Speciation of Antimony Using Chromosorb 102 Resin as a Retention Medium

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The selective retention of the Sb(III) chelate with ammonium pyrrolidine dithiocarbamate (APDC) on a column of Chromosorb 102 resin from a buffered sample solution including Sb(V) was used for the determination of Sb(III). The retained antimony was eluted with acetone. The retention of the Sb(III)-iodide compounds with sodium iodide on the Chromosorb 102 resin column from the same solution after reducing Sb(V) to Sb(III) by iodide in acidic solution was used to preconcentrate the total antimony. The retained antimony was eluted with 0.25 mol l^{-1} HNO_3. The antimony in the effluent was determined by flame atomic-absorption spectrometry. Also, the total antimony was determined directly by graphite-furnace atomic absorption spectrometry. The Sb(V) concentration could be calculated by the difference. The recoveries were ≥95%. The detection limits of a combination of the column procedure and flame AAS for antimony were 6 – 61 µg l^{-1} and comparable to 4 µg l^{-1} for a direct GFAAS measurement. The relative standard deviations were <6%. The procedure was applied to the determination of Sb(III) and Sb(V) in spiked tap water, waste-water samples and a certified copper metal with the satisfactory results.

Introduction

There is increasing demand to establish a speciation procedure for antimony in environmental samples. The importance of antimony speciation can be explained by the fact that the toxicity, bioavailability and physiological effects of antimony depend on the oxidation state. The toxicity of Sb(III) is ten-times higher than that of Sb(V).[1-3] Inorganic species of antimony are more toxic than that of organic compounds.[4] Therefore, the selective determination of Sb(III) and Sb(V) ions is very important in environmental samples, such as various water, soil and sediment samples.

The concentration level of total antimony in various environmental samples is rather low. Therefore, a selective preconcentration step is usually necessary prior to speciation and the determination of antimony species. Until now, the selective preconcentration of Sb(III) and Sb(V) has been based on solvent extraction,[5-7] coprecipitation,[8,9] ion exchange,[10] solid-phase extraction[11-15] *etc*. In comparison with each other, solid phase extraction has been proven to be a particularly useful method to determine trace amounts of metals and to improve the detection limits. Especially, solid-phase extraction based on a column technique allows preconcentration from a large sample volume, thus establishing a higher concentration factor, high recovery and low consumption of organic solvents. Different solid adsorbents, such as alumina,[11] activated carbon[16,17] and some resins,[14,15] have been successfully applied for the preconcentration of antimony species.

In our previous study, a column solid-phase extraction procedure was developed for the preconcentration of some trace heavy metal ions as their carbamate chelates with APDC from various water samples and sediments using a Chromosorb 102 resin column prior to their measurements by AAS.[18] Also, the use of a Chromosorb 102 resin column for trace preconcentration had been initiated by us.[19] This was later confirmed to be the first study by others.[20] APDC is the complexing agent most widely used for the selective preconcentration of antimony(III) using solvent extraction[21] and solid-phase extraction.[13,22] On the other hand, the solvent extraction of antimony as iodide compounds is well documented in the literature.[23] However, to the best of our knowledge, there is no report about the preconcentration of antimony based on the retention of Sb(III)-APDC and Sb(III)-iodide compounds through a Chromosorb 102 resin column.

The purpose of the present study was to explore the feasibility of Chromosorb 102 resin as a solid-phase extractant for the speciation of inorganic Sb(III) and Sb(V) species using APDC and sodium iodide in various samples, such as tap water, waste water and metal solution samples. The determination of antimony concentrations was performed by flame atomic-absorption spectrometry (FAAS) and graphite atomic-absorption spectrometry (GFAAS).

Experimental

Apparatus

Trace concentrations of Sb were determined by a Perkin Elmer Model 3110 atomic-absorption spectrometer equipped with an antimony hollow-cathode lamp. The operating conditions for antimony were: wavelength, 231.2 nm; band pass, 0.7 nm; lamp current, 14 mA; flame, 11.6 dm³ min⁻¹ air

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and acetylene 3.8 dm³ min⁻¹. The lower concentrations of antimony were determined using a Perkin Elmer atomic-absorption spectrometer (Model Analyst 100) equipped with a graphite tube atomizer (Model HGA-800, Perkin Elmer) and a Deuterium background corrector. The injection volume was 20 μl for GFAAS.

