



Propoxur (Baygon)

Method number: PV2007

Target Concentration: 0.5 mg/m³ ACGIH TLV. There is no OSHA PEL for propoxur.

Procedure: Samples are collected by drawing known volumes of air through OSHA versatile sampler (OVS-2) tubes, containing a glass fiber filter and two sections of XAD-2 adsorbent. Samples are extracted with acetonitrile and analyzed by high performance liquid chromatography (HPLC) using an ultraviolet (UV) detector.

Recommended air volume and sampling rate: 60 minutes at 1.0 L/min (60 L)

Detection limit of the overall procedure: 0.003 mg/m³ (Based on the recommended air volume.)

Status of Method: Partially Validated method. This method has been only partially evaluated and is presented for information and trial use.

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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 the OVS-2 sampling tube as a sampling device for propoxur. It follows the procedure developed for carbaryl. (Ref. 5.1)

It should be noted that in this evaluation for propoxur several other analytes were also present in the analytical procedure. These other analytes are not mentioned in this evaluation, but can be seen on the sample chromatogram. (Sec. 3.5.2)

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

The acute oral LD₅₀ for male rats is 83 mg/kg and the acute oral LD₅₀ for female rats is 86 mg/kg. The acute dermal LD₅₀ for both sexes of rats is >2400 mg/kg. (Ref. 5.2)

As with other carbamate pesticides, propoxur inhibits cholinesterase and this effect is apparently the basis of its toxic action. Carbamates are absorbed by inhalation, ingestion, and dermal penetration. (Ref. 5.2)

In a volunteer study, a single dose of 1.5 mg/kg produced mild symptoms, primarily gastrointestinal in nature, which disappeared within two hours of ingestion. The erythrocyte cholinesterase level reached a minimum of 27% of normal. (Ref. 5.2)

On the basis of the available toxicity data, propoxur has been given a TLV of 0.5 mg/m³ by the ACGIH. (Ref. 5.2)

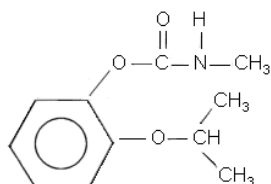
1.1.3 Potential workplace exposure

No estimate of workplace exposure was found. Propoxur is a widely used pesticide especially effective against insects affecting man and animals (i.e. cockroaches, flies, and mosquitoes). (Ref. 5.2)

1.1.4 Physical properties (Ref. 5.2 - 5.5)

CAS #:	114-26-1
Molecular weight:	209.24
Molecular formula:	C ₁₁ H ₁₅ N ₀₃
Melting point:	91.5 °C
Appearance:	white, crystalline, odorless solid
Solubility:	soluble in organic polar compounds, soluble to 0.2% in water at 20 °C
Synonyms:	apocarb, Bay 39007, Baygon, Propagon, Propygon, Suncide, Tygon Fliegenkugel, Unden
Chemical name:	2-1(1-methylethoxy) phenol methyl carbamate

Structure:



1.2 Limit defining parameters

The detection limit of the analytical procedure is 1 ng per injection. This is the amount of analyte which will give a peak whose height is approximately five times the baseline noise.

2 Sampling procedure

2.1 Apparatus

2.1.1 Samples are collected by using a personal sampling pump that can be calibrated to within $\pm 5\%$ of the recommended flow rate with the sampling device in line.

2.1.2 Samples are collected with OVS-2 tubes, which are specially made 13-mm o.d. glass tubes that are tapered to 6-mm o.d.. These tubes are packed with a 140-mg backup section and a 270-mg sampling section of cleaned XAD-2. The backup section is retained by two foam plugs and the sampling section is between one foam plug and a 13 mm diameter glass fiber filter. The glass fiber filter is held next to the sampling section by a polytetrafluoroethylene (PTFE) retainer. (See Figure 1.)

2.2 Reagents

No sampling reagents are required.

2.3 Sampling technique

2.3.1 Attach the small end of the OVS-2 sampling tube to the sampling pump with flexible, plastic tubing such that the large, front section of the sampling tube is exposed directly to the atmosphere. Do not place any tubing in front of the sampler. The sampler should be attached vertically (large end down) in the worker's breathing zone in such a manner that it does not impede work performance.

