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A review of sampling and analytical procedures for antimony and its compounds is presented. Emphasis has been placed on those methods which have application to personal air or biological samples in industrial hygiene. Two analytical techniques in particular have been used most frequently — colorimetric and atomic absorption. A need for research to develop satisfactory solid sorbent sampling techniques for stibine and other volatile antimony compounds is evident.

Sampling and analytical methods for antimony and its compounds — a review

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introduction

The use of antimony for preparation of alloys as well as for other uses has been known for thousands of years.⁽¹⁾ The primary ore is stibnite, Sb_2S_3 , which is roasted to yield antimony oxides; reduction to the metal is commonly done in a reverberatory furnace. At the present time, about half of the antimony metal produced is alloyed with lead for use in storage batteries. Other antimony alloys are used in electrical equipment, type metal, solder, and ammunition. Commercially important antimony compounds include the trioxide, trisulfide, trichloride, and pentasulfide.⁽²⁾ The U.S., which uses 25% of the world antimony production, derives half of its supply from recycling.⁽¹⁾

Because of the variety of uses of antimony, potential occupational exposures which must be monitored include not only metallic antimony, but also a variety of antimony compounds, including the stibine (SbH_3) produced by overcharging batteries.⁽³⁾ The present OSHA standards for 8-hr. time weighted average exposures are 0.5 mg/cu m (0.1 ppm) for SbH_3 and 0.5 mg/cu m (as Sb) for other forms.⁽⁴⁾ Thus, acceptable sampling and analytical methods for determination of exposure to antimony must apply to both vapors, including SbH_3 as a separate species, and solids, for a sample size of approximately 10 to 200 μ g Sb (as the metal). In addition, methods suitable for biological monitoring are important, since worker exposures may be monitored by determining

concentrations in blood, urine, hair, nails, and feces.

This paper presents a review of methods which have been applied, or have potential for application to personal industrial hygiene samples.

sampling methods

stibine and other volatile antimony compounds

At ambient temperatures, SbH_3 is a gas and the antimony halides have significant vapor pressures.⁽²⁾ Therefore, sampling methods which are efficient in the collection of gases must be devised for these compounds. Most of the sampling methods reported for atmospheres containing SbH_3 require the use of an impinger with a solution which will readily oxidize the SbH_3 . An early sampling method, reported two years after the discovery of SbH_3 in 1837, utilized an aqueous silver nitrate solution which reacted with SbH_3 to form a precipitate.⁽⁵⁾ The major product of the reaction with excess $AgNO_3$ has been reported to be the hydrated oxide, H_3SbO_3 .⁽⁶⁾ However, multiple reactions occur and the precipitate also includes metallic antimony, silver, and Ag_3Sb , making quantitation of antimony difficult. Other absorbing solutions which have a limited usefulness for analytical purposes include those of iodine,⁽⁶⁻⁸⁾ acidified chlorine,⁽⁶⁾ and silver sulfate.⁽⁹⁾

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Although SbH_3 is absorbed efficiently in concentrated iodine solutions,⁽⁸⁾ incomplete absorption has been reported at low iodine concentrations.⁽⁷⁾ The reaction in these solutions,



may be followed by observing the decolorization as the I_2 disappears. However, loss of I_2 through vaporization or oxidation of the HI causes problems in quantitative measurements. Chlorine water, acidified with 6 N HCl, has been used to collect SbH_3 but the absorption was reported to be incomplete and the samples unstable; no residual antimony was detected using colorimetric analysis after the samples were aged 12 hours.⁽⁶⁾ A silver sulfate solution containing starch, tartaric acid, sulfuric acid, and methyl fluorone was used as the basis for a meter for area monitoring of SbH_3 .⁽⁹⁾ The instrument is used for the collection and determination of SbH_3 in small (10 liter) air samples. The useful life of the reagent is only 2-3 hr, however, and the solution must be replaced frequently.

All of the above sampling methods were tested in conjunction with colorimetric analyses and shown to be of limited usefulness. Adaptations of these sampling methods for use with other types of analyses, such as atomic absorption spectrophotometry (AAS), have not been reported.

