



Expanded HPLC Method for N-Methyl Carbamates

Carbamate pesticides are widely used around the world to protect crops. In addition, they are used as biocides for industrial or other applications and in household products. Though carbamates are biodegradable compounds and bioaccumulation usually happens only to a low extent it is important to monitor produce to make sure enough time has elapsed between applying pesticides and harvest. Also, because of their high solubility, carbamates can leach into ground waters in porous soils and consequently find their way into drinking water supplies.

As part of FDA's pesticide monitoring program individual lots of domestic and imported foods and feeds are sampled and tested for pesticide residues to enforce the tolerances set by EPA. There are 11 compounds mandated by USEPA Method 531.2 for drinking water but they represent only a fraction of the carbamates that require monitoring in domestic and imported products. Methyl carbamates are separated using a reversed-phase column and then made to react with o-phthalaldehyde and a mercaptan after hydrolysis to form highly fluorescent compounds. This post-column reaction is the basis for official EPA Method 531.2 and AOAC Method 985.23.

This new expanded method is suitable for detecting a wide range of carbamates; post-column derivatization with fluorescence detection is a sensitive and selective method for residue analysis in water, food and feed samples. This method employs the same HPLC and post-column equipment and chemicals as USEPA Method 531.2 and will allow laboratories to increase the range of tested compounds.

ACKNOWLEDGEMENTS:

Pickering Laboratories would like to thank John Casanova and Frank Schenck (FDA) for their help with this project.

Post-column Analysis of Pesticides in Food and Potable Water Samples

The separation is achieved on a C₈ stationary phase with a water/Methanol gradient. Differences in selectivity of a water/Acetonitrile gradient may be used for confirmation.

METHODS

Analytical Conditions

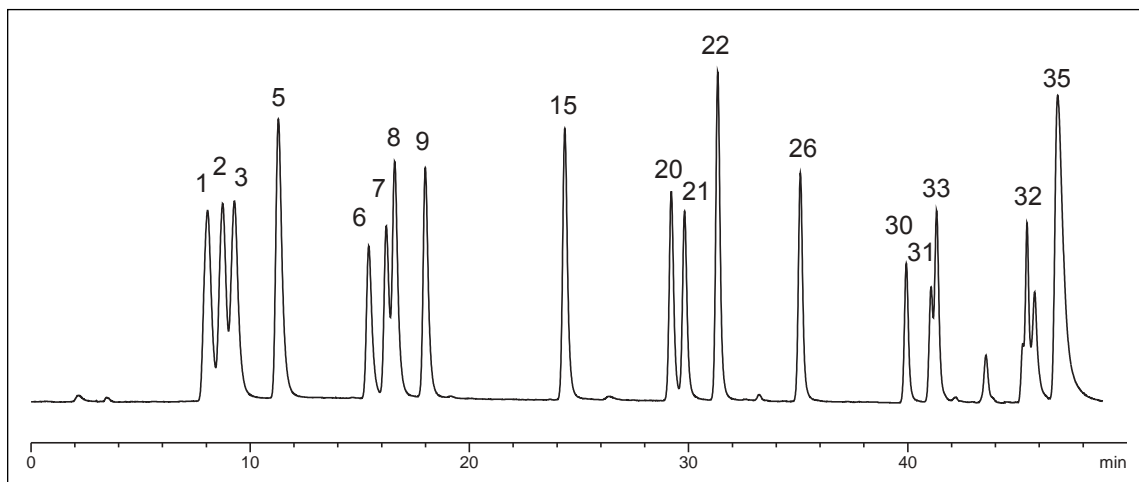
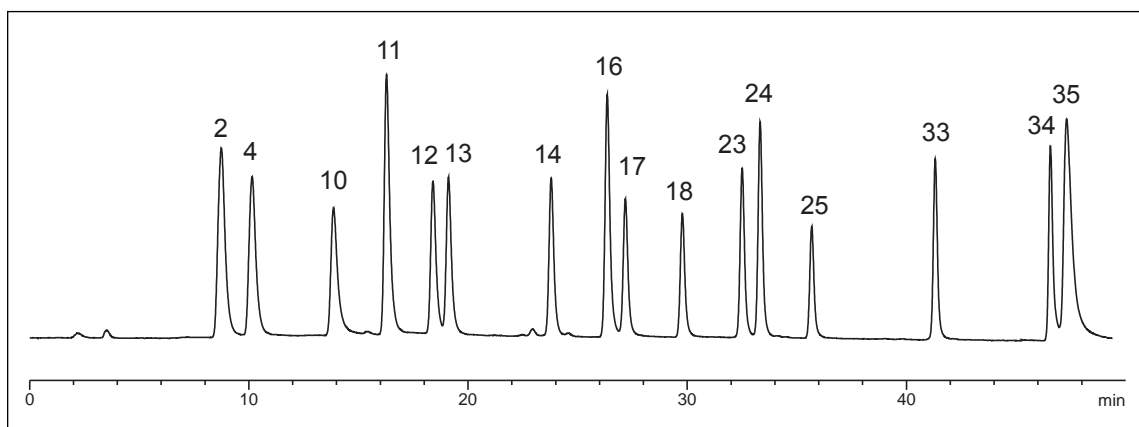
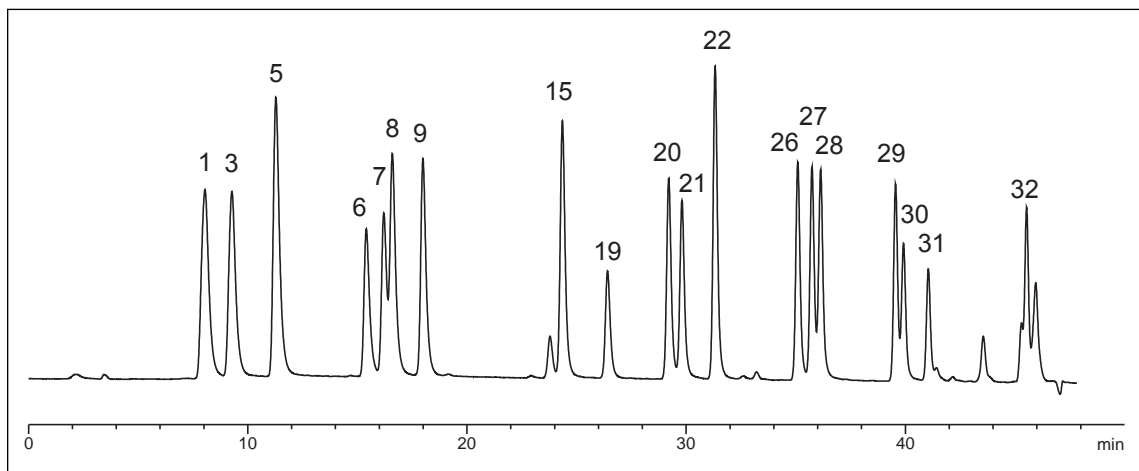
| | |
|---------------------|--|
| COLUMN: | Expanded resolution C ₈ analytical column 4x250 mm, P/N 0840250 Guard column P/N 18ECG001 |
| FLOW RATE: | 0.8 mL/min |
| COLUMN TEMPERATURE: | 45° C |
| MOBILE PHASE: | Water/Methanol |

