CHLORDANE (TECHNICAL GRADE)

Method no.:	67
Matrix:	Air
Target concentration:	0.5 mg/m^3 (OSHA PEL)
Procedure:	Samples are collected by drawing known volumes of air through OSHA Versatile Sampler tubes containing a glass fiber filter and two sections of XAD-2 adsorbent (OVS-2). Samples are extracted/desorbed with toluene and analyzed by GC using an electron capture detector.
Recommended air volume and sampling rate:	480 L at 1.0 L/min
Reliable quantitation limit:	0.064 µg/m^3
Standard error of estimate at the target concentration: (Section 4.6.)	7.7%
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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Chemist: Donald Burright

Organic Methods Evaluation Branch
OSHA Analytical Laboratory
Salt Lake City, Utah

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 technical grade chlordane. The OVS-2 is a specially prepared glass tube containing a glass fiber filter and XAD-2 resin (Section 4.9.) which will collect both vapors and aerosols. Previously, the OVS-2 tube was successfully used for a number of organophosphorus pesticides and a carbamate pesticide (Refs. 5.1. and 5.2.).

In the past, technical grade chlordane was collected on a 37-mm mixed cellulose ester membrane filter mounted in a cassette and backed up by a Chromosorb 102 sorbent tube. The analytical procedure developed by NIOSH (Method 5278) required that the sample be desorbed with toluene and analyzed by GC with an electron capture detector (ECD). (Ref. 5.3.) The NIOSH approach was to base the analysis on and γ-chlordane plus several related isomers which are typically present in technical grade chlordane.

Upon initial investigation, it was unclear if the OSHA PEL pertained to technical grade chlordane or to -α and γ-chlordane. Since the PEL was adopted from the American Conference of Government Industrial Hygienists (ACGIH) TLV, a number of the papers (Refs. 5.4.-5.6.) that were cited in the Documentation of the TLVs (Ref. 5.7.) were examined. These papers all indicate that technical grade chlordane was the material under consideration. Although technical grade chlordane was determined to be the pertinent species, the OSHA analytical approach allows for the quantitation of α- and γ-chlordane when the information is desirable. Technical grade chlordane, which has been carefully analyzed for α- and γ-chlordane content using a primary standard of pure isomers, is used to prepare analytical standards. Technical grade chlordane is preferred over α- and γ-chlordane for analytical standards because it is more readily available and it provides a chromatographic fingerprint pattern. Because most samples of technical grade chlordane have sufficiently matchable fingerprints, a sample of the bulk material is not required with each set of samples for use as the analytical standard. A bottle of technical grade chlordane can be calibrated with the primary standard and used to make standards for most samples. On the occasion when the fingerprint match between samples and the analytical standards is inadequate, bulk material from the sampling site may be required or analysis for the specific major components of technical grade chlordane can be performed.

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

Technical grade chlordane is toxic to humans by ingestion, skin absorption and inhalation. It has been estimated that the fatal oral dose for an adult lies between 6 and 60 g, with onset of symptoms within 45 min to several hours after ingestion. (Ref. 5.8.)

Technical grade chlordane is a stimulant to the central nervous system but its exact mode of action is unknown. The general symptoms are convulsions and tremors followed by depression. Cycles of excitement and depression may be repeated several times. Other symptoms are liver damage, anorexia and weight loss. (Ref. 5.8.)

Chronic poisoning in animals produces loss of appetite and degenerative lesions in the liver and renal tubules. Like most halogenated hydrocarbon insecticides, technical grade chlordane is very slowly metabolized and excreted primarily in the feces. It also is stored in body fat and is recognized as an inducer of hepatic microsomal enzyme activity. (Ref. 5.8.)

International Agency for Research on Cancer (IARC) reports on the carcinogenicity of chlordane (Ref. 5.9.): "There is sufficient evidence that technical grade chlordane is carcinogenic in mice. A report of a number of cases of cancer in humans was also available, but these data do not allow an evaluation of the carcinogenicity of technical grade chlordane to humans to be made."

1.1.3. Workplace exposure

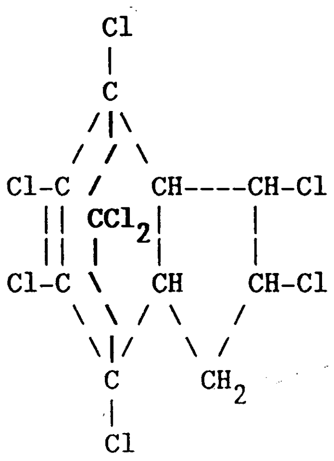
The major use of technical grade chlordane is for termite control, in which treatment of the site with a 1% emulsion is enough to give residual control for at least 5 years. The size of the work force potentially exposed is not available, but would include workers involved in the manufacture, formulation, application and transportation of technical grade chlordane. (Ref. 5.10.)

1.1.4. Physical properties (Ref. 5.9. unless otherwise indicated)

Capillary GC and GC-MS investigations show that technical grade chlordane contains at least 50 compounds, which in the majority of cases belong to the tetrahydromethanoindene or tetrahydromethanoindane system. The typical principle constituents are heptachlor, α-chlordane, γ-chlordane, α-nonachlor, β-nonachlor, α-chlordene, β-chlordene and γ-chlordene. (Ref. 5.11.)

