Method number:	PV2029
Target Concentration:	0.5 mg/m^3 OSHA permissible exposure limit (PEL), skin.
Procedure:	Samples are collected by drawing known volumes of air through OSHA versatile sampler (OVS-2) tubes, each containing a glass fiber filter and two sections of XAD-2 adsorbent. Samples are desorbed with toluene and analyzed by gas chromatography (GC) using an electron capture detector (ECD).
Recommended air volume and sampling rate:	60 minutes at 1.0 L/min (60 L)
Detection limit of the overall procedure	0.49 µg/m^3 (based on the recommended air volume and the analytical detection limit):
Status of method:	Partially Validated method. This method has been partially evaluated and is presented for information and trial use only.

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Ing-Fong Chan

Carcinogen and Pesticide Branch
OSHA Salt Lake Technical Center
Salt Lake City UT 84115-1802

1 General Discussion

1.1 Background

1.1.1 History of procedure

This evaluation was undertaken to determine the effectiveness of the OVS-2 tube as a sampling device for heptachlor. It follows the procedure developed for chlordane. (Ref. 5.1)

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

Heptachlor has an acute oral LD_{50} of 40-188 mg/kg and an acute dermal LD_{50} of 119-320 mg/kg for rats. (Ref. 5.2 to 5.4) Animal studies indicating clearly the carcinogenicity in mice and rats have been reported. (Ref. 5.2 to 5.4) Due to these and other factors, the ACGIH has set a threshold limit value (TLV) of 0.5 mg/m^3 for heptachlor with a skin notation. (Ref. 5.4) This level has also been adopted as the OSHA PEL.

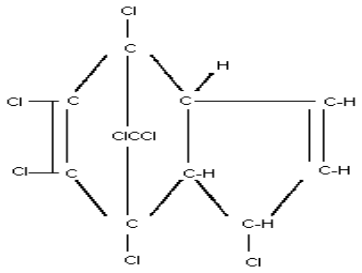
1.1.3 Potential workplace exposure

Heptachlor is used as an insecticide. In the United States, a total of 2.3 million kg of heptachlor was produced in 1980. (Ref. 5.2) No information could be found on the number of workers exposed to heptachlor.

1.1.4 Physical properties (Ref. 5.2 to 5.6)

CAS number:	76-44-8
IMIS number:	1369
Molecular weight:	373.35
Molecular formula:	$C_{10}H_5Cl_7$
Boiling point:	135 – 145 °C
Solubility:	Practically insoluble in water (0.056 mg/L), soluble in ethanol (4.5 g/100 mL), xylene (102 g/100 mL), carbon tetrachloride (112 g/100 mL), acetone (75 g/100 mL) and benzene (106 g/mL).
Chemical name:	1,4,5,6,7,8,8-Heptachloro-3a,4,7,7a-tetrahydro-4,7-methano-lH-indene
Synonyms:	3-Chlorochlordene; 3,4,5,6,7,8,8a-heptachlorodicyclo-pentadiene; 1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-4,7-endomethanoindene; 1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-4,7-methanoindene; 1(3a),4,5,6,7,8,8-heptachloro-3a(1),4,7,7a-tetrahydro-4,7-methanoindene; 3a,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-4,7-methanoindene; 1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-4,7-methyleneindene; 1,4,5,6,7,10,10-heptachloro-4,7,8,9-tetrahydro-4,7-methyleneindene; 1,4,5,6,7,10,10-heptachloro-4,7,8,9-tetrahydro-4,7-endomethyleneindene
Trade names:	Aahepta; Agroceres; Drinox; E 3314; ENT 15,152; GPKh; H34; Heptachlorane; Heptagran; Heptamul; Rhodiachlor; Velsicol 104
Appearance:	white, crystalline solid

Structure:



1.2 Limit defining parameters

The detection limit of the analytical procedure, including a 17:1 split ratio, is 0.43 pg per injection. This is the amount of analyte which will give a peak whose height is approximately five times the baseline noise.

2 Sampling Procedure

2.1 Apparatus

2.1.1 A personal sampling pump that can be calibrated to within ±5% of the recommended flow rate with the sampling device in line.

