C_6Cl_5OH MW: 266.35 CAS: 87-86-5 RTECS: SM6300000

METHOD: 5512, 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); Group I Pesticide
ACGIH: 0.5 mg/m^3 (skin)

PROPERTIES: solid; MP 190 °C; d 1.978 g/mL @ 22 °C; VP 16 Pa (0.12 mm Hg; 1700 mg/m^3) @ 100 °C

SYNONYMS: penta; PCP

SAMPLING

SAMPLER: FILTER + BUBBLER (mixed cellulose ester membrane with stainless steel backup screen/ethylene glycol)

FLOW RATE: 0.5 to 1.0 L/min

VOL-MIN: 48 L @ 0.5 mg/m^3
-MAX: 480 L

SHIPMENT: place filter in bubbler containing 15 mL ethylene glycol after sampling

SAMPLE STABILITY: at least 8 days @ 25 °C

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 0.265 to 1.130 mg/m^3 [1] (180-L samples)

BIAS: 3.0%

OVERALL PRECISION ($\hat{S}_{rT}$): 0.072

ACCURACY: ± 15% (12-28%)

MEASUREMENT

TECHNIQUE: HPLC, UV DETECTION

ANALYTE: pentachlorophenol

EXTRACTION: 10 mL methanol

INJECTION VOLUME: 20 µL

MOBILE PHASE: 60% methanol/40% water, 1.5 mL/min

COLUMN: µ-Bondapak C_{18}, 10-µm particle size, 30 cm x 3.9-mm ID

DETECTOR: UV @ 254 nm

CALIBRATION: standard solutions of pentachlorophenol in ethylene glycol and methanol

RANGE: 24 to 270 µg per sample [1]

ESTIMATED LOD: 8 µg per sample [2]

PRECISION ($\hat{S}_r$): 0.051 @ 45 to 180 µg per sample [1]

APPLICABILITY: The working range is 0.13 to 11 mg/m^3 for a 180-L air sample. This method is also applicable to STEL measurements using a 15-L sample. The method has been used to sample for pentachlorophenol in the presence of 2,3,4,6-tetrachlorophenol at a lumber yard [3].

INTERFERENCES: None identified.

OTHER METHODS: This revises Method S297 [2]. An independent analytical method provided by Vulcan Materials Co. [4] using a sampling train consisting of Zefluor filter and silica gel tube and HPLC analysis was used by a NIOSH contractor [5] for analyzing samples containing pentachlorophenol.

REAGENTS:

1. Pentachlorophenol *, ACS reagent grade.
2. Ethylene glycol, ACS reagent grade.*
3. Methanol, distilled in glass.
4. Isopropanol, distilled in glass.
5. Water, deionized and distilled.
6. Calibration stock solution, 5 mg/mL. Dissolve 50 mg pentachlorophenol in 10 mL isopropanol.

* See Special Precautions

EQUIPMENT:

1. Sampler: 37-mm cellulose ester membrane filter (0.8-µm pore size) supported by stainless steel screen in three-piece filter holder followed by a 25-mL bubbler with 15 mL ethylene glycol.
2. Personal sampling pump, 0.5 to 1 mL/min, with flexible polyethylene or PTFE tubing.
3. PTFE plugs and/or tubing.
4. Vials, glass, 20-mL with PTFE-lined caps.
5. Liquid chromatograph with a UV detector, recorder, integrator and column (page 5512-1).
6. Tweezers.
7. Syringes, 50- and 100-µL.
8. Volumetric flasks, 25-mL.
9. Pipets, 10- and 15-mL glass, delivery, with pipet bulb.
10. Graduated cylinders, glass, 25-mL.

SPECIAL PRECAUTIONS: Pentachlorophenol can irritate the eyes, can be absorbed through the skin, and can cause liver damage [6]. Ethylene glycol is very toxic. It may be harmful or fatal if absorbed through the skin or swallowed, may cause kidney damage, and is a suspect reproductive hazard [7].

