# Extractive Separation of Selenium and Tellurium Using 2,3,5-Triphenyltetrazolium Chloride

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A simple and selective solvent-extraction method has been devised for the separation of  $Se^{IV}$  and  $Te^{IV}$ , not only from each other, but also from the other accompanying elements. 2,3,5-Triphenyltetrazolium chloride has been used as an extractant for the quantitative extraction of  $Te^{IV}$  and  $Se^{IV}$  from bromide and thiocyanate medium, respectively. The formed ion-pair complex of  $Te^{IV}$  and  $Se^{IV}$  can be quantitatively extracted into a dichloromethane and 1:1 cyclohexanol-toluene solvent mixture, respectively. The optimum conditions have been evaluated based on a critical study of the acid concentration, extractant concentration, period of equilibration, effect of dilution and effect of the solvent. Water was used for the stripping of tellurium, while a fresh solution of sodium hypobromite was used for selenium. This separation procedure in conjunction with the developed methods was applied to an analysis of the selenium and tellurium content of copper concentrate, incineration ash and In–Sb alloy samples.

The separation methods are generally an integral part of an analysis scheme being used to isolate the analyte from possible interference and to concentrate the metal ion of interest from a dilute solution so as to facilitate easy measurements. Since selenium and tellurium co-exist in most materials, and since they interfere with each other in their methods of determination, many methods<sup>1-5)</sup> have been reported for their separation from each other and also from other elements. Even though extensive work has been carried out on this, in many instances the available methods are found to be inadequate for their successive separation when they are present together. Therefore, they are only useful for the separation of any one of them from the accompanying elements. It was therefore felt worthwhile to evolve an entirely new procedure which would facilitate the separation of selenium and tellurium to be effected successively with minimum manipulation so that the method can find use for the separation of these ions from a variety of practical samples.

High-molecular-weight surfactants have emerged as powerful extractants for many elements. Recently, it has been shown that 2,3,5-triphenyltetrazolium chloride (TTC) is an effective extractant for the extraction of anionic complexes of metal ions. An investigation using this reagent revealed that Te<sup>IV</sup> could be extracted as an ion-pair complex from a bromide medium into dichloromethane while Se<sup>IV</sup> was left behind in the aqueous layer. Subsequently, Se<sup>IV</sup> could be extracted as an ion-pair complex with 2,3,5-triphenyltetrazolium chloride from a thiocyanate medium into a 1:1 solvent mixture of cyclohexanol and toluene. The method is found to be rapid and provides the sequential separation of tellurium and selenium, and also from other matrix elements.

## **Experimental**

Apparatus. Activity measurements of radionuclides were car-

ried out using a Packard Tri-Carb 1500 liquid scintillation analyzer and a  $1.75\times2''$  NaI(Tl) single-channel scintillation counter. An inductively coupled plasma atomic emission spectrometer (ARL-3401) was used for the analysis of selenium (196.03 nm) and tellurium (214.3 nm).

**Reagents.** All reagents were of analytical reagent grade and doubly distilled water was used throughout the experiments.

Te<sup>IV</sup> Solution (100  $\mu g \, mL^{-1}$ ): A bulk solution of 1000  $\mu g \, mL^{-1}$  was prepared by dissolving 0.125 g of tellurium dioxide (Fluka) in 3 mL of 0.1 M (1 M=1 mol dm<sup>-3</sup>) sodium hydroxide and diluting to 100 mL using distilled water. From this, a working solution of 100  $\mu g \, mL^{-1}$  was prepared by suitable dilution with water.

Se<sup>IV</sup> Solution (100  $\mu g\,mL^{-1}$ ): A bulk solution of 1000  $\mu g\,mL^{-1}$  was prepared by dissolving 0.14 g of selenium dioxide (Fluka) in 100 mL of distilled water. From this, a working solution of 100  $\mu g\,mL^{-1}$  was prepared by suitable dilution with water.

Then, 50 mL each of a 2.0% solution of 2,3,5-triphenyltetrazolium chloride, 4 M solution of potassium bromide and 3 M solution of potassium thiocyanate were prepared by dissolving appropriate amounts of the compounds in distilled water.

