Tetraethyltin

Method number:	110
Matrix:	Air
Target concentration:	0.2 mg/m^3, TWA
Procedure:	Samples are collected by drawing known volumes of air through sampling tubes containing 100 mg of XAD-7 in the front section and 50 mg in the back section. Samples are desorbed with carbon disulfide and analyzed by GC using an FID detector.
Recommended air volume and sampling rate:	48 L at 0.2 L/min
Reliable quantitation limit:	14.4 µg/m^3
Standard error of estimate at target concentration:	6.13%
Status of method:	Evaluated method. This method has been subjected to the established evaluation procedures of the Organic Methods Evaluation Branch.

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1. General Discussion

1.1 Background

1.1.1 History

The OSHA PEL for tin is 0.1 mg/m^3 for organic tin compounds and 2 mg/m^3 for inorganic tin compounds. (Ref. 5.1) In order to evaluate the exposure to tin in the workplace, one must distinguish between the organic and the inorganic tin compounds. The early NIOSH method for organotins (Ref. 5.2), in which the workplace air is sampled with a membrane filter/charcoal tube and analyzed by colorimetry, does not do this. Until a satisfactory procedure for separating the organic tin compounds as a group from the inorganic ones is found, the specific organotins must be sampled and analyzed if we were to assess the worker exposure to organotin properly. The latest NIOSH method for organotin compounds (Ref. 5.3) specifies sampling with a glass fiber filter/XAD-2 sorbent tube, desorption with acetic acid/acetonitrile, and analysis with HPLC and graphite furnace atomic absorption spectrometer. In this procedure, specific organotin compounds are used for calibration. The method, however, may not be suitable for volatile tetraethyltin and tetramethyltin.

Although this method covers only tetraethyltin, the original goal of this work was to provide a common procedure for tetramethyltin and tetraethyltin. These volatile alkyltins lend themselves well for GC analysis. Detection using an atomic emission detector was considered first for its specific measurement of tin, but was abandoned due to lack of sensitivity. A flame ionization detector was found to provide adequate sensitivity.

A variety of sorbents were considered for a common sampler for tetramethyltin and tetraethyltin but none of them was satisfactory. Satisfactory recovery of tetraethyltin was not obtained from charcoal, carbon molecular sieve, or Anasorb 747, even with a variety of desorption solvents, including toluene, acetone, methylene chloride/methanol, and dimethyl-formamide/carbon disulfide. Polymeric sorbents such as Tenax, XAD-2, and XAD-7, on the other hand, do not collect tetramethyltin well. A common sampler for these two analytes was never found. XAD-7 was selected for sampling tetraethyltin because of its high capacity and ease of handling; it is not as prone to static electricity as XAD-2. An adequate sampler for tetramethyltin was never found. The best sampler candidate for tetramethyltin was Anasorb 747 until tests indicated the sampler capacity to be unacceptably sensitive to humidity. There was no breakthrough in six hours at 80% RH and 22°C, but at 80% RH and 25°C the 5% breakthrough point was reached in 41 minutes.

This method covers the sampling and analytical procedure for tetraethyltin. Samples are collected on XAD-7, desorbed with carbon disulfide, and analyzed by GC/FID.

1.1.2 Toxic effects (This section is for information only and should not be taken as the basis of OSHA policy.) (Ref. 5.2)

Organotin compounds differ in the severity of their toxic effects as well as in the organs they affect. The trialkyltins are the most toxic group, followed by the dialkyltins and monoalkyltins. The tetraalkyltins are metabolized to their trialkyltin homologs, so that their effects are those of the trialkyltins, with severity dependent upon the rate of metabolic conversion. Thus, an important feature of tetraalkyltin poisoning is the slow development of toxic symptoms and the long periods before death may occur.

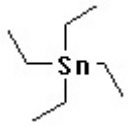
In 1951, Zeman and others reported four cases of employee exposure to unknown concentrations of tetramethyltin and tetraethyltin. Initial symptoms in all four subjects included severe headaches and nausea, with vomiting in two instances. Illnesses lasted four to ten weeks. In the most severe case of organotin poisoning, bradycardia, hypotension, and abrupt variations in the sinus rhythm of the heart were observed. These

findings suggest that these organotins are potent poisons of the circulatory system and may affect the autonomic nervous system.