The use of a solution of 6% (w/v) HgCl_2 in 6 N HCl has been reported to have a 99% efficiency for absorption of stibine.⁽⁶⁾ The samples were shown to be sufficiently stable for colorimetric analysis for a time period of at least two weeks.^(6,10-12) The products of the reaction, which are soluble in the acidic solution, were not reported but probably included the complex ion, SbCl_4^- .⁽¹²⁾ In addition, this solution can be used for atomic absorption analyses, although the sensitivity of the analysis is somewhat lower than that for aqueous solutions containing only antimony.⁽¹³⁾

Some work has been reported on the use of indicator papers using silver or gold salts^(6,14-16) for the qualitative detection of SbH_3 . Semi-quantitative measurements have been reported with the use of filter papers impregnated with a solution of AgNO_3 .⁽¹⁶⁾ Upon contact with as

little as 0.2 ppm SbH_3 , a black stain quickly develops. However, stain intensity is a function of relative air velocity and is neither uniform nor stable enough for quantitative work. In addition several other compounds, including AsH_3 , PH_3 , and H_2S also react with the reagent to produce the black stain.

For industrial hygiene purposes and personal sampling, the use of solid sorbent tubes is far preferable to the use of impingers. Very little work on the use of such sorbents for antimony compounds has been reported. Attempts to collect SbH_3 on charcoal tubes were reported to be unsuccessful, due to poor collection efficiency.⁽¹⁷⁾ The use of an oxidizing agent, such as iodine, on charcoal would take advantage of the ease with which SbH_3 is oxidized to non-volatile compounds and should be investigated. Preliminary data indicate that collection of SbH_3 on silica gel coated with HgCl_2 may be feasible; this method is compatible with colorimetric analysis as well as AAS.⁽¹⁸⁾

particulate antimony and antimony compounds

For particulate antimony and its compounds, impinger, electrostatic precipitation, or filter collection have been recommended.^(11,19,20) The collection efficiency of 0.8 μm cellulose ester membrane filters was found to be 99.7% when an antimony potassium tartrate aerosol (1 mg Sb/cu m) was sampled at 1.5 Lpm.⁽²¹⁾

Consideration of the antimony content of blank filters is important in the selection of sampling media. Some glass fiber filters have been found to contain as much as 230 ng Sb/sq cm.⁽²²⁾ These filters are not acceptable for personal sampling, in which as little as 500 ng Sb may represent a quantifiable sample. The blank for cellulose mixed ester membrane filters was found to be 0.24 ng Sb/sq cm by neutron activation analysis,⁽²³⁾ and cellulose acetate membrane filters were reported to contain 0.013 ng Sb/sq cm.⁽²⁴⁾ Another estimate by graphite furnace AAS of the antimony content of cellulose mixed ester membrane filters is 0.321 ng Sb/sq cm.⁽¹³⁾ Polystyrene filters, used for area sampling, gave blank values of less than 0.2 ng Sb/sq cm in three studies.⁽²⁴⁻²⁶⁾

TABLE I
Wet-Ashing Techniques and Recovery of Antimony

Ashing Technique	Compound	Matrix	Anal. Method	Recovery (\pm S.D.)	Ref.
HNO ₃ (140°C), HCl, Tartaric Acid	Sb	filter	AAS (flame)	100 (\pm 3)%	21
HNO ₃ (140°C), HCl, Tartaric Acid	KSbTart.	filter	AAS (flame)	101 (\pm 2)	21
HNO ₃ (140°C), HCl, Tartaric Acid	Sb ₂ S ₃	filter	AAS (flame)	103 (\pm 2)	21
HNO ₃ (100°C), Tartaric Acid	Sb ₂ O ₃	ores	AAS (flame)	102 (\pm 5)	98
HNO ₃ , HCl	Sb	steel	AAS (hydride)	99 (\pm 5)	108
HNO ₃ , HClO ₄ , H ₂ SO ₄	?	foods	AAS (hydride)	104 (\pm 7)	106
HNO ₃ , H ₂ SO ₄	Sb ₂ O ₃	urine	AgDEDC	97 (\pm 6)	55
HNO ₃ , H ₂ SO ₄	Sb ₂ O ₃	blood		90 (\pm 5)	55
HNO ₃ , HClO ₄ , H ₂ SO ₄	Sb ₂ (SO ₄) ₃	tissue	Rhodamine B	~100	59
H ₂ SO ₄ , H ₂ O ₂ , HCl	?	blood	NAA	91 (\pm 6)	73
H ₂ SO ₄ , NaNO ₃ , Na ₂ SO ₃	KSbTart.	tissue	NAA	95	74
HBr, H ₂ SO ₄	Sb ₂ O ₃	tissue	NAA	97 (\pm 2)	81
HNO ₃ , HClO ₄ , H ₂ SO ₄	?	cocoa	Radiochem.	100	28
NH ₄ Cl, H ₂ O ₂ , H ₂ SO ₄	Sb ₂ O ₃	tissue	Radiochem.	95	76