General Procedure. Extraction of  $Te^{IV}$ : An aliquot containing 100 µg of  $Te^{IV}$  was transferred into a separatory funnel. To this 2 mL of a 2% solution of 2,3,5-triphenyltetrazolium chloride and 5 mL of 4 M potassium bromide solutions were added. The mixture was diluted to 10 mL, maintaining an overall acidity of 1 M with respect to sulfuric acid. The resulting ion-pair complex was extracted for 5 min with 10 mL of dichloromethane. After discarding the aqueous layer an organic layer containing the extracted ion-pair complex of  $Te^{IV}$  was equilibrated with 10 mL of distilled water. Under this condition  $Te^{IV}$  was quantitatively stripped back into the aqueous phase and the amount of  $Te^{IV}$  was determined by following the developed radiodisplacement procedure.  $^{8}$ 

Extraction of Se<sup>IV</sup>: A 1 mL volume of a 100 μg mL<sup>-1</sup> solution of Se<sup>IV</sup> was placed in a separatory funnel. To this 1 mL of 3 M potassium thiocyanate and a 1 mL of 2% solution of 2,3,5-triphenyltetrazolium chloride were added and the mixture was diluted to

10 mL, maintaining an overall acidity of 2.7 M with respect to sulfuric acid. The formed ion-pair complex was extracted for 5 min with 10 mL of 1:1 cyclohexanol and toluene solvent mixture. After discarding the aqueous layer the organic layer was washed with 10 mL of a 0.1 M sodium acetate solution so as to remove the free thiocyanate extracted as thiocyanic acid. The organic extract was then equilibrated with 10 mL of sodium hypobromite solution prepared freshly by adding a sufficient amount of sodium hydroxide to a saturated solution of bromine water to make the solution 0.3 M in alkali. This enabled the quantitative stripping of selenium from the organic phase.

A known amount of aqueous solution containing the stripped Se<sup>IV</sup> was treated with 2 mL of concentrated nitric acid contained in a beaker. The contents were evaporated to near dryness, cooled and the residue was treated with 2 mL of 4 M hydrochloric acid. The amount of Se<sup>IV</sup> was then estimated by the radiometric method as discussed in a previous paper.<sup>9)</sup>

Sequential Separation Scheme for Te<sup>IV</sup> and Se<sup>IV</sup>: containing 100  $\mu$ g each of Te<sup>IV</sup> and Se<sup>IV</sup> were transferred into a separatory funnel followed by the addition of 5 mL of 4 M potassium bromide and 2 mL of a 2% solution of 2,3,4-triphenyltetrazolium chloride. The mixture was adjusted to a final volume of 10 mL, maintaining an overall acidity of 1 M with respect to sulfuric acid. It was then equilibrated with 10 mL of dichloromethane. Under this condition  $Te^{IV}$  was alone extracted into the organic layer as an ion-pair complex, leaving behind Se<sup>IV</sup> in the aqueous layer. After quantitatively separating the organic layer the aqueous layer was made 0.3 M with respect to potassium thiocyanate and 2.7 M with respect to sulfuric acid. To this 2 mL of a 2% solution of 2.3. 5-triphenyltetrazolium chloride was added, and the final aqueous volume was adjusted to 20 mL. It was then equilibrated for 5 min with 10 mL of 1:1 cyclohexanol and toluene. After discarding the aqueous layer the organic layer was washed with 10 mL of a 0.1 M sodium acetate solution.

From the organic extracts Te<sup>IV</sup> and Se<sup>IV</sup> were quantitatively stripped back using 10 mL of water and a hypobromite solution, respectively, as described earlier. The amounts of Te<sup>IV</sup> and Se<sup>IV</sup> were then established.

# **Results and Discussion**

**Effect of the Sulfuric Acid Concentration:** The extraction of Te<sup>IV</sup> was found to be quantitative in the range of 0.75—2.5 M and that of Se<sup>IV</sup> in the range of 2.5—4.0 M of sulfuric acid, as shown in Fig. 1.

