CHLORDANE

$C_{10}H_6Cl_8$ MW: 409.80 CAS: 57-74-9 RTECS: PB9800000

METHOD: 5510, Issue 2 **EVALUATION: FULL** **Issue 1: 15 May 1989**
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OSHA : 0.5 mg/m^3 (skin)
NIOSH: 0.5 mg/m^3 (skin); carcinogen; Group I Pesticide
ACGIH: 0.5 mg/m^3 (skin), suspect human carcinogen

PROPERTIES: liquid; d 1.59 to 1.63 g/mL @ 25 °C; BP 175 °C; VP 0.0013 Pa (1.0 x 10^{-5} mm Hg) @ 20 °C

SYNONYMS: 1,2,4,5,6,7,8,8-octachloro-3a,4,7,7a-tetrahydro-4,7-methanoindane and isomers; Toxichlor; Octachlor

SAMPLING

SAMPLER: FILTER AND SOLID SORBENT TUBE (0.8-µm cellulose ester membrane; Chromosorb 102, 100/50 mg)

FLOW RATE: 0.5 to 1 L/min

VOL-MIN: 10 L @ 0.5 mg/m^3
-MAX: 200 L

SHIPMENT: routine

SAMPLE STABILITY: > 1 week @ 25 °C [1]

FIELD BLANKS: 2 to 10 field blanks per set

MEDIA BLANKS: 2 per set

BULK SAMPLE: required

ACCURACY

RANGE STUDIED: 0.16 to 1.17 mg/m^3 [1,2] (120-L samples)

BIAS: 3.0%

OVERALL PRECISION ($\hat{S}_{rT}$): 0.070 [1,2]

ACCURACY: ± 15.3%

MEASUREMENT

TECHNIQUE: GAS CHROMOTOGRAPHY, ELECTRON CAPTURE DETECTOR (GC/ECD)

ANALYTE: Chlordane

EXTRACTION: 10 mL toluene, stand 30 min

INJECTION VOLUME: 2 µL

TEMPERATURE-INJECTION: 250 °C
-DETECTOR: 300 °C
-COLUMN: 205 °C

CARRIER GASES: 95% argon/5% methane @ 75 mL/min

COLUMN: 2 m x 4-mm ID glass packed with 1.5%, SP2250/1.95% SP2401 on 100/120 mesh

CALIBRATION: solution of analyte in toluene or hexane with internal standard

RANGE: 5 to 150 µg per sample [2]

ESTIMATED LOD: 0.1 µg per sample [3]

PRECISION ($\bar{S}_r$): 0.02 @ 6 to 120 µg per sample [2]

APPLICABILITY: The working range is 0.04 to 1.2 mg/m^3 for a 120-L sample. Chlordane, accompanied by a mixture of penta-, hexa-, hepta-, and nonachlorinated compounds, is defined by a group of five chromatographic peaks. It is necessary to de termine the percentage of Chlordane and its isomers in the standards used.

INTERFERENCES: None identified; an alternate column is 2 m x 2-mm ID glass packed with 3% QF-1 on 100/120 mesh Chrom Q.

OTHER METHODS: This is NIOSH method S278 in a revised format. [2]

REAGENTS:

1. Toluene, distilled in glass.
2. Hexane, distilled in glass.
3. Chlordane, 95%.*
4. Calibration stock solution, ca. 6 mg/mL. Dissolve 10 mg Chlordane in 1 mL toluene.
   NOTE: Since Chlordane is available only as a mixture, standardize the solution as follows:
   a. Dilute 10 µL calibration stock solution to 10 mL.
   b. Analyze by steps 11-13.
   c. Divide combined area of Chlordane peaks (Fig. 1) by total area of all peaks to determine fraction Chlordane, f.
5. Internal standard, p,p'-DDT, 98%.*
6. 95% Argon/5% methane mixture, purified.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: 37-mm, 0.8-µm pore size cellulose ester membrane filter supported by a stainless steel screen in a two-piece filter cassette holder plus a 10 cm x 8-mm OD x 6-mm ID, 20/40 Chromosorb 102 tube (front = 100 mg; back = 50 mg), separated by 3-mm silanized glass wool plug, flame sealed ends with plastic caps. Pressure drop across the tube at 1 L/min must not exceed 2.5 mm Hg. Filters and tubes are commercially available.
2. Personal sampling pump, 0.5 to 1 L/min, with flexible connecting tubing.
3. Gas chromatograph, electron capture detector, integrator and column (page 5510-1).
4. Vials, scintillation, 20-mL, PTFE-lined caps.
5. Syringe, 10-µL, readable to 0.1 µL.
6. Flasks, volumetric 10-mL.
7. Bottles, 60 mL, 40-mm ID, straight-sided with a PTFE-lined cap for extracting filter holder and screen.
8. Stopwatch.
9. Manometer.
10. Pipets, 1- and 10-mL an other convenient sizes for preparing standards.
11. Tweezers.

SPECIAL PRECAUTIONS: Chlordane and p,p'-DDT are toxic and rapidly absorbed through the skin [4]. Use gloves and eyeglasses to avoid direct contact with these compounds. Handle these chemicals and organic solvents with care in the laboratory hood. Chlordane is a potential human carcinogen [5].

