\,Cl^- + 3\,Cyanex\ \ 925 \longrightarrow \overline{TeCl_4.3\,Cyanex\ \ 925}} \qquad (1)$$

$$\mathrm{SeO^{2+} + 2\,Cl^- + 2\,Cyanex\ \ 925 \longrightarrow \overline{SeOCl_2.2\,Cyanex\ \ 925}}$$

$$(< 5.0\text{-M HCl}) \qquad (2)$$

and

$$\mathrm{SeO^{2+} + 2\,H^+ + 4\,Cl^- + 2\,Cyanex\ \ 925 \longrightarrow \overline{H_2SeOCl_4.2\,Cyanex\ \ 925}}$$

$$(> 5\text{-M HCl}) \qquad (3)$$

Similar behavior was observed for extraction of these metals with TBP.[8] The apparent extraction constant (K′) for the above reactions can be given as

$$K'_{\mathrm{Te}} = \frac{[\overline{\mathrm{TeCl_4.3\,Cyanex\ \ 925}}]}{[\mathrm{Te^{4+}}][\mathrm{Cl^-}]^4[\mathrm{Cyanex\ \ 925}]^3} \tag{4}$$

$$K'_{\mathrm{Se}} = \frac{[\overline{\mathrm{SeOCl_2.2\,Cyanex\ \ 925}}]}{[\mathrm{SeO^{2+}}][\mathrm{Cl^-}]^2[\mathrm{Cyanex\ \ 925}]^2} \tag{5}$$

$$K'_{\mathrm{Se}} = \frac{[\overline{\mathrm{H_2SOCl_4.2\,Cyanex\ \ 925}}]}{[\mathrm{SeO^{2+}}][\mathrm{H^+}]^2[\mathrm{Cl^-}]^4[\mathrm{Cyanex\ \ 925}]^2} \tag{6}$$

## Effect of Diverse Ions

The extraction of Te(IV) and Se(IV) was carried out in presence of a large number of foreign ions. The tolerance limit was set so that it affected not more than ± 2% on its extraction. The alkali, alkaline earth metal ions $Na^+$, $K^+$, $Cs^+$, $Rb^+$, $Mg^{+2}$, $Ca^{+2}$, $Ba^{+2}$ and $Sr^{+2}$ and anions such as $Cl^-$, $Br^-$, $I^-$, $SO_3{}^{-2}$, citrate, and oxalate were found more tolerable. However Fe(III), thiourea, and thiocyanate were found to be highly interfering (Table 2).

***Table 2.*** Effect of diverse ions on percentage extraction of Te(IV).

| Tolerance limits | Metal ions | |
|---|---|---|
| | Te(IV) = 100 μg | Se(IV) = 100 μg |
| 1:22 | — | $Na^+$, $K^+$, $Cs^+$, $Rb^+$, $Mg^{+2}$, $Ca^{+2}$, $Ba^{+2}$, $Sr^{+2}$ |
| 1:18 | $Na^+$, $K^+$, $Cs^+$, $Rb^+$, $Mg^{+2}$, $Ca^{+2}$, $Ba^{+2}$, $Sr^{+2}$ | $Cl^-$, $Br^-$, $I^-$, $SO_3{}^{-2}$, citrate, oxalate |
| 1:15 | $Cl^-$, $Br^-$, $I^-$, $SO_3{}^{-2}$, citrate, oxalate | $Cr^{+3}$, $Zn^{+2}$, $In^{+3}$, $Ga^{+3}$, $Th^{+3}$, $Al^{+3}$, $Sb^{+3}$, $Bi^{+3}$ |
| 1:12 | $Cr^{+3}$, $Zn^{+2}$, $In^{+3}$, $Ga^{+3}$, $Th^{+3}$ | — |
| 1:8 | $Al^{+3}$, $Sb^{+3}$, $Bi^{+3}$ | — |
| 1:0 | $Se^{+4}$, $Fe^{+3}$, thiocyanate, thiourea | $Te^{+4}$, $Fe^{+3}$, thiocyanate, thiourea |

## Effect of Stripping Agents

The stripping of metal-loaded organic phase containing Te(IV) and Se(IV) was tried with various stripping agents, such as HCl, $H_2SO_4$, $HNO_3$, $HClO_4$, $H_2O_2$, and $K_2Cr_2O_7$, in 1.0-M HCl. Only the oxidizing agents, like $HNO_3$, $H_2O_2$, and $K_2Cr_2O_7$, in 1.0-M HCl were found to completely back extract these metal ions (Table 3). $H_2O_2$ cannot be used as stripping agent since it leads to the decomposition of the extractant, Cyanex 925. Complete stripping with this oxidizing agent may be due to the oxidation of Te(IV) and Se(IV) to their higher oxidation state, Te(VI) and Se(VI).

Further, the complex species formed by Te(IV) with Cyanex 925 is comparatively more bulky than that formed with Se(IV), which seems to be the other reason for comparatively easy stripping of Te(IV) than Se(IV). It was found that Te(IV) is completely stripped (99.4%) in a single step with 6.0-M $HNO_3$, while, for Se(IV), stripping twice gives a maximum (98.5%) recovery.

