EPN 5012

\[ \text{C}_{14}\text{H}_{14}\text{NO}_4\text{PS} \quad \text{W: 323.31} \quad \text{CAS: 2104-64-5} \quad \text{RTECS: TB1925000} \]

**METHOD:** 5012, Issue 2  
**EVALUATION:** FULL  
Issue 1: 15 May 1989  
Issue 2: 15 August 1994

**OSHA:** 0.5 mg/m\(^3\) (skin)  
**NIOSH:** 0.5 mg/m\(^3\) (skin); Group I Pesticide  
**ACGIH:** 0.5 mg/m\(^3\) (skin)

**SYNONYMS:** phenylphosphonothioic acid \(\text{O}-\text{ethyl-\(\text{O}-\text{p}\)-nitrophenyl ester}\)

**PROPERTIES:** solid; MP 36 °C; d 1.268 g/mL @ 25 °C  
VP 0.04 Pa (0.0003 mm Hg, 5 mg/m\(^3\)) @ 100 °C

**SAMPLING**

**SAMPLER:** FILTER  
(glass fiber)

**FLOW RATE:** 1 to 2 L/min

**VOL-MIN:** 15 L @ 0.5 mg/m\(^3\)

**VOL-MAX:** 700 L

**FIELD TREATMENT:** transfer filters within 1 h of sampling to vial

**SHIPMENT:** in vials

**SAMPLE STABILITY:** at least 7 days @ 25 °C

**MEASUREMENT**

**TECHNIQUE:** GAS CHROMATOGRAPHY, FLAME PHOTOMETRIC

**ANALYTE:** EPN

**DESORPTION:** 15 mL isoctane

**INJECTION VOLUME:** 5 µL

**TEMPERATURE-INJECTION:** 215 °C

**-DETECTOR:** 215 °C

**-COLUMN:** 205 °C

**CARRIER GAS:** \(\text{N}_2\) or He, 60 mL/min

**COLUMN:** 2 m x 6-mm glass, packed with 3% OV-1 on 100/120 mesh Gas Chrom Q

**CALIBRATION:** solutions of the EPN in isoctane

**RANGE:** 6 to 170 µg per sample

**ESTIMATED LOD:** 2 ng per sample [1]

**PRECISION \(\hat{S}_p\):** 0.04 [1]

**ACCURACY**

**RANGE STUDIED:** 0.3 to 1.2 mg/m\(^3\) [1]  
(120-L samples)

**BIAS:** 0%

**OVERALL PRECISION \(\hat{S}_p\):** 0.06 [1,2]

**ACCURACY:** ± 11.4%

**APPLICABILITY:** The working range is 0.05 to 1.5 mg/m\(^3\) EPN for 120-L air samples. Malathion and Parathion have also been determined by this procedure.

**INTERFERENCES:** None known.

**OTHER METHODS:** This method replaces Method S285 [2]. The method of Hill and Arnold [3] has also been used for analysis of pesticides. Method 5600 is an alternative method for organophosphorus pesticides.
REAGENTS:

1. EPN.*
2. Isooctane, chromatoquality.*
3. Calibration stock solution, 15 mg/mL isooctane (prepare fresh daily):
   NOTE: 4 µL of this solution contains a mass of EPN (0.06 mg) equivalent to a 120-L air sample at OSHA PEL.
5. Hydrogen, prepurified.
6. Oxygen, purified.
7. Air, filtered, compressed.
8. Vials, 20-mL, glass with PTFE-lined caps.
9. Syringe, 10-µL, readable to 0.1 µL.
10. Volumetric flasks, 10-mL.
11. Pipets, 15-mL, with pipet bulb.
12. Tweezers.

* See SPECIAL PRECAUTIONS.

SPECIAL PRECAUTIONS: EPN is a highly toxic cholinesterase inhibitor with cumulative effects [4,5]. Special care must be taken to avoid inhalation or skin contact.

Isooctane is flammable. Prepare all samples in a well-ventilated hood.

SAMPLE PREPARATION:

4. Pipet 15 mL isooctane into each vial. Cap each vial and swirl the contents for 1 h.

CALIBRATION AND QUALITY CONTROL:

5. Prepare at least six working standards covering the analytical range of the method, by diluting aliquots of the calibration stock solutions to 15 mL with isooctane.
   a. Analyze calibration standards in triplicate with the unknowns, blanks and other quality control spiked samples following steps 7 through 9.
   b. Prepare calibration graph (peak area or peak height vs. µg of EPN).
6. Analyze recovery (R) samples with each sample set, in duplicate.
   a. Place filter media blanks in clean 20-mL vials.
   b. Inject analyte calibration stock solution directly onto the filter with a microliter syringe.
   c. Cap the vial and allow to stand overnight.
   d. Analyze according to steps 4 and 7 through 9.
   e. Calculate recovery (µg found on filter divided by µg added to filter).

MEASUREMENT:

7. Set gas chromatograph to conditions given on page 5012-1, optimizing the air, hydrogen and oxygen according to the GC manufacturer's instructions.
8. Inject 5-µL sample aliquots using solvent flush technique.
   a. Vent the solvent peak by opening the column bypass valve at the time of injection. Close the column bypass valve after the solvent peak has eluted (ca. 30 sec) and before the
analyte peak elutes.

b. Make replicate injections of samples and standards.

9. Measure peak area or peak height.

CALCULATIONS:

10. Read the mass, µg per sample, of EPN found on the sample filters, W, and average media blank filters, B, from the calibration graph.

11. Calculate the concentration, C, of EPN in the air volume sampled, V (L):

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

EVALUATION OF METHOD:

Method S285 for EPN was issued on April 26, 1976 [2]. The precision and bias were obtained by generating atmospheres of EPN at one-half, one and two times the OSHA standards [1,6]. Test atmospheres were generated using Trion-6 EPN (Wilbur Ellis Co.). Gelman Type AE glass fiber filters were used for all sampling and measurement recovery studies. Collection efficiency of the glass fiber filter was determined to be 1.0 and losses from vaporization of known amounts of EPN deposited on a glass fiber filter were negligible [4]. Filters spiked with solutions of EPN gave quantitative recoveries after storage for 7 days at 25 °C.

REFERENCES:


METHOD REVISED BY:

Paula Fey O'Connor, NIOSH/DPSE; Method S285 originally validated under NIOSH Contract CDC 99-74-45.