All pH measurements were carried out using a Nel pH 900 digital pH meter equipped with a combined glass-calomel electrode.

Reagent and solutions

All reagents were of analytical reagent grade unless otherwise stated, and doubly distilled, deionized water was used throughout the experiments.

A stock standard Sb(III) solution (1000 μg cm⁻³) was prepared by dissolving 0.2736 of potassium antimonyl tartarate (Merck) in water and diluting to 100.0 cm³. A stock standard Sb(V) solution (1000 μg cm⁻³) was prepared by dissolving 0.2160 g of potassium pyroantimonate (Merck) in water and diluting to 100.0 cm³. Working solutions for Sb(III) and Sb(V) ions were prepared daily by appropriate dilution of the stock standard solution. A palladium solution (0.5 mg cm⁻³ Pd) as matrix modifier was prepared by diluting a 1000 mg cm⁻³ Pd AAS standard solution.

The following buffer solutions were used for the preconcentration procedure: KCl/HCl buffer for pH 1; PO_4^{3-}/HPO_4^- buffer for pH 2 and 3; CH_3COO^-/CH_3COOOH buffer for pH 4 – 6; borate buffer for pH 7; NH_3/NH_4^+ buffer for pH 8 – 10.

NaI solutions with different concentration were prepared by solid NaI (Merck). The ligand APDC (Sigma) was used as 0.05% (m/v) solution in water. As the means of elution, acetone for Sb-APDC chelate and 0.25 mol dm⁻³ HNO₃ for Sb-iodide compounds were used.

Chromosorb 102 resin (Phase Separations Inc., Norwalk, CT) was used as solid-phase extractant. It is a porous styrene-divinylbenzene copolymer having a surface area in the range of 300 – 400 m² g⁻¹, 80 – 100 mesh particle size and *ca*. 0.075 – 0.060 mm pore size.

Method development

The stopcock of a glass column (100 × 10 mm) was covered with a fritted glass disk. 500 mg of Chromosorb 102 resin was slurried in water, and then poured into the column. A small amount of glass wool was placed on the disk to prevent any loss of the resin beads during sample loading. The bed height of resin in the column was approximately 1.5 cm. It was washed successively with water, acetone and water, respectively. The column was preconditioned by passing a blank solution prior to use. After each use, the resin in the column was washed with large volumes of water and stored in water for the next experiment.

The performance of column method was tested with synthetic solutions before its application to samples.

For the preconcentration of total antimony, the defined amount of NaI as a reducing agent to reduce Sb(V) and as a complexing agent was added to 50 cm³ of synthetic antimony solutions, including Sb(III) and Sb(V), in changing the concentration from 50 μg dm⁻³ to 500 μg dm⁻³ and 0.1 mol dm⁻³ HCl. The NaI and HCl concentrations in these synthetic solutions were 0.5 mol dm⁻³ and 0.1 mol dm⁻³, respectively. The solutions were loaded to the top of the preconditioned column and gravitationally passed at a flow rate of 2 cm³ min⁻¹. The column was washed with 5 – 10 cm³ of a blank solution prepared with the corresponding agent and acid solution. The retained total antimony was eluted with 10 cm³ of 0.25 mol dm⁻³

HNO₃. The effluent was evaporated to near dryness on a hot plate. The residues were then diluted to 1.0 and 5.0 cm³ using 1 mol dm⁻³ HNO₃. Antimony in these final solutions was determined by a flame atomic-absorption spectrometer.

