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Investigations of the behaviour of tellurium(IV) and selenium(IV) in ion-exchange chromatography

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Abstract

For the development of an on-column chromatographic method for the simultaneous separation and detection of selenium(IV) and tellurium(IV), their chromatographic behaviours on cation and anion exchangers as well as on multiphase cation- and anion-exchange/reversed-phase columns, have been investigated. The eluent medium consisted of diluted hydrochloric acid at different concentrations and flow-rates. The detection of selenium(IV) and tellurium(IV) was based on post-column derivatization, of the eluted species, with 1,1,3,3-tetramethylthiourea (TMTU). The TMTU concentration, pH and flow-rate was optimized in order to obtain the best sensitivity for both the analytes. The capacity factor value, k', as well as the charge of the species eluted on the different columns tested, have been obtained for all experimental conditions. It has been demonstrated that the on-column chromatographic separation between selenium(IV) and tellurium(IV) is achievable by exploiting their different elution patterns on ion exchangers. On the basis of peak symmetry and analyte resolution at different concentration ratios, the cation-exchange mode was chosen. Injecting 50 μl of samples, detection limits of 78 and 83 μg/l for tellurium and selenium, respectively were obtained. Concentrations lower than 10 μg/l could be determined by increasing the injection volume or by means of on-line preconcentration.

Keywords: Tellurium; Selenium

1. Introduction

In recent years, there has been increasing interest in trace determination of selenium (Se) and tellurium (Te). Both, from the point of view of environmental pollution, can be classified amongst very toxic and relatively accessible elements [1,2]. As for Se, the interest in its accurate determination is because of its dual role as an essential nutrient at low concentration levels and as a toxic substance at higher concentration levels. Recently, sensitive methods based on inductively coupled plasma mass spectrometry (ICP-MS) [3,4] and high-performance liquid chromatography (HPLC) after post-column derivatization [5,6] have been described for its determination. As for Te, its detection at low levels is of interest because of the formation of its alkyl derivatives [1,2]. Methods based on neutron activation analysis using extraction chromatography [7], thin-layer chromatography (TLC) [8] and spectrophotometry [9] have been described.

Se and Te occur together in environmental sam-

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ples and since their toxicity and biological activity, as well as their mobility in the environment depend on their oxidation states [10–13], their determination as total elements, using for instance the hydride generation (HG) method [14], has no meaning when speciation studies need to be performed. A few investigations are reported for the simultaneous determination of their oxidation state. Te and Se, which are situated in group VIA of the periodic system belong to the so-called metalloid elements. They can be divalent, tetravalent and hexavalent, and are generally present in aqueous solutions as anions: tellurite (TeO_3^{2-}) and selenite (SeO_3^{2-}); in hydrochloric acid (HCl) they are predominantly present as chloro-complex species [15]. Their analytical behaviour is very similar, and therefore their separation from each other is not an easy task [16]. To our knowledge, currently no on-column chromatographic method for their separation has been reported. The behaviour of aqueous solutions of components forming heteropolycompounds of Se(IV) and Te(IV) using paper chromatography has been studied and reported by Derkach and Kamerzan [17]. A spectrophotometric method based on the use of thioglycollic acid for the simultaneous determination of Se(IV) and Te(IV) has been described [18], but the sensitivity of this method is not sufficient for its general application to environmental samples. Other authors determined Se(IV) and Te(IV) by differential-pulse-polarography after the extraction of their diethyldithiocarbamates into ethyl acetate [19]. The calibration curves were linear over the concentration ranges 7.5–50 ng/ml for Se(IV) and 10–100 ng/ml for Te(IV) in the aqueous phase. The lower detection limit was 5 ng/ml for both analytes.

In this paper the feasibility of Te(IV) and Se(IV) separation by ion-exchange chromatography, using HCl as eluent, in columns which have different characteristics, such as cation-exchange (CS2, CS3, CS5, CS10) and anion-exchange (AS4, AS4A) columns as well as on multiphase cation-exchange/reversed-phase (PCX500) and anion-exchange/reversed-phase columns (PAX500), has been investigated. In all the different experimental conditions, for both the analytes, the capacity factor values, k', the charge of the species eluted and other figures of merit are reported.