CAS no.:	57-74-9
vapor pressure:	0.0013 Pa at 25°C (1×10^{-5} mm Hg)
appearance:	viscous, amber-colored liquid
density:	1.59-1.63 g/mL at 25°C
viscosity:	0.75-1.20 stokes at 55°C
refractive index:	1.56-1.57 at 25°C
solubility:	insoluble in water, soluble in most organic solvents
trade names:	1068; Aspon; Belt; CD 68; Chlordan; Chlor Kil; Chlorindan; Chlorodane; Corodane; Cortilan-neu; Dowchlor; ENT-9932; HCS 3260; Kypchlor; M140; M410; Niran; Octachlor; Octa-Klor; Oktaterr; Ortho-Klor; Synklor; Tatchlor 4; Topichlor; Toxichlor; Velsicol 1068

structural formula of α- and γ-chlordane:



1.2. Limit defining parameters (The analyte air concentrations listed throughout this method are based on an air volume of 480 L and a solvent extraction/desorption volume of 4 mL. All of the amounts listed below are for technical grade chlordane but the actual peaks analyzed were α- and γ-chlordane. The bulk material of technical grade chlordane used in this evaluation contained 6.2% α-chlordane and 7.0% γ-chlordane.)

1.2.1. Detection limit of the analytical procedure

The detection limit of the analytical procedure is 7.6 pg per injection. This is the amount of analyte which gave α- and γ-chlordane peaks with individual heights of about 5 times the height of the contaminates in the solvent (as shown in the blank). (Section 4.1.)

1.2.2. Detection limit of the overall procedure

The detection limit of the overall procedure is 30.5 ng per sample (0.064 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 30.5 ng per sample (0.064 µg/m^3). This is the smallest amount of analyte which can be quantitated within the requirements of a recovery of at least 75% and a precision (±1.96 SD) of ±25% or better. (Section 4.2.)

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 technical grade chlordane from samples used in a 15-day storage test remained above 95.6% when the samples were stored at about 22°C. (Section 4.6.) The recovery of an analyte from the collection medium during storage must be 75% or greater.

1.2.6. Precision (analytical procedure)

The pooled coefficient of variation obtained from replicate determinations of analytical standards at 0.5, 1 and 2 times the target concentration is 0.028. (Section 4.3.)

1.2.7. Precision (overall procedure)

The precision at the 95% confidence level for the 15-day ambient temperature storage test is ±15.1%. (Section 4.6.) 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 technical grade chlordane and a draft copy of this procedure were given to a chemist unassociated with this evaluation. The samples were analyzed after 65 days of storage at about -10°C. No individual sample deviated from its theoretical value by more than the precision reported in Section 1.2.7. (Section 4.7.)

1.3. Advantage

This sampling device has the capability to collect both vapor and aerosols of technical grade chlordane without having to use two separate samplers in series.

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 OVS-2 tubes, which are specially made 13-mm o.d. glass tubes that are tapered to 6-mm o.d., packed with a 140-mg backup section and a 270-mg sampling section of cleaned XAD-2, and a 13-mm diameter glass fiber filter. The backup section is retained by two foam plugs and the sampling section is between one foam plug and the glass fiber filter. The glass fiber filter is held next to the sampling section by a polytetrafluoroethylene (PTFE) retainer. (Section 4.9., Figure 4.9.4.)

2.2. Reagents

No sampling reagents are required.

2.3. Sampling technique

2.3.1. Attach the small end of the sampling tube 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. The sampler should be attached 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. The blank should be handled the same as the other samples except that no air is drawn through it.

2.4. Retention efficiency and sampler capacity

2.4.1. To test the sampler's ability to retain technical grade chlordane, twice the target concentration of technical grade chlordane, 488 µg, was liquid-spiked onto eight sampling tubes. Humid air (about 72% relative humidity) was pulled through the tubes for 1 to 16 h at 1 L/min. When the samples were analyzed, it was found that the analyte was present on the glass fiber filter and the front section of the XAD-2 at levels equal to 95-100% of the total amount spiked. No technical grade chlordane was found on any of the back sections. (Section 4.8.)

2.4.2. An aerosol of technical grade chlordane was generated and the atmosphere was sampled for 65 min with an OVS-2 tube. The front section contained 4.35 mg of technical grade chlordane and the back section contained 10.1 µg. This represented a breakthrough value of 0.2%. The amount of analyte on the front section was equivalent to a sample containing 18 times the PEL at the recommended air volume of 480 L.

2.5. Extraction and desorption efficiencies (Section 4.5.)

2.5.1. The combined extraction/desorption efficiency for technical grade chlordane from the the glass fiber filter and the large XAD-2 section at the target concentration was essentially 100%.

2.5.2. The extraction efficiency for technical grade chlordane from glass fiber filters at the target concentration was essentially 100%.

2.5.3. The average desorption efficiency for technical grade chlordane from the lot of cleaned XAD-2 adsorbent used in this evaluation over the range of 0.5 to 2 times the target concentration was 100.0%.

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

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 recommended sampling rate is 1.0 L/min. The reliable quantitation limit for a 15-min sample is 2 µg/m^3.

2.7. Interferences (sampling)

Suspected interferences 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 an electron capture detector. For this evaluation a Hewlett-Packard 5840A Gas Chromatograph with a Nickel 63 ECD and a 7671A Autosampler was used.

3.1.2. A GC column capable of separating the peaks of α- and γ-chlordane from the rest of the technical grade chlordane peaks. A 30-m x 0.53-mm i.d. (1.5-µm depth of film) DB-1 Megabore column was used in this evaluation.

