Interferences of hydride forming elements and of mercury in the determination of antimony, arsenic, selenium and tin by hydride-generation AAS

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Störungen von Hydridbildnern und von Quecksilber
bei der Bestimmung von Antimon, Arsen, Selen und Zinn
durch die Hydrid-AAS

Zusammenfassung. Mittels der Radiotracertechnik und Atomabsorptionsspektrometrie wurde der Störeinfluß unterschiedlicher Mengen (10−1000 µg) von As, Bi, Hg, Pb, Sb, Se, Sn und Te auf die Bestimmung von As, Sb, Se und Sn mit der Hydrid-AAS untersucht. Durch diese Verfahrenskombination war es möglich, das Ausmaß der Störungen durch diese Elemente sowohl im Hydrierungs- als auch im Atomisierungsschritt zu bestimmen sowie zum großen Teil auch die Gründe für ihr Auftreten aufzuklären. Keine nachweisbare Störung verursachen Hg und Pb bis zu jeweils 100 µg. Die Störung durch Bi und Te tritt hauptsächlich im Hydrierungsgefäß auf, hingegen die durch As, Sb, Se und Sn im wesentlichen in der Quarzabsorptionszelle. Bei Zinn wurde ein starker Memoryeffekt festgestellt, der aus der Ablagerung dieses Elements in der Quarzküvette resultiert und der die Bestimmung von As, Sb und Se völlig unmöglich macht. Ohne diese Störelemente liegen die Hydrierungsausbeuten für As, Sb, Se und Sn bei nahezu 100%. Bei Antimon ist allerdings eine vorherige Reduktion von Sb(V) zu Sb(III) notwendig, für die ein verbessertes Verfahren vorgeschlagen wurde. Denn durch die Radiotracertechnik konnte nachgewiesen werden, daß die während der Alterung von Sb(V)-Lösungen durch Kondensationsprozesse entstehenden Polysäuren nicht mehr hydriert werden können.

Summary. By means of the radiotracer technique supplemented by conventional absorption measurements, the interferences of As, Bi, Hg, Pb, Sb, Se, Sn and Te in amounts between 10 µg and 1 mg with the determination of As, Sb, Se and Sn by hydride generation AAS using heated quartz tube were investigated during the hydride-generation and the atomization stages. Amounts up to 100 µg of Hg and Pb do not cause any detectable interference. The interference of Bi and Te is dominant in the hydride-generation stage and that of As, Se, Sb and Sn in the atomization stage. Tin is retained to a considerable extent in the quartz tube and the resulting memory effect makes the determination of As, Sb, and Se impossible. In the absence of interfering elements, the efficiency of the formation of hydrides of As, Sb, Se and Sn was close to 100%. However, a reduction of Sb(V) to Sb(III) prior to the hydride-generation is necessary for which an improved procedure was developed.

Introduction

Mutual interferences of the hydride-forming elements in the hydride-generation AAS have been described and chemical modifications of the analyte solution for their avoidance have been recommended [2, 8, 10−12, 14, 16, 17, 19]. Welz and Melcher showed [17] that these interferences are probably not caused by competitive reactions with sodium boron hydride. Therefore, they can be expected to take place during the atomization step in the gaseous phase. At atomization temperatures achievable with a heated quartz tube (max. 1000°), the interference mechanism via radical collision seems to be of primary significance [3, 18]. Accordingly, a large excess of one hydride consumes a considerable or even the total amount of radicals of the analyte element, and thus leads to a hindrance of its atomization. Furthermore, a compound formation of arsenic and antimony was shown by molecule absorption spectra to take place at 1000°C [4].

Radiotracers have proved to be extremely useful for investigating the behavior of analytes and interfering elements in atomic absorption spectrometry [6]. However, for the investigation of interferences in the hydride-generation AAS, the radiotracer technique has been used only in the case of selenium [2] as analyte element; the results indicated that the interferences by Te, As, Sb, Bi and Sn are taking place during the atomization stage.

In this work, the radiotracer technique and conventional absorption measurements were used to investigate the extent of interferences caused by 10−1000 µg of other hydride forming elements with the determination of selenium, arsenic, antimony and tin in both the hydride-generation stage and the atomization stage.

