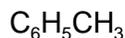


TOLUENE (diffusive sampler)

4000



MW: 92.14

CAS: 108-88-3

RTECS: XS5250000

METHOD: 4000, Issue 2

EVALUATION: FULL

Issue 1: 15 August 1987

Issue 2: 15 August 1994

OSHA : 200 ppm; C 300 ppm; P 500 ppm
NIOSH: 100 ppm; STEL 150 ppm
ACGIH: 50 ppm (skin)
 (1 ppm = 3.77 mg/m³)

PROPERTIES: liquid; d 0.866 g/mL @ 20 °C; BP 110.6 °C;
 MP -95 °C; VP 3.8 kPa (28 mmHg; 3.7% v/v)
 @ 25 °C; explosive range 1.3 to 7.1% v/v in
 air

SYNONYMS: methylbenzene; phenyl methane; toluol

| SAMPLING | MEASUREMENT |
|--|--|
| <p>SAMPLER: DIFFUSIVE SAMPLER (activated carbon)</p> <p>SAMPLE TIME-MIN: 15 min @ 200 ppm -MAX: 8 h @ 20 to 200 ppm</p> <p>SHIPMENT: see step 2 - if necessary, transfer sorbent pad to septum-capped vial; otherwise, routine</p> <p>SAMPLE STABILITY: at least 2 weeks @ 25 °C if stored in septum-capped vial</p> <p>BLANKS: 2 to 10 field blanks per set</p> <p>BULK SAMPLE: desirable</p> | <p>TECHNIQUE: GAS CHROMATOGRAPHY, FID</p> <p>ANALYTE: toluene</p> <p>DESORPTION: 1.5 mL carbon disulfide; stand 30 min</p> <p>INJECTION VOLUME: 5 µL</p> <p>TEMPERATURE-INJECTION: 175 °C -DETECTOR: 200 °C -COLUMN: 100 °C</p> <p>CARRIER GAS: He or N₂, 30 mL/min</p> <p>COLUMN: 3.6 m x 3-mm OD, SP-1000 on 80/100 mesh Chromosorb WHP</p> <p>CALIBRATION: analyte solutions in CS₂</p> <p>RANGE: 0.5 to 15 mg per sample</p> <p>ESTIMATED LOD: 0.01 mg per sample</p> <p>PRECISION (S_r): 0.022 [1]</p> |
| ACCURACY | |
| <p>RANGE STUDIED: 75 to 2250 mg/m³ [1]</p> <p>BIAS: not significant [1]</p> <p>OVERALL PRECISION (S_{rr}): 0.038 [1]</p> <p>ACCURACY: ± 7.4%</p> | |

APPLICABILITY: The working range is 13 to 660 ppm (50 to 2500 mg/m³) for a 4-hour sample. The method is applicable to STEL determinations. The method determines toluene vapor only (not aerosol). Competitive adsorption by water vapor and other volatile organic solvents affects sampler capacity.

INTERFERENCES: None identified. The chromatographic column or separation conditions may be changed to circumvent interference problems.

OTHER METHODS: NIOSH Methods 1500 and 1501 [2] employ active sampling on charcoal tubes with overall sensitivity about the same as this method.

REAGENTS:

1. Eluent: carbon disulfide (CS₂), chromatographic quality.*
2. Toluene, reagent grade.
3. Nitrogen or helium, purified.
4. Hydrogen, prepurified.
5. Air, filtered.

* See SPECIAL PRECAUTIONS

EQUIPMENT:

1. Sampler: diffusive monitor with activated charcoal collection element. Monitors must be of known sampling rate for toluene.
2. Gas chromatograph, FID, integrator and column (page 4000-1).
3. Vials, glass, 2-mL, with PTFE-lined septum caps.
4. Pipet, TD, 1.5-mL, and pipet bulb.
5. Syringes, 5-, 10-, 25- and 100- μ L.
6. Volumetric flasks, 10-mL.
7. Tweezers.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and extremely flammable (flash point =30 °C); work with it only in a fume hood.

SAMPLING:

1. Remove sampler from package and remove caps, if applicable. Attach sampler in worker's breathing zone. Make sure sampler inlets are unobstructed. Record start of sampling time. Follow manufacturer's recommendations to estimate appropriate sampling time based on relative humidity and suspected concentration of toluene.

NOTE: The uptake constant is only valid for samplers in air with ambient velocity higher than ca. 10 cm/sec. For area samples, ensure adequate air movement.

2. Terminate sampling by sealing the sampler as recommended by the manufacturer or by transferring, in a toluene-free environment, the activated charcoal collection element to a vial and sealing the vial with a septum cap. Use tweezers for handling the collection element, taking care not to touch any activated charcoal.

NOTE: Some sampler designs (e.g., 3M 3500 [3]) do not require transfer of the collection element; others (e.g., Gilian Trace Air OVM-1 and OVM-2) require transfer of sorbent pad to a sealed vial. Some sampler designs (e.g., Draeger ORSA-5) use granular sorbent.

SAMPLE PREPARATION:

3. Add 1.5 mL CS₂ to each collection element.
4. Allow to stand at least 30 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

5. Calibrate daily with at least six working standards.
 - a. Add known amounts of toluene to CS₂ in 10-mL volumetric flasks and dilute to the mark to produce toluene concentrations in the range 0.01 to 10 mg/mL.
 - b. Analyze with samples and blanks (steps 8 through 10).
 - c. Prepare calibration graph (peak area vs. mg/mL toluene).
6. Determine desorption efficiency (DE) in the calibration range at least once for each lot of samplers used. Prepare three samplers at each of five concentrations plus three media blanks.
 - a. Place the collection element of a media blank sampler into a vial and cap.
 - b. Inject a known amount of toluene directly onto the collection element with a microliter syringe.
 - c. Allow to stand overnight.
 - d. Desorb (steps 3 and 4) and analyze with working standards.
 - e. Prepare a graph of DE vs. mg toluene recovered.

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

MEASUREMENT:

- Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 4000-1.
- 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 CS₂, reanalyze, and apply the appropriate dilution factor in calculations.
- Measure peak area.

CALCULATIONS:

- From the calibration graph and desorption volume, determine the mass, mg (corrected for DE), of toluene found on the sample collection element (W) and on the average media blank (B).
NOTE: If $W > \text{sampler capacity} \times 0.33$, the sampler may be saturated [4]. Evaluate each result on the basis of other organics present in the sampled atmosphere, the concentrations of all the adsorbable organics present, the ambient relative humidity, the sampling rate for the organics present and the sampling time.
- Calculate concentration, C (mg/m³), of toluene in the air sampled:

$$C = \frac{(W - B) \cdot 10^6}{t \cdot K}, \text{ mg/m}^3$$

where: t = length of sampling period (min)
K = uptake constant (cm³ toluene/min).

NOTE: The uptake constant must be known accurately (either be supplied by the sampler manufacturer or determined by the user by calibration with standard toluene-in-air mixtures [5]). K can be estimated from the diffusion coefficient, D (cm²/min), the cross-sectional area, A (cm²), and the diffusion pathlength, L (cm), of the sampler:

$$K = \frac{D \cdot A}{L}$$

EVALUATION OF METHOD:

Precisions and biases listed in the method were determined by analyzing generated atmospheres containing various concentrations of toluene at a range of humidities and in the presence of other volatile organics [3]. Generated concentrations were independently verified.

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METHOD WRITTEN BY:

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