2.3.2 After sampling for the appropriate time, remove the sampling device and seal the tube with plastic end caps.

2.3.3 Wrap each sample end-to-end with a Form OSHA-21 seal.

2.3.4 With each set of samples, submit at least one blank. The blank should be handled the same as the other samples except that no air is drawn through it.

2.3.5 Bulk samples should be submitted for analysis in a separate container. Do not ship with the air samples.

2.4 Extraction efficiency

Two 13-mm glass fiber filters were each liquid spiked with 29.76 μg of propoxur. The two filters, along with a blank filter, were each placed in separate 4-mL vials which also contained 270 mg of XAD-2 adsorbent. These vials were stored overnight at room temperature, extracted with 2 mL of acetonitrile, and analyzed by HPLC.

The average extraction efficiency for these two filters (with the XAD-2 adsorbent present) was 94%.

2.5 Retention efficiency

Two OVS-2 tubes were each spiked with 29.76 μg of propoxur by liquid spiking the 13-mm glass fiber filter. Sixty liters of humid air were drawn through each tube. The two tubes were then

extracted as in Section 3.4. No significant breakthrough to the backup section was observed. The average retention efficiency for these two filters was 99%.

2.6 Sample storage

Two OVS-2 tubes were each spiked with 29.76 µg of propoxur as above. Sixty liters of humid air were drawn through each tube. These two tubes were stored for ten days at ambient temperature in a drawer. They were then extracted as in Section 3.4. No propoxur was recovered from the backup section of these tubes. The average recovery after ten days of storage was 90%.

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

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

2.9 Safety precautions

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 high-performance liquid chromatograph equipped with a UV detector, and manual or automatic injector. A Waters M6000A pump, Waters 710B autosampler, and Waters 440 UV detector with an extended wavelength module were used in this evaluation.

3.1.2 An HPLC column capable of separating propoxur from any interference. A 25-cm × 4.6-mm i.d. DuPont Zorbax ODS (6 µm) column was used in this evaluation.

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

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

3.1.5 Volumetric flasks, pipettes, and syringes for preparing standards, making dilutions, and performing injections.

3.2 Reagents

3.2.1 HPLC grade acetonitrile (ACN).

3.2.2 HPLC grade water. A Millipore Milli-Q system was used to prepare the water for this evaluation.

3.2.3 Propoxur, 99+% pure (EPA).

3.3 Standard preparation

Stock standard solutions are prepared by adding acetonitrile to preweighed amounts of propoxur. Working range standard solutions are prepared by diluting stock solutions with acetonitrile. Stock and dilute standards are stored in a freezer.

3.4 Sample preparation

3.4.1 Transfer the 13-mm glass fiber filter and the 270-mg section of the sampling tube to a 4-mL vial. Place the first foam plug and the 140-mg section in a separate 4-mL vial. A small glass funnel can be used to facilitate the transfer of the adsorbent. Discard the rear foam plug. Do not discard the glass sampling tube; it can be reused after it has been cleaned with surfactant or suitable solvent.

3.4.2 Add 2.0 mL of acetonitrile to each vial.

3.4.3 Seal the vials with PTFE-lined septa and allow them to extract for one hour. The vials should be shaken by hand periodically during the one-hour extraction time.

3.5 Analysis

3.5.1 Instrument conditions

Column:	25-cm × 4.6-mm i.d. stainless steel column, packed with 6 μm DuPont Zorbax ODS
Mobile Phase:	45% ACN / 55% water (v/v)
Flow rate:	1 mL/min
UV detector:	214 nm
Retention time:	5.9 min
Injection volume:	10 μL

3.5.2 Chromatogram (See Figure 2)

3.6 Interferences

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

3.6.2 Retention time on a single column is not proof of chemical identity. Analysis by an alternate HPLC column, detection at another wavelength, comparison of absorbance response ratios, and confirmation by mass spectrometry are additional means of identification.