2. Experimental

2.1. Instrumentation

A Dionex Model 4500I ion chromatographic system (Dionex, Sunnyvale, CA, USA) equipped with a pressurized post-column reagent delivery module (RDM) and a UV–Vis detector (VDM II, Dionex) was employed. The ion chromatograph was interfaced to a Dionex Autoion 450 for complete system control, data collection and reprocessing. For spectrophotometric measurements, a Varian (Australia) Cary-3 spectrophotometer was used.

The separations were carried out with IonPac CS2, CS3, CS5 and CS10 cation-exchange columns (250 mm×4.6 mm I.D.) and with IonPac AS4A and AS4 anion-exchange columns (250 mm×4.6 mm I.D.) used in conjunction with the guard columns IonPac CG2, CG3, CG5, CG10, AG4A and AG4 (50 mm×4.6 mm I.D.), respectively, and multiphase (ion-exchange and reversed-phase) OmniPac PCX-500 (cation-exchange and reversed-phase) and PAX-500 (anion-exchange and reversed-phase) columns (250 mm×4.6 mm I.D.).

The eluent and the post-column reagent solution (PCR) were mixed with the aid of a T-piece, positioned at the exit of the analytical column followed by a packed bed reaction coil of 100 μl volume.

Sample injections ranging from 50 μl to 500 μl were performed.

2.2. Reagents and standards

Hydrochloric acid (HCl), suprapur grade materials (Merck) and methanol for HPLC (Rieder de Alder) were used. 1,1,3,3,-Tetramethylthiourea (TMTU) was obtained from NovaChimica (Milan, Italy). Ultra pure water (18 MΩ/cm resistivity at 25°C) obtained by treating double-distilled water (Carlo Erba) in a UHQ-system (Elga, UK), was used throughout.

Working standards were prepared by serial daily dilution of stocking solutions, containing 1000 mg/l of Se(IV) or Bi(III) obtained from Merck. Te(IV) standard solution was prepared by dissolving 25 mg of TeO_2 (99.999%; Aldrich) in 50 ml of 10 *M* HCl

and diluting 4 ml of this solution to 100 ml with ultra pure water.

All standards, samples and reagents were prepared and stored in polyethylene containers previously cleaned and conditioned following a procedure for trace element determination [20].

2.3. Eluent solution

The eluent solution was diluted in HCl (2–220 m*M*) which was freshly prepared daily and used at a flow-rate of 1 ml/min. In the case of the Omnipac PCX-500 and PAX-500 columns, 5% methanol was also added.

2.4. Post-column reagent (PCR) solution

The PCR solution consisted of 1% (w/v) TMTU in 800 m*M* HCl. The PCR solution was prepared daily by dissolving 5 g of TMTU in 500 ml of 800 m*M* HCl. This solution was stable for at least 3 weeks if stored at 4°C or room temperature and not exposed to direct light.

The optimized PCR flow-rate was equal to 1.0 ml/min.

3. Results and discussion

3.1. Complexation of Te(IV) and Se(IV) with TMTU

TMTU is a complexing agent for Te(IV) and Se(IV) which forms an octahedral complex with TMTU in hydrochloric medium [15]:

$$TeO_2 + 4HCl + 2TMTU \rightarrow trans\text{-}Te^{IV}(TMTU)_2Cl_4 + 2H_2O \quad (1)$$

For Se(IV), the same reaction of complexation can be postulated.

In Fig. 1 the spectra obtained for Te(IV)–TMTU and Se(IV)–TMTU complexes measured against the blank reagent in HCl are shown. They both have the same pattern, and the maxima of the absorbance are located at 320 nm and 330 nm for the Se and Te complexes, respectively. In this investigation, therefore, the wavelength value of 325 nm was used.