Effect of the Potassium Bromide and Potassium Thiocyanate Concentration: The extraction of Te<sup>IV</sup> was quantitative in the range of 1.6—4.0 M potassium bromide and that of Se<sup>IV</sup> in the range of 0.25—0.5 M potassium thiocyanate.

Effect of the 2,3,5-Triphenyltetrazolium Chloride Concentration: Experimental studies showed that an overall concentration of 0.3 and 0.15% of TTC was necessary for the quantitative extraction of Te<sup>IV</sup> and Se<sup>IV</sup>, respectively, and that there was no appreciable change in the extraction of these elements by the presence of a higher concentration of TTC. However, an overall concentration of 0.4 and 0.2% of TTC was maintained in order to ensure the quantitative extraction of Te<sup>IV</sup> and Se<sup>IV</sup>, respectively, as followed in the general procedure.

**Effect of Solvent:** Studies were then performed to find

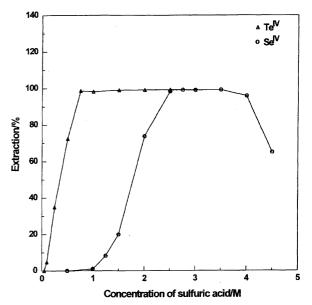


Fig. 1. Effect of sulfuric acid concentration on the extraction of Te<sup>IV</sup> and Se<sup>IV</sup> with TTC. Δ: Extractions were carried out using 100 μg of Te<sup>IV</sup> from 2 M KBr, 0.2% TTC and varying concentrations of sulfuric acid raging from 0.75—2.5 M. Solvent: 10 mL dichloromethane. ○: Extractions were carried out using 100 μg of Se<sup>IV</sup> from 0.3 M KSCN, 0.2% TTC and varying concentrations of sulfuric acid ranging from 0.5—4.5 M. Solvent: 10 mL of 1:1 cyclohexanol and toluene.

out the most suitable solvent for the extraction of the ion-pair complex of  $Te^{IV}$  and  $Se^{IV}$ . The results are summarized in Tables 1 and 2. It is clear from the data that amongst the solvent used dichloromethane offers a quantitative extraction for  $Te^{IV}$  while a 1:1 solvent mixture of toluene and cyclohexanol provides a quantitative extraction for  $Se^{IV}$ .

**Effect of Sample Volume:** The extraction was quantitative for the  $Te^{IV}$  and  $Se^{IV}$  up to an aqueous phase volume of 50 and 40 mL, respectively. The optimum equilibration time was found to be 5 min for the extraction of both  $Te^{IV}$  and  $Se^{IV}$ .

Nature of the Extracted Complexes of Te<sup>IV</sup> and Se<sup>IV</sup>: The ratios of Te<sup>IV</sup> to TTC and Se<sup>IV</sup> to TTC were established following the equilibrium shift method. <sup>10)</sup> The combining ratios of tellurium to Br<sup>-11)</sup> and selenium to SCN<sup>-12)</sup> have

Table 1. Choice of Solvent for the Extraction of Te<sup>IV</sup> with 2,3,5-Triphenyltetrazolium Chloride from Bromide and Sulfuric Acid Media

Solvent	Extraction (%)	Distribution ratio (D)
Chloroform	97.9	46.6
Dichloromethane	99.4	>100
MIBK	76.4	3.3
Benzene	4.8	0.05
Toluene	4.8	0.05
Ethyl acetate	9.0	0.1
Cyclohexane	0	

Extractions were carried out using  $100 \mu g$  of  $Te^{IV}$  from 2 M KBr, 0.2% of TTC and 1 M sulfuric acid media.

Choice of Solvent for the Extraction of Se<sup>IV</sup> with 2,3,5-Triphenyltetrazolium Chloride from Thiocyanate and Sulfuric Acid Media

Solvent	Extraction (%)	Distribution ratio (D)	
Chloroform	96.3	35.7	
Dichloromethane	95.5	21.2	
Toluene	96.2	25.3	
Benzene	96.1	25.0	
MIBK	98.1	51.6	
Cyclohexanol	98.5	64.0	
Ethyl acetate	93.3	13.9	
Pentyl acetate	97.2	34.7	
Cyclohexanol + Toluene (1:1)	99.2	>100	
Amyl alcohol	96.7	29.3	

Extractions were carried out using 100 µg of Se<sup>IV</sup> from 0.3 M KSCN, 0.2% TTC and 2.7 M sulfuric acid media.

already been reported.

