TRICHLOROETHYLENE 1022

$CCl_2{=}CHCl$ MW: 131.39 CAS: 79-01-6 RTECS: KX4550000

METHOD: 1022, Issue 2 **EVALUATION: PARTIAL**

Issue 1: 15 August 1987
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OSHA : 100 ppm; C 200 ppm; P 300 ppm
NIOSH: 25 ppm; C 2 ppm/1 h (waste anesthetic); suspect carcinogen; Group 1 Pesticide
ACGIH: 50 ppm; STEL 200 ppm; suspect carcinogen
(1 ppm = 5.37 mg/m^3 @ NTP)

PROPERTIES: liquid; d 1.46 g/mL @ 20 °C; BP 87 °C; MP -86 °C; VP 9.9 kPa (74 mm Hg; 9.8% v/v) @ 25 °C; explosive range 11 to 41% v/v in air

SYNONYMS: trichloroethene; ethylene trichloride; triclene

SAMPLING

SAMPLER: SOLID SORBENT TUBE
(coconut shell charcoal, 100 mg/50 mg)

FLOW RATE: 0.01 to 0.2 L/min

VOL-MIN: 1 L @ 100 ppm
-MAX: 30 L

SHIPMENT: routine

SAMPLE STABILITY: not determined

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 477 to 2025 mg/m^3
(3.4-L samples) [1]

BIAS: - 7.19%

OVERALL PRECISION ($\hat{S}_{rT}$): 0.082 [1]

ACCURACY: ± 19.78%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: trichloroethylene

DESORPTION: 1 mL CS_2; stand 30 min

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTION: 225 °C
-DETECTOR: 250 °C
-COLUMN: 70 °C

CARRIER GAS: N_2, 30 mL/min

COLUMN: 3 m x 3-mm OD stainless steel, packed with 10% OV-101 on 100/200 mesh Chromosorb WHP

CALIBRATION: standard solutions of trichloroethylene in CS_2

RANGE: 0.5 to 10 mg per sample

ESTIMATED LOD: 0.01 mg per sample [2]

PRECISION ($\bar{S}_r$): 0.038 @ 1.6 to 6.4 mg per sample [1]

APPLICABILITY: The working range is 27 to 875 ppm (150 to 4700 mg/m ^{3}) for a 3.4-L air sample. The method is applicable to STEL determinations. The method was used for samples containing 0.5 to 5 mg trichloroethylene from a tool-degreasing operation [2].

INTERFERENCES: None studied. Alternate columns which have been used are stainless steel, 6 m x 3 mm OD, packed with 10% SP-1000 on 80/100 mesh Supelcoport [2] and fused silica capillary, 60 m x 0.32 mm, coated with 0.25 µm OV-351 [3].

OTHER METHODS: This combines and revises methods S336 [4] and P&CAM 127 [5]. The criteria document method is similar [6]. NIOSH Method 3701 uses a portable gas chromatograph for field readout.

REAGENTS:

1. Carbon disulfide (CS_2), chromatographic quality.*
2. Trichloroethylene (TCE), reagent grade.*
3. Nitrogen, purified.
4. Hydrogen, prepurified.
5. Air, filtered, compressed.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends with plastic caps, containing two sections of 20/40 mesh activated (600 °C) coconut shell charcoal (front = 100 mg; back = 50 mg) separated by a 2-mm urethane foam plug. A silylated glass wool plug precedes the front section and a 3-mm urethane foam plug follows the back section. 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, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. Gas chromatograph, flame ionization detector, integrator, and column (see page 1022-1).
4. Vials, 2-mL, PTFE-lined septum caps.
5. Syringes, 10-µL, readable to 0.1 µL.
6. Volumetric flasks, 10-mL.
7. Pipet, TD, 1-mL.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and a serious fire and explosion hazard (flash point = -30 °C). Trichloroethylene is a suspect carcinogen and a narcotic [6,7,8]. Work with these substances only in a hood.

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 0.01 and 0.2 L/min for a total sample size of 1 to 30 L.
4. Cap the samplers. 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 CS_2 to each vial. Cap each vial.
   NOTE: A suitable internal standard, such as ethylbenzene [1], undecane [2], or octane [3] at 0.1% (v/v) may be added at this step.
7. Allow to stand 30 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards.
   a. Add known amounts of TCE to CS_2 in 10-mL volumetric flasks and dilute to the mark. Use serial dilutions as needed to obtain TCE concentrations in the range 0.01 to 10 mg/mL.
   b. Analyze with samples and blanks (steps 11 and 12).
   c. Prepare calibration graph (peak area vs. mg TCE).

9. Determine desorption efficiency (DE) at least once for each lot of sorbent used for sampling in the range of interest. Prepare three tubes at each of five concentrations plus three media blanks.
   a. Remove and discard back sorbent section of a media blank sampler.
   b. Inject a known amount (2 to 20 µL) of TCE, or a standard solution thereof in CS_2, 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 with working standards (steps 11 and 12).
   e. Prepare a graph of DE vs. mg TCE recovered.
10. Analyze three quality control blind spikes and three analyst spikes to ensure 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 1022-1. 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 an aliquot of the desorbed liquid with CS_2, reanalyze, and apply the appropriate dilution factor in calculations.
12. Measure peak area.

CALCULATIONS:

13. Determine the mass, mg (corrected for DE) of TCE 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 TCE in the air volume sampled, V (L):

$$C = \frac{(W_f + W_b - B_f - B_b) \cdot 10^3}{V}, \ \text{mg/m}^3.$$

EVALUATION OF METHOD:

Method S336 was issued on June 6, 1975 [4], and validated with generated atmospheres using a calibrated syringe drive [1]. Average recoveries were 92 to 94% (16 samples) in the range 477 to 2025 mg/m^3 for 3.4-L samples. Breakthrough volume of 18.5 L (effluent = 5% of test concentration) occurred after sampling for 99 min at 0.187 L/min from an atmosphere containing 2266 mg/m^3 trichloroethylene in dry air. Desorption efficiency for SKC Lot 105 activated coconut charcoal in the range 1.6 to 6.4 mg per sample averaged 96.4% with $\bar{S}_r = 0.7\%$ (18 samples). n-Octane was used as an internal standard in the chromatographic measurements. The semi-quartile ranges of desorption efficiencies in two rounds of the Proficiency Analytical Testing (PAT) program were 0.97 to 1.0 for charcoal tubes spiked with 0.6 to 1.1 mg trichloroethylene [9].

REFERENCES:

[1] Documentation of the NIOSH Validation Tests, S336, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977), available at Stock No. PB 274-248 from NTIS, Springfield, VA 22161.

[2] UBTL Report for NIOSH Sequence #4266-R (NIOSH, unpublished, March 26, 1984).

[3] UBTL Report for NIOSH Sequence #4266-N (NIOSH, unpublished, March 14, 1984).

[4] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, S336, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
[5] Ibid., Vol. 1., P&CAM 127, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).
[6] Criteria for a Recommended Standard...Occupational Exposure to Trichloroethylene, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 73-11025 (1973).
[7] NIOSH Current Intelligence Bulletin 2, Trichloroethylene (TCE), NIOSH (June 6, 1975), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-127 (1978).
[8] Special Occupational Hazard Review with Control Recommendations -- Trichloroethylene, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-130 (1978).
[9] Groff, J. Personal communication, NIOSH (July, 1985).

METHOD REVISED BY:

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