analysis for antimony and antimony compounds

Most analytical methods for antimony are elemental analyses. A few techniques, including electrochemical, provide discrimination between oxidation states, and chromatographic methods can be specific for individual compounds. Not all analytical methods are compatible with industrial hygiene samples. The discussion which follows emphasizes those which have been shown to be most useful in this regard, or have potential for application to industrial hygiene samples.

sample preparation

Preliminary sample treatment for industrial hygiene analyses must take into consideration the high volatilities of some antimony compounds, especially the antimony halides. Ashing of air and biological samples must be done at low temperatures and the evaporation of solutions to dryness at high temperatures must be avoided to prevent losses of antimony. In a comparison of low-temperature dry ashing with muffle furnace ashing (550°C), the recoveries determined by AAS were 99% and 46%, respectively,⁽²⁷⁾ although dry ashing has been found to yield greater than 90% recovery by others.⁽²⁸⁾ A summary of wet-ashing techniques for various sample matrices and the reported recoveries appears in Table I. The analytical methods used in these studies are considered in the following section.

The use of colorimetric analytical methods may require special precautions in ashing. For example, erratic losses of up to 70% of antimony

from tissue samples ashed in sulfuric acid have been reported.⁽²⁹⁾ The antimony was apparently not lost from solution, but was present in a chemical form (Sb(IV)) which was inert toward Rhodamine B.

Concentration of aqueous antimony solutions by solvent extraction is possible with a number of reagents.^(30,31) Antimony(V) can be extracted from concentrated hydrochloric acid solutions into isopropyl ether, and is thereby separated from antimony(III).⁽³²⁾ This principle has been applied in ring oven analysis.⁽³³⁾ Extraction of antimony(V) is also used in spectrophotometric procedures involving Brilliant Green⁽³⁴⁾ and Rhodamine B.^(19,20) Antimony(III) is selectively extracted by cupferron-chloroform solutions,⁽³⁵⁾ by benzene from sulfuric acid solutions with 0.01 M iodide,⁽³⁰⁾ and by silver diethyldithiocarbamate-carbon tetrachloride solutions,⁽³¹⁾ the latter being suitable for spectrophotometric determination. Antimony(III) and (V) are extracted by ethyl acetate from solutions containing a mixture of citric, oxalic, and hydrochloric acids.⁽³⁶⁾

Ion-exchange resins have also been used for separation and concentration of antimony in analytical procedures. Both antimony(III) and (V) are adsorbed from acid solutions (pH > 3) on Dowex 1-X10 anion exchange resin,⁽³⁷⁾ and dithiocarbamate resins were used for quantitative uptake of antimony(III).⁽³⁸⁾ In some cases, concentration of samples by freeze-drying may be convenient. Freeze-drying of natural waters containing less than 1 ppm Sb at pH 1.5 gave essentially complete recovery, although the chemical form of Sb was not specified.⁽³⁹⁾

