ALDICARB (TEMIK)

Method no.:	74
Matrix:	Air
Target concentration:	0.1 mg/m^3
Procedure:	Samples are collected by drawing known volumes of air through OSHA Versatile Samplers containing a glass fiber filter and two sections of XAD-2 adsorbent (OVS-2). Samples are extracted/desorbed with ace tone and analyzed by GC using a nitrogen/phosphorus detector.
Recommended air volume and sampling rate:	480 L at 1.0 L/min
Reliable quantitation limit:	0.25 µg/m^3
Standard error of estimate at the target concentration:	6.7% (Section 4.7.)
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

This evaluation was undertaken to determine the effectiveness of the OSHA Versatile Sampler containing XAD-2 resin (OVS-2) as a sampling device for aldicarb. The OVS-2 is a specially prepared glass tube containing a glass fiber filter and XAD-2 resin (Section 4.11.) which will collect both vapors and aerosols. The OVS-2 tube was previously evaluated and found to be an effective collection device for a number of organophosphorus pesticides, a carbamate pesticide, a natural product pesticide, and an organochlorine pesticide. (Refs. 5.1.-5.4.)

In the past, aldicarb was collected on 37-mm glass fiber filters and the extracted samples were analyzed by HPLC with UV detection. (Ref. 5.5.) This current method specifies that the samples be analyzed by GC with a nitrogen/phosphorus detector (NPD).

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

The LD_{50} value for technical aldicarb was found to be 0.9 mg/kg for male rats when applied orally. Temik 10G has an LD_{50} 400 mg/kg when wetted with saline and applied to the skin of rats. (Ref. 5.6.)

Aldicarb is a carbamate insecticide which causes cholinesterase inhibition at very low doses. It has muscarinic effects at exocrine, excretory, cardiac and bronchial sites which are exhibited overtly by salivation, lacrimation, defecation, urination, slowing of the heart and trouble with breathing. Aldicarb's chief metabolites, aldicarb sulfoxide and aldicarb sulfone, are also potent cholinesterase inhibitors. (Ref. 5.6.)

1.1.3. Workplace exposure

Occupational exposure can occur during the formulation, distribution and application of aldicarb. Aldicarb is approved for soil application around cotton, sugar beets, potatoes, peanuts, sugarcane, lily bulbs, greenhouse plants, ornamentals selected, citrus trees, pecans, sorghum, and soybeans. (Ref. 5.7.) An estimated 1.0-1.5 million pounds of aldicarb were produced in the United States in 1972. (Ref. 5.7.)

1.1.3. Physical properties and other descriptive information (Ref. 5.7.)

CAS no.:	116-06-3
molecular weight:	190.25
odor:	slightly sulfurous
melting point:	99-100°C
vapor pressure:	0.013 Pa (1 x 10^{-4} mm Hg) at 25°C
description:	white, crystalline solid
specific gravity:	1.195 at 25/20°C
flash point:	between 23-60°C
solubility:	practically insoluble in hexane, but soluble in most other organic solvents, slightly soluble in water
synonyms:	2-methyl-2-(methylthio)propanal, o-[(methylamino)carbonyl]oxime; 2-methyl-2-(methylthio)propionaldehde,0-(methylcarbamoyl)oxime; aldecarb; carbamyl; carbanolate; ENT 27093; NCIC08640; OMS 771; propanal, 2-methyl-2-(methylthio) -0- [(methylamino) carbonyl] oxime; propionaldehyde-2-methyl-2-(methylthio)-0-(methylcarbamol)oxime; sulfone aldoxycarb; Temik; UC 21149
structure:	CH_3-S-C(CH_3) $_2$-CH=N-O-CO-NH-CH_3

The analyte air concentrations throughout this method are based on the recommended sampling and analytical parameters.

1.2. Limit defining parameters

1.2.1. Detection limit of the analytical procedure

The detection limit of the analytical procedure is 60.3 pg per injection. This is the amount of analyte which gave an aldicarb peak whose area is about 5 times the area of an interference peak visible in a blank sample. (Section 4.1.)

1.2.2. Detection limit of the overall procedure is 120.5 ng per sample (0.25 µg/m^3). This is the amount of analyte spiked on the sampling device which allows recovery of an amount equivalent to the detection limit of the analytical procedure. (Section 4.2.)

1.2.3. Reliable quantitation limit

The reliable quantitation limit is 120.5 ng per sample (0.25 µg/m^3). This is the smallest amount of analyte spiked on the sampling device which can be quantitated within the requirements of a recovery of at least 75% and a precision ($\pm$1.96 SD) of $\pm$25% or better. (Section 4.3.)

The reliable quantitation limit and detection limits reported in the method are based upon optimization of the instrument for the smallest possible amount of the analyte. When the target concentration of the analyte is exceptionally higher than these limits, they may not be attainable at the routine operating parameters.

1.2.4. Instrument response to the analyte

The instrument response over the concentration range of 0.5 to 2 times the target concentration is linear. (Section 4.4.)

1.2.5. Recovery

The recovery of aldicarb from samples (liquid spiked) used in a 17-day storage test remained above 103.1% when the samples were stored in a closed drawer at about 22°C. (Section 4.5.) The recovery of an analyte from the collection medium during storage must be 75% or greater.