1.1.3 Workplace exposure

Tetraethyltin is used as a catalyst and as a metal plating agent. It is also used in flame resistant polyester. (Ref. 5.2) Workers employed in the manufacturing operations involving tetraethyltin or its application have the greatest potential for exposure.

1.1.4 Physical properties and other descriptive information (Ref. 5.2)

CAS no.:	597-64-8
synonyms:	tetraethylstannane; TET; TeET
molecular formula:	$(C_2H_5)_4Sn$
formula wt:	234.94
appearance:	clear liquid
boiling point:	180.5 - 181°C
melting point:	-136 to -125°C
specific gravity:	1.187
flash point:	53°C
solubility:	soluble in organic solvents, very slightly soluble in water
structure formula:	

The analyte air concentrations throughout this method are based on the recommended sampling and analytical parameters. The analyte concentrations are listed as those of tetraethyltin. To compare the concentration to the OSHA PEL for tin in organic compounds (0.1 mg/m^3), apply a conversion factor of 0.5052 to find the tin content.

1.2 Limit defining parameters

1.2.1 Detection limit of the analytical procedure

The detection limit of the analytical procedure is 4.8 pg. This is the amount of analyte that will give a response that is significantly different from the background response of reagent blank. (Sections 4.1 and 4.2)

1.2.2 Detection limit of the overall procedure

The detection limit of the overall procedure is 0.21 µg per sample (4.3 µg/m^3). This is the amount of analyte spiked on the sampler that will give a response that is significantly different from the background response of a sampler blank. (Sections 4.1 and 4.3)

1.2.3 Reliable quantitation limit

The reliable quantitation limit is 0.69 µg per sample (14.4 µg/m^3). This is the amount of analyte spiked on a sampler that will give a signal that is considered the lower limit for precise quantitative measurements. (Section 4.4)

1.2.4 Precision (analytical procedure)

The precision of the analytical procedure, measured as the pooled relative standard deviation over a concentration range equivalent to 0.5 to 2 times the target concentration, is 1.11%. (Section 4.5)

1.2.5 Precision (overall procedure)

The precision of the overall procedure at the 95% confidence level for the ambient temperature 15-day storage test (at the target concentration) is ±12.0%. This includes an additional 5% for sampling error. (Section 4.6)

1.2.6 Recovery

The recovery of tetraethyltin from samples used in a 15-day storage test remained above 88.1%, when the samples were stored at ambient temperature. (Section 4.7)

1.2.7 Reproducibility

Six samples, collected from a controlled test atmosphere, with a draft copy of this procedure, were submitted for analysis by an SLTC Organic Service Branch. The samples were analyzed after 2 days of storage at 5°C. No individual sample result deviated from its theoretical value by more than the precision reported in Section 1.2.5. (Section 4.8)

2. Sampling Procedure

2.1 Apparatus

2.1.1 A personal sampling pump calibrated to ±5% of the recommended flow rate with the sampling device attached.

2.1.2 Glass sampling tubes (110 mm × 6 mm o.d.) packed with two sections of XAD-7. The front section contains 100 mg and the back section contains 50 mg. The sections are held in place with glass wool plugs. For this evaluation, commercially prepared sampling tubes were purchased from SKC, Inc. (Catalog no. 226-95).

2.2 Reagents

None required.

2.3 Technique

2.3.1 Immediately before sampling, break off the ends of the sampling tube. All tubes should be from the same lot.

2.3.2 Attach the sampling tube to the sampling pump with flexible tubing.

2.3.3 Air should not pass through any hose or tubing before entering the sampling tube.

2.3.4 Cap both ends after sampling. Wrap each sample with a Form OSHA-21 seal.

2.3.5 Record the air volume for each sample.

2.3.6 Submit at least one blank with each set of samples. Blanks should be handled in the same manner as samples, except no air is drawn through them.

