C_nH_{2n+2} where n ≥ 16 MW: not pertinent CAS: 8012-95-1 RTECS: PY8030000

METHOD: 5026, Issue 2 **EVALUATION: FULL** **Issue 1: 15 August 1987**
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OSHA : 5 mg/m^3
NIOSH: 5 mg/m^3; STEL 10 mg/m^3
ACGIH: 5 mg/m^3 (as sampled by a method which does not collect vapor)

PROPERTIES: liquid; d 0.8 to 0.9 g/mL @ 20 °C; BP 360 °C; vapor pressure negligible

SYNONYMS: airborne mist of white mineral oil or the following water-insoluble petroleum-based cutting oils: cable oil; cutting oil; drawing oil; engine oil; heat-treating oils; hydraulic oils; machine oil; transformer oil.

SAMPLING

SAMPLER: MEMBRANE FILTER (37-mm diameter, 0.8- or 5-µm pore size, PVC or MCE)

FLOW RATE: 1 to 3 L/min

VOL-MIN: 20 L @ 5 mg/m^3
-MAX: 500 L

SHIPMENT: routine

SAMPLE STABILITY: stable

BLANKS: 2 to 10 field blanks per set

BULK SAMPLE: required for quantitative data

ACCURACY

RANGE STUDIED: 2.5 to 11.7 mg/m^3 [1] (100-L samples)

BIAS: - 0.84% [1,2]

OVERALL PRECISION ($\hat{S}_{rT}$): 0.065 [1]

ACCURACY: ± 11.8%

MEASUREMENT

TECHNIQUE: INFRARED SPECTROPHOTOMETRY

ANALYTE: mineral oil

EXTRACTION: 10 mL $C_2Cl_3F_3$ (Freon 113)

IR SCAN: 3200 to 2700 cm^{-1} vs. blank $C_2Cl_3F_3$

CALIBRATION: standard solutions of mineral oil in $C_2Cl_3F_3$

RANGE: 0.1 to 2.5 mg per sample

ESTIMATED LOD: 0.05 mg per sample [3]

PRECISION ($\hat{S}_r$): 0.05 [3]

APPLICABILITY: The working range is 1 to 20 mg/m^3 for a 100-L air sample. This method is applicable to all trichlorotrifluoroethane-soluble mineral oil mists, but not to (nor does OSHA's standard cover) semi-synthetic or synthe tic cutting fluids.

INTERFERENCES: Any aerosol (e.g., tobacco smoke) which absorbs infrared radiation near 2950 cm-1 interferes.

OTHER METHODS: This revises P&CAM 283 [3]. P&CAM 159 [4] and S272 [5] use similar samplers with measurement by fluorescence spectrophotometry. These methods have not been revised because of limited applicability (i.e., not all mine ral oils contain fluorescent components and other fluorescent compounds interfere). Infrared analysis overcomes both of these lim itations.

REAGENTS:

1. Trichlorotrifluoroethane ($C_2Cl_3F_3$).
2. Stock mineral oil standard, 20 mg/mL. Weigh 1.0 g of the bulk mineral oil sample into a 50-mL volumetric flask. Dilute to volume with $C_2Cl_3F_3$. Prepare in duplicate.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: membrane filter, PVC or MCE, 37-mm, 0.8- or 5-µm pore size; two-piece filter cassette.
   NOTE 1: High concentrations of oil mist may plug membrane filters. Glass fiber filters have a higher capacity for oil mist than membrane filters.
   NOTE 2: Handle filters carefully with tweezers to avoid contamination by skin oil.
2. Personal sampling pump, 1 to 3 L/min, with flexible connecting tubing.
3. Infrared spectrophotometer, double beam, dispersive, with scanning capability in the 3200-2700 cm^{-1} region, and two 10-mm spectrophotometer cells, infrared quartz with PTFE stoppers mounted in demountable cell holders.
   NOTE: Standard glass cells may be used if infrared quartz cells are not available.
4. Vials, scintillation, 20-mL, with foil-lined or PTFE-lined caps.*
5. Volumetric flasks, 10-, 25-, and 50-mL.*
6. Volumetric pipet or reagent dispenser, 10-mL.*
7. Pipets, 2- to 250-µL.
8. Tweezers.

* Rinse glassware with $C_2Cl_3F_3$. Air dry.

SPECIAL PRECAUTIONS: None.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate in the range 1 to 3 L/min for a total sample size of 20 to 500 L.
   NOTE: High concentrations of oil mist may plug membrane filters creating unacceptably high pressure drops. If this occurs, terminate sampling.
3. Collect 5 to 10 mL of unused, undiluted mineral oil in a vial. Submit with samples for standard preparation.

SAMPLE PREPARATION:

4. Using tweezers, transfer each sample or blank filter to a vial. Add 10.0 mL $C_2Cl_3F_3$. Cap and shake vigorously.

CALIBRATION AND QUALITY CONTROL:

5. Calibrate daily with at least six working standards.
   a. Add known amounts of stock mineral oil standard to $C_2Cl_3F_3$ in 10-mL volumetric flasks and dilute to the mark to obtain mineral oil concentrations in the range 5 to 250 µg/mL.
   b. Analyze with samples and blanks (step 8).
   c. Prepare calibration graph (peak absorbance vs. mg mineral oil).
6. Determine recovery (R) at least once for each lot of filters used for sampling in the range of interest. Prepare three filters at each of five levels plus three media blanks.

a. Deposit a known amount of stock mineral oil standard onto the filter. Allow solvent to evaporate.
b. Store samples overnight in filter cassettes.
c. Prepare and analyze with working standards.
d. Prepare a graph of R vs. µg mineral oil recovered.

7. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and R graph are in control.

MEASUREMENT:

8. Scan each standard solution and each blank or sample filter extract from 3200 to 2700 cm^{-1} in absorbance mode vs. $C_2Cl_3F_3$ in reference beam. Record absorbance at wavelength of largest absorbance near 2940 cm^{-1} (± 11.8%).

CALCULATIONS:

9. Determine the mass, µg (corrected for R), of mineral oil found in the sample (W) and in the average media blank (B) from the calibration graph.
10. Calculate concentration, C, of mineral oil in the air volume sampled, V (L):

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

EVALUATION OF METHOD:

The sampling portion of this method was evaluated over the range 2.5 to 11.7 mg/m^3 at 22 °C and 755 mm Hg using 100-L air samples of Gulf machine cutting oil with measurement by fluorescence spectrophotometry. Mixed cellulose ester filters, 0.8-µm pore size, were used for sampling [1,5]. The overall precision was 0.065 with an average recovery of 98%. The infrared measurement method was subsequently evaluated by NIOSH [2,3]. Precision and accuracy of the infrared and fluorescence spectrophotometric techniques are similar.

REFERENCES:

[1] Documentation of the NIOSH Validation Tests, S272, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977), available as PB 274-248 from NTIS, Springfield, VA 22161.
[2] Bolyard, M. L. Infrared Quantitation of Mineral Oil Mist in Personal Air Samples, AIH Conference, Houston, TX (1980).
[3] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 4, P&CAM 283, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).
[4] Ibid., Vol. 1, P&CAM 159, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).
[5] Ibid., Vol. 3, S272, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).

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