

Waters

Lab Highlights

Carbamate Analysis via EPA Method 531.1: Latest Revision 3.0 Specifies Waters™ Column

A significant change in the specification for an analytical column in EPA Method 531.1 *had* to be made last year. The sequence of events is illustrated below.

EPA Carbamate Separation

Figure 1a. "Before":
Typical performance of
Beckman Ultrasphere™ column
in early months of
National Pesticide Survey using
NPS Method 5.

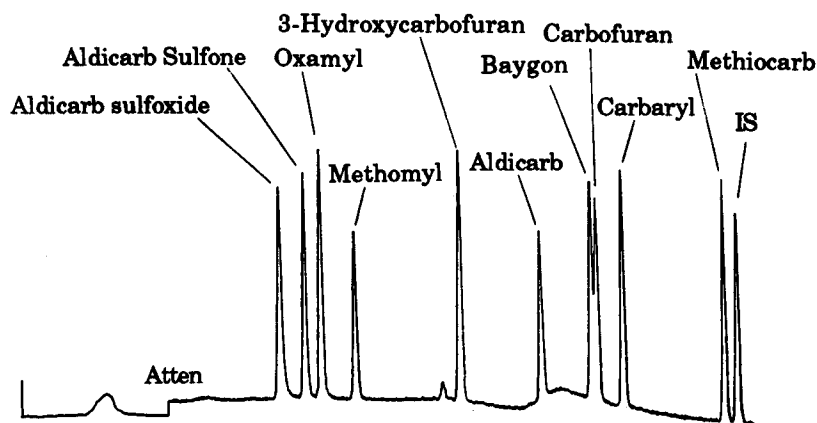


Figure 1b. "Problem":
Same separation as above on new
Ultrasphere column packed with
"improved" lot of material.

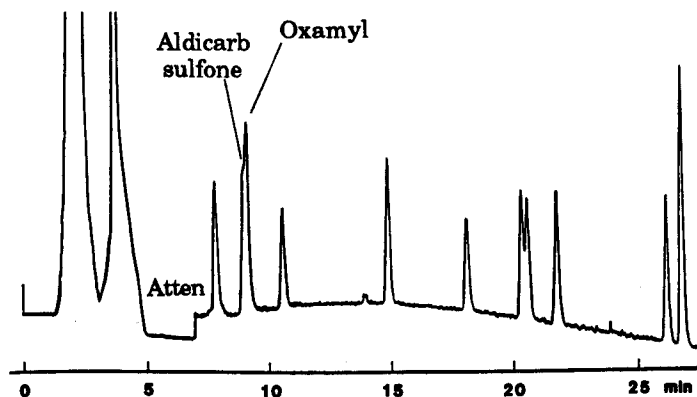
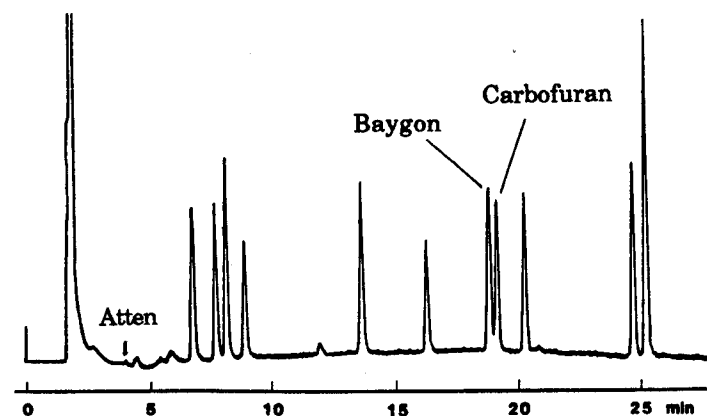


Figure 1c. "Solution":
Method 531.1, Rev. 3.0,
carbamate separation using
Waters Nova-Pak® C₁₈ Column.
Conditions: MeOH/water binary
gradient (1 mL/min; 10% MeOH
initial; hold 2 min; then 25 min
linear ramp to 80% MeOH)



*Chromatograms in Figures 1a,b,c
courtesy of R. Kent Sorrell,
USEPA, Office of Drinking Water,
Cincinnati, OH.*

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The National Pesticide Survey was undertaken by the EPA in April 1985 to learn about the magnitude of the problem of pesticide contamination in ground water. Battelle Columbus was contracted by the NPS to develop Method 5 for the determination of carbamate pesticides and related compounds via direct injection HPLC with post-column derivatization and fluorescence detection.¹ This method was tested, revised, and reissued by the EPA as Draft Method 531.1 in 1988.

After several months of evaluation, method performance had been deemed good; a sample chromatogram is shown in Figure 1a. Then, just about the time the method was to be published in final form as 531.1, Rev. 3.0², and cited in the Federal Register, the unexpected happened. When a new Ultrasphere ODS column (Beckman) was installed, it gave the separation in Figure 1b. Aldicarb sulfone and oxamyl, two critical early eluting peaks, could no longer be resolved. The problem was traced to Beckman's column manufacturing process, wherein the type of silica substrate had been changed as part of a "quality improvement" program.

The search to find a replacement column was described by Kent Sorrell at the recent Water Quality Technology Conference.³ Columns from Pickering (Carbamate Analysis column), Supelco (Supelcosil™ LC-18), and Axxiom™ (ODS) were deemed unsuitable. Accusphere™ ODS (J&W) provided a separation nearly equal to the original Ultrasphere column, but it was surpassed in performance by Waters Nova-Pak C₁₈ column as shown in Figure 1c. Baseline separation of aldicarb sulfone and oxamyl was obtained, and, in addition to a shorter elution time for the internal standard, resolution of baygon (propoxur)/carbofuran increased from R = 0.58 (Ultrasphere) to R = 1.06 (Nova-Pak).

Those chromatographers who choose to adhere to the EPA method can obtain the recommended analytical column in Method 531.1, Rev. 3.0, from Waters in an application-tested form as **Waters Carbamate Analysis Column**, 3.9 mm x 15 cm, P/N 35577.

However, for superior separation, loadability, recovery, reproducibility, and detection limits, **Waters Carbamate Analysis System** is recommended.⁴ This guaranteed system includes an optimum method which delivers baseline separation of all analytes, accomodates sample injection sizes of 1 mL for drinking water analysis, and provides two sample-per-hour throughput. **Waters Carbamate Analysis Method** easily meets or exceeds all the capability and equivalency requirements set forth in Section 10.3/10.4 of EPA Method 531.1.

NOTE: *Method 531.1 differs significantly from Waters Carbamate Analysis Method. In Method 531.1, baseline separation of all analytes is not achieved, and analysis time is longer. The major improvements embodied in Waters Carbamate Analysis Method will be described in future Lab Highlights.*

References:

1. T. Engels, National Pesticide Survey, *Method 5*, USEPA (1987).
2. R.L. Graves, *Method 531.1, Rev. 3.0*, USEPA (1989).
3. R.K. Sorrell and M.K. Zuiker, Poster paper P-5 presented at WQTC '89, Philadelphia, PA, November 1989.
4. P.D. McDonald, W.P. Leveille, A.E. Sims, W.J. Wildman, V.R. Zener, A.D. Scarchilli, WQTC '89 Proceedings Volume, American Water Works Association, in press, 1990.
