$C_{10}Cl_{10}O$ MW: 490.68 CAS: 143-50-0 RTECS: PC8575000

METHOD: 5508, Issue 2 **EVALUATION: PARTIAL** **Issue 1: 15 February 1984**
Issue 2: 15 August 1994

OSHA : no standard
NIOSH: 0.001 mg/m^3; carcinogen
ACGIH: no standard

PROPERTIES: solid; MP 350 °C (dec); VP <4 x 10^{-5} Pa (3 x 10^{-7} mm Hg; 8 µg/m^3) @ 25 °C

SYNONYMS: chlordecone, decachlorotetracyclodecanone

SAMPLING

SAMPLER: FILTER + IMPINGER (0.8-µm cellulose ester membrane + impinger with 0.1 M NaOH)

FLOW RATE: 0.5 to 1 L/min

VOL-MIN: 50 L @ 1 µg/m^3
-MAX: 600 L

SHIPMENT: routine

SAMPLE STABILITY: at least 12 days @ 25 °C

FIELD BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 2.5 to 2.6 µg/m^3 (376-L samples)

BIAS: not determined

OVERALL PRECISION ($\hat{S}_{rT}$): 0.23 [1]

ACCURACY: > ± 45%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, ECD

ANALYTE: Kepone

RECOVERY FROM FILTER: 99:1 benzene:methanol, 10 mL; stand 2 h

RECOVERY FROM IMPINGER: benzene, @ pH <7, two 5-mL portions

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTION: 225 °C
-DETECTOR: 335 °C
-COLUMN: 200 °C

CARRIER GAS: nitrogen, 70 mL/min

COLUMN: 1.8 m x 4-mm glass; 3% OV-1 on 80/100 mesh Chromosorb WHP

CALIBRATION: Kepone in 99:1 benzene:methanol

RANGE: 50 to 500 ng per sample

ESTIMATED LOD: 10 ng per sample

PRECISION ($\hat{S}_r$): 0.014 (filters); 0.060 (impingers)

APPLICABILITY: The working range is 0.001 to 25 mg/m^3 for 50-L air samples. Personal and area samples were collected by this method at a Mirex formulation plant.

INTERFERENCES: Partial conversion of Mirex into Kepone can occur by reaction with NaOH solution in the impinger and pH adjustment. A different column packing which may be used is 3% OV-210 on 80/100 mesh Chromosorb WHP.

OTHER METHODS: This method was originally designated P&CAM 225 [1,2].

REAGENTS:

1. Sodium hydroxide, 0.1 M, aqueous.
2. Benzene, pesticide quality.*
3. Methanol, pesticide quality.*
4. Benzene:methanol, 99:1 (v/v).*
5. Sulfuric acid, 0.05 M.
6. Kepone.*
7. Calibration stock solution, 50 µg/mL. Dissolve 100 mg Kepone in 99:1 benzene:methanol to make 10 mL solution. Dilute 50 µL of this solution to 10 mL with 99:1 benzene:methanol. Stable at least one week at 25 °C.
8. Impinger recovery stock solution, 1 µg/mL. Dissolve 25 mg Kepone in methanol to make 10 mL solution. Dilute 4 µL of this solution to 10 mL with methanol.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: cellulose ester membrane filter, 0.8-µm, 37-mm, in a filter holder followed by a midget impinger with 15 mL 0.1 M NaOH. (Filter holder and impinger connected with short piece of plastic tubing.)
2. Sampling pump, 0.5 to 1 L/min, with flexible connecting tubing.
3. Impinger, midget, with stem removed.
4. Gas chromatograph, electron-capture detector, integrator and column (page 5508-1).
5. Forceps, two pairs.
6. Volumetric flasks, 10-mL.
7. Separatory funnel, 125-mL.
8. Syringes, 10-µL, readable to 0.1-µL; 100-µL, readable to 1 µL.
9. pH paper.
10. Pipet, 5-mL, with pipet bulb.

SPECIAL PRECAUTIONS: Methanol and benzene are flammable.

Benzene and Kepone [3] are carcinogens.

SAMPLING:

1. Connect the outlet of the impinger containing 15 mL 0.1 M NaOH and the inlet of the sampling pump to an empty impinger without a stem to protect the sampling pump from splash over.
2. Calibrate the sampling pump with a representative sampler in line.
3. Sample at an accurately known flow rate between 0.5 and 1 L/min for a total sample size of 50 to 600 L. Do not exceed 2 mg total dust loading on the filter.
4. Separate the filter cassette and impinger for shipment to the laboratory.

