o-TERPHENYL 5021

$C_{18}H_{14}$ MW: 230.31 CAS: 84-15-1 RTECS: WZ6472000

METHOD: 5021, Issue 2	EVALUATION: PARTIAL	Issue 1: 15 May 1985 Issue 2: 15 August 1994

OSHA : C 1 ppm (o-, m-, and p-isomers)
NIOSH: C 0.5 ppm (o-, m-, and p-isomers)
ACGIH: C 0.53 ppm (o-, m-, and p-isomers)
(1 ppm = 9.42 mg/m^3)

PROPERTIES: solid; MP 58 °C; BP 332 °C; VP 0.035 Pa (0.00026 mm Hg; 0.35 ppm) @ 22 °C [1]

SYNONYMS: o-diphenyl benzene; o-triphenyl; 1,2-diphenyl benzene

SAMPLING

SAMPLER:	FILTER (2.0 µm, PTFE)
FLOW RATE:	1 to 3 L/min
VOL-MIN:	2 L
-MAX:	30 L
SHIPMENT:	routine
SAMPLE STABILITY:	not determined
FIELD BLANKS:	2 to 10 field blanks per set

ACCURACY

RANGE STUDIED:	4.5 to 20 mg/m^3 [1] (15-L samples)
BIAS:	- 4.4%
OVERALL PRECISION :	0.097 [1]
ACCURACY ($\hat{S}_{rT}$):	± 20.0%

MEASUREMENT

TECHNIQUE:	GAS CHROMATOGRAPHY, FID
ANALYTE:	o-terphenyl
DESORPTION:	CS_2
INJECTION VOLUME:	5 µL
TEMPERATURE-DETECTOR:	230 °C
-INJECTOR:	220 °C
-COLUMN:	185 °C
CARRIER:	N_2, 30 mL/min
COLUMN:	3 m x 3-mm OD stainless steel packed with 5% SE-30 on 80/100 mesh Gas Chrom W
CALIBRATION:	standard solutions of o-terphenyl in CS_2
RANGE:	0.015 to 0.5 mg per sample
ESTIMATED LOD:	0.002 mg per sample
PRECISION ($\bar{S}_r$):	0.052 @ 0.07 to 0.27 mg per sample [1]

APPLICABILITY: The working range is 0.5 to 60 mg/m^3 for a 30-L air sample. The method has been tested only with o-terphenyl.

INTERFERENCES: None identified.

OTHER METHODS: This revised Method S27 [2].

REAGENTS:

1. Carbon disulfide, chromatographic quality.*
2. ortho-Terphenyl, reagent grade.*
3. Hexane, chromatographic quality.*
4. Calibration stock solution, 1 mg/mL. Dissolve an accurately weighed 100 mg o-terphenyl in hexane and dilute to 100 mL.
5. Air, filtered, compressed.
6. Nitrogen, purified.
7. Hydrogen, prepurified.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: PTFE laminated membrane filters, 2.0-µm, 37-mm (Zefluor, Membrana Corp., Pleasonton, CA, or equivalent) backed by a gasket (37-mm OD, 32-mm ID) cut from a cellulose acetate support pad (available SKC, Inc. #225-23) in cassette filter holder.
2. Personal sampling pump, 1 to 3 L/min, with flexible connecting tubing.
3. Vials, scintillation, screw cap, 20-mL.
4. Tweezers.
5. Gas chromatograph, FID, integrator, and column (page 5021-1).
6. Beakers, 50-mL.
7. Centrifuge tubes, graduated, calibrated at 0.5 mL.
8. Volumetric flasks, 10- and 100-mL.
9. Pipets, 1.0 to 100-µL.
10. Syringe, 10-µL, readable to 0.1 µL.

SPECIAL PRECAUTIONS: Exposure to terphenyls may cause eye, skin, and respiratory irritation [3]. Carbon disulfide presents a severe hazard owing to its toxic properties and fire and explosion potential (flash point = -30 °C). All work with this solvent should be performed in a fume hood. Hexane has a flash point = -22 °C.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 1 and 3 L/min for a total sample size of 2 to 30 L.
3. Transfer the filter from the cassette within 1 h of sampling to a clean vial. Handle the filter only with clean tweezers.

SAMPLE PREPARATION:

4. Remove filter from vial and place in 50-mL beaker. Rinse vial with 1 mL CS_2.
5. Extract filter three times with 1-mL portions of CS_2.
6. Transfer the extracts and vial rinsing to a graduated centrifuge tube. Gently evaporate the solution with a stream of nitrogen to a final volume of 0.5 mL.

CALIBRATION AND QUALITY CONTROL:

7. Calibrate daily with at least six working standards over the range 0.002 to 0.5 mg o-terphenyl per sample.
   a. Add known amounts of calibration stock solution to CS_2 in 10-mL volumetric flasks and dilute to the mark.
   b. Analyze together with samples and blanks (steps 10 and 11).
   c. Prepare calibration graph (peak area vs. mg of o-terphenyl).

8. Determine recovery (R) at least once for each lot of filters used for sampling in the calibration range (step 7). Prepare three filters at each of five levels plus three media blanks.
   a. Spike a known amount of calibration stock solution directly onto the filter with a microliter pipet or syringe.
   b. Allow to stand overnight in the dark.
   c. Extract (steps 4 through 6) and analyze together with working standards (steps 10 and 11).
   d. Prepare graph of recovery vs. mg o-terphenyl recovered.
9. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and recovery graph are in control.

MEASUREMENT:

10. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 5021-1. Inject 5-µL sample aliquot manually using solvent flush technique or with autosampler.
    NOTE: If peak area is above the linear range of the working standards, dilute with CS_2, reanalyze, and apply the appropriate dilution factor in calculations.
11. Measure peak area.

CALCULATIONS:

12. Determine the mass, mg (corrected for recovery) of o-terphenyl found in the sample (W) and in the average media blank (B) from the calibration graph.
13. Calculate concentration, C, of o-terphenyl in the air volume sampled, V (L):

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

EVALUATION OF METHOD:

Method S27 was issued on November 8, 1974, and was validated for o-terphenyl with both spiked filters and an aerosol generated from a 0.25% (w/v) solution of o-terphenyl in toluene by an aspiration/spray dry technique [1]. The collection efficiency for o-terphenyl of a mixed cellulose ester membrane filter was 1.00 ± 0.01 when sampling an atmosphere containing 18.8 mg/m^3 at 1 L/min for 15 min. No o-terphenyl was detected on charcoal tubes preceded by glass fiber filters when sampling an atmosphere containing 17 mg/m^3 o-terphenyl. Storage stability of the samples was not determined.

REFERENCES:

[1] Documentation of the NIOSH Validation Tests, S27, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977), available as GPO Stock #017-033-00231-2 from Superintendent of Documents, Washington, DC 20402.
[2] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 2, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).
[3] NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, U.S. Department of Health and Human Services, Publ. (NIOSH) 81-123 (1981), available as GPO Stock #017-033-00337-8 from Superintendent of Documents, Washington, DC 20402.

METHOD REVISED BY:

B. R. Belinky, NIOSH/DPSE; S27 originally validated under NIOSH Contract CDC-99-74-45.