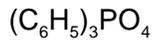


TRIPHENYL PHOSPHATE

5038



MW: 326.28

CAS: 115-86-6

RTECS: TC8400000

METHOD: 5038, Issue 1

EVALUATION: PARTIAL

Issue 1: 15 August 1994

OSHA : 3 mg/m³
NIOSH: 3 mg/m³
ACGIH: 3 mg/m³

PROPERTIES: solid; MP 50 °C; BP 245 °C
 @ 11 mm Hg; d 1.27 g/mL @ 60 °C;
 VP <0.05 kPa (<0.1 mm Hg);
 flash point 220 °C

SYNONYMS: phosphoric acid triphenyl ester; TPP

APPLICABILITY: The working range is 0.3 to 20 mg/m³ for a 100-L air sample. This method may be adapted to other phosphates of relatively low volatility with appropriate changes in chromatographic conditions.[1] A non-polar capillary column may be used for better resolution and sensitivity.

INTERFERENCES: None identified.

OTHER METHODS: This method revises S210 [1].

REAGENTS:

1. Diethyl ether, anhydrous, reagent grade.
2. Triphenyl phosphate,* reagent grade.
3. Hydrogen, purified.
4. Compressed air, prefiltered.
5. Nitrogen, purified.
6. Calibration stock solution (10-600 µg triphenyl phosphate per 10 mL diethyl ether).

* See Special Precautions

EQUIPMENT:

1. Sampler: 37-mm cellulose ester membrane filter (0.8-µm pore size) with cellulose backup pad held in a three-piece cassette filter holder supported by a cellulose backup pad.
2. Personal sampling pump @ 1 to 3 L/min, with flexible tubing.
3. Gas chromatograph equipped with a flame photometric detector, phosphorus filter, and column (p. 5038-1).
4. Electronic integrator or other suitable method for measuring peak areas.
5. Ointment jars, 2 oz., squat form with aluminum-lined screw caps.
6. Syringes, 10-µL and other convenient sizes for preparing standard solutions.
7. Pipets, 10-mL and other convenient sizes for preparing standard solutions.

SPECIAL PRECAUTIONS: Repeated exposure to triphenyl phosphate through inhalation or ingestion may cause minor changes in blood enzymes. Triphenyl phosphate also causes neurotoxic effects in animals; therefore, persons with pre-existing neuromuscular disorders may be at increased risk.[2] Handle only in a fume hood. Avoid skin contact.

SAMPLING:

1. Calibrate each personal sampling pump with a representative filter cassette in line.
2. Remove the cassette plugs and attach to the personal sampling pump with flexible tubing.
3. Sample 10 to 400 L or more of air at an accurately known flow rate of 1 to 3 L/min.
4. Seal the collected sample cassette firmly with plugs. Pack securely for shipment.

SAMPLE PREPARATION:

5. Transfer cellulose membrane filter and cellulose backup pad with tweezers to an ointment jar.
6. Pipet 10.0 mL of ether into each jar. Seal the jar immediately to minimize evaporation.
7. Allow samples to stand for at least 30 minutes with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards over the range 10 to 900 µg TPP per sample.
 - a. Add known amounts of calibration stock solution to cover the range of interest to 10.0-mL volumetric flasks and dilute to the mark with diethyl ether.
 - b. Analyze working standards together with samples and blanks (steps 11 and 12).
 - c. Prepare a calibration graph of peak area vs. µg of triphenyl phosphate per 10-mL sample.
9. Determine recovery for each lot of filters used for sampling in the concentration range of interest. Prepare three filters at each of five levels plus three media blanks.
 - a. Spike aliquot of calibration solution onto each filter.
 - b. After air-drying, extract filters in 10 mL diethyl ether (steps 5 through 7).
 - c. Analyze together with working standards.

- d. Prepare graph of recovery vs. μg triphenyl phosphate per sample.
10. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration and recovery graphs are in control.

MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 5038-1. Inject 5- μL sample aliquot using solvent flush technique or with autosampler. NOTE: If peak area is above the linear range of the calibration graph, dilute, reanalyze, and apply appropriate dilution factor in the calibration.
12. Measure peak area.

CALCULATIONS:

13. Determine mass, μg (corrected for recovery), of triphenyl phosphate found in the sample (W) and in the average media blank (B).
14. Calculate concentration (C) of triphenyl phosphate in the volume of air sampled, V (L):

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

EVALUATION OF METHOD:

This method was evaluated over the range of 1.25 to 6.99 mg/m^3 at an atmospheric temperature and pressure of 24 °C and 759 mm Hg, using a 100-L sample. Overall sampling and measurement precision, \hat{S}_{rT} , was 0.066, with average recovery of 99.1%, representing a non-significant bias. Recovery of triphenyl phosphate from filters was 99.3% for an air sample concentration of 7.0 mg/m^3 at 1.5 L/min for 66 minutes. [1,3]. A storage stability study was not conducted.

REFERENCES:

- [1] NIOSH Manual of Analytical Methods, 2nd. ed., V. 3, S210, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) CDC-99-74-45 (1978).
- [2] Occupational Health Guideline for Triphenyl Phosphate, U.S. Department of Health and Human Services (NIOSH) and U.S. Department of Labor (OSHA) (1978).
- [3] Backup Data Report for Triphenyl Phosphate, prepared under NIOSH Contract CDC-99-74-45.

METHOD REVISED BY:

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