

Post-Column LC Analysis of 23 N-Methyl Carbamates

An Expanded Method Using Water/Acetonitrile or Water/Methanol Gradients

There are a number of carbamate pesticide compounds employed worldwide which are not included in the 10 compounds mandated by USEPA Method 531.1 and AOAC Protocol 29.A05.

This Application Note describes methods for separating as many as 23 compounds, using a Pickering C8, 5 μ m silica, column with either Water/Acetonitrile or Water/Methanol gradients. Instruments, post-column derivatization and detection protocols, and reagents remain unchanged, as described Pickering's brochure, *Post-Column LC Systems for Environmental Pesticide Analysis* (B-CA5) and User's Manual 0101-0002.

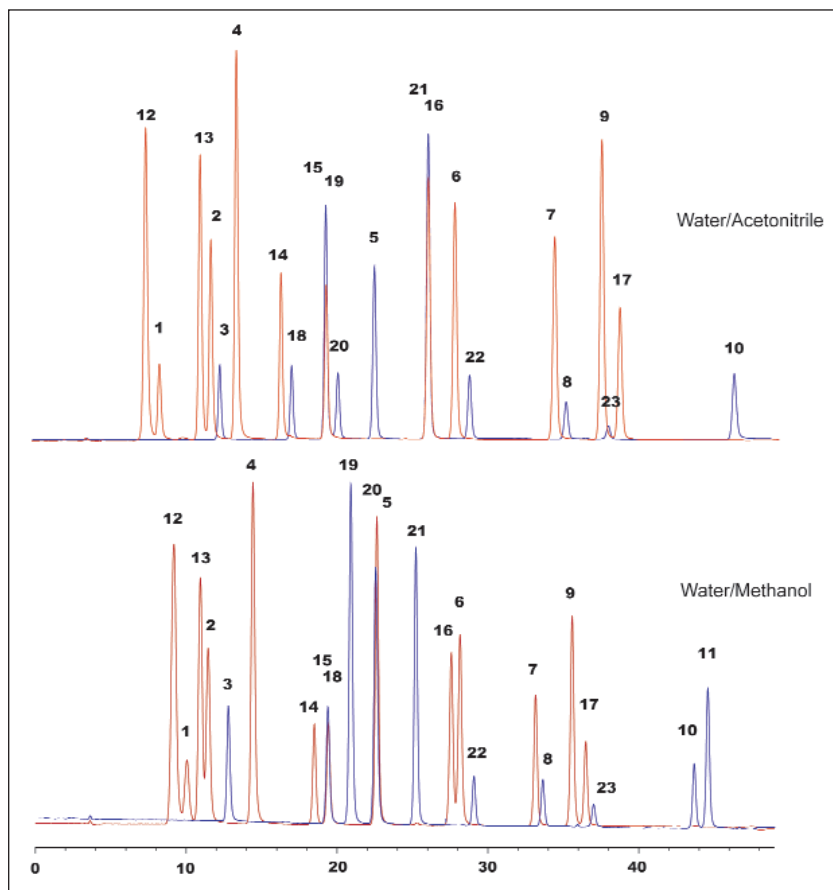
Although there are only two co-elutions in each of the solvent gradient protocols, the compounds were assigned to two separate test mixtures, A and B, in order to demonstrate the full range of separation.

The differences in selectivity between the Acetonitrile and Methanol protocols enable the user to employ both solvent systems as a means of confirming peak identification.

The following comparison of Acetonitrile to Methanol might be helpful in choosing which solvent system will be for used routinely, and which will be for confirmation:

- Acetonitrile generally exhibits higher sensitivity and baseline noise.
- It also costs more and its disposal is more restricted.

Eleven of the most widely occurring carbamate compounds and BDMC (internal standard) are supplied by Pickering Laboratories in its qualitative test mixtures, Cat. No. 1700-0063. These are designated by * in the test mixture tables below. Other compounds may be purchased from suppliers of environmental standards. A few may vary considerably in homogeneity and availability.



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Analytical Conditions: Water/Acetonitrile Protocol

COLUMN: 4.0 x 250 mm, Pickering Cat.No. 0840250
SAMPLE INJECTION: 10 µl in MeOH (for all chromatograms shown)
MOBILE PHASE: Solvent A: HPLC-grade Water
Solvent B: HPLC-grade Acetonitrile (MeCN)
PROGRAM:

Time	%H ₂ O	%MeCN	Gradient
0	90	10	
2	90	10	isocratic
46	49	51	linear
46.1	30	70	step
49	30	70	isocratic

EQUILIBRATION TIME: 13 minutes
ELUANT FLOW RATE: 0.80 mL/minute
COLUMN TEMPERATURE: 37 °C
REAGENT 1: 0.3 mL/minute, CB130 Hydrolysis Reagent
REAGENT 2: 0.3 mL/minute, OPA reagent¹ for derivatization
REACTORS: Hydrolysis: 100 °C, 500 µL
Derivatization: Ambient, 100 µL
FLOURESCENCE DETECTION: Excitation: 330 nm
Emission: 465 nm

Analytical Conditions: Water/Methanol Protocol

MOBILE PHASE: Solvent A: HPLC-grade Water
Solvent B: HPLC-grade Acetonitrile (MeOH)
PROGRAM:

Time	%H ₂ O	%MeCN	Gradient
0	88	12	
2	88	12	isocratic
42	34	66	linear
46	34	66	isocratic
46.1	0	100	step
49	0	100	isocratic

Column: 4.0 x 250 mm C8
Inject: 10ml test mix in methanol

- 1 Aldicarb sulfoxide (Standak)
- 2 Aldicarb sulfone
- 3 Oxamyl (Vydate)
- 4 Methomyl (Lannate)
- 5 3-Hydroxy carbofuran
- 6 Aldicarb (Temik)
- 7 Propoxur (Baygon)
- 8 Carbofuran (Furadan)
- 9 Carbaryl (Sevin)
- 10 Methiocarb (Mesurol)
- 11 BDMC internal standard
- 12 Butocarboxim sulfoxide
- 13 Butocarboxim sulfone
- 14 Ethiofencarb sulfoxide
- 15 Ethiofencarb sulfone
- 16 Butocarboxim
- 17 Ethiofencarb
- 18 Thiofanox Sulfoxide
- 19 Thiofanox Sulfone
- 20 Methiocarb sulfoxide
- 21 Methiocarb sulfone
- 22 3-Ketocarbofuran
- 23 Thiofanox

¹ *o*-Phthalaldehyde (O120) and Thioflour™ (3700-2000), Pickering's brand of Dimethylaminoethanethiol•HCl, in OPA Diluent (CB910)

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