2.3.7 List any compounds that could be considered potential interferences.

2.4 Sampler capacity

The capacity of the front section of the XAD-7 tube was tested by sampling from a test atmosphere of tetraethyltin (0.4 mg/m^3, 80% RH, 22°C) at 0.2 L/min. There was no breakthrough in 8 hours. (Section 4.9)

2.5 Desorption efficiency

2.5.1 The average desorption efficiency for tetraethyltin from XAD-7 over the range of 0.5 to 2.0 times the target concentration was 102.6%. (Section 4.10.1)

2.5.2 The desorption efficiencies at 0.2, 0.1, and 0.05 times the target concentration were found to be 100.2%, 104.0%, and 100.3%, respectively. (Section 4.10.1)

2.5.3 Desorbed samples remain stable for at least 24 h. (Section 4.10.2)

2.6 Recommended air volume and sampling rate

2.6.1 The recommended air volume is 48 L at 0.2 L/min.

2.6.2 For short-term sampling the recommended air volume is 3 L at 0.2 L/min.

2.6.3 When short-term samples are collected, the air concentrations equivalent to the reliable quantitation limits become larger. For example, the reliable quantitation limit is 0.230 mg/m^3 for tetraethyltin when 3 L is collected.

2.7 Interferences (sampling)

There is no known interference for sampling.

2.8 Safety precautions (sampling)

2.8.1 The sampling equipment should be attached to the worker in such a manner that it will not interfere with work performance or safety.

2.8.2 All safety practices that apply to the work area being sampled should be followed.

3. Analytical Procedure

3.1 Apparatus

3.1.1 A GC equipped with a flame ionization detector. An HP 5890 equipped with a flame ionization detector and an autosampler were used in this evaluation.

3.1.2 A capillary column capable of separating tetraethyltin and *p*-cymene from any interferences. An RTx-1 column (60 m × 0.32 mm i.d., df 1.0 µm) was used in this evaluation.

3.1.3 An electronic integrator or other suitable means of measuring detector response. The Millennium Chromatography Manager System (Waters) was used in this evaluation.

3.1.4 Glass vials, 2-mL, with poly(tetrafluoroethylene)-lined caps for desorbing samples.

3.1.5 A dispenser capable of delivering 1.0 mL of desorbing solvent.

3.2 Reagents

3.2.1 Tetraethyltin. Tetraethyltin, technical grade, was obtained from Chem Services.

3.2.2 Carbon disulfide. Carbon disulfide, *Omni Solv* grade, was obtained from E. M. Science.

3.2.3 *p*-Cymene. *p*-Cymene, 99%, was obtained from Aldrich Chemical.

3.2.4 Desorbing solvent. Add 8 µL of *p*-cymene to 1 L of carbon disulfide.

3.3 Standard preparation

3.3.1 Prepare stock standards of tetraethyltin in the desorbing solvent. Eight microliters of tetraethyltin diluted to 10 mL with the desorbing solvent will give a stock standard of 950 µg /mL.

3.3.2 Prepare analytical standards by diluting the stock standards with the desorbing solvent. A 9.5 µg/mL standard solution corresponds to the target concentration.

3.4 Sample preparation

3.4.1 Transfer the sorbent of the front and the back section to separate glass vials.

3.4.2 Add 1.0 mL of the desorbing solvent to each vial.

3.4.3 Cap the vials and shake them on a mechanical shaker for 30 min.

3.5 Analysis

3.5.1 GC conditions

column:	RTx-1 (60 m × 0.32-mm i.d., 1.0-µm df)	
oven temperature:	160°C	
injector temperature:	250°C	
detector temperature:	300°C	
injection size:	1.0 µL	
retention time:	*p*-cymene	6.05 min
	tetraethyltin	6.48 min
hydrogen:	27 mL/min	
air:	415 mL/min	
make-up gas (N_2):	29 mL/min	
split ratio:	39 : 1	

3.5.2 An internal standard calibration method is used. A calibration curve can be constructed by plotting concentration of the analyte versus ISTD-corrected response of standard injections. Bracket the samples with freshly prepared standards.

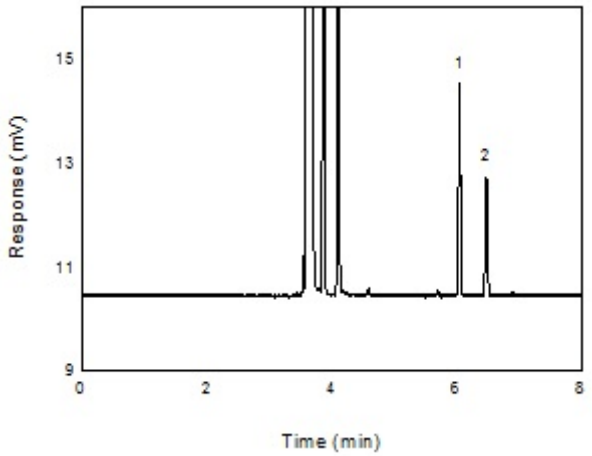


Figure 3.5.1. Chromatogram of tetraethyltin standard at the target concentration. 1 = *p*-cymene, 2 = tetraethyltin

