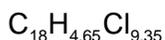


CHLORINATED TERPHENYL (60% CHLORINE)

5014



MW: 552 (average)

CAS: 11126-42-4

RTECS: TQ1390000

METHOD: 5014, Issue 2
1984

EVALUATION: PARTIAL

Issue 1: 15 February

OSHA : no PEL
NIOSH: no REL
ACGIH: no TLV
(1 ppm \approx 23 mg/m³ @ NTP)

PROPERTIES: solid; d 1.67 g/mL @ 25 °C; BP 280 to 335 °C @ 5 mm Hg (0.67 kPa); softening point 98 to 105.5 °C

SYNONYMS: chlorinated triphenyl, polychloroterphenyl, PCT, Aroclor 5460

APPLICABILITY: The working range is 0.01 to 1 mg/m³ for a 100-L air sample.

INTERFERENCES: Presence of other chlorinated terphenyls, e.g., Aroclor 5442 (42% chlorine), or other substances with similar retention and detection characteristics may interfere with the analysis.

OTHER METHODS: None evaluated by NIOSH.

REAGENTS:

1. Isooctane (2,2,4-trimethylpentane), distilled in glass.*
2. Hexane, distilled in glass.*
3. Chlorinated terphenyl (60% chlorine).*
4. Calibration stock solution, 1 mg/mL. Dilute an accurately weighed 100-mg portion of chlorinated terphenyl to 100 mL with hexane.
5. Nitrogen, 99.999%.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: 37-mm glass fiber filter (Gelman #64877 or equivalent) supported by a backup pad in a closed-face polystyrene cassette (Gelman #4338 or equivalent).
2. Personal sampling pump, 1 to 3 L/min, with flexible connecting tubing.
3. Gas chromatograph, ⁶³Ni ECD, integrator and column (page 5014-1).
4. Tweezers, stainless steel.
5. Vials, glass, 5-mL, crimp seals or caps, silicone rubber septa lined with polyperfluoroethylene.
6. Pipets, 1-, 2- and 4-mL, with pipet bulb.
7. Ultrasonic bath.
8. Balance, readable to 0.1 mg.
9. Volumetric flasks, 5-, 10-, 25- and 100-mL.
10. Syringe, 10- μ L.

SPECIAL PRECAUTIONS: Hexane (flash point = -22 °C) and isooctane (flash point = 4 °C) are highly flammable. Prepare samples and standards in well-ventilated hood.

Chlorinated terphenyls may be similar to chlorinated biphenyls in toxicity. Use protective clothing to avoid skin contact.

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 100 to 1500 L.

SAMPLE PREPARATION:

3. Transfer filter to 5-mL vial using tweezers. Discard backup pad.
4. Pipet 4.0 mL of isooctane into vial and seal immediately.
5. Agitate vial in ultrasonic bath for 30 min to complete extraction.

CALIBRATION AND QUALITY CONTROL:

6. Calibrate daily with at least six working standards over the range 1 to 100 μ g chlorinated terphenyl per sample.
 - a. Add known amounts of calibration stock solution to hexane in 10-mL volumetric flasks and dilute to the mark.
 - b. Analyze together with samples and blanks (steps 9 and 10).
 - c. Prepare calibration graph (detector response vs. μ g chlorinated terphenyl per sample).
7. Prepare two control samples at each of two levels for checking analysis of field samples.
 - a. For each control sample, inject calibration stock solution directly onto filter. Mount filter in cassette and draw ca. 30 L of analyte-free air through (to evaporate hexane). Seal cassette and store overnight.

- b. Desorb filters as in steps 3 through 5, and analyze as in steps 9 and 10.
8. Check for possible contamination during shipment of field samples by comparing results from field blanks and media blanks.

MEASUREMENT:

9. Set gas chromatograph to conditions given on page 5014-1 and optimize detector performance per manufacturer's instructions. Using given conditions, retention time of largest and most distinct peak in cluster was about 6.1 min.
10. Inject sample aliquot and measure detector response, e.g., height from base of cluster to top of most prominent peak.

CALCULATIONS:

11. Read the mass, μg , of chlorinated terphenyl found in each sample (W) and average media blank (B) from calibration graph.
12. Calculate concentration, C (mg/m^3), of chlorinated terphenyl in the air volume sampled, V (L):

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

EVALUATION OF METHOD:

Measurement precision was determined by analyzing spiked sampling media [1]. The average recovery was 1.006. There appeared to be no effect on recovery when up to 2700 L of charcoal-filtered air (40 °C and 40% relative humidity) was passed through filters spiked at the 1- μg level. Recoveries from 100-L samples collected from generated aerosol were 1.05 ($\bar{S}_r = 0.162$) after seven days storage, and 0.875 ($\bar{S}_r = 0.206$) after 14 days storage, relative to the amount found after one day. Generated concentrations were not reproducible, and independent methods for determining the true concentration appeared to provide lower and more imprecise results than this method. Because of the difficulty in determining the true concentration, overall precision and bias were not determined. References [1] and [2] provide additional information.

REFERENCES:

- [1] Graham, M. M., and H. K. Dillon. Analytical Methods Evaluation and Validation for 1,2,4-Trichlorobenzene, 1,2,4,5-Tetrachlorobenzene, Pentachlorobenzene, and Polychlorinated Terphenyls: Research Report for Polychlorinated Terphenyls, NIOSH Contract No. 210-79-0102, Southern Research Institute, Birmingham, AL, available from NTIS (1982) as PB-83-140-087.
- [2] Kimbrough, R. D., ed. "Halogenated Biphenyls, Terphenyls, Naphthalenes, Dibenzodioxins and Related Products," Topics in Environmental Health, V. 4, Elsevier/North-Holland Biomedical Press, Amsterdam, 20-21 (1980).

METHOD WRITTEN BY:

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