For the selective preconcentration of antimony(III) in presence of Sb(V), 3.0 cm³ of a 0.05% (m/v) APDC solution as a chelating agent was added to synthetic antimony solutions including Sb(III) and Sb(V) in the changing concentration. Then, the sample solutions were buffered to pH 6 using CH₃COOH/CH₃COONH₄. The solution was passed gravitationally through the column at a flow rate of about 2 cm³ min⁻¹. The column was washed with 5 – 10 cm³ of a blank solution prepared with the corresponding buffer solution. The retained antimony-carbamate complex was then eluted with 10 cm³ of acetone. The effluent was evaporated to dryness on a hot plate adjusted *ca*. 30 – 40°C and then cooled. The residue was diluted to 1.0 and 5.0 cm³ using 1 mol dm⁻³ HNO₃. Antimony in these final solutions was determined by a flame atomic-absorption spectrometer.

The Sb(V) concentration was calculated as the difference between the total antimony and Sb(III) concentrations found from the same volume of the sample and synthetic solutions.

Analysis of water and metallic copper samples

A drinking tap-water sample taken from our research laboratory was analyzed without any pretreatment. Waste-water samples were collected in pre-washed polyethylene bottles from the waste-water pools of two textile factories and an enamaleware plant. The bottles were cleaned with a detergent, double-distilled deionized water, dilute nitric acid and double-distilled-deionized water, in sequence. The samples after taking were immediately filtered through a Millipore cellulose nitrate membrane having a pore size 0.45 μm (Sartorius GmbH, Germany) and stored in pre-cleaned polyethylene bottles. The determinations of antimony in the samples were performed within three days. The preconcentration procedures given above were submitted to 600 cm³ of the drinking water for Sb(III) and 100 cm³ of the water sample for total antimony. The concentration of Sb(V) was calculated as the difference between the total antimony and the determined Sb(III) concentrations.

The total antimony in a waste water sample was also determined using graphite-furnace atomic absorption spectrometry. For this, 40 μl of a palladium solution as a matrix modifier was added to 2 cm³ of a water sample. Then, 20 μl of a sample including Pd was injected manually into a tube atomizer. The peak area of AAS was measured under the optimum conditions, such as drying at 80 – 120°C (10 s ramp) and 120°C (30 s hold); ashing at 120 – 1100°C (10 s ramp) and 1100°C (20 s hold) and atomizing at 1900°C (5 s hold).[24]

For copper, a portion (40 mg) of the certified copper sample (CRM Cu 1/3 Cu from Chine) was accurately weighed into a 50 cm³ beaker and *ca*. 0.5 cm³ of concentrated nitric acid was added to the sample. The beaker was covered with a watch-glass and the mixture was evaporated on a hot plate at about 95°C almost to dryness. The sample solution was then diluted to 10 cm³ with distilled water. The total antimony in this final solution was determined by the the procedure given above for the total antimony preconcentration.

Results and Discussion

Influence of some parameters on the preconcentration of total antimony

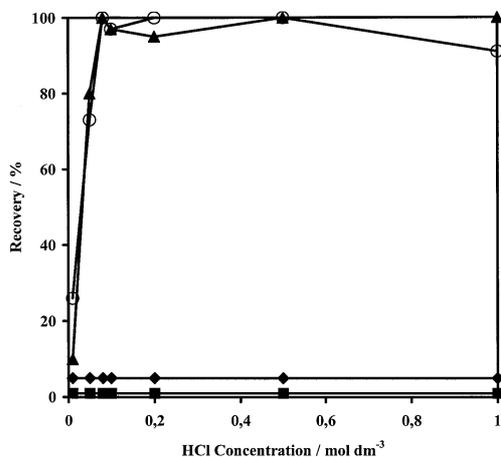It is well known that iodide ion reduces Sb(V) to Sb(III) in

Fig. 1   Effect of the HCl concentration on the recovery of Sb(III) (◆) and Sb(V) (■) from an aqueous solution; of Sb(III) (▲) and Sb(V) (○) from 0.5 mol dm^{-3} NaI solution ($n = 3$).