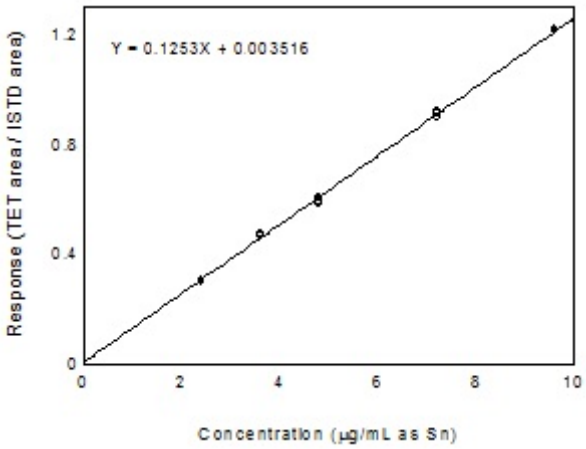


Figure 3.5.2. Calibration curve of tetraethyltin made from the data of Table 4.5.

3.6 Interferences (analytical)

3.6.1 Any compound that responds to FID and has a similar retention time as the analyte or the internal standard is a potential interference. If any potential interferences are reported, they should be considered before samples are desorbed. Generally, chromatographic conditions can be altered to separate an interference from the analyte.

3.6.3 When necessary, the identity or the purity of an analyte peak may be confirmed with additional analytical data (Section 4.11).

3.7 Calculations

The amount (in micrograms) of tetraethyltin per milliliter is obtained from the appropriate calibration curve. This amount is corrected by subtracting the amount (if any) found in the blank. The air concentration is calculated using the following formula.

$$mg/m^3 = \frac{\text{(micrograms per mL)} \times \text{(desorption volume, mL)}}{\text{(liters of air sampled)} \times \text{(desorption efficiency)}}$$

Where: Desorption volume = 1 mL
Desorption efficiency = 1.026

To compare the concentration to the OSHA PEL for tin in organic compounds (0.1 mg/m^3), apply a conversion factor of 0.5052 to find the tin content.

3.8 Safety precautions (analytical)

3.8.1 Follow the rules set down in your Chemical Hygiene Plan.

3.8.2 Avoid skin contact and inhalation of all chemicals.

3.8.3 Wear safety glasses and a lab coat at all times while in the lab area.

4. Backup Data

4.1 Determination of detection limits

Detection limits (DL), in general, are defined as the amount (or concentration) of analyte that gives a response (Y_{DL}) that is significantly different (three standard deviations (SD_{BR})) from the background response (Y_{BR}).

$$Y_{DL} - Y_{BR} = 3(SD_{BR})$$

The direct measurement of Y_{BR} and SD_{BR} in chromatographic methods is typically inconvenient and difficult because Y_{BR} is usually extremely low. Estimates of these parameters can be made with data obtained from the analysis of a series of analytical standards or samples whose responses are in the vicinity of the background response. The regression curve obtained for a plot of instrument response versus concentration of analyte will usually be linear. Assuming SD_{BR} and the precision of data about the curve are similar, the standard error of estimate (SEE) for the regression curve can be substituted for SD_{BR} in the above equation. The following calculations derive a formula for DL:

$$SEE = \sqrt{\frac{\sum (Y_{obs} - Y_{est})^2}{n - k}}$$

Y_{obs} = observed response
Y_{est} = estimated response from regression curve
n = total no. of data points
k = 2 for a linear regression curve
At point Y_{DL} on the regression curve

$$Y_{DL} = A(DL) + Y_{BR}$$

A = analytical sensitivity (slope)

therefore

$$DL = \frac{(Y_{DL} - Y_{BR})}{A}$$

Substituting 3(SEE) + Y_{BR} for Y_{DL} gives

$$DL = \frac{3(SEE)}{A}$$

4.2 Detection limit of the analytical procedure (DLAP)

The DLAP is measured as the mass of analyte actually introduced into the chromatographic column. Ten analytical standards of tetraethyltin whose concentrations was equally spaced from 0 to 0.950 µg/mL were prepared. The standard containing 0.950 µg/mL represented approximately 10 times the baseline noise. These solutions were analyzed with the recommended analytical parameters (1 µL injection, 39.1 split ratio). The data obtained were used to determine the required parameters (A and SEE) for the calculation of the DLAP. Values of 0.00289 and 0.00461 were obtained for A and SEE respectively. DLAP was calculated to be 4.8 pg.

