Stannous-2-ethyl hexanoate as Sn

Method no.:	ID-221SG
Target concentration:	0.1 mg/m^3
Procedure:	Samples are collected by drawing a known volume of air through glass fiber filters. Samples are digested with aqua regia and analyzed by atomic absorption/graphite furnace.
Recommended sampling time and sampling rate:	1-2 L/min for a total air volume of 100 L
Reliable quantitation limit:	30 µg/m^3
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 stannous-2-ethyl hexanoate on glass fiber filters. The analysis is based on the digestion of the filters with aqua regia and the analysis of Sn by atomic absorption spectroscopy.

The permissible exposure limits value (TLV) for organotin compounds has been adopted at 0.1 mg/m^3 as Sn in the workplace environment.

1.2 Uses

Stannous-2-ethyl hexanoate [$Sn(C_8H_{15}0_2)_2$] is also known as stannous-2-ethylhexoate, stannous octoate, and tin octoate. It is used as polymerization catalyst for urethane foams, a lubricant, and as a stabilizer for transformer oils.

1.3. Physical and Chemical Properties

Appearance:	Light yellow liquid
Sp.Gr.:	1.25
% Sn:	29.33
Mol. Wt.:	404.7
B.P.:	Decomposes @ 202 °C
F.P.:	142 °C

2. Range and Detection Limit

2.1 A qualitative detection limit was determined based on the OSHA AA program using 12 Sn standards ranging from 0.5 to 5.0 ppm Sn.

2.2. The OSHA AA program indicates that the qualitative detection limit should be 0.28 ppm.

3. Precision and Accuracy

3.1 Three glass fiber filters at each of two levels (1× and 2× the target level) were spiked with 10 and 20 µL of 1270 ppm stannous-2-ethyl hexanoate (as Sn) based on an air volume of 100 L and the OSHA standard of 0.1 mg/m^3. For these 6 samples, the average recovery was 98.5%. The standard deviation of the 12.7 µg spike was 0.556 and the standard deviation of the 25.4 µg spike was 0.958.

4. Interferences

4.1 Other organotin compounds will interfere if they are digestible in aqua regia.

4.2 Inorganic tin compounds will interfere.

5. Sampling Procedure

5.1 The sample is collected on a glass fiber filter with a backup pad.

5.2 The sample cassettes are plugged, sealed with an OSHA form 21, and then sent to the laboratory for analysis as soon as possible.

6. Analytical Procedure

6.1 Apparatus

6.1.1 Sample Collection

Personal sampling pumps, glass fiber filters, and sampling cassettes with back up pads.

6.1.2 Sample Analysis

Laboratory glassware to include volumetric flasks, 125 mL Erlenmeyer flasks, assorted pipettes, atomic absorption spectrophotometer, and Sn electrodeless discharge lamp.

6.2 Reagents - All reagents should be ACS analyzed reagent grade or better.

6.2.1 Nitric Acid

6.2.2 Hydrochloric Acid

6.2.3 Stock Stanneous-2-ethyl hexanoate

6.3 Safety Precautions (Reference 8.3)

6.3.1 Use caution when handling all organotin compounds, HCl, and HNO_3. The acids will cause severe burns and are harmful if inhaled. Waste acids should be diluted with water and poured down an acid drain followed by plenty of water.

6.3.2 Always use rubber gloves and, work in a fume hood. Observe warning labels on reagent bottles.

6.3.3 Avoid using glassware with chips, stars, or sharp edges. Never pipette any of these materials by mouth.

6.3.4 Before using the atomic absorption spectrophotometer, the analyst should read the operator manual and be familiar with the equipment.

6.3.5 Always wear safety glasses and never look at the flame; the intense light is harmful to the eyes.

6.3.6 Since toxic substances are vented by the flame, a fume hood must be in operation over the flame.

6.3.7 Observe care with respect to harming the equipment. Do not operate an EDL below its recommended wattage. Do not operate any of the equipment without first reading its instruction manual.

6.4 Glassware Preparation

6.4.1 All glassware must be scrupulously cleaned with concentrated HCl by soaking for several hours or overnight and then rinsing several times with deionized water.

6.5 Standard Preparation

6.5.1 A stannous-2-ethyl hexanoate spiking solution should be prepared in the following manner. Add about 0.2 grams of stannous-2-ethyl hexanoate neat liquid to 40 mL toluene in a 50 mL volumetric flask and dilute to volume with toluene. This will give a spiking solution of about 1200 ppm stannous-2-ethyl hexanoate as Sn, assuming a theoretical tin content of 29.33%. Spike with 10 to 50 µg of this solution onto a glass fiber filter and let dry. This can be taken through the digestion and analytical steps along with the samples as a check on the accuracy of the preparation and analytical steps.

