Method number:	PV2050
Target Concentration:	0.2 mg/m^3 (arbitrary). There is no OSHA PEL or ACGIH TLV for propetamphos.
Procedure:	Samples are collected by drawing known volumes of air through 37-mm glass fiber filters (GFFs). Samples are extracted with toluene and analyzed by gas chromatography (GC) using an electron capture detector (ECD).
Recommended air volume and sampling rate:	60 minutes at 1.0 L/min (60 L)
Detection limit of the overall procedure	0.02 mg/m^3 (based on the recommended air volume and the analytical detection limit):
Status of method:	Partially Validated method. This method has been partially evaluated and is presented for information and trial use only.

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1 General Discussion

1.1 Background

1.1.1 History of procedure

This evaluation was undertaken to determine the effectiveness of a GFF as a sampling media for propetamphos.

1.1.2 Toxic effects (This section is for information only and should not be taken as the basis of OSHA policy).

The following paragraph is excerpted from the book “OCCUPATIONAL DISEASES, A Guide to Their Recognition.” (Ref. 5.1)

The organic phosphorus compounds act as irreversible inhibitors of cholinesterase, thereby allowing the accumulation of large amounts of acetylcholine. When a critical level of cholinesterase depletion is reached, usually about 20% of normal, symptoms and signs of acetylcholine accumulation poisoning become manifest. Symptoms may include blurred vision, weakness, nausea, headache, abdominal cramps, chest discomfort, and diarrhea. Signs may include miosis, muscle twitching, salivation, sweating, tearing, cyanosis, convulsions, and coma. (Ref. 5.1)

Besides being absorbed following inhalation or ingestion, organophosphorus pesticides are readily absorbed through the intact skin (Ref. 5.1). When a particular pesticide has a low dermal LD_{50}, a skin notation should be added to the TLV or PEL.

Propetamphos has an acute oral LD_{50} of 119 mg/kg for male rats and an acute dermal LD_{50} of 2300 mg/kg for male rats. (Ref. 5.2)

By comparison with other organophosphorus insecticide 0.2 mg/m^3, without a skin notation was used in this evaluation.

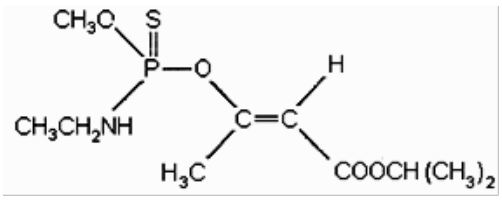
1.1.3 Potential workplace exposure

No estimate of worker exposure to propetamphos could be found. Propetamphos is used as an insecticide. (Ref. 5.2)

1.1.4 Physical properties (Ref. 5-2 - 5.4)

Molecular weight:	281.31
Molecular formula:	$C_{10}H_{20}NO_4PS$
CAS #:	31218-83-4
Boiling point:	87-89 °C
Appearance:	yellowish oily liquid
Solubility:	practically insoluble in water soluble in most organic solvents
Synonyms:	Safrotin, SAN 52139 I, Vel4283
Chemical name:	(E) 0-2-isopropoxy-carbonyl-l-methyl vinyl 0-methyl ethylphosphoramido thioatc

Structure:



1.2 Limit defining parameters

The detection limit of the analytical procedure is 0.013 ng per injection This is the amount of analyte which will give a peak whose height is approximately five times the baseline noise. This detection limit takes into account a split ratio of 32 to 1 used in the capillary GC.

2 Sampling Procedure

2.1 Apparatus

2.1.1 A personal sampling pump that can be calibrated to within ±5% of the recommended flow rate with the sampling device in line.

2.1.2 A Glass Fiber Filter, 37-mm diameter, Gelman type A or equivalent.

2.1.3 A Cassette filter holder for 37-mm filter, Millipore M000037A0, or equivalent. (See Figure 1.)

2.2 Reagents

No sampling reagents are required.

2.3 Sampling technique

2.3.1 Immediately before sampling, remove the plastic plugs from the cassette.

2.3.2 Attach the cassette to the sampling pump with flexible tubing.

2.3.3 Attach the cassette vertically in the employee's breathing zone in such a manner that it does not impede work performance.

2.3.4 After sampling for the appropriate time, remove the cassette and seal with plastic plugs.

2.3.5 Wrap each cassette end-to-end with a Form OSHA-21 seal.

2.3.6 Record the air volume for each sample, and list any possible interference.

2.3.7 Submit at least one blank for each set of sample. Handle the blank in the same manner as the sample, except no air is drawn through it.