Table 4.2
Detection Limit of the Analytical Procedure
for tetraethyltin

concentration (µg/mL)	mass on column (pg)	response
0.000	0.0	0.005117
0.095	2.4	0.006042
0.190	4.9	0.019923
0.285	7.3	0.020949
0.380	9.7	0.030539
0.475	12.1	0.029186
0.570	14.6	0.051692
0.665	17.0	0.053757
0.760	19.4	0.056698
0.855	21.9	0.069562
0.950	24.3	0.069456

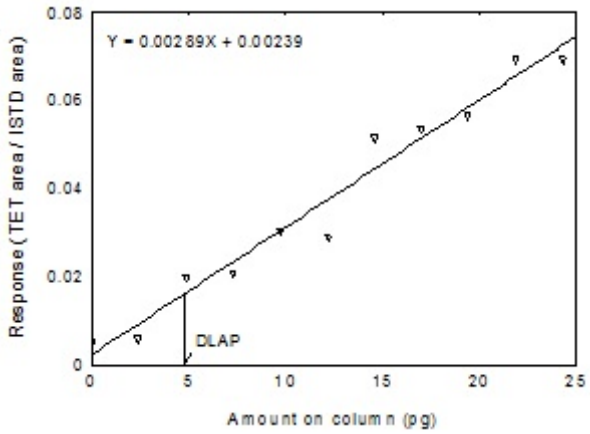


Figure 4.2. Plot of data to determine DLAP.

4.3 Detection limit of the overall procedure (DLOP)

The DLOP is measured as mass per sample and expressed as equivalent air concentration, based on the recommended sampling parameters. Ten samplers were spiked with tetraethyltin ranging from 0 to 0.950 µg. The latter amount, when spiked on a sampler, would produce a peak approximately 10 times the baseline noise for a sample blank. These samples were analyzed with the recommended analytical parameters, and the data obtained used to calculate the required parameters (A and SEE) for the calculation of the DLOP. Values of 0.0800 and 0.00549 were obtained for A and SEE respectively. DLOP was calculated to be 0.21 µg/sample (4.3 µg/m^3).

Table 4.3
Detection Limit of the Overall Procedure for Tetraethyltin

mass per sample (µg)	response
0.000	0.002780
0.095	0.003864
0.190	0.020702
0.285	0.022380
0.380	0.032509
0.475	0.035886
0.570	0.056129
0.665	0.059509
0.760	0.057776
0.855	0.061797
0.950	0.081816

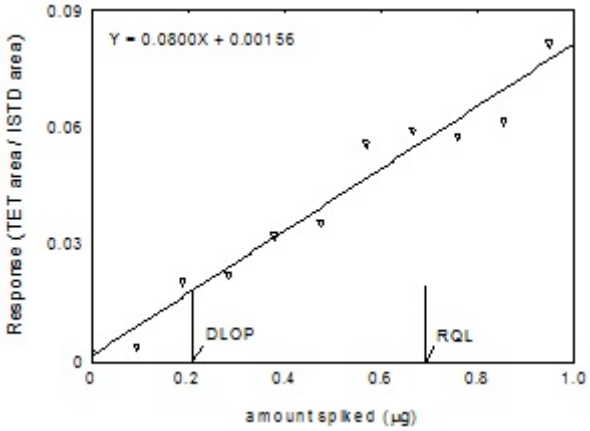


Figure 4.3. Plot of data to determine the DLOP and RQL.