3.1.3. An electronic integrator or other suitable means of measuring detector response. A Hewlett-Packard 18850A GC Terminal was 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. Chlordane (technical grade) 45%, (Dexol Industries).

3.2.2. Toluene, (American Burdick & Jackson).

3.2.3. Hexachlorobenzene (HCB) 97%, (Aldrich Chemical Co.). This may be used as the internal standard in the extracting/desorbing solution. The solution is prepared by adding 2 mg of HCB to 1 L of toluene.

3.3. Standard preparation

3.3.1. Prepare stock standards by adding toluene to preweighed amounts of technical grade chlordane, be careful to include the percentage of purity in the calculation. If α- and γ-chlordane are requested for analysis and the percentages of these compounds have not previously been determined in the technical grade chlordane stock standards,prepare primary standards by adding toluene to preweighed amounts of α- and γ-chlordane. The solutions containing the pure chlordane isomers are used to quantitate the amount of α- and γ-chlordane in the technical grade chlordane that is used to prepare the analytical standards.

3.3.2. Prepare analytical standards by injecting microliter amounts of diluted technical grade chlordane stock standards into vials containing 4.0 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 facilitate the transfer of the adsorbent. Discard the rear foam plug. Do not discard the glass sampling tube; it can be reused after cleaning with surfactant or suitable solvent.

3.4.2. Add 4.0 mL of extracting/desorbing solution to each vial.

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

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

3.5. Analysis

3.5.1. Analytical conditions

temperatures:	210°C (column) 250°C (injector) 300°C (detector)
column gas flow:	9 mL/min (95% argon/5% methane)
make-up gas flow:	24 mL/min (95% argon/5% methane)
injection size:	1.0 µL
column:	DB-1, 1.5 µm thick, 30 m x 0.53-mm i.d. fused silica (J & W Scientific)
retention time:	7.7 min for γ-chlordane 8.5 min for α-chlordane
chromatogram:	Figure 3.5.1.

3.5.2. Measure detector response using a suitable method such as electronic integration. Since there is only one source of manufactured technical grade chlordane (Velsicol Chemical Corp.), the pattern of peaks in the technical grade chlordane standards should look like the pattern in the samples. When the patterns are different use the bulk material from the sampling site as an analytical standard if the bulk material is readily available or analyze the samples for the major constituents of technical grade chlordane: heptachlor, α- and γ-chlordane, α-nonachlor, β- and γ-chlordene. (Section 4.10.) Since some formulations of technical grade chlordane have had heptachlor added to them, quantitate the amount of heptachlor separately and report the amount.

3.5.3. Use an internal standard procedure to prepare a calibration curve using several solutions 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 technical grade chlordane (the summed response for the and γ-chlordane peaks) versus the analytical standard concentrations (in terms of micrograms technical grade chlordane per milliliter). Determine the best-fit line through the data points by curve-fitting.

3.7.2. Determine the concentration, in micrograms of technical grade chlordane per milliliter, for a particular sample by comparing its detector response (the summed response for the α- and γ-chlordane peaks) to the calibration curve. Add the amount of technical grade chlordane on the backup section to the amount found on the front section. Perform blank corrections for each section before adding the results together.

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

$$\text{mg/m}^3 = (A)(B)/(C)(D)$$

where A = concentration of technical grade chlordane from Section 3.7.2.
B = extraction/desorption volume in milliliters
C = liters of air sampled
D = combined extrac./desorp. efficiency (decimal)

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

3.7.4. If the requested information is for the concentration of α- and γ-chlordane, prepare calibration curves using the information about the concentration of α- and γ-chlordane determined in Section 3.3.1.

3.8. Safety precaution (analytical)

3.8.1. Because technical grade chlordane is an animal carcinogen, handle it as if it were a human carcinogen. Observe a similar precaution with the pure isomers of α- and γ- chlordane.

3.8.2. Avoid skin contact and inhalation of all chemicals.

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

3.8.4. 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 is 7.63 pg per injection of technical grade chlordane. This amount produced peaks of α- and γ-chlordane whose individual heights are about 5 times the height of the contaminates in the solvent. The injection volume recommended in the analytical procedure (1.0 µL) was used in the determination of the detection limit for the analytical procedure. (Figure 4.1.)

4.2. Detection limit of the overall procedure and reliable quantitation limit

The detection limit of the overall procedure and the reliable quantitation limit are 30.5 ng per sample (0.064 µg/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 and in the determination of the reliable quantitation limit. Six samples were liquid-spiked with a solution containing technical grade chlordane equal to the analytical detection limit (30.5 ng/4 mL = 7.63 pg/µL x 1 µL = 7.63 pg). Since the recovery of technical grade chlordane from the samples was high and approximately equal to the detection limit of the aiilytical procedure, the detection limit of the overall procedure and reliable quantitation limit are the same.

Table 4.2
Reliable Quantitation Limit
(30.5 ng/sample)

sample	% recovered	statistics
1	108.8	
2	107.5	$\bar{X}$ = 106.0
3	107.5	SD = 3.8
4	98.9	1.96 SD = 7.4
5	108.8	
6	104.5	

4.3. Precision (analytical method only)

The precision of the analytical method was determined by multiple injections of technical grade chlordane standards and summing the area counts for the peaks of α- and γ-chlordane.