TABLE II
Analytical Methods for Antimony

Method	Detection Limit, μg^a	Working Range, μg	Precision Cv	Remarks, Matrix	Ref.
Atomic absorption (flame)	3	10-400	0.06 at 180 μg	Air particulates	21,98
Atomic absorption (hydride)	0.005	0.05-1	0.04 at 0.5 μg	in H_2 flame	101,103,106
Atomic absorption (hydride)	0.025	0.1-2	0.04 at 0.25 μg	Automated method	107
Atomic absorption (hydride)	0.0005	0.005-0.1	0.05 at 0.2 μg	T-tube burner	104
Atomic absorption (flameless)	0.02	0.05-20	0.04 at 0.25 μg	0.1 mL aliquots taken	112,115
Atomic fluorescence	0.001	0.001-0.15	~0.05 at 0.002 μg	in H_2 flame	111
Colorimetry	~0.5	1-20	0.01 at 20 μg	Brilliant Green	34
Colorimetry	0.1	1-30	0.10 at 8 μg^b	Rhodamine B	18,20,58,59,63
Colorimetry	10	10-700	0.05 at 100 μg	Iodine	7
Emission spectroscopy	2	5-500	not given	Plasma excitation	68
Neutron activation	0.03	0.1-20	~0.10	nondestructive	24,70,72,75
Polarography	~1	1-30	not given	air samples	82-84
Specific ion electrode	0.01	0.1-100	not given	total Sb(III+V)	86
X-ray fluorescence	150	150-10000	0.021 at 6500 μg	nondestructive	92

^aCalculated for a 10 mL sample volume.

^bCollaborative test Cv.

The stability of aqueous antimony solutions is related to their chemical composition. Solutions with a range of pH = 1-11 were analyzed using AAS; low recoveries were obtained within 24 hours for solutions with pH > 1.5.⁽⁴⁰⁾ Colorimetric analysis of dilute solutions of antimony in 6 N HCl (4 ppm antimony as the sulfate or potassium tartrate), showed low recoveries within one day and losses of 25-50% of the initially recoverable antimony were found after a period of 37 days.⁽⁴¹⁾ When no HCl was present, the solutions were stable for at least 50 days. This behavior was attributed to rapid hydrolysis of the hexachloroantimonate(V) ion to form mixed hydroxychloro complex anions which do not form the colored Sb complex with Brilliant Green.⁽⁴¹⁾ However, this conclusion was not confirmed by the use of other instrumental techniques such as AAS. Similar losses of recoverable antimony have been reported⁽⁶⁾ for SbH_3 collected in solutions of hydrochloric acid saturated with chlorine. After standing for only 5 hours the recovered antimony dropped to approximately 25% of that determined immediately after collection. These losses were also presumed, without confirmation by other techniques, to be due to the formation of a stable complex which did not react to form the colored Rhodamine B complex used for analysis. Alternative explanations are also possible, such as losses due to adsorption of metal ions on container surfaces; such losses have been reported for a number of heavy metals, including lead, silver and mercury.⁽⁴²⁻⁴⁴⁾

analytical techniques

The early analytical methods for antimony were based on the Berzelius-Marsh and Gutzeit processes, using zinc as a reductant with measurement of the evolved SbH_3 gravimetrically⁽⁴⁵⁾ or colorimetrically.⁽⁴⁶⁾ Several volumetric, gravimetric, and colorimetric methods are available for analysis of antimony alloys⁽⁴⁷⁾ or process streams⁽⁴⁸⁾ but most are not sensitive enough for personal samples. Table II presents a summary of quantitative techniques reported for antimony determination.

Qualitative tests include the use of silver nitrate impregnated paper,^(6,16,33) Reinsch's test,^(49,50) mercuric cyanide,^(51,52) silver diethyldithiocarbamate,⁽⁵³⁾ and copper.⁽⁵⁴⁾ The reported detection limits vary: 1 ppm in air for silver nitrate papers;^(6,16) 10-20 μg in forensic samples;^(49,54) 20 μg of bound (inorganic plus organic) antimony;⁽⁵²⁾ and 100 μg of elemental antimony.⁽⁵¹⁾