4.4 Reliable quantitation limit

The RQL is considered the lower limit for precise quantitative measurements. It is determined from the regression line data obtained for the calculation of the DLOP (Section 4.3), providing at least 75% of the analyte is recovered. The RQL is defined as the amount of analyte that gives a response (Y_{RQL}) such that

$$Y_{RQL} - Y_{BR} = 10(SD_{BR})$$

therefore

$$RQL = \frac{10(SEE)}{A}$$

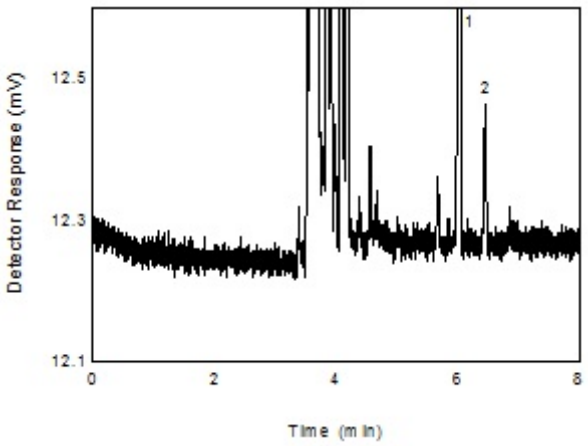


Figure 4.4. Chromatogram of tetraethyltin RQL. 1 = *p*-cymene, 2 = tetraethyltin.

RQL = 0.69 µg per sample (14.4 µg/m^3)

Recovery at this level is 106.2%.

4.5 Precision (analytical method)

The precision of the analytical procedure is defined as the pooled relative standard deviation (RSD_P). Relative standard deviations were determined from six replicate injections of analytical standards at 0.5, 0.75, 1, 1.5, and 2 times the target concentration. After assuring that the RSDs satisfy the Cochran test for homogeneity at the 95% confidence level, RSD_p was calculated.

Table 4.5
Instrument Response to Tetraethyltin

× target concn µg/mL	0.5 × 4.75	0.75 × 7.12	1 × 9.50	1.5 × 14.24	2 × 18.99
Response	0.298908	0.470980	0.603011	0.896033	1.205802
	0.299132	0.465653	0.603646	0.896054	1.214870
	0.293568	0.464225	0.598385	0.896632	1.208242
	0.297834	0.470271	0.582012	0.908579	1.207354
	0.308123	0.466955	0.590737	0.916217	1.213500
	0.302405	0.471602	0.600238	0.895729	1.209036
$\bar{X}$	0.299995	0.468281	0.596338	0.901541	1.209801
SD	0.004894	0.003079	0.008414	0.008755	0.003588
RSD %	1.63	0.66	1.41	0.97	0.30

The Cochran test for homogeneity requires the calculation of the g statistics according to the following formula:

$$g = \frac{\text{largest } RSD^2}{RSD_{0.5\times}^2 + RSD_{0.75\times}^2 + RSD_{1\times}^2 + RSD_{1.5\times}^2 + RSD_{2\times}^2} = 0.4347$$

The critical value of the *g* statistic, at the 95% confidence level, for five variances, each associated with six observations, is 0.5065. Because the *g* statistic obtained (0.4347) does not exceed this value, the RSDs within each level can be considered equal and they can be pooled (RSD_P) to give an estimated RSD for the concentration range studied.

$$RSD_p = \sqrt{\frac{5(RSD_{0.5\times}^2 + RSD_{0.75\times}^2 + RSD_{1\times}^2 + RSD_{1.5\times}^2 + RSD_{2\times}^2)}{5+5+5+5+5}} = 1.11\%$$

4.6 Precision (overall procedure)

The precision of the overall procedure is determined from the storage data in Section 4.7. The determination of the standard error of estimate (SEE_R) for a regression line plotted through the graphed storage data allows the inclusion of storage time as one of the factors affecting overall precision. The SEE_R is similar to the standard deviation, except it is a measure of dispersion of data about a regression line instead of about a mean. It is determined with the following equation:

$$SEE_R = \sqrt{\frac{\sum (Y_{obs} - Y_{est})^2}{n - k}}$$

n = total no. of data points
k = 2 for linear regression
k = 3 for quadratic regression
Y_{obs} = observed % recovery at a given time
Y_{est} = estimated % recovery from the regression line at the same given time

An additional 5% for pump error (SP) is added to the SEE_R by the addition of variances to obtain the total standard error of estimate.

$$SEE = \sqrt{(SEE_R)^2 + (SP)^2}$$

The precision at the 95% confidence level is obtained by multiplying the standard error of estimate (with pump error included) by 1.96 (the z-statistic from the standard normal distribution at the 95% confidence level). The 95% confidence intervals are drawn about their respective regression lines in the storage graphs, as shown in Figures 4.7.1 and 4.7.2. The precision of the overall procedure is ±12.0%.

