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Determination of 2-Isopropoxyphenyl Methyl Carbamate-Propoxur in Roach Control Peanut Butter by High Performance Liquid Chromatography

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Abstract

A method for the determination of 2-isopropoxyphenyl methylcarbamate, also known as Propoxur, in pesticides was developed for liquid chromatography equipped with a UV detector. Roach peanut butter sample was prepared with soxhlet extraction ten times; the standard solutions were prepared with n-butyrophenone and methanol as an internal standard, and both isopropoxyphenol (IPP) and 2-isopropoxyphenyl methylcarbamate (propoxur) as control standards. The target compound propoxur was well-separated and showed no interferences in the chromatograms. Propoxur was determined to comprise less than 2% of the sample.

Keywords: Propoxur; Baygon; Butyrophenone; 2-Isopropoxyphenol; Soxhlet extraction; High performance liquid chromatography

Introduction

Propoxur

Propoxur bait is a carbamate insecticide and was introduced by German chemical manufacturer Bayer in 1975. Propoxur is an N-methylcarbamate insecticide and acaricide. N-methyl carbamate pesticides are widely used for both agricultural and public health purposes, applied in the forms of spraying aerosols, dust and powders, pest strips, ready-to-use solutions, granular baits, and pet flea and tick collars. Propoxur is a non-systemic insecticide with a fast knockdown and long residual effect used against insect pests such as chewing and sucking insects, ants, cockroaches, crickets, flies, moths, and mosquitoes [1]. Propoxur is used extensively for hygienic purposes against such pests in homes, hotels, restaurants, and warehouses. Propoxur is highly toxic to many bird species and honeybees, and slightly toxic to aquatic species [2]. Agricultural crop applications include sugar cane, cocoa, grapes and other fruit, maize, rice, vegetables, cotton, Lucerne, forestry and ornamental [3] Propoxur is fairly soluble in water and very soluble in polar organic solvents, but only slightly soluble in non-polar organic solvents. It is hydrolyzed very slowly at pH 4, slowly at pH 7, but rather rapidly at pH 9 [3]. Propoxur decomposes at high temperature, forming methyl isocyanate (Danger! Very toxic compound) [4,5]. Propoxur is a white, crystalline, odorless solid. Solid propoxur can exist in two crystal forms (modifications I and II) but the technical material usually contains 0.95% of modification I. Propoxur is a chemical compound (Figure 1).

Materials and Experimental

Apparatus

- Soxhlet Extractor apparatus using a 250 mL round flask in a heating mantle and a Variac to control the temperature.
- Condenser, 30 cm long.
- Sartorius balance, hot-plate, glass beads.
- Cellulose extraction thimbles, 30 × 100 mm.
- Filters, 0.45 µm porosity, Acrodisc-CR (Gelman catalog # 4219, or equivalent). Syringe for pushing solution through filter.
- Rotary Evaporator- Buchi Rotavapor Model R110 equipped with a 50-62°C water bath and an aspirator-type vacuum system.
- Small chromatography type vials, 2 mL volume.
- HPLC-Water model # 6000A equipped with the following-Column: Phenomenex Prodigi ODS (3) 250 × 4.6 mm, 5 µ, 100A; Part Number 00G-4097-E0.
- Mobile phase: 80% Methanol/20% glacial acetic acid, pH 3, trace isopropyl alcohol.
- Detector: Tunable UV detector, Kratos Model SFA 339 set at 280 nm.
- System parameters: 1.010AU Full scale; Atten: 2, Digitize 6 min, temperature 25°C.

Reagents

Internal standard (IS): n-Butyrophenone, 99.0% was obtained from Sigma-Aldrich, Lot#SHBG6215V. 3.00 g was dissolved in a 100 mL volumetric flask and diluted up to volume with methanol.

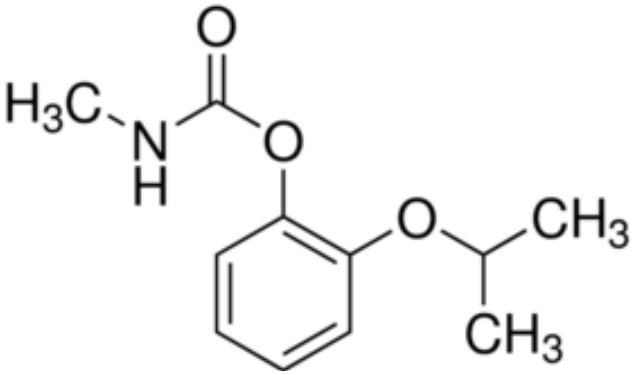

Figure 1: Structure of Propoxur.