1.2.6. Precision (analytical procedure only)

The pooled coefficient of variation obtained from replicate injections of analytical standards at 0.5, 1 and 2 times the target concentration is 0.014. (Section 4.6.)

1.2.7. Precision (overall procedure)

The precision at the 95% confidence level for the 17-day storage test is ±13.2%. (Section 4.7.) This includes an additional ±5% for sampling error. The overall procedure must provide results at the target concentration that are ±25% or better at the 95% confidence level.

1.2.8. Reproducibility

Six samples, spiked by liquid injection with aldicarb, and a draft copy of this procedure were given to a chemist unassociated with this evaluation. The samples were analyzed after one day of storage at about 22°C. No individual sample result deviated from its theoretical value by more than the precision reported in Section 1.2.7. (Section 4.8.)

1.3. Advantage

This sampling device can collect a variety of pesticides as aerosols or vapors.

1.4. Disadvantage

Currently the OVS-2 tube is not commercially available.

2. Sampling Procedure

2.1. Apparatus

2.1.1. Samples are collected by use of a personal sampling pump that can be calibrated to within ±5% of the recommended flow rate with the sampling device attached.

2.1.2. Samples are collected with glass OVS-2 tubes, which are 13-mm o.d. tubes that are tapered to 6-mm o.d. The tubes are packed with a 140-mg back section and a 270-mg front section of cleaned XAD-2 resin along with a 13-mm diameter glass fiber filter. The back or "13" section consists of XAD-2 resin and the small foam plug. The front or "A" section consists of the XAD-2 resin and the glass fiber filter. The glass fiber filter is held in place with a polytetrafluoroethylene (PTFE) retainer. (Section 4.11., Figure 4.11.4.)

2.2. Reagents

No sampling reagents are required.

2.3. Sampling technique

2.3.1. Attach the small end of the sampling device to the sampling pump with flexible, plastic tubing such that the large front section of the sampling tube is exposed directly to the atmosphere. Do not place any tubing in front of the sampler. Attach the sampler vertically (large end down) in the worker's breathing zone in such a manner that it does not impede work performance.

2.3.2. After sampling for the appropriate time, remove the sampling device and seal the tube with plastic end caps.

2.3.3. Wrap each sample end-to-end with an OSHA seal (Form 21).

2.3.4. With each set of samples submit at least one blank sample. Handle the blank sample in the same manner as the other samples except draw no air through it.

2.4. Retention efficiency

To test the sampler's ability to retain aldicarb, two times the target concentration of aldicarb (98.4 µg) was liquid-spiked onto the glass fiber filters of twelve sampling devices. Humid air (about 80% relative humidity) was pulled through the tubes at 1 L/min. Each sampling device had a different amount of air pulled through it. Breakthrough was defined as the amount of analyte on the "13" section divided by the total amount found on the sampling device. Analysis showed that the analyte was present on the "A" section at levels equal to 90-105% of the total amount spiked. Aldicarb was found on many of the "B" sections. At two times the recommended air volume, the breakthrough observed was less than 5%. (Section 4.9.)

2.5. Extraction and desorption efficiencies (Section 4.10.)

2.5.1. The combined extraction/desorption efficiency for aldicarb from the glass fiber filter and the large XAD-2 section at the target concentration was 106.8%.

2.5.2. The extraction efficiency for aldicarb from glass fiber filters at the target concentration was 97.5%.

2.5.3. The average desorption efficiency for aldicarb over the range of 0.5 to 2 times the target concentration from the lot of cleaned XAD-2 adsorbent used in this evaluation was 102.2%.

2.5.4. Extracted/desorbed samples remain stable for at least 48

2.6. Recommended air volume and sampling rate

2.6.1. The recommended air volume is 480 L.

2.6.2. The recommended air sampling rate is 1.0 L/min.

2.6.3. When short-term air samples are required, the reliable quantitation limit is 8.0 µg/m^3 for a 15-min sample collected at the recommended sampling rate.

2.7. Interferences (sampling)

Suspected interference should be reported to the laboratory with submitted samples.

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 nitrogen/phosphorus detector. For this evaluation a Hewlett-Packard 5890 Gas Chromatograph equipped with a 7673A Autosampler and an NPD was used.

3.1.2. A GC column capable of separating the aldicarb peak from potential interferences. A 30-m x 0.32-mm i.d. (1.0-µm depth of film) SPB-1 column (Supelco Inc., Bellefonte, PA) was used in this evaluation.

3.1.3. An electronic integrator or other suitable means of measuring detector response. A Hewlett-Packard 3357 computer and Hewlett-Packard 5895A GC ChemStation were used in this evaluation.

3.1.4. Two- and four-milliliter vials with PTFE-lined caps were used for sample extraction/desorption and standard preparation.

3.2. Reagents

3.2.1. Aldicarb. Aldicarb (99.0% pure) used in this evaluation was purchased from Chem Services Inc., West Chester, PA.

3.2.2. Acetone. American Burdick & Jackson "High Purity Solvent" brand acetone was used in this evaluation.

3.2.3. Triethylphosphate (TEP). TEP was purchased from ICN. This was used as the internal standard in the extracting/desorbing solution (25 microliters of TEP per liter of acetone).

