Applikationsbericht

Measurement of n-Methylcarbamoyloxime and n-Methylcarbamates in Water by Direct Aqueous Injection HPLC with Postcolumn Derivatization

Waters Corporation

This is an Application Brief and does not contain a detailed Experimental section.

Abstract

This Application brief demonstrates the measurement of n-Methylcarbamoyloxime and n-Methylcarbamates in water by direct aqueous injection HPLC with postcolumn derivatization.

Introduction

Carbamates are used worldwide as commercial pesticides for food crops. The resulting agricultural runoff can carry them into surface water, groundwater, and other drinking water resources. The United States Environmental Protection Agency (US EPA) requires that drinking water and raw surface water to be monitored for the presence of carbamate pesticides and related compounds using an established EPA Method 531.2. The European Union (EU) regulation regarding drinking water, provides a general rule for pesticides and metabolites. This regulation limits the maximum admissible concentration (MAC) at 0.1 μg/L (ppb) for each individual component, with the total concentration not to exceed 0.5 ppb. Detection of regulated compounds at ever decreasing levels is a challenge faced by many water testing laboratories. Because the concentration of these substances in real samples may be in the low parts per billion (ppb) or μg/L, optimal sensitivity is the ultimate goal.

Experimental

HPLC conditions

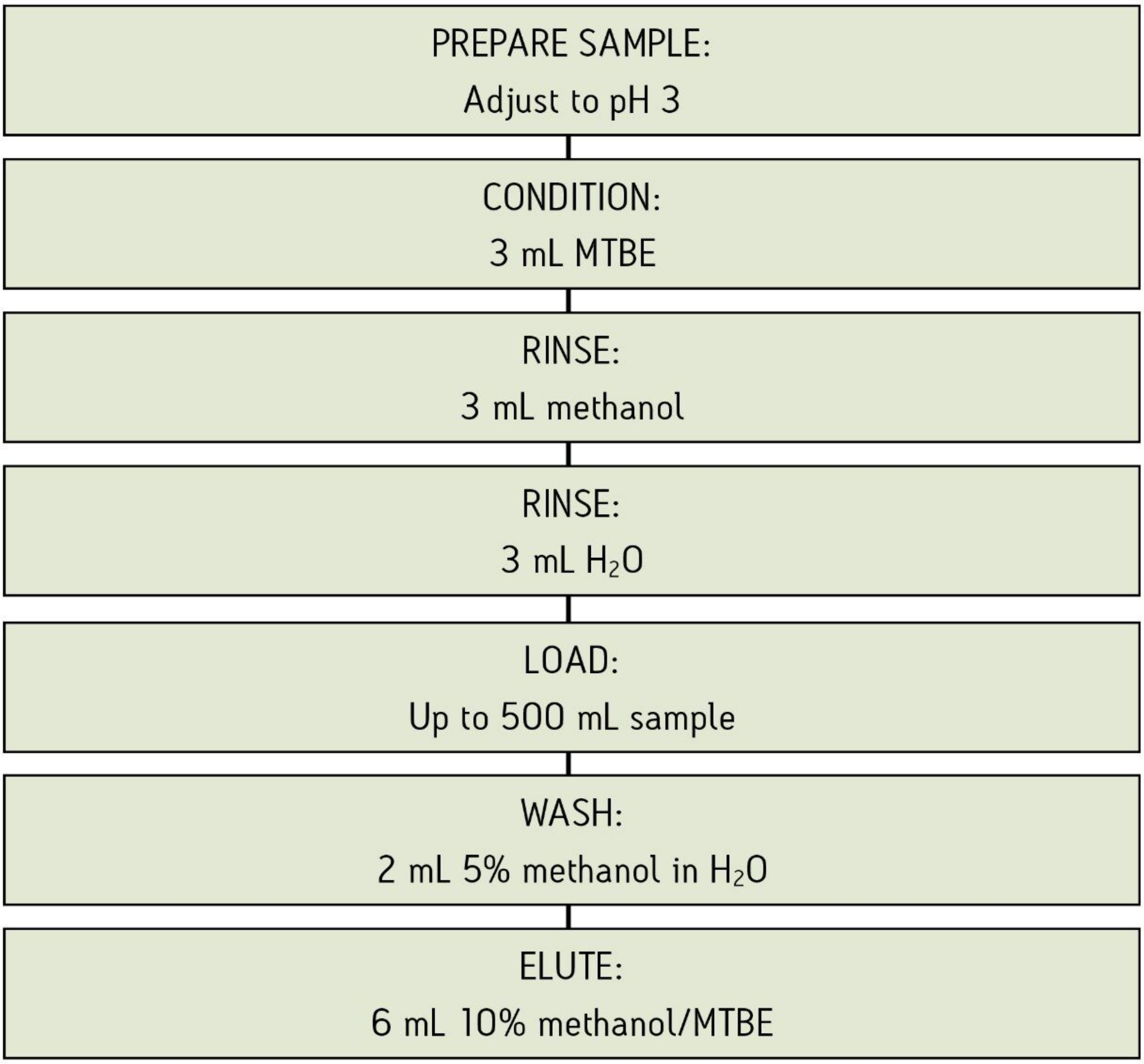
Instument:	Waters Alliance system for carbamate analysis
Eluent:	Water/methanol/acetonitrile
Column:	Waters Carbamate Analysis column 3.9 x 150 mm @ 30 °C

Injection:	400 µL of standard
Flow Rate:	1.5 mL/min
Detection:	Fluorescence, Ex- 339 nm, Em- 445 nm, Gain- 10
Data:	Waters Empower software

Sample Preparation

Oasis SPE Method for Carbamates

Method for Oasis HLB Cartridge, 6 cc, 200 mg



For LC analysis, exchange to acetonitrile, then adjust to 1 mL.