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Assemble the sampler and connect the sorbent tube (ends broken just before the connection) to the sampling pump with backup section nearest the sampling pump. Keep the sampler in a vertical position during sampling.
3. Sample at an accurately known flow rate between 0.5 and 1 L/min for a total sample size of 10 to 200 L.
4. Remove the sorbent tube from the outlet of the cassette and connect it to the inlet side of the cassette. Cap the open end of the sorbent tube and plug the outlet of the cassette. Ship sampler with appropriate blanks to laboratory.
5. In separate package, ship bulk sample of the suspected material.

SAMPLE PREPARATION:

6. Separate the sampler components for extraction as follows:
   a. Into a bottle, transfer the filter, front sorbent section and glass wool plugs. Add 10.0 mL toluene. After desorption is complete, dilute a 1-mL aliquot to 10 mL for analysis.
   b. Into a second bottle, transfer the stainless steel screen. Using a 10-mL volumetric pipet, rinse the inner surfaces of the cassette into the bottle with hexane.
   c. Into a scintillation vial, transfer the back sorbent section. Add 10.0 mL toluene.
7. Cap each container and allow to stand 30 min. with occasional swirling.
   NOTE: A suitable internal standard such as p,p'-DDT may be added, at 0.4 µg/mL, at this point.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards.
   a. Add known amounts of calibration stock solution to toluene containing internal standard in 10-mL volumetric flasks and dilute to the mark. Use serial dilutions as needed to obtain Chlordane concentrations in the range 0.01 to 15 µg/mL.
   b. Analyze the samples and blanks (steps 11 through 13).
   c. Prepare a calibration graph (ratio of the total Chlordane peak areas to peak area of internal standard vs. µg Chlordane).
9. Determine recovery at least once for each lot of filters and Chromosorb 102 used. Prepare three samplers at each of five levels plus three media blanks.
   a. Place cellulose ester membrane filter and 100 mg of Chromosorb 102 in a bottle.
   b. Add calibration stock solution to the filter and Chromosorb 102 in the container with a microliter syringe. Prepare parallel blank samples with no added analyte.
   c. Cap the bottle. Allow to stand overnight.
   d. Desorb (steps 6 and 7) and analyze with working standards (steps 11 through 13).
   e. Prepare a graph of recovery vs. µg Chlordane recovered.
10. Check recovery at two levels for each sample set in duplicate. Repeat recovery graph determination if checks do not agree to within 5% of recovery graph.

MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 5510-1.
12. Inject 2-µL sample aliquot using solvent flush technique or with autosampler. Make duplicate injections of samples and standards.
    NOTE: If peak area is above the linear range of the working standards, dilute an aliquot of the solution, reanalyze and apply the appropriate dilution factor in calculations.
13. Measure peak areas. Divide the total Chlordane peak area (sum of five peaks; see Fig. 1) by the peak area of internal standard on the same chromatogram.

CALCULATIONS:

14. Determine the mass, µg (corrected for recovery), of Chlordane found in the filter plus front sorbent section (W_f), back sorbent section (W_b), extract from cassette and screen (W_c), and average media blank filter plus front sorbent section (B_f) and back sorbent section (B_b).
    NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.
15. Calculate the concentration, C, of Chlordane in the volume of air sampled, V (L):

$$C = \frac{(W_f + W_b + W_c - B_f - B_b)}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

Method S278 was validated on June 8, 1979, [1,2,6]. The substance used to dynamically generate test atmospheres at 25 °C and 760 mm Hg was Ortho-Klor-72 (40% Chlordane), Velsicol Chemical Corporation. Collection efficiencies and recoveries were close to 1.00 in the range 6 to 120 µg per sample. No significant breakthrough was observed after 240 min of sampling an atmosphere of 1.1 mg/m^3 Chlordane at a flow rate of approximately 1 L/min. Samples spiked with Chlordane, extracted with toluene, and stored one week at room temperature gave recoveries of 96 to 100%. Overall precision ($\hat{S}_{rT}$) was 0.07. No significant bias was found.

REFERENCES:

[1] NIOSH Backup Data Report S278 (June 8, 1979) for Chlordane prepared under NIOSH Contract No. 210-76-0123 (1979).
[2] NIOSH Manual of Analytical Methods, 2nd. ed., V. 6, S278, U.S. Department of Health and Human Services, Publ. (NIOSH) 80-125 (1980).
[3] UBTL, Inc. NIOSH Seq. Report 4-999-K (July 19, 1985, unpubl).
[4] NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards. U.S. Department of Health and Human Services Publ. (NIOSH) 81-123 (1981), available as stock #PB 83-154609 from NTIS, Springfield, VA 22161.
[5] NIOSH Research Report-Development and Validation of Methods for Sampling and Analysis of Workplace Toxic Substances, U.S. Department of Health and Human Services, Publ. (NIOSH) 80-133 (1980).

METHOD REVISED BY:

Gangadhar Choudhary, Ph.D., ATSDR; S278 originally validated under NIOSH Contract No. 210-76-0123.

Figure 1. Chromatogram of Chlordane analytical standard.