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Determination of Tin in Wines and Sugar Beets by Means of Atomic Absorption Spectroscopy with Hydride Generation

Gian Pietro Molinari,* Marco Trevisan, Patrizia Natali, and Attilio A. M. Del Re

Hydride generation followed by atomic absorption spectroscopy has been used to determine the concentration of tin in 9.6% sulfuric acid solutions. These solutions were obtained by wet oxidation, with nitric and sulfuric acids, of biological samples in a Buchi 445 digestor. The effects of reaction time, hydrochloric acid presence, and pH values of the analyzed solution are outlined. The presence of hydrochloric acid is mandatory; minimum reaction times of 25 s and pH values between 0.9 and 1.25 are recommended. The four buffers potassium acetate, potassium phthalate, potassium chloride, and sodium hydrogen tartrate were compared. The first three are recommended for analysis with different sensitivities. The method allows for the determination of up to 3.3 ppb of total tin and 0.33 ppb of organic tin in the samples.

Over the past few years, quantitative determination of tin in foodstuff has attained considerable interest because of the ever-growing presence of tin both into cans on the market and in the active ingredients of pesticides, such as fentin hydroxide, fentin acetate, fenbutatin, and cyhexatin. These are all tin-based organic compounds. In many countries the law limits the presence of tin in foodstuffs.

The organic fraction of residues has greater toxicological relevance than the inorganic fraction. In fact, a concentration of 250 mg of inorganic tin/kg of food is generally considered a permissible limit in canned food (WHO, 1973), but concentrations as low as 0.15–0.03 mg of Sn/kg of food are the suggested limits for organic tin derivatives (FAO, 1979).

The total amount of tin in foodstuff is generally determined after sample mineralization. To detect organic tin, it is extracted with a slightly polar organic solvent (i.e., trichloromethane, dichloromethane, carbon tetrachloride, benzene, and toluene); the extract is then mineralized after removal of the solvent (Getzendaner and Corbin, 1973). In order to destroy the organic material, either wet oxidation (Corbin, 1970) or dry ashing (Gorsuch, 1970) techniques are available (FAO, 1979; Crosby, 1977).

The most used methods are gravimetric, titrimetric, polarographic, and spectrophotometric techniques (Crosby, 1977; FAO, 1979; AOAC, 1984).

Gravimetric and titrimetric techniques are based on the oxidation of tin, the precipitation of tin(IV) sulfide, and the conversion into oxide (for gravimetry) and into tin(II) chloride (for titrimetry). The oxide produced is weighed while the chloride is titrated by oxidation with iodine. Both methods of analysis are time consuming, demand skillful execution, and provide low sensitivity: 25 mg of total tin/sample is the minimum detectable amount (AOAC, 1984).

Polarographic methods are selective and offer good sensitivity (0.02 μg/mL). The detection limit is 0.1 μg/mL of tin, but anions, phosphate, severely interfere (Bourbon et al., 1982; Guinon and Garcia-Anton, 1985).

Spectrometric techniques are used, such as UV, visible and atomic absorption, with or without tin hydride generation (Crosby, 1977). Colorimetric techniques feature higher sensitivity (1–150 μg of tin) and are much easier to carry out. However, the interference by other metals is greater and the tin must be separated from the matrix. The most used chromogens are dithiol, pyrocathecol violet, phenylfluorone, and quercetin (Crosby, 1977; FAO, 1979). Flame atomic absorption spectrophotometry (AA) provide low sensitivity (1.5 μg/mL of tin in the final solution); heavy metals and anions, namely sulfate, severely interfere (FAO, 1979).

Other studies have described the combination of hydride generation technique with AA spectrophotometry—a technique that allows for lower detection limits (3 ng/mL of tin in the final solution) and more selectivity (Fernan-

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Table I. Instrumental Parameters for Atomic Absorption Measurements and Hydride Generation of Tin

A. Atomic Absorption	
instrument	Varian AA 475 double beam
lamp	hollow cathode, 5 mA
wavelength	286.3 nm
spectral bandwidths	0.5 nm
measurement mode	integrated peak area 25 s
air flow rate	9.5 L/min
acetylene flow rate	1.5 L/min
background correction	deuterium lamp
B. Hydride Generation	
instrument	Varian VGA, Model 65
sweep gas	nitrogen
sweep gas flow rate	2.0 L/min
reaction volume	21–23 mL
purge time	25 s
reaction time	25 s
sample volume	3 mL
buffer	0.7 M acetate
pH reaction medium	1.0–1.2
quartz cell size	length 10.0 cm, diameter 1.2 cm

dez, 1973; Thompson and Thomerson, 1974; Brodie, 1979; Evans et al., 1979; Camail et al., 1983). In these available published works, however, no reference is made to the operating conditions needed for optimal analytic response nor is there mention of pH influence, solvent medium, interfering anions, etc.