Until the widespread use of AAS, colorimetric methods were most commonly used for antimony.⁽³⁰⁾ Silver diethyldithiocarbamate (AgDEDC),⁽⁵⁵⁾ Brilliant Green,^(34,56) iodide,^(30,57) Malachite Green,⁽¹²⁾ methyl fluorone,⁽⁹⁾ and Rhodamine B^(6,19,20,58,59) have been used as reagents. A recently reported kinetic method relies on a photometric end point.⁽⁶⁰⁾ Rhodamine B colorimetry for antimony was first reported in 1941 and has been more thoroughly studied than the others.⁽⁵⁸⁾ Although this initial report recognized the potential industrial hygiene

application to analysis of tissue and air samples, sample handling procedures were not considered. The detection limit for standard solutions was reported to be 0.1 $\mu\text{g Sb}/5\text{ mL}$ solution. An improvement involving extraction of the colored antimony complex was published in 1945,⁽⁶⁾ and application to biological samples was also reported in 1945.⁽²⁹⁾ Several other applications to industrial hygiene surveys have been published.^(61,62) A collaborative test was conducted in which 10 laboratories analyzed three solutions in the range 3-8 $\mu\text{g}/\text{mL}$.⁽⁶³⁾ The overall coefficient of variation was 10.5%, with average recovery of 105%. Several modifications were made as a result of this test, including the use of chilled reagents. The revised method was subsequently recommended by the ACGIH for air and biological samples,⁽¹⁹⁾ by NIOSH for urine,⁽⁵⁹⁾ and by the Intersociety Committee for air samples.⁽²⁰⁾ Although this method has acceptable accuracy and precision, it involves considerably more sample handling and skill than are required in atomic absorption methods. Also, as stated in the preceding section, recovery is dependent upon the oxidation state of the antimony present. Digestions must be made in the presence of strong oxidizing agents to insure that all antimony is in the +5 state.^(19,20)

Analysis of airborne particulates by emission spectroscopy was described for 15 elements, including antimony.⁽⁶⁴⁾ The detection limit for conventional emission spectroscopy is in the range 10-20 $\mu\text{g Sb}$.^(65,66) A modification using hydride generation resulted in an improved detection limit of approximately 0.5 ng.⁽⁶⁷⁾ Emission spectroscopy has the advantage of multielement capability, as well as minimal matrix effects and a large dynamic range. A relatively new excitation source, the inductively coupled plasma, is being used for emission spectroscopy (ICP-OES). This technique shows promise for application to the analysis of both air particulate and biological samples.^(68,69) The detection limit for antimony is reported to be 0.2 $\mu\text{g}/\text{mL}$ in aqueous solutions.⁽⁶⁸⁾

Neutron activation analysis (NAA) has been used by several workers for simultaneous determination of several elements in atmospheric particulate matter.^(23,24,26,70,71) The advantages are good sensitivity, nondestructive analysis, minimal sample treatment, and

multielement capability, but expensive facilities and extensive data processing are needed.⁽⁷²⁾ A detection limit of 0.2 ng Sb per cellulose ester filter was determined,⁽²⁴⁾ while high blank values for polystyrene filters resulted in a detection limit of 80 ng Sb per filter.⁽⁷⁰⁾ More than 30 elements, including antimony have been determined in biological samples by NAA.⁽⁷³⁻⁸¹⁾ Accuracy of $\pm 5\%$ or better for samples containing 0.1-10 ng Sb was reported.⁽⁷⁴⁾

Instrumental photon ($> 7\text{ MeV}$ X-ray) activation analysis has been applied to air particulates.⁽²⁵⁾ Although enhanced sensitivities compared to NAA have been demonstrated for other heavy metals including As and Pb, the minimum detectable quantity of Sb was 300 ng. This value is a factor of 1.5×10^4 greater than that for NAA.

Electrochemical methods are applicable to trace quantities of antimony, and a specificity for mixtures of compounds of different valence states may be obtained. Air samples taken on glass fiber filters were analyzed polarographically; both the antimony(III) and (V) species were determined.⁽⁸²⁾ Other polarographic determinations of antimony have also been reported.⁽⁸³⁻⁸⁵⁾ An ion-selective electrode with a detection limit of 10 nM of either antimony(III) or (V) was recently reported.⁽⁸⁶⁾

Gas chromatography provides one of the few means available for determination of individual antimony compounds. Procedures have been developed for antimony trichloride,⁽⁸⁷⁾ triiodide,⁽⁸⁸⁾ pentafluoride,⁽⁸⁹⁾ and triphenyl antimony.⁽⁹⁰⁾ No applications of this potentially useful technique to analyses of industrial hygiene samples have been reported; data are also lacking in the area of solid sorbents for sampling these compounds in air.