4.7 Storage test

Storage samples were prepared by drawing samples from a controlled test atmosphere (80% relative humidity and 22°C) at 0.2 L/min for 120 min. The concentration of tetraethyltin was at two times the target concentration. Thirty-six samples were prepared. Six samples were analyzed on the day of preparation. One-half of the remaining samples were stored in a refrigerator (5°C), and the other half were stored at ambient temperature (about 22°C) in a closed drawer. At 2-4 day intervals, three samples were selected from each of the two storage sets and analyzed.

Table 4.7
Storage Test for Tetraethyltin

time (days)	percent recovery (ambient)			percent recovery (refrigerated)		
0	98.2	99.2	103.1	98.2	99.2	103.1
0	97.6	100.8	101.1	97.6	100.8	101.1
2	98.1	97.2	92.6	93.9	94.5	95.8
6	91.6	89.9	87.9	90.2	89.1	87.7
9	89.4	91.4	89.4	92.1	87.6	87.2
12	95.5	83.7	93.6	88.7	91.9	89.5
15	90.8	91.0	89.6	86.1	83.5	86.9

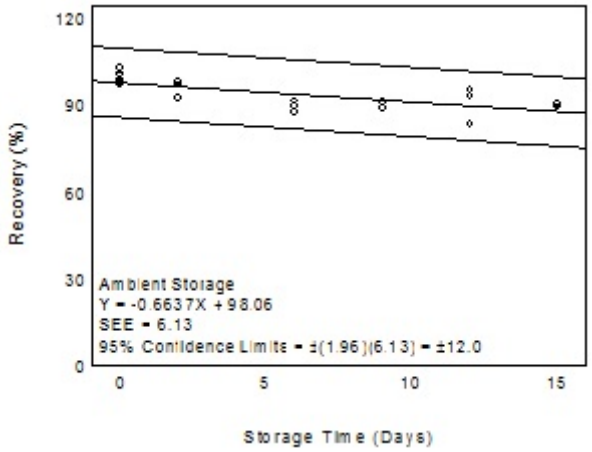


Figure 4.7.1. Ambient storage test for tetraethyltin.

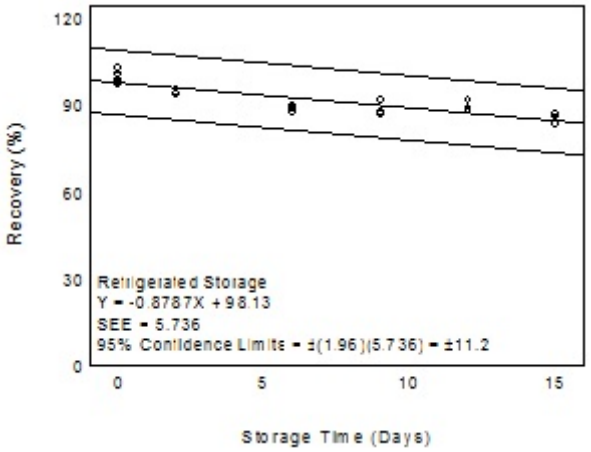


Figure 4.7.2. Refrigerated storage test for tetraethyltin.

4.8 Reproducibility

Reproducibility samples were prepared by collecting them from a controlled test atmosphere similar to that used in the storage test. The samples were submitted to an SLTC Service Branch for analysis. The samples were analyzed after being stored for 2 days at 5°C. No sample result had a deviation greater than the precision of the overall procedure determined in Section 4.7.

Table 4.8
Reproducibility Data for Tetraethyltin

µg expected	µg found	percent found	percent deviation
9.73	9.86	101.3	+1.3
9.95	10.45	105.0	+5.0
9.81	10.21	104.1	+4.1
9.89	10.19	103.0	+3.0
9.94	10.17	102.3	+2.3
9.91	10.19	102.8	+2.8

4.9 Sampler capacity

The sampling capacity of the front section of an XAD-7 tube was tested by sampling from a dynamically generated test atmosphere of tetraethyltin (0.4 mg/m^3). The samples were collected at 0.2 L/min and the relative humidity was 80% at 22°C. A complete XAD-7 sampling tube was placed in-line behind the 100-mg test section and changed at regular intervals. The 5% breakthrough point was not observed in 8 hours of sampling.