## Influence of Temperature

Extraction of Te(IV) and Se(IV) was carried out using $1 \times 10^{-1}$M Cyanex 925 in toluene in the temperature range of 303 to 353°K. For both the Te(IV) and Se(IV), aqueous acidity was maintained at both the 3.5-M HCl and 6.0-M

***Table 3.*** Effect of stripping agents on percentage recovery of Te(IV) and Se(IV).

| | | Percentage recovery (% R) | | |
|---|---|---|---|---|
| Stripping agents | Concentration (M) | Te(IV) | Se(IV) | |
| $HNO_3$ | 2.0 | 48.5 | 34.6 | 57.1[a] |
| | 4.0 | 64.6 | 56.1 | 80.8[a] |
| | 6.0 | 99.4 | 87.6 | 98.5[a] |
| | 8.0 | 99.0 | 87.1 | 98.3[a] |
| $H_2O_2$[b] (%v/V) | 0.1 | 68.2 | 46.5 | |
| | 0.5 | 99.6 | 78.1 | |
| | 1.0 | 99.6 | 98.8 | |
| $K_2Cr_2O_7$ in 1.0-M HCl | 0.01 | 85.1 | 31.4 | |
| | 0.05 | 98.8 | 68.4 | |
| | 0.1 | 98.8 | 97.1 | |

[a] Twice stripping.

[b] Extractant decomposition.

HCl. The extraction of Te(IV) was found to decrease with the temperature increase, while, in case of Se(IV), it increased with an increase in temperature. According to the Van't Hoff equation, the change in the equilibrium constant ($K'$) with temperature is given as

$$\frac{\delta(\log K')}{\delta(1/\mathrm{T})} = \frac{\Delta H}{2.303\,\mathrm{R}} \tag{7}$$

where, $\Delta H$ is the enthalpy change for the extraction reaction. The values of $\log K'$ at different temperature were calculated from Eqs. (4), (5), and (6) and plotted against $1000/\mathrm{T}\,\mathrm{K}^{-1}$ (Fig. 6). The slopes obtained from these plots

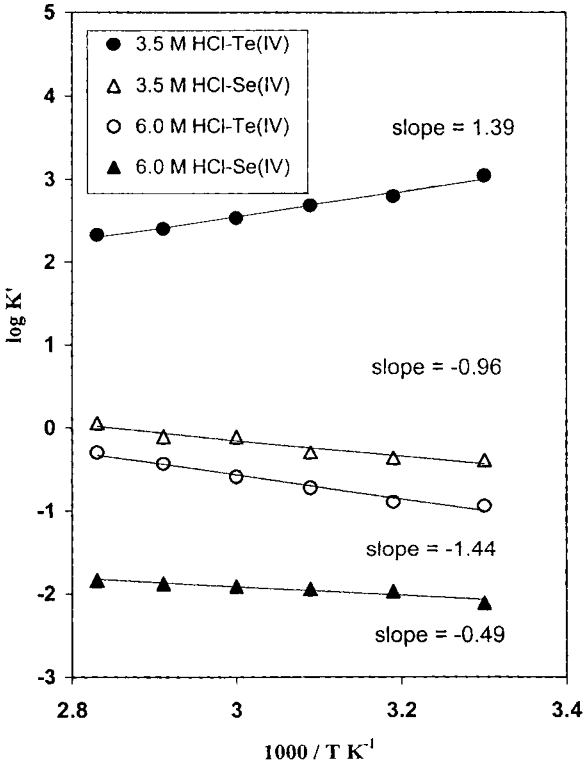


***Figure 6.*** Effect of temperature on distribution ratio (D). (Org. phase: $1 \times 10^{-1}$ M; equilibration time: 10 min.)

were found to be 1.39 and $-0.96$ for Te(IV) and Se(IV), respectively at 3.5-M HCl. However, at 6.0-M HCl, the slopes were $-1.44$ and $-0.49$ for Te(IV) and Se(IV), respectively. The corresponding $\Delta H$ values obtained were $-26.70$ kJ/mol and $+18.44$ kJ/mol for Te(IV) and Se(IV), respectively at 3.5-M HCl. This indicates that the reaction is exothermic for Te(IV) and endothermic in the case of Se(IV). The values of enthalpy change obtained at 6.0-M HCl were $+27.66$ kJ/mol and $+9.41$ kJ/mol for Te(IV) and Se(IV), respectively. The different enthalpy changes observed in the case of Se(IV) at different acidity, again, indicate different extraction reaction taking place at different acidity. Further, the enthalpy change $+18.44$ kJ/mol obtained at 3.5-M HCl is more positive in magnitude as compared to $+9.41$ kJ/mol at 6.0-M HCl, suggesting that a comparatively less stable complex of Se(IV) is formed at 3.5-M HCl.