2.1.2 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, a 270 mg sampling section of cleaned XAD-2 adsorbent, 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. (Figure 1.)

2.2 Reagents

No sampling reagents are required.

2.3 Sampling technique

2.3.1 Immediately before sampling, remove the plastic end caps from the OVS-2 tube.

2.3.2 Attach the small end of the tube to the sampling pump with flexible tubing.

2.3.3 Attach the tube vertically in the employee's breathing zone in such a manner that it does not impede work performance.

2.3.4 After sampling for the appropriate time, remove the tube and seal with plastic caps.

2.3.5 Wrap each sample end-to-end with a Form OSHA-21 seal.

2.3.6 Record the air volume for each sample, and list any possible interference.

2.3.7 Submit at least one blank for each set of samples. Handle the blank in the same manner as the samples, except no air is drawn through it.

2.3.8 Submit bulk samples for analysis in a separate container. Limit the amount of bulk submitted to one gram or one ml. Do not ship them with air samples.

2.4 Desorption efficiency (glass fiber filter and XAD-2 adsorbent)

Fifteen vials, each containing a 13-mm glass fiber filter and 270 mg of XAD-2 adsorbent, were each liquid spiked on the glass fiber filter with a 2.95 mg/mL solution of heptachlor. The first five filters were each spiked with 5 µL (.5× PEL) of the solution. The second five were each spiked with 10 µL (1× PEL) and the remaining five were each spiked with 20 µL (2× PEL) of the solution. These vials were allowed to sit overnight at ambient temperature, desorbed with 4.0 mL of toluene, shaken for 30 min and then analyzed as in Section 3. The results are listed in Table 2.4.

Table 2.4
Desorption Efficiency

sample #	µg spiked	µg found	% recovered
D1	14.80	14.12	95.4
D2	14.80	15.05	101.7
D3	14.80	13.84	93.5
D4	14.80	14.77	99.8
D5	14.80	15.88	107.3
D6	29.50	29.50	100.0
D7	29.50	30.78	104.3
D8	29.50	30.48	103.3
D9	29.50	30.58	103.7
D10	29.50	30.83	104.5
D11	59.00	58.30	98.8
D12	59.00	58.86	99.8
D13	59.00	58.73	99.5
D14	59.00	58.61	99.3
D15	59.00	58.69	99.5

average of 0.5x PEL = 99.5%
average of 1x PEL = 103.2%
average of 2x PEL = 99.4%

2.5 Retention efficiency

Six OVS-2 tubes were each liquid spiked with 20 µL (2× PEL) of a 2.95 mg/mL solution of heptachlor in toluene. These were allowed to dry for 2 hours and then 60 L of humid air (~80% relative humidity) were drawn through each tube at 1 L/min. The tubes were stored overnight in a drawer at ambient temperature, desorbed with 4.0 mL of toluene, shaken for 30 min and then analyzed as in Section 3. The results are listed in Table 2.5.

Table 2.5
Retention Efficiency

sample #	µg spiked	µg found	% recovered
R1	59.00	58.17	98.6
R2	59.00	57.02	96.6
R3	59.00	58.77	99.6
R4	59.00	58.57	99.3
R5	59.00	58.07	98.4
R6	59.00	59.21	100.4

average = 98.8%

2.6 Sample storage

Twenty-four OVS-2 tubes were each liquid spiked with 10 µL (1× PEL) of 2.95 mg/mL of heptachlor. These were allowed to dry for 2 hours and then 60 L of humid air (~80% relative humidity) were drawn through each tube at 1 L/min. Half of the tubes were stored in a drawer at ambient temperature, and the other half were stored in a freezer (-5 °C). They were extracted and analyzed as in Section 3. The results are given in Tables 2.6.1 and 2.6.2.