Standard mix preparation

Pipette 25 µL of AccuStandard mix M-531M and M-531-IS each into 100 mL of preserved reagent water for a final concentration of 25 parts per billion (ppb) per analyte. Preserved reagent water is prepared by dissolving 9.35 g of potassium dihydrogen citrate and 0.2 g of sodium thiosulfate (anhydrous) in water and diluting to 1 L.

Eluent Preparation

Filter and degas all eluents through a 0.45 µm filter.

A: Water

B: Methanol

C: Acetonitrile

Time	Flow	%A	%B	%C	Curve
Initial	1.5	88.0	12.0	0.0	-
5.30	1.5	88.0	12.0	0.0	1
5.40	1.5	68.0	16.0	16.0	5
14.00	1.5	68.0	16.0	16.0	3
16.10	1.5	50.0	25.0	25.0	7
20.00	1.5	50.0	25.0	25.0	6
22.00	1.5	88.0	12.0	0.0	5
30.00	1.5	88.0	12.0	0.0	1

Eluent gradient

Post Column Reagent Preparation

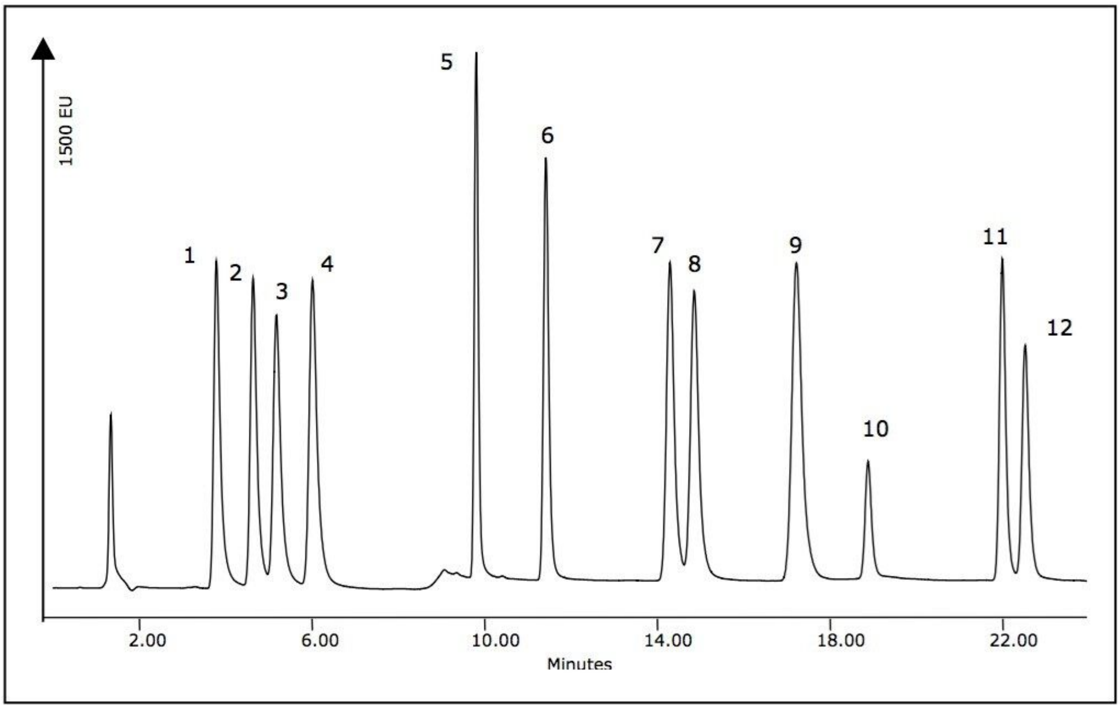
Reagent 1: 0.05 N sodium hydroxide

Dissolve 2 g NaOH in water and dilute to 1 L, filter and degas.