Table 4.3.
Technical Grade Chlordane Precision Data

x target conc. µg/sample	0.5x 122	1x 244	2x 488
area	503818	932305	1844530
counts	536341	956413	1768390
	530464	991515	1837810
	536679	1027010	1788040
	540964	951229	1822170
	546248	949299	1885210
	538045	950531	1808700
	540952	959986	1867040
	561979	939683	1873950
	509477	989442	1824240
$\bar{X}$	534497	964741	1832008
SD	16935	29058	37547
CV	0.0317	0.0301	0.0205

$\overline{CV} = 0.0279$

4.4. Instrument response to the analytes

The data in Table 4.3. is presented graphically in Figure 4.4. This figure is a calibration curve over the concentration range of 0.5 to 2 times the target concentration. The instrument response is linear over this range. The slope of the line (14200 area counts per µg/mL) is a measure of the response of the instrument to the analyte.

4.5. Extraction and desorption efficiencies

4.5.1. Extraction from glass fiber filter

The extraction efficiency of technical grade chlordane was determined by liquid-spiking six glass fiber filters with technical grade chlordane at the target concentration (244 µg/sample). These samples were stored overnight and then extracted with toluene and analyzed.

Table 4.5.1.
Extraction Efficiency
(1.0x Target Concentration)

extraction	efficiency, %
98.7	102.3
102.9	102.3
102.7	100.5

$\bar{X} = 101.8$

4.5.2. Desorption from XAD-2 adsorbent

The desorption efficiency of technical grade chlordane was determined by liquid-spiking 270-mg portions of XAD-2 adsorbent with technical grade chlordane at 0.5 to 2 times the target concentration. These samples were stored overnight and then desorbed with toluene and analyzed.

Table 4.5.2.
Desorption Efficiency

x target conc. µg/samples	0.5x 122	1x 244	2x 488
desorption efficiency,	99.7	100.7	100.9
	98.7	101.1	100.3
	99.2	100.3	99.2
	98.4	101.1	100.9
	99.0	100.4	100.3
	98.8	100.5	99.5
$\bar{X}$	99.0	100.7	100.2

The average desorption efficiency over the studied range was 100.0%.

4.5.3. Combined extraction/desorption efficiency

The combined extraction/desorption efficiency of technical grade chlordane 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 toluene and analyzed. The samples were reanalyzed two days later with new standards to test the stability of extracted/desorbed samples. The results are listed below.

Table 4.5.3.
Stability
of Extracted/Desorbed Samples

	original	48 h later
extraction/ desorption efficiency, %	102.7	99.1
	99.5	99.6
	100.3	99.0
	101.4	99.6
	100.1	98.6
	99.4	98.2
$\bar{X}$	100.6	99.0
% of original = 98.4		

4.6. Storage data

4.6.1. Storage samples were generated by liquid-spiking 36 sampling tubes with technical grade chlordane, 244.2 µg, and then pulling 20 L of humid air through them (about 74% relative humidity). One-half of the tubes was stored in a freezer (-14 °C) and the other half was stored in a closed drawer at ambient temperature (about 22 °C). Three samples from each group were analyzed periodically over a 15-day period. The results are given below and shown graphically in Figures 4.6.1.1. and 4.6.1.2.

Table 4.6.1.
Storage Tests (Liquid Spiked)

storage time (days)	ambient recovery (percent)			refrigerated recovery (percent)		
0	97.7	97.4	97.9	97.7	97.4	97.9
	97.1	97.8	97.5	97.1	97.8	97.5
3	98.8	--	98.3	97.8	97.4	97.5
6	95.5	96.5	95.2	98.4	98.6	97.4
9	96.1	96.4	96.0	96.6	95.8	96.6
13	98.0	98.1	98.0	98.2	99.1	97.9
15	98.7	--	94.8	93.2	90.7	92.6

4.6.2. Another storage test was performed by sampling a dynamically generated aerosol test atmosphere (9.6 mg/m^3 and about 80% relative humidity) of technical grade chlordane. The average value of the Day 0 samples was 202.2 µg/sample. Thirty-six samples were collected in the aerosol chamber at 1 L/min for 21 min. One half of the samples was stored at -14 °C and the other half was stored at ambient temperature in a closed drawer. Three samples of each set were analyzed periodically over a 15-day test. The results were normalized to the average of the Day 0 samples by dividing µg collected on Day 0 into the µg recovered for each sample. The normalized results are given below and shown graphically in Figures 4.6.2.1. and 4.6.2.2.

Table 4.6.2.
Storage Tests (Dynamic Atmosphere)

storage time (days)	ambient recovery (percent)			refrigerated recovery (percent)		
0	100	105	100	96.9	103	94.5
	96.9	103	94.5	100	105	100
3	87.3	102	91.4	79.9	86.1	97.2
6	93.7	103	87.9	97.8	99.3	96.9
9	99.0	100	97.9	95.2	90.4	101
13	99.9	109	94.1	92.9	103	107
15	91.7	92.6	90.6	95.5	99.3	97.9

4.7. Reproducibility data

Six samples, liquid-spiked with technical grade chlordane, were given to a chemist unassociated with this study. The samples were analyzed after being stored for 65 days at -10^{0}C. The results were not corrected for extraction/desorption efficiency and are shown in Table 4.7. None of the data had a percent deviation greater than the precision of the overall procedure of ±15.1%.

