MEVINPHOS

$(CH_3O)_2PO_2C(CH_3)=CHC(O)OCH_3$; MW: 224.27 CAS: - - RTECS:
$C_7H_{13}O_6P$

METHOD: 2503, Issue 2 **CLASS B** **Issue 1: 15 February 1984**
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OSHA : 0.01 ppm (skin)
NIOSH: Group I Pesticide [1]
ACGIH: 0.1 mg/m^3 (skin); 0.3 mg/m^3 STEL
(1 ppm = 9.16 mg/m^3 @ NTP)

PROPERTIES: liquid; d 1.25 g/mL @ 20 °C; BP 325 °C; MP 20.6 °C; VP 0.4 Pa (0.003 mm Hg; 4 ppm) 20 °C

SYNONYMS: dimethyl 2-methoxycarbonyl-1-methylethenyl phosphate; Phosdrin; CAS #7786-34-7.

SAMPLING

SAMPLER: SOLID SORBENT TUBE
(extracted Chromosorb 102, 100 mg/50 mg)

FLOW RATE: 0.2 to 1 L/min

VOL-MIN: 15 L
-MAX: 240 L

SHIPMENT: routine

SAMPLE STABILITY: at least 7 days @ 25 °C [2]

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 0.027 to 0.145 mg/m^3 [2]
(260-L samples)

BIAS: not significant [2]

OVERALL PRECISION (s_r): 0.069 [2]

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FPD

ANALYTE: phosphorus

DESORPTION: 1 mL toluene; stand 30 min

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTION: 190 °C
-DETECTOR: 215 °C
-COLUMN: 170 °C

CARRIER GAS: N_2 or He, 28 mL/min

COLUMN: 2 m x 2 mm glass; Super-Pak 20M or equivalent

CALIBRATION: Mevinphos in toluene

RANGE: 5 to 55 µg per sample [2]

ESTIMATED LOD: 0.2 µg per sample [2]

PRECISION (s_r): 0.035 [2]

APPLICABILITY: The working range is 0.003 to 0.3 ppm (0.025 to 0.28 mg/m^3) for a 200-L air sample. The method can measure a STEL concentration of < 0.4 mg/m^3.

INTERFERENCES: None identified.

OTHER METHODS: This is Methods S296 [3] in a revised format.

REAGENTS:

1. Mevinphos, analytical grade.*
2. Toluene, reagent grade.
3. Calibration stock solution, 5 mg/mL. Dilute 50 mg of Mevinphos to 10 mL with toluene. Prepare in duplicate.
4. Helium, purified.
5. Hydrogen, prepurified.
6. Air, filtered.
7. Extracted resin. Chromosorb 102 20/40 mesh (Johns Manville Corp. or equivalent) extracted in Soxhlet apparatus with 1:1 methanol:acetone solution for 2 hrs and dried at 115 °C under vacuum for 1 hr.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 10-cm long, 8-mm OD, 6-mm ID, flame sealed ends containing two sections of extracted resin (front = 100 mg; back = 50 mg) separated and retained by silylated glass wool plugs. Pressure drop across the tube at 1 L/min airflow must be less than 3.4 kPa. Tubes are commercially available.
2. Personal sampling pump, 0.2 to 1 L/min, with flexible connecting tubing.
3. Gas chromatograph, flame photometric detector with phosphorus filter, integrator and column (page 2503-1).
4. Vials, 2-mL, PTFE-lined caps.
5. Syringes, 1- to 100-µL.
6. Volumetric flasks, 10-mL.
7. Pipet, 1-mL, with pipet bulb.

SPECIAL PRECAUTIONS: Mevinphos is a cholinesterase inhibitor and can be absorbed through the skin. Wear protective gloves and suitable protective clothing when handling the pure material [1].

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the sampler immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.2 and 1 L/min for a total sample size of 15 to 240 L.
4. Cap the samplers with plastic (not rubber) caps and pack securely for shipment.

SAMPLE PREPARATION:

5. Place the front and back sorbent sections of the sampler tube in separate vials. Discard the glass wool and foam plugs.
6. Add 1.0 mL toluene to each vial. Attach cap to each vial.
7. Allow to stand 30 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least five working standards over the range 0.2 to 55 µg Mevinphos per sample.
   a. Add known amounts of calibration stock solution to toluene in 10-mL volumetric flasks and dilute to the mark.
   b. Analyze together with samples and blanks (steps 11 and 12).
   c. Prepare calibration graph (peak area vs. µg Mevinphos).

9. Determine desorption efficiency (DE) at least once for each batch of Chromosorb 102 used for sampling in the calibration range (step 8). Prepare three tubes at each of five levels plus three media blanks.
   a. Remove and discard back sorbent section of a media blank sampler.
   b. Inject a known amount of calibration stock solution directly onto front sorbent section with a microliter syringe.
   c. Cap the tube. Allow to stand overnight.
   d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
   e. Prepare a graph of DE vs. µg Mevinphos recovered.
10. Analyze three quality control blind spikes and three analyst spikes to insure that the calibration graph and DE graph are in control.

MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 2503-1. Inject sample aliquot manually using solvent flush technique or with autosampler.
    NOTE: If peak area is above the linear range of the working standards, dilute with toluene, reanalyze and apply the appropriate dilution factor in calculations.
12. Measure peak area.

CALCULATIONS:

13. Determine the mass, µg (corrected for DE) of Mevinphos found in the sample front (W_f) and back (W_b) sorbent sections, and in the average media blank front (B_f) and back (B_b) sorbent sections.
    NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.
14. Calculate concentration, C, of Mevinphos in the air volume sampled, V (L):

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

EVALUATION OF METHOD:

Method S296 [3] was issued on July 6, 1979, and validated over the range 0.027 to 0.145 mg/m^3 at 25 °C using a 240-L sample [2,4]. Overall precision, s_r was 0.0694 with an average recovery of 103.9%, representing a non-significant bias. The concentration of Mevinphos was independently verified by sampling the generator with toluene-filled bubblers and measurement by GC/FPD. Desorption efficiency was 1.04 in the range 7.0 µg to 40.0 µg per sample. After sampling an atmosphere containing 0.195 mg/m^3 at 1.0 L/min at 80% RH for 376 min, only 0.6% breakthrough was reported [2].

REFERENCES:

[1] Criteria for a Recommended Standard...Occupational Exposure During the Manufacture and Formulation of Pesticides, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-174 (1978).
[2] Backup Data Report No. S296, Phosdrin, prepared under NIOSH Contract No. 210-76-0123 (July, 1979), available as Order No. PB 81-228983 from NTIS, Springfield, VA 22161.
[3] NIOSH Manual of Analytical Methods, 2nd ed., V. 6, S296, U.S. Department of Health and Human Services, Publ. (NIOSH) 80-125 (1980).

[4] 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).

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