Te<sup>IV</sup> to TTC Ratio: Experiments were carried out using 100 µg of Te<sup>IV</sup> in 2 M potassium bromide made 0.25 M with respect to sulfuric acid and varying the concentration of TTC ranging from 0.01 to 0.06 M in a final volume of 10 mL. The solutions were equilibrated for 5 min with 10 mL of dichloromethane. The organic layer was separated and equilibrated for 5 min with 10 mL of water, and the amount of tellurium was established as described earlier. From this, the percentage extracted, and thus the distribution coefficient (D), were calculated. A log-log plot of distribution ratio vs. TTC concentration, as shown in Fig. 2, gives a straight line with a slope of 2, indicating a 1:2 mole ratio of Te<sup>IV</sup> to TTC. On the basis of the above observations it was concluded that the stoichiometry of the ion-pair is Te: 6Br: 2TTC.

v=2.09x + 2.54 0.6 0.2 -0.2 -0.6 -1.5 -1.2 -0.7 log [TTC]

Variation of distribution ratio for Te<sup>IV</sup> as a function of 2,3,5-triphenyltetrazolium chloride concentration. Extractions were carried out using 100 µg of Te<sup>IV</sup> from 2 M KBr, 0.25 M sulfuric acid and with varying amounts of TTC ranging from 0.01-0.06 M. Solvent: 10 mL of dichloromethane.

Se<sup>IV</sup> to TTC Ratio: Experiments were performed using 10 mL of aqueous solution, each containing 100 µg of selenium-75 labelled Se<sup>IV</sup> in 0.3 M with respect to potassium thiocyanate and 1.5 M with respect to sulfuric acid. The concentration of TTC was varied from  $1.5 \times 10^{-3}$  to  $7.7 \times 10^{-3}$ M. The aqueous solutions were then equilibrated for 5 min with 10 mL of 1:1 cychohexanol and toluene. The activity of 2 mL each of the organic and aqueous layers was measured using a NaI(Tl) scintillation counter. From the ratios of the activities the distribution ratio (D) was calculated. A log-log plot of the distribution ratio vs. the TTC concentration is shown in Fig. 3, which is a straight-line graph with a slope equal to 2.1, indicating a 1:2 mole ratio of Se<sup>IV</sup> to TTC. The stoichiometry of the ion-pair was found to be Se: 6SCN: 2TTC.

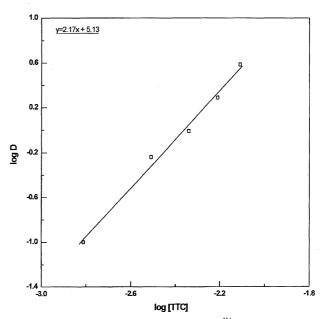


Fig. 3. Variation of distribution ratio for Se<sup>IV</sup> as a function of 2,3,5-triphenyltetrazolium chloride concentration. Extractions were carried out using 100  $\mu g$  of Se-75 labeled  $Se^{IV}$ using 0.3 M KSCN, 1.5 M sulfuric acid and with varying amounts of TTC ranging from  $1.5 \times 10^{-3}$ — $7.7 \times 10^{-3}$  M. Solvent: 10 mL of 1:1 cyclohexanol and toluene.

Effect of Stripping Agents: A variety of stripping agents such as water, hydrochloric acid, sulfuric acid, sodium hydroxide, and sodium acetate were used for the stripping of Te<sup>IV</sup> from the organic extract. The results are shown in Table 3, from which it is clear that all of the solutions examined are effective to strip Te<sup>IV</sup> from the organic layer. However, water was used to strip the extracted ion-pair complex of Te<sup>IV</sup> from the organic layer. Similar studies were carried out to find a suitable stripping solution for the quantitative stripping of Se<sup>IV</sup> from the organic extract. Amongst the various stripping solutions used, as summarized in Table 4, the use of a hypobromite solution enabled the quantitative stripping of Se<sup>IV</sup>.