Table 4.7.
Reproducibility Data

sample	µg spiked	% recovered	% deviation
1	366	98.4	-1.6
2	244	91.3	-8.7
3	366	92.4	-7.6
4	244	94.2	-5.8
5	244	92.8	-7.2
6	244	92.1	-7.9

4.8. Retention efficiency

To test the ability of the sampler to retain the analytes, eight samplers were liquid-spiked with twice the target concentration (488 µg) of technical grade chlordane. Humid air (about 72% relative humidity) was pulled through the samplers for 1 to 16 h at 1 L/min. No technical grade chlordane broke through onto the backup section of the sampling tube.

Table 4.8.
Retention of Technical Grade Chlordane

air volume (L)	µg recovered	% recovered
15.2	480.1	96.0
30.3	484.3	96.7
45.5	480.2	96.1
60.6	482.6	96.5
90.9	448.1	90.7
120.0	475.7	95.3
181.8	476.3	95.4
255.0	477.1	95.5

4.9. Preparation of the OVS-2 tube

It is anticipated that this sampler containing different adsorbents can be used to collect a broad range of airborne contaminants. For these applications the suffix will reflect the type of resin 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.

4.9.1. Apparatus

4.9.1.1:. Soxhlet extractor

4.9.1.2. Rotary evaporator

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

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

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

4.9.1.6. 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.

4.9.1.7. Glass sampling tube. The sampling tube is constructed of 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 4.9.4.)

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

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

4.9.2. Reagents

4.9.2.1. Acetonitrile, HPLC grade.

4.9.2.2. Toluene, HPLC grade.

4.9.2.3. Methanol, HPLC grade.

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

4.9.3. Cleaning of 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 acetonitrile for 24 h. Replace the contaminated acetonitrile with toluene and continue extracting for another 24 h. Replace the toluene with fresh acetonitrile 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 methanol. Filter the resin and repeat the methanol rinse procedure again. Transfer the cleaned adsorbent to a round-bottom flask and remove the methanol with the rotary evaporator. The cleaned adsorbent is now ready to be packed into sampling tubes.

4.9.4. 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 tothe tube. With the 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 4.9.4.)

4.10. Comparisons of commercially available technical grade chlordane

A study was done to compare several different sources of technical grade chlordane to check the fingerprint pattern of the chromatogram. An eight-year old solution from Dexol (D1) labeled 73% technical grade chlordane was obtained from a private source. Two other solutions were purchased from local hardware stores. One was Black Flag (BF) containing 45.3% technical grade chlordane and the other was Dexol (D2) containing 45.0% technical grade chlordane.

To determine how well the labeled concentrations of the three solutions compared to each other, two weighed aliquots of each solution were diluted with toluene. From each of the six stock solutions three dilute solutions were prepared which were approximately 0.5, 1 and 2 times the target concentration. The six dilute standards from each source (D1, BF, or D2) were then used to determine the concentration of the remaining solutions from the other two sources. The results of this round-robin analysis were then averaged and compared to the labeled percentages.

Table 4.10.
Comparison of Technical Grade Chlordane

	standard		
sample	DI	BF	D2
D1	--	78.9%	83.1%
BF	41.6%	--	47.5%
D2	39.2%	42.9%	--

The fingerprint patterns of the three solutions were similar to each other. After comparing the results, the two newer solutions were found to be close to their labeled percentages and that over the years the D1 solution had loss some of its solvent. The concentration of the D1 solution is now about 81% technical grade chlordane.

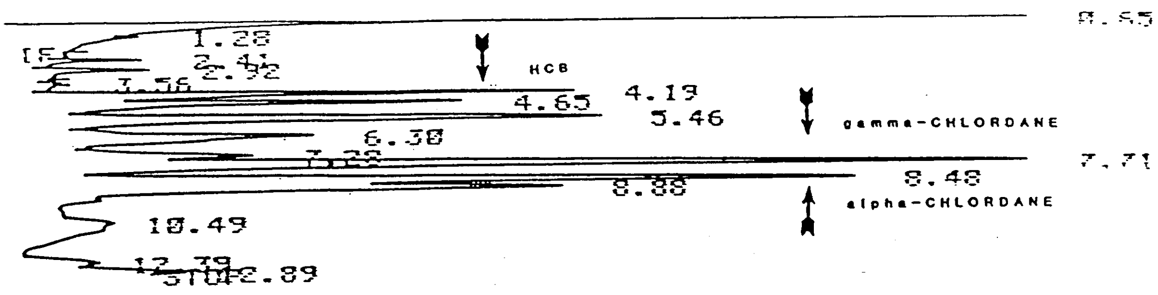


Figure 3.5.1. Chromatogram of technical grade chlordane at the target concentration.