3.3. Standard preparation

3.3.1. Prepare stock standards by adding acetone to preweighed amounts of aldicarb. Include the percent purity in the calculation.

3.3.2. Prepare analytical standards by injecting microliter amounts of diluted aldicarb stock standards into 4-mL vials containing 2.0 mL of extracting/desorbing, solution.

3.4. Sample preparation

3.4.1. Transfer the glass fiber filter and the 270-mg section of the sampling tube to a 4-mL glass vial. Place the first foam plug and the 140-mg section in a separate vial. A small glass funnel can be used to transfer the adsorbent. Discard the rear foam plug. Do not discard the glass sampling tube as it can be reused after cleaning with surfactant or suitable solvent.

3.4.2. Add 2.0 mL of extracting/desorbing solution to each vial.

3.4.3. Seal the vials with PTFE-lined caps and allow them to extract/desorb for 1 h. Shake the vials vigorously by hand several times during the extraction/desorption time.

3.4.4. If necessary, transfer some of the solution from each of the 4-mL vials to smaller glass vials suitable for an autosampler.

3.5. Analysis

3.5.1. Analytical conditions

GC conditions

temperature:	50°C (column) 200°C (injector) 220°C (detector)
temp program:	hold initial temp 1.0 min, increase temp at 25°C/min to 110°C, hold temp for 3.5 min, increase temp at 25°C/min to 135°C, hold temp for 5.0 min
column gas flow:	1.7 mL/min (helium)
septum purge:	3.8 mL/min (helium)
injection size:	1.0 µL (splitless mode)
column:	SPB-1, 30-m x 0.32-mm i.d. fused silica 1.0-µm depth of film, (Supelco Inc.)
retention time:	6.44 min (aldicarb) 11.23 min (TEP)

NPD conditions

make-up gas flow:	36 mL/min (helium)
hydrogen flow:	3.5 mL/min
air flow:	100 mL/min

chromatogram: Figure 3.5.1.

3.5.2. Measure detector response using a suitable method such as electronic integration.

3.5.3. Use an external or internal standard procedure to prepare a calibration curve using several standards over a range of concentrations. Prepare the calibration curve daily. Bracket the samples with analytical standards.

3.6. Interferences (analytical)

3.6.1. Any compound having a similar retention time as the analyte is a potential interference. Generally, chromatographic conditions can be altered to separate an interference from the analyte.

3.6.2. Retention time on a single column is not proof of chemical identity. Analysis by an alternate GC column and confirmation by mass spectrometry are additional means of identification.

3.7. Calculations

3.7.1. Prepare calibration curves from analytical standards by plotting detector response for aldicarb versus the analytical standard concentrations (in terms of micrograms aldicarb per milliliter). Determine, the best-fit line through the data points.

3.7.2. Determine the concentration, in micrograms of aldicarb per milliliter, of a sample by comparing its detector response to the calibration curve. Perform blank corrections for each section before adding the results together. Add the amount of aldicarb on the backup section to the amount found on the front section.

3.7.3. The air concentration of aldicarb can be expressed in mg/m^3 by using the following equation:

$$mg/m^3 = (A)(B)/(C)(D)$$

where A = µg/mL of aldicarb from Section 3.7.2.
B = extraction/desorption volume in milliliters
C = liters of air sampled
D = combined extraction/desorption efficiency (decimal)

The combined extraction/desorption efficiency should be determined for the particular batch of resin and lot of filter used for the sample.

3.8. Safety precautions (analytical)

3.8.1. Avoid skin contact and inhalation of all chemicals.

3.8.2 Restrict the use of all chemicals to a fume hood.

3.8.3. Wear safety glasses in all laboratory areas.

4. Backup Data

4.1. Detection limit of the analytical procedure

The detection limit of the analytical procedure, 60.3 pg per injection, is based on a 1.0-µL injection of a 60.3 ng/mL standard. This amount produced an aldicarb peak whose area is about 5 times the area of an interference peak in the blank. A chromatogram of the detection limit of the analytical procedure, and a blank are shown in Figures 4.1.1. and 4.1.2.

4.2. Detection limit of the overall procedure

The detection limit of the overall procedure is 120.5 ng per sample (0.25 $\mu g/m^3$). The injection size recommended in the analytical procedure (1.0 µL) was used in the determination of the detection limit of the overall procedure. Six samples containing a glass fiber filter and 270 mg of XAD-2 resin were liquid-spiked with 10 L of 12.05 µg/mL aldicarb in acetone. The samples were extracted/desorbed about 1 h after being spiked.

Table 4.2.
Detection Limit of the Overall Procedure

sample number	theoretical amount (ng)	amount recovered (ng)
1	120.5	103.5
2	120.5	95.5
3	120.5	92.5
4	120.5	93.9
5	120.5	100.0
6	120.5	100.3

4.3. Reliable quantitation limit data

The reliable quantitation limit is 120.5 ng per sample (0.25 µg/m^3). The injection size recommended in the analytical procedure (1.0 µL) was used in the determination of the reliable quantitation limit. Six samples containing "a glass fiber filter and 270 mg of XAD-2 resin were liquid-spiked with 10 µL of 12.05 µg/mL aldicarb in acetone. Because the recovery of aldicarb from the samples was high and approximately equal to the detection limit of the analytical procedure, the detection limit of the overall procedure and reliable quantitation are the same. They are listed below.

