Expanded HPLC Method for N-Methyl Carbamates

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

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.

The separation is achieved on a C_8 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

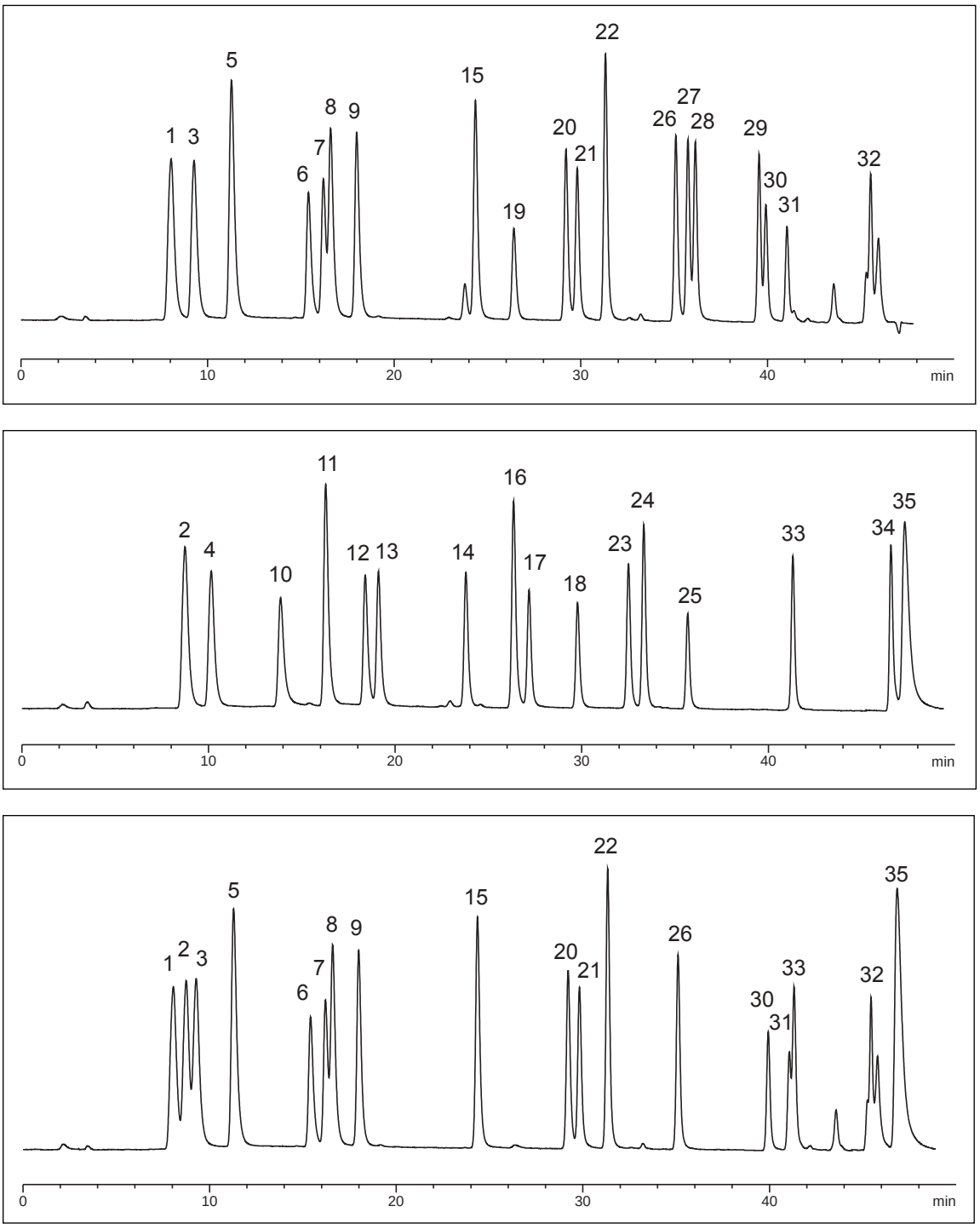
Post-column Conditions

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

1280 Space Park Way • Mountain View CA 94043 • USA
800-654-3330 • 650-694-6700 • 650-968-0749 FAX
www.pickeringlabs.com • support@pickeringlabs.com

Separation of the carbamates standard mixtures provided by FDA



Separation of the carbamates standard mixtures provided by FDA.

List of Carbamates

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