**Study of the Co-extraction of Other Ions:** The extent of the extraction of other ions that are known to extract in a bromide and thiocyanate medium and the extent to which these ions are stripped were investigated separately at the 1 mg level. The metal ions that were left behind in the

Table 3. Choice of Electrolyte Solution for the Stripping of Te<sup>IV</sup> from the Organic Extract

Stripping solution used	% Stripped
Water	99.4
1 M Hydrochloric acid	99.7
0.5 M Sulfuric acid	99.1
0.5 M Sodium hydroxide	99.4
0.5 M Sodium acetate	99.8

Each extraction was carried out using 100  $\mu g$  of  $Te^{IV}$  from 2 M KBr, 0.2% TTC and 1 M sufuric acid media with 10 mL of dichloromethane.

aqueous phase after extraction and the amount of metal ions stripped were separately established by suitable analytical procedures. The results are summarized in Tables 5 and 6.

**Separation of Tellurium from Co-extracting Ions:** The results given in Table 5 indicate that amongst the ions studied only  $Hg^{II}$ ,  $Sb^{III}$ , and  $As^{III}$  were found to be co-extracted along with  $Te^{IV}$ . However they were not stripped into water along with  $Te^{IV}$ , and thus the developed separation procedure is quite selective for  $Te^{IV}$ .

Separation of Selenium from Co-extracting Ions: Since most of the ions studied were found to be co-extracted along with selenium, attempts were made to use different stripping solutions for the selective stripping of the co-extracted ions. Ions such as  $Hg^{II}$ ,  $Co^{II}$ ,  $Ni^{II}$ , and  $In^{III}$  can be quantitatively stripped by using 10 mL of a 1% solution of sodium acetate (Table 6). Ions like  $Fe^{III}$  and  $Sb^{III}$  could be stripped using 10 mL of a 1% solution of sodium oxalate and 10 mL of a 2% solution of tartarate was found to be adequate for the quantitative stripping of  $Cu^{II}$  and  $Zn^{II}$ .

Under the above conditions the stripping of selenium was found to be very negligible. Thus, suitable stripping solutions were used for the stripping of these ions when they were present in the sample matrix along with selenium, prior to the stripping of selenium using a hypobromite solution.

Analysis of Synthetic Mixtures: The validity of the developed procedures for the separation of selenium and tellurium was assessed by analyzing synthetic mixtures containing varying amounts of several ions and these elements after applying a sequential procedure, as described earlier. The results are furnished in Table 7, from which it is evident

Table 4. Choice of Electrolyte Solution for the Stripping of Se<sup>IV</sup> from the Organic Extract

Stripping solution used	% Stripped	
Water	Nil	
5 M Nitric acid	17.5	
1 M Hydrochloric acid	Nil	
5 M Hydrochloric acid	0.5	
2.5 M Sulfuric acid	0.2	
Hydrogen peroxide (30%)	22.2	
0.1 M Sodium acetate	0.01	
0.2 M Sodium thiosulfate	35.7	
0.2 M Citrate	35.8	
0.2 M Tartrate	Nil	
0.2 M EDTA	8.8	
5% Ammonium chloride	0.05	
8% Sodium sulfite	50.0	
5% Ammonium acetate	Nil	
5% Sodium fluoride	Nil	
2% Oxalate	Nil	
2% Thiourea	20.0	
0.1 M Sodium hydroxide	2.0	
1 M Sodium hydroxide	31.2	
5 M Sodium hydroxide	36.5	
Saturated bromine water (10 mL)	95.4	
8 mL of saturated bromine water + 2 mL of 1.5 M NaOH	99.2	

Each extraction was carried out using 100  $\mu g$  of Se<sup>IV</sup> from 0.3 M KSCN, 0.2% TTC and 2.7 M sulfuric acid with 10 mL of 1:1 cyclohexanol and toluene.