Table 4.3.
Reliable Quantitation Limit
(Based on samples and data of Table 4.2.)

sample number	percent recovered	statistics
1	85.9	
2	79.2	$\bar{X}$ = 81.0
3	76.8	SD = 3.5
4	77.9	Precision = $\pm$(1.96)(3.5)
5	83.0	= $\pm$6.9
6	83.2	

4.4. Instrument response to aldicarb

The instrument response to aldicarb over the range of 0.5 to 2 times the target concentration is linear with a slope of 424 area counts per microgram per milliliter. The response to aldicarb was determined by multiple injections of aldicarb standards. The data listed below is presented graphically in Figure 4.4.

Table 4.4.
Instrument Response to Aldicarb

x target conc.	0.5x	1x	2x
µg/sample	24.1	48.2	96.4
area counts	11753	22861	42825
	11651	22868	42613
	11523	22707	42579
	11276	22603	42241
	11178	22484	41806
	11246	22640	41737
$\bar{X}$	11438	22694	42300

4.5. Storage data

Storage samples were generated by liquid-spiking 36 sampling tubes with 48.2 µg of aldicarb and then pulling 120 L of humid air (about 80% relative humidity) through them. One-half of the tubes was stored in a refrigerator (0°C) and the other half was stored in a closed drawer at ambient temperature (about 22°C). At 3- or 4-day intervals, three samples were selected from each of the two storage sets and analyzed. The results of this test showed that samples do not have to be stored at reduced temperature. The results are listed below and shown graphically in Figures 4.5.1. and 4.5.2.

Table 4.5.
Storage Test (Liquid Spiked)

storage time	% recovery					
(days)	(ambient)			(refrigerated)		
0	99.8	100.9	101.6	99.8	100.9	101.6
	101.7	111.7	106.9	101.7	111.7	106.9
4	103.7	99.2	110.7	101.4	108.2	100.7
7	96.8	99.7	106.7	110.0	96.2	100.4
11	100.0	101.4	99.4	96.2	102.7	106.7
14	109.1	104.5	104.7	103.6	105.5	99.2
17	111.4	102.7	100.3	106.6	101.4	100.6

4.6. Precision (analytical method only)

The precision of the analytical procedure is defined as the pooled coefficient of variation determined from replicate injections of aldicarb standards at 0.5, 1 and 2 times the PEL. The coefficients of variation (CV)_for the three levels and the pooled coefficient of variation (CV) are listed below.

Table 4.6.
Precision of the Analytical Method
(Based on the Data of Table 4.4.)

x target conc.	0.5x	1x	2x
µg/sample	24.1	48.2	96.4
SD (area counts)	238	151	451
CV	0.0207	0.0067	0.0107
CV = 0.014			

4.7. Precision (overall procedure)

The precision of the overall procedure is determined from the storage data. The determination of the standard error of estimate (SEE) 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 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:

$$SEE = \left[\frac{\Sigma(Y_{obs} - Y_{est})^2}{n - k}\right]^{1/2}$$

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

An additional 5% for pump error is added to the SEE by the addition of variances. The precision at the 95% confidence level is obtained by multiplying the, SEE (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 Figure 4.5.1. The data for Figure 4.5.1. was used to determine the SEE of $\pm$6.7% for aldicarb.

4.8. Reproducibility data

Six samples, liquid-spiked with aldicarb, were given to a chemist unassociated with this study. The samples were analyzed after being stored for one day at 22°C. The results were corrected for extraction/desorption efficiency and are listed below. No sample result had a percent deviation greater than the precision of the overall procedure, which is $\pm$13.2%.

Table 4.8.
Reproducibility Data

sample	µg spiked	µg recovered	% recovered	% deviation
1	48.5	44.3	91.3	-8.7
2	48.5	48.9	100.8	+0.8
3	48.5	50.0	103.1	+3.1
4	48.5	49.6	102.3	+2.3
5	48.5	44.0	90.7	-9.3
6	48.5	48.8	100.6	+0.6

4.9. Retention efficiency

To test the ability of the sampling device to retain the analyte, 12 sampling devices were liquid-spiked on the filter with 2 times the target concentration (96.4 µg) of aldicarb. Humid air (about 80% relative humidity) was pulled through the samplers at 1 L/min. Each sampling tube had humid air drawn through it for a different length of time. Breakthrough was defined as the amount of analyte on the "B" section divided by the total amount found on the sampling device.