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Transfer 15 mL ethylene glycol to a bubbler.
3. Attach outlet of filter holder to inlet arm of bubbler. Connect outlet arm of bubbler to a second empty bubbler and then to the sampling pump.
4. Sample 48 to 480 L (15 L for STEL) of air at an accurately known rate between 0.5 and 1.0 L/min.
5. Transfer filter carefully using tweezers to the bubbler. Seal bubbler for shipment in a suitable container in order to prevent damage during transit. Seal the inlet and outlet of the bubbler stem by connecting a piece of PTFE tubing between them or by inserting PTFE plugs in the inlet and outlet.
6. Collect a bulk sample (ca. 1 g) in a glass vial and ship it separately.

SAMPLE PREPARATION:

7. Transfer the liquid from the bubbler, quantitatively, to a graduated cylinder.
8. Bring volume to 15 mL with ethylene glycol. (If volume is greater than 15 mL, as would be the case when H_2O is scrubbed from humid air, record the volume and make an appropriate correction in the final calculations).
9. Just before analysis, add 10 mL methanol and mix gently but thoroughly.

CALIBRATION AND QUALITY CONTROL:

10. Calibrate daily with at least six working standards in the range 8 to 270 µg/25 mL.
    a. Add appropriate aliquots of calibration stock solution to a 60/40 (v/v) mixture of ethylene glycol and methanol.
    b. Analyze working standards together with samples and blanks (steps 13 through 15).

c. Prepare a calibration graph of peak area vs. amount (µg) of pentachlorophenol per 25 mL of sample.

11. Determine recovery for each lot of filters used for sampling in the concentration range of interest. Prepare four filters at each of five levels plus three media blanks.
    a. Spike aliquot of calibration solution onto each filter.
    b. After air-drying, extract filters in 15 mL ethylene glycol.
    c. Just before analysis, add 10 mL methanol and analyze (steps 13 through 15).
    d. Prepare graph of recovery vs. µg pentachlorophenol.
12. Check recovery at two levels for each sample set. Repeat recovery graph determination if checks do not agree to within 5% of recovery graph.

MEASUREMENT:

13. Set liquid chromatograph according to manufacturer's recommendations and to conditions given on page 5512-1.
14. Inject 20-µL sample aliquot.
    NOTE: If sample peak area exceeds the linear calibration range, dilute, and apply appropriate dilution factor in calculations.
15. Measure peak area.

CALCULATIONS:

16. Determine mass, µg (corrected for recovery), of pentachlorophenol (W) found in the sample and the average media blank (B).
17. Calculate concentration of pentachlorophenol in the air volume sampled, V (L):

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

EVALUATION OF METHOD:

This method was validated over the range 0.265 to 1.31 mg/m ^{3} at 24 °C and pressure of 761 mm Hg using 180-L samples [1,2]. Overall sampling and measurement precision, $\hat{S}_{rT}$, was 0.072, with average recovery of 105%, representing a non-significant bias. The concentration of pentachlorophenol was independently verified by direct UV analysis of sample solutions. Recovery of pentachlorophenol from filters was 101% in the range 45 to 180 µg per sample. Sample stability during storage was evaluated at 100 µg pentachlorophenol per sample. Samples showed 95.3% recovery after eight days of storage at ambient conditions compared to one-day old samples.

REFERENCES:

[1] Backup Data Report for Pentachlorophenol, prepared under NIOSH Contract 210-76-0123 (1977).
[2] NIOSH Manual of Analytical Methods, 2nd. ed., V. 4, S297, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).
[3] Analysis of NIOSH Samples for Pentachlorophenol and Tetrachlorophenol, NIOSH/MRSB Sequence #4492, Utah Biomedical Research Laboratory, Salt Lake City, UT (unpublished, 1984).
[4] Vulcan Materials Co. Analytical Backup Report #1. Determination of Pentachlorophenol in Air, Birmingham, AL 35255 (1982).

[5] Analysis of NIOSH Samples for Pentachlorophenol, NIOSH/MRSB Sequence #4065, Southern Research Institute, Birmingham, AL 35255 (1984).
[6] NIOSH/OSHA Occupational Health Guidelines for Occupational Hazards, U.S. Department of Health and Human Services, Publ. (NIOSH) 81-123 (1981), available as GPO Stock #017-033-00337-8 from Superintendent of Documents, Washington, DC 20402.
[7] CCINFO Database, Release 93-3, Record No. 41. Canadian Centre for Occupational Health and Safety, Hamilton, Ontario, Canada (1993).

METHOD REVISED BY:

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