Table 2.6.1
Sample Storage Ambient

days stored	µg spiked	µg found	% recovered
0	29.50	28.99	98.3
0	29.50	28.99	98.3
0	29.50	29.72	100.7
4	29.50	26.32	89.2
4	29.50	28.77	97.5
4	29.50	29.53	100.1
7	29.50	28.14	95.4
7	29.50	29.06	98.5
7	29.50	29.54	100.1
7	29.50	28.77	97.5
7	29.50	28.78	97.6
7	29.50	29.88	101.3

average of 0 days = 99.1%
average of 4 days = 95.6%
average of 7 days = 98.4%

Table 2.6.2
Sample Storage Freezer (- 5 ^{0}C)

days stored	µg spiked	µg found	% recovered
0	29.50	29.48	99.9
0	29.50	28.09	95.2
0	29.50	29.67	100.6
4	29.50	27.53	93.3
4	29.50	29.35	99.5
4	29.50	25.47	86.3
7	29.50	29.55	100.2
7	29.50	29.93	101.5
7	29.50	30.53	103.5
7	29.50	29.86	101.2
7	29.50	29.68	100.6
7	29.50	29.13	98.7

average of 0 days = 98.6%
average of 4 days = 93.0%
average of 7 days = 100.9%

2.7 Recommended air volume and sampling rate

2.7.1 The recommended air volume is 60 L.

2.7.2 The recommended flow rate is 1.0 L/min.

2.8 Interferences (sampling)

It is not known if any compounds will interfere with the collection of heptachlor. Any suspected interferences should be reported to the laboratory.

2.9 Safety precautions (sampling)

2.9.1 Attach the sampling equipment in such a manner that it will not interfere with work performance or employee safety.

2.9.2 Follow all safety practices that apply to the work area being sampled.

3 Analytical Procedure

3.1 Apparatus

3.1.1 A balance capable of weighing to the nearest tenth of a milligram. A Mettler HL52 balance was used in this evaluation.

3.1.2 A mechanical shaker.

3.1.3 A GC equipped with an ECD. A Hewlett Packard (HP) 5890 equipped with an autosampler was used in this evaluation.

3.1.4 A GC column capable of separating heptachlor from any interference. A 45-m × 0.25-mm i.d. (0.25 µm d_f SE-54) capillary column was used in this evaluation.

3.1.5 An electronic integrator or some other suitable means for measuring detector response. The Hewlett-Packard 3357 Laboratory Data System was used in this evaluation.

3.1.6 Volumetric flasks and pipets.

3.1.7 Vials, 2-mL and 4-mL.

3.2 Reagents

3.2.1 Toluene, reagent grade.

3.2.2 Heptachlor, reagent grade. A standard obtained from EPA (EPA # 3860, 99.8% purity) was used in this evaluation. .

3.3 Standard preparation

Prepare stock standards by adding toluene to preweighed amounts of heptachlor. Prepare working range standards by diluting stock standards with toluene. Store stock and working standards in a freezer.

3.4 Sample preparation

3.4.1 Transfer the 13-mm glass fiber filter and the 270 mg sampling section of the tube to a 4-mL vial. Place the first foam plug and the 140 mg backup section in a separate 4-mL 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.

3.4.2 Add 4.0 mL of toluene to each vial and seal with a PTFE-lined cap.

3.4.3 Shake the vials for 30 minutes on a mechanical shaker.

3.4.4 If necessary, transfer the samples to 2-mL vials for use on an HP autosampler.

3.5 Analysis

3.5.1 Instrument conditions

Column:	45-m × 0.25-mm i.d., (0.25 µm d_f SE-54)
Injector temperature:	250 °C
Column temperature:	230 °C
Detector temperature:	300 °C
Gas flows:	
Column:	2.14 mL/min hydrogen
ECD make up:	20 mL/min nitrogen
Injection volume:	1 µL
Split ratio:	17:1
Retention time:	6.75 min

3.5.2 Chromatogram (Figure 2.)

3.6 Interferences (analytical)

3.6.1 Any collected compound having a similar retention time to that of the analyte is a potential interference.

3.6.2 GC conditions may generally be varied to circumvent interferences.

3.6.3 Retention time on a single column is not proof of chemical identity. Analysis by an alternate GC column, high performance liquid chromatography (HPLC) and confirmation by mass spectrometry are additional means of identification.