Table 4.9.
Retention of Aldicarb

air volume (L)	"A" section (µg)	"B" section (µg)	total (µg)	breakthrough (%)
264	102.2	0.0	102.2	0.0
381	99.3	0.6	99.9	0.6
424	89.8	1.6	91.4	1.7
480	97.7	2.9	100.6	2.9
539	98.9	1.0	99.9	1.0
572	97.2	3.1	100.3	3.1
656	93.0	1.6	94.6	1.7
689	93.0	2.8	95.8	2.9
749	93.0	1.7	94.7	1.8
805	99.4	1.7	101.1	1.7
907	102.4	3.5	105.9	3.3
965	105.0	4.5	109.5	4.1

4.10. Extraction and desorption efficiencies

4.10.1. Extraction from glass fiber filter

The extraction efficiency of aldicarb was determined by liquid-spiking each of six glass fiber filters with 48.4 µg of aldicarb. These samples were stored overnight and then extracted with acetone and analyzed.

Table 4.10.1.
Extraction Efficiency
1x Target Concentration

sample number	µg recovered	percent recovered
1	47.3	97.7
2	47.9	99.0
3	46.1	95.2
4	47.9	99.0
5	46.7	96.5
6	47.1	97.3
$\bar{X}$		97.6

4.10.2. Desorption from XAD-2 adsorbent

The desorption efficiency (DE) of aldicarb was determined by liquid-spiking 270-mg portions of XAD-2 adsorbent with aldicarb at 0.5 to 2 times the target concentration. These samples were stored overnight and then desorbed with acetone and analyzed. The average desorption efficiency over the studied range was 102.2%.

Table 4.10.2.
Desorption Efficiency of Aldicarb

x target conc.	0.5x	1x	2x
µg/sample	24.2	48.4	96.8
DE, %	87.5	106.2	107.4
	96.5	108.3	104.6
	102.3	110.7	103.1
	103.3	109.8	101.7
	104.7	100.4	100.4
	92.8	104.8	95.0
$\bar{X}$	97.8	106.7	102.0

4.10.3. Combined extraction/desorption efficiency

The combined extraction/desorption efficiency of aldicarb was determined by liquid-spiking glass fiber filters with the target concentration and placing the filter and the large section of XAD-2 beads into a vial. The next day the samples were extracted/desorbed with acetone and analyzed. The samples were reanalyzed the following day with new standards to test the stability of the extracted/desorbed samples.

Table 4.10.3.
Stability
of Extracted/Desorbed Samples

	original	24 h later
%	107.0	110.7
	109.5	109.5
	107.9	108.1
	107.8	108.3
	103.9	102.3
	104.4	103.0
$\bar{X}$	106.8	107.0
% of original		100.2

4.11. Preparing the OVS tube

It is anticipated that this glass sampling tube can be used to collect a broad range of airborne contaminants when packed with various adsorbents. Therefore, the suffix will reflect the typeof adsorbent contained in the sampler. For example, a sampler containing Tenax will be designated OVS-T and one containing XAD-7 will be called OVS-7.

4.11.1. Apparatus

4.11.1.1. Soxhlet extractor

4.11.1.2. Rotary evaporator

4.11.1.3. Miscellaneous glassware: vacuum flask, 2-L round-bottom flask, Erlenmeyer flask, 250-mL Buchner funnel with coarse fritted disc, etc.

4.11.1.4. Urethane foam plugs, 3/8-in. x 1/2-in. diameter and 3/16-in. x 1/2-in. diameter.

4.11.1.5. Glass fiber filters, 1/2-in. or 13-mm diameter.

4.11.1.6 .PTFE retainer. The retainer is made by removing a 50° arc from a piece of PTFE tubing, 1/8-in. x 1/2-in. o.d. x 3/8-in. i.d.

4.11.1.7. Glass sampling tube. The sampling tube is constructed from two pieces of borosilicate glass tubing that have been joined together by a glass blower. One of the pieces is 50 mm x 13-mm o.d. x 11-mm i.d. The other piece is 25 mm x 6-mm o.d. x 4-mm i.d. (Figure 4.11.4.)

4.11.1.8. Plastic cap, 7/8 in. x 1/2-in. i.d. (Alliance Plastics, Inc., Erie, PA).

4.11.1.9. Plastic cap, 3/4 in. x 7/32-in. i.d. (SKC, Inc., Eighty-Four, PA).

4.11.2. Reagents

4.11.2.1. Toluene, HPLC grade.

4.11.2.2. Methanol, HPLC grade.

4.11.2.3. Acetonitrile, HPLC grade.

4.11.2.4. Amberlite XAD-2 non-ionic polymeric adsorbent, 20/60 mesh (Aldrich Chemical, Milwaukee, WI)

4.11.3. Cleaning the XAD-2 adsorbent

Add 500 g of crude XAD-2 adsorbent to a large Erlenmeyer flask and pour in enough water to cover the adsorbent. Swirl the flask to wash the beads and discard the adsorbent that floats to the surface of the water. Filter the adsorbent using a Buchner funnel. Transfer the beads back to the Erlenmeyer flask and repeat the water wash and filtration. Allow the adsorbent to air dry in the funnel for several minutes before removing the vacuum. Transfer the dried adsorbent to a Soxhlet extractor and extract the material with 1.5 L of methanol for 24 h. Replace the contaminated methanol with 1.5 L of toluene and continue extracting for another 24 h. Replace the toluene with 1.5 L of fresh methanol and continue extracting for 24 h. Each time the contaminated solvent is removed, pull air through the Soxhlet thimble to remove any trapped solvent that has not drained. Rinse the adsorbent in the thimble with some of the new solvent. After the third washing in the Soxhlet extractor, transfer the resin to an Erlenmeyer flask and swirl for 5 min with enough acetonitrile to cover the adsorbent. Filter the resin and repeat the acetonitrile rinse procedure again. Transfer the cleaned adsorbent to a round-bottom flask and remove the acetonitrile with the rotary evaporator. The cleaned adsorbent is now ready to be packed into sampling tubes.