Experimental

Instrumentation

A Perkin-Elmer MHS 20 mercury/hydride system and a Perkin-Elmer atomic absorption spectrophotometer model 400 equipped with electrodeless discharge lamps for arsenic, antimony and selenium and with a hollow cathode lamp for tin were used. The operating parameters for these instruments are summarized in Table 1. Deuterium-lamp background correction was used for each measurement. The peak area was recorded during the reaction time.

The γ-rays of the radionuclides were counted with a well-type NaI(Tl) scintillation detector of 23 cm i.d. and 28 cm i.l. (Bicron, Newbury, USA) coupled with a single channel analyzer (Berthold, Wildbad, FRG). In each experiment, a

Table 1. Operating parameters for the system consisting of MHS 20 and AAS 400

	Se	As	Sb	Sn
Wavelength (nm)	196.0	193.7	217.6	286.3
Slit (nm)	2.0	0.7	0.2	0.7
Purge I (s)	35	25	20	20
Reaction (s)	8	10	12	15
Purge II (s)	30	40	50	30

counting error $< 1\%$ was achieved by the choice of an appropriate counting rate and counting time.

Radiotracers and reagents

The radiotracer ^{76}As was produced by irradiation of $(NH_4)_3AsO_3$ in the FRM nuclear reactor of Garching, FRG, at a thermal neutron flux of 10^{13} cm^{-2} s^{-1} for 2.5 days. At the end of the irradiation, the radiotracer had a specific activity of 9.5 Ci g^{-1}. Before use, the tracer was oxidized with an excess of concentrated HNO$_3$ for 1 h at 95°C. The completeness of the oxidation was tested according to [5]. More than 98% of the arsenic was present in the pentavalent form.

For the production of $^{122/124}$Sb, SbCl$_3$ was irradiated in the FRG-2 reactor of the Nuclear Research Center of Geesthacht, FRG, at a thermal neutron flux of 8×10^{13} cm^{-2} s^{-1} for 8 days. The specific activity with regard to ^{124}Sb was 4.5 Ci g^{-1}. The antimony tracer was oxidized to pentavalent antimony with a mixture of 2 ml of H$_2$O$_2$ (30%) and 2 ml of HCl (32%) for 15 min at 95°C. The yield examination [14] proved that the tracer contained more than 99% of Sb(V). The oxidation of Sb(III) to Sb(V) by aqua regia was not quantitative.

The ^{124}Sb(III) radiotracer was prepared by addition of a mixture of 0.25 ml of 10% KI, 0.25 ml of 10% ascorbic acid and 9.5 ml 1 M HCl to the Sb(V) solution, and heating the resulting solution at 95°C for 15 min. The radiotracer ^{75}Se was obtained from NEN, Dreieich, FRG, with a specific activity of 6.2 Ci g^{-1}. The radiotracer was heated in 5 M HCl in an open vessel at 95°C for 1 h to reduce Se(VI) to Se(IV). The hydride-generation efficiency experiments [7] proved that, after the reduction step, more than 99% of selenium was present as Se(IV).

The radiotracer ^{113}Sn was supplied by Amersham Buchler, Braunschweig, FRG, with a specific activity of 4.1 Ci g^{-1} in the form of Sn(IV).

The radionuclidic purity of each radiotracer was tested by high resolution γ-ray spectrometry.

All reagents were of pro analysi grade except hydrofluoric acid which was of "selectipur" grade. They were obtained from Merck, Darmstadt, FRG. Sodium borohydride solutions were prepared from sodium borohydride powder (Riedel-de Haen/Seelze, Hannover, FRG) by dilution to 3% (stabilized with 1% NaOH) and filtration. Standard solutions were prepared from Titrisol solutions (Merck) by dilution.