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Reference standard: Propoxor (Baygon) was obtained PIC Corporation, 99.77%, Batch#140785914 (kept in frigidaire). 0.30 to 0.32 ± 0.0001 g of Baygon standard was weighed and transferred into a 50 mL volumetric flask. 10 mL internal standard solution was added and made up to volume with methanol.

Reference standard: 2-Isopropoxyphenol (IPP), 97.0% was obtained from Alfa-Aesar, Lot#10115127. 0.0030 g IPP was dissolved in a 100 mL volumetric flask and added to 20 mL internal standard solution and quantitatively filled with methanol.

- **Purified water:** Water was purified with a Milli Q System (Millipore, Milford, MA).
- **Methyl alcohol:** HPLC grade methanol was obtained from Merck.
- Acetic acid, glacial: Sigma-Aldrich, ≥ 99.0%, lot# SHBG5375.
- Sodium acetate, anhydrous: ACS, 99.0%, Alfa-Aesar, lot# Q18C027.
- **Isopropyl alcohol:** Sigma-Aldrich, USP Chemical, lot# SHBG8515V.

Mobile phase: 200 mL glacial acetic acid was mixed with 800 mL HPLC grade methanol and the pH was adjusted to 3.0 (using a pH meter) by adding sodium acetate crystals and mixing. 3-5 mL isopropyl alcohol was added and the solution was then filtered on 0.45 µm cellulose nitrate filter paper using a vacuum aspirator. After the filtrate was transferred to a mobile phase container, glass beads were added and degassed under vacuum just prior to use.

Procedure

Sample preparation

For Baygon 2.0% Bait, about 13.5-14.5 ± 0.01 g of peanut butter roach control sample was weighed into a 25-mm extraction thimble and placed in a 30-mm ID Soxhlet extractor. Boiling stones and 150 mL of methyl alcohol were placed into a 250 mL extractor flask, connected to the extractor, and extracted using 10 to 15 cycles (about 2 hours) [5]. After cooling, the flask was then disconnected from the extractor and the contents were stripped to an oil using a rotary evaporator. 40 mL of methyl alcohol were added into the flask and swirled around to dissolve the analyte, resulting in a viscous oil coating the glassware.

The solution was transferred to a 250 mL evaporation flask. The original flask was rinsed with methanol and the rinse was added to the evaporation flask. The content was stripped to a consistency of viscous oil that coated the flask and was evaporated until a solid began to crystallize. About 30-40 mL methanol was added to dissolve the oil-like propoxur into a solution and transferred into a 50 mL volumetric flask. 10 mL of Internal Standard solution was added before making up the volume to 50 mL.

All samples were filtered through a 0.45 µm porosity filter and the filtrate was collected in a vial. 1.0 mL methanol was added into two separate 2 mL injection vials using a volumetric pipette. 150 µL sample solution and 150 µL propoxur standard were added into each vial.

Instrument setup

Set up the HPLC detector at 280 nm. Check the pressure at the pump by passing degassed mobile phase for a few minutes and zeroing the detector's absorbance. Check for stability.

Injections

Inject the standard solution twice. Record the peak areas of the internal standard and IPP as I and S, respectively. Compare the ratio of Propoxur and internal standard peak areas for the two standard injections. If the ratios agree within 2.0% ± 0.1% of the average of the two (Rave), proceed with the analysis. If not, reinject the standard solution until two successive injections do agree.

Inject the sample in duplicate under the same conditions as the standard. Record the peak areas of Propoxur, IPP, and internal standard in the sample as A, A', and B, respectively. Calculate the percentage of Propoxur in the standard and in IPP. If the two values agree within 2.0% ± 0.1% of their average, report the average and the results. Otherwise, reinject the sample until this requirement is met.

Make a standard injection after every two samples (every four injections) and one more at the end of the entire run. For every standard injection, the conditions must be met, otherwise analysis must be repeated.

Results and Discussion

Equations

$$R = \frac{S}{I}$$

S=Standard peak area; I=Internal Standard peak area.

$$K = \frac{W_s \times P}{R}$$

W_s=Weight of standard (g); P=Purity of standard (%)

$$\text{Propoxur\%} = \frac{A \times K}{B \times W_{sample}}$$

A=Peak area of Propoxur standard in sample (A' for IPP); B=Peak area of internal standard in sample; W=Weight of sample (g).

Blank-methyl alcohol with internal standard (n-butyrophenone) was injected in HPLC. No interfering peak was found in the blank, which showed one peak at the retention time (3.437 mins) of the internal standard. Chromatogram is shown in Figure 2.