4.11.4. Assembly of the OVS-2 tube

Place a large foam plug in the bottom of the large end of the glass tube. Add 140 mg of cleaned XAD-2 adsorbent to the tube. With the adsorbent beads level, place the small foam plug on the beads. Add 270 mg of cleaned XAD-2 adsorbent and insert the glass fiber filter. The filter should form a small cup and touch the entire inside circumference of the tube. The PTFE retainer is inserted inside the glass tube. Gently press the PTFE retainer against the glass fiber filter. Cap the ends of the sampling tube. (Figure 4.11.4.)

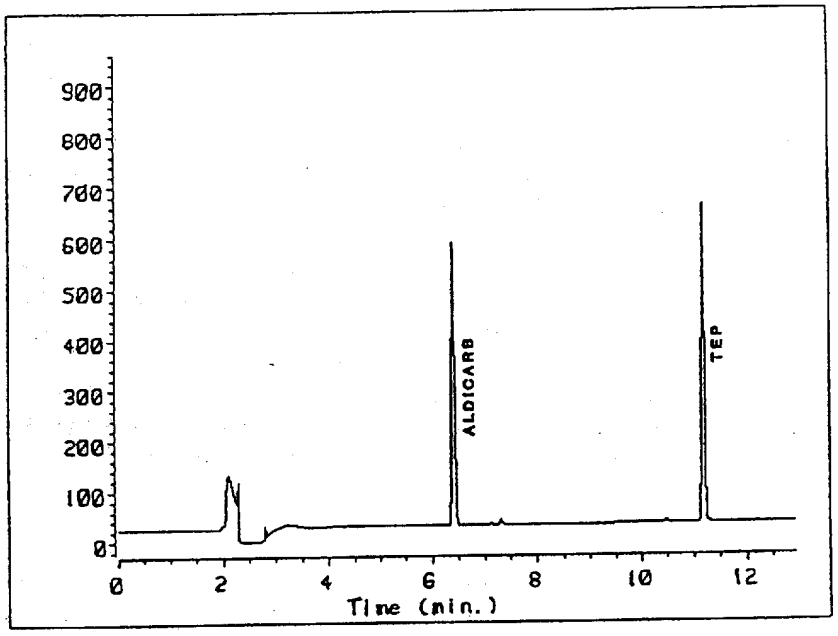

Figure 3.5.1. Chromatogram of aldicarb at the target concentration.

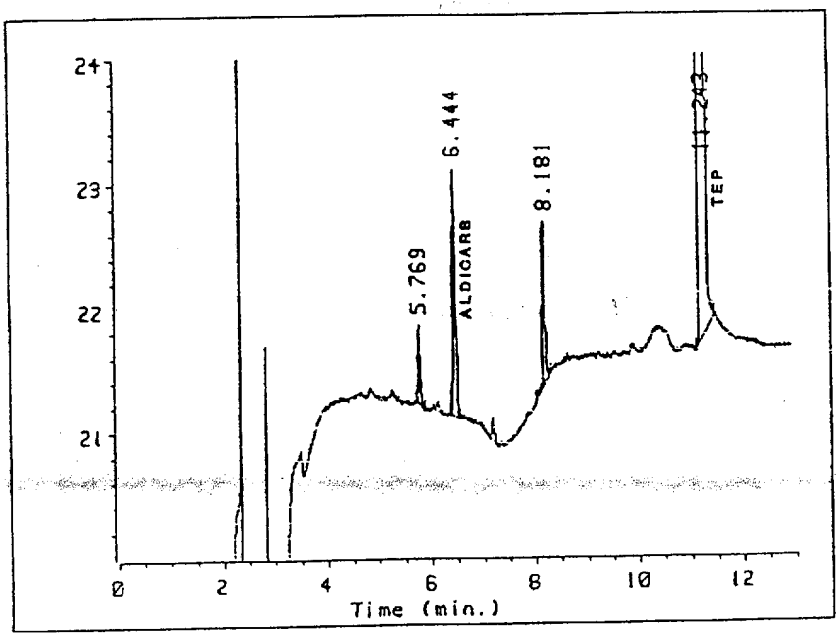

Figure 4.1.1. Chromatogram of aldicarb at the detection limit (60 pg per injection).

