FIELD APPLICATION REPORT

LIQUID CHROMATOGRAPHY

Analysis of Carbamates by HPLC – U.S. EPA Method 531.1

Authors: Vivek Dhyani

PerkinElmer, Inc. 710 Bridgeport Avenue Shelton, CT 06484

Introduction

In most countries, growing concern about the residues of pesticides in drinking water and food products is evident. Therefore, regulations limiting the concentration of pesticides in water and foodstuffs have been introduced to protect consumers from contaminated food products. Several methods are used to control these limits. HPLC is recommended for the analysis of low-volatile compounds and for compounds that are unstable when heated. **U.S. Environmental Protection Agency (EPA) Method 531.1** describes the HPLC method for the determination of carbamates in water. In this method, carbamates separated by HPLC are subjected to hydrolysis, using NaOH solution. The methylamine formed during hydrolysis is reacted with o-phthalaldehyde (OPA) and 2-mercaptoethanol to form a highly fluorescent derivative which is detected by a fluorescence detector.

Experimental

The analysis was carried out using the following conditions:

Instrument: PerkinElmer[®] Series 200 HPLC System – Pump, Autosampler and Fluorescence Detector Derivatization: Using in-line Pickering PCX5200 Post-Column Derivatization System (Pickering Laboratories, Inc.) Column: PerkinElmer Brownlee[™] Validated C-8 column, 250 X 4.6 mm, 5 µm Mobile Phase: Water/methanol gradient: (analysis time of 51 minutes with 14 minute equilibration between injections)

- Step 1: 2 minutes at 85% water
- Step 2: 40 minute linear gradient from 85% to 30% water
- Step 3: Hold at 30% water for 4 minutes
- Step 4: Step to 100% methanol; curve 0
- Step 5: Hold at 100% methanol for 5 minutes

Flow Rate: 0.8 mL/min

Temperature: 37 °C

Detector: Fluorescence Detector, with 330 nm Excitation and 465 nm Emission Injection Volume: 10 µL of 200 ppm standard



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Results

The chromatogram shown in Figure 1 below is a mixture of 12 carbamates with labeled components.

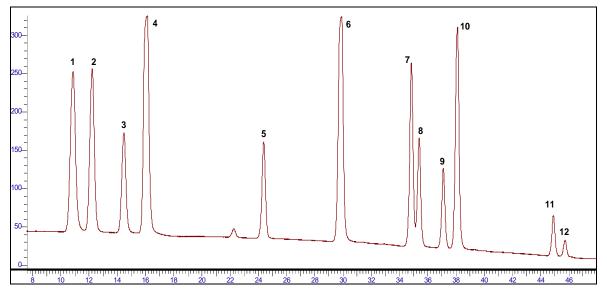


Figure 1. Peak List: 1. Aldicarb sulfoxide; 2. Aldicarb sulfone; 3. Oxamyl; 4. Methomyl; 5. 3-Hydroxy carbofuran; 6. Aldicarb; 7. Propoxur; 8. Carbofuran; 9. Carbaryl; 10. 1-Naphthol; 11. Methiocarb; 12. BDMC.

Conclusion

This application brief describes a simple, fully-automated method for the analysis of carbamates using HPLC with a Fluorescence Detector and the Pickering Post-Column Derivatization System.

PerkinElmer, Inc. 940 Winter Street Waltham, MA 02451 USA Phone: (800) 762-4000 or (+1) 203-925-4602 www.perkinelmer.com



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