3.7 Calculations

3.7.1 Construct a calibration curve by plotting detector response versus standard concentration (µg/mL) of heptachlor.

3.7.2 Determine the µg/mL of heptachlor in both sections of each sample and blank from the calibration curve.

3.7.3 Blank correct each sample by subtracting the µg/mL found in each section of the blank from the µg/mL found in the corresponding sections of the sample and then add the two sample sections together.

3.7.4 Determine the air concentration by using the following formula.

$$mg / m^3 = \frac{(\mu g / mL,\ blank\ corrected)(desorption\ volume,\ mL)}{(air\ volume,\ L)(desorption\ efficiency,\ decimal)}$$

3.8 Safety precautions (analytical)

3.8.1 Avoid skin contact and air exposure to heptachlor.

3.8.2 Avoid skin contact with all solvents.

3.8.3 Wear safety glasses at all times.

4 Recommendation for Further Study

This method should be fully validated.

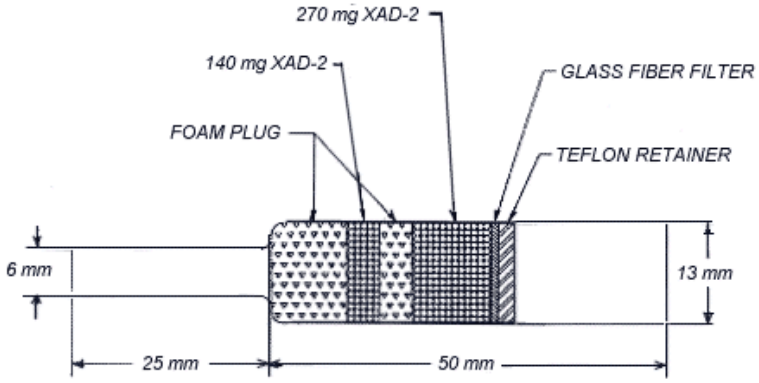


Figure 1 OVS-2 Sampling Device

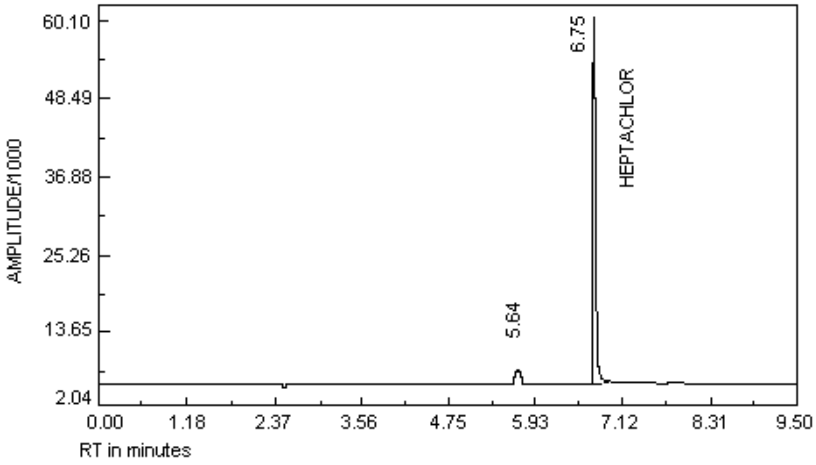


Figure 2 Chromatogram of Heptachlor

5 References

5.1 Burright, D.; Method #67, "Chlordane (Technical Grade)"; OSHA Analytical Laboratory, 1987; unpublished.

5.2 IARC Monographs on the Evaluation of the Carcinogenic Risk of Chemicals to Humans; International Agency for Research on Cancer: Lyon, 1979; Vol. 20, pp 129-153.

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5.4 Documentation of the Threshold Limit Values and Biological Exposure Indices, American Conference of Governmental Industrial Hygienist INC., fifth edition, 1986; p 296.

5.5 Farm Chemicals Handbook; Berg, Gordon L. Ed.; Meister: Willoughby, Ohio, 1986; p C123.

5.6 Merck Index, 10th ed.; Windholz, Martha ED.; Merck: Rahway, N.J., 1983; p 673.