Determination of the radioactivity

The solution to be investigated was spiked with the appropriate radiotracer and the reduction vessel was placed in the well of the detector for counting. The activity was counted before and after each hydride-generation. The silicone tube connecting the reduction vessel with the quartz tube was rolled up and also placed in the detector well. In counting the quartz tube, distinction was made between the deposition in the hot and in the cold region of the tube. That part of the tube lying outside the oven was considered as the cold region. The two ends of the quartz tube outside the oven were immersed into a mixture of 10% HF/40% HNO$_3$/50% H$_2$O for 10 min to dissolve any deposited elements. The resulting solutions and the quartz tube were then separately counted in the well-type detector. Because ^{113}Sn emits no detectable γ-rays, the activity of this radionuclide was counted via the 392-keV γ-ray of the daughter nuclide, ^{113m}In, which is in radioactive equilibrium with ^{113}Sn after at least 15 h.

Hydride-generation conditions

In the hydride-generation experiments, a solution containing 3% NaBH$_4$ and 1% NaOH was used at different acid concentrations.

Results and discussion

The mutual interactions were investigated under the aspect that the hydride-generation AAS is frequently used for the determination of hydride forming elements in environmental and biological samples that are oxidatively mineralized to destroy organic matter. In such solutions, arsenic is always present as As(V), bismuth as Bi(III), tin as Sn(IV), lead as Pb(II), and mercury as Hg(II). Selenium and tellurium can be present either in the tetravalent or the hexavalent form. With regard to the gaseous phase interference in the quartz tube, the hexavalent states are insignificant, because they are not reduced to selenide and telluride under the conditions used [7]. Sparingly soluble selenates and tellurates are not known within the group of the hydride forming elements. It can be assumed that after a oxidative decomposition, antimony can be present as Sb(III) and Sb(V). Therefore, possible interferences caused by formation of sparingly soluble selenides and tellurides during the hydride-generation stage were investigated for both valence states. Arsenic(V), selenium(IV), tin(IV), and antimony(III) were studied as the element forms to be determined.

Reduction of Sb(V) to Sb(III)

The determination of antimony via its pentavalent state should be avoided since, as the result of aging [1, 9], pentavalent antimony may condense to polyantimonic acids. This aging process reduces the efficiency of the formation of antimony hydride. For example, 10–20% of Sb(V) remained in the reduction vessel if a fresh solution was processed (reaction time 15 s, 3% NaBH$_4$/1% NaOH, reaction volume 10 ml) whereas in processing an aged solution, residues of 30–50% were measured. The hydride-generation efficiency is also dependent on the concentration of NaBH$_4$ and the reaction time. This is a typical behavior for an uncomplete hydride-generation. Therefore, a reduction of Sb(V) to Sb(III) prior to the hydride-generation is necessary.

Radiotracer experiments showed that the reduction of Sb(V) to Sb(III) with a mixture of 0.5 ml 10% KI and 9.5 ml

Table 2. Hydride-generation efficiencies (percentage residue in reduction vessel) at standard conditions and various reaction volumes

Element	Experimental conditions[a]	Residue (%) at different volumes		
		10 ml	20 ml	40 ml
As(V)	0.5 M HCl, r. t. 10 s	0.3 ± 0.5	0.5 ± 0.5	13 ± 3
Sb(III)	1 M HCl, r. t. 15 s	0.5 ± 1	2 ± 1	14 ± 2
Se(IV)	0.5 M HCl, r. t. 8 s	1 ± 1	1 ± 1	9 ± 1
Sn(IV)	sat. H_3BO_3, r. t. 15 s		0.5 ± 1	0.5 ± 1
Sn(IV)	0.5 M HCl, r. t. 15 s	0.5 ± 1	2 ± 1	
Sn(IV)	1 M HCl, r. t. 15 s	6 ± 1		
Sn(IV)	5 M HCl, r. t. 15 s	85 ± 2		

[a] r. t. reaction time

1 M HCl [15] proceeds quantitatively (> 99%) only when Sb(V) is present in monomeric form and the reduction time is at least 15 min. With shorter reaction times usually used [15], as much as 15% of Sb(V) remain unreduced. To distinguish between trivalent and pentavalent antimony in mixtures, a complexation with 1% HF was proposed. Heating the solution during the reduction to 95°C led to a deterioration of the yields for Sb(III) (decomposition of the hydrogen iodide).

