

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

METHOD: 5026, Issue 2

EVALUATION: FULL

Issue 1: 15 August 1987

Issue 2: Rev.: 15 May 1996

OSHA: 5 mg/m³
NIOSH: 5 mg/m³; STEL 10 mg/m³
ACGIH: 5 mg/m³ (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		MEASUREMENT	
SAMPLER:	MEMBRANE FILTER (37-mm diameter, 0.8- μ m MCE, 5- μ m PVC, 2- μ m PTFE, or glass fiber)	TECHNIQUE:	INFRARED SPECTROPHOTOMETRY
FLOW RATE:	1 to 3 L/min	ANALYTE:	mineral oil
VOL-MIN:	20 L @ 5 mg/m ³	EXTRACTION:	10 mL CCl ₄
-MAX:	500 L	IR SCAN:	3200 to 2700 cm ⁻¹ vs. blank CCl ₄
SHIPMENT:	routine	CALIBRATION:	standard solutions of mineral oil in CCl ₄
SAMPLE STABILITY:	stable	RANGE:	0.1 to 2.5 mg per sample
BLANKS:	2 to 10 field blanks per set	ESTIMATED LOD:	0.05 mg per sample [3]
		PRECISION (\hat{S}_r):	0.05 [3]
ACCURACY			
RANGE STUDIED:	2.5 to 11.7 mg/ m ³ [1] (100-L samples)		
BIAS:	- 0.84% [1,2]		
OVERALL PRECISION ($\hat{S}_{r,T}$):	0.065 [1]		
ACCURACY:	\pm 11.8 %		

APPLICABILITY: The working range is 1 to 20 mg/m³ for 100-L air sample. This method is applicable to all trichlorofluoroethane-soluble mineral oil mists, but not to (nor does OSHA's standard cover) semi-synthetic or synthetic 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 mineral oils contain fluorescent components and other fluorescent compounds interfere). Infrared analysis overcomes both of these limitations.

REAGENTS:

1. Carbon tetrachloride* (CCl₄), reagent grade.
2. Stock mineral oil standards, 20 mg/mL.
Weigh 1.0 g of the bulk mineral oil sample into a 50-mL volumetric flask. Dilute to volume with CCl₄. Prepare in duplicate.

* See SPECIAL PRECAUTIONS

EQUIPMENT:

1. Sampler: membrane filter, 37-mm, 0.8- μ m MCE, 5- μ m PVC, 2- μ m PTFE, or glass fiber filter, with cellulose backup pad in two-piece filter cassette.
NOTE 1: High concentrations of oil mist may plug membrane filters. Glass fibers filters have a higher capacity for oil mist and 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 maybe 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- μ L.
8. Tweezers.

* Rinse glassware with CCl₄. Air dry.

SPECIAL PRECAUTIONS: Carbon tetrachloride is a suspected human carcinogen. Handle in a fume hood.

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 CCl₄. 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 CCl₄ 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. µg 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. CCl₄ 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)

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

EVALUATION OF METHOD:

The sampling portion of this method was evaluated over the range 2.5 to 11.7 mg/m³ 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.

Issue 1 of this method (dated 8/15/1987) used Freon 113 as the solvent. However, since Freons are known atmospheric ozone depleters, carbon tetrachloride, the original solvent used in P&CAM 283, is again specified, although it is a suspected human carcinogen.

PTFE filters (e.g. Gelman Zefluor) also may be used to sample oil mists. They have lower blanks than PVC, since PTFE has fewer extractables with CCl_4 than other membrane filters.

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).

METHOD REVISED BY:

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