3.7 Calculations

3.7.1 A calibration curve is constructed by plotting detector response versus standard concentration.

3.7.2 The concentration of propoxur in a sample is determined from the calibration curve. If propoxur is found on the backup section, it is added to the amount found on the front section. Blank corrections for each section should be performed before adding the results together.

3.7.3 The air concentration is then determined by the following formula.

$$\text{mg} / \text{m}^3 = \frac{(\mu\text{g} / \text{mL in sample})(\text{extraction volume, mL})}{(\text{air volume, L})(\text{extraction efficiency, decimal})}$$

3.8 Safety precautions

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

As propoxur is a common pesticide, this method should be fully validated.

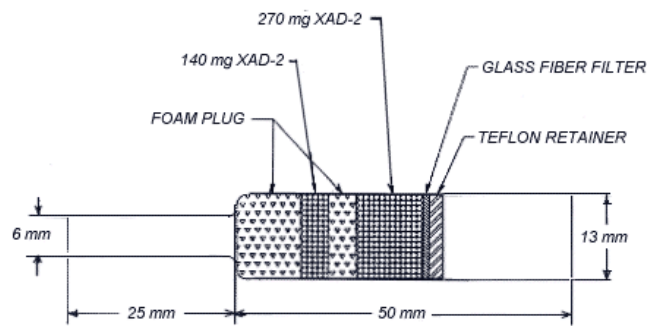


Figure 1. OVS-2 Sampling Device

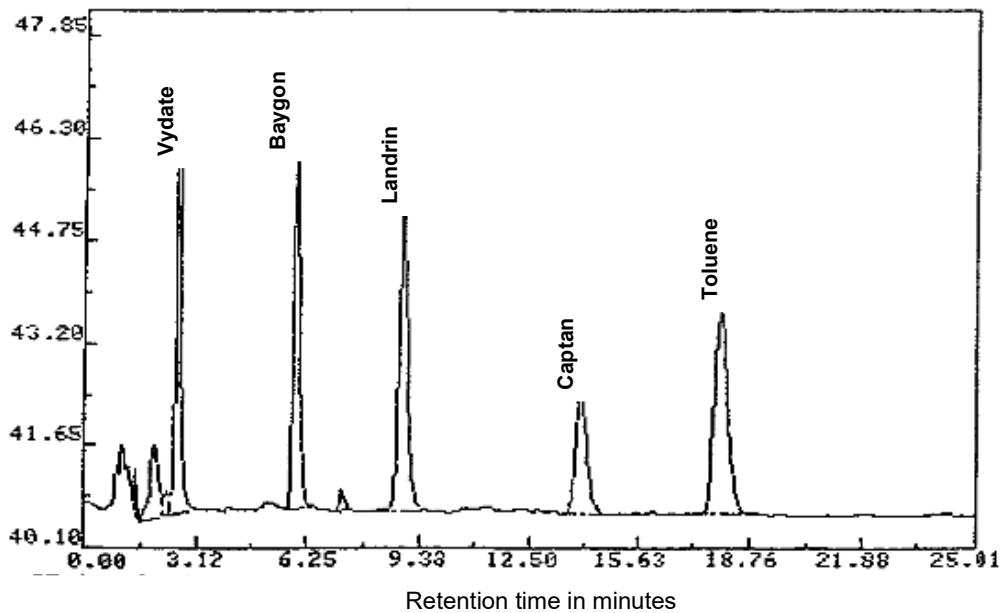


Figure 2. Chromatogram of Propoxur (with other analytes)

5 References

- 5.1 Burreight, D., Method #63, "Carbaryl (Sevin)", OSHA Analytical Laboratory, unpublished, 1987.
- 5.2 "Documentation of the Threshold Limit Values and Biological Exposure Indices," American Conference of Governmental Industrial Hygienists Inc., fifth edition, 1986.
- 5.3 Windholz, M., Ed. "Merck Index," 10th ed.; Merck and Co., Rahway, NJ, 1983.
- 5.4 "Farm Chemicals Handbook," Meister Publishing Co., 1985.
- 5.5 "Chemical Information File", U.S. Department of Labor, Occupational Safety and Health Administration, Directorate of Technical Support, June 14, 1985.