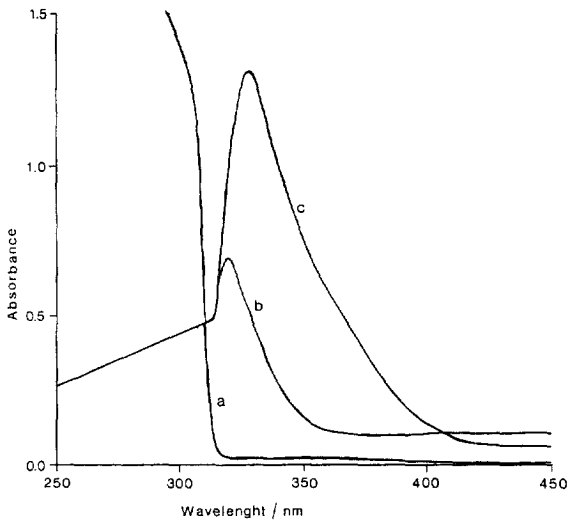

Fig. 1. Absorption spectra of Te(IV)–TMTU and Se(IV)–TMTU complexes vs. the blank reagent. (a) Blank reagent; (b) Se(IV)–TMTU complex, 5 mg/l of Se(IV); (c) Te(IV)–TMTU complexes, 20 mg/l of Te(IV).

3.2. Detection conditions

In order to optimize the conditions for the chromatographic detection, the effect of the concentration of HCl on the absorbance of the Te(IV)–TMTU and Se(IV)–TMTU complexes was studied varying the HCl concentration in the PCR solution from 200 m*M* to 1000 m*M*.

As for the absorbance of the Se(IV)–TMTU complex, no effect of the HCl concentration was observed. In contrast, for Te(IV) it was found that the absorbance of the Te(IV)–TMTU reached a maximum value for a concentration of HCl between 400–500 m*M*. In fact, stronger acidic solutions inhibit the complex formation and a concentration below 400 m*M* decreases the reaction rate [15].

In the present investigation, in order to obtain a 400–500 m*M* HCl concentration in the reaction coil where the eluent and the PCR solution are mixed with a 1:1 dilution factor, the PCR solution was prepared in 800 m*M* HCl.

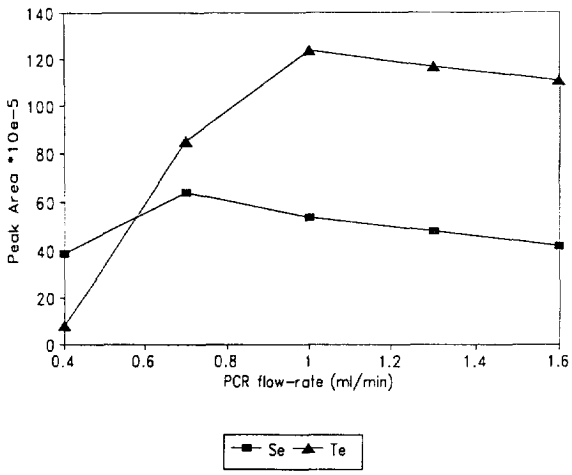

Fig. 2. Effect of PCR flow-rate on the chromatographic response (peak area) of 10 mg/l of Te(IV) and Se(IV). Injection volume 50 μl.

3.3. PCR and eluent flow-rate effects

The effects of the flow-rate of the PCR solution and of the eluent on the analytical response (peak area) of both the analytes were studied. The PCR flow-rate effect on the response of Se and Te while keeping both the eluent concentration (200 m*M*) and its flow-rate (1 ml/min) constant was investigated. As shown in Fig. 2 for Te, the highest response was obtained using a PCR flow-rate of 1.0 ml/min. For Se the maximum response was obtained for a PCR flow-rate of 0.7 ml/min. When using a flow-rate of 1.0 ml/min the decrease in the peak area of Se was 17%, while using a flow-rate of 0.7 ml/min the Te signal decreased by 46%, for their simultaneous determination a PCR flow-rate of 1.0 ml/min was chosen.

