Method no.:	ID-225SG
Target concentration:	0.1 mg/m^3
Procedure:	The sample is collected on an MCE filter (0.8 µm) and analyzed by atomic absorption with a graphite furnace.
Recommended sampling time and sampling rate:	1-2 L/min for a total air volume of 200 L
Reliable quantitation limit:	0.1 µg/mL
Status of method:	Partially evaluated method. This method has been subjected to established evaluation procedures of the Methods Development Team and is presented for information and trial use.

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Commercial manufacturers and products mentioned in this method are for descriptive use only and do not constitute endorsements by USDOL-OSHA. Similar products from other sources can be substituted.

1. Introduction

1.1 Scope

This method describes the collection and analysis of airborne triphenyltin hydroxide. It is applicable for time-weighted average exposure evaluations. The analysis is based on the technique of graphite furnace atomic absorption.

1.2 Uses

A major use of Triphenyltin Hydroxide is as a Fungicide.

1.3 Physical and Chemical Properties

$(C_6H_5)_3SnOH$

mol. wt.	-	367.02	white solid
% Sn	-	32.34%	density (20)
m.p.	-	22°C	

2. Range and Detection Limit

2.1 The lower analytical limit for TPTH is 0.1 µg/mL.

2.2 This is based on a detection limit of .02 µg/mL for graphite furnace analysis of Sn as TPTH in ethanol/ammonium hydroxide.

3. Precision and Accuracy

3.1 Precision	S = .030
3.2 Coefficient of Variation	CV=.032
3.3 Recovery	Average mean recovery = 1.032

The above are based on recovery data for eighteen glass fiber filters, spiked with TPTH in ethanol/ammonium hydroxide at ½, 1, and 2 times the PEL based on a 200 liter air volume and 0.1 mg/m³ PEL. Six samples were spiked at each level. Refer to addendum for data on recovery study.

4. Interferences

Other organotins would interfere if they are soluble in ethanol.

5. Sampling Procedure

The sample is collected on a glass fiber filter (0.8 µm, 37-mm diameter) at a flow rate of 1-2 L/min.

The recommended air volume is 200 L.

The sample cassettes are plugged, sealed with OSHA tape, labeled, and sent to the laboratory for analysis as soon as possible.

6. Analytical Procedure

6.1 Apparatus

6.1.1 Sample collection

Personal sampling pumps
AA sampling cassettes as needed

6.1.2 Sample analysis

Atomic absorption spectrophotometer
HGA graphite furnace
Electrodeless discharge lamp for Sn
Laboratory glassware

6.2 Reagents

All reagents should be ACS, analyzed reagent grade or better.

6.2.1 Ethanol

6.2.2 Ammonium hydroxide

6.2.3 Stock triphenyltin hydroxide

6.3 Safety precautions

6.3.1 Use caution when handling ammonia and organotins. TPTH is a toxic compound. Always wear rubber gloves and work under a fume hood. Waste organics should be collected in a suitable marked container and properly disposed of in the organic laboratory.

6.3.2 Avoid using glassware with chips or sharp edges. Never pipette by mouth.

6.3.3 Before using the graphite furnace, the analyst should read the operator's manual and be familiar with the equipment. Ensure that the furnace tube is properly seated, the contact rings are clean, and that cooling water is circulating. Do not exceed an atomization temperature of 2750 degrees. Heating or cooling problems could cause the tube to explode on atomization.

Always wear safety glasses and never look at the tube during atomization. Even during normal firing, the intense light is harmful to the eyes. Be aware of the high current supplied to the furnace through the copper cables; check that the insulating cover is in place over the terminals. Since toxic substances are vented by the furnace, a fume hood must be in operation over the furnace.

6.3.4 Observe care with respect to harming the equipment. Do not operate an EDL below its recommended wattage. Be certain that the purge air is circulating when using the background corrector. Do not operate any equipment without first reading its instruction manual.

6.4 Glassware Preparation

Clean the 125 mL conical flasks and the 50 mL volumetric flasks by refluxing with 1:1 nitric acid. Thoroughly rinse all glassware with D.I. water, invert, and allow to dry.

6.5 Standard Preparation

6.5.1 The procedure is to analyze the tin in triphenyltin hydroxide. Prepare standards by diluting stock TPTH ethanol/ammonia hydroxide assuming a theoretical tin content of 32.34%.