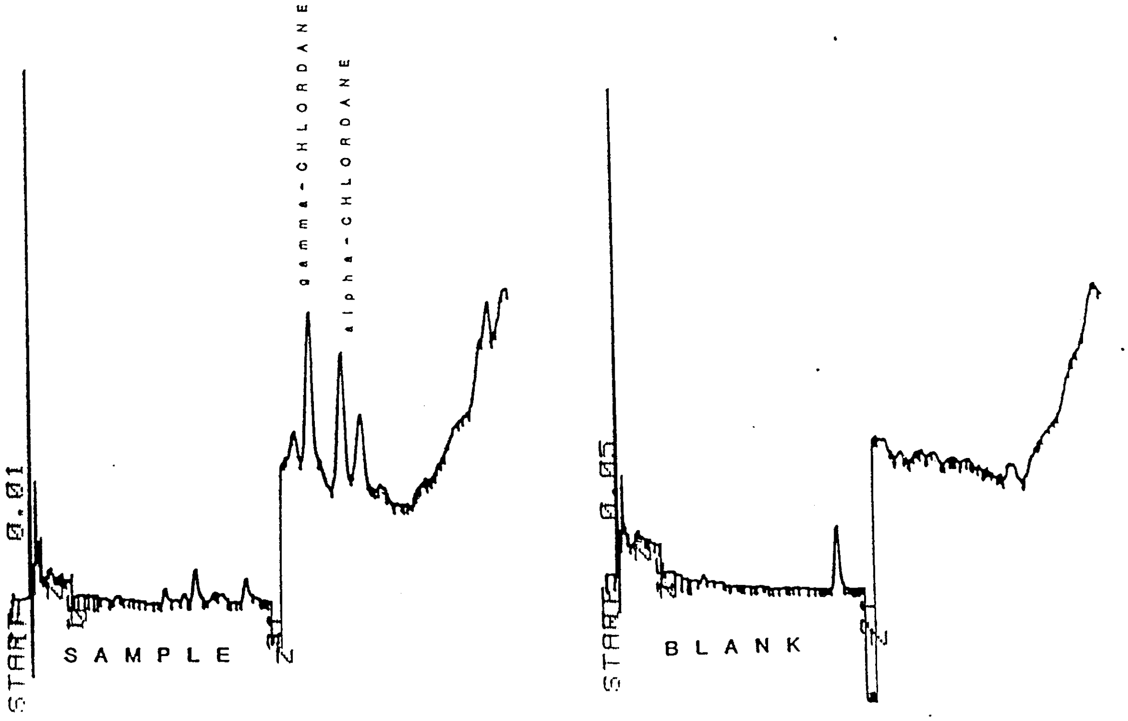


Figure 4.1. Chromatogram of technical grade chlordane at the detection limit (7.63 µg/injection) compared to a blank.

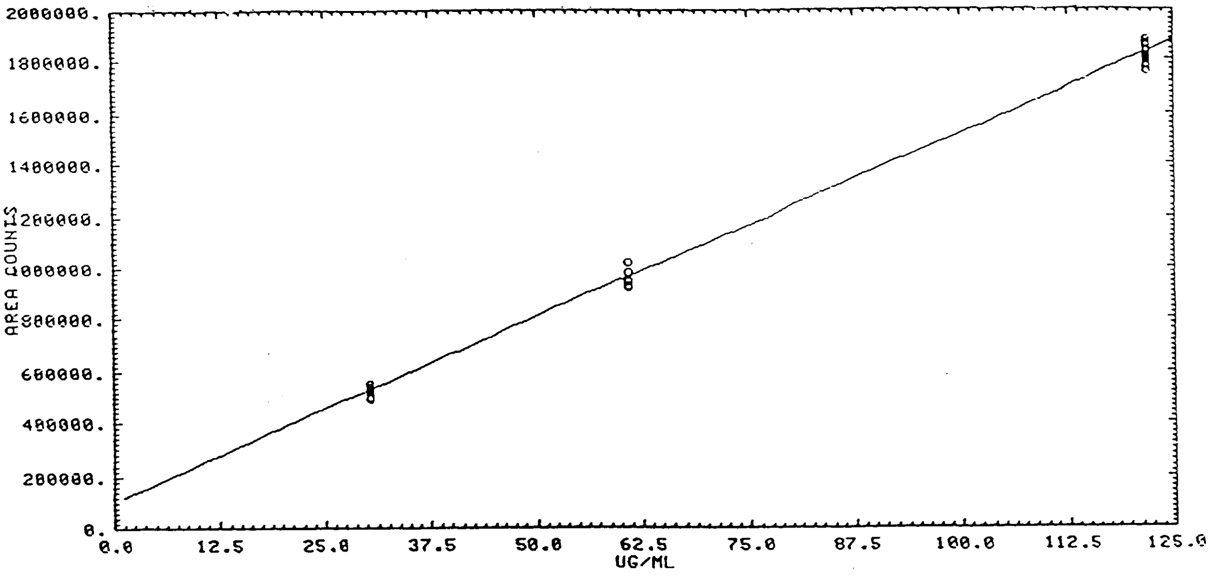


Figure 4.4. Calibration curve for technical grade chlordane, slope = 14200 area counts per microgram per milliliter.

