

CHLORINATED CAMPHENE

5039

$C_{10}H_{10}Cl_8$ (average)

MW: 414 (average)

CAS: 8001-35-2

RTECS: XW5250000

METHOD: 5039, Issue 1

EVALUATION: PARTIAL

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OSHA : 0.5 mg/m³ (skin)
NIOSH: lowest feasible (skin), carcinogen, Class I Pesticide
ACGIH: 0.5 mg/m³ (skin); STEL 1 mg/m³ (skin)
 (1 ppm = 16.9 mg/m³ @ NTP)

PROPERTIES: solid; MP 70 °C to 95 °C; BP (760 mm Hg) decomposes; sp. gr. 1.63; VP 0.03 to 0.05 kPa (0.2 to 0.4 mm Hg) @ 20 °C

SYNONYMS: toxaphene

APPLICABILITY: The working range is 0.05 to 1.5 mg/m³ for a 15-L air sample.

INTERFERENCES: Other pesticides such as aldrin, parathion, dieldrin, DDT and its metabolites, and polychlorinated biphenyls elute in the retention time band for chlorinated camphene.

OTHER METHODS: This is Method S67 [2] in a revised format. Toxaphene in air has been collected on Chromosorb 102 with acceptable recovery [3].

REAGENTS:

1. Chlorinated camphene* (toxaphene).
2. Petroleum ether*, 30 to 60 °C, suitable for pesticide residue analysis.
3. Nitrogen, purified.
4. Calibration stock solution, 0.1 mg/mL. Dissolve 5 mg of chlorinated camphene in 50 mL petroleum ether.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: 37-mm cellulose ester membrane filter (0.8- μ m pore size) supported by a cellulose backup pad in a three-piece filter holder. Do not use tenite filter holders.
2. Personal sampling pump, 0.2 to 1 L/min, with flexible connecting tubing.
3. Gas chromatograph, ⁶³Ni ECD, integrator, and column (page 5039-1).
4. Vials, 20-mL with PTFE-lined screw caps for shipping filters and pads.
5. Syringes, 10- μ L and larger sizes for preparation of standard solutions.
6. Pipet, 10-mL.
7. Volumetric flasks, 10-mL.
8. Tweezers, stainless steel.

SPECIAL PRECAUTIONS: Petroleum ether is highly flammable. Prepare samples and standards in well-ventilated hood. Overexposure to chlorinated camphene may cause nausea, mental confusion, and unconsciousness; contact with chlorinated camphene solutions may cause skin irritation [4].

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 0.2 and 1 L/min for a sample size of 2 to 30 L.
3. Carefully transfer the filter and backup pad to a screw cap vial by means of tweezers.

SAMPLE PREPARATION:

4. Pipet 10 mL petroleum ether into each vial and seal.
5. Gently swirl the vial to wet the filter and backup pad. Allow to stand 30 min.

CALIBRATION AND QUALITY CONTROL:

6. Calibrate daily with at least six working standards.
 - a. Add known amounts of calibration stock solution to petroleum ether in 10-mL volumetric flasks and dilute to the mark to obtain concentrations in the range 0.14 to 14 μ g chlorinated camphene/10 mL.
 - b. Analyze with samples and blanks (steps 9 and 10).
 - c. Prepare calibration graph (sum of peak areas in the retention time band vs. μ g chlorinated camphene).
7. Determine recovery (R) at least once for each batch of filters used for sampling in the working range. Prepare three filters at each of five levels plus three media blanks.
 - a. Deposit a known amount of calibration stock solution onto the filter. Allow filters to air dry.
 - b. Store samples overnight in vials.
 - c. Prepare (steps 4 and 5) and analyze with samples (steps 8 and 9).
 - d. Prepare a graph of R vs. μ g chlorinated camphene recovered.
8. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and R graph are in control.

MEASUREMENT:

9. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 5039-1. Inject 5- μ L aliquots manually using solvent flush technique or with autosampler. NOTE: If peak areas are outside the calibration range, make dilution of sample or standard solution, analyze and apply the dilution factor in calculations.
10. Sum the peak areas in the retention time band. Use bulk sample to identify the appropriate retention time band.

CALCULATIONS:

11. Determine the mass, μ g (corrected for R), of chlorinated camphene found in each sample (W) and average media blank (B).
12. Calculate concentration, C, of chlorinated camphene in the air volume sampled, V (L):

$$C = \frac{(W - B)}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

This method was evaluated over the range 0.22 to 1.2 mg/m³ at 22 °C and 761 mm Hg using 15-L samples [1]. No reference method was used. The collection efficiency for the filters was found to be 1.0 using a challenge concentration of 1.3 mg/m³ for 30 L. Average recoveries of chlorinated camphene from cellulose ester filters were 91% to 98% in the range 0.7 to 14 μ g per sample. Cassettes were found to be unsuitable for storage of 0.7 μ g chlorinated camphene on filters for 1 day at room temperature.

REFERENCES:

- [1] Documentation of the NIOSH Validation Tests, S67, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977), available as Stock No. PB 274-248 from NTIS, Springfield, VA 22161.
- [2] NIOSH Manual of Analytical Methods, 2nd. ed., V. 2, S67, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
- [3] Thomas, Thomas C.; Nishioka, Yoshimi A., "Sampling of Airborne Pesticides using Chromosorb 102," Bull. Environ. Contam. Toxicol., 35(4), 460-5 (1985).
- [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 No. PB 83-154609 from NTIS, Springfield, VA 22161.

METHOD REVISED BY:

James E. Arnold, NIOSH/DPSE; Method S67 originally developed under NIOSH Contract CDC-99-74-45.