6.5.2 Prepare stock solution by weighing 30.92 mg TPTH into a 100 mL volumetric flask, diluting to volume with 90% ethanol, 10% ammonium hydroxide and mix well. This is equivalent to 100 ppm Sn.

From this, a 10 ppm Sn stock solution is made by one serial 10-fold dilution.

6.5.3 Working standards are prepared from the 10 ppm Sn stock as follows:

Prepared std. (ppm)	Std, soln. used (ppm)	Aliquot (mL)	Dil. vol. (mL)
1.0	10.0	5	50
0.4	10.0	2	50
0.2	1.0	10	50
0.1	1.0	5	50
0.04	1.0	2	50
0.02	1.0	1	50

6.6 Sample Preparation

Transfer the glass fiber filter to a clean 125 mL conical flask. Add 10 mL of 90% ethanol, 10 % ammonium hydroxide to each beaker and sonicate for 5 minutes. Transfer contents to 50 ml volumetric flask. Dilute to volume with ethanol/ammonium hydroxide and invert several times to insure thorough mixing.

6.7 Analysis

6.7.1 The analysis is done by graphite furnace/AA. The instrumental parameters for determining Su in ethanol/ammonium hydroxide are as follows:

Atomic Absorption Units	
Sn wavelength	224.6 nm
Integ. time	7 sec.
Slit width	.7 low
Signal	Pk. ht.
Mode	Abs
BGC	On

Furnace Parameters

Step	Temperature	Ramp time	Hold time	Interval flow
Dry	100°C	20 S	20 S	100 mL/min
Char	800°C	20 S	20 S	100 mL/min
Atomize	2500°C	0 S	8 S	60 mL/min

(With HGA 500, program -10 chart and 0 read in atomization step)

6.7.2 Parameters are adjusted so that the 1.0 ppm standard gives a near full-scale deflection on the chart. The entire series of standards is run at the beginning and end of the analysis; a standard is also run after every fourth or fifth sample during the analysis.

6.8. Calculations

6.8.1 The OSHA Auto AA program is used for the calculations.

6.8.2 Results are reported as mg/m^3

Addendum I

A recovery study of TPTH from glass fiber filters by desorption in ethanol/ammonium hydroxide was done.

30.92 mg of TPTH was weighed into a 100 mL volumetric, diluted to volume with ethanol/ammonium hydroxide and mixed. Assuming that TFTH is 32-34% Sn, this is 100 ppm Sn as TPTH.

Six glass fiber filters were spiked at each level = ½, 1, and 2 times the PEL based on a 200 liter air volume and 0.1 mg/m³ PEL. The spikes were made as follows:

Std used (ppm Sn)	Spike Vol (ul)	Sn (g)	PEL (multiple)
100	100	10	1/2 ×
100	200	20	1 ×
100	400	40	2 ×

The filters were then desorbed in ethanol/ammonium hydroxide, diluted to 50 ml, and run on the graphite furnace as described in Section 6.7. The mean standard deviation and coefficient of variation for the recovery at each level using the OSHA "Precision and Accuracy Data" program =

PEL (multiple)	Mean Recovery	Std. Dev.	CV1
1/2 ×	0.989	.040	.040
1 ×	1.036	.012	.011
2 ×	1.072	.038	.036

The mean recoveries were then pooled =

Average mean recovery	=	1.032
Standard Deviation	=	.030
Coefficient of variation	=	.032

Addendum II

A recovery study of the loss of TPTH on glass fiber due to volatility was done. A 200 µL spike of 100 ppm Sn as TPTH was placed on six filters and attached to six personnel sampling pumps (calibrated at 2 liters per minute). 200 liters of air (100 minutes) were drawn through each. The filters were then placed into 50 mL volumetric flasks, diluted to volume with ethanol/ammonium hydroxide and run on the graphite furnace as described in Section 6.7. The recoveries are as follows:

Sample	Found	Theor.	Found/Theor.
Test-1	20.98	20.00	1.049
Test-2	20.94	20.00	1.047
Test-3	21.65	20.00	1.083
Test-4	22.49	20.00	1.125
Test-5	20.76	20.00	1.038
Test-6	21.5	20.00	1.080