2.3.8 Submit bulk samples for analysis in a separate container. Do not ship with Air sample.

2.4 Extraction efficiency

Three GFFs were each liquid spiked with 10 µL of a 1.41 mg/mL solution of propetamphos in toluene. They were then extracted with 3.0 mL of toluene and analyzed as in Section 3. The average recovery was 95.2%.

Table 2.4
Extraction Study

vial #	µg spiked	µg found	% recovered
1	14.10	13.76	97.6
2	14.10	12.60	91.0
3	14.10	12.29	96.9
4	0.00	0.00	blank

2.5 Retention efficiency

Six GFFs were each liquid spiked with 10 µL of a 1.41 mg/mL solution of propetamphos in toluene. After overnight storage in a drawer at room temperature, 60 liters of humid air were drawn through each GFF. Three of these GFFs, along with a blank GFF, were then extracted and analyzed as in Section 3. The average recovery was 90.3%. The remaining three spiked GFFs were used in the storage study.

Table 2.5
Retention Efficiency Study

sample #	µg spiked	µg found	% recovered
1	14.10	13.30	94.4
2	14.10	12.60	89.4
3	14.10	12.29	87.1
4	0.00	0.00	blank

2.6 Sample storage

The remaining three spiked GFFs from Section 2.5. (and a blank GFF) were stored for 8 days in a drawer at room temperature. They were then extracted and analyzed as in Section 3. The average recovery was 90.4%.

Table 2.6
Storage Study

sample #	µg spiked	µg found	% recovered
1	14.10	13.10	93.6
2	14.10	12.65	89.7
3	14.10	12.39	87.9
4	0.00	0.00	blank

2.7 Recommended air volume and sampling rate

2.7.1 The recommended air volume is 60 L.

2.7.2 The recommended flow rate is 1.0 L/min.

2.8 Interferences (sampling)

It is not known if any compounds will interfere with the collection of propetamphos. Suspected interferences should be reported to the laboratory with submitted samples.

2.9 Safety precautions (sampling)

2.9.1 Attach the sampling equipment in such a manner that it will not interfere with work performance or employee safety.

2.9.2 Follow all safety practices that apply to the work area being sampled.

3 Analytical Procedure

3.1 Apparatus

3.1.1 A GC with an ECD. A Hewlett-Packard 5890A GC (capillary) equipped with both an ECD and a Hewlett-Packard 7673A automatic sampler was used in this evaluation.

3.1.2 A GC column capable of resolving propetamphos from any interference. A 60-m x 0.32-mm i.d. (0.25-µm d_f SE-30) capillary column was used in this evaluation and is available from Supelco, Inc., Bellefonte, PA.

3.1.3 An electronic integrator or other suitable means of measuring detector response. A Hewlett-Packard 3392A Integrator and a Hewlett-Packard 3357 data system were used in this evaluation.

3.1.4 Vials, 4-mL and 2-mL glass with PTFE-lined septa.

3.1.5 Volumetric flasks, pipets, and syringes.

3.2 Reagents

3.2.1 Hydrogen, air, and nitrogen, GC grade.

3.2.2 Toluene, Pesticide grade.

3.2.3 Propetamphos. A 38.6% (w/w) solution in 1,3,5 trimethyl-benzene from the EPA was used in this evaluation.

3.3 Standard preparation

Prepare stock standards by adding toluene to preweighed amounts of propetamphos. Prepare working range standards by diluting stock solutions with toluene. Store stock and dilute standards in a freezer.

3.4 Sample preparation

3.4.1 Transfer the 37-mm glass fiber filter to a 4-mL vial.

3.4.2 Add 3.0 ml. of toluene to each vial.

3.4.3 Seal the vials with Polytetrafluoroethylene (PTFE) lined septa. Allow them to extract for one hour periodically shaking by hand.

3.4.4 If necessary, transfer aliquots of the samples to the vials used in GC analysis. In this evaluation, the samples were transferred to 2-mL glass vials, sealed with PTFE lined septa, and loaded on the automatic sampler.

3.5 Analysis

3.5.1 Analytical conditions

GC conditions

GC Column:	60-m x 0.32-mm i.d. (0.25-µm d_f SE-30)
Column temperature:	220 °C (Isothermal)
carrier gas:	Hydrogen
flow rate:	1.97 mL/min
split ratio:	32 to 1
retention time:	3.46 min

Injector conditions

temperature:	230 °C
volume:	1 µL

ECD conditions

Airflow rate:	100 mL/min
auxiliary gas:	Nitrogen
flow rate:	20 mL/min
temperature:	300 °C

3.5.2 Chromatogram (See Figure 2.)

3.6 Interferences (analytical)

3.6.1 Any compound having a retention time similar to that of the analyte is a potential interference. Generally, chromatographic conditions can be altered to separate interferences from the analyte.