Fig. 2   Effect of the NaI concentration on the recovery of Sb(III) (◆) and Sb(V) (■) from an aqueous solution; of Sb(III)(▲) and Sb(V) (○) from 0.1 mol dm^{-3} HCl solution ($n = 3$).

acidic media and forms stable complexes with Sb(III), such as tetraiodoantimonate(III) ion.[2,17,23,25]   Thus, the influence of the acidity and the iodide concentration as critical parameters in achieving quantitative retention of Sb(III) and Sb(V) using NaI were investigated.   In order to optimize the retention conditions for Sb(III) and Sb(V), solutions prepared with increasing HCl concentrations from 0.01 to 1.0 mol dm^{-3} in the presence of 0.5 mol dm^{-3} NaI were submitted to the column procedure.   As shown in Fig. 1, Sb(III) and Sb(V) after 0.08 mol dm^{-3} HCl were quantitatively recovered.   However, the retentions of Sb(III) and Sb(V) in the absence of NaI were almost impossible. On the other hand, as can be seen from the recovery-NaI concentration profiles for Sb(III) and Sb(V) given in Fig. 2, Sb(III) and Sb(V) were quantitatively recovered from 0.4 – 1.0 mol dm^{-3} NaI solutions prepared in the presence of 0.1 mol dm^{-3} HCl.   Even in the absence of HCl, the recovery values increase *ca*. 40% with increasing concentration of NaI until 2 mol dm^{-3}. However, after 1.0 mol dm^{-3} NaI concentration in the presence of HCl, the recovery values of Sb(III) and Sb(V) decreased with increasing concentration of NaI.   As a result, the total antimony, Sb(III) plus Sb(V), could be concentrated from a 0.5 mol dm^{-3} NaI solution prepared in 0.1 mol dm^{-3} HCl.

As can be seen from Figs. 1 and 2, the retention behaviors of Sb(III) and Sb(V) are the almost same.   For this reason it might be concluded that Sb(V) is reduced by iodide to Sb(III) in an acidic solution, and then retained as Sb(III)-iodide compounds. In light of the information given above, antimony is probably retained as an iodo complex, HSbI$_4$.   It can be concluded that the adsorption might be due to the formation of ϕ-H$^+$SbI$_4^-$ from a styrene-divinylbenzene network of Chromosorb 102 resin and the iodo complex, using of general comments given by Koshima.[15]   Also, it may be partly retained as SbI$_3$ present in the solution, due to the complex equilibrium reaction.

The retention of the analyte compound through a column filled with a low amount of resin is low due to the low adsorption capacity.   In contrast, if the amount of resin increases, the recovery is not quantitative due to an insufficient eluent volume.   Also, smaller preconcentration factor is obtained with increasing the eluent volume.   To increase the preconcentration factor, the sample volume can be increased. Thus, the resin amount and sample volume must be optimized. The influence of the Chromosorb 102 resin amounts on the recovery of the total antimony as iodide compounds was
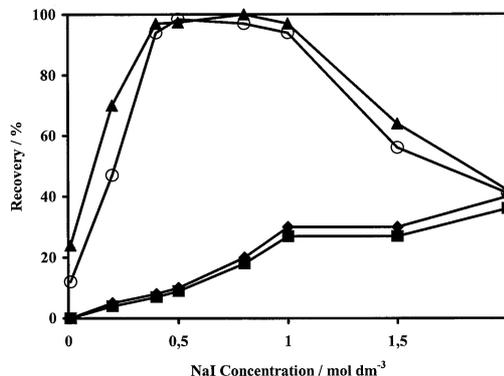
investigated ranging from 300 to 700 mg.   A quantitative recovery was obtained in the range of 400 – 600 mg. Accordingly, this result, the column was filled with 500 mg Chromosorb 102 resin.   The preconcentration of total antimony for different sample volumes was examined by following of the purposed procedure.   Sb(III) and Sb(V) as iodide compounds were quantitatively recovered up to 125 cm^3.   The preconcentration factor was found to be 62.5 for 2.0 cm^3 of the effluent volume.