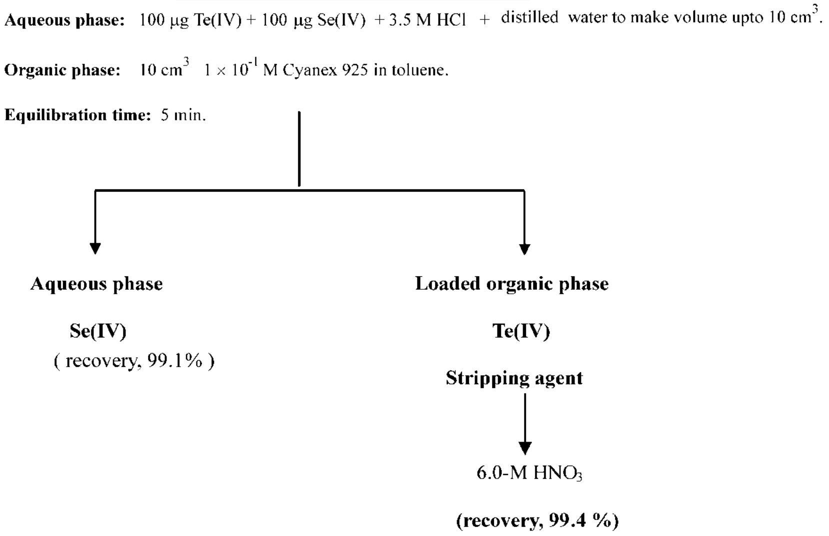
The Se(IV) extraction increased with an increase in temperature. This due to the increase in solubility of Cyanex 925–Se(IV) metal solvate species in the organic phase, while in case of Te(IV), the extraction decrease is due to a bulky, complex $\overline{\mathrm{TeCl_4.3\,Cyanex\ 925}}$ formed with three reagent molecules.

## Separation of Te(IV) from Se(IV)

The separation of Te(IV) and Se(IV) from each other was carried out by taking advantage of their different extraction conditions toward Cyanex 925. The quantitative extraction of Te(IV) was found to take place at 3.0 to 4.0 M HCl with $1 \times 10^{-1}$ M Cyanex 925. However, the extraction of Se(IV) was quantitative at comparatively higher acidity (7.0 to 8.0 M) with $5 \times 10^{-2}$ M Cyanex 925. At aqueous acidity 3.5-M HCl and with 1 to 5 minutes for an equilibration period, there was no extraction of Se(IV) was observed. This facilitated the complete separation of Te(IV) from Se(IV) at 3.5-M HCl.

For their separation, 10 cm$^3$ of aqueous solution containing a mixture of 100 μg each of Te(IV) and Se(IV) in 3.5-M HCl was equilibrated for 5 minutes with equal volume of $1 \times 10^{-1}$ M Cyanex 925. It was found that Te(IV) get completely (99.4%) extracted in organic phase, while Se(IV) remained unextracted in aqueous phase (see Flowsheet 1). Thereafter, Te(IV) from the loaded organic phase was stripped out with 6.0-M $HNO_3$. The separation of Te(IV) from Se(IV) in the various ratios was also carried out at the optimum extraction conditions required for the extraction of Te(IV). Results are given in Table 4.

**Flowsheet 1** — Separation of Te(IV) from Se(IV)



**Aqueous phase:** 100 μg Te(IV) + 100 μg Se(IV) + 3.5 M HCl + distilled water to make volume upto 10 $cm^3$.

**Organic phase:** 10 $cm^3$ $1 \times 10^{-1}$ M Cyanex 925 in toluene.

**Equilibration time:** 5 min.

***Table 4.*** Separation of Te(IV) and Se(IV) in their various ratio of mixtures.

| Mixture | μg | Amount found (μg) | Recovery (%) |
|---|---|---|---|
| Te(IV) | 25 | 24.8 | 99.4 |
| Se(IV) | 50 | 49.6 | 99.1 |
| Te(IV) | 75 | 74.5 | 99.4 |
| Se(IV) | 100 | 99.1 | 99.1 |
| Te(IV) | 100 | 99.4 | 99.4 |
| Se(IV) | 150 | 148.6 | 99.0 |
| Te(IV) | 200 | 197.5 | 98.7 |
| Se(IV) | 250 | 246.2 | 98.5 |

## CONCLUSION

The proposed methods are simple and rapid for extraction of Te(IV) and Se(IV) in the presence of a large number of foreign ions. Also, it is possible to separate Te(IV) from Se(IV) by the proposed method. As compared to TBP (0.736 M to 1.84 M),[8] the present method requires less reagent concentration for the extraction of Te(IV) and Se(IV). The complete separation of Te(IV) from Se(IV) in the present method as compared to TBP ($\beta_{Te/Se} = 78.70$) indicates that it is a better separation method. From the temperature study, the extraction reaction for Te(IV) was found to be exothermic, while, that of Se(IV), endothermic. This suggests that the temperature 303°K is efficient for the separation of Te(IV) from Se(IV).

## ACKNOWLEDGMENT

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