**METHOD: 1016, Issue 2**

**EVALUATION: PARTIAL**

<table>
<thead>
<tr>
<th>PROPERTY</th>
<th>VALUE</th>
</tr>
</thead>
<tbody>
<tr>
<td>OSHA</td>
<td>500 ppm</td>
</tr>
<tr>
<td>NIOSH</td>
<td>500 ppm</td>
</tr>
<tr>
<td>ACGIH</td>
<td>500 ppm</td>
</tr>
<tr>
<td>MW</td>
<td>203.83</td>
</tr>
</tbody>
</table>
| CAS      | (1) 76-11-9  
           | (2) 76-12-0 |
| RTECS    | (1) KI1425000  
           | (2) KI1420000 |

**PROPERTIES:**
- Solids; MP (1) 40.6 °C; (2) 25 °C
- BP (1) 91.5 °C; (2) 93 °C
- VP 5.3 kPa (40 mm Hg; 5.2% v/v) @ 20 °C; not combustible

**SYNONYMS:**
- (1): Refrigerant 112a.
- (2): Refrigerant 112.

**SAMPLING**

**SAMPLER:**
- Solid sorbent tube
  - Coconut shell charcoal, 100 mg/50 mg

**FLOW RATE:**
- 0.01 to 0.035 L/min

**VOL-MIN:**
- 0.5 L @ 500 ppm
- 2 L

**SHIEMENT:**
- Routine

**SAMPLE STABILITY:**
- Not tested

**BLANKS:**
- 2 to 10 field blanks per set

**MEASUREMENT**

**TECHNIQUE:**
- Gas chromatography, FID

**ANALYTE:**
- 1,1,1,2-tetrachloro-2,2-difluoroethane;
  - 1,1,2,2-tetrachloro-1,2-difluoroethane

**DESORPTION:**
- 1 mL CS₂; stand 30 min

**INJECTION VOLUME:**
- 5 µL

**TEMPERATURE-INJECTION:**
- (1) 185 °C  
  - Detector: 250 °C
- (2) 50 °C  
  - Column: 240 °C

**COLUMN:**
- 3 m x 3-mm OD stainless steel packed with 10% FFAP on 80/100 mesh Chromosorb WHP

**CALIBRATION:**
- Standard solutions in CS₂

**RANGE:**
- 2 to 20 mg per sample

**ESTIMATED LOD:**
- 0.3 mg per sample

**PRECISION (S):**
- (1): 0.27 @ 4 to 17 mg per sample [1]; 0.005 @ 4 to 17 mg per sample [1]

**APPLICABILITY:**
- The working range for either analyte is 120 to 1400 ppm (1000 to 12,000 mg/m³) for a 2-L air sample. These compounds are used as degreasing solvents, refrigerants, foaming agents and corrosion inhibitors. Capillary columns may be used (DB-Wax or Nukol on fused silica, 3 m x 0.32-mm, 0.5 µm film) with appropriate changes in instrumental conditions.

**INTERFERENCES:**
- None reported.

**OTHER METHODS:**
- This combines and revises Methods S131 and S132 [2].
REAGENTS:

1. Carbon disulfide (CS₂), chromatographic quality.*
2. 1,1,1,2-Tetrachloro-2,2-difluoroethane and 1,1,2,2-tetrachloro-1,2-difluoroethane, reagent grade.*
3. Hexane, chromatographic quality.
4. Calibration stock solution, 0.2 mg/µL. Dissolve 2 g analyte in CS₂ to prepare 10 mL solution. Prepare in duplicate.
5. DE stock solution, 1 mg/µL. Dissolve 10 g analyte in hexane to prepare 10 mL solution. Prepare in duplicate.
8. Air, filtered, compressed

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends, containing two sections of 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 0.035 L/min, with flexible connecting tubing.
3. File, triangular.
4. Gas chromatograph, flame ionization detector, integrator and column (page 1016-1).
5. Vials, 2-mL, PTFE-lined caps.
6. Syringes, 10-µL, readable to 0.1 µL.
7. Volumetric flasks, 10-mL.
8. Pipets, 10- to 1000-µL.

SPECIAL PRECAUTIONS: Tetrachloro-1,2-difluoroethane has been determined to be a carcinogen [3]. Both analytes react with chemically-active metals such as sodium, potassium and beryllium or with powdered magnesium, aluminum and zinc.

Hazardous products such as hydrogen chloride, hydrogen fluoride and carbon monoxide may be released when either analyte decomposes. Both analytes will attack some forms of plastics, rubber and coatings.

Carbon disulfide is toxic and flammable; work with it 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 0.035 L/min for a total sample size of 0.5 to 2 L.

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 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.
   a. Add known amounts of calibration stock solution to CS₂ in 10-mL volumetric flasks and
dilute to the mark. Use serial dilutions as needed to obtain analyte concentrations in the
range 0.3 to 20 mg/mL.
   b. Analyze with samples and blanks (steps 11 and 12).
   c. Prepare calibration graph (peak area vs. mg analyte).

9. Determine desorption efficiency (DE) at least once for each lot of sorbent used for sampling in
the range of interests. 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 (2 to 20 µL) of DE stock solution, or a serial dilution thereof, 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 analyte 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 1016-1. Inject sample aliquot manually using solvent flush technique or with
autosampler.
   NOTE: If peaks area is above the linear range of the working standards, dilute an aliquot of the
desorbed liquid with CS₂, reanalyze and apply the appropriate dilution factor in
calculations.

12. Measure peak area.

CALCULATIONS:

13. Determine the mass, mg (corrected for DE) of analyte found in the sample front (W_f) and back
(W_b) sorbent sections, and 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 analyte 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:

1,1,1,2-Tetrachloro-2,2-difluoroethane: Method S131 was issued on May 9, 1975 [2], and was validated
at 2.08, 4.17 and 8.35 mg/m³ [1]. The generated concentrations were confirmed by gas
chromatographic analysis and comparison to bag standards. The average desorption efficiency was
103.6% over the range 4 to 17 mg per sample. The breakthrough volume was 2.7 L when sampling
14,300 mg/m³ at 0.035 L/min.

1,1,2,2-Tetrachloro-1,2-difluoroethane: Method S132 was issued on May 9, 1975 [2], and was validated
at 20.05, 4.15 and 8.35 mg/m³ [1]. The generated concentrations were confirmed by gas
chromatographic analysis and comparison to bag standards. The average desorption efficiency was
105% over the range 4 to 17 mg per sample. Breakthrough had not occurred after 3 h sampling 6990
mg/m³ at 0.041 L/min. The test was stopped after 3 h.

REFERENCES:


METHOD REVISED BY:

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