The flow rate of the sample solution through the column is a very important parameter.   Because it is not only the quantitative retention, but also controls the time of preconcentration.   Thus, the effect of the sample flow rate on the efficiency of the total antimony retention on the Chromosorb 102 resin column was examined for flow rates ranging from 2 to 12 cm^3 min^{-1}.   It was found that the flow rate of the sample solutions had no influence over a flow-rate range of 2 – 4 cm^3 min^{-1} on the retention of the total antimony as iodide compounds.   At flow rates greater than 4 cm^3 min^{-1}, the decreases in the retention can be interpreted as incomplete retention of the total antimony resulting from slow sorption; 2 cm^3 min^{-1} was chosen as the flow rate of the sample solutions in subsequent experiments.

The effect of eluent types on the recovery of the total antimony as iodide compounds was investigated using various eluting solutions.   The quantitative recoveries for the total antimony were obtained with 10 cm^3 of 1 mol dm^{-3} HCl, 0.25 mol dm^{-3} HNO$_3$ and pure acetone.   To obtain a lower blank value, 0.25 mol dm^{-3} HNO$_3$ as the eluent was preferred because of more diluting solution.

Another study showed that varying the eluent flow rates over the range 1 to 5 cm^3 min^{-1} did not significantly influence the elution of the antimony species.   Therefore, an eluent flow rate of 2 cm^3 min^{-1} was chosen for the elution of the total antimony in further experiments.

Influence of some parameters on the selective preconcentration of antimony(III)

The influence of the pH on the selective preconcentration of Sb(III) as the carbamate chelate with APDC was examined using synthetic sample solutions buffered to different pH values.   For the quantitative retention of Sb(III), the optimum range of the sample pH was found to be 3 – 8 (Fig 3).   The recovery for Sb(V) in the same pH range was lower than 5%, because Sb(V) does not form a carbamate complex with APDC and is not retained through a resin column.   Due to these, pH 6
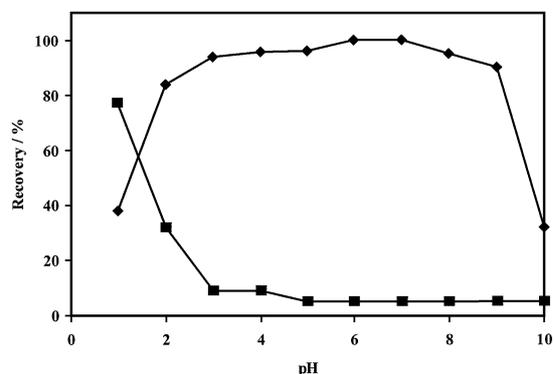
Fig. 3 Influence of the pH on the recovery of Sb(III) (◆) and Sb(V) (■) ($n = 3$).

Table 1 Effect of diverse ions on the recoveries of the total Sb and Sb(III)-APDC (Sb: 25 μg, volume: 50 cm³, $n = 3$)

Ion	Concentration/mg dm⁻³	Recovery, %	
		Total Sb	Sb(III)
Na^+	75000	100	95
Mg^{2+}	5000	101	104
Ca^{2+}	2500	96	100
K^+	2500	104	100
Fe^{2+}	250	100	—[a]
Fe^{3+}	250	50	22
Ni^{2+}	250	100	10
Pb^{2+}	100	70	22
Pb^{2+}	250	50	20
Cr^{3+}	250	100	100
$Cr_2O_7^{2-}$	250	65	<10
Cu^{2+}	100	71	15
Cu^{2+}	250	42	<10
Zn^{2+}	250	95	22
As^{3+}	250	94	100
Cd^{2+}	250	95	12
Cl^-	50000	100	100
SO_4^{2-}	100000	100	100
PO_4^{3-}	250	92	100
$C_2O_4^{2-}$	250	31	100
SCN^-	250	100	98
Citrate	250	31	100
Tartrate	250	46	100
EDTA	250	100	100
Ascorbic acid	20000	100	—[a]

a. Not examined.

was used for the selective preconcentration of Sb(III).