The eluent flow-rate effect on the response of selenium and tellurium while keeping the PCR flow-rate constant was also investigated. It was found that an increase and a decrease of the eluent flow-rate with respect to a flow-rate of 1.0 ml/min led to a decrease in the sensitivity of Te detection. This can be explained as being due to an inhibition in the complex formation when the acidity changes. Thus an eluent flow-rate of 1.0 ml/min was used in the present investigation.

3.4. Separation conditions

The separation of Te and Se in aqueous solutions as well as in HCl is complicated by their similar chemical behaviour [15,16,21].

In Table 1 the low- and high-capacity cation-exchange divinylbenzene–styrene sulphonated copolymer columns [22] and the latex agglomerated anion-exchange columns (where only the core substrate particle is sulphonated) [23] as well as on multiphase cation- and anion-exchange/reversed columns [24], tested using HCl as eluent, are reported. Characteristics such as the particle size of the packing material, the degree of crosslinking, the size of the latex particles and the hydrophobicity of the functional groups are different in each case.

As for cation-exchange, the five columns investigated were CS2, CS3, CS5, CS10 and PCX500. The elution pattern of Se and Te on these five columns was always the same. In fact, by keeping the

Table 1
Characteristics of the ion-exchange columns

Column	Capacity (μm/col)	Efficiency (*N*)	Particle size (μm)	Substrate % XL	Latex size (nm)	Latex % XL	Comments
CS2	60	1100	13.0	2.0	None		Not hydrophobic
CS3		3500	10.0		225	4.0	Hydrophilic
CS5		3000	13.0		100	2.0	Very hydrophilic
CS10	80		8.5	55.0	175	5.0	Moderately hydrophilic
PCX500	40		8.5	55.0	60	4.0	Hydrophilic
AS4		1700	15.0		100	3.5	Hydrophobic
AS4A		2000	16.0		200	0.5	Less hydrophobic than AS4
PAX500	120		8.5	55.0	200	5.0	Hydrophilic

N: plate number.
XL: cross-linking.

concentration and the flow-rate of the eluent constant, both the analytes have a retention time of around 2.1 min for selenium, and around 2.6 min for tellurium, independent of the characteristics of the column, the capacity of the exchangers being more or less similar.

In order to investigate the chromatographic behaviour of both the analytes under cation-exchange conditions, elutions on the above columns were performed varying the concentration of the HCl eluent solution in the range 2 to 220 m*M*. In Fig. 3 the typical behaviour of Se and Te on a cation exchanger, the CS10 column, for four different concentrations of HCl is shown. In all experimental conditions, Se peak eluted very close to the void volume of the columns, which, for 1.0 ml/min eluent flow-rate, corresponds to 2 min. In contrast, the retention time of the Te peak changed when the eluent concentration was varied. These results indicate that Se elutes as an anion species, i.e., is not retained on a cation exchanger, whereas Te behaves as a cation species.

In order to obtain more information on Se and Te chromatographic behaviour, also three different anion-exchange columns – AS4, AS4A and PAX500 – were tested and the k' values for both the analytes, under cation- and anion-exchange conditions, were calculated.

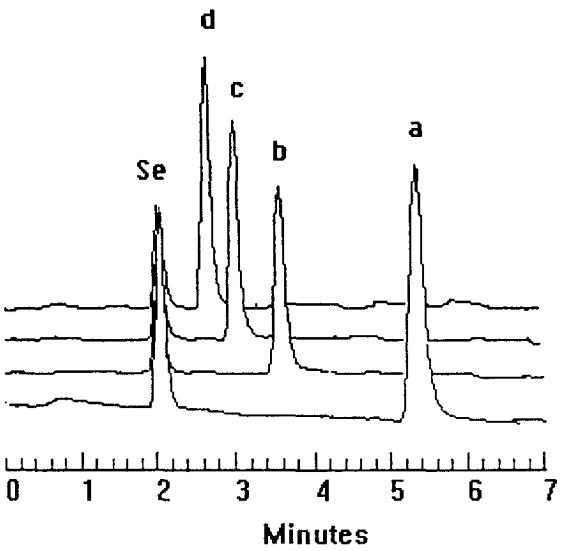

Fig. 3. Chromatographic behaviour of Se(IV) and Te(IV) on a CS10 column for different concentrations of HCl. (a) 50 m*M*; (b) 100 m*M*; (c) 150 m*M*; (d) 200 m*M*. 10 mg/l of Se and Te. Injection volume 50 μl. 0.2 AUFS. Eluent flow-rate 1.0 ml/min.