Figure 2: Blank with IS in 100 mL methanol.

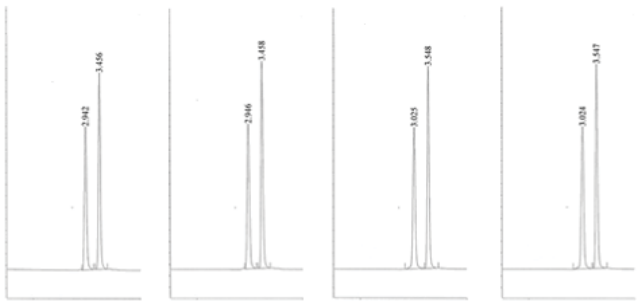

Figure 3: Propoxur control standard. 0.049 g in 50 ml with IS and methanol.

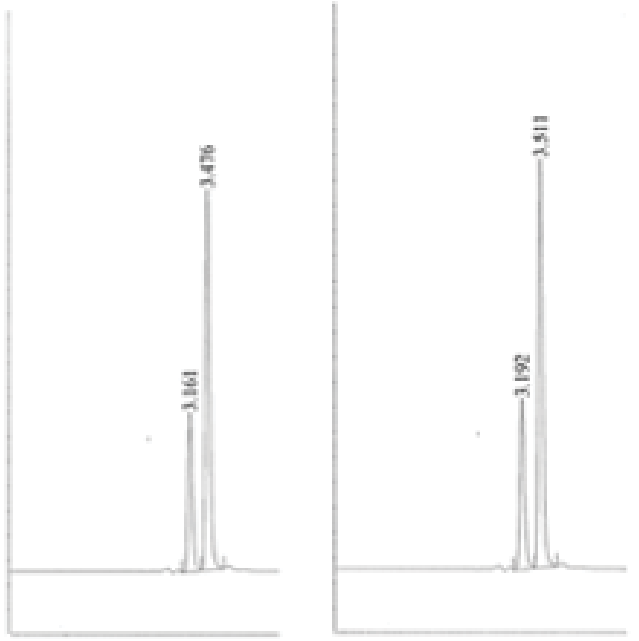

Figure 4: IPP control standard, 0.302 g in 50 ml with IS and methanol.

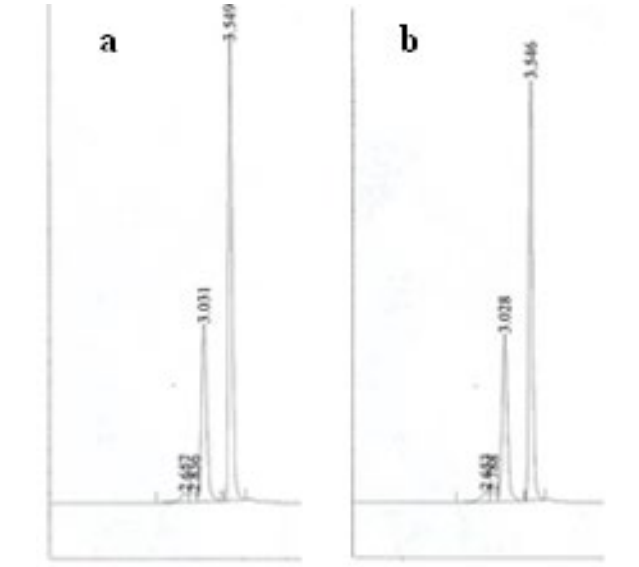

Figure 5: (a) Propoxur (Baygon) sample, 13.00680 g in 50 mL. (b) Propoxur (Baygon) sample duplicate, 13.09110 g in 50 mL.

Propoxur control standard with the IS was injected four times (Figure 3). The Propoxur standard peak retention times were at 2.942, 2.046, 3.025, and 3.024 minutes. R for each injection was found to be 0.866, 0.859, 0.867, and 0.868, respectively.

IPP control standard was injected in duplicate and had a peak before the IS (Figure 4). The IPP standard peak retention times were at 3.161 and 3.192 minutes. R for each injection was found to be 0.421 and 0.422, respectively.

Roach control peanut butter sample (propoxur) was injected in duplicate (Figures 5a and 5b). Average percent of propoxur in the sample was found to be 1.41% (w/w) and 1.48% (w/w) with a standard deviation of 0.051 and %RSD of 3.5. Average percent of IPP in roach control peanut butter sample was found to be 0.0024% (w/w) with standard deviation of 0.00005 and %RSD of 2.0. The results are within specification, which is 2% (w/w) maximum [5].

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