Because of its non-destructive nature and the minimal sample preparation required, X-ray fluorescence spectrometry would seem to be highly useful in industrial hygiene work. However, particle size and composition effects are important sources of potential errors.⁽⁹¹⁾ Calibration curves must be constructed using materials which closely match the sample particle size and composition of the matrix. After determination of the appropriate instrumental parameters, a method for antimony as Sb_2O_3 in paint pigment was

reported to have a detection limit of 15 ppm.⁽⁹²⁾ For samples which are thin films on a substrate, a detection limit of 4 ng/sq cm has been reported.⁽⁹³⁾ This limit, which was determined for samples on a Mylar[®] foil, is lower than that expected for samples on cellulose ester filters which produce greater background scatter of the incident radiation and hence increase the detection limit. Calculated detection limits of approximately 100 ng Sb/sq cm on cellulose ester filters have been reported.⁽⁹⁴⁾ Estimates of antimony burden in antimony process workers' lungs *in vivo* by X-ray fluorescence spectrometry have been made, with a detection limit equivalent to 2 mg/sq cm.⁽⁹⁵⁻⁹⁷⁾

Perhaps the most widely used technique for industrial hygiene analysis is atomic absorption spectrophotometry with its three major variations – flame AAS (direct aspiration into an air-acetylene flame), hydride generation - hydrogen flame AAS, and flameless AAS (electrically heated graphite furnace). In the range 0.26 - 1.1 mg Sb/cu m, the overall coefficient of variation for sampling on cellulose ester membrane filters and analysis by flame AAS was found to be 0.059.⁽²¹⁾ If the sample contains more than 10 mg Pb/mL, positive spectral interference will occur at a wavelength of 217.6 nm and 231.2 nm should be used for quantitation of antimony.^(21,98) The effects of copper on antimony absorption are confusing; one report ascribes positive spectral interference to 1000 ppm Cu,⁽²¹⁾ but another indicates a negative interference at the same concentration.⁽⁹⁸⁾ The absorbance of antimony solutions is reported to be depressed by the presence of sulfuric acid in the range 1-50% (v/v).⁽⁹⁸⁾ For industrial hygiene samples, an air concentration of about 0.05 µg Sb/cu m is the lowest value that can be measured accurately with a 360-liter sample using flame AAS.⁽²¹⁾ The working range for flame AAS is 10-200 µg Sb in 10 mL of solution.^(21,98,99) Instrumental sensitivities 500 times better than this are available with the use of either hydride generation or flameless techniques.

Hydride generation-flame AAS analysis for antimony was first demonstrated using zinc metal or magnesium metal plus titanium trichloride as a reductant.⁽¹⁰⁰⁻¹⁰²⁾ However, the use of sodium borohydride as a reductant, either

in the form of pellets or in solution, has advantages in ease of use and effectiveness.⁽¹⁰³⁻¹⁰⁵⁾ Sensitivity ranges from 0.6 ng Sb per sample for a modified system which concentrates the stibine⁽¹⁰⁴⁾ to 10 ng Sb per sample for direct introduction of the SbH₃ into a H₂-Ar or H₂-N₂ burner.⁽¹⁰³⁾ Hydride generation has been applied to antimony determinations in foods,⁽¹⁰⁶⁾ natural waters,⁽¹⁰⁷⁾ and steels,⁽¹⁰⁸⁾ and an automated procedure has been described.⁽¹⁰⁹⁾ Most of the published work is with antimony(III) solutions, and it is claimed that prereduction of antimony(V) to antimony(III) is essential to SbH₃ generation.^(106,108) It is not clear whether the need for prereduction is a function of solution acidity and this point deserves further study. Potential interferences from arsenic, cobalt, copper, chromium, nickel, tin, and the noble metals have been studied^(108,110) and shown to be significant, but in most cases these should not present severe problems in industrial hygiene samples. Determinations of antimony in 5% (w/v) HgCl₂ in 6 N HCl suffer from a 30% decrease in sensitivity, but have acceptable precision.⁽¹³⁾ Therefore, the use of this solution as collection medium for stibine is compatible with hydride generation AAS.