Another procedure for the reduction of Sb(V) to Sb(III) using a mixture of 0.5 ml KI/ascorbic acid (5%/5%) and 9.5 ml 1 M HCl was proposed by Peats [13]. Our radiotracer examination of this procedure revealed that at room temperature only monomeric Sb(V) is quantitatively reduced to Sb(III). However, if the reduction is carried out at 95°C, condensed Sb(V) is also reduced quantitatively to Sb(III) with a reduction time of at least 15 min. No detectable losses of antimony by volatilization were observed (< 1%). Antimony(III) produced in this way proved to be rather unstable. Back oxidation to Sb(V) occurred readily, for instance, upon keeping the solutions overnight. Within 3 h, less than 3% of Sb(III) was oxidized to Sb(V). This back oxidation is obviously caused by elementary iodine (can be observed visually) or its oxidation products, such as iodate. In addition, it was observed that fumes of iodine give rise to an interference in the quartz tube.

Hydride-generation efficiencies

Each experiment was carried out with 50 ng of the element investigated under conditions given in Table 1. Less than 1% of the labelled elements were adsorbed on the walls of the vessel and on the silicone tube. The results obtained are summarized in Table 2 as percentage residues remaining in the reduction vessel. Under standard conditions (see Table 1), all investigated elements can be quantitatively converted to hydrides. However, the efficiency of the hydride formation is effected by the composition of the reduction solution and its volume. The reduction of tin can be executed also in hydrochloric acid solutions, but only at concentrations up to 1 M. The radiotracer experiments indicate that the strong decrease of the absorbance for tin, if hydrochloric acid is used instead of boric acid, can be attributed to different hydrogen flow during the hydride-generation. While from a boric acid solution the shape of the tin signal is similar to that typical for the flame AAS, from a hydrochloric acid

solution a clearly peak-shaped signal appears. Thus, a sensitive determination of tin from hydrochloric acid solutions is possible. However, the developed hydride must be collected prior to the atomization. The above discussed behavior of tin is of significance also if it is considered as interfering element.

Deposition of elements on the quartz tube

In the case of selenium and arsenic, no detectable adsorption (< 1%) was observed at 900°C in the hot region of the quartz tube. Likewise, for all investigated elements, no loss of sensitivity was detected by absorption measurements after several runs of high amounts (up to 100 µg) of selenium and arsenic as interfering elements. No adsorption could be detected also for antimony as analyte element, but applying 100 µg of antimony as interfering element, losses of sensitivity occurred for several elements in sequential measurements. They are obviously due to adsorption of undetectable amounts of antimony (< 1%) on the quartz tube. These losses of sensitivity could be avoided by conditioning the tube with a solution containing 10% HF and 40% HNO_3.

The deposition of tin on the quartz tube is very significant. Eight consecutive hydride-generations showed that $25 \pm 13\%$ of the processed 50 ng of Sn(IV) were retained by the hot part of the tube. Three following hydride-generations, with 100 ng of inactive tin each, reduced the original residue radioactivity of the tube by about 5% per hydride-generation, and a movement from the hot into the cold part of the tube was observed. Large amounts of tin (100 µg) behaved in a similar way. Three consecutive reductions with 100 µg of tin led to a total signal depression at 50 ng of each of the investigated elements.

These results prove that samples containing large amounts of tin cannot be accurately analyzed for As, Sb and Se by using a quartz tube.

Mutual interactions

The results obtained for mutual interactions of the hydride forming elements in both stages, the hydride-generation (liquid phase) and the atomization in the quartz tube (gaseous phase) are given and discussed below for the individual elements. The results are presented in Tables 3 — 6 as percentage residue of the analyte element remaining in the reduction vessel and as percentage extinction of the analyte element, whereby the extinction measured with pure analyte solution was taken with 100% as the basis for comparison of extinctions measured with the same analyte solution to which certain amounts of other hydride forming elements were added. Also given in the tables is the reduction of extinction for pure analyte elements as the result of five consecutive previous reductions of 10 µg — 1 mg of an interfering element. We call this effect "interference memory effect". The results for extinctions and interference memory effects were obtained from absorption measurements and that for percentage residue in the reduction vessel from radiotracer experiments.

