

### Are you concerned with any of these issues?

- Is your method capable of meeting the precision and accuracy requirements for carbamates mandated by the Environmental Protection Agency in Method 531.2?
- Do you need to detect at even lower legal limits required by the European Commission or the 10X reduction mandated by the Food Quality Protection Act?
- Are you concerned about reporting false positives?
- Are you getting poor recoveries?
- Does your sample analysis time get delayed because you have to re-calibrate your system and change your processing method daily?

As scientists in the food and environmental analysis industries, you have many challenges when it comes to analyzing pesticides and related compounds. These challenges—from achieving optimal sensitivity and compound confirmation to addressing quantitation and regulatory compliance issues—can interfere with how well you do your job, how productive you can be and, more importantly, how you ensure the quality of your results.

Waters® Alliance® System for Carbamate Analysis is a complete solution for carbamate analysis. With the Alliance System for Carbamate Analysis, you will successfully analyze your samples according to government regulations the first time, saving you time and money.



*Waters Alliance® HPLC System for Carbamate Analysis consists of a 2695 Separations Module with column heater assembly, Post-Column Reaction Module, Temperature Control Module, two Reagent Managers, 2475 Scanning Fluorescence Detector, Empower™ Software and Waters Carbamate Analysis Column.*





## Waters Alliance HPLC System: Designed to Change What You Expect

Waters 2695 Separations Module offers superior solvent management by providing four-solvent blending capability, vacuum degassing of eluents, plunger seal wash, and a column heater for robustness and performance across all flow rates.

The solvent management system of the Waters 2695 Separations Module is the first to incorporate a dual piston design, in which each piston is independently software-driven, and a serial flow path. This design compensates for changes in eluent viscosity. Degassing is performed on-line in order to purge the eluent of any dissolved gases. The vacuum degasser enhances the flow precision and accuracy for both isocratic and gradient applications, resulting in more sensitive fluorescence signals. Pistons are continually washed to eliminate buffer salt buildup, extending the lifetime of the seals and minimizing seal leaks that can detract from flow precision. The result is a new standard of performance.

Compared to traditional HPLC methods for carbamates that use linear, binary gradients, Waters new technology delivers an optimized ternary gradient which provides shortened analysis time for increased sample throughput and improved resolution for enhanced quantitative precision. In addition, the substitution of acetonitrile for methanol as recommended in the Alliance system ternary gradient protocol, doubles the detection sensitivity for the last six analytes by reducing methanol-induced fluorescence quenching.

The two-stage, post-column reaction module delivers reproducible on-line derivatization. The independent column heater assembly ensures retention time reproducibility. For optimum sensitivity and excellent resolution, the Alliance HPLC System for Carbamate Analysis easily injects sample volumes up to 1 mL. For example, you can inject up to 1 mL of drinking water to improve sensitivity without pre-concentration. In addition, the Alliance System is configured with five 24-position carousels, for multiple separation methods, multiple operator use, and more convenient sample preparation.

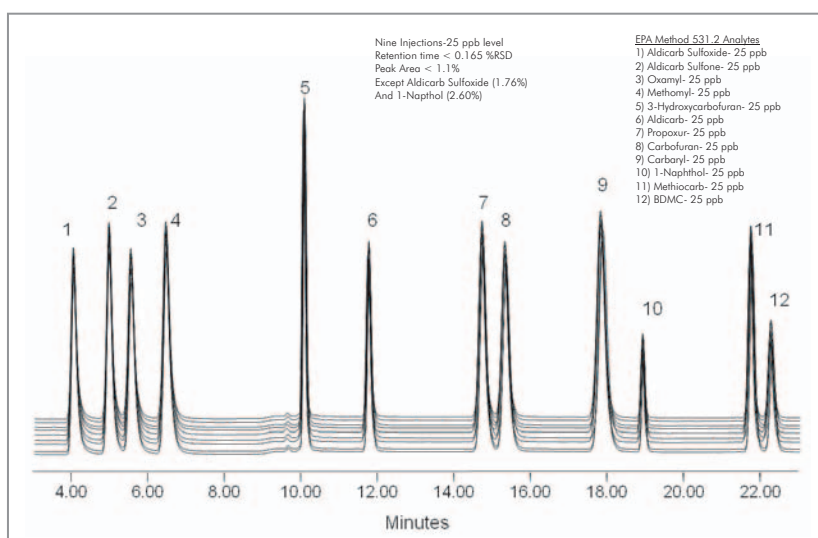


Figure 1. Alliance System for Carbamate Analysis: Reproducibility.