Table 5. Extraction and Stripping Behavior of Other Metal Ions with the Ion-Pair Complex of Te<sup>IV</sup>

Ions added (1 mg)	% Extracted	% Stripped <sup>a)</sup>	Method used
` ' '			
$Cu^{2+}$	Nil		ICP-AES
$Fe^{3+}$	0.01	_	**
$Zn^{2+}$	Nil	_	Radiometry
$Pb^{2+}$	Nil		ICP-AES
$Cd^{2+}$	Nil		Radiometry
$Al^{3+}$	Nil		ICP-AES
$Hg^{2+}$	92.3	Nil	Radiometry
$Co^{2+}$	Nil		**
Ni <sup>2+</sup>	Nil		ICP-AES
$SeO_3^{2-}$	Nil		Radiometry
Sb <sup>3+</sup>	50.5	Nil	,,
$AsO_3^{3-}$	45.4	Nil	ICP-AES

a) Water is used for stripping.

Each extraction was carried out using 100  $\mu$ g of Te<sup>IV</sup> from 2 M KBr, 0.2% TTC and 1 M sulfuric acid media with 10 mL of dichloromethane.

that the developed separation procedure works satisfactorily for the various mixtures.

Analysis of Copper Concentrate, Incineration Ash, and In–Sb Alloy Samples: The copper concentrate sample was brought into solution by treating it with concentrated nitric acid and 1:1 sulfuric acid.<sup>13)</sup> The incineration ash sample was treated with 5 mL of 40% hydrofluoric acid and 5 mL of nitric acid.<sup>14)</sup> The In–Sb alloy sample was brought on a treatment with 10 mL of concentrated hydrochloric acid followed by gentle heating.

Samples spiked with known amounts of Te<sup>IV</sup> and Se<sup>IV</sup> before decomposition was also brought into solution. Suitable solutions were subjected to separation and determination by following the procedures described earlier. Parallel studies were also carried out to determine the selenium and tellurium in the samples using ICP-AES. The results summarized in

Table 6. Extraction and Stripping Behavior of Other Metal Ions with the Ion-Pair Complex of Se<sup>IV</sup>

Ions added	% Extracted	% Stripped <sup>a)</sup>	Method used
(1 mg)			
Cu <sup>2+</sup>	98.7	98.0	ICP-AES
Fe <sup>3+</sup>	99.0	99.0	**
$Zn^{2+}$	67.2	98.0	Radiometry
$Pb^{2+}$	97.0	98.5	ICP-AES
$Cd^{2+}$	Nil		Radiometry
$Al^{3+}$	Nil		ICP-AES
$Hg^{2+}$	98.3	98.5	Radiometry
Co <sup>2+</sup>	1.0	95.0	"
Ni <sup>2+</sup>	2.0	92.0	ICP-AES
In <sup>3+</sup>	30.0	97.5	Radiometry
Sb <sup>3+</sup>	10.0	97.5	,,
$AsO_3^{3-}$	15.0	97.5	ICP-AES
$\text{TeO}_3^{2-}$	65.0	99.7	,,

a) Hypobromite solution was used for stripping.

Each extraction was carried out using 100  $\mu g$  of Se<sup>IV</sup> from 0.3 M KSCN, 0.2% TTC and 2.7 M sulfuric acid with 10 mL of 1:1 cyclohexanol and toluene.

Table 8 clearly indicate that the developed separation procedure works satisfactorily for these elements for a wide variety of samples.

#### Conclusion

The proposed extractive separation procedure is simple, rapid and suitable for the successive separation of Te<sup>IV</sup> and Se<sup>IV</sup>, not only from each other, but also from other elements. The procedure in conjunction with the methods developed for the determination of these elements provide a reliable means of analysis of samples in which they are present together. The extraction procedure developed for Te<sup>IV</sup> is highly selective, since only Hg<sup>II</sup>, Sb<sup>III</sup>, and As<sup>III</sup> were found to become coextracted. However, only tellurium from the organic layer could be stripped with water. Though many ions (viz. Cu<sup>II</sup>,