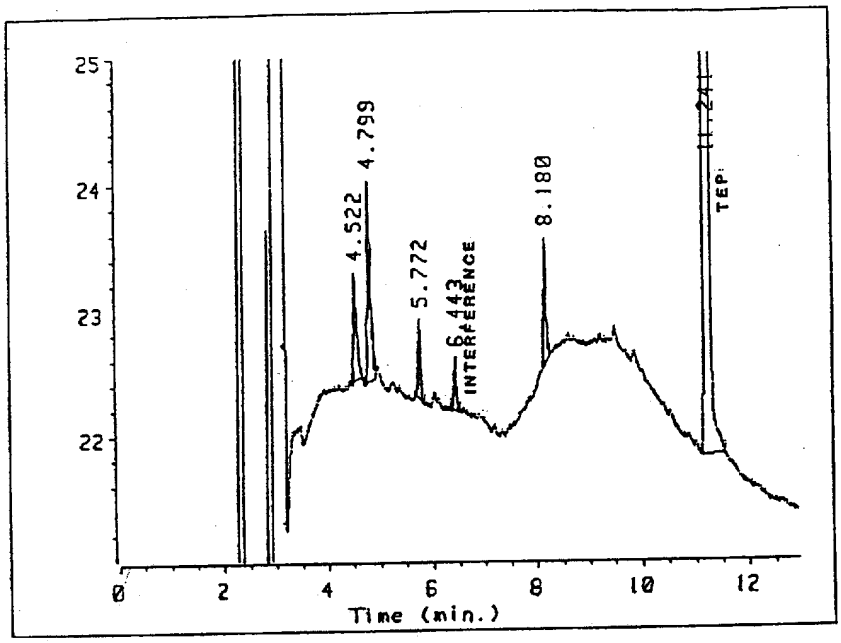

Figure 4.1.2. Chromatogram of a blank "A" section.

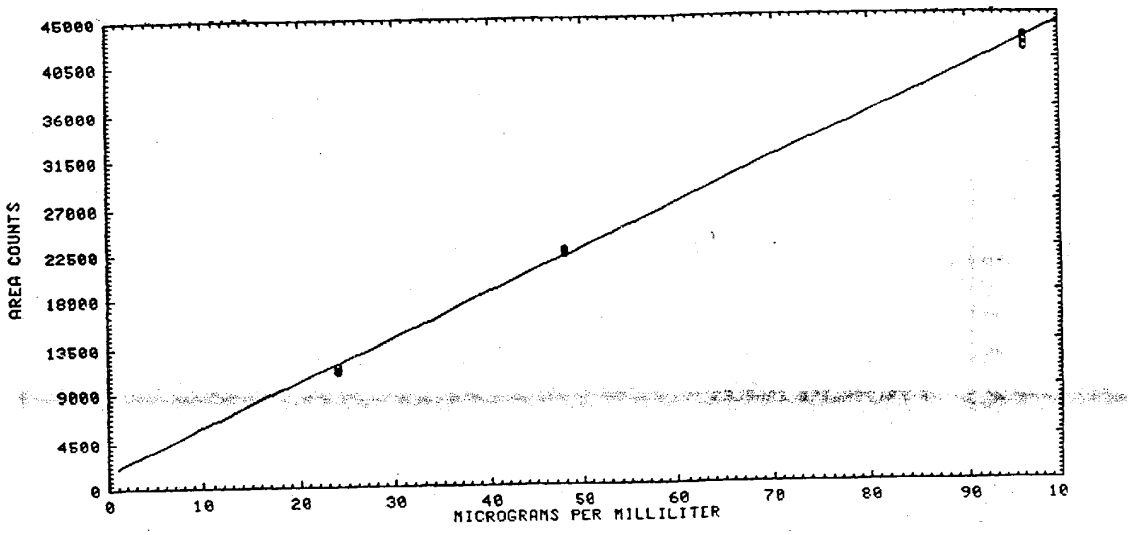

Figure 4.4. Instrument response curve for aldicarb, slope = 424 area counts per microgram per milliliter.

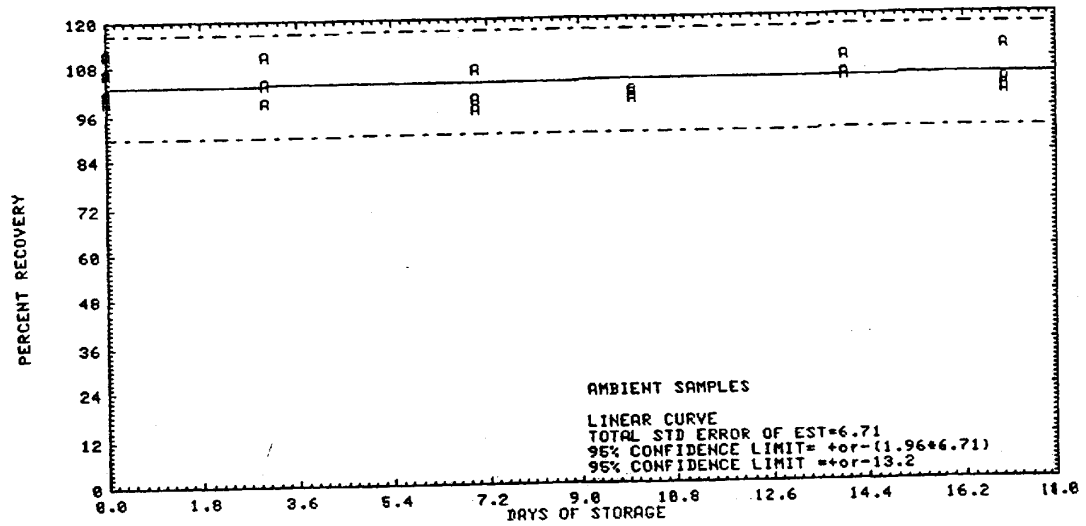

Figure 4.5.1. Ambient storage test for aldicarb, liquid-spiked.