6.5.2 Prepare the standards using an inorganic Sn stock standard in 10% HCl. Prepare a 100 ppm intermediate stock solution by diluting 10 mL of the 1000 ppm stock Sn to 100 mL with 10% HCL in a volumetric flask.

6.5.3 Prepare a 10 ppm standard by diluting 10 mL of the 100 ppm Sn standard to 100 mL with 10% HCl.

6.5.4 Prepare five working standards from the 100, 10, and 1.0 ppm standards as follows:

Std Prepared	Std Soln Used	Aliquot	Dil Vol
5.0 ppm	100.0 ppm	5 mL	100 mL
2.0 ppm	10.0 ppm	20 mL	100 mL
1.0 ppm	10.0 ppm	10 mL	100 mL
0.5 ppm	10.0 ppm	5 mL	100 mL
0.2 ppm	1.0 ppm	20 mL	100 mL

The working standards are diluted to volume with 10% HCL.

6.6 Sample Preparation

6.6.1 Transfer the glass fiber filters to separate 125 mL Erlenmeyer flasks.

6.6.2 Pipette 9 mL of concentrated hydrochloric acid into each flask, swirl, and add 2 mL nitric acid.

6.6.3 Ash the sample until nearly dry and allow to cool.

6.6.4 Quantitatively transfer the sample to a clean 10 mL volumetric flask, add 1.0 mL concentrated HCl, and dilute to volume with deionized water. This will make the final solution 10% HCl.

6.6.5 If subsequent dilutions are necessary, make them with 10% HCl.

6.7 Analysis

6.7.1 The analysis is done by atomic absorption spectrophotometry. Instrument parameters for determining tin in 10% HCl are as follows:

Atomic absorption spectrophotometer parameters

Sn wavelength	224.6 nm
Integration time	3-10 seconds
Slit width	0.2 nm
Range	UV
Flame	Hydrogen-Air

6.7.2 All calibration standards are run at the beginning and at the end of the analysis; a standard is also run after every four samples during the analysis.

6.8 Calculations

6.8.1 Either the OSHA Auto-AA program or the Auto-Colorimetric program is used for the calculations.

6.8.2 Results are reported as mg/m^3 Sn based on the total micrograms of organotin (as Sn) and the air volume.

7. Recovery Study

A recovery study was done of stannous-2-ethyl hexanoate from glass fiber filters. 0.2165g stannous-2-ethyl hexanoate was added to a 50 mL volumetric flask and diluted to the mark with toluene. This gave a 1270. µg/mL stannous-2-ethyl hexanoate (as Sn) spiking solution.

Six glass fiber filters were spiked with this 1270. µg/mL solution. The spiked filters were air dried on a test tube rack and placed on a backup pad in a plastic cassette. Three filters were spiked with 10 µL and 3 filters were spiked with 20 µL of the spiking solution which put 12.7 µg as Sn on 3 filters and 25.4 µg as Sn on 3 additional filters. This corresponded to 1× and 2× the TLV, assuming 100 liters of air was sampled. A seventh cassette, glass fiber filter and backup pad were used as an air blank. All 7 samples were treated as described in this procedure. The samples were then analyzed by atomic absorption and the statistical data are shown as follows:

Stannous-2-Ethyl Hexanoate Recovery Study Results

1×	GFF-1 = 12.46 µg	
1×	GFF-2 = 12.46 µg	
1×	GFF-3 = 13.64 µg	
	AVE. = 12.85 µg	f/t = 1.012

Standard Deviation = 0.556

2×	GFF-1 = 24.41 µg	
2×	GFF-2 = 23.13 µg	
2×	GFF-3 = 25.48 µg	
	AVE. = 24.34 µg	f/t = 0.961

Standard Deviation = 0.961

8. References

1. "Determination of Butyl Organotin Compounds in Air Samples by AAS-Graphite Furnace," Standard Test Methods, Method No. AA-62, M&T Chemicals, Inc., June 6, 1984.

2. Condensed Chemical Dictionary, G.G. Hawley, 10th Edition, p. 965, 1981.

3. Bis(Tributyltin) Oxide, OSHA Laboratory Method, Method No. ID-102-SG, Unpublished, 1984.