It is commonly accepted that, if an ion-exchange mechanism governs retention on the column, a dependence between the logarithm of the capacity factor and the logarithm of the eluent concentration is a straight line with a slope given by the ratio of charges of an analyte and an eluting ion. However, this dependence holds true only for simple monoionic eluents [25].

According to the following equation:

$$\log(k') = -(y/x) \times \log[\mathrm{E}] + cost. \quad (2)$$

(where k' is the capacity factor, y and x are the charge of the elute and the eluent, [E] is the eluent concentration and *cost.* is a constant) which is commonly used in ion chromatography investigations and which has been validated for many systems [26,27], the charge on the species which elute (eluite) was determined. This involved plotting the $\log(k')$ values vs. the $\log(E)$ and determining the slope values, y/x, where x is the charge on the species which acts as the eluent. It was taken into account that the eluent agent is the hydronium ion ($x = +1$) in cation-exchange conditions, and the chloride ion ($x = -1$) in anion-exchange conditions. In this investigation it is not necessary to consider activity effects in ion chromatography because of the dilute eluents and low sample concentrations which are routinely used. Moreover the inclusion of the activity factors would require knowledge of the activities of species in the resin phase, and these cannot be determined [26]. Notwithstanding these comments, there will be a number of occasions on which it will be necessary to reconsider activity effects.

In Table 2 the results obtained for Te and Se with all the columns tested are summarised.

3.5. Te(IV) behaviour

From the calculated charge for the Te species eluted, reported in Table 2, it appears that Te behaves in cation-exchange conditions as a monovalent cation. In anion-exchange conditions, Te elutes on AS4 and AS4A columns as a monovalent ion, which could be a cation species because it is

Table 2
Slope values (y/x), charge (y) of the eluted species and the correlation factors for Te(IV) and Se(IV) on different columns obtained by plotting the log(k') vs. log(E) according to Eq. (1)

Column	Tellurium			Selenium		
	$-y/x$	y	r	$-y/x$	y	r
CS2	1.14	1.14	−0.9993	Elution close to the void volume		
CS3	0.99	0.99	−0.9999	Elution close to the void volume		
CS5	1.16	1.16	−0.9998	Elution close to the void volume		
CS10	1.07	1.07	−0.9997	Elution close to the void volume		
PCX500	1.10	1.10	−0.9998	Elution on the void volume		
AS4	1.20	1.20	−0.9997	1.90	−1.90	−0.9998
AS4A	0.97	0.97	−0.9996	2.02	−2.02	−0.9997
PAX500	Elution on the void volume			2.00	−2.00	−0.9999

[E] varied over the range 2–200 mM.

unretained on PAX500 (Fig. 4). In all acidity conditions it elutes after 2 min.

The fact that also on AS4 and AS4A columns, Te(IV) can be partially retained, even though it behaves as a monovalent cation, is due, to some extent, to the presence on these separators of a