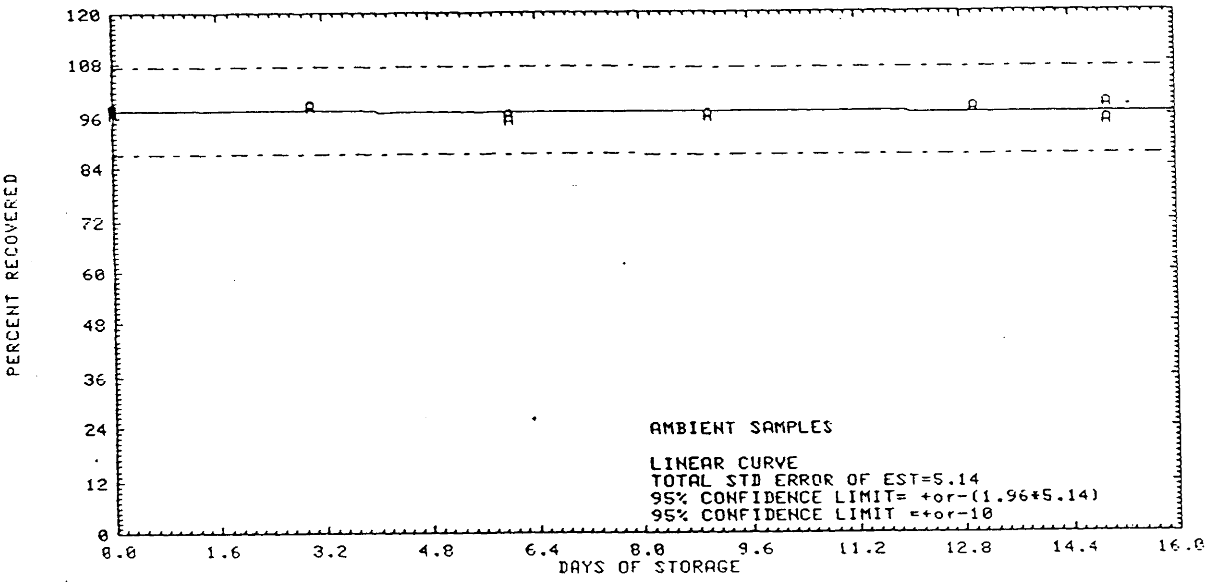


Figure 4.6.1.1. Ambient storage for technical grade chlordane, liquid spiked.

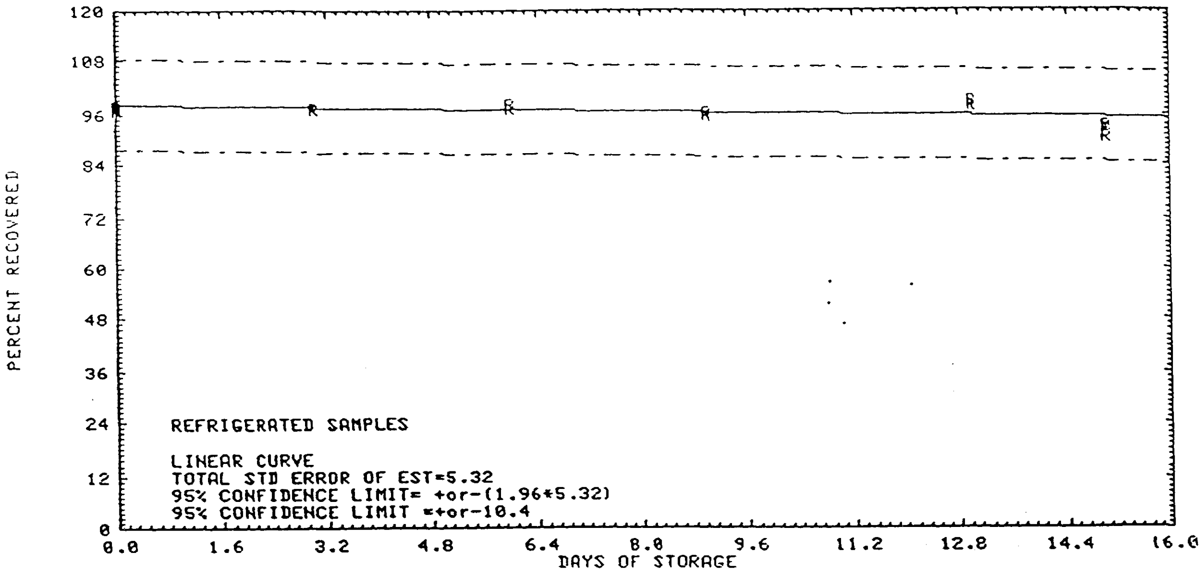


Figure 4.6.1.2. Refrigerated storage test for technical grade chlordane, liquid spiked.

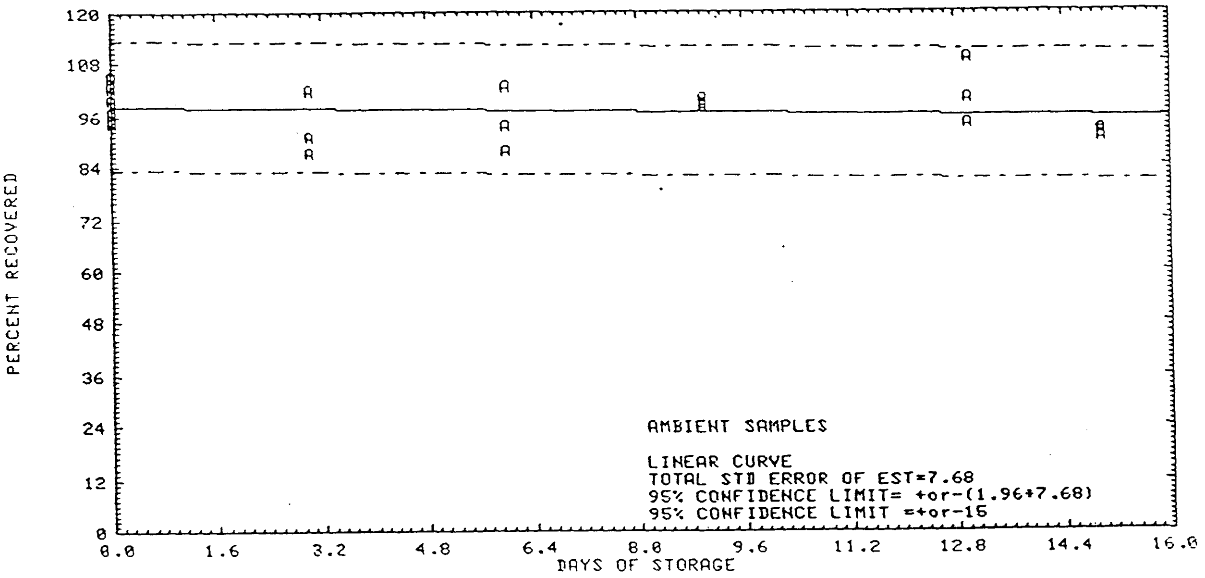


Figure 4.6.2.1. Ambient storage test for technical grade chlordane, generated aerosol.