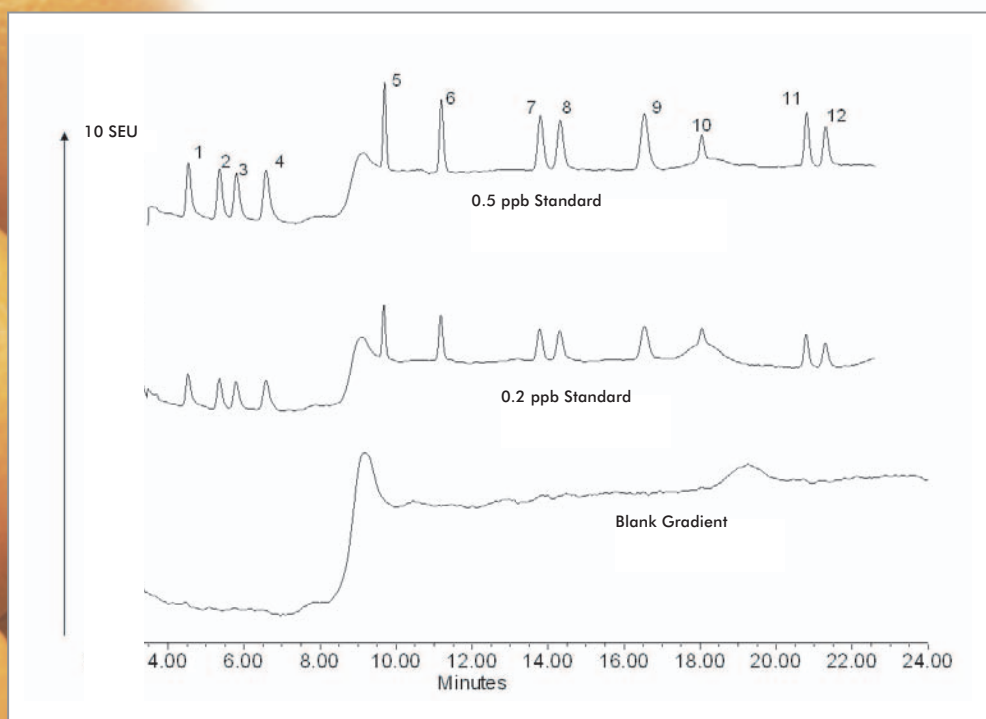


Figure 2. Waters Alliance System for Carbamate Analysis with post column fluorescence detection (PCFD) method shows excellent sensitivity in detecting carbamates at sub-ppb levels.

Peak #	Analyte	Linearity (R <sup>2</sup> )*	Retention Time (Minutes)**	Retention Time Reproducibility (% RSD)**	RT Variability (SD in sec.)
1	Aldicarb Sulfoxide	0.9999	3.77	0.20	0.48
2	Aldicarb Sulfone	0.9999	4.68	0.23	0.66
3	Oxamyl	0.9999	5.16	0.20	0.60
4	Methomyl	0.9999	6.04	0.16	0.54
5	3-Hydroxy Carbofuran	0.9999	9.83	0.07	0.42
6	Aldicarb	0.9999	11.45	0.06	0.42
7	Propoxur	0.9999	14.31	0.04	0.36
8	Carbofuran	0.9999	14.89	0.04	0.36
9	Carbaryl	0.9999	17.33	0.04	0.42
10	1-Naphthol	0.9999	18.99	0.04	0.42
11	Methiocarb	0.9999	22.01	0.02	0.30
12	BDMC	0.9999	22.56	0.02	0.30

Table 2. Typical Linearity and Retention Time Reproducibility in Preserved Reagent Water.

**Conditions:**

\* Linearity: Peak area vs ng on column R<sup>2</sup> = correlation coefficient for least squares variance analysis; 400  $\mu$ L injection volume, 2 to 20 ng on column

\*\* Retention Time Study: Average of 3 replicates; 400  $\mu$ L injection volume; 25 ppb analyte concentrations



Fluorescence Detection

Also included in this system is Waters 2475 Fluorescence Detector that provides superior sensitivity and versatile programmability. Greater sensitivity can bring many advantages to HPLC, including less sample preparation, impurity measurement that was previously undetected, and more accurate and precise results because of improved integration.



Waters 2475 Multi-λ Fluorescence Detector.