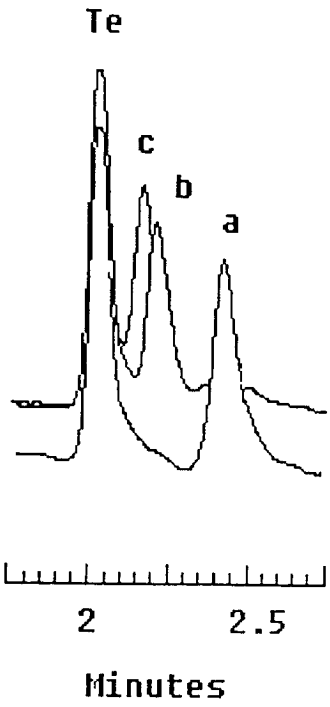

Fig. 4. Chromatographic behaviour of Se(IV) and Te(IV) on a PAX500 column for different concentrations of HCl. (a) 30 mM; (b) 40 mM; (c) 45 mM; (d) 200 mM. 10 mg/l of Se and Te. Injection volume 50 μl. 0.2 AUFS. Eluent flow-rate 1.0 ml/min.

percentage of cation-exchange sites. That Te(IV) is unretained on the PAX-500 column can be explained by considering the different structure of this multiphase separator in respect to the AS4 and AS4A columns. In fact, a new method for latex attachment was studied [24] for this multiphase column which has the advantage that there is no measurable residual cation-exchange capacity on the surface of the substrate beads. It was reported [24] that the residual cation-exchange capacity measured was less than 1 μequiv./column capacity for PAX-500, and equal to 11 μequiv./column capacity for a column of the same dimensions, made by traditional electrostatic agglomeration of aminated latex bead on PS/DVB core sulphonated surface such as AS4 and AS4A columns. All this can be explained by the presence of the following reaction in HCl:

$$TeCl_4 \rightarrow TeCl_3^+ + Cl^-$$

In fact, considering the stability constant values, K, for the species $TeCl_4$ and $TeCl_6^{2-}$ that have been reported [28] to be $K_4 = 9500$ and $K_6 = 4.5$ it can be assumed that the $TeCl_4$ species is the dominant one. Besides, studies on the crystallographic structure of $TeCl_4$ have evidenced [15,28] that it is constituted of $TeCl_3^-$ groups, explaining its electrical conductivity at the molten state [15]. Tetra halogen species or alkyl substituted halogen species give solutions that are conductive and, at the solid state can be ionic [15]. On this basis, it can be also assumed that the dissociation of $TeCl_4$ takes place in solution and that the monovalent cation $TeCl_3^+$ is the species which

elutes in hydrochloric medium during cation and/or anion-exchange mechanism.

3.6. Se(IV) behaviour

As reported in Table 2, in cation-exchange conditions, Se(IV) it is unretained and eluted very close to the void volume of all the cation-exchangers tested in the present work.

Under anion-exchange conditions, the charge value calculated for the Se species eluted on the AS4, AS4A and PAX-500 columns is −2 (Fig. 4), namely it elutes as the divalent ion, SeO_3^{2-} or $SeCl_6^{2-}$ which are present in aqueous solutions or in strong HCl, respectively. In fact, SeO_2 dissolves readily in water to give solutions which do contain seleneous acid with the $OSe(OH)_2$ structure; Raman spectra show that it is negligibly dissociated in aqueous solution while in half and fully neutralized solutions the ions $HSeO_3^-$ and SeO_3^{2-} are formed [17]. Since H_2SeO_3 dissolves in concentrated HCl, in the presence of KCl, giving K_2SeCl_6 [15], it might be supposed that the species formed in the present investigation is $SeCl_6^{2-}$. When the eluent concentration was higher than 50 m*M*, Se(IV) was almost weakly retained because it might be present as undissociated seleneous acid.

3.7. Linearity, detection limit, reproducibility and interferences

Comparing the results obtained on the different columns tested, analytical tests were performed on CS10 and CS3 columns which showed the highest efficiency and the lowest peak asymmetry, even though Se(IV) is eluted close to the void volume. Another choice might be the use of the AS4 or AS4A columns, where both the analytes are retained. But on these columns the Te peak was not very symmetric and it appeared to be even worse when real water natural samples were injected. Besides, the retention times of both the analytes where close to each other not allowing a good separation for varying concentration ratios. This was also indicated by the selectivity coefficients α, obtained as ratio of capacity factors of the two solutes in the different experimental conditions. In Table 3, as an example, the α coefficients are reported for the elution performed with 100 m*M* HCl, as can be seen the best selectivity and hence resolution is obtainable using the CS10, CS5 or the CS3 columns. On this basis, the cation-exchange mode was preferred. CS5 was not employed further due to its mixed anion- cation-exchange characteristics.

