Methyl Tin Mercaptide

Method no.:	ID-219SG
Target concentration:	0.1 mg/m^3
Procedure:	Samples are collected by drawing a known volume of air through an impinger containing 15 mL of butyl cellosolve. Samples are analyzed by atomic absorption/graphite furnace.
Recommended sampling time and sampling rate:	150 min at 1 L/min (150 L)
Reliable quantitation limit:	10 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 methyl tin mercaptide (MTM). It is applicable for time-weighted average exposure evaluations. The analysis is based on the technique of graphite furnace atomic absorption.

1.2 Physical and Chemical Properties

Appearance - clear yellow to light amber liquid
Specific gravity - 1.02 (at approx. 250 °C)
Refractive index - 1.5085 (at approx. 250 °C)
% Sn - 11%
Decomposes to form tin oxides and sulfur dioxide
Boiling point - *not distillable*

2. Range and Detection Limit

2.1 The lower analytical limit for MTM 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 MTM in butyl cellosolve.

3. Precision and Accuracy

3.1 Precision	S = .020
3.2 Coefficient of Variation	CV = 0.023
3.3 Recovery	Average mean recovery = .874

The above are based on recovery data for eighteen impingers, spiked with MTM in butyl cellosolve at .5×, 1×, and 2× the PEL based on a 150 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 butyl cellosolve.

5. Sampling Procedure

The sample is collected in an impinger containing 15 mL butyl cellosolve at a flow rate of 1 L/min.

The recommended air volume is 150 L.

The impingers are capped, 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
Impingers 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 Butyl cellosolve (2-Butoxyethanol)

6.2.2 Stock methyl tin mercaptide

6.2.3 Stock tin (1000 ppm), Scientific Products, Fisher or equivalent

6.3 Safety precautions

6.3.1 Use caution when handling butyl cellosolve and organotins. Methyl tin mercaptide is a toxic compound. Always wear rubber gloves and work in 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 Clean the 50 mL volumetric flasks by refluxing with 1:1 nitric acid. Thoroughly rinse all glassware with deionized water, invert, and allow to dry.

6.5 Standard preparation

6.5.1 The procedure is to analyze the tin in methyl tin mercaptide. Prepare a 10 ppm Sn stock solution from two serial 10-fold dilutions of 1000 ppm stock.

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

Prepared std.	**Std. stock used**	**Aliquot**	**Dil. vol.**
2.0 ppm	10 ppm	10 mL	50 mL
1.0 ppm	10 ppm	5 mL	50 mL
0.2 ppm	1 ppm	10 mL	50 mL
0.1 ppm	1 ppm	5 mL	50 mL
0.04 ppm	1 ppm	2 mL	50 mL

6.6 Sample preparation

Measure the volume of the impinger and record a sample volume. Transfer into sampling cup and analyze directly on graphite furnace. If off-scale, make all dilutions in butyl cellosolve.

6.7 Analysis

The analysis is done by graphite furnace/AA. The instrumental parameters for determining Sn in butyl cellosolve are as follows:

Atomic absorption unit:

Sn wavelength	**224.6 nm**
integ. time	10 sec.
slit width	.7 low
signal	Pk. Ut.
mode	abs.
BGC	on

Furnace parameters:

step	**temperature**	**ramp time**	**hold time**	**internal flow**
dry	100 °C	50 s	40 s	50 mL/min
char	800 °C	50 s	20 s	50 mL/min
atomize	2500 °C	0 s	8 s	30 mL/min

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

Chart = 5 mv scale, 20 mm/min

6.7.2 Parameters are adjusted so that the 2.0 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 I

A recovery study of MTM in 15 mL impinger of butyl cellosolve was done. 0.240 g MTM was weighed into a 1000 ml volumetric, diluted to volume with butyl cellosolve, and mixed. Assuming MTM is 11% Sn, this is 26.4 ppm Sn as MTM. From this a 13.2 ppm and a 6.6 ppm standard are also prepared. Six impingers were spiked at each level = .5×, 1×, and 2× the OSHA PEL based on a 150 liter air volume and 0.1 mg/m3 OSHA PEL. The spikes were made as follows:

Sample	Stock std.	ml. aliquot	final vol.	theor. Sn
0.5×-l	6.6 ppm	1.0 mL	15 mL	0.44 ppm
0.5×-2	6.6 ppm	1.0 mL	15 mL	0.44 ppm
0.5×-3	6.6 ppm	1.0 mL	15 mL	0.44 ppm
0.5×-4	6.6 ppm	1.0 mL	15 mL	0.44 ppm
0.5×-5	6.6 ppm	1.0 mL	15 mL	0.44 ppm
0.5×-6	6.6 ppm	1.0 mL	15 mL	0.44 ppm
1.0×-1	13.2 ppm	1.0 mL	15 mL	0.88 ppm
1.0×-2	13.2 ppm	1.0 mL	15 mL	0.88 ppm
1.0×-3	13.2 ppm	1.0 mL	15 mL	0.88 ppm
1.0×-4	13.2 ppm	1.0 mL	15 mL	0.88 ppm
1.0×-5	13.2 ppm	1.0 mL	15 mL	0.88 ppm
1.0×-6	13.2 ppm	1.0 mL	15 mL	0.88 ppm
2.0×-1	26.4 ppm	1.0 mL	15 mL	1.76 ppm
2.0×-2	26.4 ppm	1.0 mL	15 mL	1.76 ppm
2.0×-3	26.4 ppm	1.0 mL	15 mL	1.76 ppm
2.0×-4	26.4 ppm	1.0 mL	15 mL	1.76 ppm
2.0×-5	26.4 ppm	1.0 mL	15 mL	1.76 ppm
2.0×-6	26.4 ppm	1.0 mL	15 mL	1.76 ppm

The mean standard deviation and coefficient of variation for the recovery at each level using the OSHA "Precision and Accuracy Data" program:

Level .5× (OSHA PEL)

µg taken	µg found	AMR	
0.44	0.38	0.864	N = 6
0.44	0.40	0.909	Mean = 0.875
0.44	0.38	0.864	Std Dev = 0.019
0.44	0.38	0.864	CV_1 = 0.022
0.44	0.39	0.886	
0.44	0.38	0.864	

Level 1× (OSHA PEL)

µg taken	µg found	AMR	
0.88	0.75	0.852	N = 6
0.88	0.75	0.852	Mean = 0.845
0.88	0.75	0.852	Std Dev = 0.020
0.88	0.74	0.841	CV_1 = 0.024
0.88	0.71	0.807	
0.88	0.76	0.864	

Level 2× (OSHA PEL)

µg taken	µg found	AMR	
1.76	1.57	0.892	N = 6
1.76	1.54	0.875	Mean = 0.901
1.76	1.56	0.886	Std Dev = 0.020
1.76	1.60	0.909	CV_1 = 0.024
1.76	1.59	0.903	
1.76	1.65	0.938	