This study describes the most suitable conditions in which to form tin hydride, as well as the method to use for the quantitative determination of tin in vegetables and wines, based on AA after generating tin hydride. Three cases are discussed: (1) total tin dosage in liquid samples (wine); (2) total tin dosage in solid samples (sugar beets); (3) separate dosages of organic tin in sugar beet samples treated with tin-based fungicides used to control leaf spot.

EXPERIMENTAL SECTION

Apparatus. Atomic absorption spectrophotometer, Varian Model AA 475; instrumental parameters referred to in Table IA. Vapor generation accessory, Varian Model 65, polypropylene reaction vessel equipped with magnetic stirrer, sodium tetrahydroborate pellet dispenser, drain valve, quartz absorption T-cell, open-ended; nitrogen used to sweep the hydride vapor from reaction vessel to quartz cell, via polypropylene tubing (6-mm i.d.); operating parameters reported in Table IB. Digestor, Buchi Model 445, with 100-mL quartz flasks.

Reagents. Sodium tetrahydroborate pellets 98% (Merck). Buffer solutions: (1) Acetate, 0.7 M (pH 5.7); 67.70 g of potassium acetate dissolved in 600 mL of 0.1 N hydrochloric acid, transferred to a 1000-mL calibrated flask, and brought to volume with water. (2) Chloride, 0.1 M (pH 1.7); 7.45 g of potassium chloride dissolved in 520 mL of 0.1 N hydrochloric acid, transferred to a 1000-mL calibrated flask, and brought to volume with water. (3) Phthalate, 0.1 M (pH 2.5); 20.42 g of potassium acid phthalate dissolved in 776 mL of 0.1 N hydrochloric acid, transferred to a 1000-mL calibrated flask, and brought to volume with water. (4) Tartrate, 0.034 M (pH 3.5); 8.5 g of sodium acid tartrate dissolved in 800 mL of water, transferred to a 1000-mL calibrated flask, and brought to volume with water. Primary standard solution [1 mg of Sn(IV)/mL of sulfuric acid (3.6 N)]: Pure Sn washed with concentrated HCl and then dried; in an Erlenmeyer flask, 1.000 g of washed tin treated with 60 mL of concentrated (96%) sulfuric acid and then heated until white fumes appear; solution allowed to cool and then very carefully diluted with 150 mL of distilled water; 90 mL of concentrated sulfuric acid added; solution transferred to a 1000-mL calibrated flask and brought to volume with water; commercial standard solutions can be used if tin is dissolved in sulfuric acid. Working standard solutions: Concentrated solutions ranging from 0.0043 to 1.2 μg/mL prepared by dilution of the primary solution with 3.6 N sulfuric acid; if kept in an environment free of reducing agents, solutions stable for up to 1 month.

PROCEDURE

Sample Preparation. *1. Total Tin in Liquid Samples.* A 50-mL portion of wine was placed in a porcelain capsule, evaporated at 120 °C, and ashed in muffle kiln at 450 °C. In order to avoid distillation of tin this temperature was achieved slowly. After cooling, the residue ashes were transferred quantitatively with 3.6 N sulfuric acid to a 10-mL calibrated flask. A filtration can be necessary.

2. Total Tin in Solid Samples. A 5-g samples was minced, placed in a flask of the digester, and mineralized.

3. Organic Tin (Del Re et al., 1983). A 50-g sample was placed in a homogenizer with 5 mL of a 20% sodium chloride solution and 90 mL of trichloromethane, blended at high speed for 5 min, quantitatively transferred to a centrifuge tube, washed with 40 mL of a water/trichloromethane mixture (1/1, v/v) and then 20 mL of water, and centrifuged at 3000–3500 rpm for approximately 10 min. The supernatant was transferred to a separatory funnel, and the phases were allowed to separate; the organic phase was filtered, through Whatman No. 1 paper, into a 500-mL round-bottomed flask. The centrifuge residue and the aqueous phase were washed three times with 50-50 and 25-mL portions of trichloromethane respectively, each time centrifuging and separating. To the combined trichloromethane layers was added 5 mL of glacial acetic acid, and the mixtures were evaporated to dryness in a rotary evaporator at less than 40 °C with a vacuum. The residue was quantitatively transferred to the digestor flasks, using 2 mL of the trichloromethane/glacial acetic acid mixture (50/1, v/v) and washing twice with the same mixture. Trichloromethane was removed by evaporation in a rotary evaporator at 40 °C and the residue mineralized.