3.6.2 Retention time on a single column is not proof of chemical identity. Analysis by an alternate GC column, flame photometric detector (FPD) and confirmation by mass spectrometry are additional means of identification.

3.7 Calculations

3.7.1 Construct a calibration curve by plotting detector response versus standard concentration.

3.7.2 Determine the concentration of propetamphos in samples and blank from the calibration curve. If propetamphos is found in the blank, make a blank correction.

3.7.3 Determine the air concentration by the following formula.

$$mg/m^3 = \frac{(\mu g/mL,\ blank\ corrected)(desorption\ volume,\ mL)}{(air\ volume,\ L)(desorption\ efficiency,\ decimal)}$$

3.8 Safety precautions (analytical)

3.8.1 Avoid exposure to all standards.

3.8.2 Avoid exposure to all solvents.

3.8.3 Wear safety glasses at all times.

4 Recommendations for Further Study

4.1 An OSHA Versatile Sampler (OVS-2) packed with XAD-2 adsorbent should be evaluated as a sampling device for propetamphos.

4.2 A fully validated method should be developed.

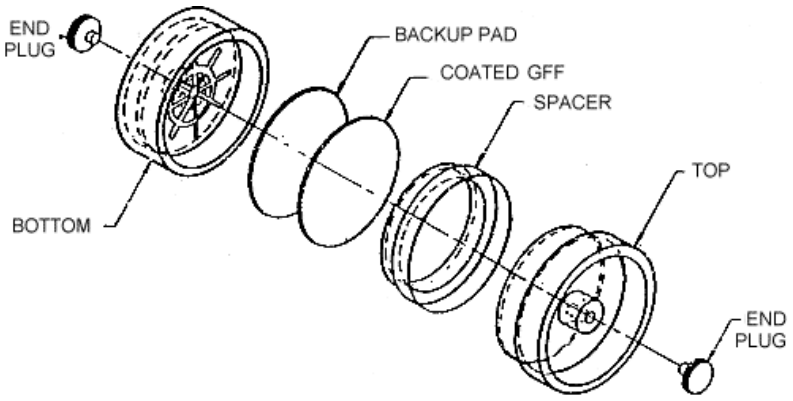


Figure 1. Sampling Device

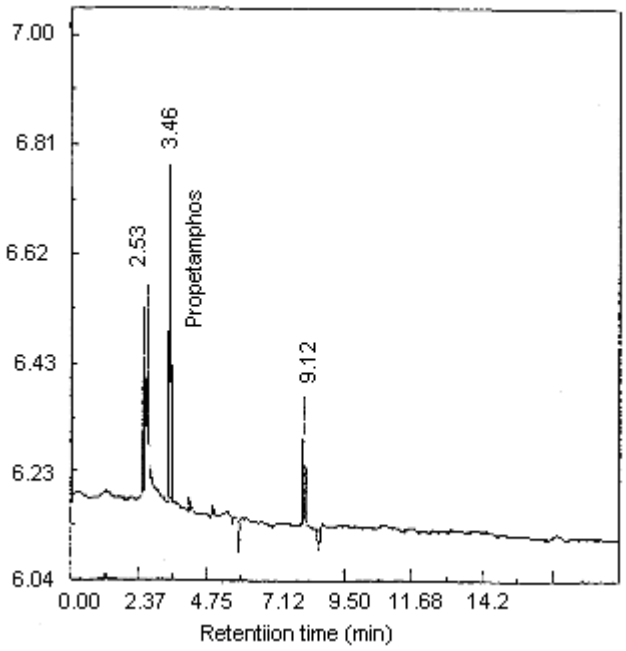


Figure 2. Chromatogram of propetamphos

5 References

5.1 "OCCUPATIONAL DISEASES, A Guide to their Recognition"; U.S. Department of Health, Education, and Welfare; Public Health Service, Public Health Service Publication No. 1097, U.S. Government Printing Office; Washington, D.C., 1964; p 245.

5.2 "Farm Chemicals Handbook"; Meister Publishing Co.; Willoughby, OH, 1985; p C195.

5.3 Windholz, M., Ed.; "Merck Index," 10th ed.; Merck and Co.; Rahway, NJ, 1983; p 1126.

5.4 Registry of Toxic Effects of Chemical Substances, 1978 Edition. (Lewis, R.J. and Tatken, R.L., Eds.) U.S. Department of Health, Education and Welfare, Public Health Services, Center for Disease Control, National Institute for Occupational Safety and Health, U.S. Government Printing Office, Washington, D.C. (1978).