Post-column Conditions

| | | | |
|----------------------|---|-----------|--------------|
| POST-COLUMN SYSTEM: | Pinnacle PCX or Vector PCX | | |
| REACTOR VOLUME: | 0.5 mL | | |
| REACTOR TEMPERATURE: | 100° C | | |
| REAGENT 1: | CB130 | | |
| REAGENT 2: | 100 mg of OPA and 2 g of Thiofluor in 950 mL of CB910 Diluent | | |
| REAGENTS FLOW RATE: | 0.3 mL/min | | |
| DETECTION: | Fluorescence detector λ_{ex} = 330 nm λ_{em} = 465 nm | | |
| HPLC GRADIENT: | Time (min) | Water (%) | Methanol (%) |
| | 0 | 85 | 15 |
| | 2 | 85 | 15 |
| | 42 | 30 | 70 |
| | 46 | 30 | 70 |
| | 46.1 | 0 | 100 |
| | 50 | 0 | 100 |
| EQUILIBRATION: | 10 min | | |

continued on back

Separation of the carbamates standard mixtures provided by FDA



Separation of the carbamates standard mixtures provided by FDA.

List of Carbamates

| | | | |
|------------------------|---------------------------|----------------------|-----------------------|
| 1 Aldicarb sulfoxide | 9 Thiofanox sulfone | 18 Bendiocarb | 27 2,3,5-Trimethacarb |
| 2 Butoxycarboxim | 10 Formetanate HCl | 19 Carbetamide | 28 3,4,5-Trimethacarb |
| 3 Aldicarb sulfone | 11 Ethiofencarb sulfoxide | 20 Propoxur | 29 Fenobucarb (BPMC) |
| 4 Oxamyl | 12 Dioxacarb | 21 Carbofuran | 30 Methiocarb |
| 5 Methomyl | 13 3-Hydroxycarbofuran | 22 Carbaryl | 31 BDMC |
| 6 Ethiofencarb sulfone | 14 Butocarbocoxim | 23 Ethiofencarb | 32 Bufencarb |
| 7 Ethidimuron | 15 Aldicarb | 24 Thiofanox | 33 Promecarb |
| 8 Thiofanox sulfoxide | 16 Metolcarb | 25 Banol | 34 Zectran |
| | 17 Cloethacarb | 26 Isoprocarb (MIPC) | 35 Aminocarb |