Mineralization in the Buchi 445 Digestor. One milliliter of concentrated sulfuric acid was added to the samples in the quartz flasks, mounted on the digestor rack, 8 mL of concentrated nitric acid was added to each flask, and the rotation was started. Heating cycles are as follows: For approximatively 4 min, 2-s heating phases in the salt bath at 260 °C were alternated with 10-s cooling phases in order to remove any residual solvent. For approximately 20 min, heating and cooling for 10 and 11 s, respectively, were alternated, making sure that the sample did not char during the immersion in salt bath. Then, the flasks stood immersed in the salt bath at 260 °C until nitric acid was completely removed. After cooling, 5 mL of water was added and the flasks again were dipped in the salt bath kept at 300 °C. Cooling, water addition, and heating to 300 °C were repeated until white fumes appeared and the nitrous fumes disappeared. After cooling, samples were quantitatively transferred to a 10-mL calibrated flask and diluted to the mark with distilled water. Samples prepared in this way can be used for spectrophotometric analysis. Should the tin content be higher than 1.5 μg/mL, they can be diluted with 3.6 N sulfuric acid.

Spectrophotometric Determination of Tin. The spectrophotometer was set to the conditions shown in Table II and the cell heated for approximately 15–20 min. The generator cell system was connected and nitrogen introduced and allowed to circulate. The instrument was set to zero. The container was filled with sodium borohydride pellets, and the apparatus and cell were checked

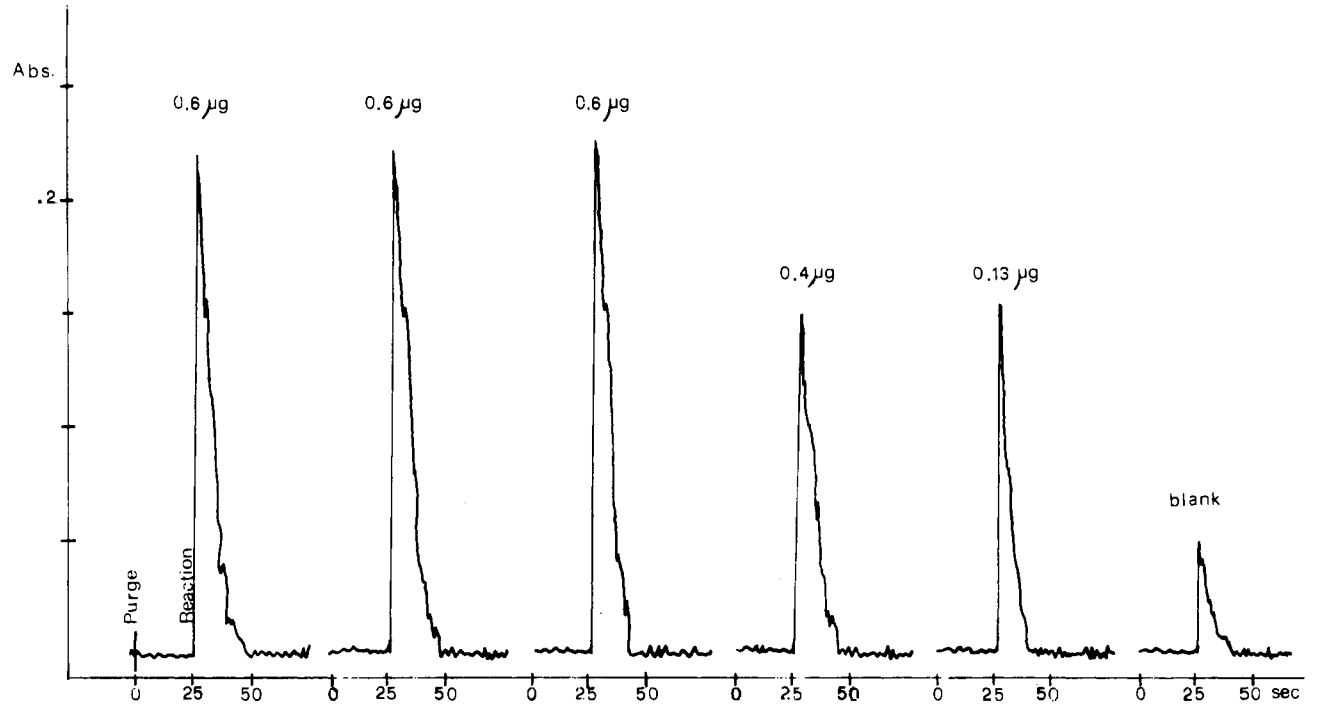

Figure 1. Absorbance as a function of time for different amounts of Sn(IV) in phthalate buffer.

