

Dibutyltin Dilaurate as Sn

Method no.: ID-218SG

Target concentration: 0.1 mg/m³

Procedure: Samples are collected by drawing a known volume of air through a mixed cellulose ester filter. Samples are extracted with toluene and analyzed by atomic absorption/graphite furnace.

Recommended sampling time and sampling rate: 500 min at 1 L/min (500 L)

Reliable quantitation limit: 5 µg/m³

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.

Date: April 2003

Methods Development Team
Industrial Hygiene Chemistry Division
OSHA Salt Lake Technical Center
Sandy UT 84070

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 dibutyltin dilaurate (DBTDL). It is applicable for time-weighted average exposure evaluations. The analysis is based on the technique of graphite furnace atomic absorption.

1.2 History

Initially, AA filters were spiked with dibutyltin dilaurate in the range of 1/2× and 1× the PEL and were then analyzed according to the NIOSH method for non-volatile organotins. However, recovery was low and imprecise, ranging from 20 to 60%. It was suspected that loss occurred due to the harsh ashing required and DBTDL's volatility at the temperatures used in the ashing. When the spiked filters were extracted with toluene and the extract run directly on the graphite furnace, recovery was quantitative.

1.3. Uses

Dibutyltin dilaurate is used as a catalyst for curing silicones, as a stabilizer for polyvinyl chloride resins, as a corrosion inhibitor, and in veterinary use, to treat tapeworms in chickens.

1.4. Physical and Chemical Properties



mol wt. - 631.55	clear, yellow liquid
% Sn - 18.79	density (20) - 1.4683
m.p. - 27°C	

2. Range and Detection Limit

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

2.2 This is based on a detection limit of .02 mg/mL for graphite furnace analysis of Sn as DBTDL in benzene.

3. Precision and Accuracy

3.1. Precision	S = .033
3.2 Coefficient of Variation	CV= .030
3.3 Recovery	Average mean recovery = 1.08

The above are based on recovery data for eighteen AA filters, spiked with DBTDL in toluene at 1/2, 1, and 2× the PEL based on a 500 L 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 toluene.

5. Sampling Procedure

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

The recommended air volume is 500 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 Toluene

6.2.2 Stock dibutyltin dilaurate

6.3 Safety Precautions

6.3.1 Use caution when handling toluene and organotins. Toluene is a central nervous system depressant and irritant of the respiratory tract and mucous membranes. Dibutyltin dilaurate 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

6.4.1 The 250 ml Phillips beakers are cleaned by refluxing with 1:1 nitric acid. All glassware is thoroughly rinsed with D.I. water, inverted, and allowed to dry.

6.5 Standard Preparation

6.5.1 The procedure is to analyze the tin in dibutyltin dilaurate. The standards are prepared by diluting stock DBTDL in toluene and assuming a theoretical tin content of 18.8%.

6.5.2 Prepare a stock solution by weighing 1.064 g DBTDL into a 100 mL volumetric flask, diluting to volume with toluene, and mixing well. This is equivalent to 2,000 ppm Sn.

From this a 5 ppm Sn stock solution is made by two serial 20-fold dilutions.

6.5.3 Working standards are prepared from the 5 ppm Sn stock as follows:

Prepared std.	Std. soln. used	Aliquot	Dil. vol.
1.0 ppm	5.0 ppm	10 mL	50 mL
0.5 ppm	5.0 ppm	5 mL	50 mL
0.2 ppm	1.0 ppm	10 mL	50 mL
0.1 ppm	1.0 ppm	5 mL	50 mL
.05 ppm	0.5 ppm	5 mL	50 mL
.02 ppm	0.2 ppm	5 mL	50 mL

6.6 Sample Preparation

6.6.1 Transfer the AA filter to a clean 125 mL conical beaker. Wash the filter with several 5 ml portions of toluene and quantitatively transfer into a 25 mL volumetric flask. Dilute to volume with toluene.

6.7 Analysis

6.7.1 The analysis is done by graphite furnace/AA. The instrumental parameters for determining Sn in toluene are as follow:

Atomic absorption unit:

Sn wavelength	286.3
integ. time	10 sec.
slit width	.2 low (3 on P.E. 306)
signal	Pk.Ht. (TCI an P.E. 306)
mode	ABS
BGC	off

Furnace parameters:

step	temperature	ramp time	hold time	internal flow
dry	100°C	50 s	40 s	60 mL/min
char	600°C	50 s	30 s	60 mL/min
atomize	2700°C	0 s	9 s	10 mL/min

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

Chart = 10 mV scale, 20mm/min.

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 Colorimetric program is used for the calculations.

6.8.2 Results are reported as mg/m³ Sn.

Addendum

A recovery study of DBTDL from AA filters by desorption in toluene was done.

5.319g DBTDL was weighed into a 500 ml volumetric, diluted to volume with toluene, and mixed. Assuming the DBTDL is 18.8% Sn, this is 2,000 ppm Sn as DBTDL. From this, 50 ml was diluted to 100 ml for a 1,000 ppm Sn standard.

Six AA filters were spiked at each level = 1/2, 1, and 2X the PEL based on a 500 L air volume and 0.1 mg/m³ PEL. The spikes were made as follows:

Std used (ppm Sn)	Spike Vol (µL)	Sn (µg)	PEL (multiple)
1,000	25	25	1/2×
1,000	50	50	1×
2,000	50	100	2×

The filters were then desorbed in toluene, 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×	1.092	.045	.042
1×	1.039	.024	.023
2×	1.095	.020	.022

The mean recoveries were then pooled =
Average Mean Recovery = 1.08
Standard Deviation = .033
Coefficient of variation .030