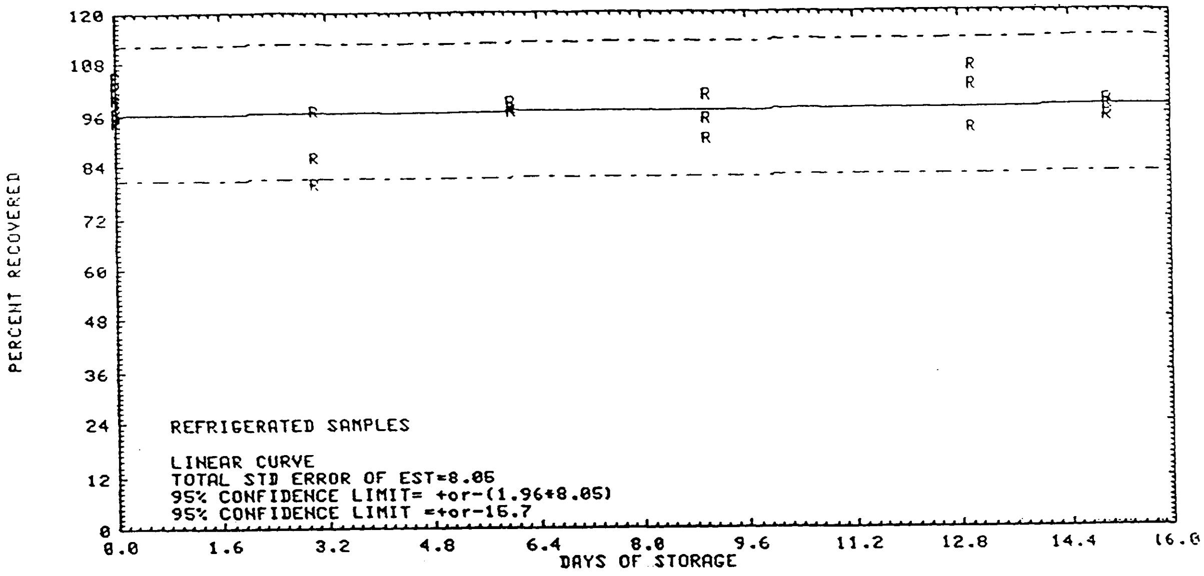


Figure 4.6.2.2. Refrigerated storage test for technical grade chlordane, generated aerosol.

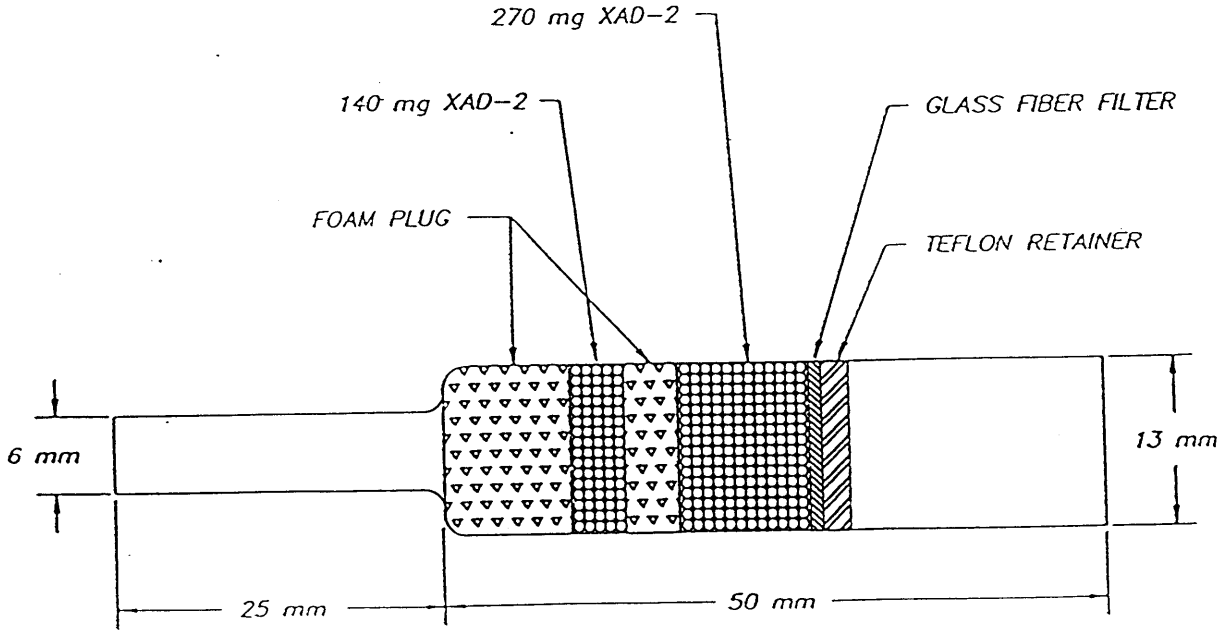


Figure 4.9.4. A drawing of an OVS-2 tube.

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