Table II. Areas Integrated from 0 to 25 s of the Absorbance/s for Different Times of Air Purge of 0.72 μg of Sn(IV) in Acetate Buffer Solution

time, s	absorbance[a]	time, s	absorbance[a]
20	1.083 ± 0.02875	30	1.090 ± 0.00949
25	1.066 ± 0.01887	35	1.036 ± 0.02466

[a] Average of three determinations ± SD.

by placing 20 mL of buffer and known volumes of 3.6 N sulfuric acid in the reaction vessel (analytical blank). The vessel was closed and allowed to stir so as to vent air from the system (purge for 25 s). The pellet was dropped into the reaction vessel and relative absorbance measured. Absorbance was measured on peak area. Peak area measurements are given in absorbance seconds; this readout mode enables one to obtain the peak area of short-duration signals from a vapor generation equipment.

The same procedure was used for both standard samples and analytical ones. Before and after every analysis, the reaction vessel must be washed thoroughly. The blank should be checked every 10–15 determinations. The absorbance range of the analytical blank is 0.25–0.5 (Table IV). After 25–30 samples, the connecting tube between reactor and cell should be cleaned with bidistilled water and allowed to dry, so as to prevent residue deposit. Sodium tetrahydroborate pellets were employed as reducing agent since they are stable and the time of analysis is very short (Brodie, 1979). As suggested by Evans et al. (1979), the quartz cell should be heated and preconditioned before the analysis, by repeated applications of the most concentrated working standard solution, until a constant response, in order to ensure that the catalytic film inside is homogeneous. One cell should be used for each element; it should be replaced after approximately 400 analyses: in fact, since it is impossible to exactly restore the catalytic film, continuous use reduces sensitivity.

RESULTS AND DISCUSSION

The analyzed wine was taken from commercially sold wines stored in iron or aluminum cans closed by tin band. The sugar beets were taken from a field trial. Six different treatments with tin-based fungicides and an untreated one

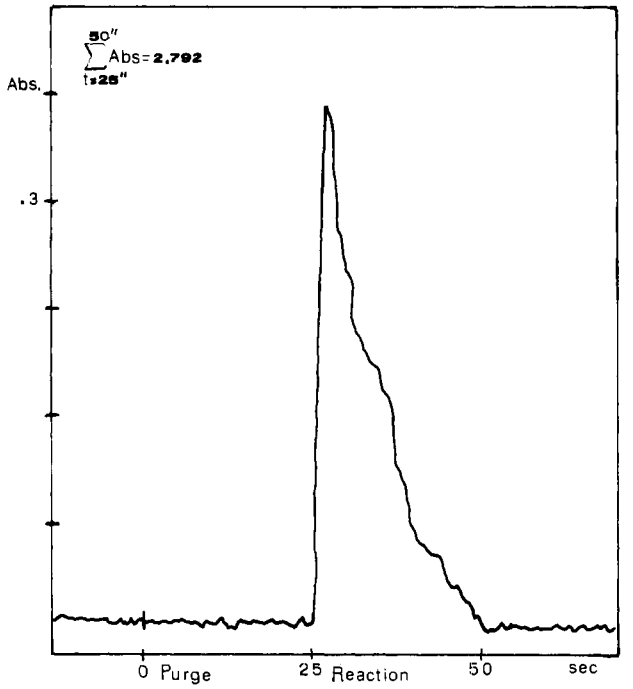

Figure 2. Absorbance as a function of time during the determination of 1.2 μg of Sn(IV) in phthalate buffer.

were compared following a Latin square scheme 7 × 7 (Table VIII). Ten tap roots picked at random from each plot were used for each analytical sample. Subsamples, taken from a triturate of the 10 tap roots, were used for analysis. More details are published elsewhere. (Molinari et al., 1984).