Table 3
Selectivity coefficient α (obtained as ratio of the capacity factors of Te and Se) for the elution of Te(IV) and Se(IV) with 100 m*M* HCl on cation and anion exchangers

Column	α
CS2	20
CS3	70
CS5	75
CS10	86
AS4	13
AS4A	0.6

For the columns PCX500 and PAX500 the α value could not calculated due to non-retention of Te and Se, respectively.

Using a PCR flow-rate of 1.0 ml/min and the 200 m*M* HCl as the eluting agent on CS10 and CS3 columns, excellent linearity ($r = -0.99998$) over 2 orders of magnitude from 100 to 10 000 μg/l of Te and Se, injecting 50 μl sample volumes was obtained. The detection limits, defined as three times the standard deviation (3σ) was calculated to be 78 and 83 μg/l for Te and Se, respectively, injecting 50 μl. Concentrations lower than 10 μg/l can be determined by increasing the injection volume, or by means of the on-line preconcentration technique.

In Fig. 5 a chromatogram obtained for a natural water sample, using a CS3 column is shown. As can be seen the separation between the two analytes, as well as their peak shape is pretty good.

The data on the reproducibility of the peak area and the retention time were obtained by performing ten injections of 50 μl of 10 mg/l Te and Se standards on four separate occasions. The relative standard deviations (R.S.D.s) for the peak area varied from 0.45 to 1.03% for Te and from 0.63 to 0.97% for Se. The R.S.D.s for the retention time varied from 0.16 up to 0.38% and from 0.13 to 0.43% for Te and Se, respectively. Thus, both peak area and retention time data were indicative of a stable and reproducible system.

As for the interferents, among the different ele-

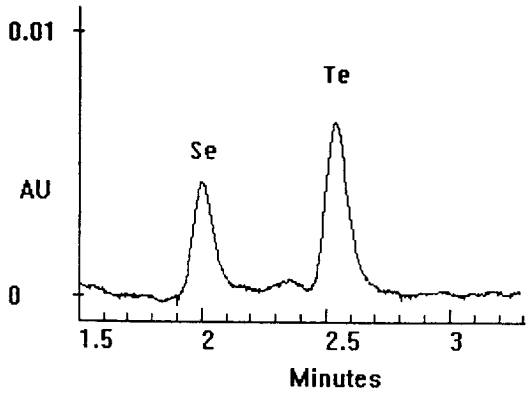

Fig. 5. Elution on CS3 column of a natural water sample containing 45 μg/l of Se(IV) and 25 μg/l of Te(IV). Injection volume 200 μl. Eluent flow-rate 1.0 ml/min.

ments tested, only Bi(III) formed a complex with TMTU which adsorbs at the same wavelength as the Se(IV)–TMTU and Te(IV)–TMTU complexes. Tests performed in these investigation showed that Bi(III) was eluted, by 200 m*M* HCl at a flow-rate of 1.0 ml/min, only after about 45 min. Therefore Bi(III) does not interfere with Se and Te determination.

4. Conclusions

The separation of Se(IV) and Te(IV) has been obtained exploiting their different ion-exchange chromatographic behaviours. The tests performed demonstrated that Se(IV) always behaves as a divalent anion and therefore on cationic phases it is not retained and elutes close to the void volume of the columns. Te(IV) behaves as a monovalent cationic species, as demonstrated by its elution pattern according to the eluent concentrations and by its elution on the void volume of the PAX500 that is a fully anionic separator. The chromatographic separation coupled with the spectrophotometric detection employed, provide a very simple method for the determination of Se(IV) and Te(IV) in natural water samples. In particular, using CS3 or CS10 for the chromatographic separation, detection limits of 78 and 83 μg/l for Te and Se, injecting 50 μl of sample, have been obtained.

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