To test the resin amount for the quantitative retention of Sb(III) with APDC, different amounts of Chromosorb 102 from 300 to 700 mg were filled into the column. The procedure was applied to synthetic solutions using these columns. The quantitative recoveries for Sb(III) were obtained over the range of 400 – 600 mg resin. Quantitative retention was not obtained by a smaller amount of resin than 400 mg. On the other hand, a larger resin amount than 600 mg prevented the elution of the quantitatively retained chelates by 10 cm³ of acetone. According to this result, the column was filled with 500 mg Chromosorb 102 resin for use in further experiments.

To deal with real samples containing very low concentrations of the antimony ions, the maximum applicable sample volume must be determined. The effect of sample volume for the selective preconcentration of Sb(III) was tested by the proposed column procedures given in the experimental section. It was found that the recovery of Sb(III) as Sb-APDC chelate was quantitative up to 1000 cm³. Hence, the preconcentration factor for Sb(III) was found to be 500 with 2.0 cm³ of the effluent volume.

As explained above, the influence of the flow rate for the sample solution as an important parameter on the retention of Sb(III) through the Chromosorb 102 resin column was investigated over the range of 2 – 12 cm³ min⁻¹. It was found that the quantitative retention of Sb(III) as the chelate with APDC was independent of the flow rates from 2 to 12 cm³ min⁻¹. As expected, this result shows that the adsorption of Sb(III)-APDC complex is more rapid kinetic than that of Sb-iodide compounds. Because Sb(III)-iodo is retained at relatively slow rates at a maximum of 4 cm³ min⁻¹. 2 cm³ min⁻¹ was chosen as the flow rate of the sample solutions in subsequent experiments.

The ligand concentration plays an important role in the retention of the analytes, because in their absence the resin does not completely retain the metal ions. However, excess ligand prevents the retentions, of metal ions due to competitive sorption between the metal-ligand complex and the ligand on the resin. This is particularly important if the ligand is a chelating agent because of its hydrophobic groups, which are effective on sorption. The influence of APDC concentration on the retention of Sb(III) was investigated over the range of 0 to 1.2×10^{-3} mol dm⁻³ APDC. The retention of Sb(III) with increasing concentration of APDC increased from 5% to 95% up to 3.6×10^{-4} mol dm⁻³ APDC. It was found that Sb was quantitatively retained over the range of 3.6×10^{-4} to 1.2×10^{-3} mol dm⁻³ APDC.

To obtain a quantitative recovery, the effect of the eluent types on the recovery of Sb(III)-APDC chelate from the column was investigated using various eluting solutions. The recovery values were under 5% with 10 cm³ of 1 mol dm⁻³ HCl and 1 mol dm⁻³ HNO₃. Sb(III)-APDC chelate was quantitatively recovered with 10 cm³ of pure acetone and 1 mol dm⁻³ HNO₃ in acetone. Acetone as an eluent was preferred in order to obtain a lower blank value.

Another study showed that varying the eluent flow rates over the range of 1 to 10 cm³ min⁻¹ did not significantly influence the elution of the antimony species. Therefore, an eluent flow rate of 2 cm³ min⁻¹ was chosen for the elution of Sb(III)-APDC chelate in further experiments.

Influence of foreign ions

Potential interferences of commonly occuring foreign ions on determination of total and selective Sb(III) in different samples were evaluated (Table 1). No interference was found from alkaline and alkaline earth metal ions over the investigated concentration range, since these ions do not react with iodide or APDC. Also, there was no interference in the presence of sulfate, chloride, thiocyanide and phosphate ions at high concentrations.

Interferences were found in the presence of Fe(III), Cu(II), Pb(II) and Cr(VI) in the preconcentration of the total antimony. It is well known that iodide ion reduces Fe(III) to Fe(II), Cu(II) to Cu(I) and Cr(VI) to Cr(III) in acidic solution.[26,27] In addition, Cu(I) and Pb(II) with iodide form insoluble precipitates, such as CuI and PbI₂.[26] However, CuI and PbI₂ in the presence of excess iodide dissolve by forming of soluble complexes, such as CuI₂⁻ and PbI₃⁻.