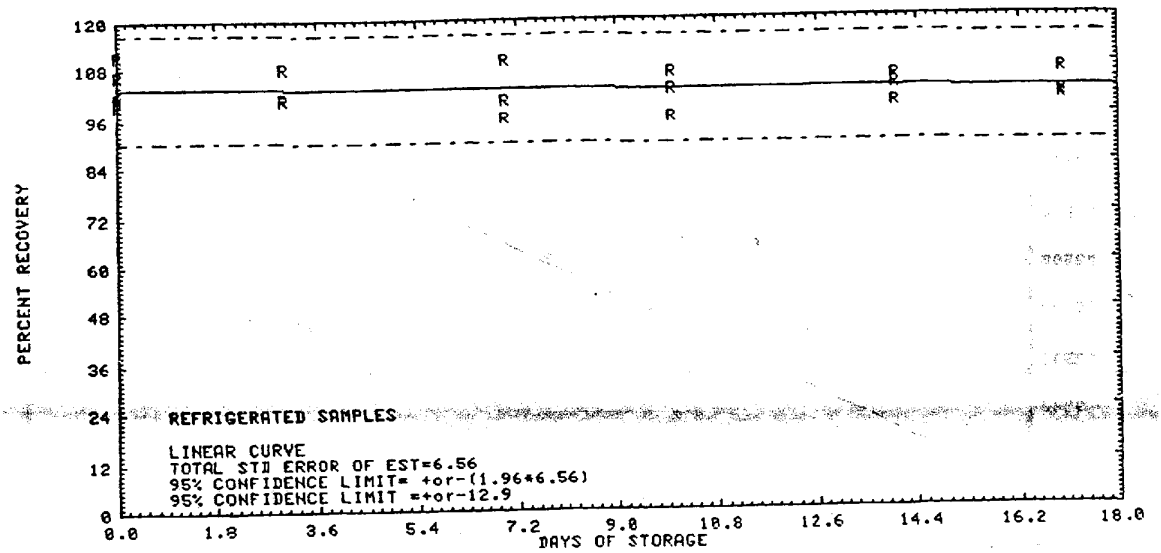

Figure 4.5.2. Refrigerated storage test for aldicarb, liquid-spiked.

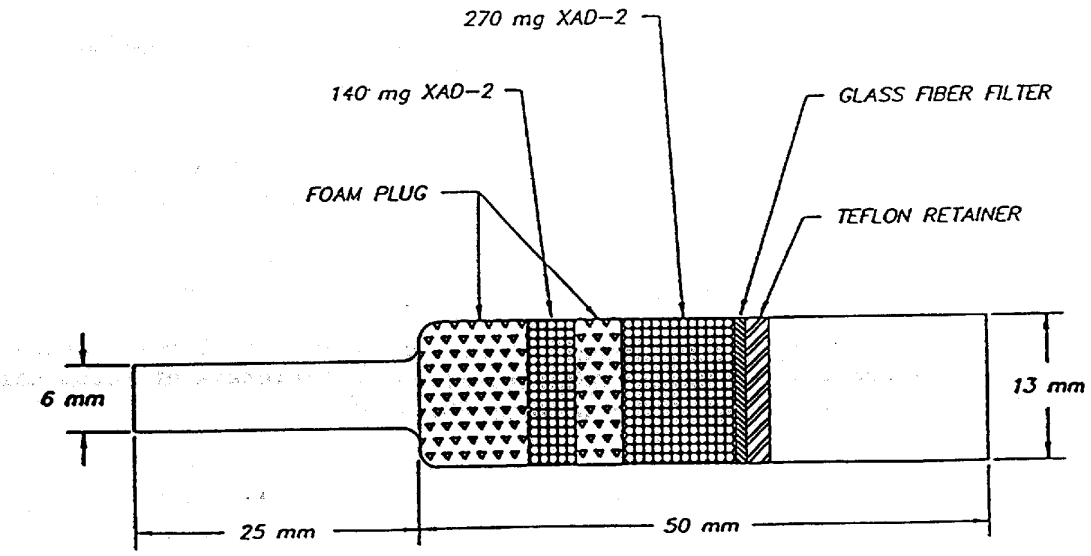

Figure 4.11.4. A drawing of an OVS-2 sampling tube.

5. References

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5.5. "Chemical Information Manual", U.S. Department of Labor, Occupational Safety and Health Administration, Directorate of Technical Support, Washington, D.C.; October 20, 1987.

5.6. "Initial Scientific and Mini economic Review of Aldicarb", U.S. Environmental Protection Agency, Office of Pesticide Programs, Criteria and Evaluation Division; Washington, D.C.; 1975; EPA-540/1-75-013.

5.7. NIOSH Registry of Toxic Effects, RTECS Online Database available through National Library of Medicine, Bethesda, MD.