Application of the hydride generation technique to atomic fluorescence resulted in a detection limit estimated at less than 0.1 ng Sb,⁽¹¹¹⁾ which is several times more sensitive than the equivalent atomic absorption technique. Although this instrumental technique obviously allows lower detection limits than flame AAS, applications to industrial hygiene samples have not been reported and the instrumentation is not commonly available.

The absolute sensitivity of flameless AAS is much greater (120 pg Sb per 5 to 100 µL aliquot) than hydride generation-flame AAS.⁽¹¹²⁻¹¹⁵⁾ However, because only small aliquots can be analyzed, sensitivity for industrial hygiene samples which must be ashed and diluted to a known volume is 5-15 ng Sb per sample, a range similar to that for hydride generation.⁽¹³⁾ Interferences from metals such as iron,⁽¹¹²⁾ titanium,⁽¹¹²⁾ lead,⁽²¹⁾ and copper⁽²¹⁾ may be expected. Reports concerning the effect of acid concentrations vary. A slight dependence of antimony absorbance on acid concentration below 0.5% acid has been reported,⁽¹¹⁵⁾ another

report indicates a strong dependence of the 217.6 nm absorbance upon acid type (HCl, HNO₃, H₂SO₄) and concentration up to 10% (w/v).⁽¹¹²⁾

summary and conclusions

It is evident that a wide variety and a large number of sampling and analytical techniques are available for antimony. In the determination of antimony and its compounds in industrial atmospheres the important factors to be considered are selection of the appropriate sampling device, careful sample treatment, and analysis by a method compatible with the sample.

Conventional cellulose ester membrane filters are preferred for sampling particulate antimony and its compounds, while for stibine either a solid sorbent device employing mercuric chloride on silica gel or an impinger containing acidic mercuric chloride solution may be used. The impinger method must be recommended at this time for sampling volatile antimony compounds other than stibine because only preliminary data is available for use of the solid sorbent sampler for these compounds and successful application of this technique has not been reported.

Sample ashing techniques are available which provide quantitative recovery of antimony from both biological and environmental samples. However, care must be taken to avoid losses from overheating. The presence of perchloric acid or other strong oxidizer is necessary for quantitative recovery when analysis is by Rhodamine B colorimetry.

The most widely used analytical techniques for antimony are Rhodamine B colorimetry and the several variations of atomic absorption spectrophotometry. The latter are generally preferred for speed and ease of operation and, in some cases, sensitivity. Both the Rhodamine B and the flame AAS analyses have been incorporated into overall sampling and analytical methods for airborne materials which have total reported coefficients of variation of less than 0.10. These techniques have also been shown to be useful for biological materials.

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Unique new toxicology program at PCPS

Dr. Gary L. Lage has been appointed Director of Toxicology Programs and Professor of Toxicology at the Philadelphia College of Pharmacy and Science (PCPS), according to Dr. John V. Bergen, College President.

Dr. Lage will direct the unique new Bachelor of Science in Toxicology degree program being implemented at PCPS. Students began course work in September, 1979.

The new degree program responds to a major unmet societal need — that is, the need for sufficient manpower to conduct the extensive toxicological evaluations of new and existing chemicals and other substances required by increasingly stringent federal regulations, such as those mandated by the Toxic Substances Control Act (TOSCA).

Moreover, the program is an especially challenging and valuable application of PCPS expertise in the health, life, and physical sciences, and reflects the College's commitment to improving life through health sciences education.

A four-year baccalaureate program, the course work in the first two-and-a-half years, including the intervening summer months, culminates in an extensive period of industrial traineeship incorporated between the third and fourth years. This traineeship is an essential component of the curriculum because of the opportunity it provides to learn about toxicological testing methodology and to obtain practical experience in approaches to safety assessment in a full-time learning-working situation.

Graduates of this program are expected to find excellent career opportunities with agricultural, chemical, cosmetic, drug, food, beverage, and petroleum industries; with governmental agencies; and with research and contract testing laboratories. Major opportunities also exist for post graduate education.