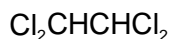


1,1,2,2-TETRACHLOROETHANE

2562



MW: 167.85

CAS: 79-34-5

RTECS: KI8575000

METHOD: 2562, Issue 1

EVALUATION: PARTIAL

Issue 1: 15 March 2003

OSHA: 5 ppm (skin)
NIOSH: 1 ppm; carcinogen; Group I Pesticide
ACGIH: 1 ppm

(1 ppm = 6.86 mg/m³ @ NTP)

PROPERTIES: liquid; d 1.586 g/mL @ 25 °C; BP 146.5 °C; MP -44 °C; VP 0.8 kPa (6 mm Hg; 0.8% v/v) @ 25 °C; nonflammable

SYNONYMS: acetylene tetrachloride; bonoform; 1,1-dichloro-2,2-dichloroethane

APPLICABILITY: The working range is 0.031 to 4.17 ppm (0.21 to 28.6 mg/m³) for a 10-L air sample [1].

INTERFERENCES: Any compound with a similar retention time under these analytical conditions is considered to be an interference.

OTHER METHODS: This method updates NIOSH Method 1019 [2] through the use of a capillary column, incorporation of Anasorb CMS sorbent tubes, and lowered sampling and analytical range with accompanying lower LOD/LOQ values [1].

REAGENTS:

1. Carbon disulfide (CS₂), chromatographic quality.*
2. 1,1,2,2-Tetrachloroethane, reagent grade.*
3. Desorption efficiency (DE) stock solution, 12.7 mg/mL. Dilute 80 µL of 1,1,2,2-tetrachloroethane to 10 mL with CS₂.
4. Helium, purified
5. Hydrogen, prepurified
6. Air, filtered, compressed

* See SPECIAL PRECAUTIONS

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, glass-sealed ends with plastic caps, containing two sections of 100/50 Anasorb CMS (front = 100 mg; back = 50 mg) separated by a 2-mm glass wool plug. Another glass wool plug precedes the front section and a 3-mm urethane foam plug follows the back section. (SKC 226-121, or equivalent).
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. Gas chromatograph, flame ionization detector, integrator, and column.
4. Vials, 2-mL, PTFE-lined caps.
5. Syringes, 10-µL, readable to 0.1 µL, 25-µL, readable to 0.5 µL, and 1000-µL, readable to 1 µL.
6. Volumetric flasks, 10-mL.
7. Pipet, TD, 1-mL.

SPECIAL PRECAUTIONS: 1,1,2,2-Tetrachloroethane is a powerful narcotic, liver poison, and suspected carcinogen, and can be absorbed through the skin [3, 4]. Carbon disulfide is toxic and a serious fire and explosion hazard (flash point = -30 °C). Work with these substances only in a hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break 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 3 to 30 L.
4. Cap the samplers. Pack securely for shipment.

SAMPLE PREPARATION:

5. Place front and back sorbent sections of the sampler tube in separate vials. Discard glass wool plugs.
6. Add 1.0 mL CS₂ to each vial. Cap each vial.
7. Allow to stand 30 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards from below the LOD to 10x the LOQ.
 - a. Add a specific amount of 1,1,2,2-tetrachloroethane to CS₂ in 10-mL volumetric flasks and dilute to the mark. Use serial dilutions as needed to obtain 1,1,2,2-tetrachloroethane concentrations in the range needed. Additional standards may be added to extend the calibration curve, if necessary.
 - b. Analyze with calibration standards, samples, and blanks (steps 11 and 12).
 - c. Prepare calibration graph (peak area vs. µg 1,1,2,2-tetrachloroethane).
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 25 µL) of DE stock solution, or a serial dilution thereof, directly onto the front sorbent section with a microliter syringe.

- c. Cap the tube. Allow to stand for 24 hours.
 - d. Desorb (steps 5 through 7) and analyze with working standards (steps 11 and 12).
 - e. Prepare a graph of DE vs. μg 1,1,2,2-tetrachloroethane recovered.
10. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and DE graph are within the limits set by the quality assurance policy and are consistent with the results given in the section entitled "Evaluation of Method".

MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 2562-1. Inject sample aliquot manually using solvent flush technique or with an 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, μg (corrected for DE) of 1,1,2,2-tetrachloroethane 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 1,1,2,2-tetrachloroethane in the air volume sampled, V (L):

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

NOTE: $\mu\text{g}/\text{L} \equiv \text{mg}/\text{m}^3$

EVALUATION OF METHOD:

NMAM 1019, Issue 1 and Issue 2:

The 4th edition Method 1019 is 2nd edition NMAM Method S124 in the 4th edition format. Method S124 was issued on May 9, 1975 [5], and validated with generated atmospheres which were calibrated by gas chromatography [6]. Average recovery was 106% at 5 ppm. Breakthrough did not occur after sampling 4 h at 0.185 L/min from an atmosphere containing 101 mg/m^3 in dry air. Desorption efficiency for SKC Lot 104 petroleum-based charcoal was 0.83, 0.87, and 0.88 at 160, 320, and 640 μg 1,1,2,2-tetrachloroethane per sample, respectively. Lower recoveries were observed for coconut shell charcoal (SKC Lot 105). In subsequent work, 1,1,2,2-tetrachloroethane was observed to degrade rapidly into trichloroethylene during storage on Pittsburgh activated carbon: 19% converted during one day and 63% converted during eight days at ambient conditions [7]. Therefore, the analyte is sensitive to the sorbent type; only the recommended sorbent should be used. Breakthrough at high relative humidity was not tested, nor was sample stability.

NMAM 2562, Issue 1

Initial work to update the NMAM method 1019 resulted in an average recovery of 88.4% over a range of 38.1 to 253.8 μg from the petroleum charcoal [1]. In order to see if the overall desorption efficiency of 1,1,2,2-tetrachloroethane could be improved at these lower levels, the sorbent was changed from petroleum charcoal to Anasorb CMS. Desorption efficiency using Anasorb CMS (SKC Lot 1879) was 91, 85, 88, and 91% at 38, 76, 165, and 254 μg 1,1,2,2-tetrachloroethane per sample, respectively. The limit of detection was found to be 0.6 μg per sample. A recovery of 87% was obtained at a level of 165 μg after storage for 28 days at ambient conditions [1].

REFERENCES:

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- [7] Arnold, J. Internal memo (NIOSH, unpublished, May 8, 1984)

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