DIONEX

Application Brief 115

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Determination of *N*-Methylcarbamates

INTRODUCTION

The *N*-methylcarbamates and *N*-methylcarbamoyloximes are among the most widely used pesticides in agriculture. Reversed-phase high-performance liquid chromatography (RP-HPLC) is the preferred method for separating them; however, HPLC with UV detection does not offer the sensitivity or specificity required for the sample matrices of interest. Therefore, postcolumn derivatization followed by fluorescence detection is used. Accordingly, U.S. Environmental Protection Agency (EPA) Method 531.2 provides guidelines for monitoring these compounds in ground and surface waters,[1] and Dionex has published a detailed method[2] that is consistent with the EPA method.

The work shown here describes a faster and more sensitive method for determining the carbamates specified in Method 531.2. The separation was performed on an Acclaim® Carbamate column, which is designed for separation of these carbamates, and the detection was performed on the FLD-3400RS fluorescence detector, which provides maximum stray light suppression for higher detection sensitivity.

Figure 1 shows the separation of a mixture of 11 carbamate pesticides and 1 internal standard (I.S.) described in EPA Method 531.2. There is baseline separation of the 12 compounds (resolutions [R_s] ≥ 1.5) and the separation is completed within 20 min. This is a faster method than those previously reported,[1, 2] due to use of an UltiMate® Rapid Separation Liquid Chromatography (RSLC) system and smaller size column.

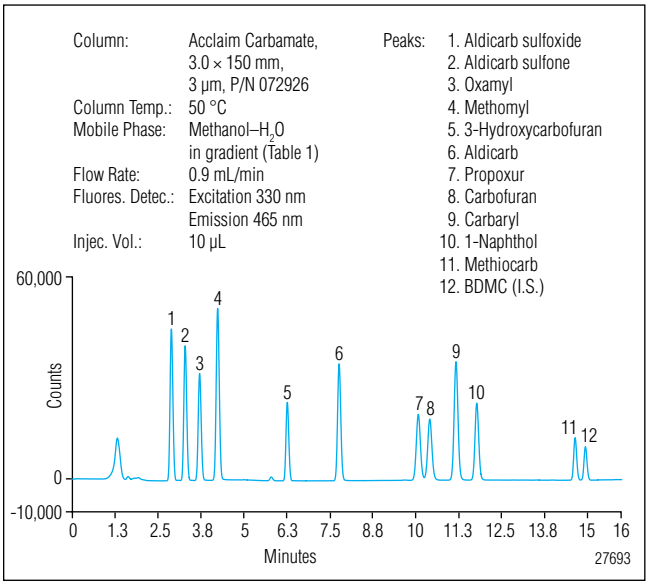

Figure 1. Chromatogram of 11 carbamate standards and an internal standard with a concentration of 35 µg/L each.

Table 1. Gradient for the Separation of Carbamates

Time (min)	Flow Rate (mL/min)	Methanol (%)	H_2O (%)	Curve
-4	0.9	14	86	5
0		14	86	
2		20	80	
8		40	60	1
13.6		70	30	5
16		70	30	

Figure 2 shows a chromatogram of a mixture of standards (0.2 µg/L each) with an injection volume of 100 µL. The EPA method requires 400 µL and the Dionex published method[2] requires 250 µL. This shows that the combination of the RSLC system and FLD-3400RS fluorescence detector provides higher method sensitivity, thus meeting the goals of the EPA method with significant time and solvent savings.

EQUIPMENT

Dionex UltiMate 3000 RSLC system including:

DGP 3600RS Pump

WPS 3000RS Autosampler

TCC-3000RS Thermostatted Column Compartment

FLD-3400RS Fluorescence Detector

Chromeleon® Chromatography Data System (CDS) software, version 6.80 SR9

Pickering PCX 5200 Derivatization Instrument (Pickering Laboratories, Inc.)

The procedure and solvents of postcolumn derivatization are the same as those specified in U.S. EPA Method 531.2.

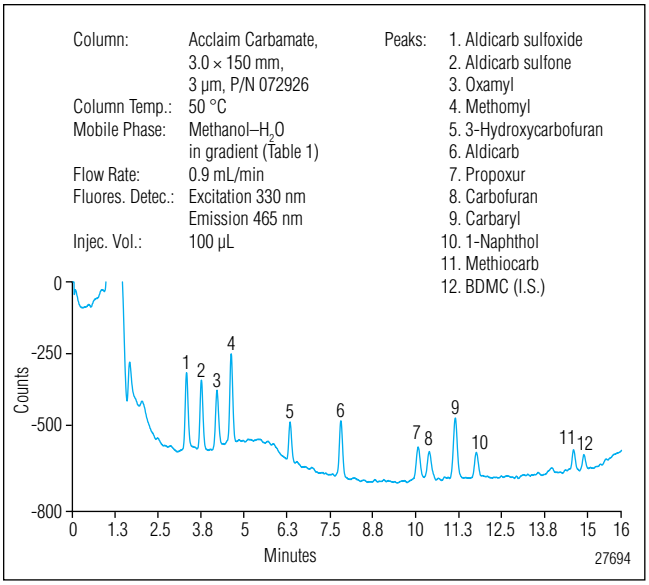

Figure 2. Chromatogram of 12 carbamate standards with a concentration of 0.2 µg/L each.

REFERENCES

1. *Measurement of* N-*Methylcarbamoyloximes and* N-*Methylcarbamates in Water by Direct Aqueous Injection HPLC with Postcolumn Derivatization*; U.S. EPA Method 531.2; Revision 1.0; Environmental Protection Agency: Cincinnati, OH, 2001.
2. Dionex Corporation, *Determination of N-Methylcarbamates by Reversed-Phase HPLC*. Application Note 96, LPN 1935, 2007, Sunnyvale, CA.