(a) Setting Operating Conditions. *Measurement Mode and Reaction Time.* Peak heights (given in absorbance units) were inadequate for tin determination. As shown in Figure 1, peaks heights were nearly the same for samples with 0.13–0.4 μg of tin. Plots of absorbance during the hydride development (Figure 2) showed reaction times of 23–25 s, with peak areas (given in absorbance × seconds) well related to tin concentrations. Therefore, the absorbance/time plots were integrated with an integration time

Table III. Areas Integrated from 0 to 25 s of the Absorbance/s of 1.2 μg of Sn(IV) in Chloride Buffer Solution and 0.6 μg of Sn(IV) in Acetate Buffer at Different pH Values[a]

pH	chloride	pH	acetate
0.75	2.6877 ± 0.5379	1.00	1.1193 ± 0.0263
0.90	3.1592 ± 0.0801	1.25	0.8947 ± 0.0232
1.05	3.0810 ± 0.0397	1.50	0.9568 ± 0.0682
1.15	2.6815 ± 0.1250		
1.25	2.1840 ± 0.1394		

[a] Average of four determinations ± SD.

Table IV. Areas Integrated from 0 to 25 s of the Absorbance/s for Different Amounts of Sn(IV) in Three Buffer Solutions at Various pH Values and Regression Lines for Each Buffer[a]

Sn(IV), μg	chloride, pH 1.05	phthalate, pH 1.00	acetate, pH 1.20
0.00	0.4935 ± 0.0269	0.3572 ± 0.0274	0.2462 ± 0.0516
0.13	0.5937 ± 0.0098	0.5830 ± 0.304	0.3562 ± 0.0290
0.40	1.1867 ± 0.0234	1.0760 ± 0.0593	0.7045 ± 0.0180
0.60	1.5855 ± 0.1045	1.5452 ± 0.0588	0.8950 ± 0.0622
1.200	2.8655 ± 0.0781	2.7827 ± 0.0150	1.6402 ± 0.0225
	param of regression: $A = a + b \times$ concn		
	$a = 0.3734$	$a = 0.3141$	$a = 0.2231$
	$b = 2.1102$	$b = 2.0381$	$b = 1.1704$
	$r = 0.9871$	$r = 0.9957$	$r = 0.9892$

[a] Averages of four determinations ± SD.

Table V. Influence of Sulfuric Acid Concentration on Response (AU) of 0.6 μg of Sn(IV) at pH 1.0

mequiv/ mL acid	summation of absorbances	av ± SD
0.17	1.488, 1.710, 1.642, 1.802	1.660 ± 0.066
0.33	1.425, 1.476, 1.594, 1.586	1.520 ± 0.042
0.47	1.170, 1.106, 1.082, 1.160	1.130 ± 0.021

Table VI. Total Tin Concentrations (ppb) in Canned Wines of the Market

type of wine	total tin	type of wine	total tin
white	nd[a]	red	10.1
white	nd	red	18.1
white	3.6	red	11.9
white	nd		
white	nd		

[a] nd = not detected. I.e., tin content less than the detection limit (3.3 ppb).

of 25 s. Shaking samples for only 20 s was not enough to give reproducible data, whereas longer shaking times were suitable (Table II). A purging time of 25 s was selected, equal to integration time.

Reaction Media. Four buffers, tartrate, chloride, acetate, and phthalate, were tested to maintain an optimal pH value in the final solution, with various volumes (1–3 mL) of sulfuric acid (9.6%). Although the instrument itself can easily detect low amounts of tin (0.0036 μg), in different conditions different detection limits can be attained. The sodium hydrogen tartrate buffer was tested, but it proved ineffective since it did not react with sodium borohydride. This is in agreement with what Brodie (1979) and Evans et al. (1979) suggest: hydrochloric acid should always be present in the reaction medium. Hydrochloric acid based buffers were selected for use: 0.1 M potassium chloride, 0.1 M potassium phthalate, 0.7 M potassium acetate.

Potassium Chloride Buffer. The pH values that produced the highest absorbance were between 0.9 and 1.05 (Table III). Both higher and lower values reduced responses and increased their variability. So, the standard average coefficients of variation are 20% at pH 0.75, 1.3% at pH 1.05, and 6.4% at pH 1.25.

Potassium Acetate Buffer. The response was lowered (20%), the pH was increased from 1.0 to 1.5, yet the variability did not increase a lot, namely from 2.3% to 7.11%. The best range is between pH 1.0 and 1.25.

Potassium Phthalate Buffer. The pattern of the phthalate buffer is similar to that of the acetate buffer one; hence, no data are presented here. Good reproducibility is shown in Figure 1.