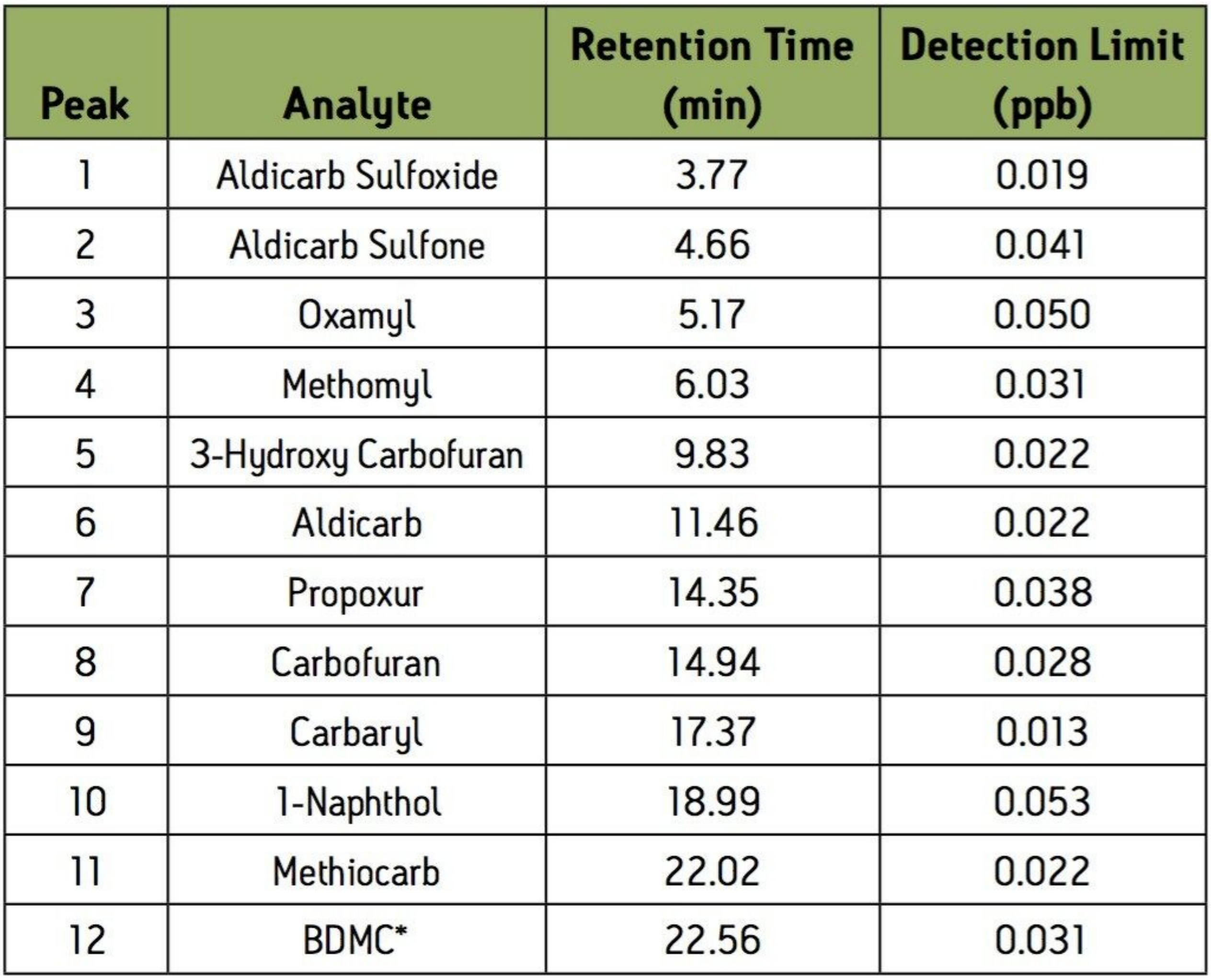
Reagent 2: PA/2-mercaptoethanol

Dissolve 19.1 g of Borax ($Na_2B_4O_7 \cdot 10H_2O$) Sodium borate in 1 L of water. To this, add a solution of 0.1 OPA (o-phthaldialdehyde) in 10 mL methanol. Swirl to dissolve, filter, and degas. To this, add 1.0 mL of 2-mercaptoethanol, ently swirl to dissolve. Protect from light. Post-column reaction temperature is 80 °C.

Results and Discussion



Standard chromatogram of 25 ppb for each analyte.



Peak	Analyte	Retention Time (min)	Detection Limit (ppb)
1	Aldicarb Sulfoxide	3.77	0.019
2	Aldicarb Sulfone	4.66	0.041
3	Oxamyl	5.17	0.050
4	Methomyl	6.03	0.031
5	3-Hydroxy Carbofuran	9.83	0.022
6	Aldicarb	11.46	0.022
7	Propoxur	14.35	0.038
8	Carbofuran	14.94	0.028
9	Carbaryl	17.37	0.013
10	1-Naphthol	18.99	0.053
11	Methiocarb	22.02	0.022
12	BDMC*	22.56	0.031

** Internal Standard*

EPA Method 531.2 target analytes.

References

1. Waters Alliance Systems for Carbamate Analysis: 720000126EN
2. Carbamates in Drinking Water: 720000609EN
3. A Fully Automatic Multi-Analyte Quantification Protocol for Carbamates – A Comparison of LC-MS and LC-MS/MS: 720000672EN

4. LC-MS of 52 Carbamates: A Fully Automated Protocol: WA20274

Featured Products

Alliance HPLC <https://www.waters.com/514248>

Empower 3 Chromatography Data Software <https://www.waters.com/waters/en_IN/513188>

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