For all analyte elements investigated, the addition of up to 100 µg of lead and mercury did not cause any detectable interferences, and no significant difference could be observed between trivalent and pentavalent antimony as interfering element. The interference memory effect caused by

100 µg of tin makes the determination of arsenic, antimony and selenium impossible (no signal).

Selenium. Table 3 summarizes the results for interferences of other hydride forming elements with the determination of selenium. If tetravalent tellurium was present in amounts higher than 50 µg, elemental tellurium precipitated during the hydride-generation (it could be observed visually). This precipitate did not interfere with the determination of selenium. Addition of more than 50 µg of Bi(III) led to visually observable precipitation of elemental bismuth. This precipitation caused such a strong interference and bad reproducibility that no meaningful selenium measurement was

Table 3. Extent of interferences of other hydride forming elements in the determination of selenium(IV) [50 ng of Se(IV) in 10 ml 0.5 M HCl] in the hydride-generation AAS

Inter-fering element	Amount (µg)	Extinction (%)	Se-residue in reduction vessel (%)	Interference memory effect (%)
As(V)	10	37 ± 5	1.5 ± 1	
As(V)	100	6 ± 3	3.5 ± 1	no
Bi(III)	10	94 ± 5	0.6 ± 0.5	
Bi(III)	50	see text	10 ± 0.5	no
Sb(III,V)	100	no signal	0.2 ± 0.5	11 ± 3
Sn(IV)	100	no signal	1 ± 1	70 ± 10
Te(IV)	100	97 ± 5	2 ± 1	no

Table 4. Interferences in the determination of As(V) by hydride-generation AAS [50 ng of As(V) in 10 ml 0.5 M HCl]

Interfering element	Amount (µg)	Extinction (%)	As-residue in reduction vessel (%)	Interference memory effect (%)
Bi(III)	100	no signal	1 ± 0.5	no
Sb(III,V)	1000	no signal	1 ± 0.5	no
Se(IV)	100	no signal	1.5 ± 0.5	no
Sn(IV)	100	no signal	0.8 ± 0.6	100 ± 10
Te(IV)	10	50 ± 10	6 ± 1	
Te(IV)	100	20 ± 10	43 ± 3	no
Te(IV)[a]	10	5 ± 5	2.5 ± 0.5	
Te(IV)[a]	100	5 ± 5	24 ± 2	no

[a] The same conditions as above but 5 M HCl

possible. No other significant interferences are occurring in the hydride-generation stage. The determination of selenium is seriously interfered by 100 µg amounts of arsenic, antimony and tin during the atomization stage. Significant interference memory effects are caused also by larger amounts of antimony.

Arsenic. The interferences of Bi, Sb, Se, Sn and Te with the determination of arsenic are summarized in Table 4. In the hydride-generation stage, the interference of larger amounts of tellurium, depending on the HCl concentration, must be considered. The additional experiments with tellurium in 5 M HCl allow the assumption that the interference caused by tellurium is due to sparingly soluble tellurides, which are better soluble in more concentrated acids. Elementary precipitated bismuth (visible from 50 µg on) does not interfere with the determination of arsenic.

Considerable interferences are caused by bismuth, antimony, selenium and tin in the atomization quartz tube. In the case of tellurium, also the extent of this interference strongly depends on the HCl concentration.

Antimony. Because, with an excess of iodide, Se(IV) and Te(IV) are reduced to the elements and Bi(III) is precipitated as BiOI, an iodide-free antimony radiotracer was produced. However, investigations with this radiotracer are only of theoretical interest as in each actual practical case both the analyte element and the interfering element are always reduced simultaneously. A freshly oxidized antimony tracer was treated with an excess of 1 ml hydroiodic acid (50%) and after 15 min with 0.2 ml conc. sulfuric acid. This solution was slowly heated until the sulfuric acid began to fume. Then 2 ml of water were added and the solution was evaporated again to remove the hydroiodic acid completely. The radiotracer obtained in this way contained 99% of Sb(III) (HF test after Ref. [14]). By using this radiotracer, no precipitation of elementary selenium could be observed. However, the Sb(III) radiotracer was stable only for about one day; after that it could not be used because of significant extent of its oxidation to Sb(V) caused by traces of iodine or iodate.