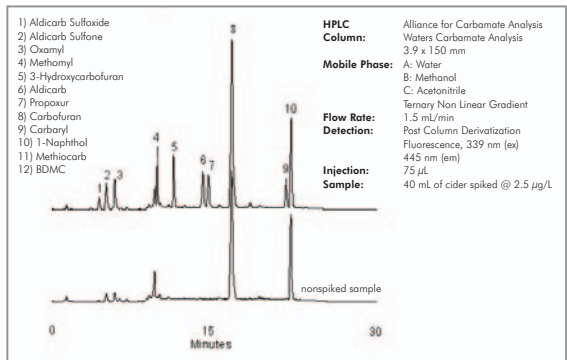


Figure 3. LC analysis of carbamates in apple cider.

2.5 µg/L 5 replicates			
Compound	% Recovery	%RSD	Blank Corrected
1) Aldicarb Sulfoxide	not recovered		
2) Aldicarb Sulfone	33.6	(14)	30
3) Oxamyl	120	(15)	80
4) Methomyl	82.6	(9.9)	60
5) 3-Hydroxycarbofuran	142	(4.9)	120
6) Aldicarb	116	(1.7)	110
7) Propoxur	116	(4.2)	110
8) Carbofuran	112	(4.2)	110
9) Carbaryl	582		140
10) Methiocarb	126	(7.6)	120

Figure 4. Results, carbamates in apple cider.

Real World Samples

The results shown in these tables were made possible with Waters Oasis® HLB sample extraction products. Oasis products provide the speed, superior results and simplicity you’re looking for—whether you’re analyzing water samples, soil or food extracts. The Alliance HPLC System for Carbamate Analysis, combined with Oasis HLB extraction cartridges, exceeds AOAC performance requirements for food analysis set forth in AOAC Official Method 985.233.



## Monitoring Carbamate Pesticides in Drinking Water

Carbamates are used worldwide as commercial pesticides on food crops. The resulting agricultural runoff can carry them into water-bearing rock formations, surface water and other drinking water resources. The United States Environmental Protection Agency (US EPA) requires that drinking water and raw source water be monitored for the presence of carbamate pesticides and related compounds using an established EPA-approved method, EPA Method 531.2. The European Union (EU) regulation, EC Directive 98/83/EC, regarding drinking water, gives a general rule for pesticides and metabolites. This regulation limits the maximum admissible concentration (MAC) at 0.1 µg/L for each individual component, total concentration 0.5 µg/L maximum. 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.

### Waters Alliance System for Carbamate Analysis

#### 2695 Separations Module

- 120-Sample capacity
- Low dispersion
- Temperature control
- Transferability of methods
- Compact, small footprint

#### Waters Post-Column Reaction System

Post-Column Reaction Module, Temperature Control Module and two Reagent Managers

- An optimized, two-stage, post-column reactor oven for online derivatization, which incorporates a proprietary reaction coil and patented countercurrent heat exchanger to minimize bandspreading and increase sensitivity

#### Waters Carbamate Analysis Column

- Designed and tested for high sensitivity and excellent resolution
- Specified as the primary column for EPA Method 531.2

#### 2475 Scanning Fluorescence Detector

- Superior signal-to-noise performance for sub-ppb sensitivity
- Fast methods development: built-in excitation and emission wavelength scanning accelerate and optimize methods development

#### Empower Data Management Software

- User interface that's easy to learn and use
- On-line help
- Fully scalable architecture
- Unique integrated database

#### Methods Guide

- Comprehensive method manual for Waters Alliance HPLC System for Carbamate Analysis

Peak Number/Analyte	EPA Method Detection Limit
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1) Aldicarb Sulfoxide	0.038 ppb
2) Aldicarb Sulfone	0.033 ppb
3) Oxamyl	0.044 ppb
4) Methomyl	0.054 ppb
5) 3-Hydroxycarbofuran	0.038 ppb
6) Aldicarb	0.049 ppb
7) Propoxur	0.061 ppb
8) Carbofuran	0.050 ppb
9) Carbaryl	0.043 ppb
10) 1-Naphthol	0.115 ppb
11) Methiocarb	0.055 ppb
12) BDMC	-

\*EPA method detection limits as outlined in the Environmental Protection Agency Federal Register, 40 CFR Ch. 1 (7-1-92 Edition) Appendix B to part 136-Definition and Procedure for the Determination of the Method Detection Limit-Revision 1.1 A spike level of 0.2 ppb in preserved reagent water was used. Calculated data determined from 7 replicates.

Table 1. EPA Method 531.2 detection limits, performed on a Waters Alliance System for Carbamate Analysis.

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# Waters

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