Table 4.9
Breakthrough of Tetraethyltin with XAD-7 Sampling Tubes

		test 1		test 2	
sampling time (min)	air volume (L)	downstream concn (mg/m^3)	breakthrough (%)	downstream concn (mg/m^3)	breakthrough (%)
30	6	0	0	0	0
90	18	0	0	0	0
150	30	0	0	0	0
210	42	0	0	0	0
270	54	0	0	0	0
330	66	0	0	0	0
390	78	0	0	0	0
450	90	0	0	0	0

4.10 Desorption efficiency and stability of desorbed samples

4.10.1 Desorption efficiency

The desorption efficiencies (DE) of tetraethyltin were determined by liquid-spiking 100-mg portions of XAD-7 with the analyte at 0.05 to 2 times the target concentration. These samples were stored overnight at ambient temperature and then desorbed and analyzed. The average desorption efficiency over the working range of 0.5 to 2 times the target concentration was 102.6%.

Table 4.10.1
Desorption Efficiency of Tetraethyltin

× target concn (µg/sample)	0.05 × 0.48	0.1 × 0.95	0.2 × 1.90	0.5 × 4.75	1.0 × 9.50	2.0 × 18.99
DE (%)	72.1	105.2	95.5	101.0	101.1	101.9
	109.6	105.8	101.9	103.8	101.5	102.0
	92.9	96.9	100.5	102.0	102.7	103.1
	107.5	106.0	99.9	106.3	102.9	99.6
	137.1	110.8	101.1	105.0	102.5	104.6
	82.5	99.4	102.1	101.3	104.4	102.1
Average	100.3	104.0	100.2	103.2	102.5	102.2

4.10.2 Stability of desorbed samples

The stability of the desorbed samples was investigated by re-analyzing the target concentration samples 24 h after initial analysis. After the original analysis was performed, three vials were recapped with new septa while the remaining three retained their punctured septa. The samples were re-analyzed with fresh standards.

Table 4.10.2
Stability of Desorbed Samples of Tetraethyltin

punctured septa replaced			punctured septa retained		
initial DE (%)	DE after one day (%)	difference	initial DE (%)	DE after one day (%)	difference
101.9	100.3	-1.6	99.6	100.0	+0.4
102.0	100.3	-1.7	104.6	102.3	-2.3
103.1	100.6	-2.5	102.1	102.0	-0.1
	average			average	
102.3	100.4	-1.9	102.1	101.4	-0.7

4.11 Qualitative analysis

Tetraethyltin may be confirmed by GC/MS using the GC conditions similar to those in Section 3.5.1.

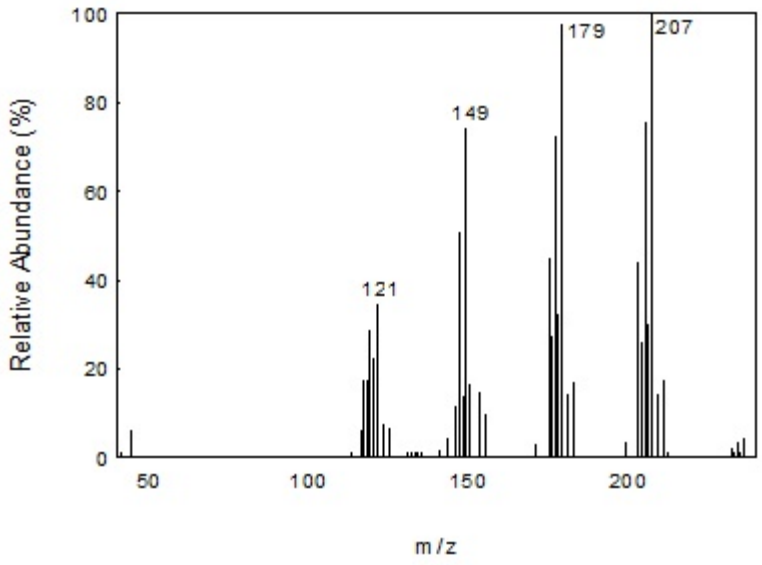


Figure 4.11. Mass spectrum of tetraethyltin.

5. References

5.1. *Air Contaminants — Permissible Exposure Limits*, Title 29, Code of Federal Regulations, Part 1910.1000.

5.2. *Criteria for a recommended standard ... Occupational exposure to organotin compounds*, U.S. Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, November 1976. DHEW (NIOSH) Publication No. 77-115.

5.3. *NIOSH Manual of Analytical Methods*, 4th ed., Method No. 5504, 1994.