Table 5 gives the interferences in the determination of antimony(III). The interferences caused by selenium and tellurium can significantly be reduced by their precipitation as elements with iodide. The precipitates do not affect the reduction of antimony. The interference caused by bismuth in the hydride-generation stage is probably increased by

Table 5
Interferences in the determination of antimony(III) by hydride-generation AAS [100 ng of Sb(III) in 10 ml 1 M HCl]

Interfering element	Amount (µg)	Admixture	Extinction (%)	Sb-residue in reduction vessel (%)	Interference memory effect (%)
As(V)	1000	KI/asc.ac.	no signal	0.5 ± 0.5	no
Bi(III)	10	no	90 ± 10	2 ± 0.5	
Bi(III)	100	no	5 ± 3	8 ± 2	no
Bi(III)	10	KI/asc.ac.	85 ± 10	6.5 ± 1	
Bi(III)	100	KI/asc.ac.	10 ± 5	18 ± 2	no
Se(IV)	100	no	no signal	7 ± 1	
Se(IV)	100	KI/asc.ac.	50 ± 10	1 ± 1	no
Sn(IV)	100	KI/asc.ac.	no signal	0.5 ± 0.5	100 ± 5
Te(IV)	100	no	90 ± 7	8 ± 2	
Te(IV)	100	KI/asc.ac.	95 ± 5	4.5 ± 1	no

Table 6
Interferences in the determination of tin(IV) by hydride-generation AAS [50 ng of Sn(IV) in 20 ml of saturated boric acid or in 10 ml of 1 M HCl]

Interfering element	Amount (µg)	Medium	Extinction (%)	Sn-residue in reduction vessel (%)	Interference memory effect (%)
As(V)	100	sat.bor.ac.	no signal	0.5 ± 0.5	no
Bi(III)	100	sat.bor.ac.	10 ± 10	1.5 ± 1	no
Sb(III,V)	100	sat.bor.ac.	no signal	0.5 ± 0.5	no
Se(IV)	100	sat.bor.ac.	50 ± 10	1 ± 0.5	no
Se(IV)	100	1 M HCl	not examined	5.5 ± 2	not examined
Te(IV)	10	sat.bor.ac.	90 ± 10	9 ± 2	
Te(IV)	100	sat.bor.ac	40 ± 20	35 ± 5	no
Te(IV)	10	1 M HCl	not examined	33 ± 3	not examined

coprecipitation of antimony with precipitating BiOI. The precipitation of elemental bismuth (visible from 50 µg on) also alters the determination of antimony. With higher amounts, bismuth considerably interferes also in the atomization stage. The interference of tin is similar as in the determination of arsenic.

Tin. Tin can also be reduced in a hydrochloric acid medium. Since the release of hydrogen selenide and hydrogen telluride from a hydrochloric acid solution is much easier than from boric acid, the interferences caused by these two elements in the hydride-generation stage were examined for a tin amount of 50 ng in 20 ml of saturated boric acid and in 10 ml of 1 M hydrochloric acid. However, the interferences in the atomization stage with reduction in hydrochloric acid were not investigated because the determination of tin using this medium is unsuitable (see above section on efficiencies).

The results for interferences in the determination of tin are surveyed in Table 6. The moderate extent of interferences caused by selenium and tellurium in boric acid can be explained by the fact that these elements are not reduced in saturated boric acid or they are reduced but are not swept out from the reduction solution as H_3BO_3 is a very weak acid. In the case of selenium, this assumption was confirmed by the radiotracer experiments. In saturated boric acid, selenium remained nearly to 100% in the reduction vessel. However, in processing 10 µg of tellurium, the residue of tin in hydrochloric acid is about four times higher than in boric acid.

The hindrance of the reduction of Te(IV) to $Te^{(-II)}$ in saturated boric acid may be a possible explanation for the above behavior.

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