SAMPLE PREPARATION:

5. Use forceps to fold and transfer the filter to a 10-mL volumetric flask.
6. Add 10.0 mL 99:1 benzene:methanol.
7. Stopper and shake the flask vigorously for 1 min. Allow the flask to stand at least 2 h.
8. Transfer the solution in the impinger to a separatory funnel. Wash the impinger with two 15-mL portions of 0.1 M NaOH, and add the washings to the separatory funnel. Add 0.05 M H_2SO_4 until the pH is less than 7.
9. Extract the Kepone with two 5-mL portions of benzene. Shake vigorously for 2 min and allow 5 min for the phases to separate. Combine the benzene layers in a 10-mL volumetric flask. Add 100 µL methanol and benzene to the mark.

CALIBRATION AND QUALITY CONTROL:

10. Calibrate daily with at least six working standards in the range 1 to 50 ng/mL.
    a. Dilute 50 µg/mL calibration stock solution with 99:1 benzene:methanol to 3.6 µg/mL.
    b. Add known amounts of 3.6 µg/mL solution to 99:1 benzene:methanol in 10-mL volumetric flasks.

c. Analyze together with samples and blanks (steps 13 through 15).
d. Prepare a calibration graph of peak area or peak height vs. ng of Kepone per sample.

11. Determine the average recovery (R) of Kepone from filters.
    a. Fortify six filters with about 7 µL of 50 µg/mL calibration stock solution.
    b. Allow the filters to air dry.
    c. Prepare samples according to steps 5 through 7.
    d. Analyze samples with standards according to steps 13 through 15.
    e. Calculate the average recovery, R (mass of Kepone found divided by mass of Kepone applied).
12. Determine the average recovery of Kepone from impingers.
    a. Fortify each of six impingers containing 15 mL 0.1 M NaOH solution with 100 µL 1-µg/mL impinger recovery stock solution.
    b. Prepare samples according to steps 8 and 9.
    c. Analyze samples with standards according to steps 13 through 15.
    d. Calculate the average recovery.

MEASUREMENT:

13. Set the gas chromatograph to conditions given on page 5508-1. t_r of Kepone = 4.6 min, and t_r of Mirex = 9.2 min for these conditions.
14. Inject 5-µL aliquots of sample solutions and standards using solvent-flush technique.
    NOTE: Rinse the syringe thoroughly after each injection with methanol and then with benzene, otherwise traces of Kepone will remain in the syringe.
15. Measure peak area or peak height.

CALCULATIONS:

16. Determine the quantities in ng of Kepone (corrected for R) on the filter (W_f), in the impinger (W_i), in the average filter blank (B_f), and in the average impinger blank (B_i) from calibration graphs based on standards analyzed with the samples.
17. Calculate the concentration, C (µg/m^3), of Kepone in the air volume sampled, V (L):

$$C = \frac{W_f + W_i - B_f - B_i}{V}, \ \mu g/m^3.$$

EVALUATION OF METHOD:

This method was tested with fortified filters, fortified NaOH solutions and controlled atmospheres near 25 °C. The overall precision including pump error was 0.234 [1]. The relative standard deviation ($\bar{S}_r$) for filters was 0.228 (10 samples, pooled) for 376-L samples at 2.5 and 2.6 µg/m^3. Quantities of Kepone found in impingers were less than 50 ng. The quantity of Kepone found in each impinger corresponded to about 0.1 ng or less for each liter of air in a sample. Collection efficiencies of impingers were roughly 50%. Average recovery of 360 ng of Kepone from filters was 0.89; $\bar{S}_r$ was 0.014 for six samples. Kepone was stable on filters at room temperature for 12 days. Average recovery of 45 ng Kepone from 45-mL aliquots of 0.1 M NaOH solution was 1.01; $\bar{S}_r$ was 0.060 for 8 samples. Average recovery of 79 ng Kepone from 10-mL aliquots of 0.1 M NaOH solution was 1.13 after 14 days of storage at room temperature. Personal and area samples (45 to 84 L) were collected by this method at a Mirex formulation plant. Mirex was found on filters in quantities of 240 to 1240 µg and in impingers in quantities of 0.3 to 16 µg. Although no Kepone was detected on filters, Kepone in quantities of 0.1 to 2 µg was found in impingers. Most or all of the Kepone in each impinger was produced by chemical conversion of Mirex. The accuracy of this method is greater than ± 45%, greatly exceeding the NIOSH criterion of ± 25%.

REFERENCES:

[1] Tucker, S.P., "Backup Data Report on Kepone," Internal NIOSH MRB Report (unpublished), May 9, 1977.
[2] NIOSH Manual of Analytical Methods, 2nd. ed., V. 1, P&CAM 225, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).
[3] NIOSH Recommended Standard for Occupational Exposure to Kepone, U.S. Department of Health, Education, and Welfare, (NIOSH), unnumbered publication (January, 1976).

METHOD REVISED BY:

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