Detection values, expressed as concentrations in biological samples, are 0.0036 μg/mL with chloride buffer and 1 mL of 3.6 N sulfuric acid solution, 0.0018 μg/mL with phthalate and 2 mL, and 0.0012 μg/mL with acetate and 3 mL.

Calibration data are given in Table IV. The phthalate and acetate buffers give better correlation coefficients than the chloride one, but sensitivities are somewhat less. That is due to the increased amounts of sulfuric acid present, that thereby significantly reduce response even at pH values best for hydride generation (Table V).

(b) Application to Analysis. In Table VI, the results of analyses on some market wines are reported.

In Table VII are reported the average concentrations as well as the minimum and maximum detected ones for organic and total tin in surgar beets. Concentrations are averages of seven independent subsamples, each taken from a batch of 10 tap roots. A comparison was made between three sections within the general area treated with usual and double doses of fentin-based pesticide and one untreated (Table VIII) following a Latin square scheme, 7 × 7. More details are published elsewhere (Molinari et al., 1984).

CONCLUSION

The mineralization technique used here has some advantages over known methods: (1) smaller amounts of acid are used, thus reducing risks of samples contamination;

Table VII. Organic and Total Tin (ppb) in Tap Roots of Sugar Beets Treated with Trifeniltin-Based Fungicides and Picked 20 and 31 Days after Treatment

	1st picking						2nd picking					
	organic			total			organic			total		
trial	av[a]	min	max	av	min	max	av	min	max	av	min	max
1	13	3	25	28	nd[b]	66	7	nd	16	52	14	92
2	15	8	26	46	26	101	13	3	23	74	40	88
3	9	4	23	22	8	39	6	nd	11	63	8	213
4	13	1	24	46	9	82	8	3	16	55	9	102
5	7	1	11	29	nd	61	8	2	15	38	7	78
6	15	4	28	76	44	148	16	3	40	44	31	69
7	4	2	7	41	7	100	6	nd	21	39	nd	100

[a] Average of seven samples. [b] nd = not detected. I.e., tin content less than the detection limit (0.33 ppb for organic tin and 3.3 ppb for total one).

Table VIII. Treatments and Doses (g of Sn/h) in the Field Trial

trial	treatment[a]	doses
1	nuarimol + fentin wp	48.2
2	nuarimol + fentin wp	96.4
3	nuarimol + fentin sc	48.2
4	nuarimol + fentin sc	96.4
5	fentin wp	48.2
6	fentin wp	96.4
7	untreated	0.0

[a] Key: wp, wettable powder; sc, concentrated solution.

(2) samples are kept at high temperature for shorter times, lessening tin losses by volatilization (Gorsuch, 1970); (3) refluxing in the digestor condenser lessens tin volatilization; (4) chances of charring organic matters are minimized; (5) time of mineralization is shortened, thus increasing analysis throughput; (6) the small amount of sulfuric acid used (1 mL) allows for greater spectrophotometric sensitivity.

The three buffers, chloride, acetate, and phthalate, can be recommended for the quantitative determination of Sn(IV) by using 20 mL of buffer and 1, 2, and 3 mL, respectively, of sulfuric acid solutions (9.6%), obtained by wet oxidation of biological samples. The 0.1 M potassium chloride buffer in 0.052 M hydrochloric acid is suitable for solutions containing 0.17 mequiv/mL of sulfuric acid and allows for the determination of 0.0036 μg/mL of Sn in the sample. The 0.1 M potassium phthalate buffer in 0.0776 M hydrochloric acid is suitable for solutions containing 0.33 mequiv/mL of sulfuric acid and allows for the determination of 0.0018 μg/mL of Sn. The 0.7 M potassium acetate buffer in 0.06 M hydrochloric acid is suitable for solutions containing 0.47 mequiv/mL of sulfuric acid and allows for 0.0012 μg/mL of Sn to be determined into the sample.

No buffer solution can be recommended as appropriate to general use because the choice depends on the total amount of sulfuric acid in the final solution, produced by the biacid (nitric and sulfuric) mineralization of biological samples. Analyses made with the acetate buffer were the most sensitive.

The biacid mineralization and AA spectrophotometry after hydride generation are combined in a procedure precise, easy, quick, and useful for analyzing many samples.

Registry No. Sn, 7440-31-5; KOAc, 127-08-2; KCl, 7447-40-7; potassium phthalate, 877-24-7; nuarimol, 63284-71-9; fentin, 668-34-8; sodium hydrogen tartrate, 526-94-3.

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