Table 7. Separation and Determination of Te<sup>IV</sup> and Se<sup>IV</sup> in Synthetic Mixtures

Composition of the synthetic	Amount found <sup>a)</sup> (µg)		Recovery (%)	
mixture (μg)	Te <sup>IV</sup>	Se <sup>IV</sup>	Te <sup>IV</sup>	Se <sup>IV</sup>
$Te^{IV} (100) + Se^{IV} (100) + Cu^{II} (500)$	$98.0 \pm 1.2$	$100.5 \pm 2.0$	98.0	100.5
$+ \text{Co}^{\text{II}} (500) + \text{Ni}^{\text{II}} (500)$ $\text{Te}^{\text{IV}} (100) + \text{Se}^{\text{IV}} (100) + \text{Hg}^{\text{II}}$	$99.5 \pm 1.5$	$99.5 \pm 2.5$	99.5	99.5
$(1000) + Pb^{II} (1000) + Fe^{III} (1000)$ $Te^{IV} (100) + Se^{IV} (100) + Fe^{III}$ $(1000) + Cu^{II} (1000) + Hg^{II} (1000)$	$98.5 \pm 1.2$	$101.0 \pm 1.5$	98.5	101.5
$Te^{IV} (100) + Se^{IV} (100) + Sb^{III} (500)$ + $As^{III} (500) + Fe^{III} (500)$	$99.0 \pm 1.4$	$98.0 \pm 2.5$	99.0	98.0

a) Average of three determinations.

Extractions were carried out using 100  $\mu g$  of Te<sup>IV</sup> from 2 M KBr, 0.2% TTC and 1 M sulfuric acid media with 10 mL of dichloromethane. Te<sup>IV</sup> was stripped back using 10 mL of water and analysed by the proposed method.

Extractions were carried out using 100  $\mu$ g of Se<sup>IV</sup> from 0.3 M KSCN, 0.2% TTC and 2.7 M sulfuric acid with 10 mL of 1:1 cyclohexanol and toluene. After washing the organic extract with 10 mL of 0.1 M sodium acetate solution twice, Se<sup>IV</sup> was stripped back using 10 mL of hypobromite solution.

Amount found  $(\mu g)^{b)} g^{-1}$ Amount added (µg) Recovery (%) Sample TeIV Te<sup>IV</sup> SeIV SeIV TeIV Se<sup>IV</sup> Copper  $172.5 \pm 4.0$  $60.5 \pm 1.5$ concentrates 500 500  $667.0 \pm 10.5$  $550.0 \pm 12.5$ 98.0 98.9 Incineration asha)  $43.3 \pm 1.0$ (CRM 176) 500 500  $506.0 \pm 9.0$  $535.0 \pm 10.0$ 101.2 98.3 In-Sb alloys  $35.0 \pm 1.5$ 500 500  $542.0 \pm 10.0$ 101.4 100.6  $503.0 \pm 12.0$ 

Table 8. Analysis of Specific Samples for the Extractive Separation Procedure

Amount of selenium and tellurium in microgram obtained by ICP-AES analysis.

Copper concentrate :

: Tellurium (170.0), Selenium (60.0).

In-Sb alloy

: Tellurium (36.5).

a) The certified value for selenium:  $41.5 \,\mu g \, g^{-1}$ . b) Average of three determinations.

Fe<sup>II</sup>, Pb<sup>II</sup>, Co<sup>II</sup>, Hg<sup>II</sup>, As<sup>III</sup>, Sb<sup>III</sup>, In<sup>III</sup>) were found to be coextracted along with Se<sup>IV</sup>, washing with a suitable stripping agent selectively stripped them without any loss of selenium. Thus, the selectivity of the method is superior compared to the existing solvent-extraction procedures involving the use of 2-mercaptobenzoxazole<sup>15)</sup> and 2-mercaptobenzothiazole.<sup>16)</sup>

The developed method is reliable, as can be seen from the complete agreement of the results observed for the analysis of various practical samples with ICP-AES. The satisfactory recovery of selenium and tellurium added to these samples also indicated that the developed method is reliable for the separation and concentration of selenium and tellurium from a solution of complex composition.

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