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Note

High-performance liquid chromatographic analysis of the photodegradation products of phosmethylan

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The influence of light is one of the most important factors that control the fate and persistence of pesticides in the environment. In order to understand the complex photodegradation pathway, we investigated the photodecomposition of O,O-dimethyl-S-[N[(*o*-chlorophenyl)butyroamido]methyl]dithiophosphate (phosmethylan)^{1,2} in dilute aqueous solution exposed to UV light. Several products were isolated and identified³. In this paper is reported a reliable, rapid high-performance liquid chromatographic (HPLC) method for the measurement of the photodegradation products.

EXPERIMENTAL

Standards

N-Hydroxymethyl-*o*-chlorobutyroanilide, *o*-chlorobutyroanilide and *o*-hydroxybutyroanilide were synthesized, 2-amino-3-chlorobutyrophenone and 4-amino-5-chlorobutyrophenone were prepared by thin-layer chromatography and *o*-chloroaniline was supplied by EGA Chemie.

Model system

A 70-l volume of distilled water containing 25 mg/kg of phosmethylan (using 50% emulsion concentrate) was exposed to UV light while fresh air was bubbled through continuously. The light source used was a Tungfram, 15 W, F-Germicid Y 16.

Extraction

A 100-ml volume of water was extracted with dichloromethane and, after evaporation to dryness, the residue was dissolved in *n*-hexane (1 ml).

High-performance liquid chromatography

An ALC 201 high-performance liquid chromatograph equipped with a refractive index (RI) detector was used. *n*-Hexane samples (20 μ l) were injected on to a 10 cm \times 4.6 mm I.D. Nevisorb column (10 μ m). The mobile phase was *n*-hexane-dioxane (97:3) (25% saturated with water), the flow-rate was 1.7 ml/min and the column temperature was 25°C. The attenuation was 8 \times .

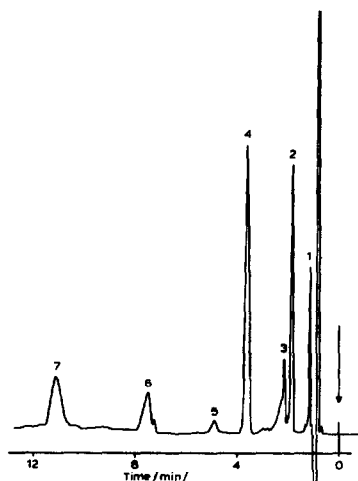


Fig. 1. HPLC trace of phosmethylan and its photodecomposition products in water. Peaks: 1 = 2-amino-3-chlorobutyrophenone; 2 = *o*-chlorobutyroanilide; 3 = *o*-chloroaniline; 4 = phosmethylan; 5 = 4-amino-5-chlorobutyrophenone; 6 = N-hydroxymethyl-*o*-chlorobutyroanilide; 7 = *o*-hydroxybutyroanilide.

RESULTS

A typical chromatogram of a water extract 18 days after treatment with phosmethylan and exposition to UV light (2 h every day) is illustrated in Fig. 1. The structural identification of the photodecomposition products was performed by direct mass spectrometry. Different eluent systems were tried [dioxane-*n*-hexane (10:90), *n*-hexane-dioxane-dibutyl ether (86:7:7), *n*-hexane-dichloromethane-isopropyl alcohol (85:15.4:0.5), *n*-hexane-dioxane-acetic acid (80:19:1), dichloromethane-*n*-hexane (70:3), *n*-hexane-dioxane (97:3) saturated with water] and the best resolution was obtained with *n*-hexane-dioxane (97:3). To prevent tailing and for complete separation, the eluent was 25% saturated with water.

There were no major interfering compounds in the extracts. The limits of detection were 1.5 μg for the parent compound and 0.3, 0.65, 0.6, 0.7, 3.0 and 1.6 μg for 2-amino-3-chlorobutyrophenone, *o*-chlorobutyroanilide, *o*-chloroaniline, 4-amino-5-chlorobutyrophenone, N-hydroxymethyl-*o*-chlorobutyroanilide and *o*-hydroxybutyroanilide, respectively.

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