When the optimized amount of sodium iodide (0.5 mol dm⁻³

Table 2   Determination of Sb(III) and total antimony in spiked tap water and waste-water samples ($n = 3$, sample volume 50 cm³)

Sample	Added/μg dm⁻³		Found/μg dm⁻³		Recovery, %	
	Sb(III)	Total Sb	Sb(III)	Total Sb	Sb(III)	Total Sb
Tap water	0	0	0	0	—	—
	100	200	100	207.0	100	103
	200	300	200	295.0	100	98
Waste-water	0	0	0	20.7	—	—
	100	100	96	116.7	96	96
	200	200	200	210.3	100	95

NaI) was added as a complex agent to the antimony solution in the presence of these ions, antimony was not completely recovered because of the iodide deficiency. For this reason, the interferences of Cu(II) and Pb(II) were eliminated due to the formation of their soluble complexes in the presence of excess iodide. Iron(III) interference was not eliminated using EDTA, SCN⁻ and phosphate having no interference on the retention of the Sb(III)-iodo complex. However, the interference of Fe(III) was eliminated by reducing Fe(III) to Fe(II) with ascorbic acid. Chromium(VI) interference was also prevented in the presence of ascorbic acid. The interference of oxalate, citrate and tartarate may be explained by more stable complex formation.[23]

The interferences of Cu(II), Fe(III), Ni(II), Pb(II), Cd(II) and Zn(II) on preconcentration of Sb(III) by APDC were eliminated by EDTA. Sb(III) was not completely recovered in the presence of $Cr_2O_7^{2-}$. However, the effect of Cr(VI) on the retention of Sb(III)-APDC was prevented using ascorbic acid.

The results given above suggest that the proposed method should be suitable for the determination of total antimony and Sb(III) in various samples having different complex matrices, such as waste-water and some pure metal solutions.

Analytical performance of the proposed procedures

Recovery tests were performed for 100 cm³ of tap water and waste-water samples. The results are shown in Table 2. Good agreement was obtained between the recovery of analyte for spiked and control samples using the experimental procedures for Sb(III) and total Sb. The recovery values calculated for the standard additions were quantitative, thus confirming the accuracy of the procedure and the absence of any matrix effects. Also, the accuracy for total antimony was tested by the direct determination of total antimony in waste-water using GFAAS. As can be seen from the results given in the application, the total antimony concentrations found with GFAAS and the preconcentration are in good agreement with each other.

The validation method for the total antimony determination was checked with the analysis of a standard reference material (CRM Cu I/III copper). The copper sample was dissolved according to a procedure given in the experimental section. The solid-phase extraction procedure for the total antimony was applied to a copper solution. The antimony content was found to be 0.020 ± 0.001 as the mean plus standard deviation (n: 7, m/m%). The relative standard deviation (RSD) was found to be 5%. Good agreement was obtained between the estimated content by the proposed method and the certified value, 0.020 m/m%, for Sb. Also, this result indicates that the developed column solid-phase extraction method for Sb is not affected by copper until 4000 μg cm⁻³ Cu.

The limits of detections for the total antimony and Sb(III) determinations by a combination of the preconcentration

procedures and flame AAS were 61 and 6 μg dm⁻³, respectively, based on three times (n: 10) the standard deviation of blanks. The detection limit found by a GFAAS measurement was 4 μg dm⁻³.

Application

To evaluate the usefulness of the proposed method, it was applied to an analysis of the total antimony drinking water and waste-water samples. It was found that the concentrations of Sb(III) and the total Sb in drinking water and waste-water taken from textile factories were lower than the detection limits. The mean and standard deviation of the concentration of total antimony in a waste-water sample taken from an enamelware factory was found to be 20.7 ± 0.9 μg dm⁻³ (n: 5, RSD: 4.3%) by applying of a combination of the preconcentration procedure and flame AAS. The total antimony content of the same waste-water sample was found to be 21.4 ± 1.2 μg dm⁻³ (n: 5, RSD: 5.6%) by a direct GFAAS determination. There was good agreement between two concentrations.

In conclusion, the results obtained in this work indicate that Chromosorb 102 resin can be used as an effective solid-phase extractant for the preconcentration of total antimony as Sb(III)+Sb(V) by sodium iodide and of Sb(III) by APDC from various water samples and high-salinity solutions. The concentration of Sb(V) was calculated based on the difference for its speciation. There is no need to use an additive complexing agent to determine the total antimony and to obtain speciation information. This avoids the risk of contamination and improves the detection limits. The detection limits, particularly for Sb(III), obtained with the column preconcentration procedures are comparable with that of GFAAS measurements.

References

1. T. D. Luckey and B. Venugopal, "*Metal Toxicity in Mammals: Physiologic and Chemical Bases for Metal Toxicity*", **1977**, Plenum Press, New York.
2. H. B. Hou and H. Narasaki, *Anal. Sci.*, **1999**, *11*, 159.
3. K. L. Stemmer, *Pharm. Ther. A*, **1976**, *11*, 57.
4. P. Smichowski, Y. Madrid, and C. Camara, *Fresenius J. Anal. Chem.*, **1998**, *360*, 623.
5. T. Kamada and Y. Yamamoto, *Talanta*, **1977**, *24*, 330.
6. K. Subramarian and J. C. Meranguer, *Anal. Chim. Acta*, **1981**, *124*, 131.
7. A. Menen Garcia, M. C. Perez Rodriguez, J. E. Sanchez-Uria, and A. Sanz-Medel, *Fresenius J. Anal. Chem.*, **1995**, *353*, 128.
8. A. H. Abu-Hillal and J. P. Riley, *Anal. Chim. Acta*, **1981**, *131*, 175.
9. Y. C. Sun, J. Y. Yang, Y. F. Lin, and M. H. Yang, *Anal. Chim. Acta*, **1993**, *276*, 33.
10. J. Shida and S. Umeki, *Anal. Sci.*, **1999**, *15*, 1033.
11. P. Smichowski, M. B. de La Calle-Guntinas, Y. Madrid, M. G. Cabo, and C. Camara, *Spectrochim. Acta*, **1994**, *49B*, 1040.
12. S. Arpadjan, L. Vuchkova, and E. Kostadinova, *Analyst*, **1997**, *122*, 243.
13. S. Garbos, M. Rzepecka, E. Bulska, and A. Hulanicki, *Spectrochim. Acta*, **1999**, *54B*, 873.
14. S. Garbos, E. Bulska, and A. Hulanicki, *Atom. Spectr.*, **2000**, *21*, 128.
15. H. Koshima, *Anal. Sci.*, **1986**, *2*, 255.

16. H. Koshima and H. Onishi, *Anal. Sci.*, **1985**, *1*, 237.
17. T. Kubota, A. Kawakami, T. Saagar, N. Ookubo, and T. Okutani, *Talanta*, **2001**, *53*, 1117.
18. S. Saracoglu and L. Elci, *Anal. Chim. Acta*, **2002**, *452*, 77.
19. L. Elci, Z. Arslan, and J. F. Tyson, *Spectrochim. Acta*, **2000**, *55B*, 1109.
20. Y. Chai, G. Jiang, and J. Liu, *Analyst*, **2001**, *126*, 1678.
21. C. H. Chung, E. Iwamoto, M. Yamamoto, and Y. Yamamoto, *Spectrochim. Acta*, **1984**, *39B*, 459.
22. R. E. Sturgeon, S. N. Willie, and S. S. Berman, *Anal. Chim.*, **1985**, *57*, 6.
23. Z. Marczenco, "*Separation and Spectrophotometric Determination of Elements*", **1986**, John Wiley & Sons, New York.
24. S. Saracoglu, Ph. D. Thesis, Erciyes University, Kayseri-Turkey, **2001**.
25. R. A. Washington, *Analyst*, **1965**, *90*, 502.
26. J. S. Fritz and G. H. Schenk, "*Quantitative Analytical Chemistry*", 4th ed., **1979**, Allyn and Bacon Inc., Boston.
27. T. Gündüz, "*Kantitatif Analiz Laboratuvar Kitabı*", **1984**, Ankara Univ. Press, Ankara.